Concrete for a Sustainable Agriculture

Proceedings of the 7th International Symposium

September 18-21st 2011
Québec City, Canada

Edited by:
Stéphane Godbout
Organizing Committee Chair
Regional director, CSBE
and
Lise Potvin, Vice-Chair

September 2011

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Institut de recherche et de développement en agroenvironnement

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Message du maire de Québec

Québec est fière d'accueillir le 7e Symposium international sur le béton, matériau de base pour une agriculture durable, de la Commission internationale du génie rural! C'est avec un grand plaisir que je souhaite à tous les participants la plus cordiale des bienvenues.

S'il est un domaine où recherche et développement durable vont de pair, c'est bien celui de l'agriculture! À l'heure où les questions d'ordre environnemental et économique exigent innovation et savoir-faire de la part des intervenants, cette rencontre porte la promesse d'échanges enrichissants qui serviront l'avancement des connaissances et l'efficacité des pratiques.

Avec son riche patrimoine, ses quartiers animés et sa tradition d'hospitalité, Québec constitue le lieu idéal pour joindre l'utile à l'agréable. Entre fleuve et montagnes, la ville vous accueille, parée de ses plus belles couleurs. Je vous invite à en découvrir les nombreux attraits, où l'art, la culture, le plein air et la gastronomie occupent une place de choix.

Félicitations aux organisateurs de l'événement!

À tous et à toutes, je souhaite un excellent séjour parmi nous!

Le maire de Québec,

Régis Labeaume

Québec History and Heritage

Here the History is Beautiful
Many tourist-friendly destinations beckon to the imagination by appealing to one's sense of wonder and discovery, but few are as innately captivating as Québec City. This fortified city on the bank of the St. Lawrence River offers a perfect balance between things to see and do.

Geography
Mother Nature has been generous to Québec City. The St. Lawrence River flows past Montréal, becoming narrow as it passes between the cliffs on which are perched Québec City and Lévis before continuing, broad and mighty, on its journey to the Altantic Ocean. The geographical diverse landscape enhances the region's beauty. Nestled in the heart of the St. Lawrence Valley, Québec City is divided into two distinct areas: Upper Town, which is perched on a promontory overlooking the river, and Lower Town, which lies on the riverbank where the city's founders lived long ago

Old Québec
There is an abundance of attractions in Québec City. Its most famous district, Old Québec, is the cradle of French civilization in North America. The winding streets and bustling public squares date back over 400 years to the founding of the city by French explorer Samuel de Champlain.

Today, Old Québec is famous the world over for its European charm and well-preserved architectural treasures. Delightful shops and cafés line the winding streets, where horse-drawn carriages clip clop steadily along, street performers entertain passersby and people out for a stroll admire the view from the promenade running along the top of the cliff.

The entire district, which is best explored on foot, is a living history book, and every garden, building and street corner is its own chapter. In 1985, UNESCO recognized the significant historical value of Old Québec by designating it a World Heritage Site.

Centuries of Human Contact
The Québec City region has been a point of contact between different peoples and nations from around the world for hundreds of years:

- First Nations have always been present in the Greater Québec Area. They were the first to occupy the site prior to encountering the Europeans, whose arrival transformed the continent and the Native way of life.
- France, the first source of European immigrants to Québec and the country of origin for the founders of Québec City and New France.
- The British Isles became the new political masters of Québec City in 1763. Many immigrants travelled from the British Isles to settle across North America, with a number of Irish immigrants choosing to live in the Greater Québec City area.
- The United States, our neighbours to the south, were first our rivals, then our allies and are now essential partners for the economic vitality of Québec City, the province of Québec and Canada.

The 1600s: for Millennia, a Crossroads for Humanity

For thousands of years, First Nations had been visiting this site, which was known as "Kébec", an Native word meaning "the narrowing of the river". They were drawn by the site's natural resources and strategic importance, as it is located at the confluence of several major rivers. Many rich and distinct cultures benefited from the trade networks and political alliances established with groups living deep in the heart of the continent. When Jacques Cartier sailed up the river in 1535, he came upon the Iroquois village of Stadacona, a community of farmers and fishers, near Cape Diamant. A century later, Algonquin nomads were camped at the site of what is today Québec City. The reserve for the Huron-Wendat nation is known as the village of Wendake and is located within the city limits. A must for visitors interested in traditional Native cuisine and culture.

The Founding of the City

When Samuel de Champlain disembarked at the foot of Cape Diamant on July 3, 1608, the village described by Cartier had disappeared. Based on an alliance he established with the Algonquin in 1603, the explorer had free reign to build a small fort, which he called an "abitation". This group of buildings surrounded by a palisade formed the core of a small village that slowly expanded over the following decades, at roughly the same time as British pioneers were settling in Virginia. By the mid-seventeenth century, Québec City's population was only a few hundred souls. The settlement only started to flourish in 1663, when the king of France intervened and appointed Jean Talon as the colony's intendant. By the end of the French Regime, Québec City, the economic, religious, military and political hub of New France, had over 7,000 inhabitants.

The 1700s: the changing of the Crown

On September 13, 1759, British and French forces clashed in the Battle of the Plains of Abraham, with the British emerging victorious. This battle brought an end to the siege of Québec City. The site of the conflict is now a city park. New France surrendered to the British troops the following year. With the signing of the Treaty of Paris in 1763, the colony was ceded to the British monarch. Québec City thus became the capital of the Province of Québec and a major port of entry into British North America. To define relations between the French Canadian catholic population and the British protestant leaders, a political framework was developed and enshrined in the Québec Act of 1774, which was later replaced by the Constitutional Act of 1791. The latter authorized the creation of the first legislative assembly in a British colony. The current fortifications surrounding the Old City were constructed during the British Regime as protection against an American invasion. Today, Québec City is the only fortified city north of Mexico.

The 1800s: Québec City, Port of Entry into North America

Québec City was booming in the early nineteenth century, thanks to the shipbuilding industry, maritime trade and immigration. The city population grew five times larger in less than fifty years (from 8,968 in 1805 to 45,940 in 1851), making it the third largest port in North America, after New York and New Orleans. Starting in the 1830s, approximately 30,000 immigrants disembarked in Québec City annually before continuing their journey to elsewhere in Canada or to the United States.
The Confederation of Canada was established in 1867. Québec City was named the provincial capital and gradually evolved into an important commercial and cultural hub. Unfortunately, the city's new status could not prevent the economic decline resulting from the drop in wood exports and the slowdown of the naval construction industry, nor counter the void left by the permanent withdrawal of the British garrison from the Citadel in 1871. During this period, several stately buildings were constructed, including the Parliament Building, City Hall and the Château Frontenac. Note that Québec is the only predominantly French-speaking Canadian province.

The 1900s: now Four Cnturies
The turn of the twentieth century heralded an economic upswing. The industrial and manufacturing sectors flourished in Lower Town. Québec City was becoming cosmopolitan, boasting tramways, major department stores and expanding outlying communities. The rapid growth of the civil service in the 1960s contributed in large part to the transformation of the provincial capital. Québec City, as it appears today, began take shape: a government town with a sizable service sector, that is R&D-intensive and whose cultural scene continues to draw visitors from around the world.

The Capital and the World
In 1908, Québec City celebrated its three-hundred-year anniversary with historical re-enactments and parades in which 18,000 British, French, American and Canadian soldiers marched as friends at the same spot where, years earlier, they had been mortal enemies. These allies met again in the city in 1943 and 1944, when American President Franklin D. Roosevelt and British Prime Minister Winston Churchill participated in the Québec Conferences. In 1945, Food and Agriculture Organization (FAO) of the United Nations was created in the provincial capital. Since then, Québec City has regularly hosted major international meetings, such as two Sommets de la Francophonie (once in 1987 and again in October 2008), as well as the Summit of the Americas in 2001.

Now We are Turned to the Future
With a view to acknowledging the past and preparing for the future, Québec City commemorated the turning moments of its history during its 400th anniversary celebrations in 2008: the evolution of its population, culture, economy, politics and urban life. Motivated by the year-long celebrations, the entire region is now committed to ensuring its future will be as glorious as its past

http://www.quebecregion.com/en/media/tools_resources/article/history_heritage_article?a=med

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Preface Dr. Stéphane Godbout

Agriculture and particularly the livestock production are changing rapidly in today’s world. Since few years, the impact of animal production on the environment has become a matter of concern. During the past ten years, a lot of complaints have been reported about odour and animal welfare. In the current scenario, people are also concerned by the countryside and patrimonial value of the agricultural building (architectural landscape). Based on this fact, not only the gas and odour emissions or animal welfare will be a concern, but also the building appearance and design will play a key role for the development of agriculture.

The concrete is already largely used in the building construction and its characteristics give to this material a huge potential to be a part of the future. The different new cements and the approaches and technologies presently available like nanotechnologies brings newer opportunities to create specific agricultural and ecofriendly concrete that are more sustainable and ecological.

The seventh edition of the “Concrete for a sustainable agriculture Symposium” will be a very amazing opportunity to find a technique for making concrete better and more ecological. The program of the symposium is well related to the people’s concerns such as:

- Concrete deterioration and durability;
- Eco-design;
- Life cycle;
- Animal welfare.

This symposium will also offer the possibility to exchange knowledge and practical experiences about the use and role of the concrete in agriculture. We hope that this symposium will give us useful scientific information and give us an opportunity to build a relationship and networking.

We don’t have to forget that agriculture is not just a way to produce. Agriculture is also there to protect the land, occupy the territory and to draw the rural landscape.

Enjoy your stay in Quebec.

Stéphane Godbout, ing., P.Eng., Ph.D. and agr.
Local organizing committee chair
Regional director, CSBE

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Keynote Papers

7th International Symposium on Cement Based Materials for a Sustainable Agriculture (CIGR International Symposium)
Hosted by the Canadian Society for Bioengineering (CSBE/SCGAB)
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CONCRETE FOR FARM BUILDINGS; PRACTICAL ASPECTS AND OPTIMUM USAGE

Yves Choinière, ing., agr., P. Eng., Les Consultants Yves Choinière inc.

Summary of the conference
The engineers involved in the design of concrete structures for farm applications have to select the proper concrete mixtures in function of each application.

In practice, the designer is facing rural realities such as:

- Availability of the selected concrete type knowing that most rural concrete manufacturing plants have limited selection.
- The quality and experience of the agricultural building contractor with regard to concrete pouring and placing, and especially, proper curing.
- The agricultural producer requiring the most economical concrete mix.
- Lack of site inspection to insure the quality of the concrete work.

Immediate failure of concrete elements are rare in agriculture, most problems occur after an extended period of time due to different factors. The main problems encountered are associated with cracks, surface erosion and general deterioration of certain concrete structures. With regard to new and existing farm buildings, silage and manure storages, the main areas of research needed are:

- The development of concrete repair materials such as surface additives, crack fillers, polymeric membrane or other products to reduce the rate of concrete surface deterioration
- The review of the concrete type selection methods in function of the usage. The main problems with on-farm concrete usage are undesired surface cracks. Cracks allow contaminants to penetrate and attack the reinforcing steel and interior concrete.
- Research on fibre reinforced concrete not only for strength but for workability and resistance to cracks.
- Research new concrete type to reduce bacterial contamination, odour and gas production.
- Research in construction methods to maximize the use of pre-manufactured concrete elements and secure higher concrete quality.
- To develop methods for concrete recycling following demolition.

With regard to policy and regulatory aspects, recent events revealed that site inspection and quality control is crucial in order to secure that contractors respect the designer's specifications.
For future farm buildings, there will be a more intensive usage of concrete for the construction of walls, ceiling, partitions and other structural elements. Fire resistance and expected durability are the main selection criteria.

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A MICROBIAL BASED SYSTEM DEVELOPED FOR SELF-SEALING CONCRETE CRACKS

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CSAS11010 –Topic VII.

ABSTRACT Concrete has a relatively low tensile strength which makes it prone to cracking. Without immediate and proper treatment, cracks tend to expand further and eventually require costly repair. Engineering structural elements in such a way, that they would respond to crack formation with an autonomous self-healing action, would therefore be highly desirable. Based on previous experience with microbial induced calcium carbonate precipitation for consolidation of concrete and stone, the use of bacteria as self-healing agent is tested. Diatomaceous earth (DE) was used as the carrier to protect bacteria since the bacterial activity decreases a lot in the high pH concrete environment. First the ureolytic activity of the bacteria after being immobilized in DE, was tested at neutral pH and in a cement suspension which was used to mimic the high pH environment inside concrete. The DE immobilized bacteria were shown to remain active in cement suspension and decompose a significant amount of the urea (60%) that was provided as a nutrient. Un-immobilized bacteria almost lost all ureolytic activity in the cement suspension. By means of light microscopy, it was observed that cracks of width 0.15 mm to 0.17 mm in the specimens added with DE immobilized bacteria, were completely filled by the precipitation. The precipitation was primarily confirmed to be $CaCO_3$ based on the results of Energy Dispersive Spectrum analysis. The addition of diatomaceous earth did not decrease the tensile and compressive strength of the specimens.

Keywords: Bacillus sphaericus, diatomaceous earth, ureolytic activity, cement slurry, microbial $CaCO_3$, crack sealing

INTRODUCTION Crack repair is of crucial importance to concrete since cracks are the main path for the penetration of corrosive substances which greatly decrease the service life of concrete structures. Nowadays, a promising method to repair concrete cracks is being investigated. The idea is that repair work will be done by the concrete itself when cracks appear, through a self-healing process. Cracks will be healed by certain kinds of healing agents released from the concrete when cracking happens. Compared with the commonly used repair method which follows the procedure of detection, monitoring and repair, the self-healing method is cheaper over the structure's life-cycle since the later maintenance would be greatly saved (Neville 1996). Organic polymer (Dry et al., 1994&1996), super-absorbent polymer (Lee et al., 2010), expansive agents (Yamada et al., 2007) and so on are being investigated as self-healing materials for cracks. Another alternative self-healing material is microbial carbonation precipitation. Some bacteria can produce or induce bio-minerals during their growth and metabolism. Under suitable conditions, most bacteria are capable of inducing carbonate precipitation

(Stocks-Fischer et al., 1999; Rodriguez-Navarro et al., 2003; Castanier et al., 1999; Hammes et al., 2003). In our study, an ureolytic strain, *Bacillus sphaericus*, was used. Based on previous research (Dick et al., 2006), it is able to precipitate calcium carbonate ($CaCO_3$) in its micro-environment by the decomposition of urea ($CO(NH_2)_2$) into ammonium (NH_4^+) and carbonate (CO_3^{2-}) (see eq. (1) ~ (3)). The precipitated bio-$CaCO_3$ has a good potential to be used to heal concrete cracks because it is natural, environmentally friendly and compatible with the concrete matrix. The bacteria should be added into concrete during the process of mixing. When cracks appear, bacteria inside concrete would be activated to precipitate $CaCO_3$ to fill the cracks. However, bacterial cells cannot be added into concrete directly because the activity decreases a lot in the harsh environment of concrete (mainly due to the high pH of cement). Besides that, bacterial cells might be destroyed during the process of hydration because the micro-pores become smaller and smaller during hydration, and this might squeeze the bacteria inside the pores (Jonkers et al., 2010). In this work, diatomaceous earth (DE) was used as a carrier for the bacteria to protect them from high pH environment.

$$CO\left(NH_2\right)_2 + 2H_2O \xrightarrow{Bacteria} CO_3^{2-} + 2NH_4^+ \quad (1)$$

$$Ca^{2+} + Cell \rightarrow Cell - Ca^{2+} \quad (2)$$

$$Cell - Ca^{2+} + CO_3^{2-} \rightarrow Cell - CaCO_3 \downarrow \quad (3)$$

The aim of the experiments was to examine the protective effect of DE. The experiment was first performed in a mimic high pH concrete environment (a cement suspension, 20g/L). Then the DE immobilized bacteria were added to mortar specimens to evaluate the self-healing efficiency by microscopy analysis.

Material and Methods

Bacterial strain *Bacillus sphaericus* LMG 22557 (Belgian coordinated collection of microorganisms, Ghent) was used based on previous research. This strain has a high urease activity (40mM urea hydrolyzed $OD^{-1}.h^{-1}$) and can produce $CaCO_3$ in a controlled way (De Muynck et al., 2010). The medium used to grow *Bacillus sphaericus* was based on yeast extract and urea. The yeast extract medium was first autoclaved for 20 min at 120°C and then added to the filter sterilized urea solution. The final concentrations of yeast extract and urea were 20g/L. Cultures were incubated at 28°C on a shaker at 120 rpm for 24 h. Bacterial cells were harvested by centrifuging the 24 h-old grown culture and were resuspended in saline solution (NaCl, 8.5 g/L), represented as bacterial suspension (BS, 10^9cells/mL).

Diatomaceous earth DE particles (Dicalite Europe NV, Belgium) in the study had different kinds of irregular shapes and the particle size distribution ranged from 4μm to 20μm. There were a large number of pores on the surface. The size of these surface pores was around 0.1μm to 0.5μm. Therefore, DE had a high specific surface area of 29 m^2/g determined by Nitrogen gas adsorption (BELSPORP-Mini II).

Ureolytic activity of DE immobilized bacteria

Immobilization of bacteria to DE DE was added to the bacterial suspension in a falcon tube. The concentration of DE was 20% (w/v, weight of DE/volume of BS). Then the falcon tube was placed on a shaker (100rpm, 28˚C) for 1 hour or 4 hours.

Ureolytic activity of DE immobilized bacteria The ureolytic activity of the bacteria was indicated by the amount of urea decomposed by bacteria in the urea medium, which was composed of urea (20g/L) and yeast extract (20g/L). The ureolytic activity was examined in neutral pH and high pH environment.

A neutral pH was obtained by adjusting the pH of the urea medium to 7.0 by use of a 1M NaOH solution. To mimic the high pH environment inside concrete, cement powder was added to the urea medium (Ordinary Portland Cement CEM I 52.5N, 20g/L). The mixture was referred to as "cement suspension". The cement suspension was put on the shaker (100rpm) for about 1 day till the pH reached a stable value (12.5). Subsequently, the mixture of DE immobilized bacteria in the falcon tube was added to the neutral pH urea medium and the cement suspension. As a reference, the same amount of un-immobilized bacteria (free cells) was also added to the media at the same time. The amount of urea decomposed was determined by the total ammonium nitrogen (TAN) in the urea media. One mole of urea ($CO(NH_2)_2$) produces 2 moles of NH_4^+; the amount of NH_4^+ can hence indicate the amount of urea decomposed. TAN concentrations were measured calorimetrically by the method of Nessler (Ivanov et al., 2005). Triplicates were used in the experiment.

Self-healing of mortar specimens by means of DE immobilized bacteria

Mortar specimens Four series of mortar specimens (40mm x 40mm x 160mm) were made with a water to cement ratio of 0.5 and a sand to cement ratio of 3. The cement used was the same as that used for making cement suspension. The sand was normal sand (CEN-NORMSAND DIN EN 196-1). The components of the specimens in each series are shown in Table 1.

Table 1. Mortar specimens in each series

Series	Cement (g)	Sand (g)	Water (g)	DE (g)	Nutrients (g)	BS (mL)
1 (R)	450	1350	225	0	0	0
2 (DE5)	450	1350	225	22.5	0	0
3 (DE5N)	450	1350	225	22.5	37.375	0
4 (DE5BN)	450	1350	112.5	22.5	37.375	112.5

The nutrients were made of 1.125g yeast extract, 11.25g urea and 25g $Ca(NO_3)_2$ and the total weight was 37.375g.

The amount of materials in Table 1 was for 3 prisms. In each series, 9 prisms were made. Three of them were made without reinforcements, which were subjected to the strength test after 28 days based on the European standard EN 196-1. Six of them were added with reinforcements to make realistic cracks in specimens. The specimens with reinforcements were made as follows: a 10 mm mortar layer was first brought into the moulds, which was then compacted by means of vibration. Two reinforcement bars (D=2mm, L=140mm) were then placed on top of it. Afterwards, the moulds were completely filled with mortar and vibrated (Wang et al., 2010). All moulds were put in an air-conditioned room with a temperature of 20°C and a relative humidity of more than 90% for 24h. The mortar specimens were placed in the same air-conditioned room (curing room) after demoulding.

<u>Creation of realistic cracks</u> After 14 days, prisms with reinforcements inside (6 in each series) were taken out from the curing room and were subject to a three-point bending test for the aim of making realistic cracks. A linear variable differential transformer (LVDT) was first attached to the bottom of the specimens by use of a fast drying glue (Schnellklebstoff X 60, HBM) to measure the crack width. The crack width was increased with a velocity of 0.0005 mm/sec until a crack of 0.3 mm was obtained. Subsequently, the load was released and the remaining crack width was about 0.15mm to 0.17mm. Afterwards, the cracked specimens were immersed into different media. Three of them were immersed into water and three were immersed into the deposition medium (made of urea and $Ca(NO_3)_2$, 0.2M) for 40 days.

Healing efficiency The crack healing in different series of specimens were observed under a light microscopy (Leica S8AP0) which was connected with a camera (Leica DFC295), which was used to take photographs of the cracks under the specific magnification. The materials formed in cracks were also characterized for its morphology and composition by use of Scanning Electron Microscope (FEI QUANTA 200F SEM) and Energy Dispersive Spectrometer (EDAX, America). Before examination, samples were completely dried in the oven at 40 ˚C for 2 days and were gold coated by a Baltec SCD030 Sputter Coater.

Results and Discussion

Ureolytic activity of DE immobilized bacteria at different pH As shown in Fig.1, no urea was decomposed when no bacteria were added to the urea media. At neutral pH, both un-immobilized (free cells) and DE immobilized bacteria showed a high ureolytic activity (about 95% of urea was decomposed). There was no difference between using free cells or immobilized bacteria. However, when in the high pH cement suspension, the ureolytic activity of un-immobilized bacteria was greatly decreased. The amount of decomposed urea decreased from 95% (19 g/L) to less than 5% (1 g/L). Yet the DE immobilized bacteria kept much higher ureolytic activity than the free cells. Still 60% (12 g/L) of the urea was decomposed. The values of decomposed urea on the 3rd day were slightly lower than those after 1 day. This was due to the volatilization loss caused by the transformation of NH_4^+ to NH_3. It can be seen from Fig.1 that DE has a protective effect for bacteria in the harsh environment. The protective effect was related with the mixing time of BS and DE. As shown in Fig.2, the mixing time had no influence on ureolytic activity of DE immobilized bacteria in neutral pH. However, limited protective effect for bacteria in cement suspension was observed when the mixing time was too short (0h) or too long (4h). The mixing time of 1h was more appropriate.

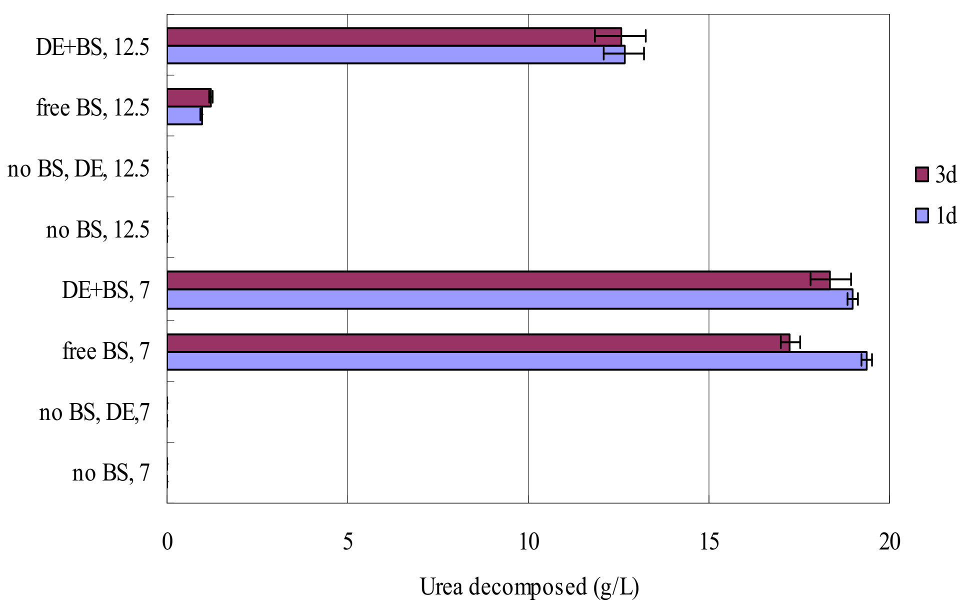
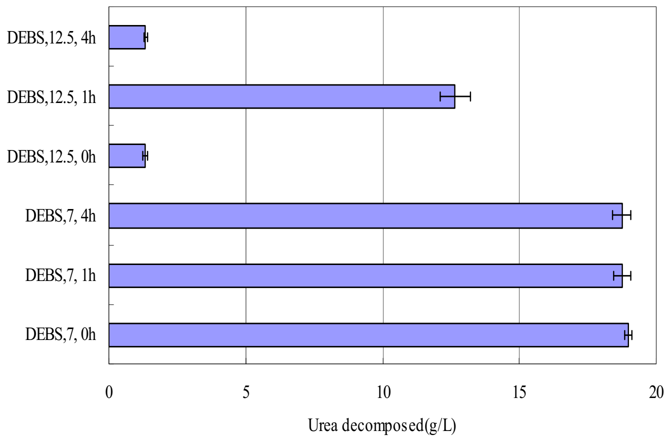


Fig.1 Ureolytic activity of un-immobilized (free BS) and DE immobilized bacteria (DE+BS) in neutral pH (7) and high pH cement suspension (12.5)



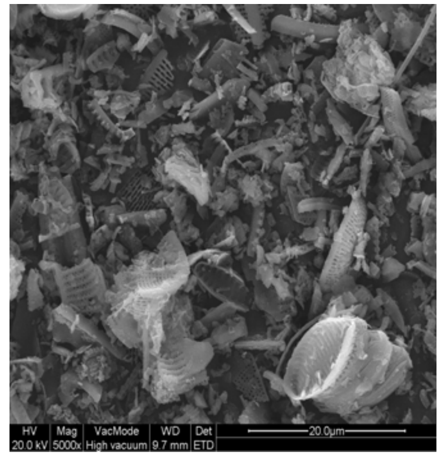


Fig.2 Ureolytic activity of DE immobilized Fig.3 Morphology of DE particles after different mixing time

DE particles have a very porous structure (See Fig.3). The pores are on the surface and have a nano-size. That is the reason why DE has such a high specific surface area. Most of the pores are only 0.1-0.5μm which is not large enough to hold bacterial cells (1-2μm). Therefore, bacterial cells were mainly sorbed on the surface of the particles. If the mixing time is too short, bacterial cells have not enough time to attach to the particle surface; yet if the mixing time is too long, bacteria might release from the surface.

Influence of the biological agents on mechanical properties of mortar specimens It can be seen from Fig.4 (a) that there is no obvious difference in tensile strength of the specimens in all series. The addition of DE, DE with nutrients or DE with nutrients and bacteria did not decrease the tensile strength of the specimens. Similarly, the compressive strength of the specimens was not decreased either after the addition of self-healing agents. DE powders used in the experiments have a high specific surface area and have a strong ability to absorb water. So if the water to cement ratio of all specimens was kept the same, in the early stage of hydration, the real water to cement ratio of the specimens containing DE was lower than 0.5 because some water was absorbed by DE. In the late stage of hydration, water was released from DE particles again to fulfill complete hydration. In fact, DE itself has a potential to be used as an elegant internal medium for water storage and a healing material for repairing micro-cracks by stimulating the hydration of unhydrated cement particles. Therefore the addition of DE should have a positive effect on the specimens. It should be noted that although urea and other nutrients had a little negative effect on the strength of the mortar specimens, this negative effect could be counteracted by the positive effect from DE.

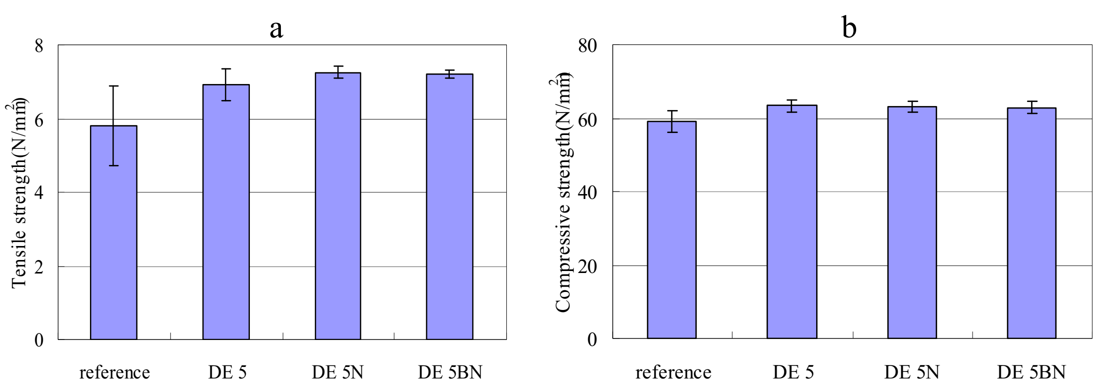


Fig.4 mechanical properties of the mortar specimens in different series

Visualization of crack filling The specimens in the same series had similar crack filling states. Hence, one representative image of each series is shown (Fig.5). Almost no precipitation formed in the cracks of the specimens of series 1 (R), 2 (DE5) and 3 (DE5N) immersed in water. It was noticed that the cracks in the specimens containing DE immobilized bacteria (series DE5BN) were partly filled after the immersion in water (Fig.5 (d)). And much more precipitation was formed in the cracks when the specimens (with DE immobilized bacteria) were immersed in the deposition medium. The cracks were almost completely filled (Fig.5(h)). There were also some white deposits formed in the cracks of the specimens without bacteria when they were immersed in the deposition medium (Fig.5 (e), (f) and (g)). But the amount was quite limited. In view of the crack filling in different kinds of specimens, it can be seen that there was self-healing in the specimens with DE immobilized bacteria. The healing effect was more effective when the specimens were immersed in the medium consisting of urea and Ca^{2+}. Bacteria can not only decompose urea from the crack wall of the specimens (incorporated during mortar mixing), but also use the urea and Ca^{2+} from the deposition medium to produce more calcium carbonate. That's why more precipitation formed and completely filled the cracks in the specimens (with bacteria) immersed in the medium.

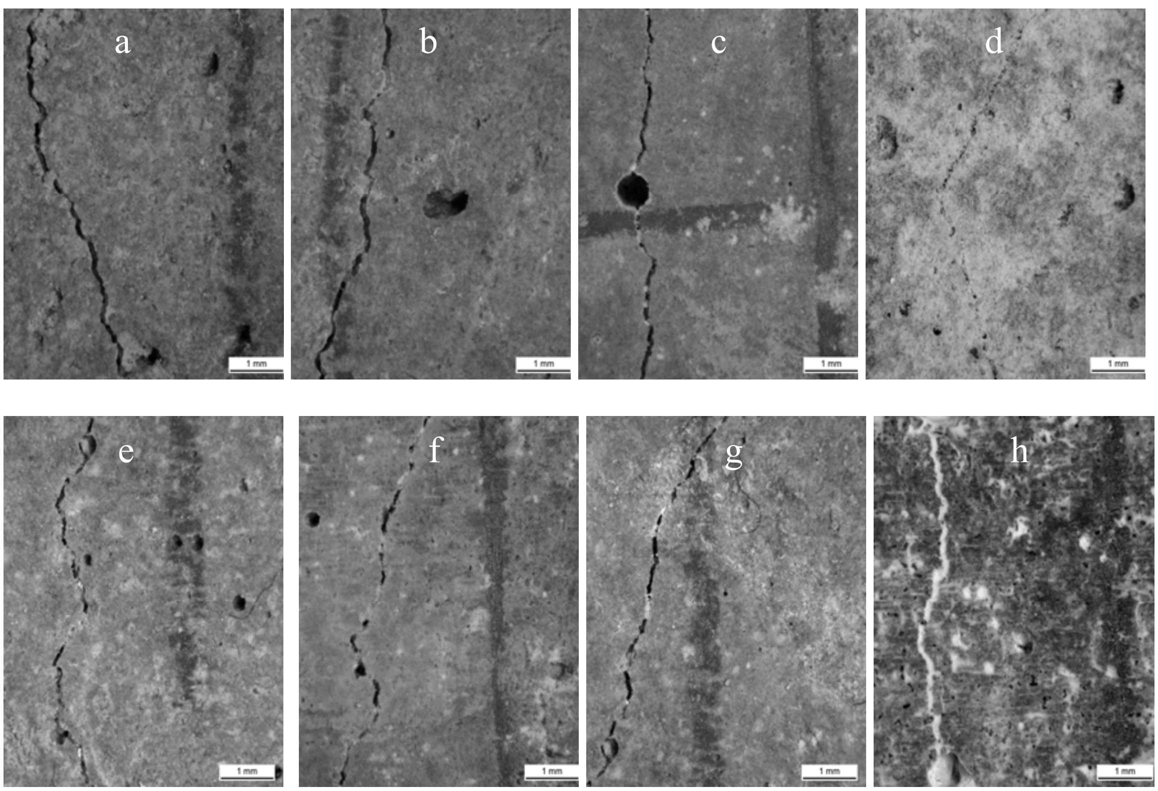


Fig.5 Light microscopy images of cracks in different specimens after 40 days immersion (a, b, c, d: specimens of series 1, 2, 3, and 4 immersed in water; e, f, g, h: specimens of series 1, 2, 3, and 4 immersed in the deposition medium)

SEM analysis of precipitation in the cracks There was no precipitation formed in the cracks of the reference specimens and the specimens only with DE, which corresponded with the microscopy results in Fig.5 (a) and (b). Particles with irregular triangular or rectangular prisms were observed in the specimens with DE immobilized bacteria which were immersed in water (Fig.6 (c) and (d)). The particle size was about 2μm to 10μm. When the specimens were immersed in the deposition medium, the morphology of the precipitation obtained was quite different. Some of the particles showed a hexagonal slab shape with a size of about 30µm (Fig.6 (e)). Some had spherical or flower-shape with a size about 5µm to 10µm. These particles were primarily confirmed to be $CaCO_3$ based on the results from EDS. It was noticed that there was kind of indents in some particles (shown by the white arrows in Fig.6 (c) and (e)), which might be due to the bacterial remains.

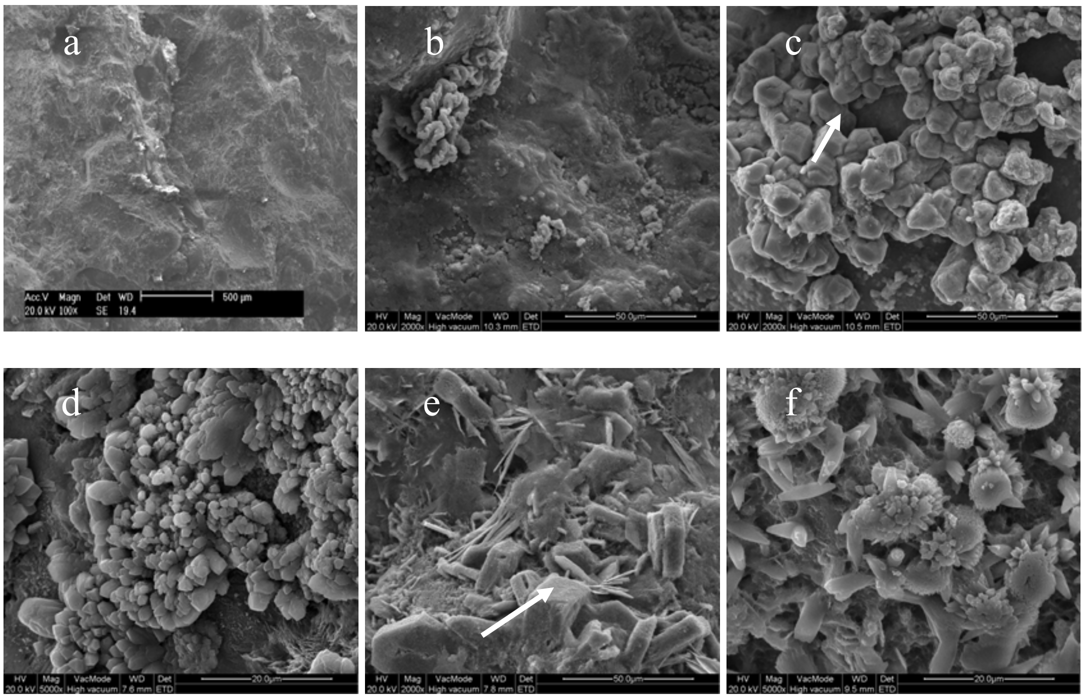


Fig.6 SEM images of the precipitation in the cracks (a, b are taken from the reference specimens and the specimens only with DE immersed in water, respectively; c and d show the specimens with DE immobilized bacteria immersed in water; e and f are from the specimens with DE immobilized bacteria immersed in the deposition medium)

CONCLUSION Diatomaceous earth (DE) was found to have a protective effect for the bacteria in the high pH cement environment. The possible mechanism is that DE particles have a strong capacity to sorb bacterial cells on the surfaces due to their high specific surface area. After sorption, DE provided a kind of micro-environment around the bacteria, in which the local pH was less aggressive than that in the whole cement environment and thus bacteria could still decompose urea. The time for mixing DE and BS should be around 1 hour. Cracks with a width of 0.15-0.17mm in mortar specimens were partly or completely filled by the aid of DE immobilized bacteria depending on the immersion media. The precipitation in the cracks was mainly composed of calcium carbonate.

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DESIGNING THE FARMS OF TOMORROW, LESSONS FROM RESEARCHES ON LANDSCAPE TRANSFORMATIONS AND PERCEPTIONS

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While the protection of the heritage of agriculture is always a challenge and a necessity, contemporary rural and agricultural transformations as for example, new environmental challenges also invite to create the heritage of tomorrow. On the basis on the results of studies conducted in the south of Québec, this presentation seeks to draw lessons to construct the heritage of tomorrow from researches on the temporal dynamics and the perceptions of landscapes. In a first time, taking the measure of the social transformations that have crossed rural areas during the last decade, it is proposed to better understand why create the heritage of tomorrow and what should be the benefits to agriculture. In a second time, on the basis of analysis that reveal the rate of change that have affected agricultural landscapes over time, it shows that landscapes have always been in a process of continuous transformations according to the evolution of values, politics and technological advances. In a third time, the results of studies concerning inhabitant's perception of agricultural landscapes will reveal the most valued and the most controversial elements of these landscapes. Factors that influence the perceptions will also be highlighting. Concerning these factors, it appears that it is first the experience of individual with and within the landscape and the trust in the agricultural practices that play a fundamental role, more than the formal aesthetics of landscapes. Ultimately, the project that invites the landscape to construct the heritage of tomorrow if first of all to guide the future asking the question: what landscape do we want for tomorrow?

Concrete Durability

Section 1

IMPACT OF ACETIC ACID ATTACK ON THE CHEMICAL, PHYSICAL AND MINERALOGICAL EVOLUTION OF CEMENT PASTES

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CSAS11012 – Topic 1: Concrete Deterioration

ABSTRACT Concrete for agricultural construction is often subject to aggressive environmental conditions. This paper aims to analyze the mechanisms of cementitious matrix degradation by acetic acid solution and to identify the cement composition parameters influencing the durability of agricultural concrete. The effect of alumina cement (AC) and of supplementary cementing materials (SCM) is evaluated in order to verify the chemical stability of cement paste in an acetic acid environment and to propose realistic concrete mix design. This study concentrated on six types of hardened cement pastes made with a general use Portland cement (GU), GU blended with CA/silica fume (SF)/low-calcium fly ash (FA)/ground granulated blast furnace slag (GBFS) and metakaolin (MK) at various replacement levels, which were immersed in an acetic acid solution. The degradation mechanisms were investigated using mass loss and altered depth measurements and Electron Microprobe, X-Ray Diffraction and Mercury Porosimetry analysis. The results show that (i) The GU pastes present a very severe alteration. (ii) The cement matrix is completely removed during the immersion of alumina cement pastes. (iii) Silica fume and fly ash did not improve the chemical stability of pastes. (iv) Addition of slag or metakaolin reduces considerably the degradation of cement pastes. In a general way, the alteration of cement pastes subjected to an acetic acid solution is translated to a decalcification of the altered zone, an increase of the porosity and a progressive dissolution of all phases. Acid resistance of cement pastes was considerably improved by incorporation of slag or metakaolin.

Keywords: Acetic Acid, Cement paste, Alumina cement, Supplementary cementing materials, Mechanisms, Electronic microprobe, X-ray diffraction, Porosity.

INTRODUCTION Intensification of farming practice is at the origin of environmental problems directly linked to the excess of effluents such as liquid manure and ensilage effluent (Bertron 2004). Although animal excrement recycling was recognized as a practice that maintains and improves the fertility of soils (Martinez and Le Bosec 2000), current policies aim for storage in water-tight silos, often built in concrete.

The agricultural effluents are quickly transformed under the effect of bacteria into organic acids (acetic, propionic, butyric and iso-butyric acids). The most aggressive acids seem to be those that produce easily soluble calcium salts, such as acetic acid (Biczok 1960, Moskvin et al. 1971, Moskvin et al. 1980). According to Bertron (2004), an acetic acid solution of pH 4 mimics well the aggressiveness of organic acids of liquid manure.

Agricultural effluents constitute a severe chemical threat toward the concrete of agricultural structures. In contact with an acetic acid solution, concrete will undergo an acido-basic reaction leading to the formation of soluble to very soluble salts in water (Kleinlogel 1960, Lange 1985, Zivika and Bajza 2001) and to the leaching of hydrates (Bertron et al. 2005a, Oueslati and Duchesne 2009) resulting in an increase of porosity and permeability of the material, a decrease in mechanical resistance and a corrosion of the reinforcement (Bertron et al.2005b, Zivika 2006, Berke 1989).

The use of alumina cement (AC) and supplementary cementing materials (SCM) can improve concrete properties such as workability, durability and permeability (Baron and Ollivier 1992). The use of SCM such as silica fume and fly ash has been found to improve the resistance of concrete to acid attack because of the reduced presence of calcium hydroxide, which is most vulnerable to acid attack (Mehta 1985).

The aim of the study is to compare the acid durability of blended cement pastes immersed in acetic acid at a pH of 4. Chemical, physical, and mineralogical evolution of blended cements were investigated as well as the microstructure of the samples.

MATERIALS AND METHODS The binders used are formulated from a general use Portland cement (GU) blended with a special high alumina cement (AC) renowned as acid resistant and different supplementary cementing materials. The high alumina cement was used as 60 and 80% replacement by mass of GU cement. Table 1 presents the substitution rate of the GU cement by different types of SCMs and chemical composition of binders are presented in table 2.

Table 1. Substitution rates of GU cement by different SCMs.

SCM	Substitution rate (%) (by mass)
Silica fume (SF)	10 - 15
Type F fly ash (FA)	50 - 70
Ground granulated blast furnace slag (GBFS)	60 - 80
Metakaolin (MK)	15 - 20

Samples preparation and treatment Cement pastes were prepared at water to binder mass ratio of 0.27. Cylindrical samples (50 mm in diameter by 100 mm height) were demolded 24h after pouring and stored in wet room at 23°C and 100% RH for 27 days. Test samples were then immersed at a solid/liquid volume ratio of 1/15 for 3 months in 0.5M acetic acid solution at a pH of 4 adjusted using concentrated NaOH solution. To maintain the pH of the solution at a value of 4, the solution was renewed on a weekly basis throughout the duration of the experiment. After 3 months of immersion in acetic acid, images of samples were taken prior to other preparation.

Table 2. Chemical composition of binders (%).

Binder	CaO	SiO_2	Fe_2O_3	Al_2O_3	MgO	MnO	K_2O	Na_2O	TiO_2	SO_3
GU	62.5	19.6	2.27	4.9	2.61	0.05	0.9	0.24	0.25	2.57
AC	36.82	4.61	16.21	38.47	0.68	0.77	<0.02	0.04	1.75	-
SF	0.50	94.17	0.32	0.21	0.47	0.07	1.17	-	-	0.12
FA	1.87	42.2	27.60	21.60	0.92	-	2.55	0.66	-	1.10
MK	0.03	51.65	0.68	44.7	0.08	-	0.34	0.08	0.12	-
GBFS	37.31	36.77	0.85	7.77	13.91	1.02	0.43	0.31	0.36	-

Test methods - Altered Depth and Mass Loss measurements The altered depths and relative mass losses of the paste specimens were measured after the acid immersion. To quantify the altered depth, three specimens were sawn perpendicularly to their axis and some phenolphthalein was sprayed on the plane sections. The depth of the colour change, considered to correspond to the altered depth, was measured with an optical videomicroscope on several points distributed around the perimeter of the cylinders. The mass loss measurements were carried out on three immersed specimens that had first been wiped.

Test methods - Microstructure Analysis The microstructure of the altered samples was observed under a JEOL JSM-840A Scanning, Electron Microscope equipped with an energy dispersive X-ray analysis system (EDXA). The sound and altered zones of GBFS and MK paste samples were used for the determination of the porosity by mercury intrusion porosimetry (MIP) (Micromeritics AutoPore IV model) applying pressures up to (60000psi) on subsamples dried at 80°C for 72 hours previous to porosity measurement.

Test methods - Chemical and Mineralogical Analysis The chemical modifications of the specimen were analyzed by a Cameca SX-100 electron microprobe at an accelerating voltage of 15 kV, a current strength of 10 nA and a scanning volume of 5*5*5 m^3. The measurements were performed on polished sections from the altered surface to the centre or sound portion of the specimen. Particular care was given to the selection of the points analyzed in order to measure only the hydrated paste and to avoid residual anhydrous grains. Ca, Si, Al, Fe, Mg, S, Na, K, Ti and O concentrations were measured. The chemical data are expressed as percentages of oxide amounts in the volume sounded. Data in the altered zone were corrected according to the titanium content to represent the absolute variation of each element as suggested by Bertron et al. (2009). Mineralogical analyses were completed on solid phase samples analysed a Siemens D5000 X-ray diffractometer using Cu K radiation generated at 30 mA and 40 kV. Specimens were step-scanned as random powder mounts from 8-68° 2θ at 0.02°2θ steps integrated at 1.2 s $step^{-1}$. Analyses were performed on the sound and altered zones.

RESULTS AND DISCUSSION

Altered Depth and Mass Loss measurements Figure 1 shows the aspect of paste specimens after 3 months of immersion in the acetic acid solution. The pastes containing GBFS or MK kept their integrity. However, FA and SF pastes present important deterioration while the GU control and AC pastes present important dissolution of their superficial zone.

Figure 1. General aspect of paste samples after 3 months of immersion in acetic acid solution.

Figure 2 shows a section through the GU control sample after 3 months of immersion in the acetic acid solution (0.5M, pH 4). Three zones can be identified. A sound zone, marked Zone 1, was considered as being the basic zone where the color turns violet during the phenolphthalein test. An intermediate zone, marked Zone 2, and the external zone (zone 3) which is very porous and had a very weak mechanical strength. Zone 3 is very susceptible to drying shrinkage and presents large open cracks.

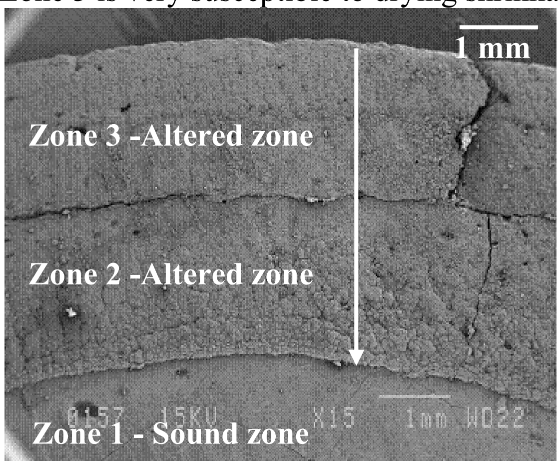

Figure 2. Secondary electron images of a section through a specimen. Zone 3 is the external altered zone, zone 2 is a more compact zone while zone 1 is the sound zone that turns violet during the phenolphthalein test.

Figure 3 presents the development of altered depths for different pastes tested. Differences observed between the various specimens are significant with altered depth measurements on FA and SF samples which are more important of that measured on GU control specimens. MK, GBFS and AC reduce the extent of the acid aggression compared to control sample. It is important to note that the superficial zone of AC and GU samples were completely dissolved during acid immersion but altered depth measurements reported in figure 3 take into account the surface dissolution.

Figure 4 shows the mass losses for all specimen tested. Replacement of a part of the Portland cement by MK or GBFS reduced considerably the mass losses for samples stored in acidic solution. The use of SF, FA or AC increased the mass losses compared to GU control specimen. According to mass loss and altered depth results, further experiments will only be conducted on MK and GBFS specimens, that present better performance against acetic acid solution.

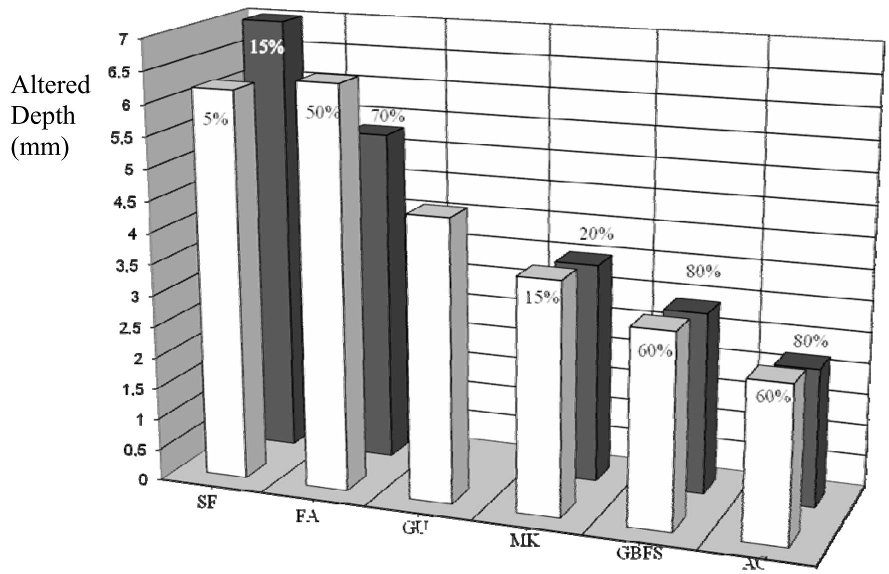

Figure 3. Altered depth of paste samples immersed 3 months in acetic acid solution.

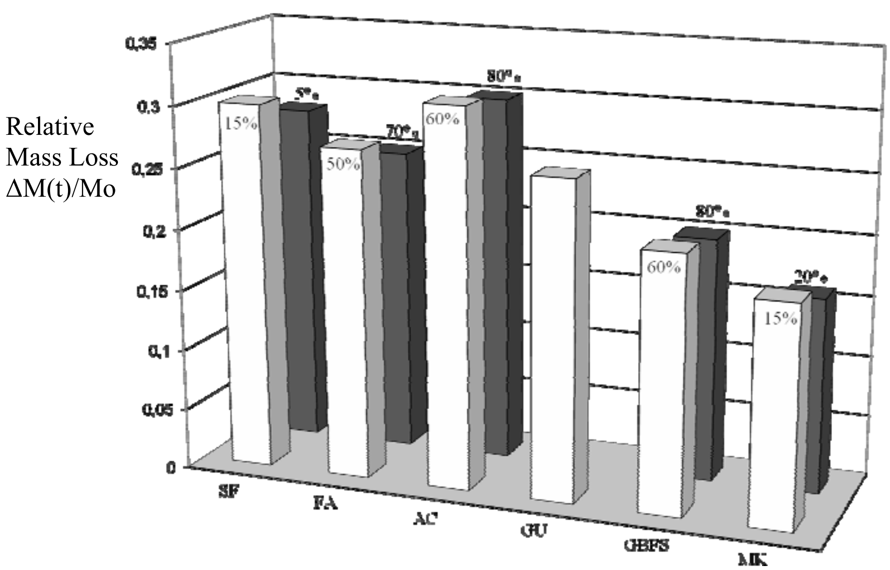

Figure 4. Relative mass loss of paste samples immersed 3 months in acetic acid solution.

Microstructure Analysis Figure 5 present the cumulative pore volume for the sound zones of pastes made with GBFS and MK measured by mercury intrusion. The sound zone was delimited from the colour change of the phenolphthalein indicator. Data for the GU control is given as comparison. Figure 6 presents the same data for the altered zone of sample immediately in contact with the acid solution.

In sound zones, the GBFS samples present the most important total porosity without any improvement of the size of pores, contrary to MK samples which allows a slight decrease of the total volume of pores but a large contribution to the refinement of pores. For each of these SCM additions, we notice an improvement of the porosity with the increase of the rate of cement replacement.

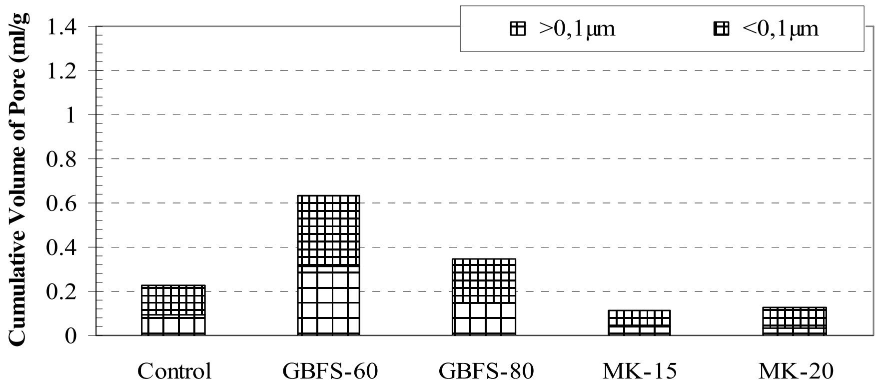

Figure 5. Porosity of the sound zones of samples immersed 3 months in acetic acid solution.

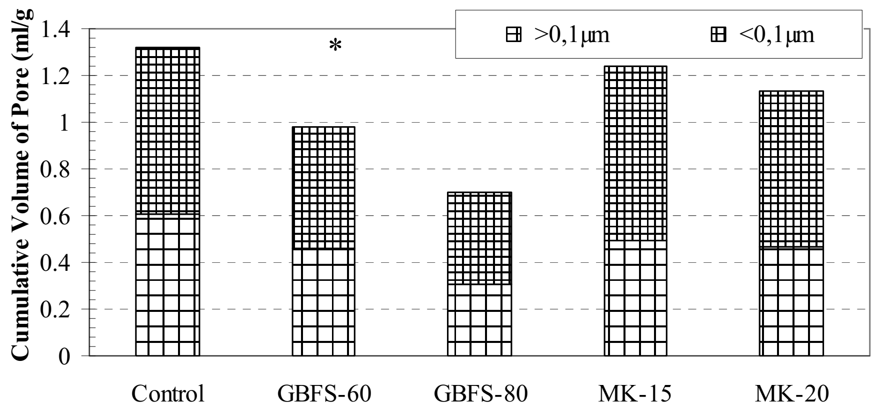

Figure 6. Porosity of the altered zones of samples immersed 3 months in acetic acid solution. (note - control sample - intermediate zone).

In altered zones, we note a net increase of the porosity for all specimens. The GU control sample presents the most important total porosity which should be more significant considering that the zone studied was an intermediate zone because the most degraded external zone was totally lost in solution. The altered zone of the MK samples was largely affected by the aggressive solution with total porosity values closed to that obtained with the intermediate zone of the GU control. However, the cumulative volume of pores smaller than 0.1 µm is more important for MK samples which will be beneficial according to transport properties of porous material. The samples containing GBFS present the weakest total porosities of all the tested samples knowing that their contribution to the refinement of pores is low.

Chemical and Mineralogical Analysis The chemical analysis of cement pastes that were immersed in the aggressive solution is given in figure 7. Electron microprobe chemical data are given as absolute percentages of oxide amount as a function of the distance from the surface of the specimen to their sound zone. Figure 8 shows the mineralogical characterization of the corresponding zones analysed by XRD.

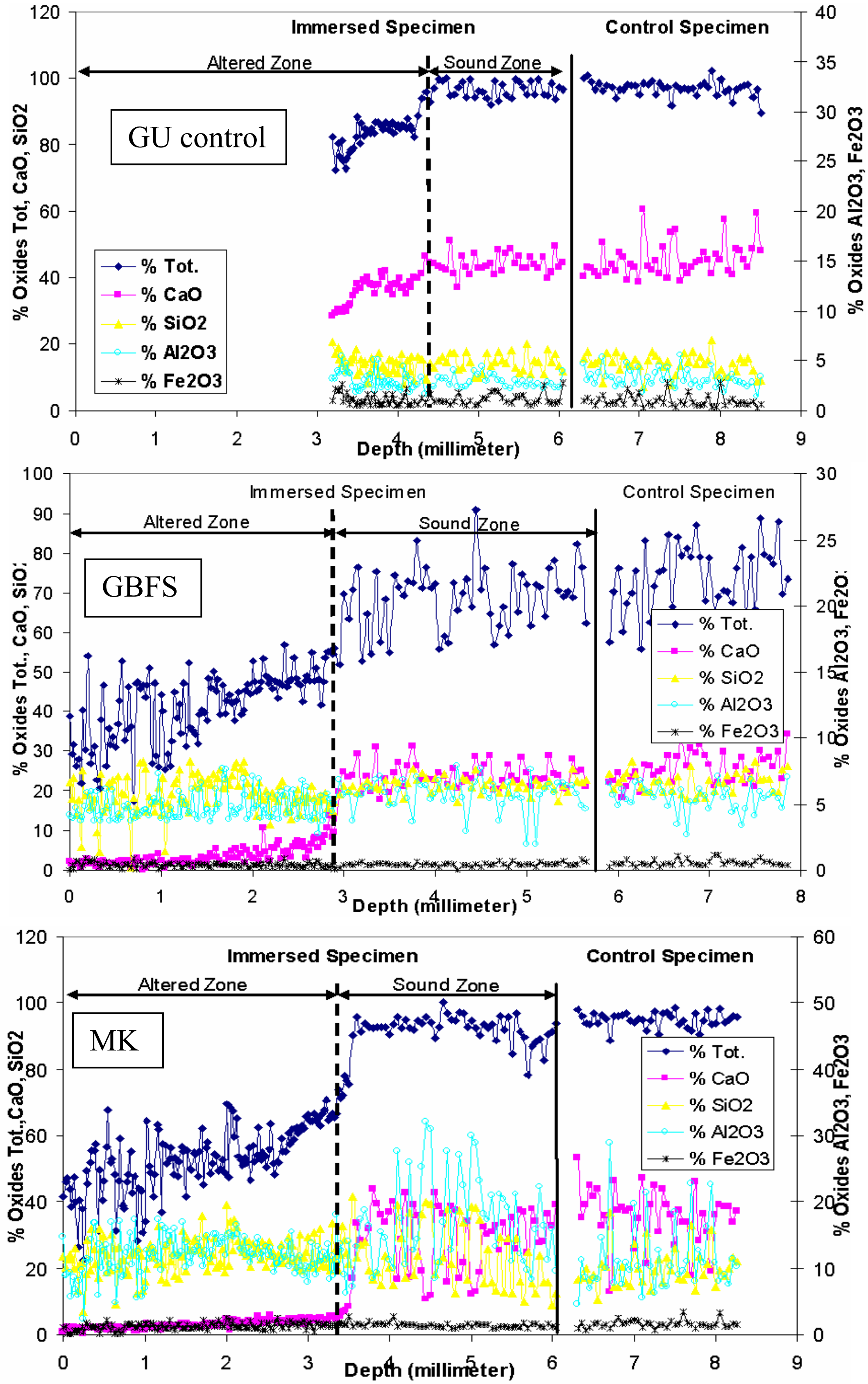

Figure 7. Chemical composition profiles analysed by electron microprobe on paste specimen according to the distance to the surface of the specimen.

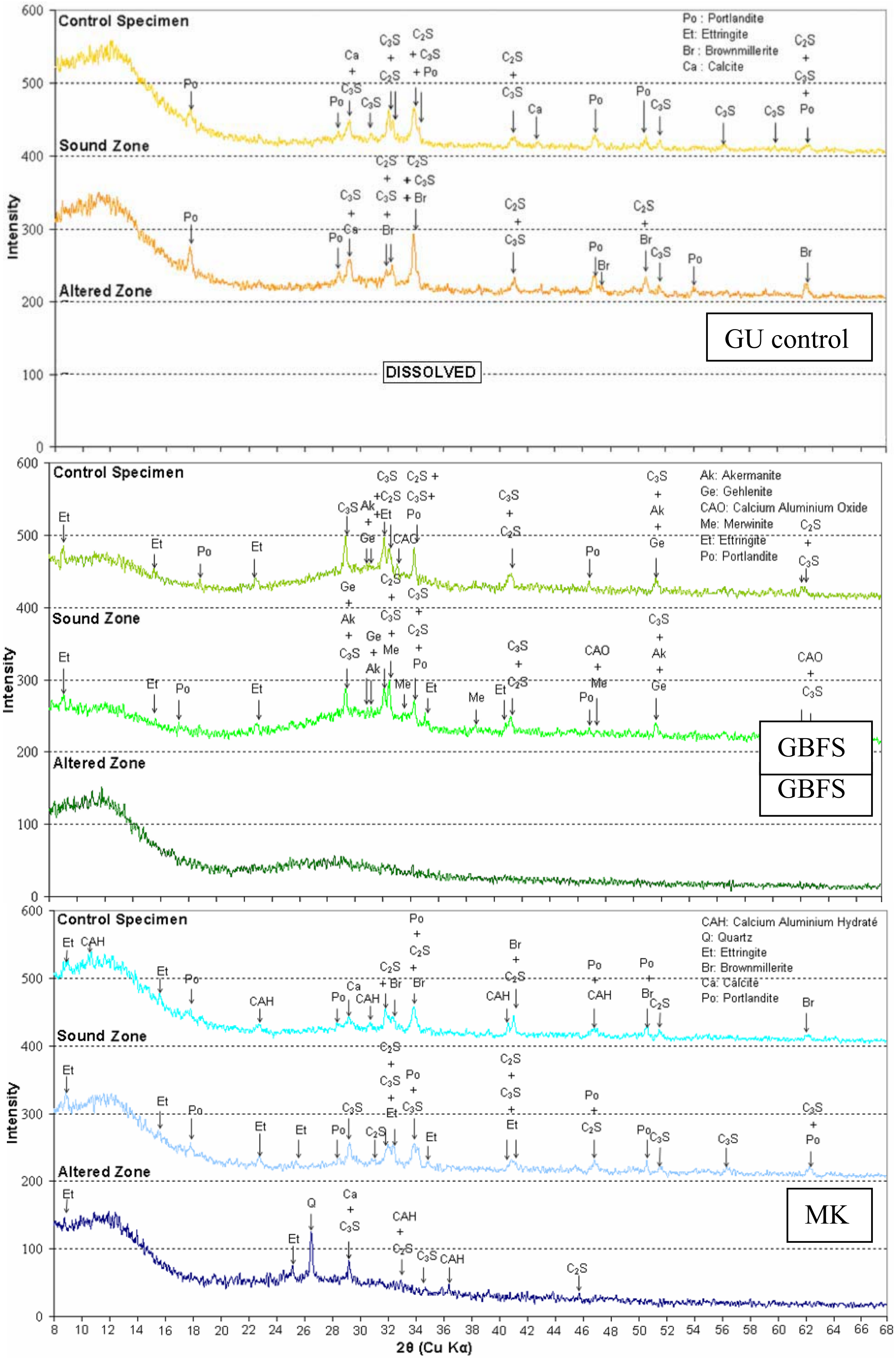

Figure 8. Mineralogical analysis by XRD of paste specimens after 3 months of acid immersion.

Electron microprobe data show that the sound zone has the same chemical composition as the control specimen that was kept in the wet room for the entire period of testing. The change in color of the phenolphthalein indicator matched a brutal drop of the CaO amount and a large decrease of the total amount of oxides. Altered depth measurements done by observation of the indicator color change corresponds exactly to the CaO drop measures by the electron microprobe. The altered zone of each sample is almost completely decalcified and the total amount of oxides drops reaching values of 30 and 40% for GBFS and MK samples, respectively. Lower value of the total amount of oxides is expected for the GU control sample as 25% was measured on paste samples cured for 3 months in a wet room prior to acid immersion (Oueslati 2011). The drop in CaO content is directly linked to the dissolution of portlandite ($Ca(OH)_2$) and to the decalcification of the C-S-H phase, followed by the complete dissolution of C-S-H. This alteration mechanism is confirmed by XRD results.

XRD data shows that GU sound zone is composed of portlandite, ettringite, C_2S, C_3S, brownmillerite and calcite. C-S-H, a poorly crystalline phase, is represented by a large hump centered on a reticular distance of 3.04Å. GBFS sound zone is composed of the same phases with the addition of akermanite, gehlenite and merwinite that are usual slag's phases. Compared to GU sound zone, a calcium aluminate hydrate phase is present in the MK sample. The altered zone of the GBFS sample is completely amorphous with a more pronounced hump centered close to quartz main line. MK altered zone is also more amorphous but still contains low intensity ettringite and anhydrous phases lines and we note the appearance of the quartz main line. Calcium-bearing phases are altered during acid attack and the altered surface tends to be covered by an amorphous phase which acts as a diffusive protective barrier against further attack.

CONCLUSION Agricultural effluents cause severe chemical attack on concrete structures. The attack causes a decalcification of the matrix and a progressive dissolution of the hydrated and anhydrous cement phases. In this study, the effects of supplementary cementing materials and alumina cement incorporation on behaviour of cement pastes immersed in acidic solution have been investigated.

Results indicate that acetic acid resistance of cement pastes could be improved significantly by incorporation of GBFS or MK. These samples were the ones which presented the weakest altered depth and mass loss compared to GU control, SF, AC and FA samples. Except for SF, the best performance against acidic attack was obtained with the higher Portland cement replacement level.

The acid attack is translated into a decalcification in the altered zone and a modification of the microstructure as measured by MIP. In sound zones, the GBFS samples present the most important total porosity without any improvement of the size of pores, contrary to the MK samples which allows slightly decreasing the total volume of pores but especially contributing to the refinement of pores. However, the altered zones of GBFS samples are by far less porous than for others samples which is important in reducing the kinetic of alteration. Both SCMs, GBFS and MK, improve the general behaviour of cement paste against organic acid media.

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DETERIORATION OF CEMENTITIOUS MATERIALS BY ORGANIC ACIDS IN AGRICULTURAL EFFLUENTS: EXPERIMENTS AND MODELLING

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CSAS11 17 – Topic I: Concrete deterioration

ABSTRACT Combining experiments with modelling, this paper investigates how Portland cement pastes are attacked when exposed to three aggressive solutions made of acetic acid, oxalic acid or a mixture: 1/3 oxalic - 2/3 acetic acids, the mixture being more representative of real effluents such as sugar cane molasses. Oxalic acid is known to have a protective effect toward OPC matrices whereas acetic acid has a strong leaching effect. The aim was to better characterize the degradation mechanisms by discriminating between acidic attack and complexation effects, determining the cement phase evolution, and assessing leaching sensitivity versus the solubility and molar volume of calcium salts. Under acidic pH, oxalate anions are stronger complexing agents of aluminium and calcium cations than are acetate anions. Calcium oxalate salt precipitates whatever the pH, while there is no precipitation of the highly soluble calcium acetate salts. Both oxalic acid alone and the acid mixture were protective, showing the predominant role of oxalic acid. Experiments and modelling led to similar results in terms of degradation rate and depth.

Keywords: acetic acid, alteration, concrete, organic acid attack, modelling, oxalic acid.

1. INTRODUCTION

Carboxylic acids present in agricultural and food-processing industry effluents, such as liquid manure, silage juices, and sugar cane and sugar beet molasses, severely attack concrete facilities such as storage silos, treatment plants, and animal houses. Therefore, understanding the mechanisms of concrete degradation by organic acids is of prime importance in the development of concretes that perform well in these environments. Experimental attack on concrete by quite a large range of organic acids showed that the degradation mechanisms were highly dependent on the acid and linked to the characteristics of their associated salts and complexes as well as those of the acid themselves (Larreur-Cayol et al. 2011a, Bertron et al. 2005, 2009). The dissociation constant of an acid and the solubility of its salt are known to directly influence how aggressive the acid is. When the calcium salt is soluble (case of acetic and lactic acids), the degradation mechanisms are comparable to those induced by strong acids with soluble calcium salts such as nitric and hydrochloric acids When the calcium salt is slightly soluble to insoluble, the effect of the deterioration may be protective or aggravating (case of tartaric, citric, oxalic acids – Larreur-Cayol et al. 2011a).

The dependency of degradation on the poly-acidity and the physical properties of the salts is not well understood yet. By combining experiments with modelling, this paper investigates how Portland cement pastes are attacked when exposed to one of three aggressive solutions: acetic acid, oxalic acid or a mixture of 1/3 oxalic, 2/3 acetic acids, the last being more representative of real effluents such as sugar cane molasses. Degradation rate and mechanisms were analyzed using mass losses, degraded depths, XRD and EPMA measurements. The first section gives a general analysis of the influence of different acid properties (pKa, complexation by the conjugated bases, solubility and molar volume of the salts) on the degradation mechanisms by means of calculated speciation and solubility diagrams. The rate and depth of the cement paste degradation are modelled in a second stage.

2 MATERIAL AND EXPERIMENTS

The specimens were CEM I 52.5 N cement pastes (Table 1) with a water/cement ratio of 0.27. They were cast in cylindrical moulds 75 mm high and 25 mm in diameter. The specimens were taken out of their moulds 24 h after pouring and stored in water at 20 °C for 28 days. They were then immersed in the aggressive solutions for one year at 20 °C, the acid concentration being kept constant by a regular renewal of the solution.

The acid concentration of the aggressive solutions was fixed to 0.28 mol/L (Table 2), in consistency with our previous studies (Bertron et al. 2005, 2007, Larreur-Cayol et al. 2011a). This corresponds to the highest total concentration of acids measured in effluents. The pH of the acetic acid solution was brought up to 4 (i.e. the lowest pH found in effluents) by adding NaOH. Such a pH increase was not feasible for the oxalic-type solution since oxalate anion interacts strongly with sodium (or potassium) cation. The pH of the oxalic acid and mixture solutions were thus kept at their strongly acidic initial value (Table 2).

3. MODELLING APPROACH AND DATABASE

The HYTEC reactive transport model (van der Lee et al. 2003) takes into account: i) the aqueous chemical mechanisms occurring both in the cement pore water and the leaching reactor, ii) the mineralogical alteration of the cement matrix, and iii) the coupled evolution of porosity and diffusivity.

Table 1. Chemical composition of the Portland cement.

	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	TiO_2	Na_2O	K_2O	SO_3	l.o.i.
CEM I 52.5 N	66.8	20.8	4.9	2.9	1.4	0.3	0.1	0.6	2.7	2.1

Table 2. Compositions of the aggressive solutions.

	Acetic	Oxalic	Mixture (acetic 2/3-oxalic1/3)
Acid concentration (mol/L)	0.28	0.28	0.28 (0.187/0.093)
NaOH concentration (mol/L)	0.06	0	0
pH	4	0.85	1.2

The change in specific volume of the cement phases can be calculated and HYTEC accounts for pore clogging by carbonation or, on the contrary, for porosity increase by leaching of hydrates. The evolution of the effective diffusion coefficient is related to porosity change through Archie's empirical law.

The chemical reactions were calculated at thermodynamic equilibrium, i.e. chemical reactions were assumed to be instantaneous compared to diffusion. The MINTEQ thermodynamic database - release 2.20 - was chosen for this study. One additional step was to select relevant data from the literature for the acid/base and complexation properties of the carboxylic acids. Table 3 details the equilibrium formation constants of the Al-Ca organometallic and hydroxyl complexes used in the present calculations, while Table 4 gives the physicochemical properties of the calcium organic salts. The database was also enriched with additional thermodynamic constants for cement-type minerals. The decalcification of calcium and silicate hydrate (C-S-H) was modelled by assuming a set of three types of C-S-H, or $C_xS_yH_z$ (C/S ratios of 0.8, 1.1 and 1.8).

A two-dimensional simulation in cylindrical symmetry was performed to model the cement specimens and the leaching reactor, complying with the experimental solution/solid ratio. In the model, the porosity of the cement pastes was set to 10% and the effective diffusion coefficient to 10^{-12} m^2/s. The modelled hydrated cement paste was composed of C-S-H 1.8 (51 wt.%), portlandite (18%), ettringite (13%), C_3FH_6 (6%), hydrogarnet (5%), monocarboaluminate (5%) and hydrotalcite (2%).

4 RESULTS AND DISCUSSION

4.1 Al and Ca chemistry in presence of acetic and oxalic acids

4.1.1 Al and Ca complexation

Figure 1 shows the calculated distribution of the aqueous species as a function of pH in presence of acetic acid at 0.28 mol/L and for calcium and aluminium concentrations representative of a cement pore solution. It is worth emphasizing that the precipitation of solid phases is not taken into account for the sake of simplicity. At pH 1, i.e. well below the pKa of acetic acid (4.76), the protonated form of acetic acid is predominant and, therefore, calcium is present as free Ca2+ cations only. As pH increases, the acetate concentration rises and partly complexes calcium, or even totally complexes aluminium. However, under more alkaline conditions, the inorganic hydroxyl complexes of aluminium eventually become the most stable species. Not considered here, gibbsite willprecipitate between 5 and 11 under the present chemical conditions.

The range of pKa values of the oxalic acid (1.28 and 4.28) implies that oxalate complexes are formed at lower pH than acetate ones. In presence of oxalic acid, aluminium complexation is a major process over the pH range 1 to 9, the latter value being the pH above which the $Al(OH)^{4-}$ hydroxyl complex is the most stable species (Fig. 1). At pH around 1, calcium is not yet complexed, which may suggest that it could be more available to interact in another way (e.g. calcium oxalate precipitation, which is not considered in these calculations). Calcium oxalate complexes are the main aqueous species above pH 3. There are no significant differences between the complexation of calcium and aluminium in the oxalic acid solution or the acid mixture (Fig. 2) despite a concentration of acetic acid twice that of oxalic acid in the mixture. Oxalate is a very strong complexing agent compared to acetate.

Table 3. Equilibrium constants of reactions (mass balance equations) corresponding to the formation of the main Al-Ca complexes considered in the modelling.

Species	Reaction	Log K (25°C)
H-acetate(aq)	$H^+ + C_2H_3O_2^- \rightarrow C_2H_4O_2$ ($C_2H_4O_2 = CH_3\text{-}COOH$)	4.76
$Al\text{-}acetate^{2+}$	$Al^{3+} + C_2H_3O_2^- \rightarrow AlC_2H_3O_2^{2+}$	2.75
$Al\text{-}(acetate)_2^+$	$Al^{3+} + 2\ C_2H_3O_2^- \rightarrow Al(C_2H_3O_2)_2^+$	4.60
$AlOH\text{-}acetate^+$	$Al^{3+} + C_2H_3O_2^- + H_2O \rightarrow AlOHC_2H_3O_2^+ + H^+$	-0.15
$Al_2(OH)_2\text{-}acetate^{3+}$	$2Al^{3+} + C_2H_3O_2^- + 2H_2O \rightarrow Al_2(OH)_2C_2H_3O_2^{3+} + 2H^+$	-2.41
$Ca\text{-}acetate^+$	$Ca^{2+} + C_2H_3O_2^- \rightarrow CaC_2H_3O_2^+$	1.18
$Ca\text{-}(acetate)_2(aq)$	$Ca^{2+} + 2C_2H_3O_2^- \rightarrow Ca(C_2H_3O_2)_2$	1.40
$H_2\text{-}oxalate(aq)$	$2H^+ + C_2O_4^{2-} \rightarrow C_2H_2O_4$ ($C_2H_2O_4$ = HOOC-COOH)	5.52*
$H\text{-}oxalate^-$	$H^+ + C_2O_4^{2-} \rightarrow C_2HO_4^-$	4.27
$Al\text{-}oxalate^+$	$Al^{3+} + C_2O_4^{2-} \rightarrow AlC_2O_4^+$	7.73
$Al\text{-}(oxalate)_2^{2-}$	$Al^{3+} + 2C_2O_4^{2-} \rightarrow Al(C_2O_4)_2^-$	13.41
$Al\text{-}(oxalate)_3^{3-}$	$Al^{3+} + 3C_2O_4^{2-} \rightarrow Al(C_2O_4)_3^{3-}$	17.09
$Al\text{-}Hoxalate^{2+}$	$Al^{3+} + C_2O_4^{2-} \rightarrow AlC_2HO_4^{2+}$	7.46
AlOH-oxalate(aq)	$Al^{3+} + C_2O_4^{2-} + H_2O \rightarrow AlOHC_2O_4 + H^+$	2.57
$AlOH\text{-}(oxalate)_2^{2-}$	$Al^{3+} + 2C_2O_4^{2-} + H_2O \rightarrow AlOH(C_2O_4)_2^{2-} + H^+$	6.84
$Al(OH)_2\text{-}oxalate^-$	$Al^{3+} + C_2O_4^{2-} + 2H_2O \rightarrow Al(OH)_2C_2O_4^- + 2H^+$	-3.12

Ca-oxalate(aq)	$Ca^{2+} + C_2O_4^{2-} \rightarrow CaC_2O_4$	3.19
Ca-(oxalate)$_2^{2-}$	$Ca^{2+} + 2C_2O_4^{2-} \rightarrow Ca(C_2O_4)_2^{2-}$	4.02
Al-hydroxyl^{2+}	$Al^{3+} + OH^- \rightarrow AlOH^{2+}$	9.00
Al-(hydroxyl)$_2^+$	$Al^{3+} + 2OH^- \rightarrow Al(OH)_2^+$	17.71
Al-(hydroxyl)$_3$	$Al^{3+} + 3OH^- \rightarrow Al(OH)_3$	25.31
Al-(hydroxyl)$_4^-$	$Al^{3+} + 4OH^- \rightarrow Al(OH)_4^-$	33.31
Ca-hydroxyl$^+$	$Ca^{2+} + OH^- \rightarrow CaOH^+$	1.30

* Corresponding to $pK_{a1} = 1.2$ and $pK_{a2} = 4.2$ for the oxalic dicarboxylic acid.

Table 4. Physicochemical properties of calcium organic salts considered in the modelling.

Mineral	Reaction	LogK (25°C)	Density [kg/m^3]	Molar volume [cm^3/mol]
Ca-(Acetate)$_2$	$Ca^{2+} + 2C_2H_3O_2^- \rightarrow Ca(C_2H_3O_2)_2$	3.63	1500	105.4
Ca-(Oxalate):H_2O	$Ca^{2+} + C_2O_4^{2-} \rightarrow Ca\text{-}C_2O_4{:}H_2O$	8.73	2260	66.4
Ca-(Oxalate):2H_2O	$Ca^{2+} + C_2O_4^{2-} \rightarrow Ca\text{-}C_2O_4{:}2H_2O$	8.3	2020	81.2

4.1.2 Al and Ca solubility diagrams

The combination of dissolved calcium ions with the conjugated bases of the carboxylic acids may lead to the formation of salts, which primarily influence the mechanisms and aggressiveness of the acidic attack. In other words, salt stability versus pH and acid concentration is a key issue for the resistance of cementitious materials in contact with agricultural environments.

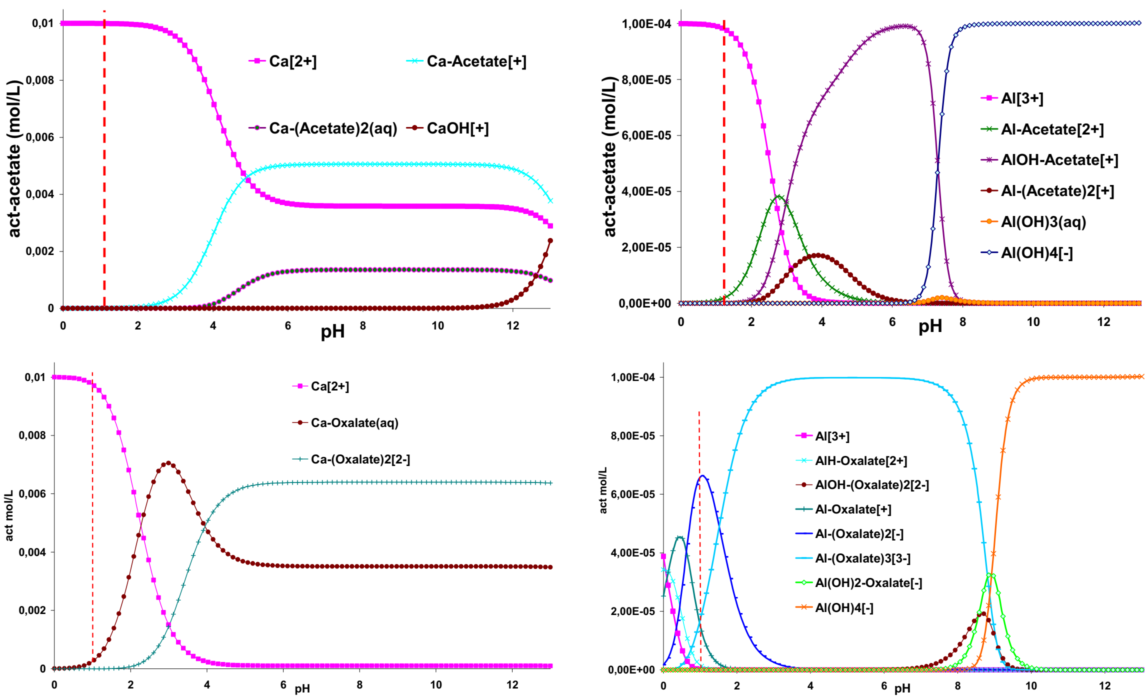

Figure 1. Calculated pH dependency of Ca and Al complexation in presence of acetic acid (top) and oxalic acid (bottom) at 25 °C without considering any solid phase precipitation. Total concentration in mol/L: [Ca] = 10^{-2}, [Al] = 10^{-4}, [acetic acid] = $2.8\text{x}10^{-1}$, [oxalic acid] = $2.8\text{x}10^{-1}$.

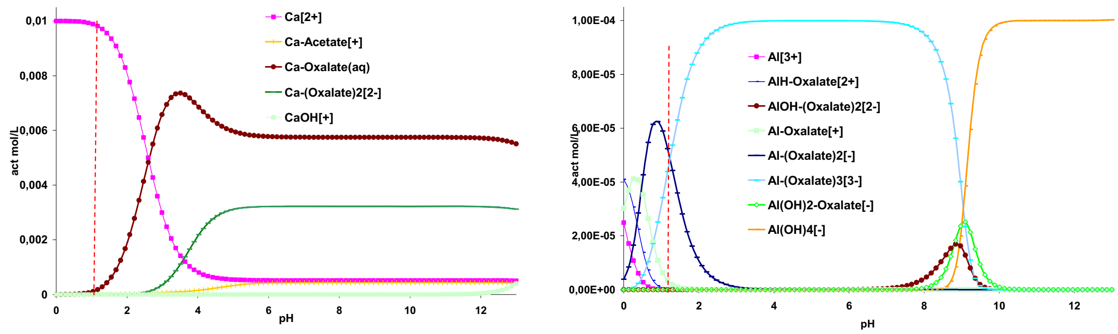

Figure 2. Calculated pH dependency of Ca and Al complexation in the acid mixture at 25 °C. Total concentration [Ca] = 10^{-2}, [Al] = 10^{-4}, [acetic acid] = 1.86×10^{-1}, [oxalic acid] = 9.4×10^{-2}.

Figure 3 shows the calculated solubility diagrams of calcium and aluminium in acetic acid and oxalic acid solutions. The former acid does not lead to any salt precipitation in the concentration range 10^{-7} – 10^{-2} mol/L. Gibbsite, an inorganic aluminium hydroxide, may precipitate in the pH range 4 – 11 as already mentioned.

Calcium oxalate salt can precipitate over a wide pH range, already for low acid concentrations, and even at the expense of portlandite in higher acid concentrations. Aluminium precipitation is again driven by gibbsite. However, compared to acetic acid, the aluminium complexation by oxalate narrows the stability domain of gibbsite in the acidic pH range. In this respect, De Windt et al. (2010) have shown that aluminium complexation by carboxylic acid has to be considered to estimate the proportion of aluminium inserted into the silica gel layer that may be formed during the acid attack of Portland cement pastes.

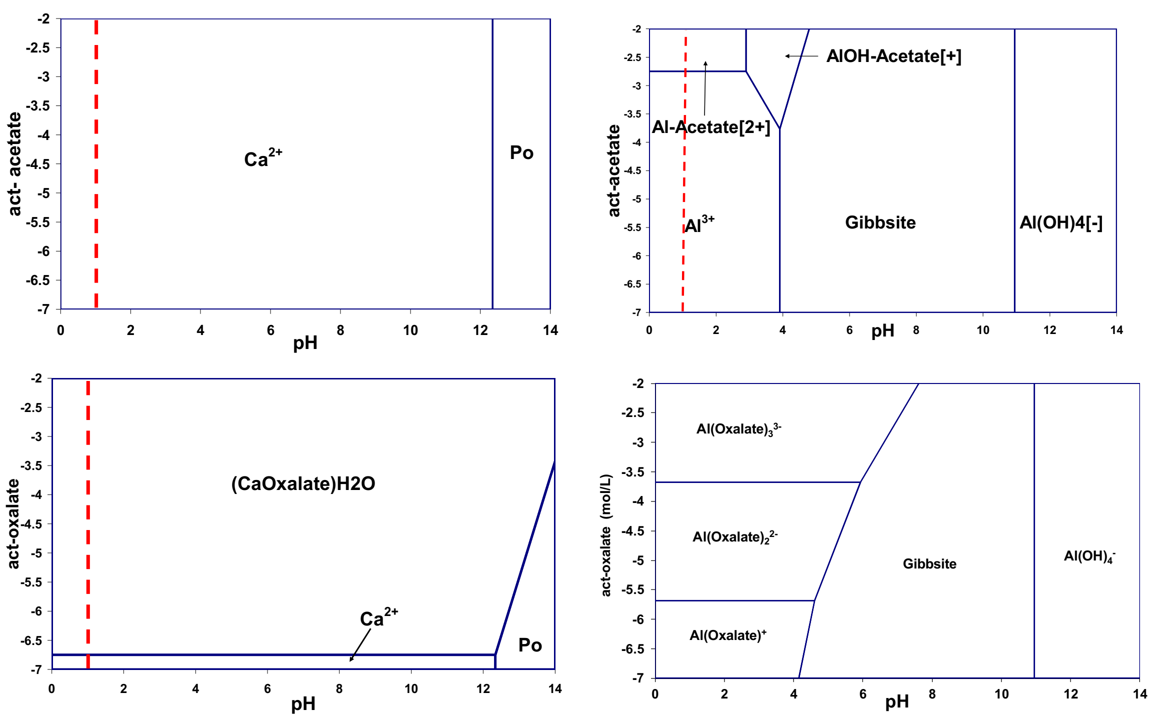

Figure 3. Calculated solubility diagrams of Ca and Al in acetic acid solution (top) and oxalic acid solution (bottom) at 25 °C. *Po* stands for portlandite.

4.2 Deterioration of the cement paste by acetic and oxalic acids

4.2.1 Macroscopic observations and microscopic analysis The samples kept in acetic acid had an orangey colour (Fig. 4). The outer layer showed some cracks but this layer adhered to the specimen and was not dissolved during the attack. There was no visible precipitation of salts, in agreement with Section 4.1.2. The cracking was probably due to the shrinkage of the degraded zone resulting from the loss of calcium during the attack.

Neither oxalic acid alone nor the mixture of acids was aggressive: no macroscopic damage (change of colour, erosion, loss of mass…) was observed on the cement pastes during the experiment. Nevertheless, the aspect of the specimens immersed in oxalic acid alone was different from the appearance of those immersed in the mixture of acids. The latter were covered by macroscopically observable Ca-oxalate salts, in agreement with Section 4.1.2. The salts were identified as Ca-oxalate mono- and di-hydrates (i.e. whewellite and weddelite, respectively) using XRD (Larreur-Cayol et al 2011b). These salts could be easily collected from the surface by simple scraping. The surface of the specimens immersed in the pure oxalic acid solution was grey and slightly rough but the precipitation of salt, identified by XRD as Ca-oxalate mono-hydrate, was not visible with the naked eye (Larreur-Cayol et al. 2011a). It was assumed that, because of its low solubility and a suitable molar volume, the precipitation of this salt sealed the capillary porosity. Despite the different aspects of the specimens, the chemical profiles analyzed with electron microprobe were the same for oxalic acid solutions and for the mixture of acids (Larreur-Cayol et al. 2011b).

Acetic acid alone | Oxalic acid alone | Oxalic + acetic acids

Figure 5. Aspects of paste specimens after 2 months of immersion in the three aggressive solutions.

4.2.2 Degradation rate and extent of the mineralogical alteration The concentration gradient between the reactor solution and the cement pore water causes ionic diffusion. In particular, the organic acids readily diffuse into the cement pores, bringing about hydrate dissolution. Both the experimental analysis and the reactive transport modelling indicate that acetic acid strongly accelerates hydrate dissolution by acidic hydrolysis (Fig. 6). The thickness of the degraded zone was many times larger than the degradation thickness found in a control test with pure water.

The deepest degradation front corresponds to portlandite and ettringite dissolution as well as C-S-H decalcification (Fig.6), corresponding to a net loss of calcium from the cement specimen to the reactor solution. The outermost degraded zone consists of alumina and silica gels. The cement phase C_3FH_6 is transformed into a ferric hydrous phase, but without modifying the total iron content of the specimen. A transitory front of a small amount of Ca-acetate salt and calcite progresses over time within the cement matrix. The modelling accuracy for degradation thickness and rate is consistently enhanced when the increase of diffusivity in the degraded zone is considered. In particular, the porosity of the altered gel layer is so high that the degradation rate follows a linear time dependency and not a diffusional root square one.

In contrast, the degradation induced by oxalic acid is restricted to the very surface of the cement specimen both experimentally and in the calculations (Fig. 6). This is related to the fast precipitation of calcium oxalate salt close to the cement/reactor interface, but located inside the cement matrix, acting as a protective diffusive barrier. The protecting effect of calcium oxalate salt also occurs in the modelling of the acid mixture.

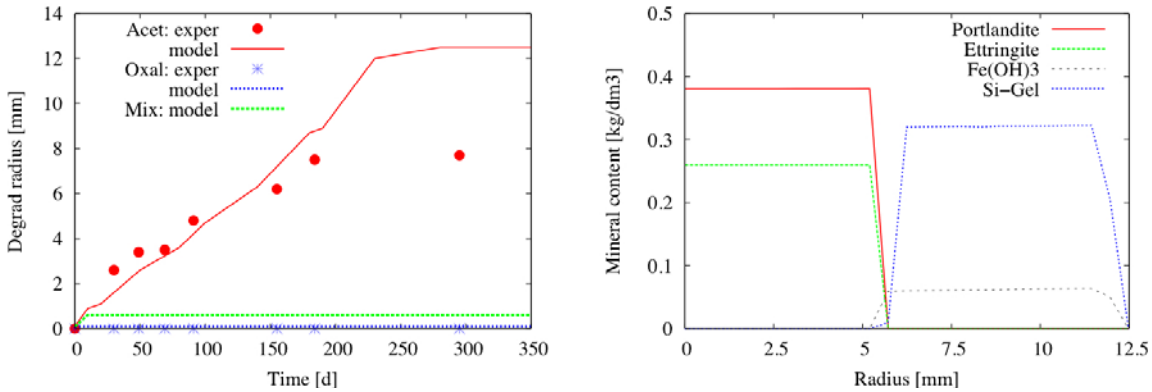

Figure 6. *Left:* Evolution of the degraded layer depths over time for Portland cement paste immersed in pure acetic or pure oxalic solutions, or in a mixture of these acids; *Right*: amounts of some cement phases and Ca-oxalate calculated after 90 days for acetic acid.

5. CONCLUSION AND PERPECTIVES

Oxalic acid exerts a protective action on Portland cement, both as a pure solution and in an acid mixture, due to the precipitation of calcium oxalate salt in the capillary porosity. The modelling reveals that Ca-oxalate salts precipitate at the cement surface and are stable whatever the pH. Therefore, they may offer great protection in various media. In contrast, the acetic acid solution leads to degradation to of the cement paste to a significant depth by an acidic hydrolysis process. Modelling indicates that the precipitation of Ca-acetate salt within the cement matrix is temporary if it exists at all.

Modelling based on intrinsic physicochemical properties (complexation constant, poly-acidity constant, salt formation constant and molar volume) of the different aqueous and solid species involved in the interaction between the cementitious materials and the organic acid solution can be a useful tool to investigate the main degradation mechanisms and, reciprocally, to help in the designing of more resistant concrete. A similar combined experimental/modelling study is in progress for a complementary set of carboxylic acids (citric, succinic and tartaric).

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INFLUENCE OF WASTES CONTENT ON PROPERTIES OF POLYMER CONCRETE

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CSAS11013 – Topic 1: Concrete Deterioration

ABSTRACT. In the building materials industry new materials have occurred and their application is in continuous development. Polymer concrete is one of the new composite materials which has special properties and offers the possibility of improving them by using different types of compounds. Near the binder and aggregates, in the polymer concrete mix can be used various types of fillers or fibers, which are usually wastes or by-products. In this study, epoxy resin was used as binder for obtaining polymer concrete. In each composition, two types of wastes were introduced as filler: fly ash (FA) and silica fume (SUF). The compositions used in the present research derived from a previous study which investigated a large number of mixes using different dosages of resin and only one type of waste as filler. The mechanical characteristics of the obtained composite polymer concrete such as compressive strength, flexural strength and split tensile strength were investigated. The experimental results shown that for the same dosage of epoxy resin the maximum compressive strength and split tensile strength were obtained for maximum fly ash dosage, while the maximum flexural strength was obtained for a medium FA dosage. The use of composite polymer concrete (PC) as construction material in various structures, including farm building applications, involves several environmental, economical and social benefits. Thermal insulation properties of the PC are superior to those of classical concrete, leading to the enhancement of thermal comfort, simultaneously with the energy cost savings. Also, using PC could enhance the waste management strategy.
Keywords: *construction, polymer concrete, properties, waste*

INTRODUCTION. In the last 50 years, the interest in the manufacture and the use of composite polymer concretes (PC) in various applications was continuously evolving. On the other hand, enhanced waste management strategies are required for handling huge amounts of wastes generated from different industries, such as silica fume (SUF) and fly ash (FA) from ferrosilicon factories and thermoelectric plants. These wastes were already used in concrete and mortar mixture, mainly to improve their mechanical properties, texture and workability (Thomas et al., 1998; Bentz et al., 2011). SUF and FA demonstrated potential to be used as fillers in PC matrix as well. The recovery and the reuse of these wastes will reduce the environmental impact due their disposal on landfills. Moreover, the manufacture of one tone of Portland cement used in conventional concrete releases about 0.8 tones of CO_2 into atmosphere. According to Gladel and Fauteux (2007), the cement industry contributes to about of 5-7% of global greenhouse gases emissions and global warming. In contrast, PC is realized of polymer binder instead of cement. Therefore, replacing ordinary concrete with PC has an important environmental benefit, but can be more expensive with respect to its ingredients and dosages. In addition to polymer binder, the PC recipe includes also aggregates such as crushed stone, gravel, limestone, chalk, silica fume, granite, quartz, expanded glass, etc. (Hing, 2007). Various types of resin can be used in the PC matrix, such as: epoxy resin (Abdel-Fattah and El-Hawary, 1999; Reis, 2004) polyester resin (Varughese and Chaturvedi, 1996), furan resin (Muthukumar and Mohan, 2005), poly(methylmethacrylate) resin (Blaga and Beaudoin, 1985).

PC composites are usually characterized by small thermal conductivity, good chemical/corrosion resistance, very low water sorption, good abrasion resistance and marked freeze-thaw stability. Particularly, thermal insulation properties are superior to those of classical concrete, leading to the enhancement of thermal comfort, simultaneously with the energy cost savings (Gorninski et al., 2007). Therefore PC composites are recommended in certain special applications, such as for industrial flooring, skid-resistant overlays in highways, epoxy plaster for exterior walls, resurfacing of deteriorated structures, farm structures etc. (Hing, 2007; Islam et al., 2011; Golestaneh et al., 2010).

The properties of PC are generally determined by the nature and the amount of the polymer binder and by the type and dosage of filler (Barbuta, 2007). For improving the properties of polymer concrete various fiber reinforcements are used as well (glass, carbon fibers, PVA, etc.) (Shao et al., 2005; Reis, 2007). Various types of filler can be used in the composition of polymer concrete, such as: silica fume, fly ash, phosphogyps, talc, limestone powder, magnetite fume etc. (Harja et al., 2008, Koleva et al., 2008). In the literature are presented the studies with one single type of filler. The presence of filler is important because it can improve the performances of material (Varughese and Chaturvedi 1996; Reis and Ferreira, 2004; Harja et al. 2009a; Barbuta et. al. 2010a; Barbuta et al. 2010b; Barbuta et al. 2009). It should be noted that the availability of fillers such as fly ash and silica fume in a geographical region are not always available or sufficient, depending on its industrial sector (Gladel and Fautex, 2007). In such case, the use of waste mixtures in the PC matrix may compensate this drawback. The correct combination of silica fume, fly ash, superplasticizer and polymeric additions may have synergistic effects, resulting in the construction material performing well in specific applications (Souza Almeida and Sichieri, 2006). Recent studies on fly ash as filler in epoxy mortar suggest that it can replace quartz filler with improved mechanical properties.

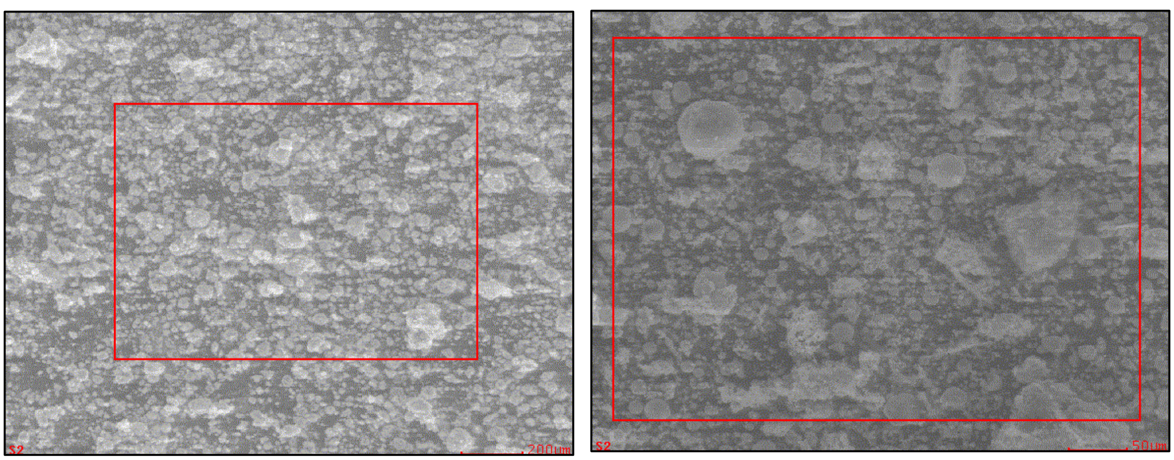
In this study, epoxy resin was used as binder for obtaining composite polymer concrete. In each composition, two types of wastes were introduced as filler: fly ash (FA) and silica fume (SUF). The compositions used in the present research derived from a previous study which investigated a large number of mixes using different dosages of resin and only one type of waste as filler. The mechanical characteristics of the obtained composite polymer concrete such as compressive strength, flexural strength and split tensile strength were investigated. In addition, it is shown that other properties such as the thermal conductivity can be further considered in the evaluation of the PC as sustainable alternative to conventional concrete.

EXPERIMENTAL PROCEDURE

Different PC compositions were prepared by slow mixing of ROPOXID 701 epoxy thermosetting resin (EP) combined with ROMANID 407 polyamide hardener firstly with river gravel crushed aggregates, then with silica fume (SUF) and/or fly ash (FA) at different ratios in a mechanical mixer (Barbuta and Lepadatu 2008; Lepadatu et al., 2010). Selected PC mixture compositions and corresponding performances are depicted in the next section. Two sizes of crushed aggregates were used in these experiments: 0-4 mm (Sort I) and 4-8 mm (Sort II). The range of the resin content used for the experiments was adopted with respect to workability condition and the product cost (Barbuta and Lepadatu 2008).

The fillers SUF and FA were provided as by-products from romanian ferrosilicon alloy factory and thermal power plant. SUF and FA have the following typical characteristics: particles size of 0.01-0.5 μm (SUF) and 0.01-100 μm (FA); density of 2250 kg/m^3 (SUF) and 2400-2550 kg/m^3 (FA); specific surface of 13000 m^2/kg (SUF) (Abul Bari et al., 2008; Baltakys et al., 2007; Barbuta et al., 2010b).
The morphology, chemical and mineralogical composition of these wastes were analyzed by SEM/EDX and XRD, using electronic microscope QUANTA 3D series AL99/D8229 and difractometer X'PERT PRO MRD (PANalytical Holland).

The scanning electronic microscopy (SEM) for SUF and FA is presented in Figure 1 and indicates in both cases a quite homogenous arrangement of spherical fly ash and/or irregular silica particles, which is suitable for polymer concrete applications (Harja et al., 2009b). The chemical compositions of SUF and FA are presented in Figure 2 and Table 1. As can be seen in Figure 2, both wastes contain Si, O, Al, Fe, Ca, K and trace of Mn, Mg and Ti (Harja et al., 2009b; Harja et al., 2009c, Harja et al., 2010).
Mechanical characteristics of PC were determined according to standard procedures (EN 12390/2001). For each tested composition, PC cubes of 70.7 mm and prisms of 210x70x70 mm sides were poured and tested for mechanical characteristics at 14 days. Thus, three cubes were tested for compressive strength, three cubes for split tensile strength and three prisms for flexural strength. Medium values were considered for PC performance evaluation.



Silica fume

Fly ash

Figure 1. Electronic microscopy for wastes

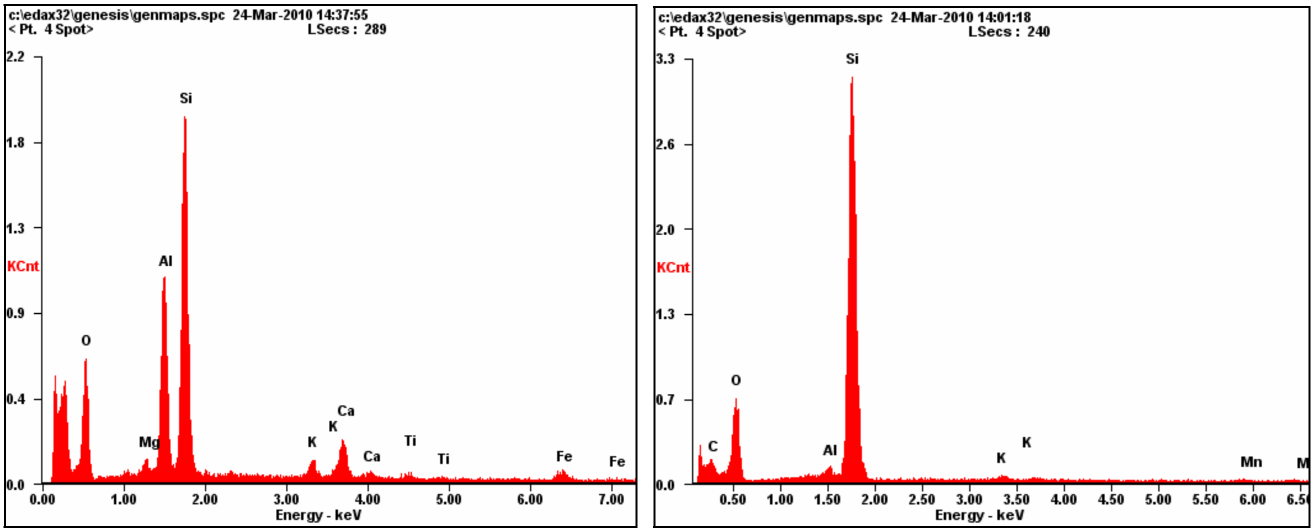


Figure 2. Chemical composition for wastes

Table 1. Chemical composition of wastes, %

Composition*	Ash	Silica fume
SiO_2	55.2	86-95
Fe_2O_3	7	0.85-2.5
Al_2O_3	22.6	1.3-4
CaO	8.3	0.4- 0.8
MgO	1.1	0.6-1.5
MnO	-	0.3-1.2
TiO_2	0.8	-
C	2.4	1.1-2.5

*balance: residues traces

RESULT AND DISCUSSION

Table 2 presents the PC mixture compositions considered for PC performance evaluation in terms of mechanical characteristics. As can be seen, same mixture ratios were used for each type of waste-rich polymer concrete (PC with SUF+FA (BPSF), PC with SUF (BPS) and PC with FA (BPF)), allowing their performance comparison.

Table 2. Mixture PC design combinations, %

Mixture No.	EP resin, %	Filler, %	Sort I, %	Sort II, %	FA+SUF			fc, MPa		Fti, MPa		ftd, MPa	
					fc,MPa	fti,MPa	ftd,MPa	SUF	FA	SUF	FA	SUF	FA
2	12.4	12.8	37.4	37.4	60.4	16.1	6.8	58.8	69.8	12.3	14.0	6.9	7.2
3	12.4	6.4	43.8	37.4	50.2	14.3	6	59.6	58	16.8	16.7	7.7	5.8
6	15.6	6.4	40.6	37.4	44.6	15.2	6.2	58.6	64.5	17.6	16.8	6.3	7.3
11	16.4	7.2	38.2	38.2	38.8	12.1	5.7	58.8	67.8	15.6	15.7	7.3	6.4
12	13.2	10.4	38.2	38.2	44.5	13.8	5.2	63.2	60.6	14.4	16.5	6.9	5.9
15	14	8.0	39.0	39.0	36.1	12.2	5.6	57.8	61.1	14.8	13.7	6.5	7.9

Compressive strength

Figure 3 presents the compressive strength (f_c) of polymer concretes as a function of epoxy resin content, when a minimum content of filler (i.e. 6.4%) is used. As can be seen, with the increase of the resin content, f_c decreases for BPSF, but increases for BPF and has no significant effect on BPS. According to Table 2, fc for BPSF is lower than for BPS or BPF, any given PC composition, except the mixture 2 where comparable performances are observed. The maximum f_c value for BPSF is obtained when a minimum content of epoxy resin and a maximum content of filler are used in PC matrix (mixture 2). The increase of filler content for a minimum dosage of resin (12.4%) has no significant effect on fc, except for BPSF and BPF where an fc increase is recorded (Figure 4). Therefore, the increasing of epoxy resin dosage or the decreasing of filler dosage results in the diminishing of compressive strength for BPSF. Apparently, resin agglomeration can occur at high resin content, leading to the increase of PC porosity and thus the decrease of PC performances (Koleva et al., 2008).

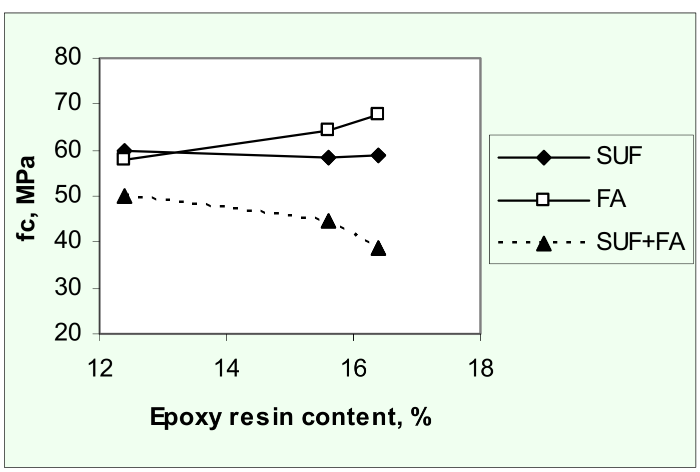


Figure 3. Compressive strength variation for CPM with 6.4% filler, as a function of epoxy resin content

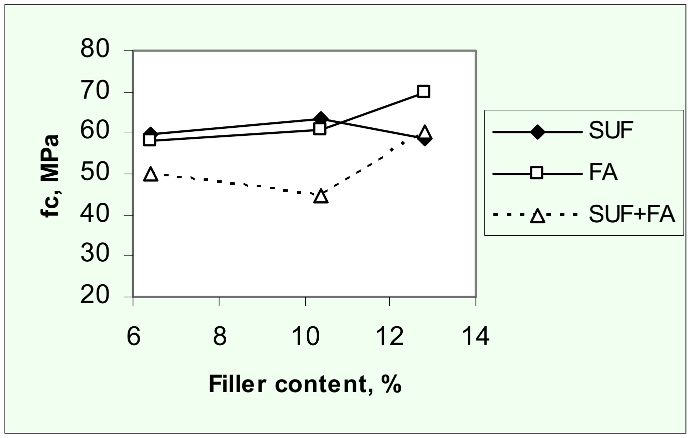


Figure 4. Compressive strength variation for CPM with 12.4% epoxy resin, as a function of filler content

Overall, the fc of all PC compositions is about 1-3 folds higher than that of ordinary Portland cement concrete (32.5 MPa). According to Golestaneh et al. (2010), the above mentioned superior PC performance is likely due the higher compactness of the aggregates and filler. Same authors reported for PC a 4 folds higher fc than for ordinary concrete when silica powder is used as filler. Thus, the type and the dosage of components have an important influence on the PC compressive strength.

Flexural strength

Figure 5 presents the flexural strength (f_{ti}) of PC as a function of epoxy resin content, when a minimum content of filler (i.e. 6.4%) is used in PC matrix. As can be seen, f_{ti} slightly increases with the increase of EP content up to 15.6%, after which it significantly decreases especially for BPSF. According to Table 2, f_{ti} for BPSF is lower than for BPS or BPF for all PC compositions, except for the mixture 2 where a comparable performance is obtained. In contrast to BPS and BPF, the decreasing of filler dosage results in the diminishing of flexural strength of BPSF (Figure 6).

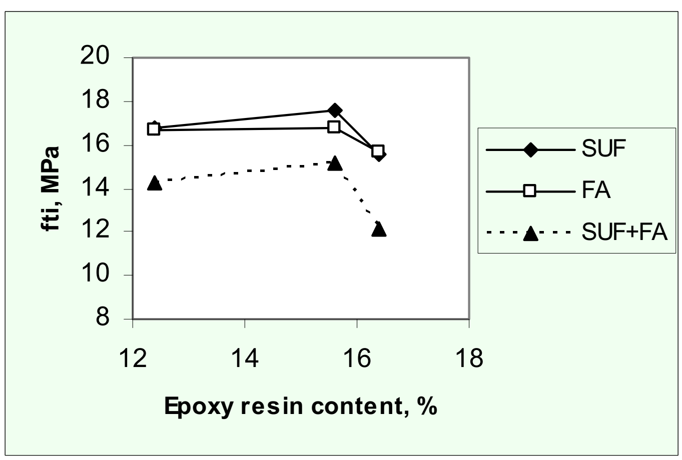


Figure 5. Flexural strength variation for polymer concrete with 6.4% filler, as a function of epoxy resin content

Therefore, the maximum value of f_{ti} for BPSF is obtained for minimum content of epoxy resin and maximum content of filler. Overall, the maximum f_{ti} values are close to those reported by Golestaneh et al. (2010) and about 4 folds higher than of conventional concrete. Reis (2009) shown that the addition of supplementary materials such as textile fibers in BPF based compositions can decrease both f_c and f_{ti}. Thus, the type and the dosage of components play an important role in PC flexural strength performance.

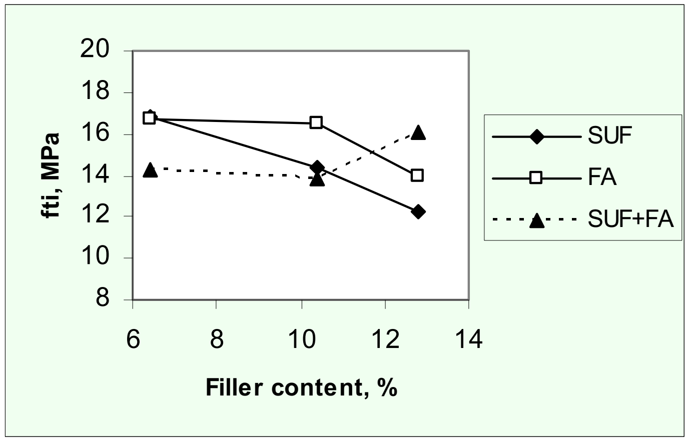


Figure 6. Flexural strength variation for polymer concrete with 12.4% epoxy resin, as a function of filler content

Split tensile strength

Figure 7 presents the split tensile strength (f_{td}) of PC as a function of epoxy resin content, when a minimum content of filler (i.e. 6.4%) is used in PC matrix. As can be seen, f_{td} for BPSF is still lower and slightly decreases with the increase of resin content after 15.6%, than for BPS and BPF that show quite opposite behavior. According to Table 2, f_{td} for BPSF is lower than for BPS or BPF for all PC compositions, except for the mixture 2 where a comparable performance is obtained. For the same resin content, f_{td} diminishes with the decrease of filler content for BPSF and BPF, but slightly increase for BPS (Figure 8). Therefore, the maximum value of f_{td} for BPSF is obtained for minimum content of epoxy resin and maximum content of filler. It should be noted that the maximum obtained f_{td} values are about 2 folds higher than of conventional concrete (3.42 MPa) and about 2 folds lower than of the polymer concrete with silica powder reported in the study of Golestaneh et al. (2010).

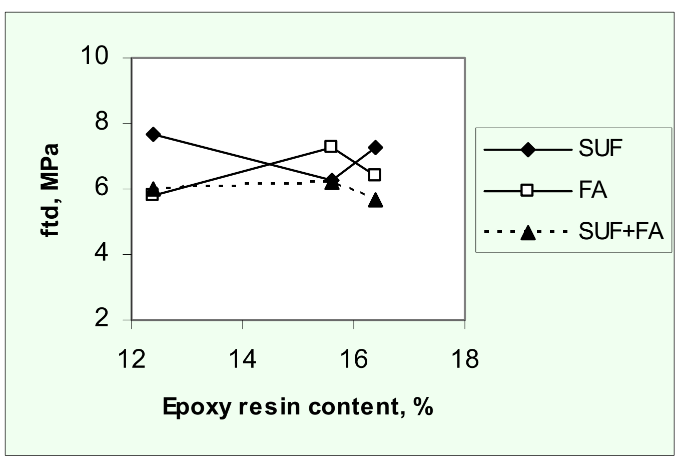


Figure 7. Split tensile strength variation for polymer concrete with 6.4% filler, as a function of epoxy resin content

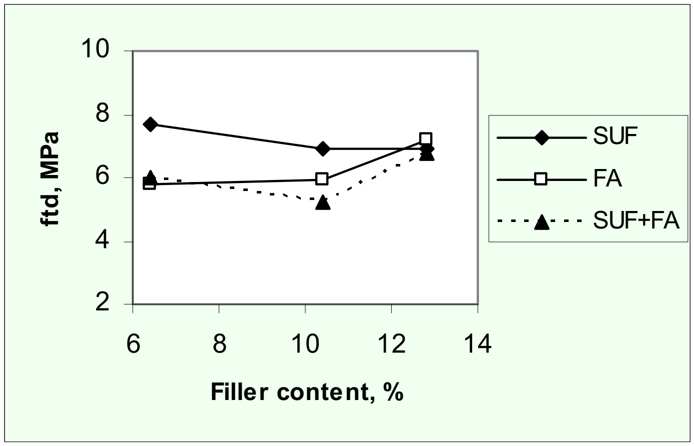


Figure 8. Split tensile strength variation for polymer concrete with 12.4% epoxy resin, as a function of filler content

The behavior of BPSF in compression, flexure and split tensile tests appears to be more close to that of BPF than of BPS. The experimental results shown that for the same dosage of epoxy resin the maximum compressive strength and split tensile strength were obtained for maximum FA dosage, while the maximum flexural strength was obtained for a medium FA dosage. Although the slightly inferior mechanical properties of BPSF suggest a possible adverse effect of SUF on BPSF compactness (possibly due the increase of irregular particles agglomerates), the BPSF performance is still comparable to other PC and superior to ordinary concrete. Moreover, all tests have shown that for realizing composite polymer concrete it is not necessary a high quantity of polymer, thus the total price of the composite can be competitive.

Moreover, the thermal conductivity for ordinary concrete takes values between 1.39-4.33 W/m K. Thermal conductivity for micro-concrete is 0.47 W/m K, for polymer concrete is 0.441 W/m K and for polymer concrete with SUF and FA (containing wastes) is.0.432 W/m K (Bentz et al., 2011). As can be seen, BPSF presents thermal insulated properties superior than ordinary concrete. Thus, replacing ordinary concrete with BPFS in various structures, including farm building applications, could increase also the thermal comfort at lower costs and as well the durability under aggressive environmental conditions.

CONCLUSION. Polymer concrete with SUF+FA (BPFS) has good mechanical properties compared with other types of polymer concrete in terms of both mechanical and thermal properties. Different types of wastes combinations can be used in high dosages as filler in PC composition. The experimental results shown that for the same dosage of epoxy resin the maximum compressive strength and split tensile strength were obtained for maximum FA dosage, while the maximum flexural strength was obtained for a medium FA dosage. Replacing ordinary concrete with BPFS in various structures, including farm building applications, involves several environmental, economical and social benefits. Also, using polymer concrete could enhance the waste management strategy.

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DURABILITY OF DIFFERENT BINDERS IN SYNTHETIC AGRICULTURAL EFFLUENTS

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CSAS11 18 –Topic I: Concrete deterioration, Topic VII: LCA and concrete used in sustainable farm building

ABSTRACT: This paper analyses the resistance of different binders (CEM I, CEMI with metakaolin, CEM III/A, CEM III/C, Calcium Aluminate Cements) in various acids (acetic, citric and oxalic acids) with different aggressiveness toward the cementitious matrix. The effect of a mixture of acids (acetic + oxalic) is also investigated. Alteration kinetics was assessed through mass losses and degraded layer depth measurements. Degradation mechanisms were investigated using electron probe micro-analyzer (EPMA) and X-ray diffraction (XRD). The results point out that the increasing order of binder durability is not the same in the different solutions. The degradation mechanisms are highly dependent on the nature of the binder.
Keywords: synthetic effluents, binder, secondary cementing materials, pozzolanic additions.

INTRODUCTION

Because of its low cost, watertightness, good thermal inertia, etc., concrete is widely used in agro-industrial environments. It is notably used for the construction of structures intended for the collection, storage and treatment of liquid waste (liquid manure, molasses, silage juices, etc.). However, it suffers severe damage, notably linked with acid attacks caused by a wide range of organic acids in the effluents.

The organic acids react with the hydrated and anhydrous phases of the cementitious matrix, mainly leading to the formation of calcium salts. These acids and their Ca-salts have very varied physicochemical properties, which result in significantly different alteration mechanisms and aggressiveness toward cement pastes, e.g. those of citric, acetic and oxalic acids. The issue of the formulation of durable concretes is thus complex as performance must be assessed considering, first, each acid taken separately, for a better understanding of attack mechanisms, and then using mixtures of acid representative of real effluents.

In a previous work, the attack by oxalic, tartaric, acetic and citric acids on CEM I pastes was investigated (Larreur-Cayol et al. 2011a). This study pointed out that the first parameter to be considered to evaluate an acid's aggressiveness was the solubility of its Ca-salts. When the salt is soluble (case of Ca-acetate) it does not protect the cement matrix. However, when the salt is poorly soluble or insoluble (as for Ca-oxalate or Ca-citrate) it is not necessarily protective either. In this case Larreur-Cayol et al. (2011a) showed a correlation between the protective/deleterious effect of the salt and its molar volume. Moreover, the study revealed that other parameters, such as the poly-acidity of the acid, the mesoscopic shape of the salt and its affinity for the matrix, could also influence the aggressiveness of the acids. It was found that oxalic, acetic and citric acids had very different effects on the matrix. The low or null aggressiveness of oxalic acid was considered to be linked with the formation, in a thin outer layer of the cement matrix, of calcium oxalate salt from calcium hydroxide and, presumably, at least a part of calcium silicate hydrates. It was assumed that, thanks to its low solubility and a suitable molar volume, the formation of this salt acted to seal the capillary porosity of the matrix. Acetic acid had intermediate aggressiveness. The attack resulted in an almost complete decalcification of the matrix and in the dissolution of all the hydrated and anhydrous phases. Citric acid was the most aggressive for CEM I pastes. Slightly soluble Ca-citrate salt precipitated but (i) it was expansive (high molar volume compared to that of $Ca(OH)_2$ or C-S-H) and (ii) it did not adhere to the cement matrix and thus was not protective. Finally, it appeared that Portland cement paste did not offer satisfactory resistance to the attack by acetic and citric acids (Larreur-Cayol et al. 2011a).

This paper investigates the behaviour of four types of binders (CEM I, CEM I with metakaolin additions, CEM III/A and C, and Calcium Aluminate Cement) in the above three acids, alone and mixed. The kinetics and mechanisms of alterations were analysed (mass losses, degraded depths, XRD, scanning electron microscope SEM, EPMA) during the immersion tests.

MATERIALS AND METHODS

2.1 Cementitious materials

Paste specimens were made with the following binders: CEM I, CEM I 52.5 N with different metakaolin substitution rates (10, 20 and 30% of metakaolin obtained by a rapid calcination of ground kaolin by the flash process); Calcium Aluminate Cements (CAC 1 and 2); CEM III/A and CEM III/C 32.5 N. The pastes were made with a water/cement ratio of 0.27. They were cast in cylindrical moulds 75 mm high and 25 mm in diameter. The specimens were taken out of their moulds 24 h after pouring and stored in water at 20°C for 27 days. They were then immersed in the aggressive solutions for 3 months. In parallel, control specimens were kept in water at 20°C.

Table 1. Composition of the various raw materials and binders

	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	TiO_2	Na_2O	K_2O	MnO	SO_3	l.o.i.
CEM I 52.5 N	66.8	20.8	4.9	2.9	1.4	0.3	0.1	0.6	traces	2.7	2.1
Metakaolin	1.1	67.1	26.8	2.6	0.1	1.4	traces	n.a.	traces	n.a.	0.8
CEM I+10% MK	60.2	25.4	7.1	2.9	1.3	0.4	0.1	0.5	traces	2.4	2
CEM I+20% MK	53.7	30.1	9.3	2.8	1.1	0.5	traces	0.5	traces	2.2	1.8
CEM I+30% MK	47.0	34.7	11.5	2.8	1.0	0.6	traces	0.4	traces	1.9	1.4
CEM III/A 32.5 R	56.2	26.2	7.2	1.4	3.6	0.3	0.2	0.3	0.1	2.6	1.3
CEM III/C 32.5 N	46.2	32.2	9.3	0.9	6.0	n.a.	0.5	0.5	n.a.	1.6	1.8
CAC 1*											
CAC 2	37.3	4.5	39.3	15.3	1.0	3.0	0.1	0.2	n.a.	0.3	n.a.

l.o.i..: loss on ignition. n.a.: data not available.
The composition of CAC1 will be provided in the final version of the paper. We apologize for the inconvenience.

2.2 Composition of the aggressive solutions

The aggressive solutions were made with a concentration of acid of 0.28 M in accordance with our previous investigations (Bertron et al. 2005, 2007, Larreur-Cayol et al. 2011a) (table 2). This concentration is the highest total concentration of acids found in effluents. Different quantities of sodium hydroxide were added to bring the pH up to 4 (minimum value in the effluents), except for oxalic acid and for the mixture of acetic and oxalic acids because the reaction between sodium hydroxide and oxalic acid leads to the formation of sodium oxalate salt. Na-oxalate is insoluble in water and it thus disturbs the assessment of the binders' performance. The pHs of the oxalic acid solution and of the mixture were therefore kept at their initial values of 0.85 and 1.2. Large amounts of NaOH were added to the citric acid solution because of the poly-acidity of this acid.

Table 2. Compositions of the aggressive solutions

Acid	Acetic	Citric	Oxalic	Mixture (acetic 2/3-oxalic1/3)
Concentration of acids (mol/L)	0.28	0.28	0.28	0.28 (0.1866/0.0933)
Concentration of NaOH (mol/L)	0.06	0.330	0	0
pH	4	4	0.85	1.2

The specimens were immersed in the aggressive solutions in static conditions (no agitation), to recreate the hydraulic conditions occurring in silos, ponds and other effluent storage structures. The durability of the various binders listed in section 2.1 was assessed in each of the 3 solutions: acetic, citric and oxalic acids. In the mixture of acids, CEM I was the only binder tested. Acetic and citric acid solutions were renewed as soon as the pH of one of them reached 4.5. The pH of the oxalic acid and the mixed acid solution did not change over the time of immersion. Nevertheless, the solutions were renewed at the same time as the others.

2.3 Test methods

2.3.1 Alteration kinetics

Relative mass losses and altered depths were monitored over time throughout the experiment. The mass loss measurements were made on two specimens at each solution renewal. Two cement paste cylinders were taken out of the solution, briefly wiped with absorbent paper and then weighed using a weighing device with an accuracy of ±1 mg. Altered depths were measured on two slices of specimens sawn perpendicularly to their axes. Phenolphthalein was sprayed on to the plane sections and its change in colour was the criterion used to distinguish the degraded zone from the sound zone. The cross sections were scanned and the degraded layer depths were measured using Gimp® software.

2.3.2 Degradation mechanisms (chemical and mineralogical)

The chemical changes of the specimens were analysed by electron microprobe (Cameca SX 50, operating conditions: 15 kV, 20 nA) after 1 month of immersion. Two samples were considered for each acid. Two control specimens were also analysed 5 weeks after pouring. To explore the mineralogical changes, samples were analysed by XRD (Siemens D5000, 35 kV, 30 nA) after one month of immersion. A control specimen was also analysed 5 weeks after pouring. The detailed procedures of these tests are given in (Larreur-Cayol et al. 2011a).

3 RESULTS AND DISCUSSION

3.1 Macroscopic observations

Figure 1 shows the aspects of some specimens after 2 months of immersion in the various aggressive solutions.

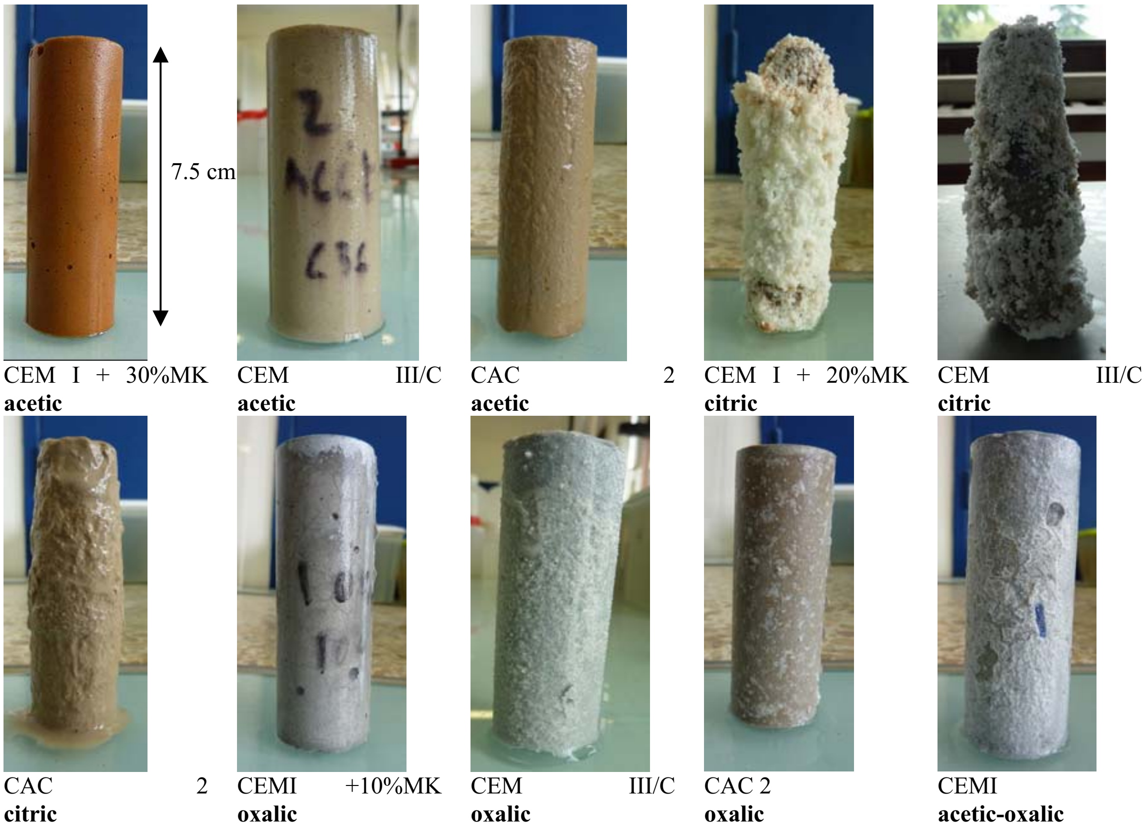


Figure 1: Aspects of paste specimens after 2 months of immersion in the various aggressive solutions (specimens were all 7.5 cm high).

In citric acid, there was rapid erosion of all the samples (CEM I, CEM I+MK, CAC, CEM III). White salts formed on the surface of CEM I+MK and CEM III specimens and accumulated in the bottom of the tank, but they did not adhere to the matrix (they were easily removed by gently scratching the surface). The formation of salt was not visible at the surface of CAC specimens but some salt was found in small quantities at the bottom of the tank.

In acetic acid, CEM I+MK became orange (probably because of ferrous oxide) as was observed in previous studies (Larreur-Cayol et al. 2011a, Bertron et al. 2005, 2007) with pure CEM I. CEM III pastes were whitish, similarly to observations by Bertron et al. (2005, 2007). Neither type of binder was dissolved during the attack and the outer parts of the specimens, although porous and with highly reduced mechanical strength, kept their initial shape. The outer layer of CAC specimens was progressively dissolved in this acid.

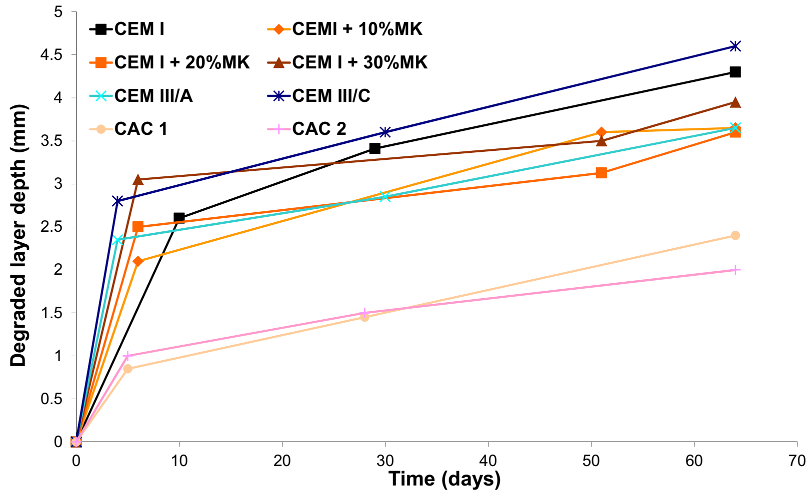
Oxalic acid was not aggressive toward CEM I + MK, CEM III/A and CAC specimens, as had been observed for CEM I by Larreur-Cayol et al. (2011a). But CEM III/C pastes were attacked and macroscopic, non-adherent salts continuously formed on the surface of the specimens while the outer layer was progressively dissolved.

Regarding the mixture of acetic and oxalic acids, a thin layer of white salts was observed all over the surface of CEM I specimens, unlike what was observed in oxalic acid alone.

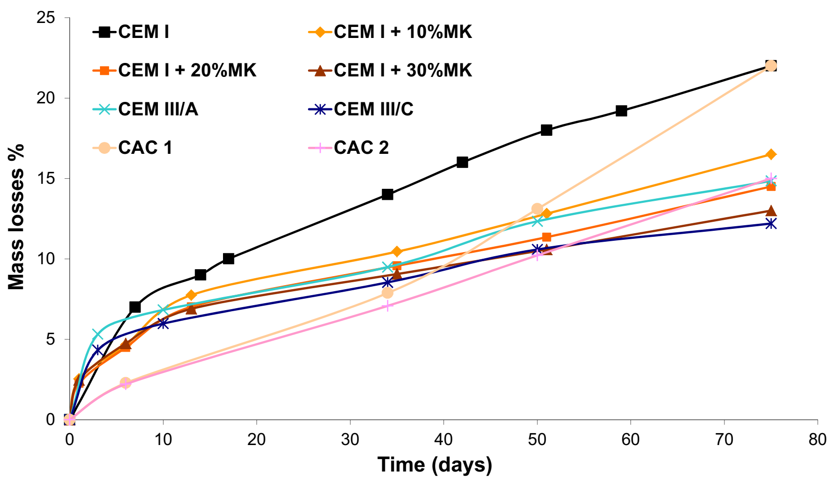
3.2 Degradation kinetics

3.2.1 Acetic acid

Figure 2 shows the evolution over time of mass losses and altered layer depths of the various specimens immersed in acetic acid.



a)



b)

Figure 2. Degradation kinetics of the various binders immersed in acetic acid according to time of immersion: a) degraded layer depths b) relative mass losses

It should be noted first that mass losses and degraded layer depth indicators did not give the same order of durability for the various binders. Notably, CAC cements appeared to be the most resistant of the binders studied if the degraded layer depth indicator was considered. In contrast, their behaviour was similar to that of the others at the end of the experiment when mass losses were considered. Another difference could be observed regarding CEM III/C: it had the lowest mass losses of all the binders but its degraded layer depths were the highest.

Actually, degraded layer kinetics expresses the progression of the degradation front inside the matrix. On the other hand, mass losses are dependent on (i) the location of the degradation front, (ii) the behaviour of the matrix during the attack (dissolution, precipitation of salt or simple release of calcium with preservation of the Al-bearing silica gel skeleton) and (iii) in case of preservation of the Si-gel and no precipitation of salt (case of acetic acid), the amount of CaO of the binder.

The first parameter to be considered should be the degraded layer kinetics as it expresses the progression of the degradation front toward the reinforcements of the structure, which, when corroded, lead to the ruin of the facilities. Mass losses should be considered for a given degraded layer depth. Mass loss should be considered for a given degraded layer depth as it then reveals the intensity of alteration of the degraded layer.

Considering degraded layer depths, CACs were the most resistant, CEM III/C and CEM I were the least resistant. CEM I + MK and CEM III/A had very similar and intermediary behavior.

CEM I showed the largest mass losses because of its highest degraded layer kinetics and highest CaO content among all the binders. The addition of metakaolin reduced mass losses. At the end of the experiment, CEM III/C had the lowest mass losses because of its low calcium content and the stability of slag residual grains during the attack (Bertron et al. 2005). Mass losses of CAC were moderate at the beginning of the experiment but they increased very quickly because of the partial dissolution of the altered layer. Mass losses of CAC1 were far higher than those of CAC2 because the degraded layer of CAC2 was more stable than that of CAC1 during the attack.

The more favourable behaviour of CAC binders may be explained by their mineralogical and chemical composition. The main constituent of CAC cements is CA (monocalciumaluminate), the hydration of which leads to the formation of four hydrated phases CAH_{10}, C_2AH_8, C_3AH_6 and AH_3. During the conversion that occurs for 14 days after the beginning of hydration, the metastable phases CAH_{10} and C_2AH_8 are converted into a stable assemblage (C_3AH_6 + AH_3) (Fryda et al. 2008). AH_3 is stable at pH between 3 and 4. The dissolution of the calcium component of the other hydrates leads to the formation of additional quantities of this phase, which infiltrate the porosity and protect the matrix from further attack (Scrivener et al. 1999).

3.2.2 Citric acid

Figure 3 gives the mass losses over time of all the binders immersed in citric acid. Degraded layer depths are not provided as the grading of the binders was the same, the degraded layers of all the binders tested being dissolved during the attack. After three months, the highest degraded depths were measured on CEM I+30%MK (12 mm, specimens almost totally dissolved), the lowest were measured on CEM III/C (3 mm). Mass losses were higher in citric acid than in acetic acid, which indicated a more severe attack. CEM III/A and CAC were the most resistant while CEM I+MK and CEM I were the least resistant. The addition of metakaolin seemed to improve the resistance of the material up to a 20% substitution rate. Increasing the metakaolin substitution rate from 20% to 30% induced higher degradation rates.

The attack of CEM I paste by citric acid led to the formation of calcium citrate tetra-hydrate salt, $Ca_3(C_6H_5O_7)_2.4H_2O$ (Larreur-Cayol et al. 2011a). The precipitation of the salt, the molar volume of which is very high (518.4 cm^3/mol (Walsdorf et al. 1989)) compared to those of C-S-H and CH (between 108 and 125 and 33 cm^3/mol respectively (Taylor 1990, Tennis and Jennings 2000)), leads to the break-up of the outer part of the cementitious matrix. This, in turn, at least partially explains the very high alteration kinetics of the specimens as the progression of the alteration front is not slowed down by the diffusion of the aggressive species through the altered zone, a phenomenon usually observed for other acids. Moreover, the calcium citrate salt does not adhere to the matrix; it falls from the specimen and collects in great quantities at the bottom of the tank.

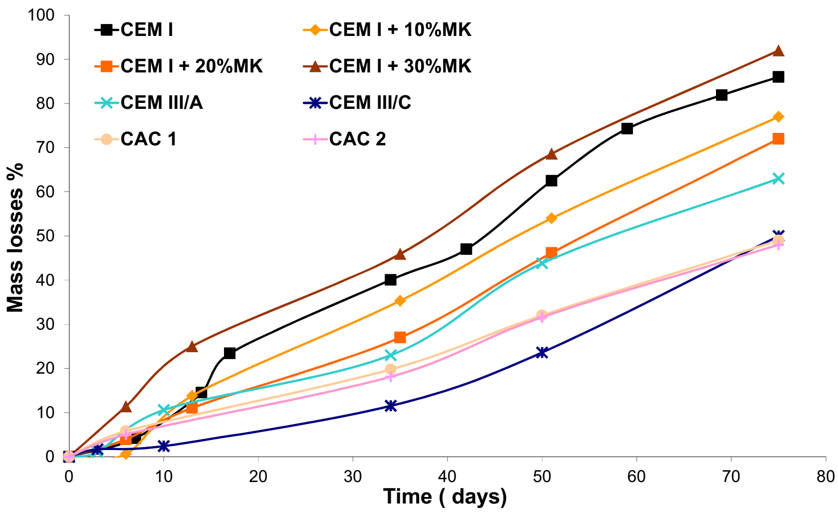


Figure 3. Relative mass losses of the various binders immersed in citric acid according to time of immersion

The better behaviour of CAC toward citric acid attack may be linked (i) to the stability of AH_3, initially present in the matrix and newly formed by the dissolution of other hydrated phases as explained above and (ii) to the molar volume of C_3AH_6 and AH_3 (150 and 156 cm^3/mol respectively (Fryda et al. 2008)), higher than that of C-S-H and CH. The gap between the molar volume of Ca-citrate salts and the reactive phases is reduced as compared to Portland cement pastes. This may explain why the degradation is less intense. The better behaviour of CEM III/C is not understood yet.

3.2.3 Oxalic acid

Figure 4 gives the mass losses over time of all the binders immersed in oxalic acid. Degraded layer depths are not provided as they remained at zero throughout the experiments for all the binders except for CEM III/C, the degraded layer depth of which was 1.2 mm after 3 months of immersion. Oxalic acid was not aggressive for any of the binders except CEM III/C. Neither mass loss nor degraded layer was observed on CEM I, CEM I+MK or CEM III/A. Calcium aluminate cements even increased in mass, which suggested the precipitation of significant amounts of adherent salt. As the pH of the attack was lower here than the pH at which the hydrates C_3AH_6 and AH_3 are stable, they were dissolved, probably to form Ca and/or Al-oxalate salts. Substantial attack of CEM III/C was observed in the first month of immersion (degraded layer of about 1.2 mm and 4% mass loss), the kinetics being strongly reduced during the rest of the experiment. It should be noted that oxalic acid is the least aggressive of the acids tested despite the lowest pH. This underlines the fact that pH is not the only criteria of aggressiveness of an acidic medium contrary to the classification of aggressive environments in EN 206-1 standard.

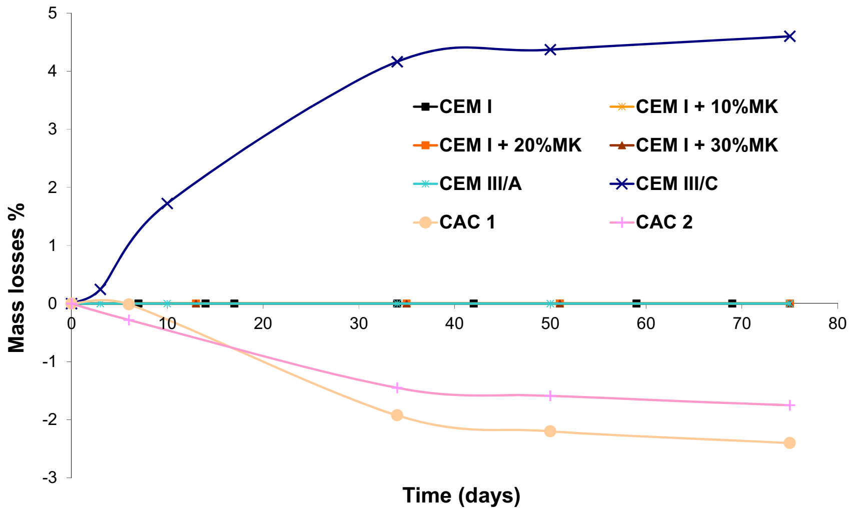


Figure 4. Relative mass losses of the various binders immersed in oxalic acid according to time of immersion

3.3 Degradation mechanisms in oxalic acid

3.3.1 Oxalic acid alone

Figure 5 shows the composition of oxides of a CEM III/C sample immersed in the oxalic acid solution for 4 weeks, according to the distance to the surface in contact with the aggressive solution. Three zones can be observed: (i) Zone 1, or the sound zone, showed no chemical changes; its composition was identical to that of the control sample; (ii) Zone 2 was a transition zone, characterised by enrichment in sulphur oxide SO_3 and slight decalcification. Zones 1 and 2 caused the phenolphthalein to change colour; (iii) Zone 3, or the altered zone, showed lower CaO content suggesting a partial decalcification of C-S-H. The presence of a significant amount of CaO in this zone (contrary to what was observed in citric or acetic acid (Larreur-Cayol et al., 2011a)) indicated the precipitation of calcium salt. This was confirmed by XRD analyses showing the precipitation of Ca-oxalate. The chemical profiles of CEM III/C in oxalic acid were noticeably different from those of CEM I, CEM I+MK and CEM III/A (results not provided), which showed a calcium content that remained unchanged throughout the specimens. For all these binders, XRD showed that Ca-oxalate mono-hydrate precipitated in the specimen because of the dissolution of $Ca(OH)_2$ and, presumably, at least a part of the C-S-H. This result underlines the key role of $Ca(OH)_2$ in the attack by oxalic acid. In cementitious materials incorporating slag, especially at high replacement levels, $Ca(OH)_2$ is consumed to form C-S-H during hydration.

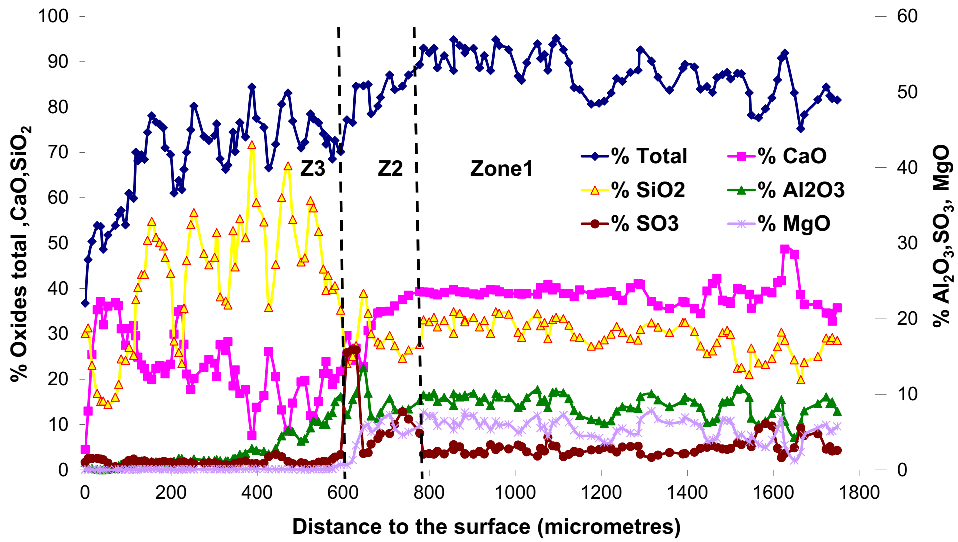


Figure 5. Chemical composition profile of a CEM III/C paste immersed in oxalic acid for one month.

Slag cement pastes are known to be more chemically resistant than OPC paste, notably in acidic conditions, as obtained with acetic acid for example (Oueslati 2011). However, in the case of attack by oxalic acid, the absence of $Ca(OH)_2$ is prejudicial to the durability of the matrix. Actually, the chemical stability of the Ca-bearing phase of the solid (and not only the chemical nature of the cation combining with the oxalate anion to form an insoluble salt) is of prime importance in the impact of the oxalic acid attack on the matrix. For the oxalate salt to have the most beneficial effect, its formation should occur at as high a pH as possible, so that the further hydrolysis of the material caused by hydronium ion released by oxalic acid can be prevented. In cement paste, $Ca(OH)_2$ is the least stable hydrated phase of the paste, its dissolution occurring from pH 12.5 in the interstitial solution (and $[Ca^{2+}]=20*10^{-3}$ M) according to Moranville et al. (2004).

3.3.2 Mixture of acetic and oxalic acids

Figure 6 shows the oxide composition of a CEM I sample immersed in the acid mixture (acetic 2/3, oxalic 1/3) for 4 weeks, according to the distance to the surface in contact with the aggressive solution. The chemical profiles of the CEM I specimens immersed in the mixture of acids show that there was no change in the chemical composition of the matrix and, notably, no decrease in the calcium content of the outer part of the specimen. This is typical of the attack of CEM I paste by oxalic acid alone and clearly different from that of acetic acid (Larreur-Cayol et al. 2011a, Bertron et al. 2005, 2007). This suggests that oxalic acid has a preponderant effect over acetic acid in the attack. This is in accordance with the relative values of the reaction constants of Ca-acetate and Ca-oxalate formation (De Windt et al. 2011, Larreur-Cayol et al. 2011b). Nevertheless, the aspect of the specimens immersed in oxalic acid alone was different from the appearance of those immersed in the mixture of acids, the latter being covered by macroscopically observable salts. The surface of the former was grey and slightly rough but the precipitation of salt, identified as Ca-oxalate mono-hydrate through XRD, was not visible with the unaided eye. Actually, the salts precipitated in the mixture of acids (figure 7) were calcium oxalate mono- and di-hydrate, whewellite and weddelite. Whewellite is the monoclinic form of Ca-oxalate, while the tetragonal weddelite is a less stable form. Weddelite and whewellite very often occur together on the surface of calcareous or non-calcareous historical buildings that have undergone biodeteriation, notably in the Mediterranean urban environment (Jorge Villar et al. 2003, Monte 2003). This difference in the precipitation form of Ca-oxalate between oxalic acid alone and the acetic-oxalic mixture has not been explained yet. However, it should be noted that both forms seem to be protective for the cementitious matrix.

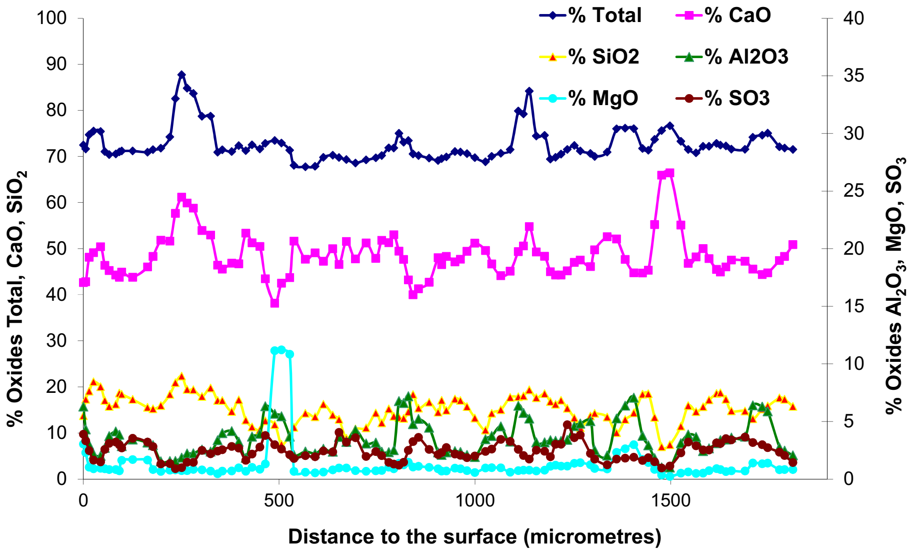


Figure 6. Chemical composition profile of a Portland cement paste immersed in the mixture of oxalic and acetic acids (1/3-2/3) for one month.

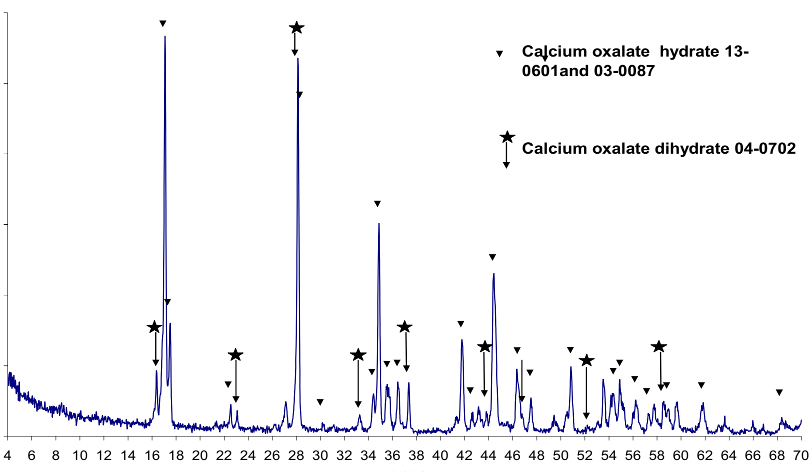


Figure 7: XRD traces of the outer layer of CEM I pastes immersed in the mixture of oxalic and acetic acids (1/3-2/3) for one month.

CONCLUSION This paper underlines the key role of different parameters influencing binders' resistance in acid media, and notably the chemical stability of the cementitious phases involved in the formation of the acids' salts and the difference of molar volume between the salts and the cementitious phases at the expense of which they formed. The decreasing order of durability in acetic and citric acids was as follows: CAC > CEM III > CEM I+MK > CEM I. The addition of MK seemed to reduce the degradation. However, in citric acid, an optimum substitution rate was reached at 20 %; beyond this value the degradation worsened. The better performance of CACs may be linked to the stability of the hydrated phase AH_3 at the pH of the attack. In oxalic acid, CEM III/C was the only binder to be noticeably attacked, probably because of the absence of $Ca(OH)_2$ in the hydrated matrix. The degradation mechanisms in the mixture of acetic and oxalic acids were very similar to those in oxalic acid alone, showing the preponderant effect of oxalic acid in the attack. However, a difference was noted between the two solutions in the nature of the Ca-oxalate salts precipitated.

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AGRO-INDUSTRIAL WASTES FOR THE ACHIEVEMENT OF IMPROVED PROPERTIES IN CEMENT BASED COMPOSITES

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CSAS11030 – **Topic I: Concrete Deterioration.**

ABSTRACT Agro-industrial wastes have recently attracted a great deal of attention as potential alternative of raw material applied to different rural buildings. This paper summarizes the results of some researches carried out by the Rural Constructions Group at the University of Sao Paulo, Brazil focused toward the development of fibre reinforced cement incorporating agroindustrial wastes. Studies have been developed in two main themes regarding (i) ashes with pozzolanic activity as partial substitutes of the ordinary Portland cement (OPC) and (ii) vegetable fibres for the improvement of physical and mechanical performance of such cementitious composites. Some examples of these two groups of residual materials are the use of ashes of sugar cane waste, from rice husk and swine deep bedding, and fibres extracted from the husk of the green coconut, bamboo pulp obtained by organosolv process and recycled Kraft pulp recovered from cement bags. The work shows the procedures of processing of these materials in the way that they can be successfully used in the production of the cement based composites. The extrusion is also evaluated as a potential process for the production of cementitious composites due to the improvements achieved in the mechanical and microstructural behavior. The modification of the matrices by accelerated carbonation was applied in the initial ages after fabrication, resulting in the upgrade of the mechanical behavior of the resulting materials.
Keywords: fibre reinforced cement composite, vegetable fibre, sugar cane waste, bamboo, swine deep bedding, ashes.

INTRODUCTION Composite materials constituted by natural fibres and ashes with pozzolanic activity constitute a current area of interest in the materials science concerning with the sustainability especially for applications in agriculture environment. This work presents the results of some researches carried out by the Rural Constructions Group at the University of Sao Paulo in Pirassununga campus, Brazil, toward the development of cement based materials incorporating agroindustrial wastes, and resulting in potential improvement of the physical and mechanical performance of the resulting elements.

Ashes from deep bedding swine production

The raising of pigs in deep bedding system is an alternative in which the waste is composted *in situ* for the reduction of the risk of contamination (air, water and soil) and better agronomic value (Oliveira, 1999). In the animal production cycles, there is the possibility of burning the resulting bedding material in order to produce thermal energy with the consequent generation of ashes. Studies were carried out with the swine deep bedding ashes (SDBA) as a partial substitute for ordinary Portland cement (OPC) and their potential as pozzolanic addition (Oliveira, 2010).

Fibres and ashes from the cogeneration energy plants

The sugar cane bagasse fibre (SCBF) is one of the most important renewable resource and cellulosic by-products from the agriculture industries in Brazil at the moment. Teixeira (2010) studied the application of such residues in composite materials produced by the extrusion process. This method constitutes and innovation for the production of cementitious composites with the achievement of higher performance if compared with traditional molding processes, as the compaction by vibration. The extrusion method can be associated to several advantages such as: low-cost equipments, large production capacity, low water/cement ratio of the matrix and low porosity of the extruded composites with the consequent improved fibre-matrix interface due to the mechanical compaction of the mixture (Shao et al., 2000).

Bamboo micro-fibre from organosolv pulping

The production of bamboo fibre by the organosolv process was studied by Correia (2011) using the variables time vs temperature in order to find the optimum condition for the pulping process based on the chemical, physical and morphological characteristics of the resulting pulp. The adjustments in the pulp characteristics were compatible to those required for the use as fibrous reinforcement in cementitious matrices. The organosolv pulping method was chosen as an alternative to the Kraft process, due to the use of organic ethanol-water based reagents during the cooking step, resulting in the facility of solvent recovery at the end of the process.

Coir macro-fibres from the green coconut husks

The use of fibres taken from the residual husks after the extraction of the green coconut water was investigated in different formulations containing ordinary Portland cement, rice husk ash and cellulosic pulp from recycled cement bags (Pereira, 2011).

Protection of vegetable fibre in aggressive cement matrix

The mechanism of carbonation can be described as the diffusion of CO_2 from the atmosphere through unsaturated pores of the cementitious matrix. The CO_2 is dissolved in the aqueous phase in the pores and transformed into carbonic acid H_2CO_3, which is dissociated in HCO^{3-} and CO^{3-} ions. Additionally, the calcium hydroxide $Ca(OH)_2$ is dissolved in Ca^{2+} and OH^- ions, resulting in the precipitation of calcium carbonate $CaCO_3$ (Peter et al., 2008). According to Berger and Klemm (1972), accelerated carbonation immediately after molding of the individual cement compounds resulted in the high strength development of the silicate phases. The accelerated carbonation of the cementitious matrix at early ages was investigated as an alternative procedure to mitigate the degradation of the vegetable fibre by the reduction of the alkalinity of the matrix, resulting in the significant improvement of the mechanical properties (Almeida et al., 2010). The carbonation process is also a potential initiative to CO_2 sequestration in the industry, and as an attempt to improve the durability of the resulting vegetable fibre-cement products.

EXPERIMENTAL The methodology developed for each study with applications of pozzolanic ashes and vegetable fibres are summarily described in this work as well as further information can be reached in the literature.

Ashes from swine deep bedding applied in conjunction with chopped sisal fibre

The swine deep bedding was burned in a furnace with controlled heating ramp of 5°C/min and permanence for 3 h at 600°C. The ashes were ground in a ball mill with porcelain jar. The sisal fibre was separated and cut in a knife mill, in order to homogenize the dimensions in approximately 4 mm of length, and then 1.7% by dry mass was added to the formulation. The swine deep bedding ashes replaced 30% by mass of the ordinary Portland cement. The homogenization of the formulations was carried out in a Hypo planetary mortar mixer with nominal capacity of 40 kg. The experimental pads were conformed using a vibrating table and immediately sealed in plastic bags for the two initial days of curing in saturated air followed by immersion in water for the subsequent 26 days. The pads were then cut and the specimens tested in saturated condition at 28 days of age for determination of the mechanical and physical properties (Oliveira, 2010).

Fibres and ashes from the cogeneration energy plants

Composites were produced with levels between 0% and 5% as primary reinforcement of wastes from cogeneration plants. Sugar cane bagasse (*Saccharum officinarum*) fibre used in this study was taken from the Baldin Agroenergia plant in Pirassununga, estate of Sao Paulo, Brazil. Extrusion was used in the production of cementitious composites with different cross section geometries and small scale production. The matrix was composed of CPV ARI cement according to Brazilian Standards NBR 5733 (1991) (equivalent to type III cement, ASTM C150, 2009), crystalline silica "filler" and sugar cane fibre. Unbleached eucalyptus Kraft pulp was applied as the secondary reinforcement. The amount of 1% by mass of hydroxypropyl methyl cellulose (HPMC) and carboxylated polyether (surfactant) were added as rheological modifiers to promote pseudo-plastic behaviour of the cement paste which, in turn, enabled the extrusion process. The Auger type extrusion vacuum helical screw equipment (Gelenski brand, MVIG-05 model) was used for the lab production. The specimens were immersed in water for curing. Mechanical and physical performances of the cement composites were evaluated at 28 days of age and after 200 accelerated ageing cycles (Teixeira, 2010).

Bamboo organosolv pulp in Portland cement with mineral addition

The cementitious composites were produced with 25% of metakaolin by mass and reinforced with 8% of bamboo organosolv pulp. The best pulping condition was the cooking temperature of 190^{o}C for 2 h. The lab pads were produced using the slurry vacuum de-watering followed by pressing technique, as a crude reproduction of the Hatschek process (Savastano Jr. et al., 2000). After the initial period of wet curing the composites where subjected to 200 accelerated ageing soak & dry cycles for the durability studies (Correia, 2011).

Coir macro-fibres from the coconut husk and pozzolanic addition

The green coconut waste fibre was investigated with different proportions of ordinary Portland cement (OPC), rice husk ash (RHA) as pozzolanic addition, and cellulosic kraft pulp from recycled cement bags. The fibres were donated by the Coco Verde Industry from Rio de Janeiro, Brazil (Pereira, 2011). The composites were molded using the slurry dewatering and pressing technique and sealed in plastic bags for the two initial days of curing in saturated air followed by immersion in water for the subsequent 26 days. In the completion of 28 days, the mechanical and physical characterizations were carried out.

Accelerated carbonation technique to improve durability

The study of early-stage accelerated carbonation of the cementitious matrix used 10% (dry mass) of unbleached eucalyptus cellulosic pulp, 77.2% of Portland cement type CPV-ARI (Brazilian Standards NBR 5733, 1991), and 12.8% of ground carbonate material. After molding by the slurry dewatering and pressing technique the composites were subjected to the accelerated carbonation after two days from manufacturing. The carbonation treatment was performed for three days in the climatic chamber that was totally saturated with successive CO_2 cycles. The composites were then kept in saturated air at 25°C for curing until 28 days from manufacture. Mechanical and physical properties were assessed after 28 days of age and 200 accelerated ageing cycles.

Mechanical bending test

Modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE) and specific energy (SE) of the composites were evaluated by four point bending configuration test as detailed by Savastano Jr. et al. (2000).

Physical characterization tests

Water absorption, apparent porosity (AP) and bulk density (BD) were obtained from the average of ten specimens for each mix design, following the procedures specified by ASTM C 948 (1981) Standards. Soak/dry accelerated ageing cycles

The accelerated ageing test involved comparative analysis of physical and mecha

composites performance, before and after soak/dry cycles. Specimens were successively immersed in water at 20 ± 5°C during 170 min, followed by the interval of 10 min, and then exposed to temperature of 70 ± 5°C for 170 min in a ventilated oven and finally left to the final interval of 10 min. This procedure was based on recommendations of the EN 494 (1994) Standards and was performed up to 200 cycles in the attempt to compare the degradation in the specimens for each mix design.

RESULTS AND DISCUSSION

Densification of composites containing mineral addition

The composites prepared with swine deep bedding ashes (SDBA) as a partial substitute for ordinary Portland cement (OPC) and short sisal fibres presented higher toughness (specific energy of 100 J/m^2) than the reference composites without SDBA and sisal fibres (specific energy of 40 J/m^2) at 28 days of age.

Table 1 shows the results of bamboo pulps analyzed by a Pulptec™ MFA-500 Morphology Fibre and Shive Analyser MorFiTrac, as described in details by Tonoli et al. (2009). This equipment consists basically of a charge-coupled device (CCD) camera that captures the images of the fibre/water suspension and records them for further analysis with the software that carries out the measurements and statistical corrections. Test cellulose sheets were prepared in a Pulmac ASF-C1 for the individual fibre strength test and the fibre bonding index, which was measured in a zero-span tester Pulmac Z2400-C1.

Table 1 – Physical characterization of the bamboo pulp (Correia, 2010).

Parameters	Pulping time 2 h
Length weighted (mm)	0.8
Width(µm)	19.8
Fines (%)	62.3
Zero-span tensile strength index (N.m/g)	204.6

The composites with bamboo cellulosic pulp presented average value of modulus of rupture equal to 7.7 MPa after 8 days of curing which increased to 8.3 MPa after 200 accelerated ageing cycles due to the reduction of apparent porosity. The corresponding average values of the apparent porosity decreased from 27% by volume at 8 days of age to 19% by volume after 200 accelerated ageing cycles as a result of the precipitation of the hydration products into the pores of the matrix and of the fibre-matrix interface.

Effect of pozzolanic activity in the matrix with coir macro-fibres

Composites containing residues from green coconut fibre as the main reinforcement for cement based composites presented better values of modulus of rupture (MOR) at 28 days – 15.7 MPa in average – when using 50% of rice husk ash as partial replacement of cement. This mechanical result was considered significantly superior if compared to MOR equal to 12.5 MPa for the composites without the green coconut fibre.

Effect of extrusion processing on behavior of composites with fibres and ashes from the cogeneration energy plants

Table 2 shows the mean values and standard deviations of specific energy (SE), water absorption (WA) and bulk density (BD) of composites reinforced with sugar cane fibre with 28 days of curing and after 200 ageing cycles.

Table 2 - Mean values and standard deviations of specific energy (SE), water absorption (WA) and bulk density (BD) of composites reinforced with sugar cane fibre. Composites tested after 28 days of curing.

Amount of fibre (% by mass)	Condition	SE (J/m^2)	WA (%)	BD (g/cm^3)
0	28 days	378 ± 27	16.54 ± 0.29	1.73 ± 0.01
5		630 ± 51	18.76 ± 0.38	1.64 ± 0.01
0	200 cycles	280 ± 111	11.64 ± 0.77	1.78 ± 0.01
5		404 ± 58	13.53 ± 0.60	1.66 ± 0.01

The extruded composites with 5% of SCBF presented higher fracture energy, leading to higher specific energy (SE) if compared to the reference without fibre. Mechanical results of the composites after 200 ageing cycles indicate the pore refinement and densification of the fibre-matrix transition zone due to the continued hydration of the cement matrix. The re-hydration products of cement phases around the SCBF that occurred through the cycles of ageing explain the higher bulk density of the composite (Table 2).

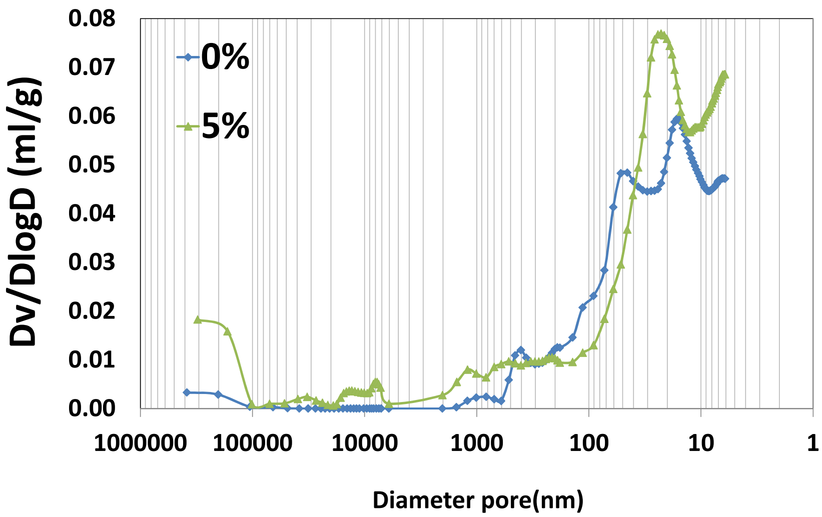


Figure 1 shows the behavior of the pore size distribution of cementitious composites extruded with 0% and 5% by mass of SCBF. Peaks between 6 and 40 nm showed great variation between the two different levels of reinforcement SCBF. Composites with 5% of fibre had higher peaks in relation to plain matrix without fibre. This statement can be explained by the fibres inside the composite which leads to higher porosity and therefore to major defects.

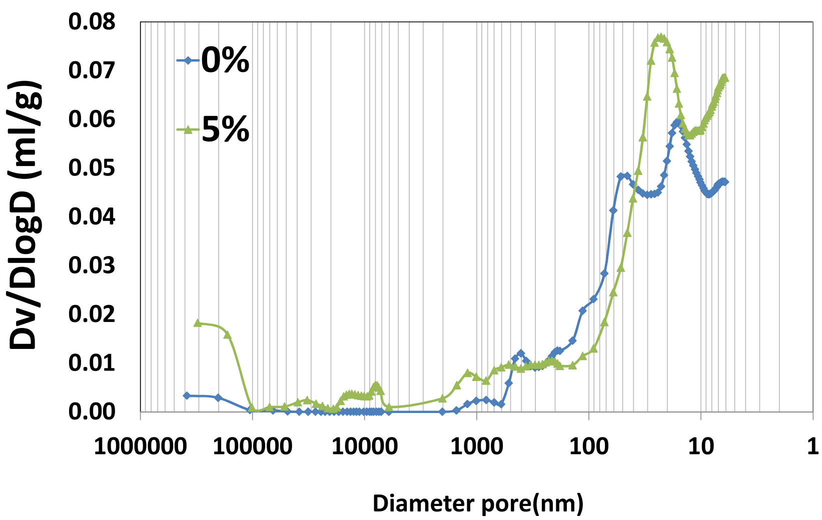


Figure 1 – Mercury intrusion porosimetry (MIP) in composites extruded with 0% and 5% of sugar cane bagasse fibre with 28 days of age.

The peaks between 6 and 100 nm are related to the typical pores distribution of the matrix formed during hydration. These results were also found by Tonoli et al. (2007) that studied fibre cement with different levels of refining of cellulosic fibres. The range between 10^4 and 10^5 nm (large pores) can be attributed to air entrapment due to the inclusion of high levels of SCBF. The range from 100 to 10000 nm diameter was associated to low porosity, contrarily to results presented by Tonoli et al. (2007) that studied fibre cement boards with different levels of refining of cellulosic pulps. This difference can be explained by the manufacturing based on the extrusion process that submits the mixture to high pressure promoting its compaction and decreasing of pore sizing especially in the region close to the external surface.

Protection of vegetable fibre in aggressive cement matrix

Early age accelerated carbonation cured composites presented average value of modulus of rupture of 18.2 MPa and for the non carbonated samples the average value was 9.1 MPa. Figure 2 shows the typical stress vs. specific deflection curves obtained from the mechanical bending test. The carbonated composites presented improved mechanical behavior at 28 days and after 200 ageing cycles.

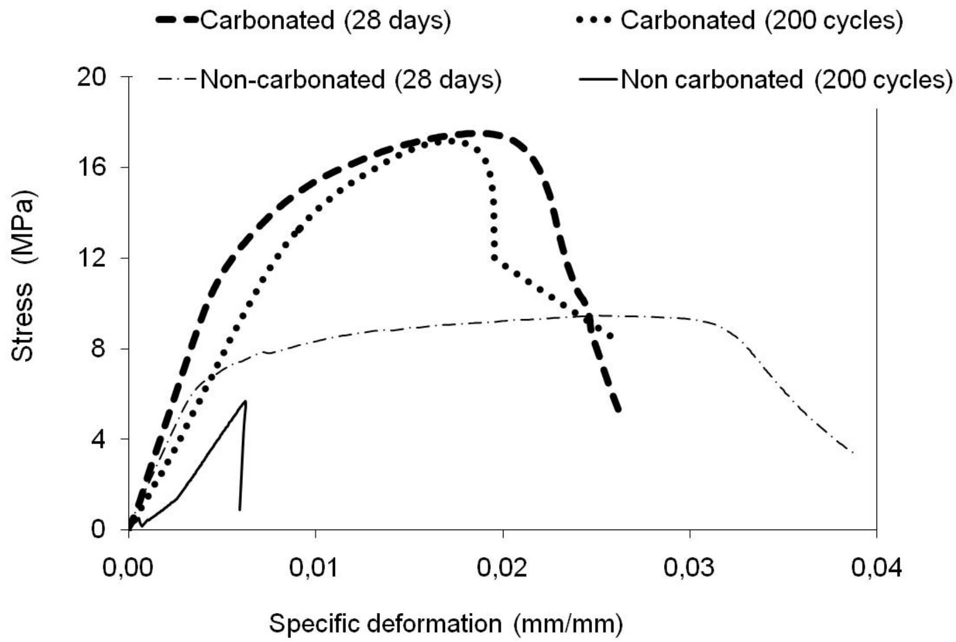
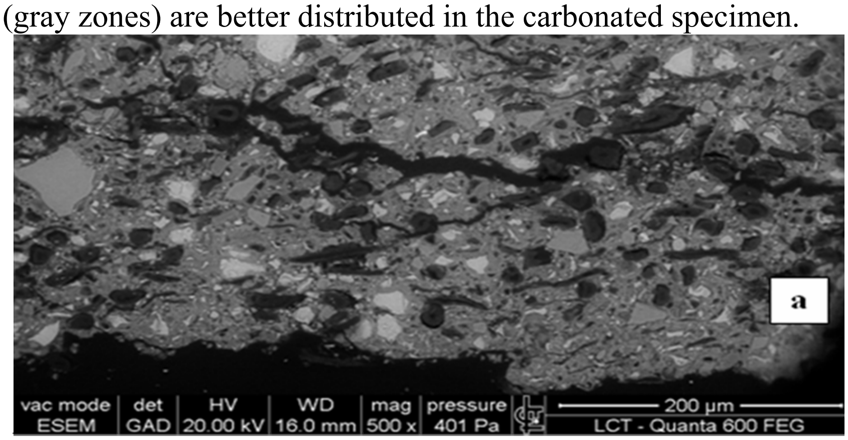


Figure 2. Typical stress vs. specific deflection curves of accelerated carbonated and non-carbonated composites.

The effect of the accelerated carbonation in the microstructure was examined using environmental scanning electronic microscopy (ESEM) of polished cross-section, in the gaseous analytical detector (GAD) mode. The preparation of the samples is described in Savastano Jr. et al. (2005). The polished cross-section observed in Figure 3(b) shows the microstructure of the carbonated specimen more homogeneous and compact (due to the lower amount of voids represented by the darker irregular regions of the micrograph) in comparison to the non-carbonated specimen at Figure 3(a). The cement hydrated products (gray zones) are better distributed in the carbonated specimen.



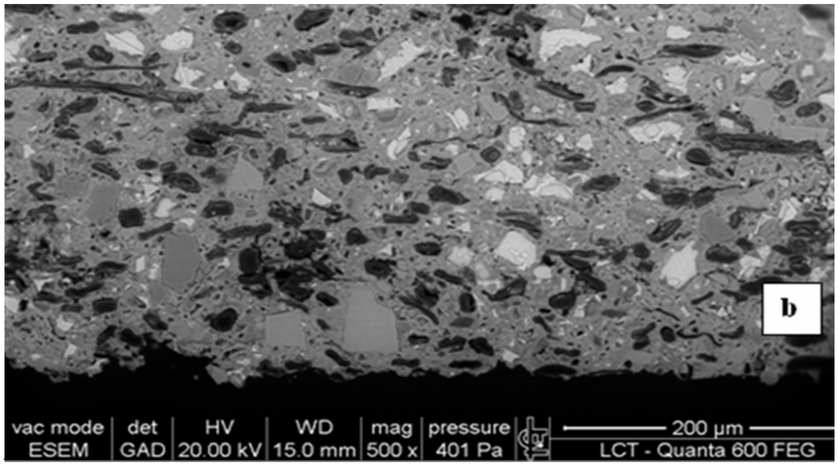


Figure 3. ESEM-GAD micrographs of the polished cross-section of the (a) non-carbonated and (b) carbonated composites.

The carbonation process at early ages resulted in the denser matrix, lower porosity and decrease in the $Ca(OH)_2$ content, what can explain the improvement in the achieved results (Almeida et al., 2010).

CONCLUSION The studies carried out demonstrated that different residues can be added to the cement based materials for a sustainable agriculture using the appropriate manufacturing processes. These examples show the possibility of improving the physical and mechanical properties of the composites for specific applications, including rural constructions. The following materials from renewable resource showed potentialities for application:

- The composites prepared with swine deep bedding ashes (SDBA) replacing 30% by mass of Portland cement (PC) and containing 1.7% of short sisal fibres presented more than twice higher toughness than the reference matrix without SDBA and fibres in the initial ages (28 days).
- Coir macro-fibres from the coconut waste husk determined an increase of more than 25% in the modulus of rupture in the initial ages of composites using 50% of rice husk ash as partial replacement of Portland cement and cellulosic pulps from recycled cement bags as processing fibres.
- The average value of modulus of rupture of the composites reinforced with bamboo organosolv pulp was increased in approximately 8% after 200 soak & dry accelerated ageing cycles, due to the precipitation of products of hydration into the pores of the matrix and increased adhesion between the matrix and the micro-fibres.
- Fibres and ashes from the cogeneration energy plants applied to the extruded composites with 5% of SCBF presented higher fracture energy and specific energy (approximately 42% for the composites after 200 ageing cycles) compared to the reference without fibre.
- Protection of vegetable fibre in aggressive cement matrix by early age accelerated carbonation contributed to significant improvement of the mechanical behavior and durability after 200 accelerated cycles, as a result of the denser matrix, lower porosity and decrease in the Ca(OH)2 content.

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Storage Structures and Rural Pavement

Section 2

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DESIGN RECOMMENDATIONS FOR REINFORCED CONCRETE CYLINDRICAL MANURE TANKS

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CSAS11035 – Topic VI: Air and Water Tightness in Concrete Structures

ABSTRACT In Canada 14% of all livestock farms have liquid manure storage systems; 77% of the storage tanks used are concrete and 75% of these concrete tanks have no cover. The design of cylindrical reinforced concrete manure tanks does not appear to be covered by any formal standard or building code in Canada. The Province of Manitoba has adopted the provisions of ACI 350, "Concrete Sanitary Structures". However, American standards do not fit well with the Canadian concrete design code CSA A23.3. Furthermore, ACI 350 was written for and by municipal engineers and tends to be quite conservative. This paper is concerned with the selection of the wall thickness and wall reinforcement for cylindrical liquid manure tanks in which cracking of the wall must be kept to a minimum to prevent leakage. The various loads involved in the design of the wall are reviewed. Design criteria are presented to limit the circumferential tensile stress in the concrete from lateral wall pressure, shrinkage and temperature gradients in the wall; to limit the tension in the hoop reinforcement; and to limit width of cracks in the concrete if they do occur.
Keywords: Concrete, cylindrical manure tank, structural design.

INTRODUCTION Many farm operators find it necessary to incorporate a manure storage facility in their manure handling system because manure can not be applied to the land all year around. Many of these storage facilities are reinforced concrete tanks large enough to store the manure produced over six to nine months. The determination of tank size is described in detail in the Canadian Farm Buildings Handbook (Anon., 1988; Darby, 2003).

In a survey of some 16,000 livestock farmers in Canada (Bourque & Koroluk, 2003) it was found that 14% had liquid manure storage systems. The hog farmer had the highest proportion at almost 86%; beef cattle farmers used almost exclusively solid manure systems.

Quebec had the highest percentage of farms with liquid manure storage systems because of the prominence of hog and dairy industries. The construction of liquid manure tanks in the prairie provinces has been steadily increasing in recent years. Darby (2003) provides an excellent summary of the design specifications and standards in Canada.

Cylindrical reinforced concrete structures have for years provided an economical and serviceable container for the storage of liquid manure. Typically, the height of the wall is small compared to the diameter of the tank and the walls are subjected to a combination of circumferential tension forces and vertical bending moments. The design and construction strive to have loads that are axisymmetric because non-axisymmetric loads are very difficult to deal with on these thin shell structures.

Non-prestressed reinforced concrete design of structural components neglects the contribution of the concrete in tension. The concrete is assumed cracked in the tension zones under service loads and the reinforcing steel takes care of the tensile stresses present. However, significant cracks in containers with liquid manure could leads to leakage of the content and pollution of the environment, especially the groundwater. Hence, the National Farm Building Code (CCBFC, 1995) requires that "manure storage tank walls shall be designed and constructed to prevent leakage of the contents". Some provincial regulators, like Ontario, require a second barrier in the form of liners.

The objective of this paper is to review the various loads that a cylindrical tank wall must be able to resist and to address the question of support condition that might be appropriate at the bottom of the wall. Design aids for determining hoop tensions will be referenced.

WALL LOADS The following are the loads, other than dead load, that must be considered in the design of a liquid manure tank wall:

- Outward horizontal liquid pressure from the manure at an equivalent fluid density of 10 kN/m^3. The tank should be assumed filled to the top of the wall unless there is a positive overflow device preventing this from happening.
- Inward horizontal soil pressures based on the equivalent fluid densities listed in Table 1. Below the assumed groundwater table the equivalent fluid density for the first three materials in Table 1 may be taken as 50% of those shown. Of course, the water pressure below the water table must be added to the soil pressure. The equivalent fluid density of poorly drained soft silts and clays includes the liquid pressure.

Table 1. Equivalent fluid density for different soil types, kN/m^3 (CCBFC, 1995).

Soil Type	Equivalent fluid density
Clean sand and gravel, well-drained	4.7
Sand and gravel with fines, restricted permeability	5.7
Silt, residual silts and clays	7.0
Soft silts and clays, poorly drained	16.0

Inward hydraulic pressure from groundwater increasing with depth below the highest known groundwater table at the rate of 9.8 kN/m^2 per m depth. If there is uncertainty about the highest groundwater table it is prudent to assume it to be at the level of the local undisturbed ground level (exclusive of any berming). This could be reduced if granular backfill and a perimeter drainage system is used.

Uplift on all parts of a tank below the assumed design groundwater table. If a manure tank can be emptied during periods of high groundwater table, it is necessary to provide some means of relieving the water pressure. This can best be accomplished by providing a compacted granular fill base for the floor and a sub-floor drainage system tied to a perimeter drain. As an added measure of safety, a pressure relief plug can be formed into the floor. If the drainage system does not relieve the upward hydraulic pressure on the floor because of a blockage or a submerged outlet, the plug should pop up and allow water to enter the tank. If this happens, care should be taken to ensure that the plug has been properly reseated before the tank is put back into service.

The National Building Code (CCBFC 2005) and the National Farm Building Code (CCBFC 1995) indicate that where the backfill may be subject to vehicular loads within 1.5 m of a manure tank wall, such as manure tankers or trucks, the walls must be designed for a horizontal surcharge load of 5 kPa, applied uniformly below ground level.

Weight of the manure on the footing and/or floor.
Uniform snow and rain loads and other live loads if the liquid manure tank is covered. Snow and rain loads will depend on location (CCBFC, 2005). If a storage tank cover is not exposed to vehicular traffic it must be designed for the dead plus snow loads, or dead load plus 2.0kPa, whichever is greater. If the tank cover may receive vehicular traffic, the minimum uniformly distributed live load for farm machinery traffic is 7.0 kPa. Where it is anticipated that the cover will be occupied by either loaded trailers, trucks or farm tractors having a mass in excess of 6000 kg (including the weight of mounted equipment),the live load must not be less than 10 kPa (CCBFC, 2005). Alternatively, a concentrated wheel load of 23 kN per wheel, applied over an area of 750 mm by 750 mm located so as to cause maximum effects must be considered.

Forces caused by expansion of ice that may form on the surface of the manure. Although the 1995 NFBC specifies that ice loading must designed for in areas of the country where ice forms in liquid manure tanks, there is little guidance as to the magnitude of loading except for climates similar to that of the Quebec City area. Papers by Jofriet and Johnson (1995b) and by Jofriet et al. (1996b) dealt with ice loading in some detail.

Temperature gradients in the tank wall and creep and shrinkage of the concrete.

LOAD COMBINATIONS When designing a tank wall, the following major live load combinations must be considered:

- Outward pressure of the manure on the wall plus ice pressure. The inward soil loading on the wall may not be used to offset of the manure pressure unless thoroughly compacted granular backfill is used (CCBFC, 1995). Cohesive soils when used for backfill tend to shrink away from the wall making their support of the manure pressure for the life of the structure uncertain. If compacted granular backfill is used the maximum resistance that may be subtracted from the manure pressure should be a low estimate of the active pressure. Inward groundwater pressure should never be used to offset manure pressure. Of course, if the tank is to be tested for leakage before backfilling, the outward liquid pressure will act alone.
- Inward pressure on an empty tank resulting from a combination of ground water pressure, the soil backfill and the surcharge due to vehicles travelling within 1.5 m of the wall.
- In both load combinations other loads must be considered such as dead loads, live load reactions on the wall from a possible tank cover, temperature variations in the structure and structural components, creep and shrinkage of the concrete. In the second load combination uneven levels of backfill must be taken into account. This is very difficult to analyze and the author recommends that backfill levels be kept the same all around a cylindrial tank both during construction and after.

INTERNAL FORCE ANALYSIS. The inward soil pressure from the backfill and the groundwater pressure will cause a circumferential compression in a cylindrical tank wall. Providing these loads are uniform around the tank perimeter, the hoop compression will be uniform. For concrete tanks this hoop compression and the resulting hoop compressive stresses are small compared to the compressive strength of concrete. For instance, for a 50 m diameter, 2.5 m deep tank backfilled with a poorly drained soft silt or clay, the maximum compressive stress will be about 2 MPa. For a wall made of 30

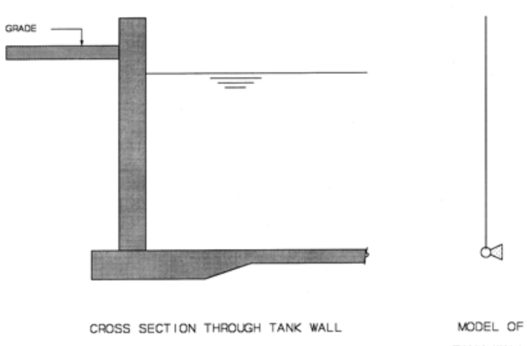
MPa concrete this is not excessive providing the inward pressure is approximately uniform around the circumference of the tank. If not, the wall may buckle much like a column or similar compression member.

In the case where a tank wall is supported at the top by a cover as well as at the bottom, the wall will function mainly as a vertical beam spanning between roof and footing. A conservative assumption for determining the span moment is that the wall is hinged top and bottom; however some fixity should be assumed at the bottom of the wall and the resulting bending moment should be used to design the vertical dowels from the footing. The hoop tension will be negligible. The determination of the bending moments from the outward manure pressure and the inward soil/groundwater pressure is simple and does not deserve further discussion.

The majority of tanks do not have a cover and these need to be considered in more depth. The outward manure pressure causes circumferential tension in the cylindrical wall and the hoop tension needs to be resisted by reinforcing steel, and to a certain extent by the concrete (Jofriet et al, 1987). An accurate estimate of the hoop tension in the wall and its distribution over the height is essential. Once the hoop tension is determined the hoop reinforcing steel and the wall thickness can be determined using the recommendation made by Jofriet et al. (1987).

The hoop tension is a function of the outward pressure on the wall and on the manner in which the wall is assumed supported at the bottom, i.e. the boundary condition at the bottom of the wall. If the floor of the tank and the wall footing are a homogeneous unit placed before the tank wall is constructed (Fig. 1) and if the floor is suitably reinforced to withstand the radial reaction from the wall there will be very little opportunity for the wall to displace radially at the bottom and a hinged support is a good assumption. The hoop tension resulting from the manure pressure can then be determined with the aid of the appropriate table in PCA (1993).

A common construction method for liquid manure tanks is to have the wall and footing separated from the tank floor by a joint allowing the wall and its footing to move independent of the floor (Fig. 2). The advantage of this type of construction is that the floor can be placed after the wall and that unforeseen uplift pressures underneath the tank only affect the floor and not the entire tank.



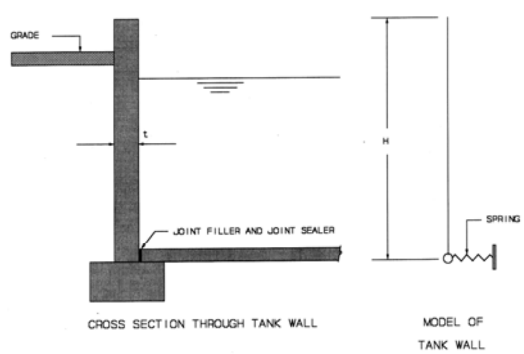


Fig. 1 Cross section of wall integral with floor Fig. 2 Wall with independent floor slab

Some (e.g. Mullen, 1981) have assumed that the wall can slide freely at the bottom so that the hoop tension at each level in the wall equals the pressure at that level multiplied by the tank radius and the radial reaction at the bottom of the wall is zero. This assumption is not accurate because:1) there is always some horizontal reaction at the bottom of the footing from friction and 2) the footing at the bottom of the wall is larger and hence stiffer than the wall providing additional resistance to free radial displacement.

A hinged assumption with zero radial displacement, on the other hand, is not realistic either if the construction procedure is as indicated in Fig. 2 or if the floor in Fig. 1 is not reinforced and likely to crack. The "real" boundary condition therefore lies somewhere between a hinged joint and a perfectly sliding support.

The horizontal reaction from friction is a function of the vertical force in the wall from dead load and downward friction from the backfill. The radial reaction may be as high as 50 to 75 percent of the vertical force. The effect of the footing stiffness exceeding that of the wall is equivalent to a radial spring support at the bottom of the wall (see Fig. 2). Assuming that there is a radial shear force, F_r (N/m), at the wall-footing interface, the hoop tension in the footing,

$T_f, = F_r D/2$: (1)

in which D is the tank diameter. The hoop tensile stress in the footing can be obtained by dividing the hoop tensile force by the footing area, A_f, and the hoop strain in the footing can be calculated, assuming linear elastic behaviour, as $F_r D/(2A_f E_c)$ where E_c is Young's modulus of concrete. The radial displacement of the footing, and of the bottom of the wall, due to F_r alone is the hoop strain multiplied by the tank radius or $F_f D^2/(4A_f E_c)$

If the footing is represented by a linear spring with stiffness k' (N/m per m circumference), then a radial reaction F_r would result in a radial displacement F_r/ k'.and thus:

$k' = 4A_f E_c / D^2$ (2)

Radial springs with spring stiffness k' N/m per metre (Eq. 2) must be applied uniformly around the perimeter of the tank wall at the bottom. The total spring stiffness, k, is determined by multiplying k' by the circumference of the tank, πD:

$k = 4\pi A_f E_c / D$ (3)

Hoop tension in the wall is simple to determine assuming a sliding base. When the wall base is assumed hinged, hoop tension can be determined from tables in "Circular Tanks without Prestressing" (PCA, 1993). When the support at the bottom of a tank wall is a radial spring the hoop tension is more difficult to find. A design-aid based on a number of linear eleastic axisymmetric finite element analyses in a cylindrical wall with height H, diameter D and wall thickness t are presented by Jofriet and Johnson (1995a).and Jofriet et al. (1996a). The wall load is a triangular hydraulic load (liquid weight w kN/m^3) increasing from zero at the top of the wall to wH kN/m^2 at the bottom. For each of the 10 tank geometries expressed by the non-dimensional ratio H^2/Dt (0.4,0.8,1.2,1.6,2.0,3.0,4.0,5.0,6.0 and 8.0),four spring stiffnesses were considered ($k = c\pi t^2 E_c / D$.where c was 9, 18, 36 and 72)

WALL DESIGN. The wall of a cylindrical cast-in-place concrete manure tank without a cover is circumferentially predominantly a tension member and vertically a bending member. Once the hoop tension has been determined, it can then be used to select a wall of adequate strength. A design criterion is required to avoid failure and to reduce to a minimum the development of vertical tensile cracking of the wall. Alternatively, the concrete silo or tank can be equipped with an effective waterproof liner. A cost analysis can provide the most economical solution.

To guard against yield of the reinforcing steel, i.e. failure of the wall,, an ultimate strength design assuming a concrete cracked section will provide the amount of circumferential reinforcing steel at each level of the wall. The load factors should be as specified in the current building code.(e.g. CCBFC 2005) and the strength reduction factor of 0.85 should be applied to the yield strength of the reinforcing steel. The circumferential steel can be evenly distributed between in- and outside face of wall.

The Portland Cement Association publication for the design of non-prestressed tanks (PCA 1993) suggests that the wall thickness of reinforced concrete tanks should be determined by limiting the circumferential tensile stress in the concrete. Many design engineers appear to neglect this concept, and the recommendations of ACI Committee 350 (2006) do not make direct reference to this procedure.

Using this criterion, the wall thickness can be determined by assuring that the tensile strength of the equivalent concrete cross section ($A_c + (n-1)A_s$), multiplied by a strength reduction factor,.is greater than the maximum tensile stress in the concrete due to hoop_ tension effects, including lateral manure pressure, ice pressure if appropriate, shrinkage. creep, and thermal gradients, and possibly reduced by soil pressure. Jofriet et al. (1987) recommended a direct tensile strength of $0.32(f'_c)^{2/3}$ (f'_c is the concrete 28-day cylinder strength) and a strength reduction factor of 0.75. Jofriet et al. (1987) have also made recommendation for shrinkage and creep, and for thermal gradient stresses. The load factors should be as specified in the current building code.

Tensile stresses in the wall due to shrinkage are a function of the percentage reinforcing steel in the wall. These unavoidable tensile stresses can be kept to about 0.5 MPa by limiting the percentage steel to 1% (Jofriet et al., 1987), i.e. the wall thickness should be selected such to limit the amount of hoop steel to 1% of the concrete cross-sectional area.

Temperature gradients can also add considerably to the tension stresses in the wall, especially if the heating or cooling is non-uniform around the perimeter (Godbout et al., 1995). Much of this can be avoided or minimized by backfilling to the top of the wall, realizing that this means safety fencing on top of the wall.

Vertical steel will be required to resist bending moments caused by inward and outward radial loads, and by temperature gradients. The design of the vertical steel will be a standard bending member design. The difficulty is what boundary conditions to base the bending moments on. Construction as in Fig. 1 will lead to considerable bending moments at the wall/footing interface. A fully fixed assumption is of course conservative, but safe; moments can be obtained using PCA (1993). Even if the construction is as pictured in Fig. 2 a prudent designer will assume a certain amount of fixity at the bottom of the wall. Much smaller moments of the opposite sign will occur somewhere between footing and top of wall; a conservative assumption for the determination of these moments is that the wall is hinged at the bottom.

The reinforcing steel should be distributed in such a way as to limit crack widths. Cracking can occur owing to lack of concentricity of the tank. variations in wall thickness, and moments in the^tank wall due to unsymmetrical heating and shrinkage. Reasonable limits on crack widths are those suggested by ACI Committee 350 (2006):

W_{max} < 0.25 mm for noncorrosive liquids
W_{max} < 0.20 mm for corrosive liquids

where W_{max} = maximum crack width. Both lateral load and restrained deformations due to temperature gradients should be used. For the determination of crack width due to direct tension the recommendation by Broms and Lutz (1965) is recommended (ACI Committee 350, 2006), for bending the CSA Standard formula applies. Broms and Lutz recommend that:

$W_{max} = \varepsilon_s c[16 + (s/c)^2]^{1/2}$
where W_{mm} is the maximum crack width, *c* is the cover to the centre of the steel, ε_s is the strain in the reinforcing steel assuming a cracked section, and *s* is the spacing of the hoop steel. The use of small diameter bars at small spacings is recommended.

FOOTING DESIGN The radial reaction, F_r, at the bottom of the wall must be carried by the wall footing as a radial force. The radial force F_r can be resisted by friction between the bottom of the footing and the soil, by passive soil pressure against the vertical side of the footing, by placing the footing against undisturbed ground, or by reinforcing the footing with hoop steel to resist the resulting hoop tension (Eq. 1), or a combination of these. The hoop force, T_f, resulting from the radial force is simply the difference between the total hoop tension in the wall for the case being considered and that assuming a perfectly sliding base ($0.5wRH^2$). In the latter case the force F_r, and hence the footing hoop tension T_f, is zero.

The total hoop force in a wall with geometry, H^2/Dt, and spring support, k, at the bottom can be found easily by integrating the hoop forces plotted in Figs. 4 to 13 in Jofriet et al. (1996a) over the height of the wall, i.e. by finding the area under the curves. Since the hoop force plots are non-dimensional, the areas so found have to be multiplied by wRH^2. Table 2 lists values of the difference for all wall geometries and all spring stiffnesses provided in Jofriet et al. (1996a).

The hoop force in the footing thus calculated assumes that the entire force F_r is resisted by hoop tension (or compression if the load is radially inward). The reduction of this force by friction at the bottom of the footing is easy to estimate assuming a conservatively small coefficient of friction of say 0.4 to 0.5. The reduction from a passive soil pressure along the side of the footing is more difficult to calculate. It will be a function of the radial displacement and the type of soil. Of course, for any passive pressure to develop, the soil must be undisturbed and the footing concrete must be placed directly in contact with the undisturbed soil.

Table 2 Values of radial wall reaction for various wall geometries and bottom supports

H^2/Dt	Spring stiffness $k = 4\pi A_f E_c / D$				
	0	9	18	36	72	∞
0.4	0	0.128	0.168	0.199	0.221	0.245
0.8	0	0.115	0.154	0.186	0.208	0.235
1.2	0	0.100	0.138	0.170	0.192	0.221
1.6	0	0.088	0.124	0.154	0.176	0.205
2.0	0	0.087	0.120	0.148	0.169	0.195
3.0	0	0.072	0.100	0.124	0.141	0.163
4.0	0	0.067	0.091	0.111	0.124	0.142
5.0	0	0.064	0.086	0.103	0.114	0.129
6.0	0	0.060	0.079	0.094	0.103	0.115
8.0	0	0.055	0.071	0.082	0.089	0.099

SUMMARY The most important forces that the wall of a cylindrical liquid manure tank must be designed for were reviewed. The determination of the internal hoop forces in the wall of a cylindrical liquid manure tank is difficult if the boundary support at the bottom of the wall is other than sliding or hinged. References are provided to aid designers who want to consider a more realistic boundary condition. This paper also provides the designer sufficient information to calculate the horizontal shear force transferred from the wall to the footing given certain boundary conditions at the bottom of the wall. Friction and passive earth pressure can provide horizontal reactions at the wall footing; the frictional resistance is easy to calculate; the passive earth pressure can only be estimated as being less than the upper bound value. The net radial force equals the horizontal shear force minus the reactive forces; it must be resisted by the footing ring and will result in hoop tension to be resisted by reinforcing steel.

The paper includes design criteria to be used for the determination of the amount of circumferential (hoop) steel in the cylindrical wall, as well as the thickness of this wall. The vertical steel in the wall can be calculated using standard bending member analysis; the determination of the positive and negative bending moment will require considerable judgment on the part of the designer. The hoop steel in the footing should be determined from the net radial force on the footing providing a net hoop tension in the footing.

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7th International Symposium on Cement Based Materials for a Sustainable Agriculture
(CIGR International Symposium)
Hosted by the Canadian Society for Bioengineering (CSBE/SCGAB)
Québec City, Canada September 18-21st 2011

CEMENT BASED PAVEMENTS FOR INDUSTRIAL AND RURAL PAVING APPLICATIONS

Yves Brousseau

Abstract: Cement based pavements for industrial and rural paving applications : Roller Compacted Concrete (RCC), Full Depth Reclamation (FDR) and Cement Treated Based (CTB) / Les pavages à base de ciment pour les applications industrielles et rurales : le Béton Compacté au Rouleau (BCR), le Retraitement Haute Performance (FDR) et la Fondation Stabilisée au Ciment (FSC).

DEVELOPMENT OF APPROPRIATE TECHNOLOGIES FOR RAINWATER HARVESTING SYSTEMS

Marina Boldo Lisboa, Marcio Andrade, Henrique Lisboa

Abstract: In the context of increasing pressure on water resources, bringing consequences for both qualitative and quantitative aspects, the development of appropriate technologies for rainwater harvesting systems appears as a promising alternative. This work aims to develop an innovative technology for the construction of large rainwater storage tanks, with low cost constructive, in the region West of Santa Catarina, Brazil. This paper presents a new technology of construction of cylindrical tanks, supported on the ground, with slabs of slate mounted juxtaposed and surrounded by a structural steel armor and a thin-screen with an aperture enabling the manual application of the mortar, similar to the ferrocement craft. Were deployed three rainwater storage tanks with capacities of 50,000 liters, 80,000 liters and 250,000 liters, in three different cities of West of Santa Catarina. The first two tanks were executed in public schools and store rainwater for non-potable purposes, i.e. the discharge of toilets, washing sidewalks and irrigation of gardens. The larger tank was executed in a pig installation and store rainwater for livestock drinking and hygiene of these installations. This method allowed the implementation of constructive tank with greater ease and speed, better performance, structural and low cost constructive. It should provide greater durability than the ferrocement tank, since the water is not in direct contact with the mortar lining of the tank wall. As the slate is a rock inert, the tank armor is more protected.

Keywords: Rainwater storage tank, Slate, Ferrocement.

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AN ECOLOGICAL CONCRETE-PRODUCT STUDY BASED ON FROG AND SNAKE BEHAVIOUR ON VARIOUS SURFACES WITH SLANTS OF SMALL DRAINAGE CANALS IN PADDY FIELD

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CSAS11011 – Topic V: Water and Wastewater management

ABSTRACT Irrigation and drainage concrete-product canal have been used in paddy field. Diverse small animal like frog and snake inhabit surround paddy field as composing an ecosystem. Since canal have been set variously as dividing the habitat of small animal, they surely fall into small canal when migrating to breeding grounds, and then are carried to other place by water or dead in dried canal bottom. They cannot escape from canal with right angled smooth-surface wall. Using frog and snake, the experiment of their behaviour on various surfaces (6-type roughness: plane, very little, small and middle roughness, large-porous-roughness, and large-porous-roughness with moss) with 4 slants, i.e. 0, 30, 60, 90 degrees, was carried out to make clear concrete-block conditions which small animal can escape from canal. As the result, frog slid down at 90-degree slant on plane rough and very little rough blocks, and the jumping and crawling behaviour was found except above situation. Frog preferred the jumping in larger slant (but below 60 degrees) on rough surface and the crawling increased gradually. On the other hand, snake slid down at 60- and 90-degree slants on plane and very little rough blocks, and also at 90-degree slant on small and middle rough blocks. Snake could move by using their scale in case of larger rough block in spite of the slant. Consequently, the concrete product block with gentle slope and some roughness need ecologically to install partly in places of long-distance drainage canal.
Keywords: Concrete product block, Drainage canal, Frog, Slant, Snake, Surface roughness.

INTRODUCTION

In Japan, the water-use system has been planed strictly and rationally by using irrigation and drainage canals in flat land (Hasegawa and Tabuchi, 1995), since paddy field area is very wide. In lower mountainous and village area, paddy field area is generally small and long-and-narrow, and the irrigation water is carried to the fields by pipe-line system and then the water after irrigating is led into drainage canal. In the past, these paddy fields have held diverse animals and plants ecologically, but recently this ecology has been changed the style because of using various agricultural machines and agricultural chemical for a higher rice production (Fujioka and Lane, 1997; WCMC, 1998).

The biodiversity is rich in lower mountainous and village area, called "Satoyama", rather than in flat land area, because Satoyama includes not only crop land, paddy field and grassland but also forestry, bush and many streams. Therefore, Satoyama is admitted a notable area with biodiversity holding a food chain, as focusing Satoyama Initiative in COP10 (Conference of the Parties (for Biological Diversity) held in Nagoya, October 2010), introduced in the film (Green TV Japan, 2010). Recent agricultural projects need the concept of environment friendly and landscape. For example, as conservation in Satoyama, extremely rare plant and small animal etc. must be now preserved, while investigating them before starting the project.

Many small animals as frog, snake and heron inhabit surround paddy field, while composing the ecosystem with biodiversity. In Japan, to establish a higher rice production, various irrigation and drainage canals have been constructed by using a lot of concrete product block, for example, U- and V-shaped flumes. However, we can find often fallen frogs in drainage canals in paddy field for irrigating period (Hosokawa *et al*., 2009 and 2010). These canals have been actually divided small animal's habitat surround paddy field. Small animals migrate to their breeding grounds at late spring period, and some of them surely fall into canal and are usually carried to other place by water in canal, or unfortunately dead in dried canal bottom without water. Small animals fell into canal are difficult to escape from there because U-shaped flumes have structural 3-plane surfaces, i.e. both side walls with almost right angle and flat bottom. Water-use canal must flow much water by using smooth-surfaced flume inside the cross section area. Therefore, the restriction by canal in paddy field must be influenced to small animal's migration, to divide the habitat, and also to reduce their herds. To keep the biodiversity, canal structure with small-animal friendly should be improved from the characteristics of small animal behaviour.

The purposes of this study are to make clear the canal-wall properties to preserve frog and snake from the experiments on various surface roughness and slants of concrete blocks, and to find out some improving points from the investigation of two-type ecological drainage canals for frog's escaping in Satoyama area of Japan.

MATERIALS AND METHODS

Body form of frog and snake, and various surfaces of block in the experiment

Frogs and snakes are often found in small canals in paddy field in a flat and uneven land. In Japan, they and other small animal are composed a food chain there, as kite captures snake, mouse and frog, snake captures mouse, frog and other small animal, and then frog captures worms and other small animal. When they migrate to other field, they sometimes fall into canal and cannot almost escape from there, because canal structure have smooth-surface and high wall.

In the experiment, we used frogs and snakes captured in the university campus, as shown in Figure 1. This frog was "Japanese tree frog", Hyla japonica, and this snake was "Japanese striped snake", Elaphe quadrivirgata. As Table 1 indicates their body forms, the mean of length, weight, height and width of frog was 2.7cm, 1.7g, 1.5cm, and 1.3cm, respectively, and snake's weight, height and girth was 126.5cm, 191.9g, 2.3cm, and 7.8cm (in the girth), respectively.

Figure 1. Frog (Japanese tree frog, left) and snake (Japanese striped snake, right).

To study surface condition and angled slant of concrete block which frog and snake can escape or clime up from the drainage canal easily, various uneven surface conditions were made by using slate board and 25mm- and 40mm-size coarse aggregate concrete. The block surface area in all experiment was 90cm×110cm, closed by 20cm-height wooden boards.

Table 1 Body form (mean±SD) of small animals in the experiment

Small animal	Number	Length (cm)	Weight (g)	Height (cm)	Width (cm)	Remark
Frog	14	2.7±0.4	1.7±0.5	1.5±0.2	1.3±0.2	Japanese tree frog
Snake	2	126.5	191.9	2.3	7.8*	Japanese striped snake

* girth

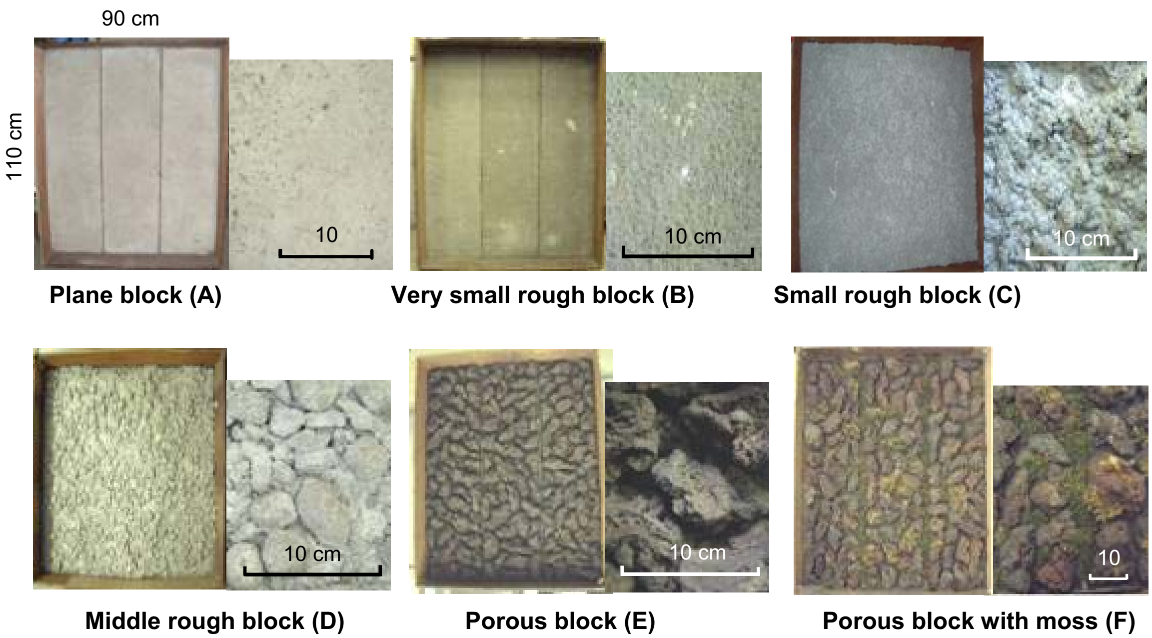

Figure 2. 6-type surfaces of concrete blocks in the experiment.

As shown in Figure 2, plane block (A) was composed by only 3-slate boards without roughness. In three rough-surface blocks, very small rough block (B) was made by coating the mortal (cement vs. river sand of 1:3 and 65% W/C by a weight mortar mix proportion) on a slate board. Small rough block (C) was made by 25mm-size river gravel concrete, and also middle rough block (D) was made by 40mm-size one, based on 60% W/C. Furthermore, large rough porous block (E) was made with crushed eruptive rocks from the skirts of Mt. Fuji, glued on a slate board, and large rough porous block with moss (F) was fixed the moss into rock's gaps and pockets on E block. The moss was Brachytheciaceae (*Bryhnia novae-angliae*) taken from rocks covered with moss in downstream of the Oirase river in Aomori prefecture (Iwatsuki and Mizutani, 1994).

As blocks and surfaces were composed by various materials, the thickness and the maximum of thickness as block's roughness were necessarily different due to the materials, as shown in Table 2. Surface roughness was indicated by the thickness and the maximum thickness at 5 random lines on blocks. However, for small animal, it is important to walk (crawl) on the surface of block. Figure 3 is surface condition on one line at will each block as an example. E and F blocks indicated large uneven surfaces being so hard for small animal to crawl.

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Measurement and analysis of small animal behaviour on 6-type surfaces of blocks

According to measure small animal behaviour on 6-type surfaces of blocks, the digital video camera (Panasonic NV-DB1) was used for 8 hours on each block and at slants of 0, 30, 60 and 90 degrees. The video camera was fixed to the top of angle iron flame as taking small animal behaviour film in 90cm×110cm area at 170cm high above the block surface, and the each slant was made by the angle iron flame, as shown in Figure 4.

The video film was taken in the room under about 400 lures illumination from fluorescent lights at the ceiling, 20±1ºC temperature (adding 25ºC temperatures in F block only) and 80±5% humidity after making the block surface wet by sprinkling water for a short time. Small animal was changed each other every one hour as being under less stress, after sitting on the center of block at the start.

Watching one-hour-video film of small animal behaviour on a 14-inch-TV monitor, the behaviour pattern, the moved distance and the staying time on block surfaces were measured and then analyzed. The behaviour pattern was measured the time spent in the jumping and crawling behaviour. The moved distance was measured the length by a

Table 2. Surface condition and thickness of concrete blocks in the experiment.

Surface condition	Plane block	Rough block			Large rough block	
		Very small	Small	Middle	Porous	Porous with moss
Mark	A	B	C	D	E	F
Material of block	Slate board	Mortal coating on slate board	Concrete with 20mm-coarse aggregate	Concrete with 40mm-coarse aggregate	Crushed eruptive rocks glued on slate board	Crushed eruptive rocks glued on slate board
Thickness (mm)	0.8±0.5	1.9±1.2	21.6±5.8	21.7±9.0	26.0±19.2	27.2±15.6
Max. of thickness (mm)	2.5	5.2	35.8	42.4	71.5	64.1

curvimeter while tracing the behaviour tracks in each jumping and crawling on the plastic transparency sheet. Then, the total staying time was measured the total time for one hour in each cell by using traced behaviour tracks on the sheet, while dividing 90cm×110cm area into 9 cells of each block.

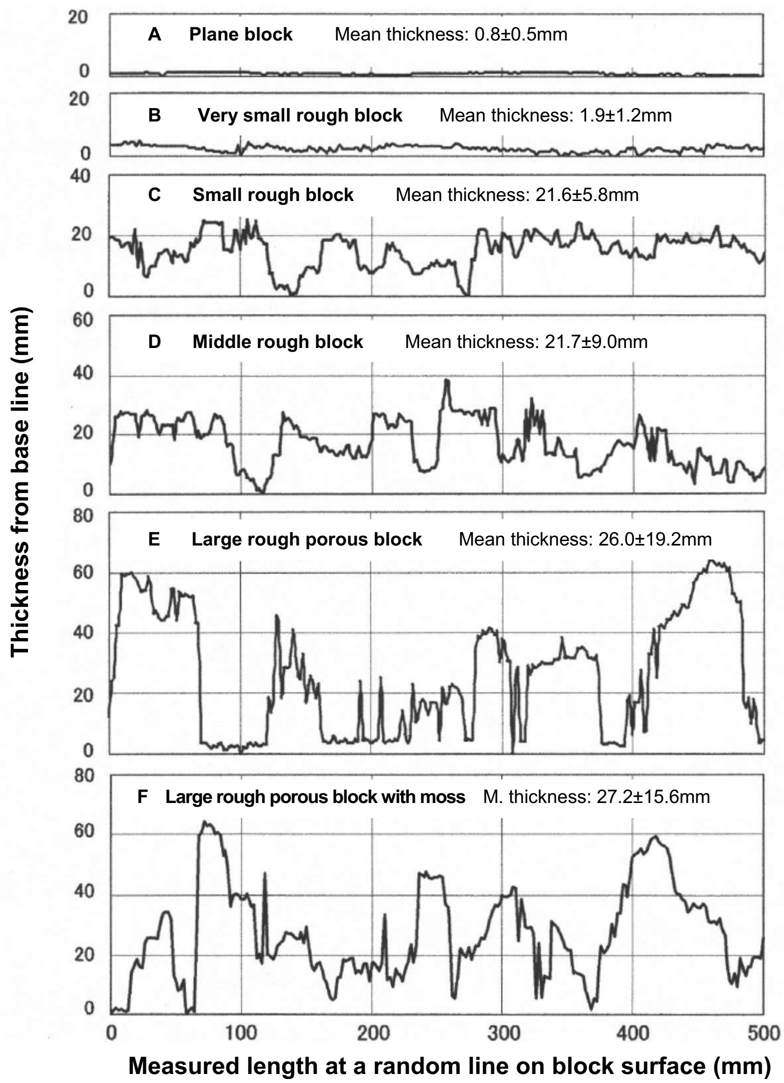

Figure 3. Surface conditions (example: one 500mm-line) of blocks.

If small animal escaped from the flame area, the investigation was continued from the center of block again after taking back to the center by the observer. As the variation of behaviour was not small actually, the behaviour film in each small animal was taken at least 5 times as the coefficient of variation became within 40%. During the observation of behaviour, the observer waited out of the room except the escaping treatment while watching behaviour on the TV monitor. During the experiment, feeding was never stopped as frog ate worm and snake ate mice always under a wet environment in their waiting rooms.

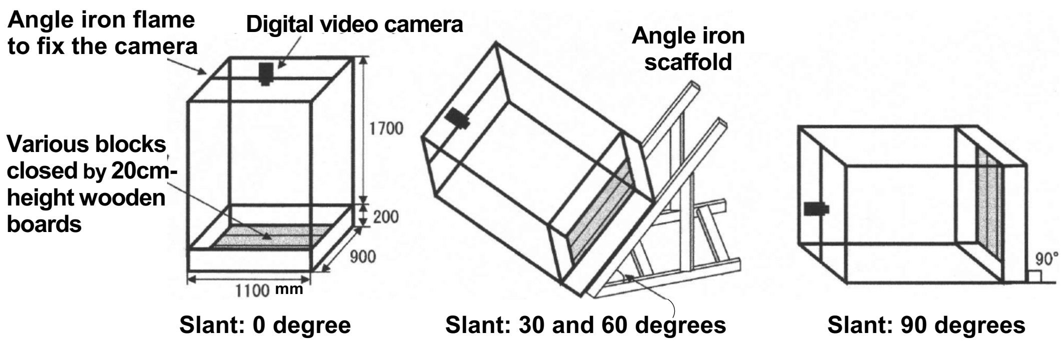

Figure 4. Method of recording digital-video behaviour on various blocks at each slant.

Investigation of ecological small drainage canals

Ecological small canals have been constructed in Satoyama by the agricultural land and environment improvement project, and some were reported about its effects and improvements. It is necessary to study about the actual condition and the effective function after the projects.

Ecological small drainage canals, constructed in Satoyama area of Tateyama city, Chiba prefecture, 2006, were investigated in September 2010. There were two-type canals, i.e. V-shaped (trapezium) porous concrete block canal and U-shaped concrete product canal with partly both-side expanded slopes by concrete floor and small log. The investigation was carried out the function, material and structure and then improvement points from the view of small animal escaping behaviour.

RESULTS AND DISCUSSION

Characteristics of small animal behaviour on various block surfaces

1. Behaviour pattern small animal on block Figure 5 indicates the distribution of frog's behaviour patterns for one hour. The behaviour of frog is only two patterns, i.e. jumping and crawling. Frog slid down at 90-degree slant on plane rough and very little rough blocks (A and B blocks), though frog has suckers on top of each finger. Except for above situation, we can found the jumping and crawling behaviours. As being in smaller slant below 60 degrees on rough surfaces (C~D blocks), the jumping one decreased but the crawling one increased gradually. The large rough surface of E and F blocks was too big for frog, and particularly frog stayed in gaps and pockets for a long time at 90-degree slant. In case of 25ºC temperatures in F block, its tendency was same, but frog increased the jumping behaviour particularly at 60- and 90-degree slants. It was estimated that warm environment led frog to lively behaviour.

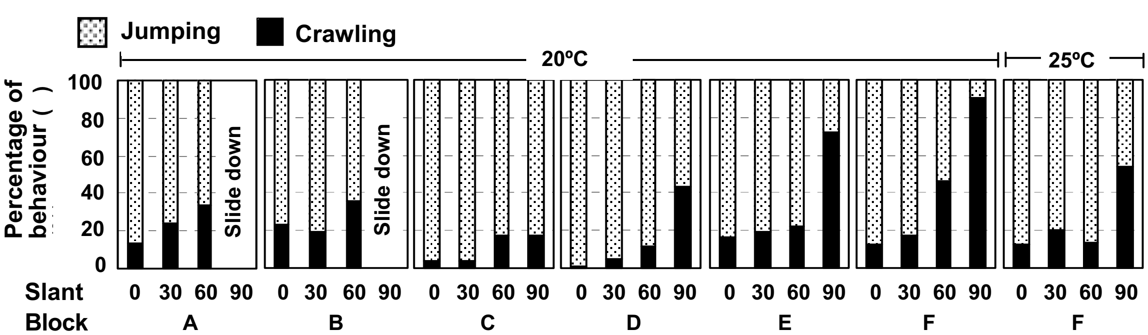

Figure 5. Distribution of frog's behaviour pattern for one hour.

On the other hand, snake slid down at 60- and 90-degree slants on plane and very little rough blocks, and also at 90-degree slant on small and middle rough blocks. Snake could move by using their scales in case of larger rough block in spite of the slant.

Therefore, from these results, according to help the escaping behaviour, the surface of block needs middle and large roughness if anything and the slant is suitable below 60 degrees at least. Furthermore, the surface is important that frog need gaps or pockets and also tall moss or plant to hide them, and snake need rough to hold themselves in spite of the slant.

2. Moved distance of small animal on block Figure 6 indicates the moved distance for one hour by frog and snake. In case of A block and flat (0 degree slant), frog moved about 13.5m/hour causing by the jumping mostly, and snake moved 12.5m/hour by the crawling only. The moved distance by both animals decreased obviously as the slant and the surface roughness became large. Although frog's behaviour changed from the jumping to the crawling when the surface roughness became large as shown in Figure 3, the moved distance was caused by the jumping rather than by the crawling at lower slants. In particular, the moved distance in E and F blocks was shorter than other smaller surface blocks. As there were many gaps and pockets on E and F blocks, a frog stayed or hid in a pocket with moss of F block's surface as shown in Figure 7.

Snake have only the crawling behaviour, and there was a clear tendency that the moved distance increased as the slant and the surface roughness became small. Snake were apt to slide down on small rough surfaces at steep slants (A and B blocks). However, we can found some moved distance by crawling because the large rough surfaces (E and F blocks) made with crushed eruptive rocks, on where being skilfully holding or hanging by using their scales.

Both frog and snake behaviour in 25ºC temperatures in F block made longer the moved distance, rather than in 20ºC one. It was estimated that a warm environment in the range of 25ºC temperatures may lead a lively migration. It is estimated that 20ºC-temperature environments was a little cold for small animal in the experiment.

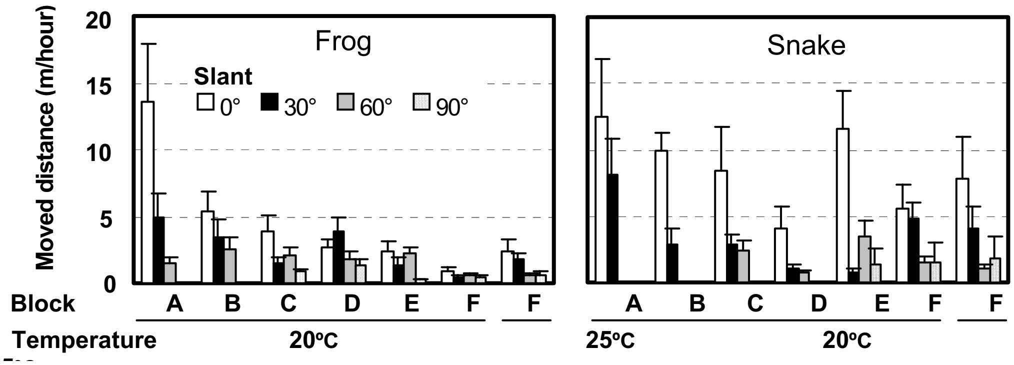

Figure 6. Moved distance for one hour by frog and snake.

Figure 7. Frog stayed or hid in a pocket with moss of F block's surface.

3. Total staying time of small animal on block Figure 8 indicates the percentage of distribution of total staying time for one hour on each cell of blocks. The slide down of behaviour by frog and snake were shown in the figure, and frog slide down in A and B blocks at 90 degree slant and snake slide down in A~D blocks at 90 degree slant and A and B blocks at 60 degree slant. Although snake's sliding down behaviours were conspicuous than frog's one, the heavy body weight of about 200g was influenced to these phenomenon, compared with the frog's body weight of 1.7g.

In the distribution of frog's total staying time, we cannot find a clear tendency. However, in rough and middle rough blocks at larger slants, frog was apt to stay at upper site. In some cells, frog stayed for a long time in specified gaps and pockets to hide the body.

In the distribution of snake's total staying time, we cannot find a clear tendency. There was a small tendency that snake liked the corner. The behaviour differences under warm environment and greened with moss were not exactly. Snake can escape from canal with rough surfaces to hold the body by using the scale, and can move at lower slant lively actually.

These results lead that a slope with gentle slant and rough surface should be necessary in a part of long distance canal, as it will cost to construct a long distance slope for only the small animal escaping.

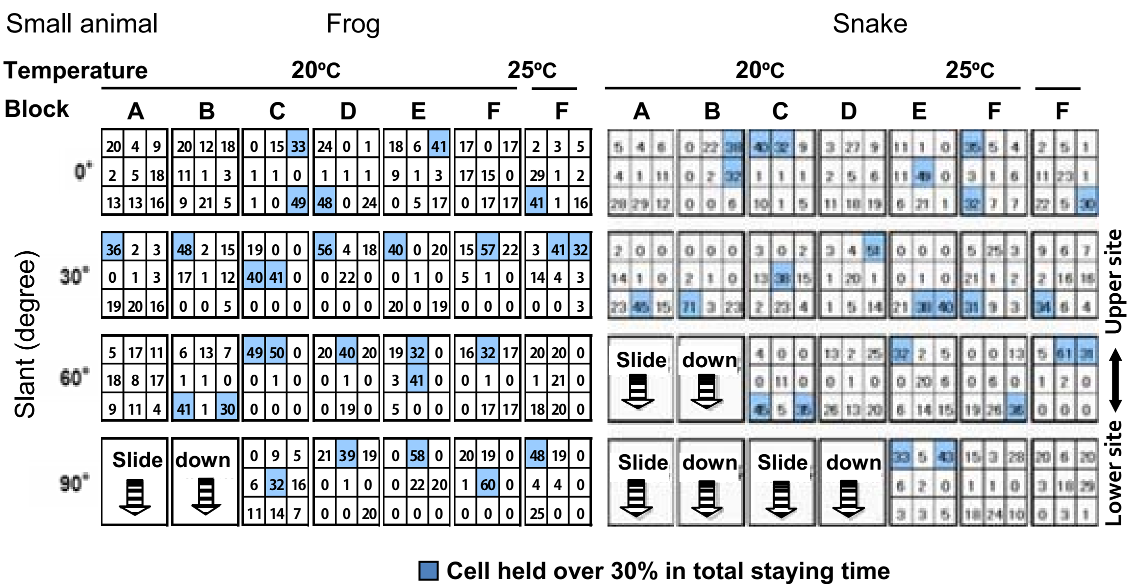

Figure 8. Percentage of total staying time for one hour on each cell of block.

Function requiring in ecological small drainage canals

Figure 9 shows two ecological drainage canals investigated in Satoyama area of Tateyama city, Chiba prefecture. These canals were constructed or installed partly as small drainage canal in the paddy field in 2006 by the agricultural land improvement project.

The left in Figure 9 is V-shaped (trapezium) drainage canal by porous concrete block. This block had a crawling function by their gentle slant and uneven surface, but was easily dry and just covered with less moss and wild plant because of less wet-keeping function. On the other hand, the right in Figure 9 is U-shaped drainage canal by concrete product with both-side expanded slope for escaping. Two slops were made of rough concrete floor and logs to escape from water surface, each other. Although frog can swim after falling into canal, almost frog is flowed down to the downstream only. In the upper canal near the expanded canal, the slanted drop facility like a chute was constructed to awake a falling frog at the expanded canal, and the partly-expanding canal can reduce a water velocity which frog may escape by using slopes. However, the actual expanded canal was too short and narrow where the water whirls to be caused to wake frog falling on. The log in a slope was exposure against frog to be found by snake and bird, and was also slightly bigger (too high) for frog.

The exposure slopes may need to be covered with moss and lower plant in case of no weeding to hide the small animal body, and the one-side expanded place installed in ecological canal is better than the both-side expansion, because no farmer hope to be reduced a part of area of paddy field by expanding canal, even partly.

Figure 9. V-shaped (trapezium) drainage canal of porous concrete block (left), and U-shaped drainage canal of concrete product with both-side expanded slope for escaping (right).

CONCLUSION

It is suggested that the behaviour of frog and snake was obviously restricted by concrete product block with smooth surface of the inside two-wall in drainage canal. Particularly, the crawling behaviour by frog and snake was impossible in plane and small rough surfaces at 90-degree slant. Also, the moved distance by both small animals decreased in the above condition. Frog preferred not only gentle slope but also gaps and pockets to hide the body and also the surface covered with moss. On the other hand, snake preferred gentle slant and rough surfaces, and used their scale skilfully to crawl and hang themselves on rough surfaces.

It is important to observe the behaviour after frog and snake fell down into drainage canal. In general, they try to swim in water to escape at first after falling, and then almost are flowed down only to the downstream while a little swimming. In the actual ecological drainage canal, its structural and surface improvements are required as frog can awake by the water whirls at the drop facility in the upper canal, and can climb up a gentle and rough slope covered with moss and low plant to hide them effectively.

Furthermore, the concrete product flume with easy escaping function may be installed at a part of drainage canal ecologically, for example, at the installing interval of one block (3~5m length) per 100m drainage canal, because it is difficult costly to construct them in a long distant slope in whole paddy field.

The future subject is to study on the frog jumping ability and the function and structure of drainage canal with one side expansion system for frog to escape easily (Hosokawa *et al*., 2009 and 2010). Then next study is a canal-wall to be easily covered with moss and low plant as shelter, from the view of small animal escaping behaviour and concrete material.

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APPLICATION OF CONCRETE-POLYMER COMPOSITES FOR LIVESTOCK WASTE AND MANURE MANAGEMENT FACILITIES

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CSAS11008 –**Concrete Deterioration**

ABSTRACT This study is to overview the methods of applying concrete-polymer composites, one of the novel construction materials, to livestock waste and manure management facilities through extensive literature studies. The result showed that polymer-modified concrete is not suitable for this application due to its low acid resistance, but it is suitable for repair and rehabilitation of deteriorated cement concrete livestock facilities since the mechanical properties of polymer-modified concrete are comparable to those of conventional portland cement concrete. On the other hand, polymer concrete and polymer-impregnated concrete are beneficial in chemical resistance, strength, and watertightness, and are effective in new construction of livestock facilities which require enhanced durability.
Keywords: livestock waste, manure management, facilities, concrete-polymer composites, polymer-modified concrete, polymer concrete, polymer-impregnated concrete

INTRODUCTION

Livestock facilities play a very important role in productivity increase of livestock products and efficient management of livestock. Such facilities are divided into animal production, feed storage, and waste and manure disposal depending on the purpose of usage. Among them, waste and manure disposal facilities could suffer from severe corrosion damage due to ingress of deleterious ingredients, which may lead to durability problems. Especially, in the case of group feeding of livestock such as swine or beef cattle which produce a large amount of waste, the durability problem can be even more serious.

Wood, steel, aluminum, or plastic may be suitably chosen as construction materials for livestock facilities, but portland cement concrete is most widely used because of the various advantages such as ease of use, good durability, and reasonable cost (Whitaker, 1979).

Generally, freezing and thawing, carbonation, alkali-aggregate reaction, and salt crystallization are described as the main causes of cement concrete deterioration (Mehta et al., 2006). In livestock waste and manure management facilities, however, deterioration induced by hydrogen sulfide is dominant because the hydrogen sulfide tends to convert to sulfuric acid under the biological action of sulphatizing bacteria. The building materials which can compensate for this deficiency of conventional

cement concrete are concrete-polymer composites. Concrete-polymer composites have been commonly used as construction materials for civil infrastructures (Fowler, 2003), but few cases utilizing in livestock facilities have been found. Of course, concrete-polymer composites are not utilized as widely as cement concrete because the per-unit-volume price is more expensive. However, if the advantages of the cement-polymer composites are appropriately used, concrete-polymer composites could be good alternatives for producing durable and economical livestock facilities.
In this study, the deterioration mechanisms of livestock facilities made of conventional cement concrete, characteristics of concrete-polymer composites, types of livestock waste and manure storage facilities, and application methods of concrete-polymer composites are discussed.

DETERIORATION MECHANISMS OF CEMENT CONCRETE
When livestock facilities made of cement concrete experiences deterioration, cracking, surface corrosion, delamination, and other types of distresses typically occur. This is because the deleterious ingredients penetrating from the outside reacts with the hydration products and thus degrades the performance of concrete. However, the greatest risk to the livestock facilities made of cement concrete is the deterioration caused by hydrogen sulfide resulting from sulfide reduction by anaerobic bacteria which comes from fermentation of livestock waste. The sulfuric acid formed by sulfate reducing bacteria residing in livestock waste reacts with alkali in concrete, inducing the corrosion and deterioration of concrete. The identified mechanisms of corrosion by sulfuric acid have been described by several researchers (Morita, 2002 and Mehta et al., 2006).

1) Formation of sulfide (biological action)
When livestock waste is in the anaerobic state, sulfate reducing bacteria inhabiting in the concrete surface-bonded bio-film is activated and sulfate ion in the livestock waste is reduced to sulfide (Formula 1):

$$SO_4^{2-}+2C+2H_2O \rightarrow 2HCO_3^{-}+H_2S \quad (1)$$

Formation of hydrogen sulfide is a biochemical reaction in which sulfate ion is used as the electronic acceptor, and the organism is used as an electronic donator by sulfate reducing bacteria. It is known that in such reactions, organisms used by sulfate-reducing bacteria selectively ingest certain organic acids formed by hydrolysis and organic acid bacteria. Thus, when assuming that a sufficient amount of sulfate-reducing bacteria exists within livestock waste, the content of sulfate ion and the type and content of organism play an important role in the formation of hydrogen sulfide.

2) Formation of sulfuric acid (biological action)
Hydrogen sulfide melts onto the concrete surface by surface condensation and changes to sulfuric acid by biological action of sulphatizing bacteria (Formula 2):

$$H_2O+2O_2 \rightarrow H_2SO_4 \quad (2)$$

The sulphatizing bacteria does not require organisms but requires oxygen in the air when forming sulfuric acid with autotrophic bacteria. The general growth environment of sulphatizing bacteria is between 10℃ and 30℃ and in many cases around 30℃. It does not grow well in a dry environment but grows well in humid environment, so it acts lively in places where the surface condensation occurs.

3) Deterioration of concrete by sulfuric acid corrosion (chemical and physical action)
Sulfuric acid formed by sulphatizing bacteria reacts with calcium hydroxide, the alkaline component in the hydrated cement paste matrix, on the concrete surface, and forms calcium sulfate (Formula 3).

The formed calcium sulfate continues the chemical reaction with tricalcium aluminate ($3CaOAl_2O_3$) contained in the cement paste and forms ettringite (Formula 4). When the ettringite is formed, a large number of water molecules are chemically combined and undergoes 300 to 400% volume expansion, ultimately resulting in damage of concrete.

$$Ca(OH)_2+H_2SO_4 \rightarrow CaSO_4.2H_2O \quad (3)$$

$$3(CaSo_4\cdot 2H_2O)+3CaO\cdot Al_2O_3+26H_20 \rightarrow 3CaO\cdot Al_2O_3CaSO_4\cdot 32H_2O \quad (4)$$

CHACTERISTICS OF CONCRETE-POLYMER COMPOSITES

1) General properties (Chandra et al., 1994. Wahby, 2003. Czarnecki, 2007)
Concrete-polymer composites are the materials made by replacing and strengthening a part or all of the cement hydrate binder of cement concrete with polymers. Concrete-polymer composites are generally classified into the following three types depending on the principles of process technology:

① Polymer-modified concrete (PMC)

② Polymer concrete (PC)

③ Polymer-impregnated concrete (PIC)

PMC is the composite material fabricated by partially replacing and strengthening the cement hydrates of conventional concrete with polymeric modifiers or admixtures such as polymer latexes or dispersions, redispersible polymer powders, water-soluble polymers, liquid resins, and monomers. PMC has a monolithic co-matrix in which the organic polymer matrix and cement gel matrix are homogenized.

The properties of PMC can be significantly improved to be superior to conventional cement concrete. There are various factors affecting the properties of fresh and hardened PMC such as type of polymer, polymer-cement ratio, water-cement ratio, air content, and curing conditions.

PC has the microstructure in which the cement hydrate binders of cement concrete are completely replaced with polymeric binders. Therefore, PC develops characteristics very different from those of the cement concrete. The properties of the fresh and hardened PC are affected by factors such as polymeric binder types, binder formulations and mix proportions, and curing conditions.

PIC is a composite material made by impregnating monomers into the hardened cement concrete. The monomers are subsequently polymerized, strengthening the cement hydrate binders with the resultant polymers. Polystyrene and polymethyl methacrylate monomers are used as the common monomeric impregnants for PIC. The properties of PIC mainly depend on the polymer impregnation depth and polymer loading. Although there are some differences in the properties between fully and partially impregnated ones, the major properties are consistent as follows:

① Excellent watertightness and freeze-thaw durability

② Good chemical resistance

③ Excellent abrasion resistance and impact resistance

Table 1. Properties of concrete-polymer composites compared to cement concrete

Property	Portland cement concrete	Polymer-modified concrete	Polymer concrete	Polymer-impregnated concrete
Compressive strength, MPa	10-60(>60*)	10-75	40-150	100-200
Flexural strength, MPa	1.5-7(>7*)	3-12	4-50	7-35
Tensile strength ,MPa	0.6-3.0(>3*)	4-9	4-20	4-17
Modulus of elasticity, GPa	15-30	10-25	7-45	30-50
Poisson`s coefficient	0.11-0.21	0.23-0.33	0.16-0.33	0.20-0.25
Coeff. of linear thermal expansion, $10^{-6}.K^{-1}$	10-12	11-15	10-35	10-17
Water absorbability, %	4-10	1-3	0.5-3	0.5-1.5
Chemical resistance	poor/average	average/good	very good/excellent	good/very good

*For High Performance Concrete
Source : Czarnecki, L. 2007. Concrete-Polymer Composites : Trends Shaking the Future. Int. J. Soc. Mater. Eng. 15(1):95

2) Chemical resistance (Chandra et al., 1994. IMRI, 1986)

The chemical resistance of PMC is dependent on the characteristics of polymers incorporated, i.e., the nature of chemicals and polymer-cement ratio. Most PMC is damaged by inorganic or organic acids and sulfates as they contain cement hydrates that are vulnerable to those chemical agents, but resists alkalies and various salts. The chemical resistance of PMC is generally rated as good to fats and oils, but poor to organic solvents. Table 2 shows the conceptual evaluation of the chemical resistance of typical PMC.

Table 2. Chemical resistance of polymer-modified concretes

Type	Evaluation value (points*)				
	Acid	Alkali	Salt	Solvent	Oil and fat, mineral Oil
Unmodified	1	8~10	1~7	5~7	7~10
SBR-modified	1~2	10	5~10	2~3	8~10
PAE-modified	1~2	10	5~10	2~3	7~9
EVA-modified	1~2	8~10	5~10	5~7	8~10

*Conceptual evaluation criteria for chemical resistance, indicating from 10 points for "Excellent" to 1 point for "Failed".
Source: Chandra, S., and Y. Ohama. 1994. Polymers in Concrete. CRC Press.: 130

Since PC has a watertight or impermeable microstructure without the low chemical-resistant ingredients such as cement hydrates, it generally has a high chemical resistance compared to cement concrete, as indicated in Table 3. Such high resistance relies on the nature and amount of the polymeric binders, the properties of aggregates, and the nature of chemicals used. The resistance of PC to oxidizing agents is inferior.

Table 3. Chemical resistance of polymer concretes

Type	Evaluation value (points*)				
	Acid	Alkali	Salt	Solvent	Oil and fat, mineral Oil
Polyester	8~9	3~4	9~10	4~6	7~9
Epoxy	9~10	9~10	10	6~7	9
Furan	9~10	9~10	10	7~8	8
Acrylic	8~9	8~9	9~10	5~6	7~9
Cement	1	8~10	1~7	5~7	7~10

*Conceptual evaluation criteria for chemical resistance, indicating from 10 points for "Excellent" to 1 point for "Failed".
Source: Chandra, S., and Y. Ohama. 1994. Polymers in Concrete. CRC Press.: 141

PIC is superior to cement concrete in chemical resistance as well as in watertightness and freezing-thawing durability. The comparison of chemical resistance with unimpregnated (controlled) concrete is summarized in Table 4. The results show that the corrosion resistance to sulfate is excellent. Also, the corrosion resistance to acid turns out to be good.

Table 4. Chemical resistance of polymer-impregnated concrete

Property	Control Undried	Dried	MMA	Styrene	MMA :10% TMPTMA	Acrylonitrile	Chlorostyrene
Sulfate attack							
No. of days	480	605	720	690	630	540	3200
% weight loss	0.466	0.522	0.006	0.030	0.003	0.032	0.009
Acid resistance (15% HCL)							
No. of days	105	106	805	8-5	709	623	292
% weight loss	27	26	9	12	7	12	8
Acid resistance (15% H_2SO_4)							
No. of days	49	77	119	77	--	70	77
% weight loss	35	30	26	29	--	30	26

Source (excerpt): ACI Committee 548, 1977. Polymers in Concrete: 54

TYPES OF LIVESTOCK WASTE AND MANURE STORAGE FACILITIES

Livestock waste and manure management facilities are essential for most farms to meet the environmental regulations and to hold the waste and manure until a convenient or advantageous time to spread it on the land. Types of facilities include (Whitaker, 1979):

1) Slotted Floors

Slotted floors are important in housing for several types of livestock. It is generally conceded that the use of slotted floors has made confinement housing for swine an economically viable system.

Regardless of whether manure is stored in a pot under the floor, drained, or scraped away, the slotted floor provides a very efficient method of removing the manure from under the animals. The slotted floor has a number of other advantages. It allows greater animal density, and at the same time the animals remain cleaner.

2) Pit storages
Pit storages are located under the slotted-floor buildings. The problem of odors and toxic gases at the time of agitation discourages many from using this method. Hydrogen sulfide and ammonia are strong irritants and can be lethal in sufficient concentrations. Carbon dioxide and methane are odorless but cause asphyxiation.

3) Holding ponds or tanks
These are used to hold manure up to six months or longer. Some anaerobic decomposition may occur, but the primary purpose is simply storage. Holding ponds should be located so that there is no possibility of polluting streams or lakes. In areas of porous soil structure, sealing may be necessary.

Tanks may be either above ground, requiring the manure to be pumped from the barn, or below grade into which the manure flows by gravity. Both ponds and tanks present potential odor problems when they are agitated and pumped.

4) Lagoons
Lagoons are designed to cause bacterial decomposition of the manure. Anaerobic lagoons have a deficiency of air and frequently cause some odor problems. Often they become simply holding ponds that need to be pumped periodically. Aerobic lagoons require sufficient surface area to enable the sun and air to bring about complete decomposition leaving the water clear and without scum or odor.

5) Settling basins
These are used as temporary detention ponds to allow settling of a high percentage of the solid material from yard runoff as it flows into a lagoon. The basin is cleaned periodically and dries out between storms.

6) Oxidation ditches
Oxidation ditches are equipped with large agitators that stir air into the manure. Considerable decomposition results, and odors are reduced to a minimum. However, costs are high, management is difficult, and much nutrient value is lost.

7) Gutters
Gutters should be scraped and flushed down daily. Deep gutters are allowed to accumulate the material scraped and flushed into them for one to three days. They are essentially self-cleaning when allowed to drain. The pit under slotted floors is emptied either by pumping into above ground tanks or allowed to drain periodically into below- grade storage.

APPLICATION OF CONCRETE-POLYMER COMPOSITES

Livestock waste and manure management facilities are typically made of cement concrete. Concrete-polymer composites can be efficiently incorporated to enhance the durability of such facilities in the following ways.

PMC, as shown in Table 1, is beneficial in that it has good adhesion to existing concrete as well as has low water absorbability even though there is some variation depending on the nature of polymers added. Also, the mechanical properties of PMC are similar to those of cement concrete. However, as shown in Table 2, it is vulnerable to acid and solvent in the aspect of chemical resistance. Accordingly, PMC is not suitable for the use in manure storage facilities which emit hydrogen sulfide gas, but rather it can be used for repair and rehabilitation of the deteriorated livestock waste and manure management facilities. Also, PMC can be an effective building material for livestock facilities for feed and bedding. PC generally has much improved mechanical properties and chemical resistance than cement concrete as seen in Tables 2 and 3, respectively. Also, it has good watertightness, adhesion, abrasion resistance and impact resistance, and has a short setting time. Thanks to these advantages, PC is widely used in construction works, especially in precast products including sewage manholes and pipes. Also, it is applicable to the emergency repair of damaged concrete structures at job sites (Yeon, 2010). Based on these beneficial properties, PC could be utilized in livestock waste and manure management facilities such as pit storages (including slotted floors), holding ponds or tanks, lagoons, settling basins, oxidation ditches, gutters and other facilities in the form of precast products. If the precast member is too massive, it can be properly segmented. The PC also can be produced as cast-in-place using formwork, and it is advantageous in reducing the thickness of a member by 65 to 80% due to its high strength (Yeon, 2010).

PIC, as shown in Table 1, has low water absorption as well as enhanced mechanical properties. Polymer-impregnation is used to strengthen the surface area, 3 to 5 mm from the surface of a member, of the precast or in-place concrete members by impregnating and polymerizing the monomer. As shown in Table 4, PIC has excellent chemical resistance compared to the untreated control. There were some constructability problems in the past when radiation or thermal polymerization method was used, but currently, polymer impregnation can be easily achieved even at room temperature when polar monomers such methacrylic acid is used (Yeon, 2006). This method can only be applied to newly constructed cement concrete structures and requires sufficient duration and temperature of surface drying prior to impregnating the polymers. If the advantages of PIC are well ensured, it will be greatly useful for the construction of longer-lasting and better-performing livestock waste and manure management facilities.

CONCLUSIONS

This study was conducted by analyzing the experimental data reported in the literature to propose the methods of applying concrete-polymer composites, a novel construction material, to livestock waste and manure management facilities.

This study showed that polymer-modified concrete (PMC) is superior in adhesion but showed little difference from cement concrete in chemical resistance. Thus, it is desirable to use PMC only for repair of damaged cement concrete livestock facilities.

Polymer concrete (PC) is superior in both mechanical properties and chemical resistance, and can be suitably used in all kinds of livestock facilities. PC can be used as both precast and cast-in-place. Also, in the case of massive precast members, it can be segmented for convenience.

Polymer-impregnated concrete (PIC) can be effectively used in livestock facilities using the partial impregnation method. To achieve the impregnation of monomers, sufficient drying of the concrete surface is required.

The manufacturing process and construction methods were not presented within the scope of this study, but a wide range of information regarding them is available in the various literatures. Also, this paper did not include the result of cost analysis. However, sufficient economic efficiency could be ensured by selecting the proper type or application method of concrete-polymer composites.

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Live Cycle Analysis

Section 3

LIFE CYCLE ANALYSIS

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CSAS11019 – LCA and Concrete used Sustainable Farm Building

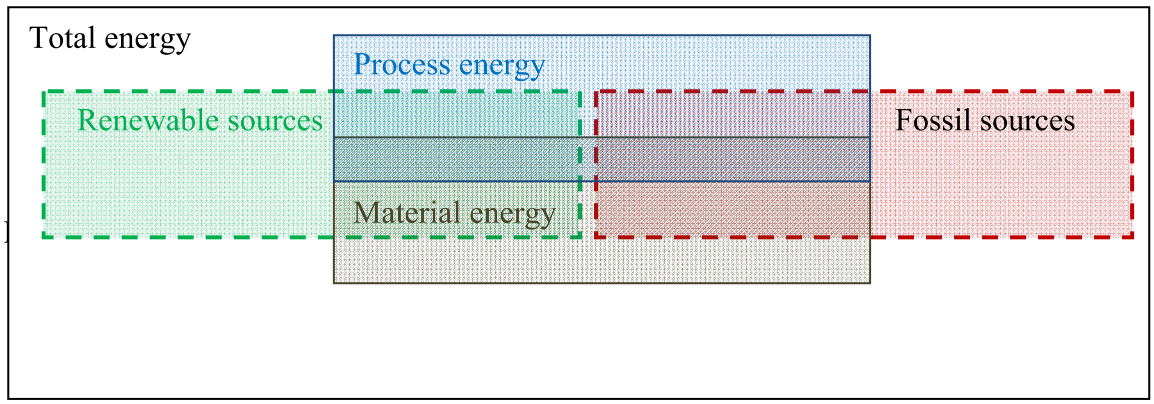
ABSTRACT The French pig and pork institute (IFIP) took part in a project called Eco-conception of livestock housing. The first step of this project was to perform a complete life cycle analysis (LCA) in order to evaluate the environmental impact of the existing French pig houses. The main part of this project was to aggregate the impact of the whole building materials over a 30 years time life. Because of the lack of data, only energy used and the green house gases (GHG) emission have been calculated. Two main types of structures were compared, either concrete paneling or monolithic brick paneling. Higher energy consumption was estimated for concrete paneling than for monolithic brick paneling (i.e., 56.9 and 52.6 $MJ.m^{-2}.year^{-1}$, respectively). The CO_2 equivalent release by concrete paneling was higher than by monolithic brick paneling (4.8 vs 4.3 kg eq. $CO_2.m^{-2}.year^{-1}$). Plastic was also compared to concrete for pen separation. In this case, concrete separations have a better impact than plastic ones. However, the energetic impact and GHG emission by pig house is very weak compared to the global farm balance, which includes food, direct energy utilization, fertilizer, pesticides, fungicide use and manure management; indeed, buildings contribution to total energy consumption is less than 3% and it represents less than 1% of the global farm GHG emission.
Keywords: Life cycle analysis, pig house GHG, pig house energy use

INTRODUCTION In France, there is a label for low emitting houses for human beings, but nothing exists for livestock housing. Therefore, three animal French institutes started a project, so-called “Green building and livestock housing”, in order to transpose the residential sector label into livestock housing. The first step was to calculate the livestock housing environmental impact.
Method

The method used was an aggregation of the Green house gas (GHG) emissions and energy use of the whole building materials. The balance was evaluated from data collected from two pig units. Each of them was a 200 sows farrowing to finishing farm. In the first farm, the barns were built with concrete paneling, whereas the second one was built with monolithic paneling. Three different criteria were used in the evaluation: (i) with or without sow electronic feeder (SEF) in the gestation unit, (ii) monolithic panels or concrete panels, and (iii) plastic slatted floor or concrete slatted floor in the post weaning unit. At the end, eight different types of pig houses were considered.

LCA method The determination of the energy use and the whole GHG emission were based on life cycle analysis (LCA) that allows the calculation of the environmental impact from cradle to grave. In the present project neither equipment (fans, feeders, etc.) nor the energy use over the building life were taken into account for LCA.

Energy definition According to the LCA method, two kinds of energy use are considered: the energy used in process and the energy contained in the material itself (only when the material had its own calorific power, like wood for example). Energy use in process corresponds roughly to the primary energy required to extract, transform, transport, the raw material (fig 1). Total energy use can originate from renewable or fossil sources.



Energy calculation In this project, only previously existing LCA for material were used. They came from a French database (www.inies.fr). The process energy was taken into account only to assess the environmental impact of French pig house. In this database, most of final products LCA concern the residential sector. In order to adapt these data for pig housing, raw materials environmental impact were recalculated from the final product. For example, raw concrete LCA has been calculated from concrete beam LCA.

Green house gas definition Green house effect is a phenomenon which prevents infrared ray to escape towards space across the atmosphere. The green house effect is advantageous when it helps to maintain temperature above 14°C on Earth. It can rapidly become a risk factor for climate regulation when it starts to increase heavily. The impact of an activity on green house effect is expressed in kg of CO_2. In the case of pig house building, GHG emission comes from total energy use as describe previously. For building materials this factor is obtained from the "INIES" database.

Limitation of the method Calculation of energy use and GHG emission are based on linear extrapolations of a final product impact from a raw material. In fact, a complete LCA on a pig house building is presently impossible because of the lack of data available on specific pig house building materials. That is the reason why the data used to evaluate the environmental impact of pig house have been modified from existing data. Therefore, our results could be considered as a first approach but not as a precise one.

Assessment of the environmental impact of pig house Calculation of environmental impact has been carried out in five successive steps: 1. pig house materials description; 2. quantification of each material previously listed on the whole life of the pig house; 3. estimation of environmental impact of every material used for the pig house building by using previous LCA study results, database (INIES, ECOINVENT, etc.), 4. Aggregation of the whole environmental impact of all materials on the pig house time life, 5. Analysis of results.

RESULTS The work concerns only the most representative type of pig house in France, as far as pigs are considered. With regard to the sows, even when they are still individually housed in most cases (around 70% of buildings), the evaluation of the environmental impact was performed from farms with group-housed sows because of the new welfare regulation that will impose this system from 2013 onwards. The quantification of each material took into account the manure storage facilities.

Pig house material description All the main materials which are used to build a pig house were defined (Table 1), based on the experience of the engineers working on this project. Neither copper used for electrical network nor plastic pipes for water supply were taken into account.

Table 1. Materials used in pig house and their time of life

Item	units	Time of life (years)
Monolithic	m^3	30
Fibrocement	m^2	30
Carpenter wood	m^2	30
Sandwich panels	m^2	30
Concrete without steel	kg	30
Plastic	kg	15
Polystyrene	kg	30
Steel	kg	30
Stainless steel	kg	30
Galvenized steel	kg	30
Cast iron	kg	15

Quantification of each material for 8 kinds of building To assess the quantity of each material listed into the Table 1, a complete building was drawn in “Google Sketch ‘up” (Cf. Figures 2 and 3).

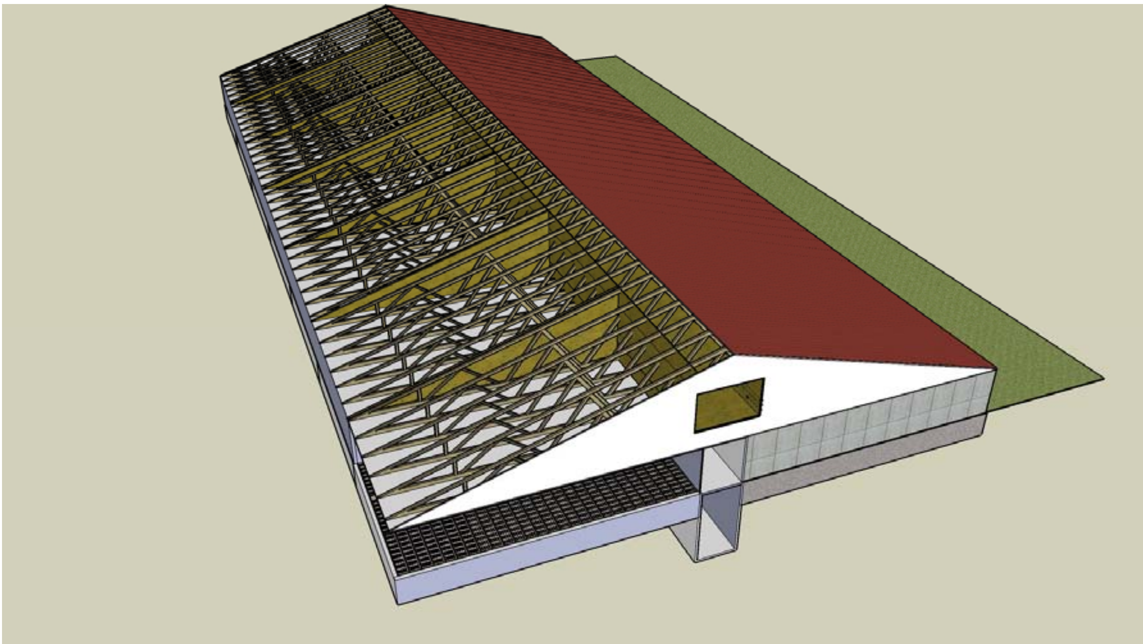


Figure 2. Post-weaning and finishing building

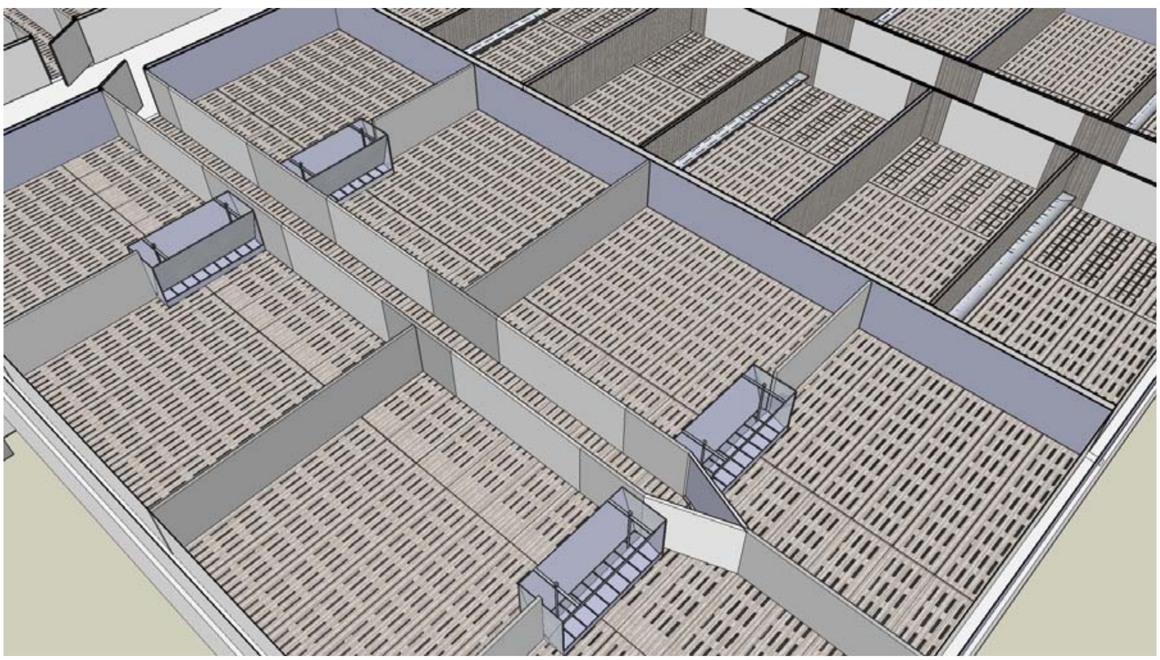


Figure 3. Post-weaning pen

After drawing all the building, we use the metric tools to quantify the volume of each material. Depending on the units use in material existing LCA we convert volume into the right unit. For example, insulation (Polystyrene) is express in kg in existing LCA, so we use the density of insulation material to convert from m^3 to kg. The table 2 shows the quantification results.

Table 2. Amount of material use in pig house

Materials denomination	Units	With SEF				Without SEF			
		Monolithic panels		Concrete panels		Monolithic panels		Concrete panels	
		PSF*	CSF**	PSF	CSF	PSF	CSF	PSF	CSF
Monolithic	m^3/sow	8,64	8,64	0,00	0,00	8,64	8,64	0,00	0,00
Concrete without steel	kg/sow	8756,00	9164,00	10819,00	11227,00	8294,00	8702,00	9693,00	10100,00
Plastic	kg/sow	106,00	34,00	106,00	34,00	106,00	34,00	105,00	34,00
Polystyrene	kg/sow	101,00	101,00	101,00	101,00	98,00	98,00	98,00	98,00
Inox steel	kg/sow	16,00	16,00	16,00	16,00	15,00	15,00	15,00	15,00
Galvenized steel	kg/sow	60,00	60,00	60,00	60,00	60,00	60,00	60,00	60,00
Cast iron	kg/sow	16,00	16,00	16,00	16,00	16,00	16,00	16,00	16,00
Fibrocement	m^2/sow	14,00	14,00	14,00	14,00	13,00	13,00	13,00	13,00
Steel	kg/sow	211,00	232,00	264,00	285,00	211,00	221,00	247,00	257,00
Carpenter wood	m^2/sow	205,00	205,00	204,00	204,00	196,00	196,00	195,00	195,00
Sandwich panels	m^2/sow	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

* PSF : Plastic slatted floor in post-weaning
** CSF : Concrete slatted floor in post-weaning

Estimation of environmental impact of each material After having the full list of materials needed to build a pig house we start to look into existing database in order to define the environmental impact of each of those materials. The main part of LCA comes from "INIES" database, but some of them come from other database. The table 3 shows the resources centers available to give environmental impacts of building materials.

Unfortunately, materials uses in pig house are not directly parts of those databases. That is why we needed to modify some of them to fulfill the table 1 with GHG emission and energy use for each material. The results are shown in table 4.

Synthesis of pig house environmental impact As all the materials and there impacts are known, it is possible to aggregate the results. The table 5 gives the balance sheet of the energy use and GHG emission for the eight building studied.

Table 3. Non exhaustive list of resources center giving environmental impact of building materials.

Name	Website	Comments
Envirobat Méditerranée	http://www.envirobat-mad.net	Ressources center for builders and building designers
VAD Rhône-alpes	http://www/ville-amenagement-durable.org	Materials chips (fees access)
INIES	http://inies.fr	Database with 470 environmental chips about 3000 final product. (free access)
Ecoinvent	http://ecoinvent.ch	LCA database known for its homogeneity (fee access).
DEAM Ecobilan	http://www.ecobilan.com/fr_deam.php	Private engineering company specialized in LCA. Database with fee access.
Association Canadienne de la construction – LEEP	http://www.cca-acc.com/greenbuilding/rating/indexfr.html	Ressources center of Canadian construction association (CCA). Evaluation system LEED Canada-NC1.0

Table 4. Energy use and GHG emission of pig house materials

Materials denomination	Units	Process energy use MJ/units	GHG emissions kg eq CO_2/units
Monolithic	m^3	7,05	0,92
Concrete without steel	kg	1,27	0,14
Plastic	kg	58,00	2,15
Polystyrene	kg	57,00	4,14
Inox steel	kg	35,00	1,31
Galvenized steel	kg	26,00	1,31
Cast iron	kg	25,00	1,28
Fibrocement	m²	3,46	0,32
Steel	kg	0,21	0,01
Carpenter wood	m²	0,083	0,00
Sandwich panels	m²	12,82	0,66

Table 5. Energy use and GHG emission per year of eight king of pig house

Materials denomination	Units	With SEF				Without SEF			
		Monolithic panels		Concrete panels		Monolithic panels		Concrete panels	
		PSF*	CSF**	PSF	CSF	PSF	CSF	PSF	CSF
Primary energy	MJ/sow	857,0	735,0	942,0	820,0	829,0	707,0	886,0	764,0
GHG emission	CO_2 eq kg/sow	67,0	64,0	77,0	73,0	65,0	61,0	71,0	68,0
Primary energy	MJ/m²	57,1	49,0	62,8	54,7	55,3	47,1	59,0	50,9
GHG emission	CO_2 eq kg/m²	4,5	4,3	5,1	4,9	4,3	4,1	4,7	4,5

* PSF : Plastic slatted floor in post-weaning
** CSF : Concrete slatted floor in post-weaning

Analysis of the results We choose to express the results per year in order to compare the need of energy to build the pig house and the energy directly consume when the pig house is in producing phase (electricity, gas, fuel need to run the farm).

The average of energy consumption is 817,5 MJ/sow/year. Pig house which have SEF are using less energy in building since the average is 796,0 MJ/sow/year for SEF farm against 838 MJ/sow/year with sow stall. These results come from that building with SEF system needs less huts separation than sow stall system. The kind of slatted floor has the main impact on energy use since with plastic slatted floor in post-weaning there is an average of 878,0 MJ/sow/year against 756,0 MJ/sow/year for concrete slatted floor. In all the case, plastic has a very bad carbon footprint; therefore, all building techniques using plastic instead of other materials will be charge with a poor environmental balance sheet. Finally, the monolithic panels uses less energy than concrete panels with respectively 782,0 MJ/sow/year and 853 MJ/sow/year. The analysis is strictly the same for GHG emission.

The French pig LCA has been made by S. ESPAGNOL the results are not yet published but it shows that to build a pig house your energy needs are 0,323 MJ/kg of pork and your CO_2 emission is 0,028 kg CO_2/kg of pork in comparison of the global environmental balance sheet of 11,771 MJ/kg of pork and 2,827 kg CO_2/kg of pork.

Conclusion To conclude, the best environmental way to build a pig house is using monolithic panels with SEF system and concrete slatted floor. In fact those results are specific to French pig house because of their way of building. By now, only the farrowing to finish farm pig house have been studied, to improve the results it will be interesting to do the same analysis physiological stage per physiological stage.

To go further, energy use and GHG emission for building a pig house are very low compare to the full environmental balance sheet which include; animal food, producing energy needs, manure management and all the other farm incomes. In a full environmental balance sheet the build of a pig house uses less than 3% of total energy yearly needed and less than 1% of global GHG emission.

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LIFE CYCLE ANALYSIS OF CONCRETE USE IN WALL ASSEMBLIES OF AGRICULTURAL BUILDINGS

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CSAS11032 – LCA and Concrete used Sustainable Farm Building

ABSTRACT Wood is the most popular material used in wall and roof assemblies in agricultural buildings in Canada. There has been some development in the last few years and concrete is now used in some wall assemblies. The main objective of this paper was to compare greenhouse gas (GHG) emissions of two wall assemblies (a typical wall assembly built with wood studs and a wall assembly built with concrete) used in the construction of agricultural buildings following a life cycle analysis (LCA) approach. The results were used to estimate GHG produced by the construction of buildings and manure tanks on a typical pig farm. Emissions produced by the construction of buildings and manure tanks were then compared with the emissions produced each year by the activities occurring on the farm. Total GHG emissions from the wood structure and the concrete walls were respectively 140 and 57 kg CO_2e/m^2. Results showed that for the wood structure wall, 80% of the emissions (113 out of 140 kg CO_2e/m^2) were associated with the steel exterior cladding. Total GHG emissions from a pig barn built with wood structure and concrete walls were respectively 823 and 688 t CO_2e. The barn with the concrete walls reduces GHG emissions by 16%. On a 25 years basis, the construction of the buildings and the manure tanks represents between 1.7 and 3% of the total GHG emissions produced on the farm.

Keywords: life cycle analysis, concrete, pig building

INTRODUCTION

In Canada, concrete is mainly used on farms for the construction of agricultural buildings, manure tanks and silos. Concrete is practically the only material used in the construction of the footing, foundation and floors in most of the agricultural buildings. Wood is the most popular material used in wall and roof assemblies. There has been some development in the last few years and concrete is now used in wall assemblies.

Life cycle analysis (LCA) on pig production showed that animal housing and equipment were not the major contributors to the greenhouse gas emissions produced on a farm. Basset-Mens and van der Werf (2005) showed that building construction accounted for between 0.7 and 1.4% of the greenhouse gas (GHG) produced in pig production in France. In an environmental assessment of Danish pork, Dalgaard (2007) stated that the contribution from animal housing and equipment was less than one percent for all impact categories.

OBJECTIVES

The main objective of this paper was to compare GHG emissions of two wall assemblies (a North American typical wall assembly built with wood studs and a wall assembly built with concrete) used in the construction of agricultural buildings following a LCA approach. The results were used to estimate GHG produced by the construction of buildings and manure tanks on a typical pig farm. Emissions produced by the construction of buildings and manure tanks were then compared with the emissions produced each year by the activities occurring on the farm.

METHODOLOGY

Wall assemblies The typical wall assembly was a steel exterior cladding followed by an air space, a wood structural panel (sheathing), a wood structure (38 x 140 mm at 600 mm on centre) with fibreglass insulation, a polyethylene membrane used as a vapour barrier, an air space and a high-density polyethylene (HDPE) water resistant wall finish.

The assembly of the concrete wall consisted of two layers (76 mm) of cast-in-place concrete separated with a layer of expanded polystyrene insulation (76 mm). In that case, concrete served as the exterior and the interior finish.

A diagram of the wall assemblies is presented in figure 1.

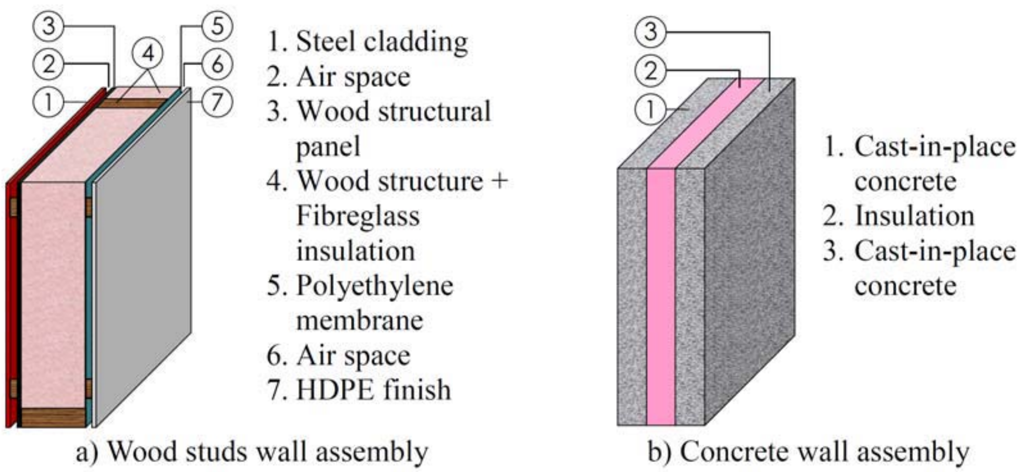

Figure 1. Wall assemblies

Emissions per m² of wall Emissions per m² of wall were estimated using the "EcoCalculator for Commercial Assemblies" developed by the Athena Institute (Athena Institute, 2008). According to Athena Institute (2008), the EcoCalculator offers instant LCA results for hundreds of common building assemblies. The results embedded in the EcoCalculator are based on detailed assessments completed with the ATHENA® Impact Estimator for Buildings, which in turn uses Athena's own datasets. The calculator takes into account: resource extraction and processing, product manufacturing, on–site construction of assemblies, all related transportation, maintenance and replacement cycles over an assumed building service life and structural system demolition and transportation to landfill (Athena Institute, 2008).

The wall assemblies studied in this paper were not included in the EcoCalculator, therefore other calculations were made to take into account the materials used in the walls. The wood stud wall assembly studied in this paper was derived from wall assembly #64 of the EcoCalculator while the concrete wall assembly was derived from wall assembly #23 of the EcoCalculator.

Emissions for the whole farm The analysis was completed in order to verify the impact of the wall assemblies on the emissions produced by the construction of an entire building. Emissions from the manure tank construction were also computed. The results obtained from the construction of the buildings and the manure tanks were compared to the emissions produced by the management of a typical pig farm. The farm was a farrow-to-finish operation of 417 sows producing 8,608 pigs per year (Hamelin et al., 2009). The maternity consisted of a building of 1,375 m^2 and the nursery was a building of 300 m^2. Two buildings of 1,235 m^2 each were required for the growing-finishing.

Except for the wall assemblies, it was assumed that the buildings were all identical with a footing and a foundation made of concrete, a partly slatted floor also made of concrete and a roof made with wood trusses covered with steel. Emissions from construction of the building were computed by combining the results obtained from the studied walls and the EcoCalculator used to evaluate the emissions from the footing, foundation, floor and roof.

Emissions associated with the construction of the manure tanks required for the storage of manure produced on the farm were derived using the values presented in the EcoCalculator. Three manure tanks were required to store the manure produced on the farm. Each tank had a diameter of 36 meters and a depth of 3.7 meters.

Emissions associated with the management of the farm have already been the subject of a LCA and the results were presented in Pelletier et al. (2010). The analysis included emissions from pigs, energy consumed on the farm, land application of manure and fertilizers, crop production and harvesting.

RESULTS

Emissions per m^2 of wall Total GHG emissions from the wood structure and the concrete walls were respectively 140 and 57 kg CO_2e/m^2. Table 1 presents detailed GHG emission results for the two wall assemblies. Results show that for the wood structure wall, 80% of the emissions (113 out of 140 kg CO_2e/m^2) were associated with the steel exterior cladding.

Table 1. GHG emission results for the studied wall assemblies in kg CO_2e/m^2

	Wood structure wall	Concrete wall
Exterior	113	21.5**
Insulation	13*	14
Inside	14	21.5**
Total	140	57

* Include: wood structural panel, wood structure, fibreglass insulation and polyethylene membrane.

** Concrete only.

Emissions for the whole farm

Barn construction Total GHG emissions from a pig barn built with wood structure and concrete walls were respectively 823 and 688 t CO_2e. Table 2 presents detailed GHG emission results for the two barns. The barn with the concrete walls reduced GHG emissions by 16%. Results showed that for the barn with wood structure walls, 28% of the emissions were associated with the wall assembly.

Greenhouse gas emissions produced by the footing, foundations and floor represented 55 and 79% of the total emissions for a pig barn built with wood studs and concrete walls.

Table 2. Total GHG emissions from a pig barn built with wood structure and concrete walls in tonnes of CO_2e

	Barn with wood structure walls	Barn with concrete walls
Roof	138	138
Walls	229	94
Inside walls	4	4
Footing, foundation and floor	452	452
Total	823	688

Manure storage tank construction The construction of the three manure storage tanks produced 144 t CO_2e.

Farm operation Total GHG emissions produced by the pig farm presented in this paper, varied form 1,339 and 2,295 t CO_2e/year (Table 3). The emissions were affected by the type of soil and the phosphorus content of the soil where the manure was spread. On a 25 year basis, the construction of the buildings and the manure tanks represents between 1.7 and 3% of the total GHG emissions produced on the farm.

Table 3. GHG emissions from the construction of the barns and manure tanks and from the farm operations

	Barn with wood structure walls	Barn with concrete walls
Barn construction	823 t CO_2e	688 t CO_2e
Manure tank construction	144 t CO_2e	144 t CO_2e
Sub-total for infrastructures	967 t CO_2e	832 t CO_2e
Farm operations	1,339 to 2,295 t CO_2e/yr	1,339 to 2,295 t CO_2e/yr

DISCUSSION

In North America, although the use of concrete in the construction of agricultural building is indisputable, its use is mostly limited to foundations and floors while walls are still built with wood and steel.

The use of concrete provides several advantages such as: availability, durability, ease of maintenance and weather resistant. In addition, concrete walls significantly reduce the problems associated with rodents. These benefits have not been shown by the LCA. Therefore, demonstrating the overall benefits of the use of concrete for the construction of the walls would be relevant.

The use of concrete could be beneficial in the development of new concepts of farm buildings including manure management and energy and water recovery. The flexibility of concrete opens the door to an unlimited number of concepts. The use of concrete in the building design would then contribute to reduce emissions over the entire farm.

CONCLUSION

The main objective of this paper was to compare GHG emissions of two wall assemblies used in the construction of agricultural buildings following a LCA approach. Total GHG emissions from the wood structure and the concrete walls were respectively 140 and 57 kg CO_2e/m^2. Results show that for the wood structure wall, 80% of the emissions (113 out of 140 kg CO_2e/m^2) were associated with the steel exterior cladding. Total GHG emissions from a pig barn built with wood structure and concrete walls were respectively 823 and 688 t CO_2e. The barn with the concrete walls reduced GHG emissions by 16%. On a 25 years basis, the construction of the buildings and the manure tanks represents between 1.7 and 3% of the total GHG emissions produced on the farm.

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ECO-CONCEPTION GUIDELINES FOR LIVESTOCK HOUSING

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CSAS11020 –Topic IV: Concrete and Green Building (ex. Label and Environmental Declaration)

ABSTRACT Contrary to livestock housing sector, several eco-conception guidelines already exist for services and residential sectors. Agricultural sector is evolving to a better environmental management, including buildings. Our work was to adapt existing guidelines to livestock housing. A guideline was developed divided in 4 points: Merging buildings and landscape for ecological building conception or renovation; Materials, resources and nuisance management in order to decrease raw material use to limit pollution and improve recycling; Energy, water and waste: reduce the needs, promote the renewable energy and limit pollution; Comfort and health in order to preserve the staff and animals welfare. A charter was elaborated with 68 points. A building project fulfills the main aims of the charter if 44 points are respected: 23 points which are obligatory and 21 more chosen among the 45 other points. The main part of the project was to make a guideline which works with all kind of animal husbandry and flexible enough to introduce an eco-conception thinking in a large number of project including both new and renovated buildings. With this charter, buildings which are mainly built in concrete can also fulfill the 44 required points. In conclusion, the charter has been successfully tested even if it appear that livestock housing constructors are not yet ready to work in this way. It also showed us that farmers are not ready to pay an extra price to make their building more sustainable because of the lack of investment returns.
Keywords: eco-conception, Green livestock building label

INTRODUCTION In France, a label exists for low emission houses in the residential sector. As nothing existed in livestock housing, three French animal institutes have started a project: “Green building and livestock housing”. The main aim of this project was to transpose the residential sector label to livestock housing. Compared to residential sector, the livestock housing has 2 mains distinctions: it is a producing building and the animal welfare needs to be taken into account.

Definition of eco-conception Eco-conception is about creating an environmentally friendly building. French eco-conception principles mainly take their origin in the high environmental quality label (HEQ) but also in others charters from France or foreign countries.

An eco-building has to minimize its environmental impact during the build but also during the building use. The building has to be part of the landscape by valorizing the best of natural environment and using as much as possible the local resources. The HEQ label is, in France, the first clear charter leading us to this kind of building. This label is about global management of building in order to minimize negative impact on the external environment as well as creating a satisfying inside climate.

There are 14 targets to deal with in order to obtain the HEQ label. These targets are split into 4 sections, Eco-building, eco-management, inside comfort and welfare of inhabitants.

Method There was no real scientific method to fulfill the aim of this project. Work meetings, brainstorming and interviews with breeders and livestock advisers were the main tools used. However, before starting to produce a new green building label for the livestock housing, we began by searching existing labels even if they were not related to livestock housing.

After the inventory of existing label, the differences of livestock housing have been listed in order to take into account the fact that livestock housing is used to produce animal products. Then, a charter has been made with technical index cards for each point of the charter. In fact, within existing labels, the points are not clear enough and can be interpreted in many ways. That is why a technical index card per point explains with examples what this point means.

Finally, we tested the charter and technical index cards with farmers to improve it and to know if each point were clear enough for the future users of the charter.

Results After a year of work we managed to create a label called "Eco-construire un bâtiment d'élevage" (ECBE).

ECBE is a label (CF. Appendix A) more flexible compared to HEQ and it takes into account distinction of livestock housing, comfort and welfare of animals as well as the comfort and welfare of workers. Breeders choose the targets they are willing to fulfill among several points. ECBE is split into 4 sections: 1-landscape merger; 2- Materials, resources and nuisance management in order to decrease raw material use to limit pollution and improve recycling; 3- Energy, water and waste of activities: restrict needs, restrict pollution emissions, encourage renewable energy sources; 4- Comfort and health: preserve health of personnel and of animals, improve their comfort.

Among the 68 points, the farmer has to fulfill 23 necessary points (Cf. Appendix A "lines with an X in the last column") and choose 21 other points among the 45 remaining points. Each point is equal to another. In fact, there is no balance between the points. This label is a matter of progress and not a perfect charter. With this way of thinking, breeders who want to use this charter can improve their building by choosing the items in the charter in agreement with their will.

During the first test of the charter breeders have been afraid because they saw in this label, new environmental pressures but after a while they thought that this charter could be a helpful tool to manage a new construction. The pig breeders didn't want to put a label for their pig house through this charter but they would like to use it as a building checklist during the construction.

Conclusion The ECBE Label is a new notion in agricultural sector. For some French government subsidies, the environmental impact of livestock housing is taken into account. As nothing existed, the subsidies were given to farmers who were using wood in their livestock housing. That is why this project was about creating an eco-policy label in order to prove that pig houses mainly built in concrete panels are able to be as good as wood building since they fulfill a package of goals (energy and water management for example) .

ECBE Label is more about making progress in ways of building livestock houses than having very few livestock houses extremely respectful of the environment. In fact, breeders are not ready to pay extra money in order to have an ECBE label. What they want is more about saving money by reducing energy use, water consumption. In that, the ECBE label gives them all the key steps when they start a new building project. Finally, this guideline will not be used as a label but more as a tool to assist builders.

Appendix A **ECBE Label « ECO-CONSTRUIRE UN BÂTIMENT D'ELEVAGE »**
Precondition: Constitution of a multidisciplinary team (builders, architect, advisers, constructors …)

I Landscape merger							
I 1 Relation of the building with the site –							
1-1 I try to use existing building							**X**
1-2 I take apart useless buildings							
1-3 I take into account natural and environmental qualities of the site to establish the building							
I 2 Circulation and movements							
2-1 I restrict vehicles movement around the site							
2-2 I organize accesses for deliveries, pick-ups and garbage collection							**X**
2-3 I organize circuits towards plots and I adapt rotation of crops in order to reduce distance							
2-4 I study the possibility of crops exchanges to optimize establishment							
I 3 Waters of rain and streaming water							
3-1 During the building conception, I take into account the hydrology of the site							
3-2 I accomplish collect network of water from roofs and streaming water (trenches)							**X**
3-3 I separate waters from roof and mucky streaming waters							**X ou R** according to context
3-4 I collect and use waters from roof.							
I 4 Impact on neighborhood during the building life							
4-1 I accomplish an architectural plan (choice of the shape of the building, of materials, of colours …)							
4-2 For the realization of a new building: I restrict discomfort (noise, dusts, smells)							X
I 5 Biodiversity on the built site							
5-1 I keep and I develop green areas with local species of vegetation							
I 6 Local resources							
6-1 I choose the system of building which is adapted to local resources (straw, …)							
II Materials, resources and nuisances of building: Restriction of raw materials use, disposal and optimize recycling							
II 1 Economy of materials							
1-1 I optimize the building weight and the quantities of materials needed for the building (building, annexes and manners)							**X**
1-2 I try to find different uses of areas when it is possible							
II 2 Materials and technology of building							
2-1 I take into account the life cycle analysis of materials use							**X**

in the building.							
2-2 I choose eco-label materials preferably							
2-3 I apply the principle of circumspection to materials introducing health risks							
II 3 Waste of construction site							
3-1 I prove that firms organized sorting, management of the waste and recovery of the polluted effluents of the construction site							
3-2 I prove that the firm definitely considered and estimated the cost of treatment of waste in the documents answering the tender							
3-3 I consider the management of debris and the demolition debris with the firms present on the construction site							
II 4 Nuisances of construction site (noise, dusts, circulation, visual aspect)							
4-1 With firms, I organize the construction site to minimize nuisances and I ensure that this organization is carried out during the construction site							
4-2 I inform the neighborhood of my construction project and take into account their opinions							
4-3 I choose materials and useful techniques with few nuisances							
4-4 I ask that firms promise to use devices respecting acoustical regulation							
II 5 Organization of the Construction site							
5-1 I am informed that I must name a SPH (Security Protection of Health) coordinator							**R**
III Energy, water and waste of activities: restrict needs, restrict polluting emissions, favour renewable energy sources							
III 1 ENERGY SAVING							
1-1 I try to predict my future energy consumption							**X**
1-2 I accomplish an energy diagnosis, once the building is functioning							**X**
1-3 I make my electrical installation in conformity with standards							**R**
1-4 I set up data loggers (electricity, gas, fuel oil) for the building							**X**
1-5 I set up devices of recovery of warmth or bursars in energy							
1-6 I keep first and foremost the resolutions of natural aeration (except if heating or health pressures)							
1-7 If need be, I assess and I optimize the systems of heating and aeration							
1-8 I optimize the manure management by competitive means to avoid over consumptions of energy							
1-9 I favour natural lighting							
1-10 I choose lighting systems with low consumption and piloted according to the needs							**X**
III 2 Use of renewable energy sources							
2-1 I enquire to know the different resolutions of use of renewable energy sources adapted to my needs (usage, potency …)							
2-2 I try to use renewable energy first (hot water, electricity, heat pump, biomass).							
III 3 Consumption of water							
3-1 I accomplish a prediction of water consumption							

3-2 I check my water consumption, once the building is functioning							
3-3 I install a specific water meter for my livestock house							**X**
3-4 I consider equipments and practices with low water consumption							
III 4 Waste, manure and gas emission							
4-1 I set up a system of selective waste sorting if a network of waste sorting is available.							**X**
4-2 I choose materials with low maintenance level in order to restrict waste due to the maintenance of the building							
4-3 I respect the procedures of maintenance, doses of products and I try to limit their use							**X**
4-4 I create a protected storage areas for dead animals							**R**
4-5 I manage organic manure use							**R**
4-6 I set up equipment or practices reducing the gas emissions							
4-7 I choose a system of treatment of clear manure							
III 5 Building maintenance							
5-1 I preserve my building to maintain technical results							
5-2 I assure maintenance by nonpolluting techniques and without nuisances							
IV Comfort and health: preserve health of personnel and of animals, improve them comfort							
IV 1 Bioclimatic requirements = temperature, hygrometry, speed of air, gas							
1-1 I specify climate needs according to the physiological stage of the animals and I implement all necessary means to success							
1-2 I consider a air renewal system good enough to prevent against stream of fresh air							**X**
1-3 I study the workstations ergonomic and I optimize it							
IV 2 Acoustical comfort							
2-1 I restrict the sources of noise							**X**
2-2 I accomplish a soundproofing of equipments and motors							
IV 3 Lighting comfort							
3-1 I bring adequate quantity and quality of light, for workers and animals							
3-2 I choose bright colors which leads to a calm animals behavior							
IV 4 Health quality							
4-1 I choose materials with less VOC , ether of glycol, and formaldehydes than recommendation by the WHO							
4-2 I design my building for a maintenance and an easy cleanliness (cleaning and disinfection)							**X**
4-3 I set up a health barrier for the access to animal husbandry (with disinfection)							**X**
4-4 I avoid crossroads between clean and dirty circuits (principle of the step forward)							
4-5 I design my building so as to respect the good practices of animal husbandry (I am certified Charter of the good practices of animal husbandry or I try to apply the Best Available Techniques)							
IV 5 Water quality							
5-1 I accomplish the annual analyses of drinking water							**R**
5-2 I protect the surrounding water wells from all potential contamination							

Concrete Floors for Animal Housing

Section 4

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COMPARISON OF FOUR MEASUREMENT TECHNIQUES FOR ASSESSING SURFACE ROUGHNESS OF CONCRETE FLOORS FOR ANIMAL HOUSING

Stéphane Godbout, Lorie Hamelin, Heiko Georg, A. Avalos Ramirez, Frédéric Pelletier

Abstract

The quality of concrete floors used for animal housing is a key determinant of animal welfare and health, and consequently for the overall production performance. A core parameter to characterize concrete floors quality and their propensity to represent a risk for animal welfare is surface roughness. Yet, various methods exist for determining floor surface roughness and their accessibility to farmers and to some extent concrete floors suppliers for animal houses is rather variable. This study compares two different measurement methods for assessing concrete floors surface roughness, varying in complexity and precision: (i) laser-based measurements; and (ii) sand patch method. The roughness measurements were performed on a set of concrete slatted floors for fattening pigs which have never been used in pig houses. This study concluded that the sand patch method is a quite simply and economic alternative to determine surface roughness when an accurate method is not available. However, in the future, in order to increase the precision, research works have to be done.

Keywords: concrete, floor, surface roughness.

Introduction

The physical properties and quality of materials used for floors in animal houses strongly influence the animal welfare and health, as well as the overall production performance of farms. Applegate (1988) indicates that an ideal material for floors in animal houses should minimize animal discomfort, injury or disease and should not become deformed, deteriorated or requires extraordinary maintenance. Concrete presents several of these characteristics and it is widely used as floor material in pig houses.
A core parameter that defines the concrete floors quality and their propensity to represent a risk for animal welfare is surface roughness. The floor has to be not too rough (abrasion of claws) but also not too smooth (risk of slipping and falling) in order to minimize foot and skin lesions in all age of animals (Zoric et al., 2008; Hanson et al., 1999). In fact, floors with high surface roughness reduce animal slipping, but may cause acute lameness if too smooth.

Given the economical importance rough floors may have for the farmer, it could be desirable to measure floor surfaces roughness directly on-farm. This would allow the farmer to identify and replace the slats suspected to be too rough.

There are various methods to determine floor surface roughness and their accessibility to farmers and to some extent concrete floors suppliers for animal houses is rather variable. Many techniques available are not reliable for on-farm measurement. The availability of a cheap and friendly use technique would be interesting for farmer concerned by the floor characteristic over the time.

Two techniques are well documented in the literature: the sand patch and laser beam method. The first one use the average texture depth measure the volume of the sand filling the grooves (De Belie, 1995). The laser beam is a high-precision method using the heights of the peaks and valleys of the surface to determine the roughness (Franck and De Belie, 2006).

The aim of this paper is to validate the results obtained with this low tech technology with those obtained from a “high-tech” and well established and high-precision technology.

Materials and method

Concrete element

For all the measurements to be carried, same concrete slats were used. These slats are intended for fattening pig barn and have never been used. They have been kept outside from June 2006 to November 2007 and thus submitted to the meteorological conditions found in Braunschweig (Germany) between these dates. This fact is believed not to be a disturbance, the elements can be considered as new elements after first or second wet cleaning.

The concrete used has concrete strength class (Germany) C 40/ C50 and it was reinforced with two steel bars. The concrete slats were produced according DIN/EN 12737 with a maximum load per animal of 250 kg per slat. The global dimensions of the slats are shown in figure 1.

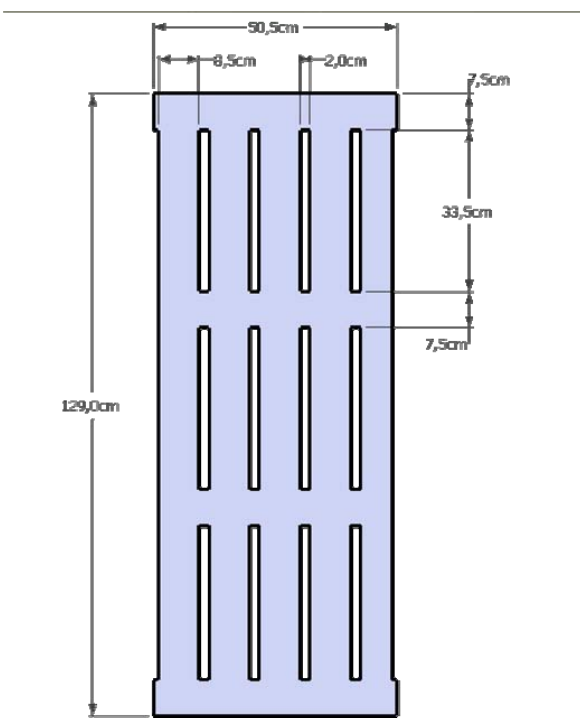

Figure 1. Illustration of the commercial slatted floor element used in this study

Slat elements have been sliced into smaller slats in order to have uniform samples of 8.5 by 33.5 cm. A total of 27 samples have been used .

Before testing, all slats samples were washed in order to ensure uniformity between all slats as well as to get rid of the contamination that occurred during the slicing of the slats. A cleaning procedure adapted from Broes (1999) has been carried out.
Roughness measurements

Roughness average (R_a)

This study focuses on the measurement of the roughness average (R_a) only. This represents the arithmetic average of all the surfaces areas between the profile and its center line, divided by a reference length selected to include important roughness features, but to avoid errors of form (Figure 2). In other words, R_a represents the average length of the peaks found along the profile.

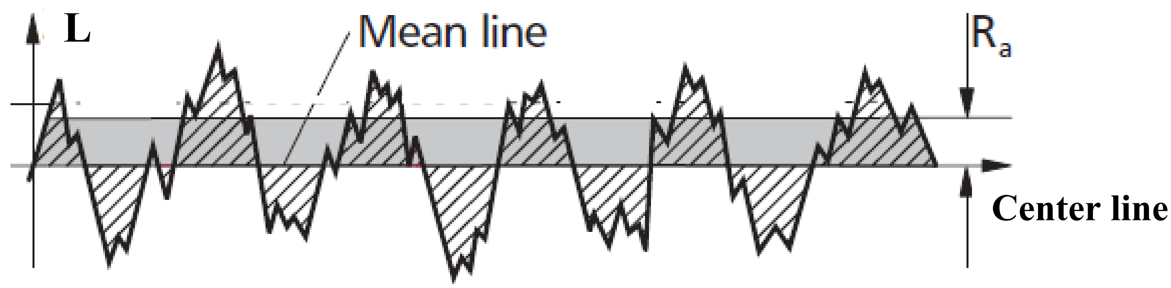

Figure 2. Roughness average, R_a

Sand-patch method

This method consists of spreading a predefined volume (V) of fine sand on the floor surface and estimate R_a through measuring the area of the patch (A). The roughness is then simply determined as $R_a = A / V$. This concept is further described in De Belie (1995) as well as in De Belie et al. (2000) and has been highlighted as a reliable method for assessing floor roughness by Franck et al. (2007).

A graduated cylinder of 10 ml (± 0.05 ml) was used to measure 1 ml of sand, resulting in a standard volume of 0.855 cm^3 for all tests. The sand was gently and uniformly distributed with a scraper over the slat surface in order to form a rectangular surface, as illustrated in Figure 3. For each slat, the longitudinal edge of the rectangle (Y) was measured in 6 different locations and the average was used to determine the rectangle area. Similarly, the horizontal edge (X) was measured in two different locations and averaged.

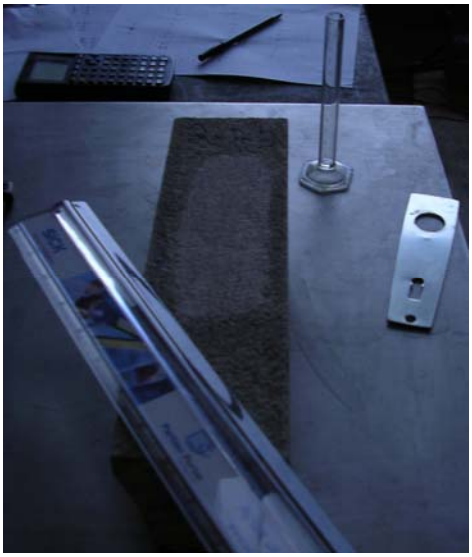

Figure 3. Sand application on the concrete element

Laser beam

The roughness of all concrete samples have also been measured through the automated laser measurement device developed at the Magnel Laboratory for Concrete Research (Ghent, Belgium) and described, among others, in Franck et al. (2007). With this device (Figure 4), the height of the surface peaks and valleys is determined with a high precision laser beam mounted on a table allowing controlling the distance between the laser beam and the samples in the X and Y direction. It allows measuring R_a with an accuracy of 7 µm (Franck and De Belie, 2006).

Figure 4. The automated laser measurement device with stepping motors (bottom and right) and concrete floor samples on the test bed (Franck and De Belie, 2006).

Results and discussion

Two different methods (low and high tech methods) were used to determine the surface roughness of concrete slats.

Table 1 shows that the values obtained in the presented study using both methods were in the range of values reported in the literature for concrete floors, which are from 190 to 900 µm for the sand patch method, and from 80 to 900 µm for the laser beam method. In the case of the laser beam method, the device used for the present study was the same one used by Franck et al. (2007) and the results obtained here were in the range of values reported by them.

The surface roughness determined with sand patch method presented values from 27.4 to 94.4 µm, while those obtained with laser beam varied from 71 to 92 µm (table 1). This shows that the values of surface roughness presented higher variability when they were determined using the sand patch method than with the laser beam. It was probably due to the precision of each method and the random error which should be higher for sand patch than for laser beam method. If we consider that the laser beam method gives more accurate values than the sand patch method, the results obtained in the present study show that the average of surface roughness is underestimated.

The difference between the results obtained by these two methods is related to many factors, two can be discussed here. First, the size of sand particles and human factors related to the cone manipulation produce high variations among the different determinations for the same material. Second, in the present study, a rectangular patch has been used instead of the circular patch used by the De Belie (1995) and Franck et al. (2007). For slat element, the rectangular patch is probably more what farmers are willing to use but may be it compromises the accuracy and can explain partly the underestimation shows in table 1.

Table 1 Surface roughness for concrete floors measured using sand patch and laser beam methods.

Method	Range of values of surface roughness (µm)	Mean of surface roughness (µm)	Standard deviation (µm)	Reference
Sand Patch	27.4 - 94.4	54.5	± 20.9	This study
Sand Patch[a]	190 - 590	-	-	Franck et al., 2007
Sand Patch[a]	200 - 900	-	-	De Belie, 1997
Laser beam	71 - 92	85.3	5.1	This study
Laser beam[a, b]	80 - 296	-	-	Franck et al., 2007
Laser beam[a]	50 - 900	-	-	De Belie and Monteny, 1998

a: The range of values corresponds to different concrete materials.
b: The surface roughness was measured with the same device used for the present study.

Conclusion

Two different methods were used to determine the surface roughness of concrete slats. The sand patch method can easily be adapted and performed in farms; it does not require complex equipment. The other method is based on the use of a laser beam. It is more complex to perform than the sand patch method and it requires sophisticated equipment in order to calculate the actual value of surface roughness.

The sand patch method is a quite simple and economic alternative to determine surface roughness when an accurate method is not available. However, in the future, in order to increase the precision, research works have to be done.

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SKID RESISTANCE AND DURABILITY OF COATED AND UNCOATED CONCRETE FLOORS IN DAIRY CATTLE BUILDINGS

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CSAS11037 – Section 4: Concrete Floors for Animal Housing.

ABSTRACT Passageways in cattle buildings should provide durable and non-slippery surfaces to allow cattle an unrestrained locomotion and expression of behaviour. In order to evaluate walking areas of cattle buildings and their grip or skid resistance, measures with a Skid Resistance Tester to obtain SRT – values were performed on several dairy farms in Germany. Uncoated concrete, brushed concrete, epoxy resin coating and mastic asphalt as coating of concrete was investigated. Results demonstrated, that even high quality concrete had low SRT – values and thus low grip, whereas mastic asphalt showed high SRT – values, meaning good grip. Processing uncoated concrete surface and epoxy resin coating lead to higher SRT – values compared to mastic asphalt. Due to changes of micro-roughness of mastic asphalt, attention should be paid, to keep passageways as clean and dry as possible.

Keywords: SRT, skid resistance tester, cattle housing, floor characteristics

INTRODUCTION Interaction of bovine claws and different floor types in cattle housing is an important factor regarding claw health, lameness and locomotion behaviour of cattle. In many loose housing cattle buildings concrete flooring is widely used for passageways. But even high quality concrete is eroded after a few years chemically by faeces and mechanically by scrapers. Concrete floors become slippery and affect walking behaviour of cattle negatively. One solution to improve surface properties of concrete us in animal houses is coating by epoxy resins or a layer of mastic asphalt. To compare those solutions and to show the durability we measured different flooring surfaces of different ages two times after 1 year. To characterize the floor properties we use the skid resistance test, a pendulum friction testing method, which was developed for measuring road surfaces. Concrete flooring skid resistance is one of the multiple choices to characterize flooring properties. The skid resistance tester was used by Nilsson et al. (1988) to measure grip of floors in dairy buildings. Franck et al. (2007) compared SRT - values of floor surfaces with different friction measurement techniques and measured the surface roughness by using a laser beam. In this study, the SRT - values showed a positive correlation with dynamic coefficient of friction under wet conditions and it shows significant correlations with roughness. Van der Tol et al (2005) considered dynamic forces when measuring the coefficient of friction .Their results show, that the friction of concrete floors is not sufficient for unrestrained cattle locomotion. The authors recommend special attention to the design of concrete floors. Thus the aim of study was to measure SRT – values of different concrete floors under practical conditions to evaluate effects of coating and grinding compared to uncoated concrete.

MATERIAL AND METHODS SRT Measures were performed in six different cattle buildings providing floors of uncoated concrete, brushed concrete, epoxy resin coating and mastic asphalt. The cattle buildings were located in the federal state of Thuringia in Germany. Measures were done using a Skid Resistance Tester (Figure 1). The SRT value is a kind of dynamic friction measuring method.. The pendulum (Figure 1 and Figure 2) is accelerated up to 2.5 ms^{-1}, a slider with a rubber block at the pendulum scratches on the watered floor for an equal distance. Depending on the floor roughness, the pendulum is de-accelerated and moves a pointer at a scale, the so called SRT - Value. A value of zero is obtained without any friction or free swinging of the pendulum arm. The surface has to be watered and cleaned before the measure. Figure 3 shows the SRT Tester in its working environment on a passageway of a cattle building.

SRT-Values were obtained from 40 different points of evaluated floors. A minimum of seven repetitions per measuring point was set to achieve reliable results. In total, 320 single SRT – Values were measured. Data were recorded using a data sheet and transferred to an Excel-sheet to calculate temperature adjusted SRT – Values per measuring point. Mean values per floor type were calculated in the same data sheet as well as standard deviation.

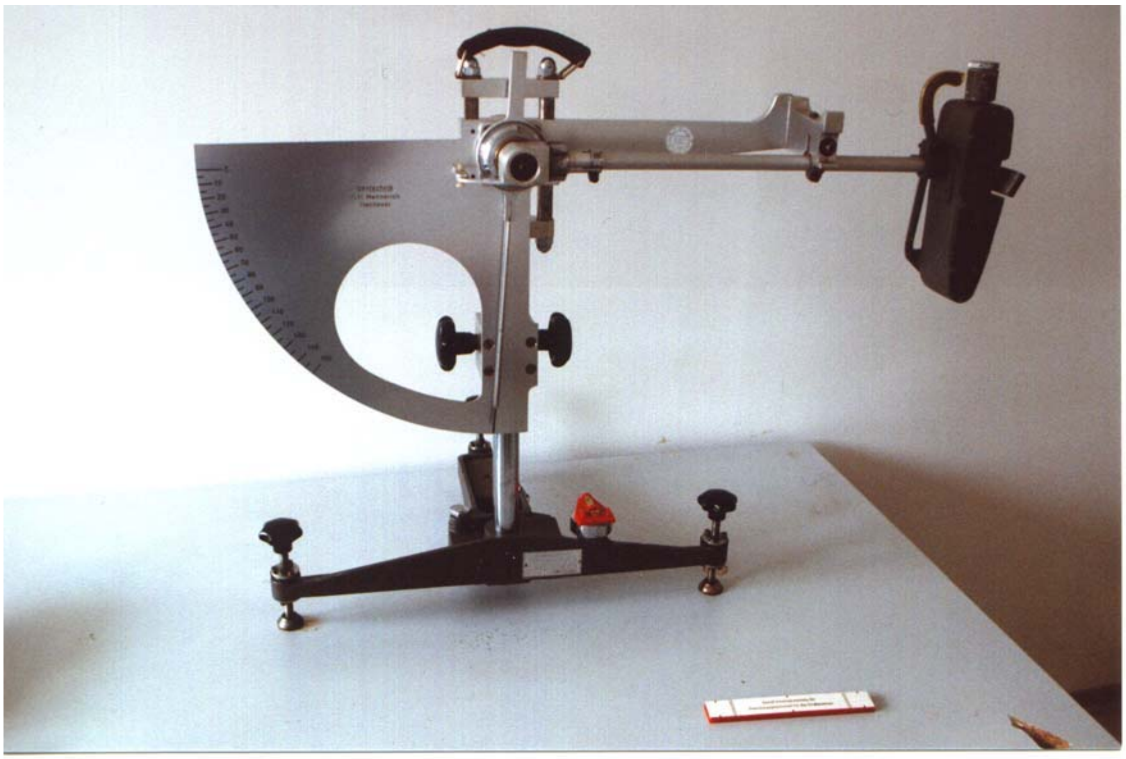

Figure 1. Pendulum tester to measure skid resistance (SRT) and tools for sliding length adjustment.

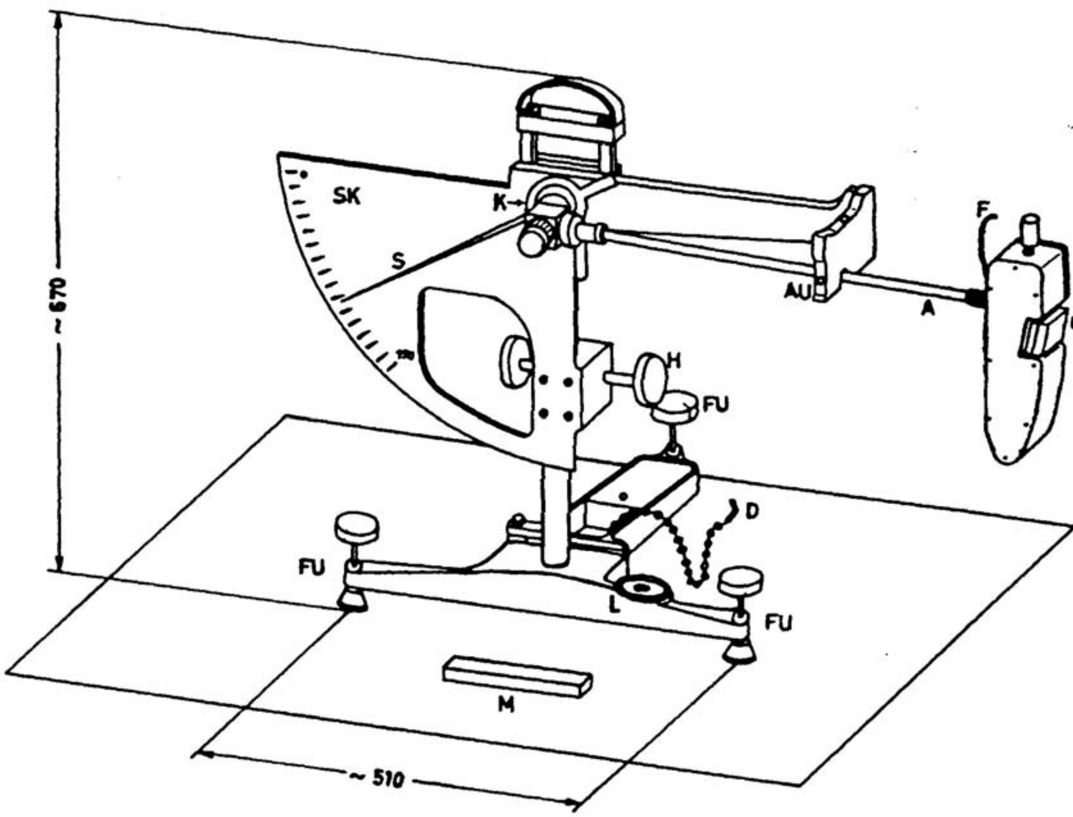

Figure 2: Scheme of Pendulum tester for skid resistance (SRT) with leveling screws (FU), spirit level (L), scale (SK), pointer (S), pendulum arm (A), release catch and release button (AU), rubber slider (G), control for vertical movement (H) and scale for sliding length adjustment (H).

Figure 3: Pendulum tester to measure skid resistance (SRT) on a wet concrete floor of a cattle building..

RESULTS .For a comparison of SRT Values it is necessary to know how to interpret them. In Table 1 a characterization of SRT – Values shows, that acceptable flooring for cattle starts with SRT – values of 50 to 60.

Table 1. Interpretation of SRT-Values.

SRT-Value	Grip
> 70	excellent, maybe abrasive
60 - 70	good
50 - 60	sufficient
40 - 50	not sufficient
< 40	slippery

The results from our Skid Resistance Tester are shown in Figure 4. Six years old concrete and concrete with epoxy resin coating (1 year old) with SRT <= 40 showed poor grip values. The floors were suitable as walking surfaces in cattle buildings. A recently installed brushed concrete had acceptable grip at SRT of 49, whereas mastic asphalt coating resulted in excellent good and sufficient grip. Mastic asphalt has even if it is 5 years old good grip properties.

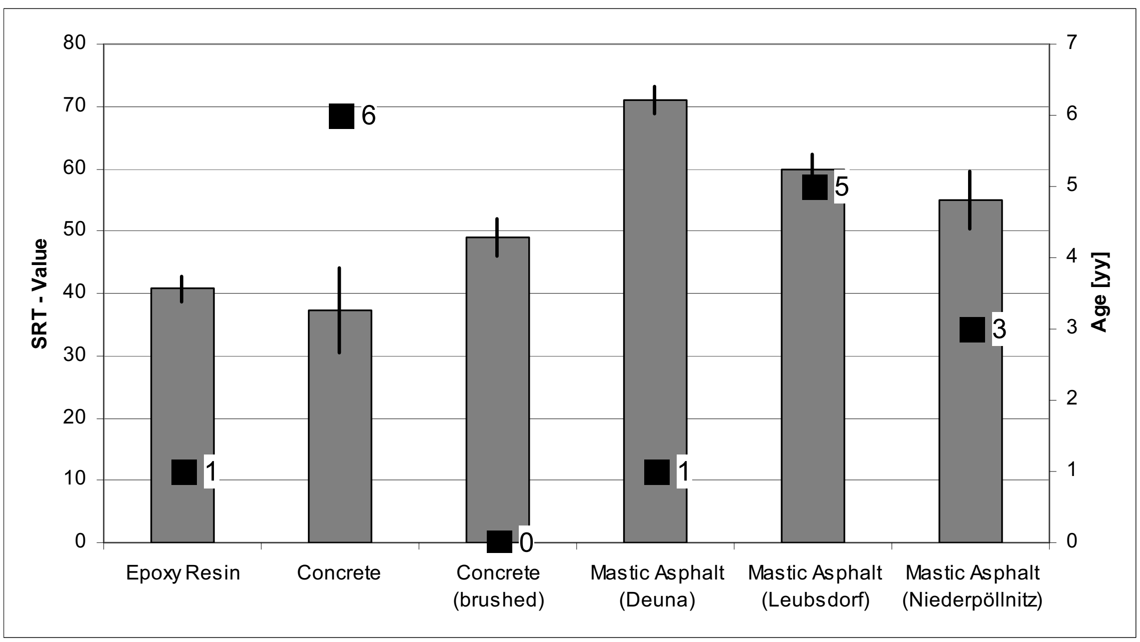

Figure 4. SRT – Values for 6 different floor types (grey column) and age in years (black square).

In order to show how farmers could improve a poor grip of concrete floors, we measured concrete floors and epoxy coated floors before and after renovation. The results are shown in Figure 5. Grinding of poor screeded concrete resulted in good grip of SRT = 55. An epoxy resin coated floor with slippery properties was sandblasted to improve grip. The sandblasted epoxy floor had increased SRT – values of 44 which maybe sufficient.

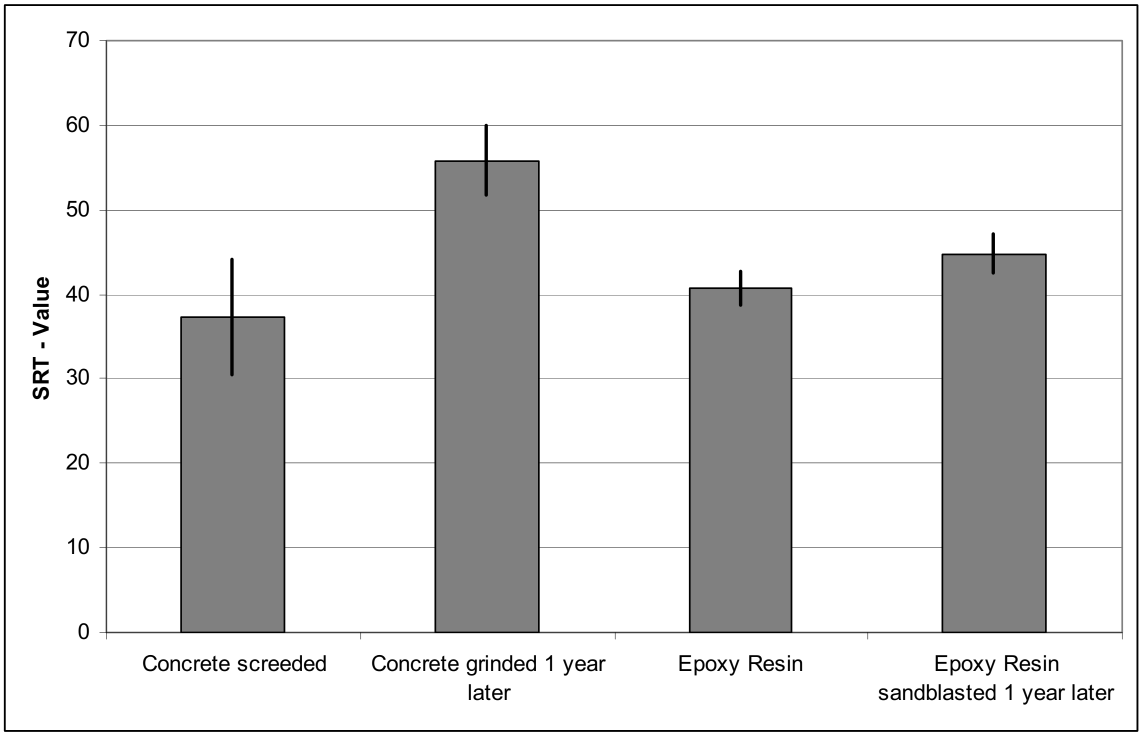

Figure 5: Concrete and epoxy resin coated concrete before and after processing.

DISCUSSION Results of SRT – values indicate that uncoated concrete should either be coated with mastic asphalt in order to get durable grip or – if left uncoated – finished by using a brush. Considering results of Franck et al. (2007) mastic asphalt can maintain grip for a long time. The reason can be a changed micro-roughness, even mastic asphalt gets eroded by cleaning device and faeces. As a result, "peaks" of rigid components remain within the surface and thus lead to constantly high SRT – values. Concrete will remain or erode to a slippery surface within the same time. Our results seem to be in line with those of Van der Tol et al. (2005), who stated, that the dynamic coefficient of friction, which is highly correlated with SRT – values, is not suitable for an unrestrained cattle locomotion. Epoxy resin as coating remained even after sandblasting relatively slippery and had lower SRT – values compared to grinded concrete surface.

CONCLUSION.Mastic asphalt seems to be a durable coating with high grip for a period of 8 to 10 years, even if the micro-roughness will be changed. Due to possibly higher abrasiveness of older mastic asphalt, wet passageways for cattle should be avoide in order to keep the claw dry and more resistant against erosion. Especially feeding passageways, where cows spend 5 – 6 hours per day, should be cleaned more frequently when using mastic asphalt. Even high quality concrete is eroded after a short period of 2 – 3 years and will be no longer a suitable surface for passageways in cattle buildings. Grinding may be an option to improve grip of uncoated concrete but can become costly. Epoxy coating in our case showed poor SRT results.

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AN INVESTIGATION ON THE EFFECTS OF NUMBER OF CHICKS AND VENTILATION SYSTEM TYPE ON ENERGY EFFICIENCY IN YAZD BROILER FARMS

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ABSTRACT The objective of this study was estimation of energy consumption during various stages of broiler production and analysis the effects of numbers of chick and ventilation system type and concrete floor design on energy efficiency of these units. Data were collected from 44 growers by using a face to face questionnaire method during January–February 2010. Collected data were classified into three groups according to the numbers of chicks and two groups according to type of ventilation system. The effects of numbers of chicks at three levels (less 10000 bird, 10000 to 20000 bird and more 20000 bird) and ventilation system type at two levels (tunnel and cross ventilation) were analyzed on energy efficiency. The results indicated energy ratio, energy productivity and specific energy were 0.15, 0.014 kg MJ^{-1} and 71.95 MJ kg^{-1}, respectively. The results of ANOVA presented that the number of chicks had significant effect on energy efficiency ($P<0.01$) and level 3 (more 20000 bird) had the biggest energy ratio. The effect of ventilation system type was significant at 5% level and tunnel ventilation system that used in 61% of units had the bigger energy efficiency.
Keywords: Broiler, Energy Efficiency, Concrete Floor, Ventilation System.

INTRODUCTION Sustainable agriculture is a production system in which the management and conservation of the resources base and the orientation of technological and institutional components changes in such a manner as to ensure the attainment and continued satisfaction of human needs for the present and the future generations (Food and Agricultural Organization, 1991). Nowadays hens are inter-breaded, so chicks in a short period reach to desirable weight. The intensity of energy use on broiler farms is high and studies on input-output energy pattern on broiler farms are very important. Efficient use of agricultural product energies helps to achieve increased production and productivity and contributes to the profitability and competitiveness of agriculture sustainability in rural living (Singh et al., 2002). Energy is used in almost all facets of living and in all countries, and makes possible the existence of ecosystems, human civilizations and life itself. Different regions and societies adapt to their environments and determine their own energy resources and energy uses. The standards of life achieved in countries are often a function of energy related factors. On the other hand, energy can exist in many forms, and can be converted from one form to another with energy conversion technologies. We use energy carriers, produced from energy sources, in all aspects of living (Toklu et al., 2010).

The aim of the present study is to investigate the energy input and output per 1000 bird for the production of broiler in Yazd province, Iran, and to analysis the effects of numbers of chicks at three levels (less 10000 bird, 10000 to 20000 bird and more 20000 bird) and ventilation system type at two levels (tunnel and cross ventilation) were on energy efficiency.

MATERIALS AND METHODS Yazd province with the area of 7215ha (4.37% of total area of country) located in the center of Iran within 29°, 48' to 33°, 30' latitude and 54°, 45' to 56°, 30' longitude. Data were collected from broiler farms in Yazd province by using a face-to-face questionnaire during January–February 2010. To determine the broiler farm numbers, the Neyman method and Stratified random sampling technique were applied (Yamane, 1967):

$$n = \frac{\sum (N_h S_h)}{N^2 D^2 + \sum N_h S_h^2} \qquad (1)$$

where n is the required sample size, N is the number of holdings in target population, N_h is the number of the population in the h stratified, S_h^2 is the variance of the h stratified, d is the precision where ($\bar{x} - \bar{X}$), z is the reliability coefficient (1.96 which represent the 95% reliability) and $D^2 = d^2 / z^2$.

To calculate the sample size, permissible error of 5% within confidence level of 95% was used. Therefore the size of 44 was considered as sampling size and 44 broiler farms where selected randomly.

To calculate the energy inputs and output of each broiler farms, all of inputs and output quantified per unit (e.g. 1000 bird). The energy equivalents of inputs and output (Table 1) were used to determine the amount of energy inputs and output. In this research energy inputs were including chicks, human labor, machinery, diesel fuel, feeds and electricity and energy output was the energy of broiler and manure.

Table 1. Energy equivalents of inputs and outputs in broiler production.

Inputs	**Unit**	**Energy equivalent (MJ (1000bird)$^{-1}$)**	**Reference**
A. Inputs			
Chick	kg	10.33	(Heidari et al., 2011)
Human labor	h	1.96	(Heidari and Omid, 2011)
Machinery			
(a) Electric motor	kg	64.8	(Chauhan et al., 2006)
(b) Steel	kg	62.7	(Chauhan et al., 2006)
(c) Polyethylene	kg	46.3	(Kittle, 1993)
Diesel fuel	l	47.8	(Kitani, 1999)
Feeds			
(a) Maize	kg	7.9	(Atilgan and Hayati 2006)
(b) Soybean meal	kg	12.06	(Atilgan and Hayati, 2006)
(c) Di calcium Phosphate	kg	10	(Alrwis and Francis, 2003)
(d) Fatty acid	kg	9	(Berg, 2002)
Minerals and vitamins	m^3	1.59	(Heidari et al., 2011)
Electricity	kWh	3.6	(Heidari and Omid, 2011)
B. Outputs			
Broiler	kg	10.33	(Heidari et al., 2011)
Manure	kg	0.3	(Kizilaslan, 2009)

Following the calculation of energy inputs and output, energy ratio (energy use efficiency), energy productivity and net energy were determined (Heidari and Omid, 2011):

$$\text{Energy use efficiency} = \frac{\text{Energy Output (MJ (1000 bird)}^{-1})}{\text{Energy Input (MJ (1000 bird)}^{-1})} \quad (2)$$

$$\text{Energy productivity} = \frac{\text{Yield (kg (1000 bird)}^{-1})}{\text{Energy Input (MJ (1000 bird)}^{-1})} \quad (3)$$

$$\text{Net energy} = \text{Energy Output (MJ (1000 bird)}^{-1}) - \text{Energy Input (MJ (1000 bird)}^{-1}) \quad (4)$$

Ventilation System types Proper ventilated housing is essential for profitable poultry production. There are basically five reasons why we must ventilate poultry houses:

1- To remove heat.
2- To remove excess moisture
3- To minimize dust and odors.
4- To limit the build up of harmful gases such as ammonia and carbon dioxide.
5- To provide oxygen for respiration.

Of these five, the two most important are removing built up heat and moisture. The time of the year determines which of these is of primary concern.

There are two types of ventilation system in broiler houses. The type of inlets will depend on the type of ventilation. It is important that the direction of the air jets be in harmony with overall project. Inlets are operated by an automatic control panel, so no operator is required.

Cross ventilation

Fans are installed on one side of the shed and the air inlets on the opposite side. It is a valid system for high stock density, when a large number of fans are necessary. The air inlet is designed to supply two jets, one for winter which goes over the batteries, and the other one for summer, which goes all over the shed. It is a very ingenious system based on the principle of a high-speed air stream near the ceiling and low speed air stream at bird level to recycle bird body heat in winter. In summer air speed is higher, creating a cooling effect.

Tunnel ventilation

This is the ideal system for broilers, layers and chicks. Fans are installed in one end wall and air inlets are installed in the opposite end wall. This system also features low air speed in winter and high air speed in summer. It is a perfect solution for areas with very cold or very warm climate. The system permits control of the minimum air quantity, so it is an ideal solution when it is necessary to heat and to recycle indoor air. The system is equipped with jet diffusers, and fans which draw in outdoor air and recycle indoor air. With a polypropylene perforated duct which runs the length of the barn, the jet diffusers attain a uniform distribution of minimum air volumes. The possibility to control the minimum air volume is very important to retain environmental heat and to reduce feed consumption without damaging bird welfare. This ventilation system is easily adapted to existing barns because installation is quite simple. It may also include a central heating system with warm air generator connected to the jet diffusers, as well as a cooling system with pad cooling or high-pressure nozzles.

We analyzed the effects of numbers of chicks at three levels (less 10000 bird, 10000 to 20000 bird and more 20000 bird) and ventilation system type at two levels (tunnel and cross ventilation) on energy efficiency of broiler farms.

The data analysis was carried out with the help of the Excel 2007 spreadsheet, SPSS 16.0 software.

RESULTS AND DISCUSSION The inputs used in broiler production and their energy equivalents with output energy rates are shown in the Tables 2 and 3.

Table 2 Energy consumptions of inputs and output in broiler production.

Inputs/Outputs	Unit	Quantity per unit (1000bird)	Total energy equivalent MJ $(1000bird)^{-1}$	Percentage
A. Input				
1. Chick	kg	51.50	531.96	0.28
2. Human labor	h	65.27	127.93	0.07
3. Machinery	kg	3.54	196.06	0.10
4. Diesel fuel	L	2314.49	110632.79	59.20
5. Feed	kg	5501.49	59311.40	31.74
6. Electricity	kWh	4468.26	16085.73	8.61
B. Output				
1. Broiler	kg	2601.82	26876.78	97.87
2. Manure	kg	1948.11	584.43	2.13

Table 3 Energy output-input ratio and forms in broiler production.

Items	unit	Broiler
Energy use efficiency	-	0.15
Energy productivity	kg MJ^{-1}	0.01
Specific energy	MJ kg	71.95
Net energy	MJ $(1000bird)^{-1}$	-159424.66

Total energy used in various operations during broiler production was 186885.87 MJ $(1000bird)^{-1}$. The energy use efficiency, energy productivity and net energy of broiler production were shown in Table 3. Energy use efficiency (energy ratio) was calculated as 0.15, showing the inefficiency use of energy in the broiler production. By raising the meat yield and by decreasing energy inputs consumption the energy ratio can be increased.

The result of ANOVA of the effects of number of chicks and ventilation system type on energy efficiency has been indicated in Tables 4 and 5.

Table 4 ANOVA of effect of number of chicks on energy efficiency.

Variables	df	SS	MS	F
Number of chicks	2	0.008	0.004	8.329*
Error	41	0.020	0.0005	
Total	44	1.025		

*Significance at 1% level.

Table 5 ANOVA of ventilation system type on energy efficiency.

Variables	df	SS	MS	F
Ventilation system type	1	0.003	0.003	4.679**
Error	43	0.025	0.001	
Total	44	1.025		

**Significance at 5% level.

The number of chicks had significant effect on energy efficiency ($P<0.01$) and level 3 (more 20000 bird) had the biggest energy ratio. The effect of ventilation system type was significant at 5% level and tunnel ventilation system that used in 61% of units had the bigger energy efficiency.

During the summer, the most important thing that a ventilation system does is to remove heat from the house. Birds give off heat as they grow. During cold weather, or when chicks are little, this heat is beneficial. During the summer, however, bird body heat, combined with the heat from outside air and from solar radiation, causes reduced feed consumption, reduced growth, and increased mortality. Ventilation and cooling systems remove the heat added to a broiler house by bird body heat and by solar radiation. As birds near market weight, they give off relatively large amounts of heat.

During the winter, the most important function of a ventilation system is to remove moisture from the house while conserving heat produced by the birds and brooders. When a house is closed, the ventilation system must be operated on a timer to remove the moisture produced by birds or damp conditions and condensation will result.

CONCLUSION The objective of this study was to estimation energy consumption and analysis the effects of number of chicks and ventilation system type on energy efficiency of broiler farms in Yazd Province, Iran. Based on the results of the investigations, the following conclusions were drawn:
1. The total energy consumption in broiler production was 186885.87 MJ $(1000\text{bird})^{-1}$.
2. The results indicated energy ratio, energy productivity and specific energy were 0.15, 0.014 kg MJ^{-1} and 71.95 MJ kg^{-1}, respectively.
3. The impacts of number of chicks and ventilation system type were significant on energy efficiency.
4. Tunnel ventilation system had the bigger energy efficiency.
5. The new poultry house flooring system eliminates the need to use litter, such as sawdust and wood chips on the floor. This results in a much drier and more concentrated type of manure between broods.

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AMMONIA EMISSION FROM A 15 YEAR OLD CONCRETE SLATTED FLOOR IN A DAIRY BARN

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CSAS11XXX –Topic 1: Concrete Deterioration

ABSTRACT Ammonia emissions caused by animal husbandry highly contributes to eutrophication and acidification of the environment. In order to investigate the ammonia emission from building material in a dairy barn, a used, 15-year-old concrete slatted floor was investigated. For the measurements of the emitted ammonia a new approach of isolating the slatted floor from other sources of ammonia in the barn was used. Emitted ammonia from the soiled floor, as well as wet and dry high pressure cleaned flooring material was quantitatively investigated in a commercially run dairy barn. The values of emitted ammonia from the concrete itself were 6.4 mg NH_3 h^{-1} m^{-2} (wet concrete) and 1.5 mg NH_3 h^{-1} m^{-2} (dry concrete), respectively. The roughness of the tread area and the surface of the slat interspaces were compared between old and new concrete slats. While the tread area of the old slat was smoother than from the new one, the interspace surface of the old slat was rougher than from the new slat.

Keywords: ammonia emission, concrete, slatted floor, dairy housing

INTRODUCTION Ammonia emission is well known to have negative impacts on the environment because of acidification and eutrophication of ecosystems. A major source of ammonia emission is animal husbandry (Groot Koerkamp et al., 1998). Approximately 80 % of the total emission is originated from animal housing as well as storage and application of faeces. Especially naturally ventilated cattle barns do contribute to the total ammonia emission at a large scale. Ammonia is mainly generated from urea in the animal's urine. It gets released in an enzymatic process when urease (generated by microorganisms in the faeces) degrades urea into ammonia and carbon dioxide. Braam and Swierstra (1999) observed that increasing roughness of concrete specimen also increases urease activity, therefore, ammonia emission.

There are many approaches of cleaning slatted floors actively or passively in dairy barns in order to reduce ammonia emission. Hamelin et al. (2010) investigated different designs of concrete slats. They found that introducing a notch into the shape contributes positively to the cleaning effect. Määttä et al. (2008) used varying coatings for slatted floors and found epoxy-coated concrete the most promising material in order to enhance the cleanliness of the slatted floor. Kroodsma et al. (1993) used water in order to flush the slatted floor. He achieved significant effects on reducing the ammonia emission. Braam et al. (1997) found the beneficial use of a manure scraper on flooring in a dairy barn. Retz et al.

(2010) used a special cleaning device combining a manure scraper and water flushing to reduce ammonia emission. However, in order to evaluate these cleaning techniques properly it is useful to know how much ammonia gets inevitably emitted from the concrete material itself after a long use in a barn. It is assumed that despite any common cleaning technique, a certain amount of microorganisms, feces and urine remains in the pores of concrete surfaces emitting a certain amount of ammonia. Especially worn surfaces which underwent mechanical abrasion show a decreased cleanability (Määttä et al., 2009). Pelletier et al. (2005) and Pereira et al. (2011) found values up to 175 mg NH_3 m^{-2} h^{-1} of ammonia emission from soiled concrete used in swine buildings and from a scale model of a slatted floor soiled with cow manure, respectively.

Objective Aim of the study was to determine how much ammonia emission is generated only from the concrete material of a 15-year old slatted floor.

MATERIAL AND METHODS

Experimental Site The experiment was carried out in a naturally ventilated, organic dairy barn located in Labenz, Schleswig-Holstein, Germany. The experimental site of the barn consisted of two compartments with a manure storage pit under the slatted floor and straw beddings for ca 90 Holstein-Friesian.

Slatted floor material To measure the ammonia emissions exclusively from the slatted floor, exchangeable concrete floor elements (55 cm x 62 cm x 20 cm) were cut out of the existing floor. Table 1 gives an overview of the technical data of the slat elements. To hold the cut pieces of the slatted floor at the same level as the remaining floor, a metal support was build and placed underneath each test slat. Thus, the single elements could be isolated from the rest of the barn to avoid influences from other sources of ammonia emissions like the slurry pit, the cubicles or the cows themselves during the measurements.

During this study, slat elements with different treatments were investigated: naturally soiled elements, high-pressure cleaned slat elements when they were still wet as well as high-pressure cleaned slat elements when they were dried. For the measurements of the ammonia emission from soiled slat elements, test slats were taken directly out of the barn after they had been naturally soiled with faeces and urine by the cows. In order to get an average value of the ammonia emission from the soiled slat elements, three slats were measured at six occasions, respectively.

In order to measure the ammonia emission from only the concrete material of the slatted floor, test elements were cleaned with a high-pressure cleaner (Kärcher, Model HDS, Winnenden, Germany) to completely remove faeces and urine from the slat surface. This was considered to be the most thorough cleaning technique commonly used. During three repetitions, three slat elements were used, respectively.

After the cleaning, each slat element was placed in a 400-liter basin filled with water for one minute in order to ensure an even wetting if the surface of the slats. After the wetting, the slats were placed into the measuring chambers for 24 hours. The ammonia emission of the cleaned slats was measured in analogous manner as the soiled slat elements. After the measurement of the wet elements, the slats were dried for 48 hours at room temperature and measured again for 24 hours in order to investigate the ammonia emission from the dry concrete.

Table 1. Technical data of the slat elements.

Type of slatted floor	Miesner, R55/20
Date of installation in the barn	1995
Concrete strength	C 45
Dimensions of original slat	55 x 270 x 20 cm
Dimensions of test element	55 x 60 x 20 cm
Weight of original slat	ca. 344 kg
Weight of test element	ca. 77.4 kg
Total surface of the test element	ca. 1.4 m^2
Surface of the tread area of the test element	ca. 0.3 m^2

Measurement and Analysis of ammonia emissions The present study used the method of measuring the ammonia emission from the slat elements described in Retz et al. (2010). After removing the slat elements from the barn, they were stored in an air-conditioned 20 ft office container in airtight PE-measuring chambers (120 cm x 80 cm x 60 cm) with an acrylic glass lid. During the measurement, each chambers contained one slat element respectively. Gas-washing bottles (500 ml) were installed at the air inlet and at the outlet of the chambers, connected with silicone "Tygon®"-tubes (Saint-Gobain Performance Plastics, Northboro, MA, USA; inner diameter: 8 mm, outer diameter: 16 mm). All bottles were filled with 250 mg of sulphuric acid (0.1n). The two bottles at the air outlet of the measuring chambers were connected in series to absorb the ammonia emitted from the slats. Membrane pumps (Iwaki, Model APN-085LVX1-E4, Tokyo, Japan) sucked the air out of the chambers and through the gas-washing bottles during 24 hours time period. An air flow meter (Ritter, Model TG5, PVC, Bochum, Germany) at each chamber measured the volume of the air sucked through.

After 24 hours (Elzing and Monteny, 1997) the bottles with the sulphuric acid were weighed to get the total amount of the acid that was left after partly evaporation. Thereafter, the acid was filled in 100 ml PE-bottles. The product of the reaction between ammonia and sulphuric acid is ammonium sulphate.

Using a VDI standard method (VDI, 1974), the concentration of the ammonium sulphate was calculated with a segment flow analyzer (Skalar, Model San^{++}, Breda, The Netherlands).

Roughness of the slat elements Surface roughness measurements were performed according to the DIN 4768. A 15-year-old slat element from the barn was compared to a new one which has not been in use before in a barn. The tread areas as well as the surface of the slat interspaces were examined with a manual measuring device (resolution: 0.001 mm) on the slat elements, the used and the new one, respectively. Per surface area, measurements every 0.5 mm were taken trough on a test line of 75 mm and repeated four times at different locations of the test element. The roughness is defined as the arithmetic mean of the absolute of the distances from the surface to a reference line (Braam and Swierstra, 1999, DIN 4768, 1990):

$$R_a = \frac{1}{l} \int_0^l |y(x) - y_{ref}| \, dx$$

where R_a is the surface roughness, l the measuring length, y(x) the distance from the surface to a reference line and y_{ref} the position of the reference line.

A comparison of the means was performed with the software package SAS 9.1 (SAS Institute Inc., Cary, NC, USA).

Figure 1. Cleaned slat element and concrete surface of the tread area of a 15-year-old slatted floor.

Results and Discussion

The results show a mean ammonia emission of 51.4 ± 23.1 mg NH_3 h^{-1} m^{-2} from the untreated soiled slat elements (table 2). In comparison, the emission from the wet high pressure cleaned slat element was 6.4 ± 0.7 mg NH_3 h^{-1} m^{-2}. After being dried, the emission was reduced to 1.5 ± 0.5 mg NH_3 h^{-1} m^{-2}. Hence, a thorough high pressure cleaning allows a reduction of 87.5 % (wet) and 97.1 % (dry) of the ammonia emission.

It seems that during the drying-process, solved ammonia gets released with the evaporating water (Hamelin et al., 2010), leading to a lower emission of ammonia when the concrete is dry. De Foy et al. (2004) found a positive relation between water absorption of materials and the number of micro-organisms responsible for odour-emission.

The values of the soiled slat elements are lower than the data found in the literature. However, measurements with the scale model (Pereira et al., 2011) were performed straight after the application of the manure on the concrete material while in this study emissions have already occurred in the barn before the slat elements were measured inside the measuring chambers.

Table 2. Ammonia emission from the soiled and high-pressure cleaned slat elements calculated per m^2 tread area.

Concrete	Condition	mg NH_3 h^{-1} m^{-2}	
soiled	wet	51.4	± 23.1
High-pressure cleaned	wet	6.4	± 0.7
High-pressure cleaned	dry	1.5	± 0.5

The roughness measurement of the slat surface shows a significant difference between the tread areas of the old and the new slat elements (table 3). The old slat has a statistically significant lower R_a-value than the new one. This clearly shows the effect of use and abrasion of the concrete through the animals in the 15 years of use. However, highest R_a-values of 114 ± 59 were found at the vertical surface of the interspaces of the slat elements. The vertical slat surface of the new, untreated concrete shows a R_a-value of 83 ± 59 which is significantly lower than the value from the slat elements already in use. Corrosion of the concrete through acidic compounds in the manure and urine is likely to be the reason of the higher roughness of the used slats (Bertron et al., 2010). The surface of the tread area represents

19.4 % of the total emitting surface. A higher R_a-value of the interspaces, therefore, affects the roughness and porosity of the total surface of the concrete slats. The high roughness of the slat elements contributes to the clinging of manure on the surface of the slat elements when soiled. Even though the slat elements had been cleaned thoroughly with the high pressure cleaner, small amounts of manure and microorganisms are likely to remain in the pores of the concrete, contributing to the ammonia emission. Pelletier et al. (2002) found high water-sorption values combined with elevated numbers of microorganisms in concrete. Braam and Swierstra (1999) also found increasing urease activity with increasing roughness of concrete elements.

Table 3. Surface roughness R_a of the new and 15-year old slat elements at different locations

State of use	Location of measurement	R_a (µm)	sig.*
old	tread area	49 ± 30	A
old	surface of slat interspace	114 ± 59	C
new	tread area	94 ± 46	BC
new	surface of slat interspace	83 ± 59	B

*different letters indicate a statistical significant difference between the means.

CONCLUSION After 15 years of use, dry concrete slatted floor shows an emission of ammonia of 1.5 mg NH_3 h^{-1} m^{-2} when dried and 6.4 mg NH_3 h^{-1} m^{-2} when wetted. This gives a basic emission of the concrete material that cannot be undergone by common cleaning procedures in a dairy barn. It can also be assumed that worn concrete slats, showing higher roughness at the interspace surfaces than new ones, result in higher emission due to higher porosity.

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Ecological Concrete

Section 5

7th International Symposium on Cement Based Materials for a Sustainable Agriculture (CIGR International Symposium)
Hosted by the Canadian Society for Bioengineering (CSBE/SCGAB)
Québec City, Canada September 18-21st 2011

ASSESSMENT OF THE PERFORMANCE OF COTTONISED FLAX IN NATURAL FIBRE REINFORCED CEMENTITIOUS COMPOSITES

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CSAS11005 – Topic IV: Concrete and Green Building.
ABSTRACT Cottonisation of flax not only makes flax usable for producing textiles, it also further divides the technical fibre into (bundles of) elementary fibres and removes partly the alkali-sensitive pectin and hemicellulose. An experimental investigation was performed to assess the characteristics of cottonised flax in fibre reinforced cementitious composites. The fibre and composite properties are compared with technical flax and PVA fibres. Strength/strain curves of the fibres and composites are compared to evaluate the mechanical performance. Not only tensile strength and stiffness but also first crack strength and peak stress, work of fracture as a measure for multiple cracking and the visual closure of individual cracks by self healing are analysed. Cottonisation of flax enhances modulus of elasticity, peak stress and strength at first crack of concrete, in comparison to technical flax fibres.
Keywords: microstructure, tensile strength, stiffness, peak stress, multiple cracking, durability, cottonised flax, natural fibres, fibre reinforced, cementitious

INTRODUCTION The production of natural ‘green’ fibres has a low environmental impact. The fibres are also renewable and biologically degradable with good mechanical properties. Some plant varieties, such as flax, thrive in several climates and are therefore easily to cultivate.

The polymers which are found in natural fibres are cellulose, hemicellulose, lignin and pectin (Figure 1). The lignin and pectin act as the bonding agents (Baley, 2002). The fibres contain chains of cellulose wrapped together by microfibrils and have a polygonal cross-section. In the middle a central canal is found, the lumen (Sedan et al., 2008). The mechanical properties depend on the crystal structure, the cellulose, the degree of crystallisation and polymerisation, the angle of the microfibrils, the porosity and the size of the lumen. The fibre strength reduces with the moisture content, and the stiffness with increasing uptake of water in the fibre pores since it reduces the cohesion of the fibrils. The lumen is partly responsible for the water uptake (Baley, 2002). The tensile strength and the modulus of elasticity decrease as the fibre diameter increases (Bodros & Baley, 2008).

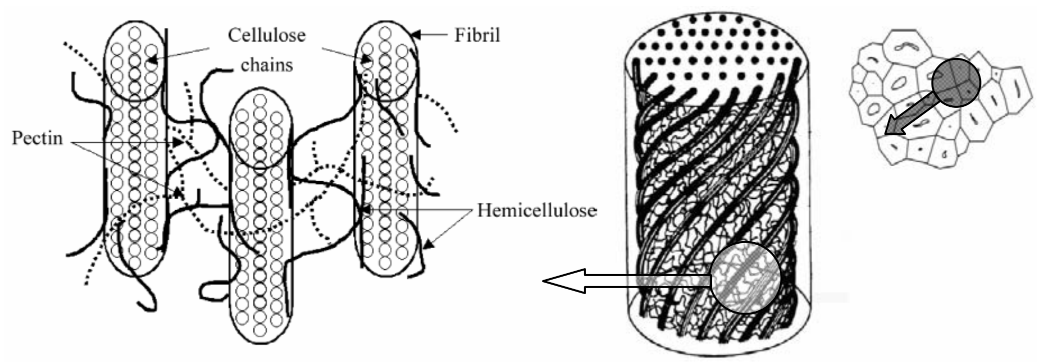
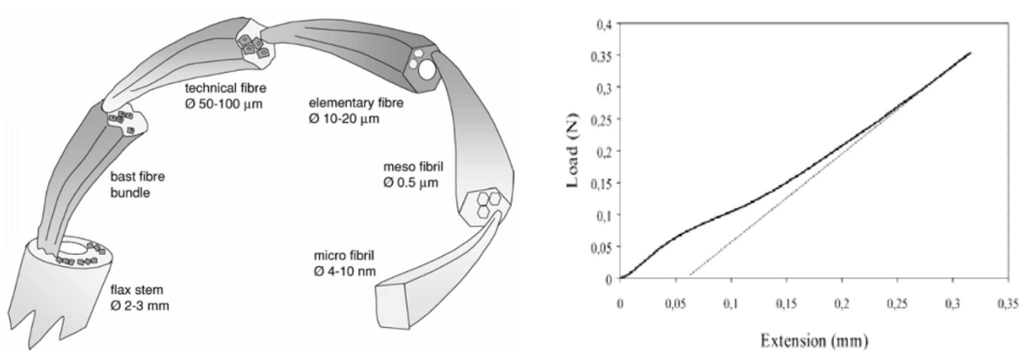


Figure 1. Polygonal cross-section of natural fibres with microfibrils and polymers after Baley (2002) and Sedan et al. (2008).

The presence of impenetrable fibres in concrete ensures a water film on the fibre wall. This is the wall-effect at the interface (Savastano & Agopyan, 1999 and Liao et al., 2004). The water/cement ratio is therefore higher in the regions near the fibres with the respect to the matrix around them. The crystalline products formed in the transition zone therefore consist of larger calcium hydroxide crystals. For natural fibre composites, the transition zone is porous, cracked and rich in $Ca(OH)_2$ macro crystals.

The studied fibres in this research are the flax fibres. Flax is primarily cultivated for its seeds from which oil is obtained. The stems are often considered as waste material. But the fibres in the stem can be used in fibre reinforced concrete with good mechanical properties. The flax fibre is one of the most durable and strong natural fibres. The difference with synthetic fibres is the water uptake of the fibre and its more hydrophilic nature. This ensures a better bond with the cement matrix (Boghossian & Wegner, 2008). The one meter long technical fibre inside the flax stem (Figure 2a) consists of bundles of elementary fibres bound together with pectin and hemicellulose. Due to the bundle effect, the technical fibre is 57 % stronger than the elementary fibre (Bos et al., 2002). The diagram in Figure 2b shows an initial non-linear elastic behaviour. This is attributed to the orientation and sliding of the microfibrils (10-11°) with the axis of strain (Baley, 2002). The mechanical properties of the flax fibres show higher variability due to the natural origin. Some properties are listed in Table 1.



(a) (b)

Figure 2. Composition of a flax stem (a) and a typical force/displacement diagram (b) after Baley (2002), Bodros & Baley (2008) and Bos et al. (2002).

Table 1. Mean mechanical properties of elementary flax (Baley, 2002; Bodros & Baley, 2008; Boghossian & Wegner, 2008; Bos et al., 2002) and PVA-fibre (Li, 2008).

	Flax fibre	***PVA-fibre***
Diameter	**10-60 µm**	**39 µm**
Tensile strength	**840-1800 MPa**	**806-1620 MPa**
Modulus of elasticity	**50-100 GPa**	**21.8-60 GPa**
Maximum strain	**1.8-3.2 %**	**6 %**
Water uptake	**95 %**	
Density	**1.5 g/cm³**	**1.3 g/cm³**

The process of cottonisation lowers the amount of technical fibres and alters the amount of elementary fibres. The pectin and hemicellulose are partly removed. The flax fibres are compared with the synthetic uncoated PVA-fibre (PolyVinylAlcohol). The hydrophobic nature of the PVA-fibre gives a strong chemical bond with the cement matrix and originates from a hydroxyl group in the molecular chain of the PVA-fibre. The mean mechanical properties are also given in Table 1.

Fibre reinforced cementitious composites exhibit multiple cracking. First a crack is formed, next fibre bridging action takes over the load and the crack is stopped. The load increases and the matrix cracks at another location. This goes on until the fibre bridging cannot take the alteration of the load (Li, 2008).

MATERIALS AND METHODS

Materials The studied mortar mixtures were composed of OPC (571 kg/m³), fly ash (685 kg/m³), silica sand 0/2 (456 kg/m³), water (332 kg/m³), a polycarboxylate superplasticizer (Glenium 51, conc. 35 %) and several volume percent of fibres. The PVA-fibres with a length of 6 mm and the flax fibres were obtained from the Belgian companies Redco nv and Debruyne nv, respectively. The technical fibre had been cut down into approximately 5 cm long pieces. The raw material needed further processing. This included cutting into 2-3 cm long fibres and removal of all the clay and other unwanted natural materials. The three types of fibres are shown in Figure 3.

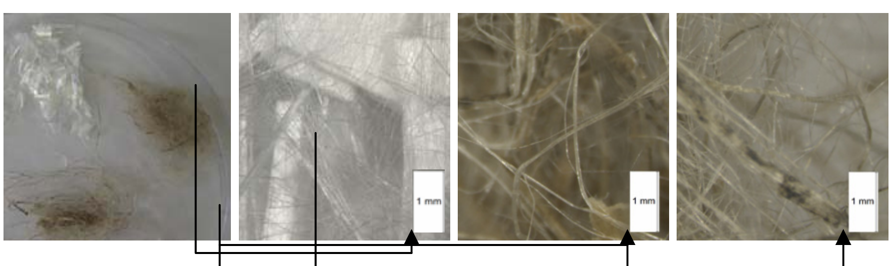


Figure 3. Typical view of PVA-fibres, technical flax fibres and cottonised flax fibres

Every series of mixtures was composed out of three 160·40·15 mm³ samples. These were sliced out of two 160·40·40 mm³ mortar bars. The code *C* next to the name of a mixture refers to a control sample. *PVA2 C* and *PVA2* had the above mentioned composition with 15 kg/m³ superplasticizer and

2 vol.% PVA-fibres (26 kg/m³). *PVA1* contained 1 vol.% PVA-fibre (13 kg/m³) and 10 kg/m³ superplasticizer. *PVA0.5* contained 6.5 kg/m³ and 5 kg/m³ of the respective ingredients. *VL1 C*, *VLc1 C*, *VL1* and *VLc1* had 1 vol.% (15 kg/m³) technical flax fibre (VL) and cottonised flax fibre (VLc) and 15 kg/m³ superplasticizer. *VL0.5* and *VLc0.5* contained half as much fibres and superplasticizer.

Methods

Mixing procedure First, all solid components (cement, fly ash and silica sand) were weighed and mixed dry with a mortar blender (Testing, Bluhm & Feuerherdt). Water was added to the dry mix in 10 seconds and the mixture was further mixed during one minute at 140 rpm. The superplasticizer was then added and the composition was again mixed during one minute. The edges of the mixing bowl were scraped during 30 seconds and the mixing velocity was altered to 285 rpm. The mixture was mixed during five more minutes until it became homogenous. When the mixture was consistent and uniform, the fibres were slowly added during two minutes mixing at 140 rpm, followed by mixing during one more minute at 140 rpm and three minutes at 285 rpm. The mortar was cast in the prismatic moulds and the samples were compacted on a vibration table for two minutes. The samples were demoulded after 24 hours and were stored at a relative humidity of more than 90 % and a temperature of 20±2 °C until the age of 28 days.

Four point bending test At the age of 28 days, the specimens were cracked using a four point bending test (Figure 4), except for the control samples. Fibre reinforced composites excite multiple cracking behaviour, and this can be examined using a four point bending test. A servo hydraulic testing system (Walter+Bai DB 250/15) ensured a displacement-controlled test (0.002 mm/s to imitate a quasi static load). Every specimen was cracked until maximum multiple cracking capability and strain softening. Force-displacement curves were transformed into stress-strain curves by relating the vertical displacement to the strain in the curved lower surface of the prisms during bending.

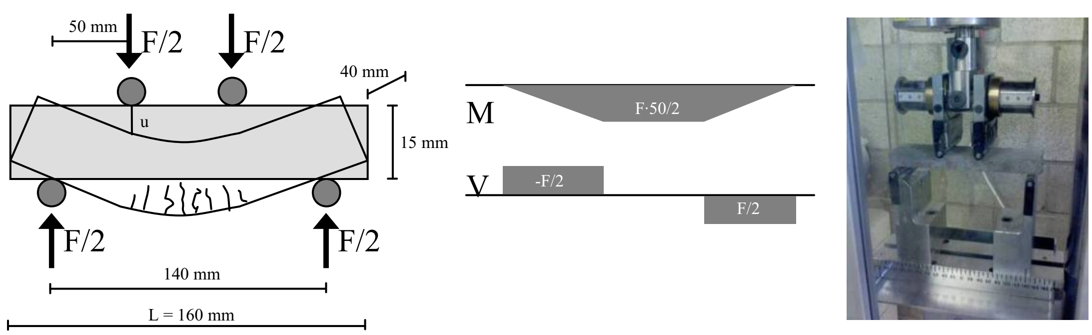


Figure 4. Schematization of the test setup, acting moments and shear forces and testing machine used for the four point bending test.

Curing After cracking, the samples were cured by applying wet/dry cycles. One day they were stored in water at a temperature of 20±2 °C, the next day at a relative humidity of 60 % and a temperature of 20±2 °C. The samples were investigated using an optical microscope (Leica S8 APO with DFC 295 camera) and the crack width was measured. After 28 days of curing, the specimens were loaded until failure with the four point bending test to investigate the self-healing capability. Prior to the final loading, the crack width was again measured to visually judge the amount of crack closure. Control specimens were not cracked at an age of 28 days but underwent the same curing conditions as other specimens. At an age of 56 days, control specimens were cracked and immediately reloaded until final failure.

Parameters investigated The parameters that were investigated in the stress/strain curves were the total working force W (surface under the stress/displacement curve) as a measurement of multiple cracking, the peak stress σ_{cu}, the strain at first cracking ε_{fc}, the first cracking stress σ_{fc} and the modulus of elasticity E (slope of the stress/strain curve). The measurement for the self-healing capacity was the regain of E, σ_{cu}, multiple cracking and σ_{fc}. At reloading, unhealed specimens tend to follow the unloading curve. Therefore the regain property is expressed as the quotient of the difference of the reloading minus the unloading property over the difference between the preloading minus the unloading property. The regained multiple cracking is expressed as the reloading over the preloading total working force.

All parameters were compared using the statistical program SPSS® with 5 % significance level. Multiple averages were compared with an ANOVA. The homogeneity of the variances was controlled with a Levene test. The post hoc test for data with homogeneous variances was a Student-Newman-Keuls test and if not homogeneous a Dunnett's T3.

RESULTS AND DISCUSSION

Workability and hardening properties The determining factor of the workability was the step of fibre addition during processing. The workability increased by lowering the fibre amount. The workability of mixtures with flax fibres was lower than for the mixtures with the synthetic PVA-fibres. That is due to the higher fluid uptake by the flax fibre. Due to the smaller diameters of cottonised flax, the workability of mixtures with *VLc* was lower than the mixtures with *VL*. Only a maximum of 1 vol.% flax fibres was manageable. Mixtures with PVA-fibres had hardened after a day. The hardening with cottonised flax was comparable. With technical flax however, the hardening lasted 2 days.

Crack initiators for multiple cracking To enhance the formation of multiple cracks, micro defects can be added. Bast fibre leftovers function as crack initiators, as can be seen in Figure 5a. These bast fibres do not bind with the cement matrix and are pulled out completely after loading until failure (Figure 5b). The pulled out length was sometimes 1.5 cm.

(a) (b)

Figure 5. Bast fibre leftovers as crack initiators (a) and totally pulled out bast fibre (b).

Stress/strain curves *PVA2 C* and *PVA2* exhibited multiple cracking with formation of 4 to 6 cracks with an average crack width of 6-35 µm and a pronounced strain hardening effect. Figure 6 shows the stress/strain curves of a mixture with 1 vol.% PVA-fibre. A small hardening effect is seen in *PVA1 (3)*. A crack forms and bridging action of the fibres takes over. This continues and the stress alters until either the bridging or the cement matrix cannot take the load. A second crack was formed and the process started all over again. Now the bridging capacity of the first crack was insufficient to take up the load. A mixture with cottonised flax (Figure 7) does not show any form of strain hardening. One crack is formed and the crack opens. Figure 8 shows the stress/strain curves of a *VL1* mixture. A small hardening effect is seen and reflected in the formation of two cracks.

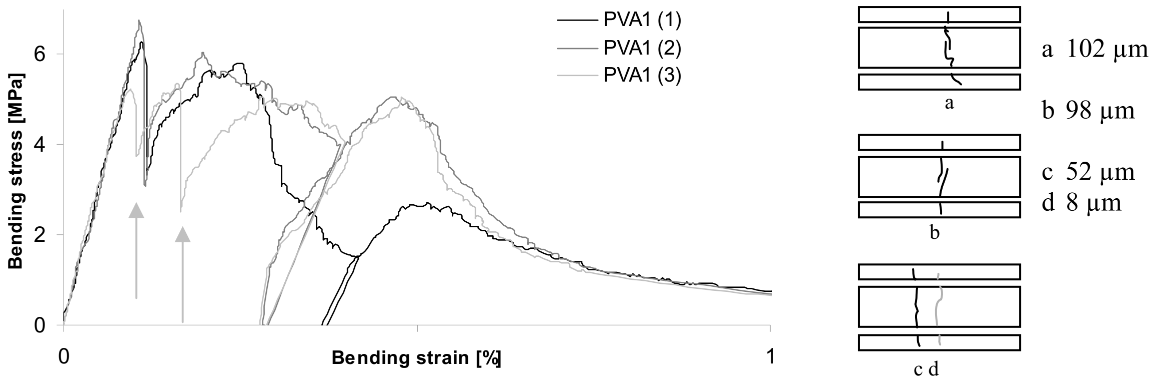


Figure 6. Stress/strain curve of a 1 vol.% PVA-fibre mixture (arrows indicate formation of the first and second crack in *PVA1 (3)*).

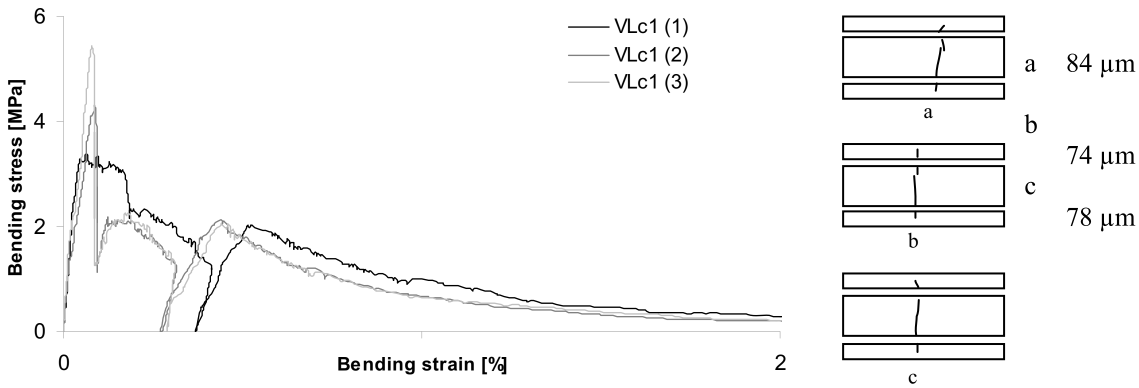


Figure 7. Stress/strain curve of a mixture with 1 vol.% of cottonised flax fibre.

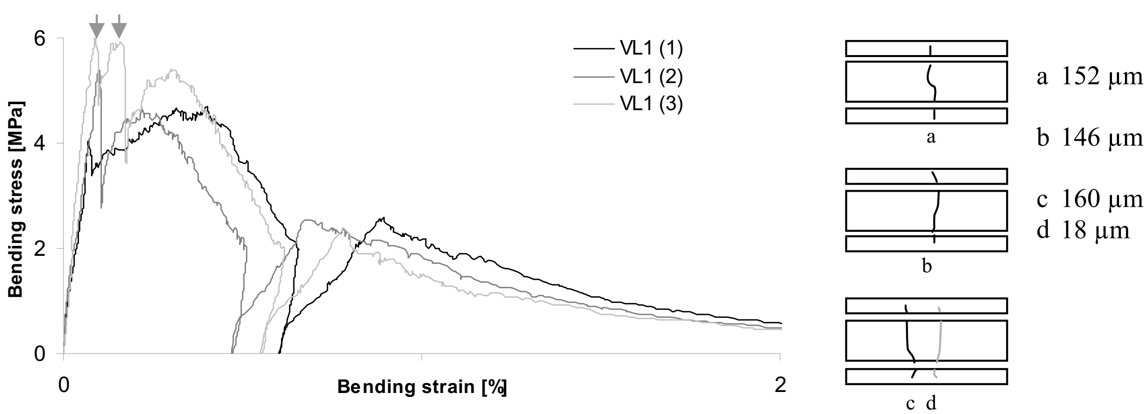


Figure 8. Stress/strain curve of a mixture with 1 vol.% of technical flax fibre (arrows indicate the formation of two cracks in *VL1 (3)*).

Comparison of the mechanical properties

Stiffness *PVA2 C* has an E-modulus larger than other PVA-mixtures (Figure 9). Control specimens were not precracked, so this points at further hydration in the wet/dry cycles. There is a significant difference between *VLc1* and *PVA1*, but none between *VL1* and *PVA1*. On a significance level of 8.4 %

there is a difference between the two mixtures with flax. On this level can be concluded that mixtures with cottonised flax show a higher modulus of elasticity than those with technical flax fibres.
The regain in stiffness shows that *PVA1* and *PVA0.5* exhibit more regain than *PVA2*. This is because the stress induced at precracking of the *PVA2* samples was larger because of the multiple cracking capability. The final crack width in *PVA2* samples is higher due to a higher bridging capacity of more fibres. Mixtures with a similar amount of flax fibres show a lower regain in stiffness but do not differ from each other.

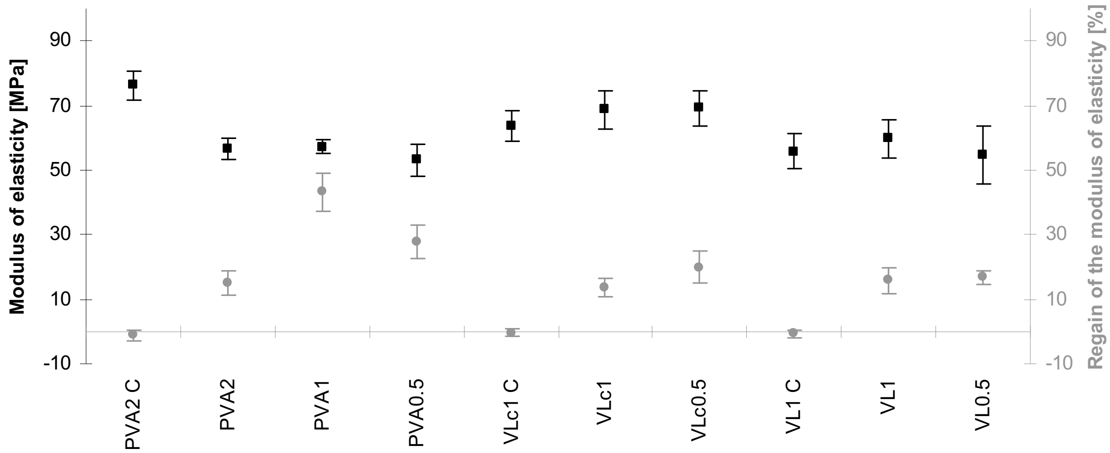


Figure 9. Mean modulus of elasticity of the mixtures at 28 days (56 days for the controls) and mean regain of the modulus with the standard deviation.

Peak stress Figure 10 shows the mean peak stress σ_{cu} and the strength regain. There is no significant difference of peak stress at different vol.% of PVA-fibres. The peak stress of control specimens is higher than for the test specimens. This is due to further hydration, of both the Portland cement and the puzzolanic fly ash. Samples with flax fibres show a lower peak stress than with PVA-fibres. *VLc0.5* differs at a significance level of 9.1 % from *VL0.5* and *VLc1* at 24.6 % from *VL1*. *VLc1 C* however differs significantly from *VL1 C*. Mixtures with cottonised flax show therefore a tendency of having a higher peak load than with technical flax fibres.

The regain of the peak load of control specimens is slightly negative. There is no significant difference between the fibres. All mixtures show similar recovery with regard to the peak load.

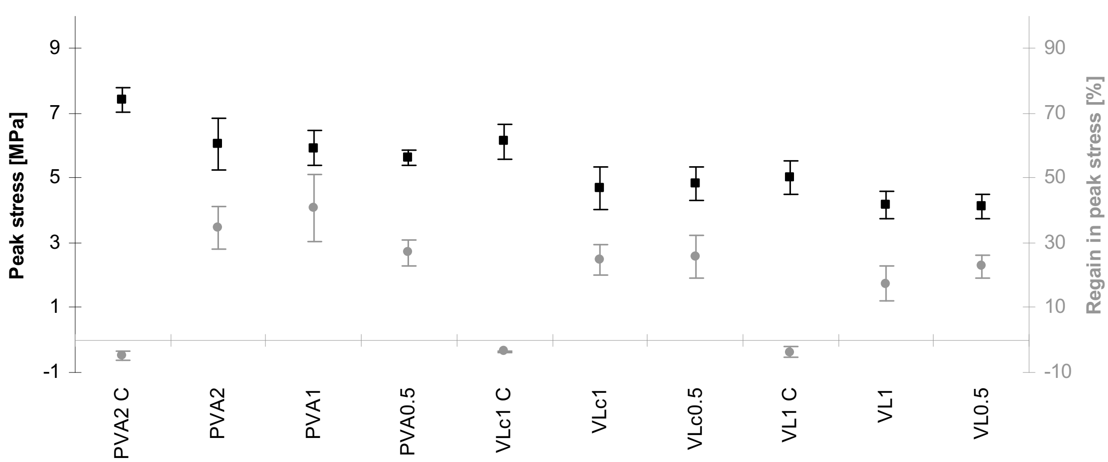


Figure 10. Mean peak stress of the mixtures and mean regain of the peak stress with the standard deviation.

Multiple cracking Lowering the volume percent of fibres gives a lower amount of total working force (Figure 11). There is not enough bridging capacity to induce multiple cracking at low amounts of fibres. Mixtures with 1 vol.% technical flax fibre have a better multiple cracking capability than those with 1 vol.% cottonised flax fibres and PVA-fibres. The difference between the flax fibres is due to the higher amount of bast fibres and technical fibres which have lower bonding capacity with the cement matrix in the mixture *VL1*.
The control mixtures give a regain of 12 % due to the smoothing of the stress/strain curves at reloading. Also *VLc0.5* shows no regain in working force because it had almost none in the beginning. The difference between the technical and the cottonised flax fibre is due to the fact that the cottonised form did not exhibit great multiple cracking behaviour.

First cracking strength Control mixtures in Figure 12 always show a higher first cracking strength because of the further hydration. The only significant difference between the mixtures is found between *VLc1 C* and *VL1 C*. The control mixture with cottonised flax fibre shows a higher first cracking strength than the one with the technical flax fibre. The regain of the first cracking strength does not differ significantly. There is a maximum regain of the stress at crack initiation of 40 % for PVA2. Mixes PVA1, VLc0.5 and VLc1 all show a similar regain of around 30 %. The regain for VL0.5 is somewhat lower, but not significantly different. The difference between VLc1 and VL1 is significant.

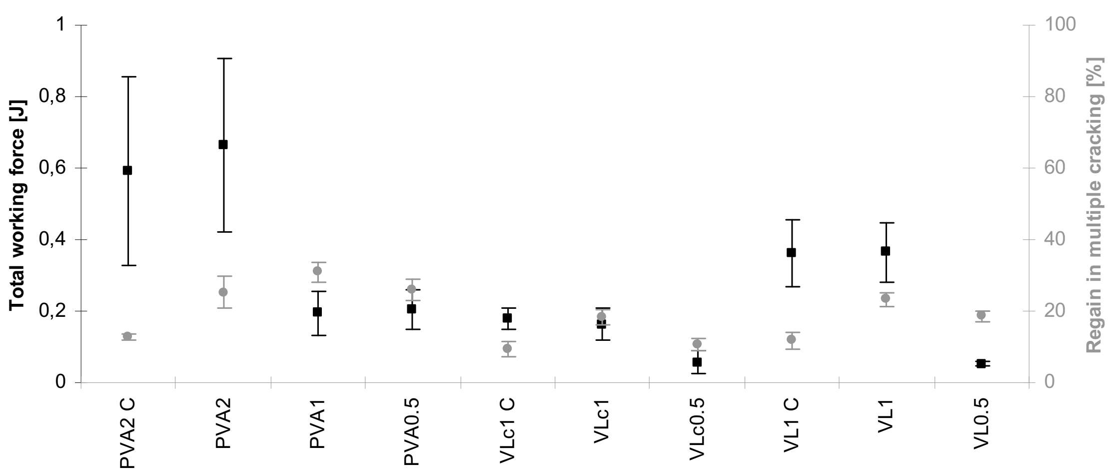


Figure 11. Mean amount of the total working force at preloading of the mixtures and mean regain of the total working force with the standard deviation.

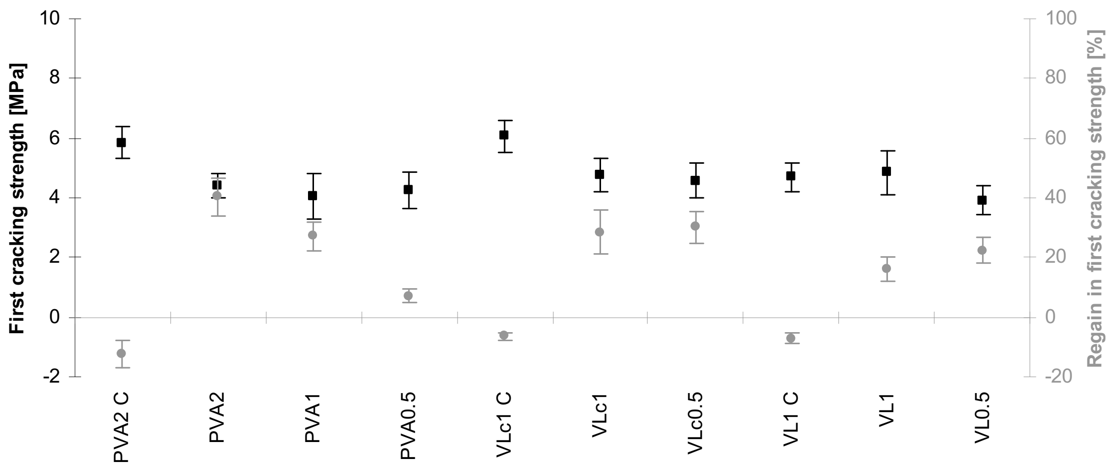


Figure 12. Mean first cracking strength of the mixtures and mean regain of the first cracking strength with the standard deviation.

Visual closure of the cracks Microscopic investigation of the cracks shows a great likeliness between mixtures with PVA and flax fibres (Figure 13). The crack closure is independent of the fibre type, and is completely determined by the initial crack width.

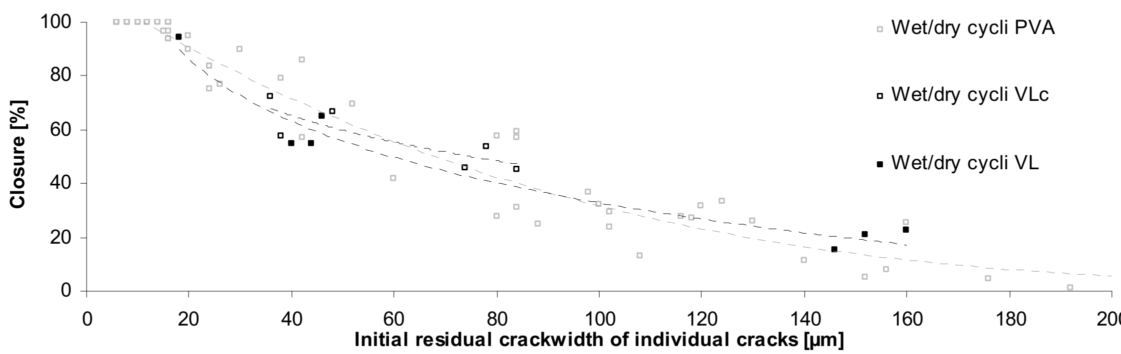


Figure 13. Visual measurement of the crack healing for individual cracks.

CONCLUSION

Workability. Mixtures with flax fibres show a lower workability than those with synthetic PVA fibres. The mixtures with cottonised flax were the least workable and a maximum of one volume percent is manageable.

Stiffness. At a significance level of 8.4 %, it can be concluded that mixtures with cottonised flax show a higher modulus of elasticity than those with technical flax fibres and a comparable stiffness with the samples containing PVA-fibres. Mixtures with flax fibres show a lower regain in stiffness than the mixtures with PVA-fibres but do not differ from each other.

Peak stress. Samples with flax fibres show a lower peak stress than PVA-fibres. Mixtures with cottonised flax show a tendency of a higher peak load than those with technical flax fibres. There is no significant difference between the fibres, with regard to strength regain efficiency.

Multiple cracking. Mixtures with the technical flax fibre have a better multiple cracking capability than the mixtures with cottonised flax fibres and PVA-fibres. Bast fibre leftovers also ease the formation of cracks. Double cracking was observed for one sample containing 1 vol.% technical flax fibre.

First cracking strength. The control mixture with cottonised flax fibre shows a higher first cracking strength than the one with the technical flax fibre. The regain of the first cracking strength does not differ significantly.

Visual closure. The visual closure of the cracks is independent of the sort of fibre used in this investigation, but depends mainly on the initial crack width.

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PROPERTIES OF UNFIRED BRICKS INCORPORATING WASTE OYSTER-SHELL LIMES AND BASALT FIBERS

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CSAS11029 Topic IV: Concrete and Green Building (ex. Label and Environmental Declaration)

ABSTRACT: Large amounts of oyster-shells are being abandoned and accumulated along the coastal areas all over the world, leading to an environmental problem and a serious health hazard. However, there is a possibility that the combination use of fly ash and oyster-shells lime (OSL) may produce a kind of low cost brick, which is free of baking. Presently, the mechanical behaviours of this kind of brick are investigated. The strength development up to 360 days were studied for the brick with an addition of 0~20 wt.% OSL together with 3 wt.% basalt fibre. Results show that the optimum addition of OSL is about 10~15 wt.%. In this case, 28-days' compressive and flexural strengths of the brick are about 30~35MPa and 4~5MPa, respectively. XRD test shows that Ca constituent in OSL is higher than lime, partially explaining the higher gains in strengths of the brick with higher OSL addition. Since drying and rewetting of bricks usually lead to degrade, strength variation, deformation (shrinkage/expansion) and water absorption of the brick were also studied after ten dry-wet cycles. It is shown that both the compressive and flexural strength may slightly increase, as compared with those bricks in a room condition. The reason is that the higher temperatures during dry process (80ºC) may lead to higher rates of reaction (hydration) when moisture is enough in the bricks, resulting in a strength increase.

Keywords: Brick free of baking, Oyster-shells lime; Basalt fibre; Dry-wet test, Shrinkage deformation, XRD

INTRODUCTION

Due to the present global concerns for sustainable development that has arisen from extensive environmental problem coupled with the rapid pace of technological development within the building sector. There is a growing interest in unfired building materials (UBM) with good physical material behavior, with respect to an energy conscious and ecological design, which fulfils serviceability requirements for all strength and durability [1]. The unfired building materials technology relies on the use of an activated industrial by-product or renewable natural resources. There are a number of patents on the use of fly ash–lime mixtures for making unfired bricks were proposed [2–4].

However, there was a little use of waste oyster-shells (OS) produced from fishery, which accumulated from the coastal areas all over the world. The accumulated OS becomes a source of environmental damage and pollution by leakage of water as well as difficulty of landfill security and maintenance and control of landfill [5-6]. In China, the annual OS production is more than 3.0×10^5 tons because of its vigorous aquiculture industry. However, only a very few by-products are currently utilized, and the rest is left as solid waste in landfills (Fig.1), causing a serious environmental problems. Since OS contains 95wt.% CaO, calcined OS lime (OSL) may be used as the replacement of lime in producing

UFB [7]. Other place has demonstrated that it is feasible of utilizing fly ash and OSL to produce a low cost unfired bricks (UBL) in open air.

The present investigation is directed towards the strength development to 360 days for the bricks in which lime was subsittuted by up to 20 wt.% OSL. Experimental results showed that the strength development of UBL was affected by the OSL content, and the optimum OSL addition was determined as 10-15 wt.%. The UBL are usually used in a dry-wet environment for a long term, therefore, this paper would study the properties of the strengths, the (shrinkage/expansion) deformation and water absorption. XRD was used to study the changes in crystallinity and appearance or disappearance of phases for UBL after 28 days water curing.

Drying and rewetting is an important factor leading to the brick degradation. UBLs cured under lab condition were compared with that subjected to a series of ten DW cycles, both the compressive and flexural strength were investigated. At the same time, deformation (shrinkage/expansion) and water absorption of bricks subjected to ten DW cycles were also investigated.

1. Experimental program

1.1. Materials and mix proportion

The chemical analysis and physical properties of cement, fly ash, slag, lime and OSL are presented in Tab.1. The fineness modulus of river sand was 1.65. The superplasticizer (SP) was liquor of the phenolic aldehyde. Its density was 1.1 g/cm^3 and has 31% solid content. Fibers introduced into the matrix to minimize dry shrinkage or crack were BF with average lengths of 10~20 mm (Tab.2). The mix proportions of the UBL are given in Tab.3.

1.2. Specimen preparation and test methods

The weighed quantity of fly ash passing through 4.75 mm sieve was thoroughly mixed in a dry state. In order to keep the OSL and lime from carbonizing, the weighed quantity OSL and lime were kept in 1% sodium hydroxide solution for 3–4 hours, the slurry was sieved through 1.18 mm sieve. First, fly ash, cement, slag, sand, fibers and SP were mixed uniformly by using a mortar mixer. Then, the hydrated lime and the OSL slurry, and water were added to the mixture and mixed thoroughly by kneading until the mass attained uniform consistency. The content of lime and OSL shown in Table 2 was in terms of dry weights of the ingredients. UBLs were made using the size 40 mm×40 mm×160 mm for strength test, and they were compacted by vibration. The specimens were cured by covering with a wet cloth, till sufficient strength gained (usually 3 days), and than the specimens were demolded and transferred to curing tanks filled with a 0.1% $Ca(OH)_2$ solution. The UBL were taken out from the solution 3 days prior to strength testing. For each mixture, six specimens were used for both flexural strength and compressive strength test. The load was applied continuously at a steady rate of 2.5 kN/s.

Details of dry–water cycles are described as follows (Fig.2): samples with a size of 40×40 ×160 mm were submitted to 10 dry–wet cycles. Two days, one cycle was carried out: 8 hours immersed in water, 40 hours dried at 80°C in an air dry oven. The weight and length values (Fig.2) of samples were tested, and for each mixture, three specimens were used for dry–wet cycle resistance test. After 10 cycles, both the compressive strength and the flexural strength were analyzed and compared to those cured in air. The water absorption rate (ΔW_i) is calculated by:

$$\Delta W_i = (W_{wi} - W_{di}) / W_{di} \times 100\%$$

Where W_{wi} is the weight after 8 hours immersed in water, W_{di} is the weight after 40 hours dried at 80°C in an air dry oven.

The drying shrinkage rate (ΔL_i) is obtained from the following equation:

$$\Delta L_i = (L_{di} - L_{wi}) / L_{di} \times 100\%$$

Where L_{wi} is the length after 8 hours immersed in water, L_{di} is the length after 40 hours dried at 80°C in an air dry oven.

XRD patterns of the UBM were recorded using a D/max 2550V X-ray diffractometer (CuKa radiation, 40 kV, 450 mA) in a scanning range of 5~70° in 2θ scale. The testing rate that was applied was 0.02°/s for all specimens. Identification of the hydration products was carried out by using a Diffrac-At Database.

2. Result and discussion

2.1 Strength development

The replacement amount (lime was substituted by OSL) for bricks UBLa, UBLb, UBLc, UBLd and UBLe ranged from 0 to 20 wt%. The development of the flexural strength are shown in Fig.3. The results indicated that as the replacement amount increased there was a significant change in the flexural strength. The 28-day flexural strength for the brick with 10 wt% OSL replacement (UBLc) was 4.1 MPa, while that for brick without OSL replacement (UBLa) was only 2.6 MPa. The brick with 15 wt% OSL replacement (UBLd) had a high flexural strength (4.0 Mpa), while that for brick with 20 wt% OSL replacement (UBLa) was only 2.8 MPa. Fig.3 shows that there was significant gain in strength over the first 180 days. However, at later ages, the strength of all bricks gain slowly with time. At about 360 days, the gain in strength above the 28-day value was 18% and 16% for UBLc and UBLd, respectively. However, the gain in strength for the other bricks exceeded 20%.The highest gain in strength of 38% was that for UBLa, followed closely by the UBLe (31%).

The compressive strength test results for the various unfired brick mixes are shown in Figs.4. The results generally showed that there was a continuous increase in strength with time for UBLs. The 28-days average compressive strength was highest for the UBLd (34.2 MPa), followed closely by the UBLc (32.6 MPa). The brick incorporating 20wt.% OSL, which had the lowest average compressive strength value of 26.8 MPa at 28 days, are still possible used, for environmental conservation has come into focus. Bricks made with 15 wt.% OSL had an average compressive strength of 43.5 MPa at 360 days showing an increase in strength of 21% when compared to the 28-day strength. The compressive strength of UBLc was 42.8 MPa at 360 days showing an increase of 24%, while the average strength for the UBLa was 39.9 Mpa (an increase of 46%).

2.2 Dry-wet cycles

The weight water absorption of samples with 40×40 ×160 mm was tested after each dry-wet cycle, and the results were shown in Fig.5. The results show that the biggest water absorption for all these samples less then 8 wt.% is in relatively acceptable limit compared to the widely used construction bricks had an approximate water absorption value more than 15 wt.% . The water absorption decrease with the dry-wet cycles increase, and UBLe had the highest water absorption about 7.5 wt.% at first cycle, whereas UBLa had the lowest absorption, and, followed by UBLd, UBLc and UBLb. After 10 dry-wet cycles, UBLa still had the lowest water absorption, followed by UBLd, UBLc and UBLb. The UBLe had the highest water absorption about 4.5%.

The length deformation also tested after each dry-wet cycle, and the results were shown in Fig.6. It demonstrates that the length was usually shrinkage even though these samples were saturated absorption. The length shrinkage value increase along with the increase cycle times, and the shrinkage value gently changed after 7~8 cycles. For all the tested samples, the UBLc and UBLd have a bigger shrinkage value, the UBLe has the biggest shrinkage value, and UBLa has the smallest shrinkage value at first few cycles. However, the total shrinkage values after 10 cycles for UBLc and UBLd were approximately the same with UBLa.

The compressive strength and the flexural strength after 10 dry-wet cycles were showed in Table.4. Interestingly, the strengths of the specimens submitted to 10 drying-watering cycles were higher than those cured in open air. For UBLa, the compressive strength of 39.3MPa after 10 dry-wet cycles was 22% higher that cured in open air. For UBLc and UBLd, the compressive strengths also increased 16% after dry-wet cycles. UBLb had the highest strengths improved with 28%. The influence of dry-wet on flexural strength is lower than on compressive strength, and the increase value is among 6~17%. The reason behind this behaviour is that the higher temperatures affect the bricks in two ways: 1) higher temperatures can lead to micro-cracking at the sand-cementitious paste interface due to the different coefficients of thermal expansion, resulting in strength degrade; 2) higher temperatures can also lead to higher rates of reaction (hydration) when there is enough moisture in the bricks, hence, higher strengths. It is worth noting that higher temperatures also increase the carbonation, which can lead to an increase in the strength of bricks.

2.3 X-Ray diffraction analysis

Fig. 7a~b shows a typical XRD pattern of lime and OSL, and the results show that there are only $CaCO_3$ and $Ca(OH)_2$ crystallinity peaks for OSL. However, for lime, besides of $CaCO_3$ and $Ca(OH)_2$, there are unknown crystallinity peaks. This may partially explain why there were higher gains in strengths for bricks with high OSL contains. The higher OSL affect the bricks in two ways: 1) act as alkaline sharpened agent based on the chemical kinetics theory to increase the rate of reaction (hydration) and increase the strength; 2) increase the carbonation can also lead to an increase in the strengths of bricks.

CONCLUSION

OSL represents a highly polluting effluent in most ostrea-culture producing countries. The valorization and the recycling of this waste could be a partial solution to the negative environmental impact. Its incorporation into the brick-making industry can contribute to this solution. The main purpose of this study was to assess both the strength development and environmental applicability when incorporating OSL in unfired fly ash bricks. For this, both compressive and flexural strength up to 360 days, shrinkage/expansion deformation and the water absorption as well as the strength variation characteristics after suffered 10 dry-wet cycles were considered in these laboratory experiments.

In this study, the following conclusions were obtained:

1) The optimum OSL addition is determined as 10~15 wt.%. The 28-day compressive strength and flexural strength of the new brick are higher than 30 MPa and 4 MPa, respectively.
2) The water absorption of UBLs ranges from 6 to 8 wt.% at first dry-wet cycle, and the absorption decreased along with increasing the cycle times, after 10 cycles, the mass water absorption ranges from 4~6 wt.%.

3) These tested bricks were usually shrinkage even if saturated absorption, and the shrinkage value also decreased along with increasing the cycle times. This phenomenon perhaps can be used to reduce shrinkage crack in construction engineering.
4) Both the compressive strength and the flexural strength increased with the increasing the dry-wet cycles, this may be the fly ash action rates were activated by the higher temperature (80°C) during dry process.
5) XRD pattern shows that apart form $CaCO_3$ and $Ca(OH)_2$ crystallinity peaks there are unknown crystallinity peaks in lime. While there are only $CaCO_3$ and $Ca(OH)_2$ crystallinity peaks in OSL. The higher Ca contain for OSL is the main reason that there were higher gains in strengths for bricks with high OSL replacement.

The result shows that the use of OSL for producing new low cost unfired fly ash brick in open air is feasible and effective. Furthermore, it can bring better social and economic benefit for the production of bricks with the addition of OSL other than lime.

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Appendix A

Table 1 Chemical and physical properties of cementitious materials

Chemical analysis (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Specific surface, Blaine (m^2/kg)	28 day compressive strength (MPa)
Cement (PC)	19.5	4.4	6.22	65.9	1.5	462	45.9
Fly ash (FA)	32.5	19.1	8.2	34.7	1.98	565	-
Slag	37.35	11.59	0.67	40.98	8.04	483	-
OSL	4.6	1.1	0.1	92.8	0.5	15000	-
Lime	-	-		63.56	3.38	13000	

Table 2 Physical and mechanic properties of BF

Bulk density (g/cm^3)	Tensile strength (MPa)	Elastic modulus (Gpa)	Softening point(℃)	Elongation percentage (%)	Single dia.(μm)	Conductivity factor (W / m·K)	Operating temperature range (℃)
2.65	4100～45000	225	960	3.1％	7～17,	0.031～0.038	-260～500

Table 3 Mix proportions of the UBL

	FA	PC	Lime	Slag	OSL	GY	SBR/C* (%)	BF/C* (%)	Sand/C *	W/C *	SP/C* (%)
UBLa	45	15	20	15	0	5	3	0.1	0.5	0.3	0.5
UBLb	45	15	15	15	5	5	3	0.1	0.5	0.3	0.5
UBLc	45	15	10	15	10	5	3	0.1	0.5	0.3	0.5
UBLd	45	15	5	15	15	5	3	0.1	0.5	0.3	0.5
UBLe	45	15	0	15	20	5	3	0.1	0.5	0.3	0.5

* C means cementitous materials which include fly ash(FA), cement(PC), lime, slag, gypsum(GY), OSL; SP means water-reducing agent

Table 4 Strengths of UBLs submitted to 10 dry-wet cycles

组号	R_c (MPa)		*△R_c	R_f (MPa)		*△R_f
	R_{cn}	R_{cd}		R_{fn}	R_{fd}	
UBLa	32.2	39.3	1.22	3.2	3.7	1.17
UBLb	32.5	41.6	1.28	3.9	4.3	1.11
UBLc	36.6	42.5	1.16	4.6	4.8	1.05
UBLd	37.3	43.3	1.16	4.3	4.6	1.08
UBLe	31.7	39.3	1.24	3.1	3.4	1.10

*△R_c means the rate of compressive strengths submitted to 10 dry-wet cycles (R_{cd}) to that cured in open air (R_{cn}).

*△R_f means the rate of flexural strengths submitted to 10 dry-wet cycles (R_{cd}) to that cured in open air (R_{cn}).

Fig.1 Schematic of OS landfills

Fig.2 Schematic diagram for drying shrinkage properties testing for UBLs

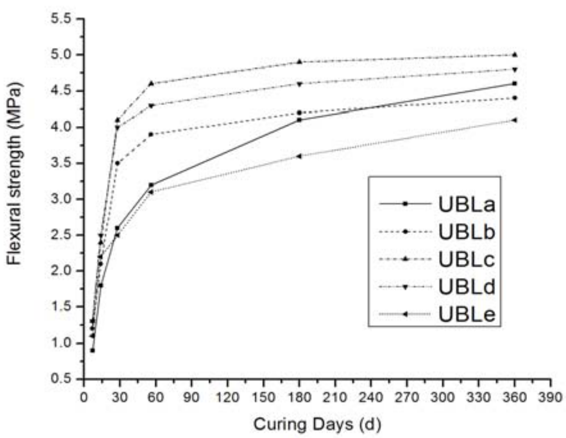

Fig.3 Development of flexural strengths of UBLs up to 360 days

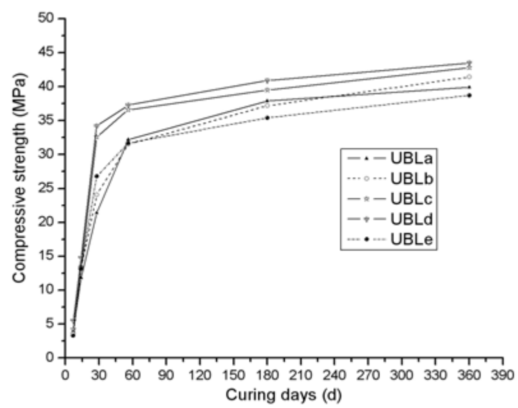

Fig.4 Development of compressive strengths of UBLs up to 360 days

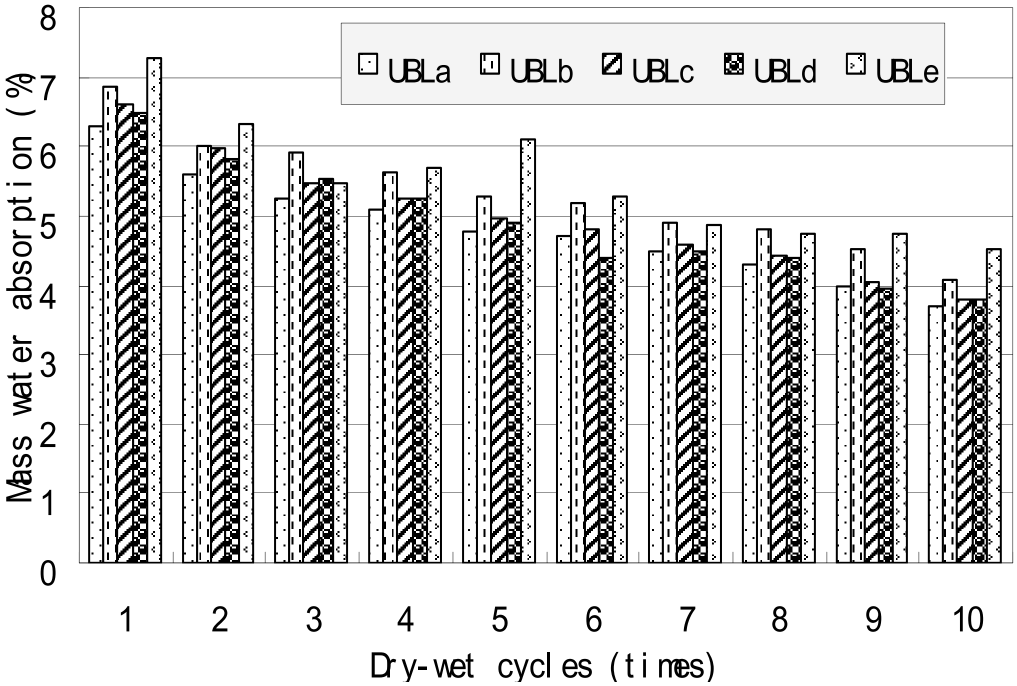

Fig.5 Mass water absorption of UBLs under dry-wet cycles

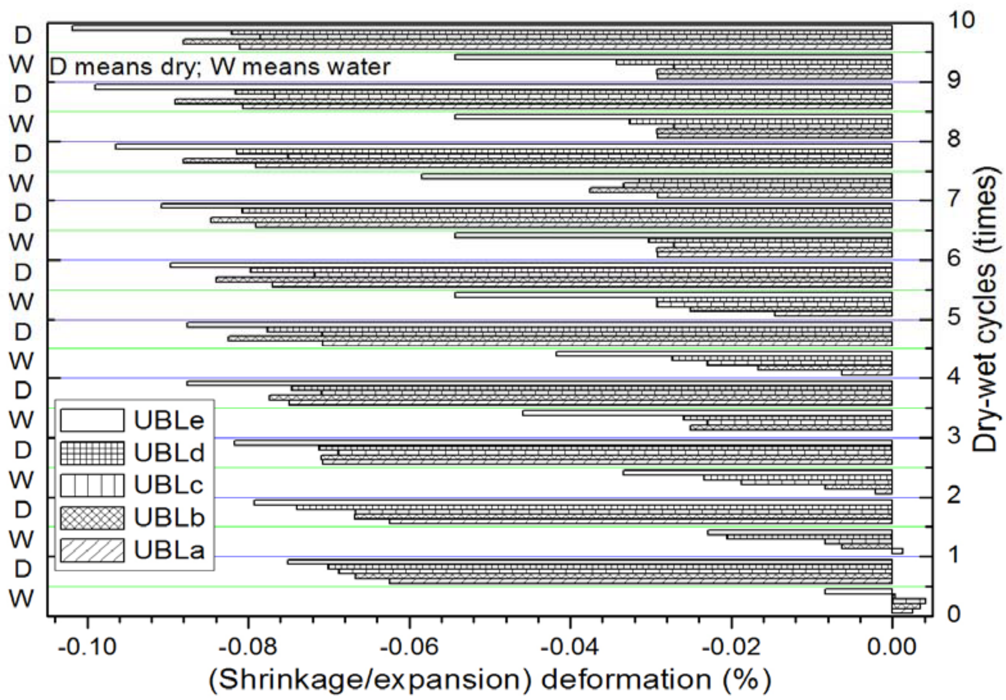

Fig.6 Shrinkage/expansion deformation of UBLs under dry-wet cycles

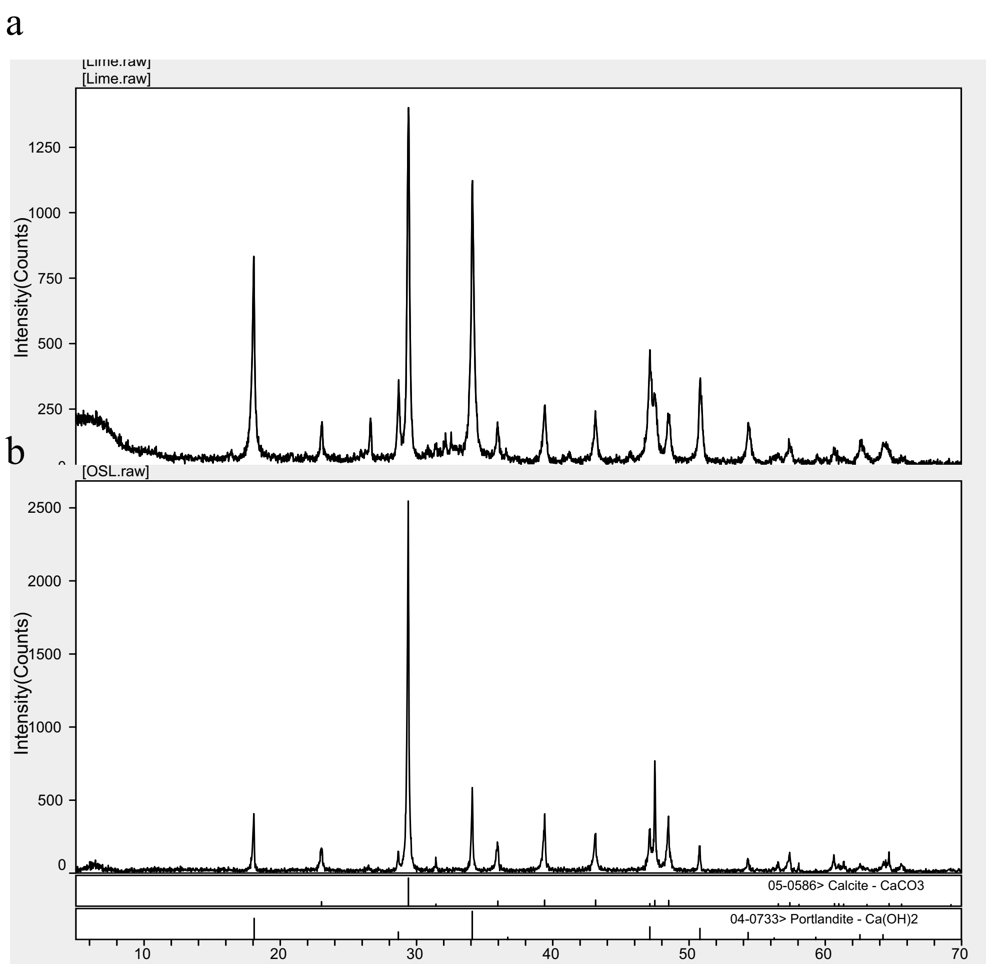

Fig.7 (a) and (b) XRD patterns of Lime and OSL

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BEHAVIOR OF RECYCLED CONCRETES IN AGRICULTURAL ENVIRONMENTAL

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CSAS11- 7 – Topic VII

ABSTRACT: This research work intends to analyse and predict the capacity of recycled concretes which incorporate ceramic waste in substitution of conventional coarse aggregate to resist the action of the environment, chemical, physical or biological attacks or any other process. Results obtained in the mechanical tests (compressive and splitting tensile strength tests) are higher when using recycled materials. An increase in strength resistance between 5 and 25% has been observed as the percentage of substitution grows.Regarding the study of the durability it is shown that incorporating the recycled ceramic aggregate does not affect durability in a negative way, but, on the contrary, these concretes behave in a similar manner than standard concretes. As conclusions, it is worth to use this type of ceramic waste when manufacturing structural concrete in agricultural environment. These recycled concretes can be considered as durable, as they maintain their original shape and strength capacity all through their life period.
Keywords: ceramic, recycled, concrete, durability, strength

INTRODUCTION

Cement matrices (paste, mortar and concrete) are widely used in agricultural and cattle-rearing environments (Massana Guitart, 2010) and in the agro-food industry. Their use extends from the structural elements of constructions to elements such as wall coverings, feeding troughs, flooring, slurry storage tanks, and from mass or reinforced concrete to bricks with a mortar coating.

These cement matrices (mainly concrete) are exposed to a wide variety of aggressive agents (Pera et al., 1998), of a physical, chemical and biological nature, which may cause their degradation. For this reason, the correct design and production of these matrices is of great importance in order to ensure adequate durability throughout their service life and to reduce repair and maintenance costs to a minimum.

It is fundamental that such matrices present good behaviour for the installations to function correctly, since degradation implies considerable economic losses due to temporary suspension of activities and possible injuries to staff or to animals housed in the buildings, and has the potential for causing serious environmental impacts as a result of possible soil and underground water pollution.

In this kind of environment, attacks are usually of a chemical nature, directly related to contact with the chemical substances (Bertron et al., 2007), (Sanchez et al., 2009), (Larreur-Cayol et al., 2011) present in waste water from various sources, mainly animal excrements, silage effluents, cleaning and disinfection products, acids from the dairy industry and juices from the fruit industry. In addition, less serious damage has also been identified caused by the presence of waste from the remains of animal feed, raw materials and fertilizers, etc.

As a result of waste management policies, the reuse of various wastes (industrial, construction and demolition, etc.) is currently being considered by the construction sector as an alternative to the use of raw materials (Hendriks and Janssen, 2003), (Bianchini et al., 2005), (Gonzalez-Fonteboa and Martinez-Abella, 2005, Aquilar et al., 2007), (Poon and Chan, 2007, Poon et al., 2007), (Dapena et al., 2011). The consequent reduction in consumption of natural resources would lead to considerable economic, social and environmental advantages.

With respect to the waste generated by the ceramics industry (bricks, tiles, sanitary ware, etc.), various research projects are investigating the use of this kind of waste as a raw material in the production of cement, whether incorporating it as an active additive or as fine or coarse recycled aggregate in the production of mortar and concrete (Ay and Unal, 2000), (de Rojas et al., 2001a, de Rojas et al., 2001b, de Rojas et al., 2006, De Rojas et al., 2007, Frias et al., 2008), (Correira et al., 2006; Pereira, 2007), (Khalaf and DeVenny, 2005, Khalaf, 2006) y (Juan et al., 2010, Medina et al., 2011). To date, the results obtained have been satisfactory in both cases.

The aim of the present research was to study the viability of reusing waste from the ceramic sanitary ware industry as recycled coarse aggregate in partial substitution (20 and 25%) of natural coarse aggregate in the production of concretes for use primarily in agro-industry environments. This paper presents the results obtained for the corresponding mechanical, porosity, penetration of water under pressure and oxygen permeability assays.

EXPERIMENTAL PROCEDURE

MATERIALS

Aggregates

The natural aggregates employed can be sub-divided into two categories: the coarse fraction of boulder (gravel), corresponding to a fraction size of 4/20 mm in size (figure 1a), and the fine fraction (sand), with grains of less than 4 mm in size.

The recycled ceramic aggregate employed came from a ceramic sanitary ware factory. This ceramic waste was subjected to a crushing process using a jaw crusher, and was then sieved to extract the fraction of 4/12.5 mm in size. The aggregate obtained presented irregular shapes with marked edges (figure 1b), mainly due to the properties of the original product (generally of reduced thickness) and to the crushing process.

a) b)

Figure 1: Coarse aggregates: a) Gravel, b) Recycled ceramic

The physical and mechanical properties of the aggregates used are shown in Table 1, and complied with the requisites established by European standard EN 12620 (Asociación Española de Normalización y Certificación, 2009) and in Chapter III of the Spanish Instructions for Structural Concrete (EHE-08) (Comisión Permanente del Hormigón, 2008).

Table 1: Physical and mechanical properties of coarse aggregates

Characteristic	Gravel	Ceramic	EN 12620 / EHE-08
Grading modulus	6.93	6.17	-
Maximum size (mm) (EN 933-1)	20	12.5	-
Fine content (%)	0.22	0.16	<1.5
Dry sample real density (kg/dm^3) (EN 1097-6)	2.63	2.39	-
Water absorption (%) (EN 1097-6)	0.23	0.55	$\leq$ 5
Flakiness Index (%) (EN 933-3)	3	23	< 35
"Los Ángeles" coefficient (%) (EN 1097-2)	33	20	$\leq$ 40
Total porosity (%) (MIP)	0.23	0.32	-

Cement

The cement used was pure Portland cement (type CEM I 52.5 R), which fulfils the specifications given in the Instructions for the Authorization of Cements (Instrucción de Recepción de Cementos: RC-08) (Presidencia, 19/06/2008).

Concrete mixtures

Three types of concrete were mixed; a reference concrete (RC) and two concretes containing recycled aggregates in the proportions 20 and 25% (in weight). These were denominated CC-20 and CC-25, respectively. The design and calculation of these mixes was carried out using the de la Peña method (Arredondo, 1968), in which a characteristic compressive strength of 30 MPa and a constant water content is established according to the desired consistency (slump class between 60 and 90 mm) and maximum size of the aggregate (20 mm). Mix proportions of the various components are given in Table 2.

Table 2: Mix proportions of concretes

Type concrete	Materials (kg/m^3)				
	Sand	Gravel	Ceramic	Cement	Water
Reference concrete (RC)	716.51	1115.82	0.00	398.52	205.00
Concrete containing 20% recycled aggregate (CC-20)	725.81	892.66	216.43	387.64	205.00
Concrete containing 25% recycled aggregate (CC-25)	728.14	836.87	270.53	384.91	205.00

Experimental procedure

In this study, the following properties of hardened concretes were analysed: compressive strength and tensile splitting strength, pore size distribution (American Society for Testing and Materials, 2004), penetration of water under pressure (EN 12390-8) and permeability to oxygen (UNE 83981). It was tested 3 specimens for each type of concrete, altogether 9 specimens for each test, except for permeability to oxygen that 27 micro specimens were tested.

In order to determine total porosity of the concretes, a technique known as Mercury Intrusion Porosimetry (MIP) was employed using a Micromeritics mercury porosimeter, model 9500, which reaches a pressure of 33,000 psia (228 MPa), enabling determination of pore size from 6 to 0.005 µm.

RESULTS

Mechanical strength

The values obtained from compressive and tensile splitting strength at 28 days for the different concretes assayed are given in Figure 2. It can be observed that the incorporation of recycled ceramic aggregate improved mechanical behaviour, both as regards compressive and tensile strength, of the recycled concretes (CC) compared to the reference concrete (RC). Furthermore, as can be seen from Figure 2, the greater the percentage of substitution, the more marked the improvement. Thus, the concrete produced with a 25% substitution (CC-25) presented an increase in compressive and tensile strength of 12%, and of 25% compared to the control concrete (RC).

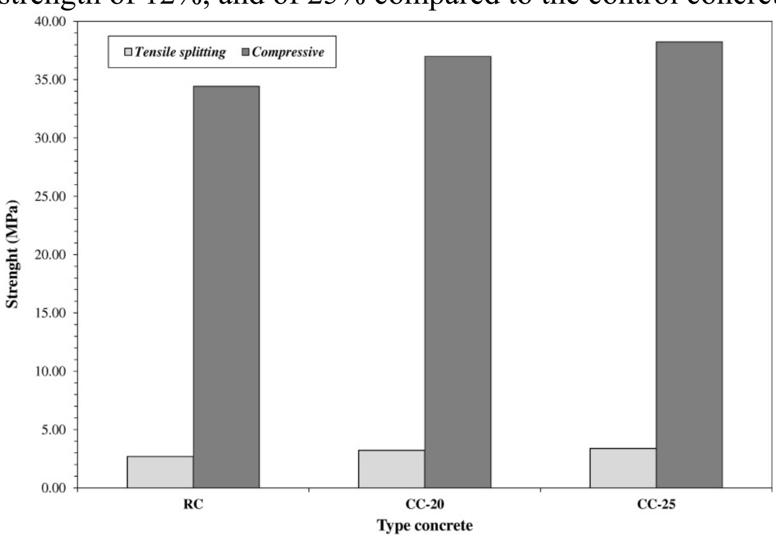

Figure 2: Compressive and tensile splitting strength at 28 days

Porosity

Table 3 and Figure 3 present the results obtained for porosity of the different concretes assayed. As can be observed in Table 3, partial substitution of natural gravel by recycled ceramic aggregate did not lead to a significant increase in porosity.

Table 3: Total porosity

Type concrete	Porosity (%)
RC	15.72
CC-20	16.21
CC-25	16.38

As regards pore size distribution (Figure 3), it can be seen that as the percentage of ceramic aggregate incorporated rose, the percentage of mesopores increased (0.05 < Φ < 0.002 μm) whilst the percentage of macropores decreased (Φ >0.05 μm), indicating a refinement of the pore system.

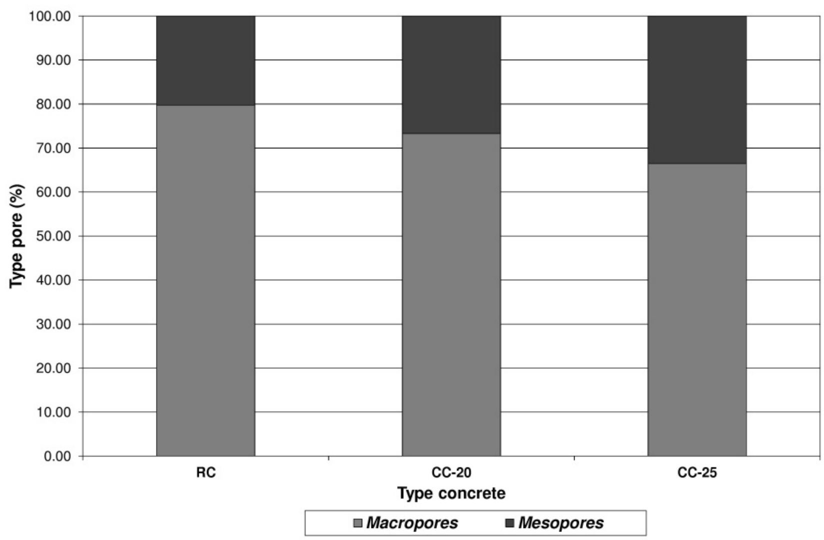

Figure 3: Pore size distribution

Penetration of water under pressure

The results for maximum depth obtained in the study of impermeability to water are shown in Figure 4. It can be observed that maximum depth remained almost constant for all the concretes assayed, and that the incorporation of ceramic aggregate was not associated with a negative effect. Furthermore, all three cases comply with the limits for maximum depth established in section 37.3.3 “Resistance to water concrete” in the EHE-08, indicating that these recycled concretes are impermeable to water.

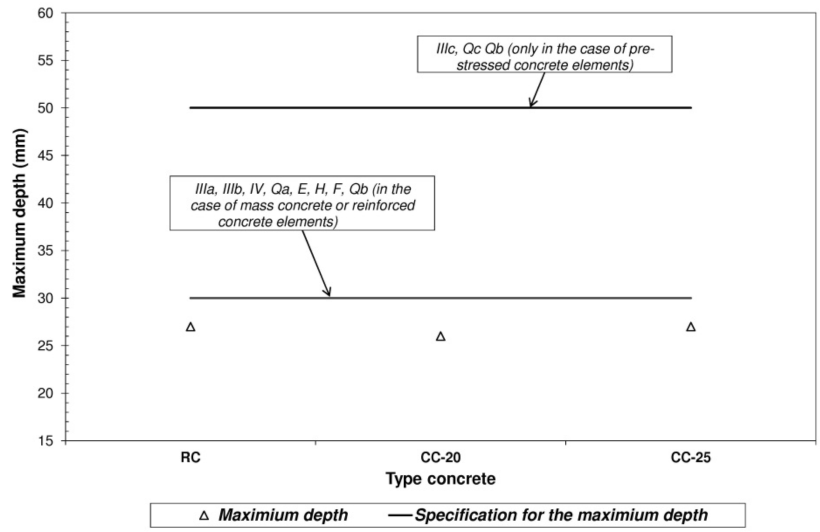

Figure 4: Mean and maximum depth of penetration of water under pressure

Permeability to oxygen

The results obtained for permeability to oxygen are shown in Table 4. As can be seen, a slight increase in permeability to oxygen was observed as the percentage of ceramic aggregate incorporated rose. However, permeability coefficient values remained virtually constant, since only a 2.66% increase was observed for the 25% ceramic aggregate substitution concrete (CC-25), compared to the control concrete (RC).

Table 4: Permeability to oxygen results

Type concrete	**$K*10^{-17}$ (m^2)**
RC	6,40
CC-20	6,49
CC-25	6,57

This slight increase was directly related to the effect of incorporating ceramic aggregate on the pore system of the new concretes, and to the characteristics of the oxygen molecules which are primarily spread through the capillary system.

It should be noted that these values fall within the established range ($K= 10^{-18}$-10^{-17} m^2) (Buyle-Bodin and Hadjieva-Zaharieva, 2002) for normal concretes.

CONCLUSIONS

The results obtained in the present research indicate that:

- Recycled ceramic aggregate presents suitable physical and mechanical characteristics for use as aggregate in the production of structural concretes.
- The recycled concretes show improved mechanical behaviour in comparison with the control concrete, presenting an increase in compressive and tensile strength as the percentage of substitution rose.
- The recycled concretes comply with the maximum water penetration requirements established in the EHE-08 for concretes in order to be considered sufficiently impermeable for use in all classes of environmental exposure.

- The reduction in resistance to oxygen penetration did not have negative effects on the durability of the concretes, since the resulting values were within the range of normal values.
- The recycled concretes may prove to be optimal for structural use in agro-industry environments, but require further study (durability).

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USE OF RESIDUES OF THE OIL INDUSTRY TO IMPROVE THE THERMAL PERFORMANCE OF CEMENT MORTARS

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CSAS11014 – Concrete and Green Building.

ABSTRACT Italy, along with Spain, is one of the major olive oil producing nations in the world. The olive oil production sector has reached a level of excellence in the food industry, especially in the south of Italy. Yet, compared to other important margins of development of the sector, there are a number of critical issues that strongly condition its expansion. The causes are mainly due to the difficulty of disposing the processing waste in an environmentally sustainable way. A particularly serious problem is the disposal of pomace. This substance is composed of residues from the pulp, skin and bits of pits mixed with vegetation water. Its moisture is highly dependent on the extraction technology and possesses a high pollution load. The techniques that have demonstrated a high environmental performance are based on the reuse of basic components of the residue. In particular, taking appropriate precautions, the olive oil mill wastewater is spread on agricultural land and becomes a useful source of nutrients for some plant species; the solid organs, such as pulp and skin, can be used for animal feed or to mend agricultural land; finally, olive stones, subjected to appropriate treatments, can be used as fuel. The size of olive stones in granular form ranges from 2 to 5 mm, depending on the type of crusher used, and, according to the degree of residual moisture, they have a calorific value ranging from 4000 to 4800 kcal/kg. The spread of its use as fuel is limited by reasons, such as the high production of fumes, the variability of calorific value and the need to adopt specific burners. A particular use of this vegetable substance may be as inert addition to cement mortar in order to improve its thermophysical characteristics and the weight per unit volume. The present paper analyzes the thermal performance of cement mortars with the addition of olive stones oil residue and proposes a series of solutions to achieve the best performance. The use of vegetable substances to produce lightweight concrete and to improve its final thermal performance is a commonly adopted solution. In particular, wood processing residues, such as wood chips or sawdust, cork and plant fibres are often used. The use of olive stones would allow, on the one hand, to reuse processing waste difficult to dispose of and, on the other hand, to improve the thermal characteristics of the cement mortars.
Keywords: Olive Mill Wastewater, Olive Stones, Thermal property.

INTRODUCTION Studies conducted by the United Nations Environment Programme (UNEP) have shown that buildings account for 30-40% of the world's energy consumption, 90% of which occurs during the phase of utilization, while the remaining part is consumed during the lifecycle of building materials (UNEP, 2007). The use of non-renewable energy resources is one of the main causes of greenhouse gas emission in the world. The fourth Intergovernmental Panel in Climate Change (IPCC) report indicates that construction is the sector with the greatest possibilities of saving, since, through negative-cost interventions, it is possible to reduce emissions by 30% within 2020. A considerable part of this energy is commonly used to control microclimate conditions, while another part is used to extract raw materials, to transport them, to make building components and to dispose of them. An estimate of the European Commission has shown that, if the current building methods are maintained, about 549 Mtep will be consumed for housing and for the tertiary sector in 2020; while, if a correct saving and energy efficiency policy were implemented and aimed at promoting technical solutions of thermal insulation and improvement of electrical equipment, a saving of 27% in housing and of 30% in tertiary building could be obtained. Therefore, interventions in this important productive sector should concern, on the one hand, the promotion of building techniques and of low environmental impact local materials and, on the other hand, the production of shells with limited heat loss. The peculiarities of the buildings for agricultural productions, such as the place, where agricultural and agri-food products are stored, and cattlesheds, would require the adoption of materials and building solutions which could allow a saving of energy resources as well as adequate internal microclimate conditions.

OBJECTIVES This paper is aimed at analyzing and evaluating a particular solution for the improvement of the thermal performance of the cement mortar commonly used to produce technical elements in the building sector. This solution envisages the addition of a part of olive stone granules, which are residues from the olive pressing during olive oil production, to mortar aggregates. The main objectives of this paper are to test the operational feasibility of the proposed solution and to verify, by comparing thermal conductance values, the improvement of the mortar thermal performance resulting from the addition of this part of processing residue coming from the oil industry. In order to evaluate the main thermophysical parameters of the tested mortar, a portable testing device has been created. Thanks to its simple functioning and high portability, it will allow to carry out rapid and low-cost preliminary evaluations of the thermal properties of the technical solutions to be adopted also with other materials. This opportunity is particularly useful in the rural context, where the wide availability of natural materials and of traditional building solutions encourages the preliminary comparison of the expected thermal performances.

MATERIALS AND METHODS The olive stone is the internal part of the drupe (almond), which is crushed during olive pressing and then eliminated with the olive processing residues (pomace). The olive stone can be eliminated directly from the olive paste in order to obtain sweeter and more oxidation-stable oil. However, at present, the greatest quantities are separated directly from the virgin pomace produced in oil mills (Fig. 1) or derive from the dedusting of the exhausted pomace produced in pomace factories. The olive stone is the solid part of pomace; its size depends on the pressing process adopted. Generally, it is in granules, whose size ranges from 2 to 5 mm. Its quantity ranges from 25% to 42% of the weight of the pressed olives, depending on the extractive technology used (Di Giovacchino, 2001), (Table 1). Its function during the extraction process is particularly useful, since it allows to obtain a more draining olive paste thus facilitating the extraction of the oil from the mass. Furthermore, its presence contributes to enrich the extracted oil with peroxides enhancing the slightly bitter tones of its taste. Thanks to its quite good calorific value, which is about 4100 kcal/kg, the olive stone can be used effectively as a fuel, above all in the boilers of oil mills, though it requires adequate burners and its combustion produces a greater amount of fumes compared to other kinds of biomasses. The olive stone deriving from the exhausted pomace produced in pomace factories has different characteristics (Table 2). As a matter of fact, the particular chemical processes adopted to extract the last traces of oil from virgin pomace make the olive stone unsuitable to be used in small boilers, owing to lighting problems, emissions of fumes and bad smells, clogging of exchangers and chimneys. One of the most interesting uses of the olive stone is its mixing with clay to increase the heat insulating power of bricks (Hai Alami A., 2010). Other studies have shown that the addition of the olive stone to bituminous concrete mixtures improves stripping resistance, durability and water and freeze-thaw resistance (Al-Masaeid, HR, 1994).

Figure 1. Olive stone resulting from physical separation from virgin pomace.

Literature reports the use of olive stone to produce Activated carbon (Sánchez, M.L.D. et al, 2006), as an additive for resins (Siracusa, G. et alt., 2001) and as an abrasive (Dawson, D., 2006). The olive stone used in the tests carried out for this paper derives from the centrifugal separation of "Carolea" and "Ottobratica" olive pomace. After separation, it has undergone a natural drying process in heaps located in the open but under a shelter. The tested olive stone has shown 23.67% humidity, a granule size ranging from 1 to 5 mm, a bulk density of 653 kg/m^3 and seemingly no impurity or dust. The evaluation of the change in the cement mortar thermal performance has been carried out at an experimental level by measuring the steady-state thermal conductivity of two cement mortar boards: the one was used as a reference and was built with traditional mortar; the other was built by adding a percentage of olive stone, equal to 70% of the dry mixture, to mortar aggregates. This percentage is the limit value below which wet mortar is adequately workable even on vertical surfaces. The mortar mixture is sold as dry premixture obtained with an automatic dosage of 25% grey Portland cement, 10% lime and 65% sand, grain size 2.5 mm. Max. 28 days after being mixed with water, it reaches a compressive strength of 17.65 MPa. The test mortar boards measured 0.30 x 0.30 x 0.020 m (± 0.002) and were built in the middle of a bigger plywood board supported by a fibreglass net (weighting 1.40 N/m^2) (fig. 2). Water was added to the premixed mortar in order to obtain plastic consistency. The quantity of water added was different for the two specimens. In the first specimen, the necessary quantity of water was 22.00% by weight; in the second specimen, where 70% by weight of dry olive stone was added, the necessary water was 29.50% (Table 3), owing to the needed hydration of the olive stone. Template filling was carried out in successive layers level with the two sides of the boards. The maturing of the mortar specimens took place in a humidity and temperature controlled environment during over 28 days. Before the test, the residual moisture in the mortar was 0.87%, in the reference specimen, and 0.50%, in the mortar mixed with olive stone.

Testing device The thermophysical parameters of the specimens were measured in an indoor environment. This solution allowed to obtain rapidly a steady-state heat flux and, therefore, to limit the time, needed to collect thermal data for each specimen, at 72 hours. Conductance was calculated using the values of the heat flux and of the surface temperatures of the mortar subjected to a thermal gradient of over 20°C. To that end, a contact heat fluxmeter and three contact probes were used to measure temperatures. The heat fluxmeter and the probes were connected with a data logger to collect data at 10' intervals (fig. 2). After 72 hours, collected data were acquired in a PC and processed through the programme "InfloFlux" (developed by ANIT National Association for Thermal Insulation and soundproofing). The processing procedure is based on the instantaneous calculation of conductance by means of (1)

$$C_t = \frac{\phi(t)}{T_h(t) - T_c(t)} \quad (1)$$

where $\phi(t)$ is the instantaneous heat flux measured at time t, $T_h(t)$ is the instantaneous temperature at time t in the hot side, T_c (t) is the instantaneous temperature at time t on the cold side. The calculation method used is the progressive averages that implies the calculation of parameters as mean values of the n measurements taken at the previous times and, therefore, (1) can be rewritten as follows:

$$C_t = \frac{\overline{\phi}(t)}{\overline{T}_h(t) - \overline{T}_c(t)} \quad (2)$$

where $\overline{T}_h(t) = \frac{1}{n}\sum_{i=1}^{n} T_h(t_i)$, $\overline{T}_c(t) = \frac{1}{n}\sum_{i=1}^{n} T_c(t_i)$, $\overline{\phi}(t) = \frac{1}{n}\sum_{i=1}^{n} \phi(t_i)$

In order to properly apply the method of progressive averages, it is necessary to check the presence of certain conditions, such as $\phi(t_i)$>5 W/m^2, a difference in temperature between the hot and cold spaces higher than 10°C, a thermal time lag in the wall below 10 hours (ISO 9869). With a view to guaranteeing the above-mentioned conditions, a specific testing device was designed and constructed. The main characteristics of that testing device are its portability and simple use. It is based on the use of a portable cold space on which mortar boards are fixed. The cold space, which, thanks to a cooling system, has an inner temperature of about 2°C and simulates the typical Mediterranean winter thermal conditions, though on stable conditions, is placed in an indoor environment with a temperature at least 20 °C higher than that which is kept in the cold space. The heat flux resulting from the difference in temperature between the two environments passes through the mortar board and is measured by the heat fluxmeter, while the surface temperatures are registered by three contact thermometers, one located on the hot side and two located on the cold side.

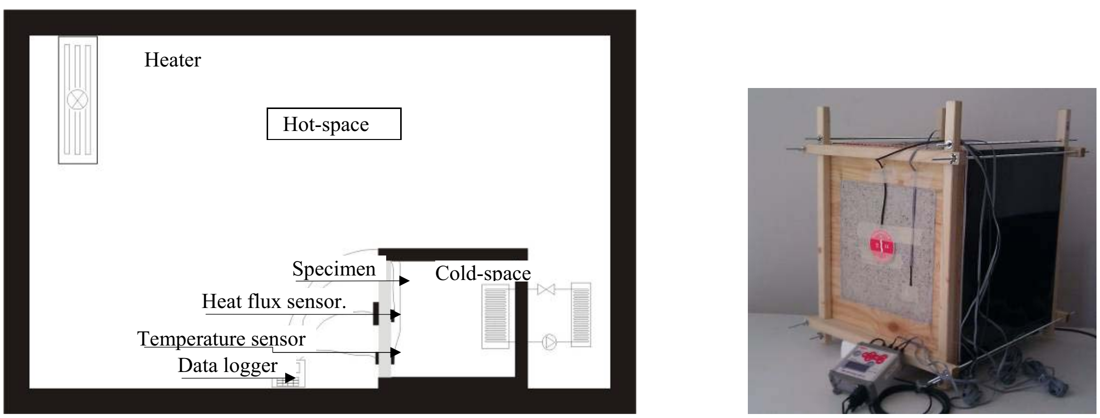

Figure 2. Testing device used for the comparative analyses of conductance in the mortar board

Table 1. Percentages of components of virgin olive pomace out of 100 kg of pressed olives and according to the used extraction technology.

Products	Centrifugation		Pressure
	Three phases	Two phases	
	%	%	%
Total pomace	45-55	70-80	30-40
Water	40-55	55-65	24-30
Oil	3.0-5.0	3.0-5.0	5.5-8.0
Pulpy part	18-20	12-15	25-28
Woody part	34-38	25-28	40-42

Table 2. Characteristics of exhausted pomace after solvent extraction.

Products	%
Oil	0.5 -1.0
Olive stone	45 - 60
Skin	8 - 12
Dust	20 - 30
Humidity	7 - 12

Table 3. Composition of fibres (%) in the olive stone and in the seed (the range of the fibre fraction is expressed in grams per 100 g of dry matter) (Heredia et al, 1987).

	Stone	Seed
Cellulose	29.79-34.35	2.36-3.91
Lignin	20.63-25.11	2.19-4.60
Hemicellulose	21.45-27.64	4.02-8.95
Ash	0.01-0.68	0.03-0.13

Execution of tests Boards were alternatively fixed to the cold space for thermal conductance analyses. After starting the cooling system of the cold space and the heating system of the hot space, a thermal camera registered the distribution of temperatures on the external surface of the specimen and the possible presence of heat losses or heat bridges. Moreover, this analysis allowed to check the correct positioning of the temperature and heat flux sensors (fig. 3). When the device reached steady-state conditions, the data logger, which was used to acquire the measured thermophysical values at fixed 10’ intervals, was also started. The possibility to consult the data logger for real-time measurements enabled to check when the heat flux reached a steady state. This condition is reached when the registered heat flux variations oscillate in the neighbourhood of the horizontal asymptote with a maximum width of 0.05 $Wm^{-2}K^{-1}$. When the above-mentioned condition was reached, the values acquired and stored by the data logger were transferred to the personal computer to calculate the conductance values with the method of progressive averages (ISO 9869).

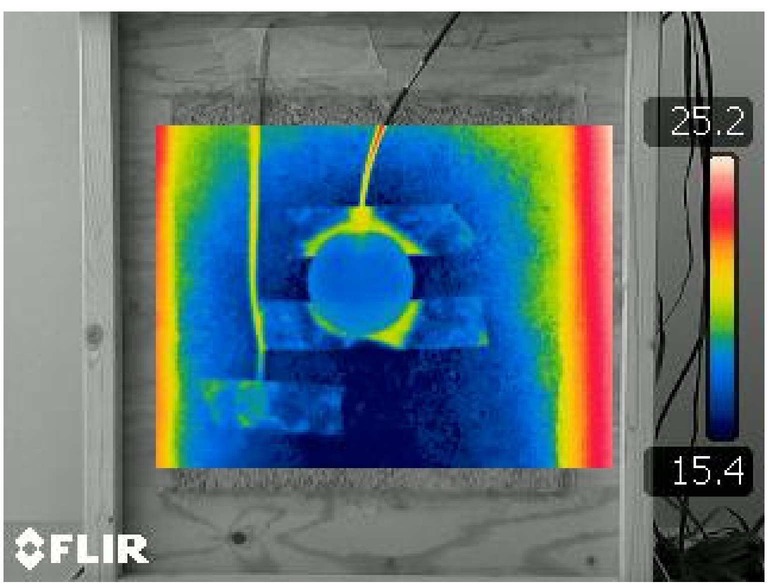

Figure 3. Thermographic analysis of the specimen

RESULTS AND DISCUSSION The steady-state conditions of the heat flux were reached in about 94 hours for the specimen in traditional mortar without olive stone, while they were reached in about 72 hours for the specimen to which olive stone was added. By means of automatic calculation, the registered thermophysical values enabled to determine the average conductance and heat transmission values. Processing resulted in an average conductance value of 55.56 $Wm^{-2}K^{-1}$, for the specimen in traditional mortar, and in an average conductance value of 16.20 $Wm^{-2}K^{-1}$, for the specimen with olive stone (Table 3).

Table 3. Values obtained from processing the thermophysical parameters registered during tests

	Traditional Mortar	Mortar with Olive Stone
Average flux (°C)	86.00	72.30
Average hot side temperature (°C)	14.16	17.31
Average cold side temperature (°C)	12.61	12.85
Average conductance ($Wm^{-2}K^{-1}$)	55.56	16.20

Moreover, graphs about the time variations in the processed values allow to highlight certain thermophysical properties of the two tested materials (fig.4).

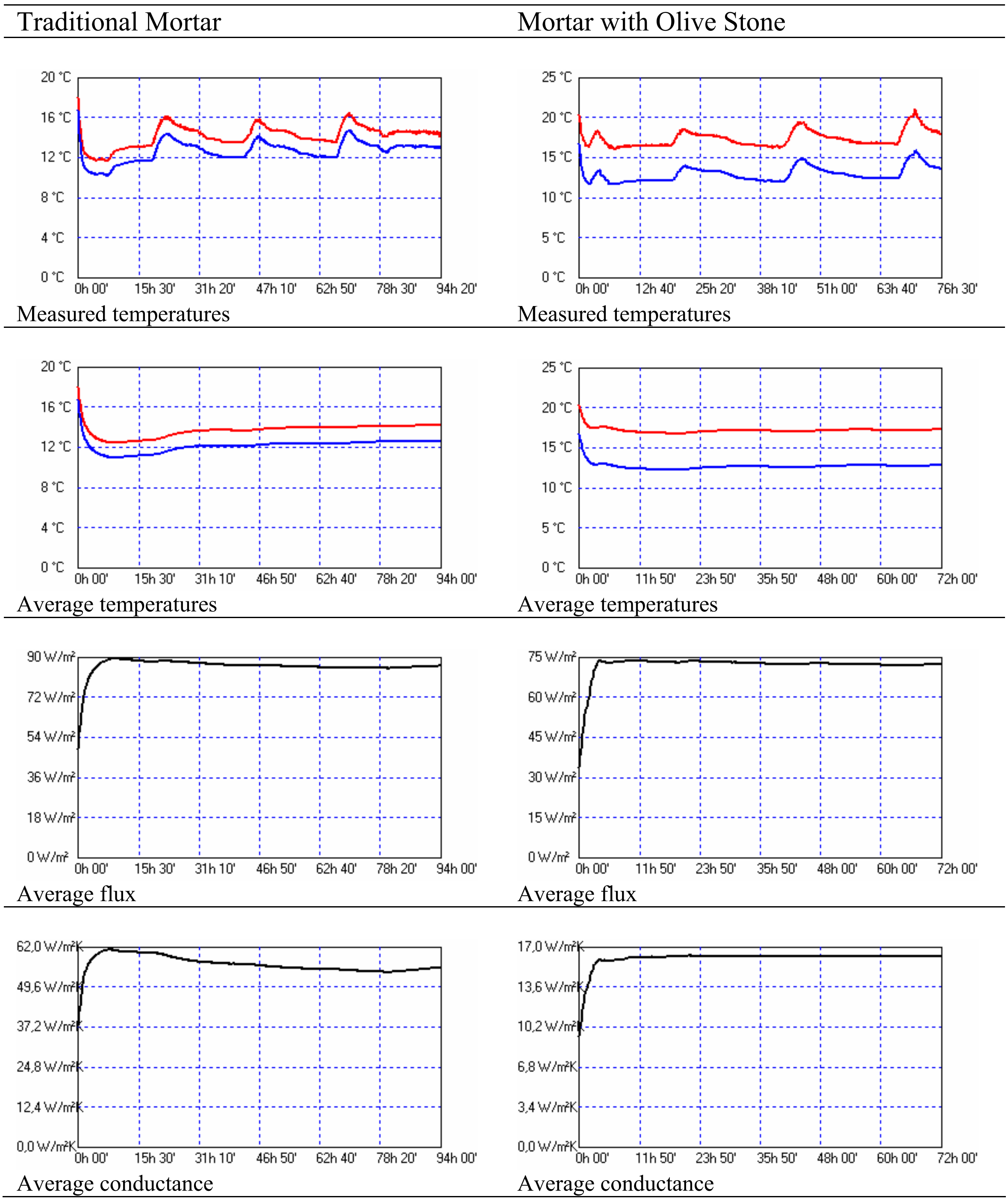

Figure 4. Time profile of the thermophysical parameters registered during tests for traditional mortar and for the mortar with olive stone. The red line indicates the values of the hot side of the specimen, while the blue line indicates those of its cold side.

The mortar with olive stone showed a conductance value of about 30% of the traditional one, i.e. the addition of a quantity olive stone equal to 70% by weight of dry mortar mixture leads to a decrease of about 70% the average conductance value.

Furthermore, the trend of the average time values of temperature and conductance shows an improved thermal behaviour, i.e. the mortar with olive stone shows a more constant thermal behavior in time and, above all, it reaches thermal equilibrium more rapidly, thus assuring better thermal performances in a shorter time.

This behaviour may be related to the decrease in the density (507.02 kg/m^3) value caused by the olive stone and to the consequent decrease in the mortar thermal capacity.

CONCLUSION The addition of olive stone to traditional mortar led to an overall improvement of thermal performances. In particular, a considerable decrease in thermal conductance and a higher overall thermal capacity were noticed. The improved thermophysical characteristics make this kind of mortar suitable for the construction of horizontal partitions and heat insulating walls both in new buildings and in the restoration of rural buildings. This solution would allow, on the one hand, to improve the overall thermal behaviour of the building by generating considerable energy saving thanks to the internal climate control, and, on the other hand, to re-use an agro-industrial residue that, otherwise, should be disposed of. This composition of mortar could be useful for several applications in the building sector, e.g. as floor screed or basic plaster for exterior wall coating. However, it should be carefully protected from water vapour and humidity. In fact, though to a limited extent, cement mortar is permeable to water and vapour that, if absorbed by olive stone granules, would significantly vary their thermal conductivity as well as the overall durability of mortar, since the vegetable substance may decay in time (Coatanlem, P, 2006). To that end, it is crucial to envisage adequate solutions to impermeabilize the mortar surfaces exposed to humidity, e.g. by treating exterior plaster walls with waterproofing finishes. Previous studies showed that in the case of wood chipping mortar, a material with physicochemical characteristics similar to those of olive stone, the preventive treatment of woodchips with sodium silicate solutions allowed to increase mortar durability and resistance in time (Coatanlem, P, 2006) as well as to increase its resistance to fungi and pests. Further research and analysis to evaluate thermal performances for different percentages of olive stone are currently in progress. Moreover, in order to verify a more general and complete applicability of the solution in the field of rural construction, the following important characteristics will be also analyzed: fire resistance, improvement of acoustic performances, shock resistance, durability and compatibility with other building materials.

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VALUE-ADDITION OF RESIDUAL ASHES FROM DIFFERENT BIOMASS ORIGINS IN CEMENT BASED MATERIALS: A COMPARATIVE STUDY

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CSAS11036 – Section 5: Ecological Concrete

ABSTRACT The rising fuel prices and growing concern about global warming associated with greenhouse gases emissions have increased the interest for clean bioenergy production. Conventionally, the use of biomass as an energy source is considered as carbon neutral or low carbon, which has potential of sustainable energy supply in future. However, production of ash from different bioenergy transformation processes can become an important source of pollution if appropriate disposal schemes are not implemented. The utilization of ashes from biomass origins in cement based materials could be an environmentally friendly and valuable approach. Currently, fly ash from coal is used in cement production. However, utilisation of biomass ashes as cement constituents is limited due to ASTM norm C618. In this research, ashes from different biomasses produced by different transformation processes have beencompared and discussed. For example, ash obtained from biochar by pyrolysis of swine manure has high percentages of elements such as K, P, Ca, and Mg which are suitable for soil fertility but not useful in cement manufacture. However, application of this ash on agricultural soil is restricted by norms to avoid overloading of nutrients in soil as well as leaching of heavy metals in natural water sources. Similarly, ashes from commercial wood pellets and switchgrass could be suitable for agricultural as well as for construction materials. Properties such as particle-size and bulk density of these ashes can be favourable for using them as substitute for sand and filler materials in construction materials.
Keywords: Ash, biomass, cement, combustion, bioenergy, swine, willow, switchgrass, wood pellet

INTRODUCTION The rising fossil fuel prices and growing concern about global warming associated with greenhouse gas emissions has increased the interest for energy production from renewable sources (e.g. biomass, solar, wind, etc.). Conventionally, the use of biomass as an energy source is considered as carbon neutral or low carbon because the CO_2 emitted during the transformation of material to recover the energy, is absorbed again by the biomass through the photosynthesis process. Though, such consideration is true only if the production of biomass is carried out in a sustainable way.

Ash-forming elements are present in biomass as salts, bound in the carbon structure (inherent ash) as mineral particles from dirt and clay introduced into the biomass fuel during harvesting or transportation (entrained ash) (Van Loo and Koppejan, 2008). During combustion, a fraction of such elements is volatilized and released to the gas phase. Later they form the fine mode of the fly ash characterized by a particle size of < 1µm. On the other hand, the non-volatile ash compounds remain in the char and results in residual ash particles with a wide range of compositions, shapes and sizes, related to the characteristic of the parent mineral particles. A fraction of the residual ash will be entrained with the flue gas and form the coarse part of fly ash (> 5 µm), while the other fraction will stay on the grate and form bottom ash (Van Loo and Koppejan, 2008).

The increased utilisation of biomass materials for the production of heat and power, especially in large power plants, increase the amount of ashes derived from such production. The review carried out by Reijnders (2005) listed the pollutants and hazardous elements that ash may contain. These include significant quantities of relatively mobile inorganic compounds (e.g. As, Cu, Cd), elements that give rise to significant aqueous leaching (Ba, Br, Ca, Cl, F, Fe, K, Mg, Na, Mn, P and S) and persistent hazardous organic pollutants (POP) such as polycyclic aromatic compounds (PAH), polychlorinated bifenyls (PCB) and chlorinated dioxins and benzofurans. Thus it is necessary to implement appropriate schemes for utilisation of ash disposal.

Nowadays, uses of ashes from different origins include soil stabilisation, mine backfill and agriculture. Benefits and barriers in each case are presented by Smith (2005). Ashes have also emerged as construction or geotechnical materials or have been proposed for such applications. Ashes, principally from the combustion of coal, had been used to replace a portion of cement in the concrete. The ASTM norm C618 covers fly ash for use in concrete where cementitious or pozzolanic action is desired. Using ash in concrete shows distinct quality advantages: improves workability, reduces segregation, bleeding, heat evolution and permeability, inhibits alkali-aggregate reaction, and enhances sulphate resistance (FHWA, 2011). There are additionally economic and ecological benefits.

Currently, most of fly ash used in cement production comes from coal. The quality of these ashes is controlled by standard specifications or classification systems. Usually, the ASTM norm C618 is applied. Additional standards include the AASHTO M 295 and the EN 450. However standard specifications were not specifically prepared for biomass or biomass co-firing ash (Van Loo and Koppejan, 2008). Even the use of biomass ash in concrete is prohibited by the ASTM norm C618 (Wang and Baxter, 2007). According to Rajamma *et al.*(2009), chemistry and mineralogy of biomass ashes differ from those of coal ashes. Although in some researches where results from the characterisation and analysis of biomass ash or co-firing biomass ash showed a similarity compared to the coal fly ash and to the specifications given by norms (Wang and Baxter, 2007; Wang *et al*., 2008; Rajamma, *et al*., 2009 and Esteves, *et al*., 2011). Thus the exclusion of biomass fly ash in concrete by norms seems inappropriate. Standards in a number of European countries have been drafted to permit the ashes from the co-combustion of coal with biomass to be used in concrete production (Van Loo and Koppejan, 2008).

ASTM norm C618 defines fly ash as the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gasses. Two major classes of fly ash are specified in ASTM C618 on the basis of their chemical composition resulting from the type of coal burned: Class F (normally produced from burning anthracite or bituminous coal) and Class C (normally produced from the burning of subbituminous coal and lignite). Class C fly ash usually has cementitious properties in addition to pozzolanic properties due to free lime, whereas Class F is pozzolanic fly ash and shows rarely cementitious properties when mixed with water alone. Table 1 shows the chemical and physical requirements listed in the ASTM C618 specification.

Table 1. ASTM Specification C618-08a. Chemical requirements

Chemical properties	**Class**	
	F	**C**
$SiO_2 + Al_2O_3 + Fe_2O_3$, (Σ), min., %	70.0	50.0
SO_3, max, %	5.0	5.0
Moisture content, max, %	3.0	3.0
Loss on ignition, max, %	6*	6.0

* The use of Class F pozzolan containing up to 12.0 % loss on ignition may be approved.

This research focuses on the use of ashes from biomass origins in cement based materials as an environmentally friendly and valuable approach. A comparative analysis of ashes from different biomass origins via different thermal transformation processes is discussed.

MATERIAL AND METHOD

Biomasses The ashes used come from four different biomasses: (i) commercial wood (a mix of black spruce and grey pine pellets), (ii) dried solid fraction of pig manure (SFPM), (iii) switchgrass, and (iv) willow. All biomasses, which were previously pelleted, were submitted under direct combustion via a pellet heater.

One additional biomass was evaluated. There is the biochar from the SFPM, when it was ubmitted into a pyrolysis process. The swine manure, procured from the collection pit of a local swine farm (St-Lambert, Quebec) in the form of 35% dry solids of swine manure, was pre-treated prior to the thermochemical conversion via pyrolysis. For this, the samples were put in an oven maintained at 105°C for a maximum of 72 hours on aluminium trays or until a constant weight . The dried samples were ground in a custom rotary cutter using 2 mm mesh size screen. The particles thus obtained was then transferred to a closed plastic container until used in the pyrolysis process. The physical and chemical properties of the biomasses are listed in table 2. The SFPM has the most important ash content of all biomasses tested under direct combustion (8.8 and 9.57 % d.b. at 750^{o}C and 1100^{o}C respectively), and the wood has the less important (0.5 and 0.38 % d.b. at 750^{o}C and 1100^{o}C respectively).

Table 2. Physical and chemical properties of the biomasses

		Wood	SFPM	Switchgrass	Willow	Biochar
Physical properties						
Diameter	(mm)	6.5	8.4	5.5	6.8	
Humidity	(% w.b)	6.6	10.5	14.1	12.7	1.97[a]
Density	(g/cm^3)	1.1	1.26	0.94	1.03	
Bulk density	(kg/m^3)	686	769	509	590	
Ash content (750^{o}C)	(% d.b.)	0.5	8.8	3.7	2.8	15.3[a]
Ash content (1100^{o}C)	(% d.b.)	0.38	9.57	4.3	4.0	
High heating value	(MJ/kg w.b.)	17.9	15.6	18.7	18	
Chemical properties						
SiO_2	(% w.b.)	0.041	0.986	2.405	0.716	6.13
Al_2O_3	(% w.b.)	0.014	0.180	0.168	0.153	0.95
Fe_2O_3	(% w.b.)	0.005	0.521	0.062	0.053	2.16
CaO	(% w.b.)	0.153	1.945	0.624	1.367	16.2
Na_2O	(% w.b.)	0.010	0.343	0.033	0.034	5.62
MgO	(% w.b.)	0.028	0.698	0.153	0.159	10.0
P_2O_5	(% w.b.)	0.006	1.464	0.157	0.215	20.9
SO_3[b]	(% w.b.)	0.008	0.834	0.089	0.145	
K_2O	(% w.b.)	0.047	1.321	0.229	0.588	20.9
Cl	(mg/kg)	13.8	3053	129	34.4	
C	(% w.b.)	47.1	40.5	43.5	45.1	36.9 (d.b)[a]
N	(% w.b.)	0.108	2.26	0.624	0.584	3.24 (d.b)[a]
TiO_2	(% w.b.)	0.000	0.009	0.009	0.005	0.05
V_2O_5	(% w.b.)	0.000	0.001	0.000	0.000	
CrO_3	(% w.b.)	0.000	0.003	0.001	0.001	0.03
MnO	(% w.b.)	0.015	0.025	0.010	0.013	0.31
CoO	(% w.b.)	0.000	0.000	0.000	0.000	
NiO	(% w.b.)	0.000	0.001	0.000	0.000	
CuO	(% w.b.)	0.000	0.022	0.001	0.001	
ZnO	(% w.b.)	0.001	0.039	0.004	0.027	
SrO	(% w.b.)	0.000	0.004	0.002	0.004	
ZrO_2	(% w.b.)	0.000	0.001	0.000	0.001	
Nb_2O_5	(% w.b.)	0.000	0.000	0.000	0.000	
BaO	(% w.b.)	0.001	0.001	0.002	0.004	

[a] Values corresponding to swine raw manure

Thermal processes

Direct combustion of the pellet biomass The direct combustion experiments were carried out in a 60,000 BTU/h (17.58 kW) output biomass pellet heater (Enviro; model: Omega). The method used is part of a study aiming to evaluate the emissions and the energy produced when such biomasses are employed for heat production by direct combustion (Godbout *et al*. 2011a; 2011b). In order to test the biomasses under their own optimal combustion conditions, a preliminary experiment were carried out to determinate the ideal input air flow for each biomass (Godbout *et al*., 2011a). The stove control board allowed five different burning rates (amount of pellets per unit of time), from which, three rates were used in the present experiments: (i) maximal, (ii) intermediate and (iii) minimal burning rate. Each test included the biomass burning at the three rates continuously. The experiments were carried out burning each biomass during 50 min at each burning rate (in steady state) with their own ideal input air flow. Three repetitions were carried out for each. Before each test and at each burning rate change, the biomass burned during 60 and 50 min respectively in order to get stable burning conditions. Moreover, the pellet heater was installed on a scale (± 0.05 kg) which collected the weight at regular intervals during the combustion process. After each test, the ash in the stove (bottom ash) and the ash in the chimney (fly ash) were collected and weighed.

Heating by oven after the biomass pyrolysis The Auger pyrolysis reactor used in this study is an interesting biomass pyrolysis technique which is a continuous type, equipped with computer control to maintain temperature of the pyrolysis zone at required level. The automated reactor control system also provided collection of data for temperature of different zones, pressure and rotational velocity of the Auger reactor. The heating block consisted of a solid copper block with lengthwise rod-type heating elements of about 300 W/inch capacity with 10 inches in length. The biochar collection system used gravity settling of heavier char particles from the pyrolysis vapor, alternatively, for a higher scale process this can be suitably replaced with a cyclone separator. Finally, the pyrolysis vapor was condensed using a cylindrical type condenser of about 12 L volume with a spiral cooling element (-10 to -15°C recirculating fluid) and a 2 L round bottom flask heat quenching system with 250 ml initial liquid volume. A vapor trap was installed before condenser to collect heavier bio-oil fraction and another vapor trap was installed after the condenser to trap the remainder lighter bio-oil fraction, if any. The pyrolysis vapor was extracted from the pyrolysis zone using a vacuum pump at the end of the vapor exit. The inside pressure of around 500 mm Hg during the pyrolysis operation was observed to be effective in evacuating the pyrolysis vapor. The biomass feeding control was calibrated with respect to particle size of the dried swine manure and it was verified at the end of each pyrolysis batch by measuring the remaining biomass. The whole Auger pyrolysis reactor system was cleaned at the end of each batch to minimize cross contamination of samples. The ash obtained from swine manure biomass was resulted from the combustion of the pyrolysis product, biochar. For this, the biochar was put in a crucible and heated in an oven at 550°C for 30 min followed by 950°C for another 30 min.

Characterization of ashes. Elemental composition of ashes were studied by X-ray fluorescence spectrometry (XRF). Chlorine content was measured by titration with silver nitrate ($AgNO_3$). C and N content was determined using a TruSpec analyser (LECO). The norm ASTM C311-11 was followed in order to determine the loss on ignition (LOI). In order to determine moisture content (MC), the collected ashes samples were dried in a laboratory oven at 105^{o}C. The ashes were not sieved before the analysis. On the other hand, the mineral analysis of the biochar ash samples was performed at a local mineralogy laboratory (COREM, Quebec City) as per standard procedures.

RESULTS AND DISCUSSION

Characterisation of fly ash from the direct combustion of the raw biomass. The total recovered ashes (fly and bottom ash) were 0.9 %, 10.2 %, 4.1 % and 4.1 % of the burned biomass, for wood, SFPM, switchgrass and willow respectively. Fly ash recovered represented a very small portion of total recovered ashes. In average, fly ashes recovered included 1.0 % and 2.8 % of total recovered ashes. Total biomass fly ash presented visually different colors. In the case of ashes recovered from wood, switchgrass and willow, they are predominantly grey but their tone is markedly different. The wood ashes are darker than those of the willow, and the switchgrass ashes are darker than the other two. The ashes from SFPM had a particularly different color. They presented a light brown color with some white fine particles produced by the metal oxides. Additionally, some particles completely unburned were noticed into the switchgrass ash samples. That could be caused by the fact that the pellets of such biomass were very fragile. Thus, high quantities of dust and fractured material were produced that they were easily and quickly transported by the airflow once arrived into the combustion chamber.

The chemical properties of the sampled biomass fly ashes are listed in Table 3. Results showed that all samples were predominantly made of SiO_2 (11.4−41.9%), CaO (16.5−29.3%) and C (9.6−14%). Generally, the ashes' chemical properties were proportional to those of the initial biomass. For instance, switchgrass showed a significantly high SiO_2 content before and after the combustion. Furthemore, the trace elements with the most important contents (ZnO, MnO and TiO_2) were the same in the biomasses and in the ashes. On the other hand, high Cl content were found in fly ashes (13,806−35,223 mg/kg).

When compared with ASTM chemical requirements, the fly ashes of the evaluated biomasses did not meet all ASTM parameters in order to be classified by such norms. In fact, none of the fly ashes meet the minimal sum of SiO_2 + Al_2O_3 + Fe_2O_3 content and the LOI parameter. The switchgrass fly ashes showed a sum value close to ASTM class C designation, i.e. 47.7 %. In fact, the switchgrass fly ash presented a high and predominantly SiO_2 content (41.9 %). With respect to LOI results, no biomass respected the maximal allowed value to ASTM requirements (6.0 % max.). The switchgrass ashes had the closest value (14.8 %). In the other hand, the SO_3 and the moisture contents of fly ashes of all tested biomasses were into the ASTM limit values. However, because fly ash from our biomasses did not meet all chemical ASTM requirements, they should not be used in concrete materials as replacement of a part of the cement. In fact, such ashes would not have an enough pozzolanic propriety because of the important deficit in Al and Fe content.

Table 3. Chemical requirements of ASTM specification (C618-08a) for fly-ash and properties of the biomass fly ash samples.

		ASTM requirements		Biomass fly ash properties			
		F	C	Wood	SFPM	Switchgrass	Willow
SiO_2	(% w.b.)			11.4	13.0	41.9	16.4
Al_2O_3	(% w.b.)			2.3	2.0	3.8	2.6
Fe_2O_3	(% w.b.)			3.7	4.3	2.0	2.4
Σ^a	(% w.b.)	70.0	50.0	17.3	19.3	47.7	21.4
CaO	(% w.b.)			24.8	20.4	16.5	29.3
Na_2O	(% w.b.)			1.9	2.9	1.4	1.5
MgO	(% w.b.)			5.8	6.9	3.8	5.1
P_2O_5	(% w.b.)			9.5	12.8	4.6	7.7
SO_3^b	(% w.b.)	5.0	5.0	4.4	3.9	1.5	3.2
K_2O	(% w.b.)			2.6	5.1	4.1	4.1
Cl	(mg/kg)			34,314	35,223	13,436	13,806
C	(% w.b.)			14	11.5	9.6	10.7
N	(% w.b.)			0.646	0.883	0.366	0.494
TiO_2	(% w.b.)			0.13	0.12	0.23	0.14
V_2O_5	(% w.b.)			0.01	0.01	0.00	0.01
CrO_3	(% w.b.)			0.09	0.06	0.05	0.06
MnO	(% w.b.)			0.65	0.46	0.40	0.46
CoO	(% w.b.)			0.00	0.00	0.00	0.00
NiO	(% w.b.)			0.03	0.02	0.01	0.02
CuO	(% w.b.)			0.06	0.08	0.03	0.06
ZnO	(% w.b.)			1.25	0.97	0.45	0.78
SrO	(% w.b.)			0.07	0.06	0.05	0.09
ZrO_2	(% w.b.)			0.01	0.01	0.01	0.01
Nb_2O_5	(% w.b.)			0.00	0.00	0.00	0.00
BaO	(% w.b.)			0.10	0.06	0.07	0.10
PbO	(% w.b.)			0.00	0.00	0.00	0.00
MC^c	(% w.b.)	3.0	3.0	1.6	1.3	0.8	0.8
LOI^d	(%)	6.0^e	6.0	22.6	22.2	14.8	20.3

[a] Sum of SiO_2 + Al_2O_3 + Fe_2O_3, (min, %); [b] SO_3 (max, %); [c] Moisture Content (max, %); [d] Loss on Ignition (max, %); [e] The use of Class F pozzolan containing up to 12.0 % loss on ignition may be approved.

Characterisation of bottom ash from the direct combustion of the raw biomass. Higher quantity of ash was recovered from the bottom of the heater, between 97.2 % and 99% of total ashes recovered. As expected, the wood produced the less (0.9 % w.b. of which 97.2 % were bottom ash) and the SFPM produced the most (10.2% w.b. of which 99 % were bottom ash). The switchgrass and willow produced both 4.1 % w.b. of which 98 % were in bottom ash.

Visually, bottom ashes showed the same color difference as corresponding fly ashes. Equally, CaO, SiO_2 and C were the principal chemical elements constituting bottom ashes for each biomass as fly ashes (table 4). However, the P_2O_5 and K_2O content in SFPM ash were more important than C. In fact, important quantities of such elements were present in the initial biomass. Thus, these elements are hardly decomposed and volatilized during combustion; they remain attached to the solid waste fraction. Additionally, wood ash presented a significant high carbon content (41.7 %). Based on the organic matter and carbon content ratio (1.2), it can be deduced that C in wood ash is present in a graphitic form.

Table 4. Chemical requirements of ASTM specification (C618-08a) for fly-ash and properties of the biomass bottom ash samples.

		ASTM requirements		Biomass bottom ash properties			
		F	C	Wood	SFPM	Switchgrass	Willow
SiO_2	(% w.b.)			6.7	10.8	55.2	17.2
Al_2O_3	(% w.b.)			1.5	2.0	4.5	3.1
Fe_2O_3	(% w.b.)			4.2	5.7	4.2	1.7
Σ[a]	(% w.b.)	70.0[a]	50.0[a]	12.5	18.5	63.9	22.0
CaO	(% w.b.)			19.7	22.0	16.2	30.2
Na_2O	(% w.b.)			1.1	3.8	1.0	1.0
MgO	(% w.b.)			3.7	8.0	3.7	3.6
P_2O_5	(% w.b.)			1.8	16.4	3.8	5.1
SO_3	(% w.b.)	5.0[b]	5.0[b]	1.4	6.1	0.3	1.7
K_2O	(% w.b.)			5.0	11.0	5.2	11.0
Cl	(mg/kg)			1,238	11,829	543	594
C	(% w.b.)			41.7	8.2	2.6	13.1
N	(% w.b.)			0.3	0.3	0.1	0.3
TiO_2	(% w.b.)			0.07	0.11	0.26	0.13
V_2O_5	(% w.b.)			0.00	0.01	0.01	0.00
CrO_3	(% w.b.)			0.33	0.06	0.30	0.07
MnO	(% w.b.)			1.79	0.29	0.25	0.30
CoO	(% w.b.)			0.00	0.00	0.00	0.00
NiO	(% w.b.)			0.10	0.03	0.49	0.02
CuO	(% w.b.)			0.04	0.21	0.02	0.02
ZnO	(% w.b.)			0.17	0.27	0.07	0.24
SrO	(% w.b.)			0.06	0.05	0.04	0.09
ZrO_2	(% w.b.)			0.01	0.01	0.01	0.02
Nb_2O_5	(% w.b.)			0.00	0.00	0.00	0.00
BaO	(% w.b.)			0.15	0.02	0.05	0.10
PbO	(% w.b.)			0.00	0.00	0.00	0.00
MC[c]	(% w.b.)	3.0[c]	3.0[c]	1.6	0.3	0.3	0.4
LOI[d]	(%)	6.0[d,e]	6.0[d]	50.1	10.8	3.9	21.6

[a] Sum of $SiO_2 + Al_2O_3 + Fe_2O_3$, (min, %); [b] SO_3 (max, %); [c] Moisture Content (max, %); [d] Loss on Ignition (max, %); [e] The use of Class F pozzolan containing up to 12.0 % loss on ignition may be approved.

With regard to Cl content, SFPM showed a value significantly higher than other biomasses ($Pr<0.0001$). The wood, willow and switchgrass ashes did not present significant differences among themselves ($Pr>0.1$) due to the large variability in the data. The cause of such variability was not identified. On the other hand, relative high values were found for MnO (0.3−1.79 %), ZnO (0.07−0.27 %) and TiO_2 (0.07−0.26 %). The same trace elements were reported in fly ash.

Excluding switchgrass, the biomasses bottom ash could not be classified into the ASTM norm because of the high values of LOI (10.8−50.1 %) and also, in the case of the SFPM ash, with the higher SO_3 content (6.1 %). The switchgrass bottom ash met the requirements for class C designation. Critical properties found in fly ash were different than in bottom ash for switchgrass. In fact, ash had a significantly higher SiO_2 content (55.2%) and a low LOI result (3.9%) that allowed being classified as class C ash. Thus it should be showing pozzolanic behavior in a concrete material. Further studies should be focused in the study of the physical properties of concrete materials when using switchgrass bottom ash.

Characterisation of ash from biomass submitted to a pyrolysis process. The ash composition of minerals in biochar ash is showed in table 5. Results evidently suggest that it might not be a suitable option for integration with concrete constituents as major constituents such as SiO_2, Al_2O_3, Fe_2O_3 had substantially lower concentrations than required as per ASTM norms for concrete. In principle, the ash obtained from biochar from pyrolysis of animal manure is very different from those of wood and other biomasses. On the other hand, it might be interesting to investigate the maximum concentration of ash from biochar by pyrolysis of animal manure, which can be mixed with concrete without compromising structural properties and environmental safety. As a matter of fact, ash obtained from biochar by pyrolysis of animal manure is recommended for agricultural land and regarded as safe; therefore, it might also be safe for mixing with concrete.

Table 5. Chemical properties of the biochar samples from swine manure pyrolysis (%).

Constituent	
SiO_2	6.13
Al_2O_3	0.95
Fe_2O_3	2.16
CaO	16.2
MgO	10.0
Na_2O	5.62
K_2O	20.9
TiO_2	0.05
MnO	0.31
P_2O_5	20.9
Cr_2O_3	0.03
LOI	11.3
Total	**83.25**

CONCLUSION The chemical proprieties of biomass ash were analyzed to determine their potential use as a replacement of a part of the cement in the concrete. The biomasses tested included wood, solid fraction of pig manure, switchgrass and willow. Results led to the following conclusions: 1.There is a close relation between the chemical properties of the initial biomass and both fly and bottom ashes. 2. Biomass fly ash present high Cl content (13,806−35,223 mg/kg). 3. Biomass fly ash has not enough $SiO_2 + Al_2O_3 + Fe_2O_3$ content and LOI value to meet ASTM specifications. Thus they are not suitable as cement replacement. 4. Cl content in biomass ash was high and very variable. 5. Switchgrass bottom ash show a potential use in concrete materials. In fact it met ASTM chemical specifications for the class C designation. However, further studies should be focused in the study of the physical properties of concrete materials when using such ash. 6. Ash from biochar produced from swine manure pyrolysis is not suitable for integration with concrete constituents. Further studies could consider studying the chemical proprieties of a biomass ash with coal fly ash mix.

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