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DEVELOPMENT OF AN AGRICULTURAL-BASED BIOPOLYMER PRODUCT

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Abstract

University of Saskatchewan researchers have developed a biocomposite material which includes tailings from flour production. The inclusion of flour tailings reduces the overall material cost of the biocomposite. The physical characteristics of samples made from various compositions were compared, and used to optimize the composition of the biocomposite. Water absorption, durometer hardness, and impact resistance of the biocomposites were studied using American Society for Testing Materials (ASTM) guidelines. Data indicated that samples containing approximately 40% flour tailings (by mass) and approximately one quarter polyethylene (by mass) demonstrated the best combination of desirable characteristics.

Keywords. Biopolymer, biocomposite, polyethylene, flour, flax fiber, thermo-molding, water absorption, durometer hardness, impact resistance

Introduction

A biocomposite has been developed at the University of Saskatchewan that contains tailings from flour production. The new product is intended for use as an environmentally mindful alternative to conventional plastics. As explained by Johnson et al., polymers are often coupled with filler materials that may affect physical characteristics of the compounded material. The flour tailings function as filler for the biocomposite, in order to reduce material costs and to improve its physical properties. The goal of the current research is the optimization of ingredient proportions in order to produce the most satisfactory physical properties in the final product.

Development of novel biocomposite materials has been reported by a number of research institutions. The ACRES (Affordable Composites from Renewable Resources) group at the University of Delaware has developed a vacuum-assisted resin transfer molding process. This process is used to bind natural fibres and soybean oil resin into mats, intended for use in construction, furniture manufacturing, and in automobile parts (O'Donnell et al., 2004). As reported by Domenek et al. (2004), research in France has led to development of homogeneous, transparent, strong, and water resistant films obtained through casting or thermal film forming processes. These films contain inexpensive and biodegradable wheat gluten. American researchers have created composites that contain soy flour, glycerol, polyester amide, and pineapple leaf fiber (Liu et al., 2005). Pineapple leaf fibre was selected for its high tensile strength, high cellulose content, high degree of crystallinity, and wide availability in the southern USA. The composites were formed by a sequence of extrusion, pelleting, and injection molding.

Physical properties of the composite material have been examined and are being reported. Water absorption, durometer hardness, and impact resistance were examined using American Standard for Testing Materials (ASTM) standards. Water absorption of the material is influenced by the hydrophilic nature of its biological components. Durometer hardness testing is based on the inversely proportional relationship between penetration of a testing mechanism and relative hardness. Impact resistance of a material quantifies its resistance to sudden impact.

Materials and Methods

The biocomposite under consideration is a blend of flour tailings, pea starch, flax fibre, guar gum, D-sorbitol, sodium benzoate, paraffin wax, and one or more forms of virgin or recycled polyethylene, or polyvinyl alcohol (PVA).

Each component affects overall stability of the final product. Guar gum is a hydrocolloid, intended to bond and waterproof the product surface. D-sorbitol is an alcohol sugar, and functions in the bonding of the final product and in the maintenance of the moisture content. Sodium benzoate functions as a preservative. The remaining components of the material were included in varying amounts, with optimization of the composition as the primary focus of this work. Pea starch was included in the formula in order to form a material matrix. Flax fibre contains cellulose, and is intended to strengthen the material and add structural integrity by bonding with the starch. Paraffin wax, an alkane hydrocarbon, decreases product solubility in water and aids in product release from the thermo-moulder. The conventional and recycled polymers were incorporated in order to stabilize the material. Finally, glycerol was included in some samples to aid with product release from the mold.

Samples were thermo-molded in a 30 ton press. The materials were mixed for five minutes, to ensure a homogeneous blend. The thermo-molder was heated to 132°C (270°F) before the

sample was placed in the mold. Each thermo-molded sample had a mass of 300 g, and each ingredient was added as a mass percentage. Water was misted on the top and bottom of the mold, to ensure gel formation by the guar gum, resulting in encapsulation of the material. The mold was closed, and pressure was applied to the material for 12 minutes. Pressure applied to the biocomposite was maintained at approximately 4 MPa. The process used in the development of this biocomposite is similar to the thermal casting method of Domenek et al. (2004).

Samples were cooled and stored at laboratory conditions (23°C and 50% relative humidity) until mechanical testing was completed. The relative rates of water absorption, hardness, and impact resistance of the samples were recorded. The specimens used for mechanical testing were selected in an identical manner from each sample, and obtained using a conventional band saw.

As demonstrated by Liu et al. (2005), ASTM Standard D570 (Standard Test Method for Water Absorption of Plastics) (ASTM, 1998a) quantifies the water absorption by biocomposite samples. In the current work, samples were weighed and had geometric measurements taken prior to exposure, after two hours of water absorption, after 24 hours of water absorption, and after a reconditioning (drying) period of 24 hours. Specimens were immersed in 50 mL of distilled water at room temperature, as shown in Figure 1.



Figure 1. Water absorption testing of biocomposite samples.

Hodson et al. (2001) note the destructive nature of durometer testing. However, sample destruction was considered inconsequential for the purposes of this work. ASTM Standard D2240 (ASTM, 1998b), the Standard Test Method for Rubber Property – Durometer Hardness was used to quantify the relative hardness of samples. The hardness of five locations of each sample was recorded, using a type DO durometer. Testing was done at the geometric center of whole specimens, and at four equidistant sites. Manual operation was employed, and the readings were averaged to provide one hardness value for each sample.

ASTM Standard D5628 (ASTM, 1998c), the Standard Test Method for Impact Resistance of Flat, Rigid Plastic Specimens by Means of a Falling Dart was used to rank the samples by their ability to resist sudden impact. This method was chosen over the Charpy impact method because of the impracticability of notched specimen preparation for the Charpy test (Chang et al., 2004). Three replicates from each sample were tested. The mechanism used for testing has a 1 m drop height and a flat platform on which the specimen is placed, as shown below in Figure 2. Impact was delivered by a conical dart, either stainless steel (mass = 996 g) or aluminum (mass

= 350 g). Up to three different levels of testing were completed for each sample type, using variable height and variable mass techniques. Samples which cracked or split were recorded as failures. The mechanism of failure (brittle or ductile) was also documented.



Figure 2. Impact dart and apparatus base.

Results and Discussion

Data from each mechanical test was compiled and analyzed. Water absorption data included changes in specimen mass and specimen surface area after two and 24 hours of soaking in distilled water at laboratory conditions (temperature of 23°C and 50% relative humidity). Water absorption by the biocomposite is important for its proposed end use. The desirable result is that the biocomposite be capable of absorbing an appreciable volume of water, while maintaining structural stability. The average increase in mass after two hours of water exposure was 34.6%. After 24 hours of water exposure, the average increase in mass was 52.6%.

Recycled polyethylene and PVA appear to impart the best water absorption characteristics in the biocomposite samples, given that samples which contained recycled plastic milk jug material or PVA as the plastic binder absorbed significant amounts of water. The relationship linking water absorption and glycerol content was inconsistent, and could not be defined.

Dimensional stability was quantified by measuring the change in surface area of the biocomposite specimens after the water absorption process. The desirable result is that there is minimal change in surface area after reconditioning the specimens. After the two and 24 hour soaking periods, the average change in surface area was 3.1% and 19.2%, respectively. Virgin polyethylene was the plastic stabilizing material in the samples with greatest dimensional stability, and the majority of samples with above average stability also contained glycerol.

After 24 hours of reconditioning (drying), the final mass of the biocomposite specimens was recorded. Overall mass change indicates the quantity of material solubilized during testing. A balance is required between the level of water absorption by biocomposite samples and their respective solubility during testing. Therefore, changes in mass and surface area of specimens after the reconditioning period should be minimized. Current work did not demonstrate a relationship between material composition and sequential wetting and drying. Mass changes were due to material disintegration in the water, and size changes were due to sample cracking

or curling upon drying. In general, most samples lost a small percentage of their mass after 24 hours of water exposure.

Durometer hardness values range between zero and 100. The average readings ranged between 31 and 63, where a higher reading indicates a harder material. It is desirable for the material to have a hardness value greater than the average value of 51. Hardness of individual biocomposites is variable, depending on sample composition. Current data indicates that the hardest samples contain little or no glycerol. In addition, the optimum plastic stabilizers appear to be virgin polyethylene and PVA.

Impact resistance testing was completed at six possible energy levels, with a maximum of three levels being applied to each sample. These energy levels are listed in Table 1. Samples were first exposed to a moderate (type C) test, and energy application was either increased or decreased, depending on the result of the first energy application.

Table 1. Impact resistance test energy levels.

Test Code	Dart Type	Height (mm)	Energy (J)
A	Aluminum	127	0.44
B	Steel	127	1.24
C	Aluminum	254	0.87
D	Steel	254	2.48
E	Aluminum	381	1.31
F	Steel	381	3.72

Test results were recorded as “Pass” or “Fail”. Any specimen which exhibited evidence of cracking or damage as a result of impact was recorded as a failure. Impact resistance testing was examined in order to find the samples which were most resistant to sudden impact, and which failed in a ductile manner. Few samples were able to sustain the impact from the tests which applied higher energies on impact (types D, E, and/or F). A relationship linking sample composition and impact resistance did not result from this research.

Conclusions

The data under consideration has led to a number of conclusions regarding the newly developed biocomposite. Water absorption data indicated that the various biocomposite samples could absorb high levels of water throughout the exposure period. Samples that absorbed high amounts of water contained recycled polyethylene or PVA. No conclusion regarding the influence of glycerol content on water absorption by the material can be obtained from current data. Dimensional stability was also considered during water absorption testing. High polyethylene and paraffin wax content provided specimens with the best stability. Data from durometer hardness testing indicates that virgin polyethylene and PVA are superior plasticizers. Reduced glycerol content appears to increase relative hardness of the material. Impact resistance data is not indicative of a relationship between formulation and impact resistance

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