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Physicochemical Characteristics of Densified Untreated and Microwave Pretreated Canola Straw Grind

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ABSTRACT The goal of this study was to determine the ability of microwave-chemical pretreatment to enhance compression characteristics and densification process of canola straw grind. In this study, ground canola straw samples were immersed in alkaline (sodium hydroxide and calcium hydroxide) solutions and then exposed to microwave radiation. Chemical composition, bulk, and particle densities of samples were determined before and after pretreatment. Untreated and microwave pretreated samples with 12% (w.b.) moisture content were compressed at four levels of compressive forces (1000, 2000, 3000 and 4000 N) in a plunger die assembly and the compression and relaxation test data were recorded. The specific energy required for compression and extrusion of pellets produced from untreated and pretreated canola straw grinds was calculated. The fracture load and tensile strength of the pellets were also evaluated to investigate the hardness of the pellets. Chemical composition analysis showed that microwave pretreatment was significantly able to disintegrate the lignocellulosic structure of canola straw grinds. Data analysis also revealed that the pellets made from microwave pretreated biomass grinds had a significantly higher density and tensile strength than untreated samples.

Keywords: Pelleting, Densification, Canola straw, Microwave pretreatment, Chemical pretreatment.

INTRODUCTION The role of biofuel has been strongly enhanced by their consideration in the climate change debate, as well as opportunities for rural development and improved energy security. Both developed and developing countries have embarked on programs to develop the

technology and infrastructure to economically and sustainably produce biofuel from biomass. Increasing the energy security, reduction in greenhouse gas emissions, use of renewable resources, and establishment of a carbohydrate-based chemical process industry are the main benefits of using lignocellulosic biomass as a stable energy source. Agricultural residue is example of lignocellulosic biomass that could be used as a fuel for energy generation.

Because agricultural biomass is of irregular shape and size and low bulk density, it is very difficult to handle, transport, store, and utilize in its original form. Densification of biomass into durable compacts is an effective solution to these problems and it can reduce material waste. Densification of biomass is an efficient method for a number of reasons such as improvement of handling and storage characteristics, lowering transportation costs, enhancing volumetric calorific value, improvement of control over the combustion process, increasing the uniformity of physical properties for making uniform, clean and stable pellets for production of environment-friendly fuel (Granada et al., 2002; Sokhansanj et al., 2005). Biomass can be compressed and stabilized by 7 to 10 times the density of standard bales by the application of pressures between 400–800 MPa during the densification process . The bulk density of loose and standard baled straw is approximately 40 and 100 kg/m^3, respectively and can be increased to 600-1200 kg/m^3 by the densification process (Demirbas 2001; Tripathi et al. 1998).

Binding compounds are very important in compacting materials and manufacture a consistent, durable and high quality pellet. Thus, natural or synthetic binding agents are often added to agricultural biomass before densification process to enhance the quality of pellets. Many biomass materials contain natural components such as lignin that contribute to binding the particles but they may not be available in a form or state or abundant enough to remarkably contribute to product binding in densification process. Therefore, a pretreatment process is required to change the structure of biomass materials and release desirable binding materials.

A number of pretreatments have been developed and applied to change the lignocellulosic structure of agricultural residue to improve production of ethanol and other chemicals. These pretreatments include physical, biological, and chemical methods, such as steam explosion, liquid hot water, dilute acid, flow-through acid pretreatment, lime, ammonia fiber/freeze explosion, and ammonia recycle percolation. Most of these methods involve high temperature, which is usually achieved through convection- or conduction-based heating. Dielectric heating including microwave irradiation is an alternative method to conventional heating. Microwave irradiation has been applied as an efficient pretreatment technique to enhance the enzymatic hydrolysis of biomass materials. Some studies have demonstrated that microwave irradiation can change the structure of lignocellulosic materials so that it disintegrates the lignin and hemicellulose, changes the ultrastructure of cellulose and enhances the enzymatic hydrolysis of biomass materials (Azuma et al., 1984). Ooshima et al. (1984), Azuma et al. (1984) and Kitchaiya et al. (2003) reported an improvement in enzymatic hydrolysis of rice straw and sugar cane bagasse pretreated by microwave in the presence of water in comparison to untreated biomass. Alkali pretreatment is a typical chemical pretreatment method for lignocellulosic materials based on the chemical reaction between alkali and lignocellulosic materials. Sodium hydroxide (Carrillo et al. 2005) and lime, or calcium hydroxide (Kim and Holtzapple 2006) have been investigated for alteration of lignocellulose. When biomass is treated by dilute alkaline solution, swelling increases the internal surface area of the material. Swelling causes a decrease in the degree of polymerization, separation of structural linkage between lignin and carbohydrates, and disruption of lignin structure. Combination of microwave irradiation and dilute alkali treatment is an alternative method for pretreatment of lignocellulosic materials at lower temperature. This pretreatment improves the enzymatic hydrolysis of biomass materials by accelerating the reactions during the pretreatment process compared with the conventional heating chemical pretreatment process (Zhu et al., 2006; Hu and Wen, 2008; Keshwani et al. 2007). While all aforementioned studies were intended for the

pretreatment of lignocellulosic biomass for further enzymatic hydrolysis or saccharification, they may be used or modified as a pretreatment for densification process. Therefore, the main objectives of this research were to investigate the effect of microwave pretreatment on densification process of canola straw grind and study the effect of different alkaline solutions for microwave pretreatment and applied compressive load on densification of pretreated canola straw.

MATERIALS AND METHODS

Sample preparation

Canola straw was acquired in small square bales with dimensions of 0.45×0.35×1.00 m from the Central Butte area of Saskatchewan, Canada in the summer of 2008. Before pretreatment of straw samples by microwave irradiation, they were ground using a hammer mill (Glen Mills Inc., Clifton, NJ) powered by a 1.5 kW electric motor with a screen opening size of 1.6 mm. The average moisture content of ground canola straw was 6.84% (wb). The moisture content of the ground samples was measured following the procedure mentioned in ASABE Standard S358.2 (ASABE, 2006), where the sample was oven-dried for 24 h at 103±2˚C.

Microwave pretreatment

Canola straw samples were pretreated in a domestic microwave oven made by (Panasonic Model NNC980W, Panasonic Canada Ltd, Mississauga, ON, Canada). The cavity dimensions of the microwave were 0.24×0.41× 0.42 m; it is equipped with a rotary device that rotates a 381 mm diameter circular ceramic plate. Twenty grams of ground canola straw sample was immersed in 180 g of alkali (1% (w/v) NaOH and 1% (w/v) $Ca(OH)_2$). The mixture was placed in a 600 mL beaker and positioned at the center of the rotating circular ceramic plate in the microwave oven for treatment at 713 W (full power). The mixture was exposed to microwave radiation until the moisture content of materials reached to appropriate level (12% w.b.) for densification process.

Chemical analysis

Chemical analysis of pretreated and untreated ground canola straw samples was performed at the Dept. of Animal and Poultry Science Laboratory (University of Saskatchewan, Saskatoon, SK, Canada). The analysis included the determination of crude protein, starch, lignin, acid detergent fibre (ADF), neutral detergent fibre (NDF), and total ash contents. Association of Official Analytical Chemists (AOAC) standard method 984.13 (AOAC, 2005a) was used to determine the crude protein content of the biomass and total starch content was measured using AOAC standard method 996.11 (AOAC, 1998). AOAC standard method 973.18 (AOAC, 2005b) was followed to determine lignin and ADF contents, whereas, NDF was determined using the method of Van Soest *et al.* (1991). The total ash content was evaluated using AOAC standard method 942.05 (AOAC, 1990). The percentage cellulose was calculated indirectly from percentage ADF and lignin (%ADF minus %lignin) in the same manner used by Mani et al. (2006). Hemicellulose percentage was also calculated indirectly from percentage NDF and ADF (% NDF minus %ADF) (Mani et al., 2006).

Bulk and particle density analysis

The bulk density of pretreated and untreated canola straw samples was calculated from the mass and volume of a standard cylindrical steel container (SWA951, Superior Scale Co. Ltd., Winnipeg, MB) with 0.5 L volume that was filled with the sample after passing through a funnel. Since the sample was fluffy and did not flow down readily through the funnel, a thin steel rod was used to clear the blockage in the funnel. After filling the cup, excess biomass material was

removed and the sample was not compacted in any way. A gas multi-pycnometer (QuantaChrome, Boynton Beach, FL) was used to determine the particle density of pretreated and untreated ground canola straw samples by calculating the displaced volume of nitrogen gas by a known mass of material. Five replicates were performed for both bulk and particle density measurements.

Experimental set-up and densification procedure

Pretreated and untreated canola straw samples were compressed and pelletized in a single pelleting unit. This unit consisted of a steel cylindrical die with 6.35 mm internal diameter and 125 mm internal chamber length and a plunger/piston connected to the upper moving crosshead of an Instron Model 1011 (Instron Corp.,Canton, MA) universal testing machine which provided the load necessary to compress the biomass samples. The die was equipped with a heating element which was wound around it. Two type-T thermocouples were also installed; one attached to a temperature controller which regulated the power input to the heater, and the other monitored and verified the temperature of the die wall. The cylindrical die was slip fitted into a stainless steel base with a hole matching the outer diameter of die. This steel base supported the whole assembly and allowed the plunger to move straight down with no lateral movement during compression process.

Approximately 0.5-0.6 g of pretreated or untreated of canola straw samples was loaded into the cylindrical die. The temperature of cylindrical die was regulated at 95±1˚C to simulate frictional heating during commercial pelleting process (Mani et al., 2006). Compressive force was applied using the Instron machine fitted with a 5000 N load cell and four pre-set loads of 1000, 2000, 3000 and 4000 N were used to compress the materials. The plunger compressed the sample using a crosshead speed of 50 mm/min. After compression and achieving the pre-set load, the plunger was stopped and held in position for 60 s, constituting the relaxation test. The force–deformation data during compression and the force–time data during stress relaxation were logged in the computer.

To calculate the specific extrusion energy after completion of relaxation test, the steel base was removed and another steel plate with a centrally located hole was replaced under the cylindrical die for support. The newly formed pellet was ejected from the die at 50 mm/min via load application using the plunger. During this time, the force-displacement data was recorded and specific extrusion energy was calculated based on the Mani et al. (2006) method. The compression test was replicated ten times for all samples.

Pellet density

The density of each newly formed pellet after ejection from the die was calculated after measuring its mass and volume. Pellet mass was measured using a digital scale and diameter and length of pellet were measured using a digital caliper to calculate the volume.

Tensile strength test

Diametral compression test was used to evaluate the tensile strength of the formed pellets from microwave pretreated and untreated canola straw samples which was used by Tabil and Sokhansanj (1997). Three layers of blotting paper were fastened to lower steel platen and upper steel plunger attached to Instron machine. Pellets were cut diametrically into tablets with a thickness of approximately 2 mm using a scalpel. An individual tablet was placed on its edge on the lower padded platen and compressed by upper plunger until failure occurred. The Instron was fitted with a 5000 N load cell and compressed the samples at a crosshead speed of 1 mm/min. The fracture type was very important to determine the correct tensile strength. Based on the Fell and Newton (1970), only fractures causing the tablets to break or crack in two halves along the loading

axis were accepted and other fracture modes were discarded. Upon failure, the fracture force was recorded and the tensile strength of tablets was calculated using following equation:

$$\delta_x = \frac{2F}{\pi dl} \quad (1)$$

where δ_x is tensile (horizontal) stress (Pa); F is load at fracture (N); d is tablet diameter (m) and l is tablet thickness (m). This test was replicated ten times for each sample.

RESULTS AND DISCUSSION

Chemical composition

Table 1 shows the effect of microwave-chemical pretreatment on the chemical composition of canola straw grind. The main components of ground canola straw are cellulose, lignin, and hemicellulose with 58.83, 13.24 and 11.92 %, respectively and protein, starch, and ash include slight segment of canola straw grind. Microwave-chemical pretreatment caused remarkable changes in lignocellulosic structure of canola straw grind that resulted in significant reduction in cellulose, hemicellulose, and lignin contents. It has been reported that alkali treatment dissolves hemicellulose and lignin of lignocellulosic materials and microwave irradiation accelerates this reaction (Jackson, 1977; Zhu et al., 2005; Kumar et al., 2009; Lesoing et al., 1980). The data enumerated in Table 1 shows this fact. It is obvious that canola straw samples pretreated by microwave-alkali technique particularly sodium hydroxide solution have less cellulose, hemicellulose, and lignin compared to untreated samples. Microwave-NaOH pretreatment was more effective than microwave-$Ca(OH)_2$ to hydrolyze and dissolve lignocellulosic components as reported by Lesoing et al. (1980) and Keshwani et al. (2007). While protein and starch of samples did not change significantly after microwave pretreatment, higher ash content was recorded in grind samples pretreated by microwave-alkali treatment compared to untreated samples. It was observed that samples pretreated with NaOH solution had higher ash content than those pretreated with $Ca(OH)_2$ solution.

Bulk and particle density of canola straw samples

Table 2 gives the bulk and particle densities of untreated and microwave pretreated canola straw grind samples. Canola straw grind samples pretreated by microwave-chemical technique resulted in higher bulk and particle densities than untreated one. Bulk and particle densities of canola straw grind increased from 151.30 to 160.35 kg/m^3 and from 1290.73 to 1367.33 kg/m^3, respectively after microwave-NaOH pretreatment and they increased to 153.68 kg/m^3 and 1342.26 kg/m^3, respectively after microwave-$Ca(OH)_2$ pretreatment. Bulk and particle densities of biomass pretreated by microwave-chemical method increased that is related to increasing of depolymerized components and ash content after pretreatment. Canola straw samples pretreated by microwave-NaOH resulted in higher bulk and particle densities compared to samples pretreated by microwave-$Ca(OH)_2$ that could be more favourable to hydrolysis. However, ash content of microwave-NaOH pretreated samples was the highest.

Pellet density

Table 3 shows the effect of microwave pretreatment and compressive load on density of canola straw grind pellets. Microwave-chemical pretreated canola straw grind revealed remarkably higher pellet density than untreated samples so that the pellet density of untreated canola pellet sample compressed at 4000 N increased from 992 kg/m^3 to as high as 1335 kg/m^3 after pretreatment using microwave-NaOH. Sodium hydroxide solution was more efficient compared to calcium hydroxide to

increase the initial density of the pellets. In general, it can be concluded that the pellet density of microwave-alkali pretreated canola straw is significantly greater than that of untreated biomass and this corresponds to higher particle density, more hydrolyzed components and ash contents. The density of all compacts significantly increased with an increase in applied compressive load from 1000 to 4000 N for untreated and pretreated biomass. For example, the pellet density of untreated and pretreated using microwave-NaOH increased from 749 to 992 kg/m^3 and from 1124 to 1335 kg/m^3, respectively when the compressive load increased from 1000 to 4000 N. An increase in compressive load results in plastic deformation of ground particles and consequently results in pellets that have densities closer to their respective particle densities.

Compression and extrusion energy

Table 3 shows the effect of microwave pretreatment and applied compressive load on specific energy required for compression and extrusion of untreated and pretreated canola straw. For all untreated and pretreated feedstocks, the compression, extrusion, and total specific energy significantly increased with an increase in applied compressive load. The results indicated that at any specific applied compressive load, biomass pretreated by microwave-NaOH needed less specific energy for compression. The specific energy required for compression of canola straw increased from 12.13 to 23.37 MJ/t and from 13.48 to 25.93 MJ/t, respectively for biomass pretreated by microwave-NaOH and microwave-$Ca(OH)_2$ and increased from 13.63 to 28.05 MJ/t for untreated biomass when the applied compressive load increased from 1000 to 4000 N. More specific energy was required to compress the canola straw samples than to extrude them for all pretreated and untreated samples. The data analysis showed that the effect of microwave-chemical pretreatment was significant on specific energy required for extrusion; more energy was needed to extrude the samples pretreated using microwave-NaOH compared to untreated samples or those pretreated by microwave- $Ca(OH)_2$. Generally, the results revealed that the total energy required for compression and extrusion of canola straw samples pretreated by microwave-$Ca(OH)_2$ was less than untreated samples and samples pretreated by microwave-NaOH required more energy.

Tensile strength of pellets

Table 4 shows the results of diametral compression of pellets formed from untreated and microwave pretreated canola straw grind at different applied compressive loads. Fracture load and tensile strength significantly increased with an increase in applied pressure from 1000 to 4000 N for all untreated and pretreated feedstocks. At any specific compressive load, microwave-chemical pretreatment significantly increased fracture load and tensile strength of pellets formed from canola straw. For example, the tensile strength of untreated pellets formed at 4000 N was 0.62 MPa compared to 4.47 MPa for pellets formed after pretreatment of canola straw grind by combination of microwave and 1% NaOH solution. Canola straw grind samples pretreated by microwave-$Ca(OH)_2$ showed higher tensile strength than untreated samples but not higher than the samples pretreated by microwave-NaOH. This means that $Ca(OH)_2$ solution is not strong as NaOH solution to disintegrate the structure of lignocellulosic materials or depolymerize/dissolve the components involved in binding.

Conclusions

The following conclusions can be drawn from this study:

1. Canola straw grind samples pretreated by microwave/alkali technique had lower cellulose, hemicellulose, and lignin content than untreated samples.

2. The solubilization of cellulose, hemicellulos,e and lignin of canola straw samples increased after microwave/alkali pretreatment.
3. The ash content of canola grind samples pretreated by microwave/alkali technique, in particular pretreated by NaOH solution, increased significantly.
4. Canola straw grind samples pretreated by microwave/chemical technique had significantly higher bulk and particle densities than untreated sample.
5. The density values of pellets was as low as 992 kg/m^3 for untreated canola grind pellets to as high as 1335 kg/m^3 in pellet samples pretreated by a combination of microwave and NaOH.
6. The total energy required for compression and extrusion of canola straw grind samples pretreated by microwave-NaOH was higher than untreated samples.
7. The tensile strength of untreated pellets was very low (0.62 MPa) compared to the high tensile strength of microwave/alkali pretreated pellets (4.47 MPa).

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Table 1. Chemical composition of untreated and microwave pretreated of canola straw grind.

Composition (%)[a]	Untreated	Microwave pretreated	
		NaOH 1%	$Ca(OH)_2$ 1%
Protein	2.10	1.91	1.84
Starch	3.70	3.02	3.62
Ash	1.91	13.98	8.09
Cellulose	58.83	46.73	47.49
Hemicellulose	11.92	7.48	8.80
Lignin	13.24	12.92	12.79

[a] dry matter

Table 2. Bulk and particle densities of untreated and microwave pretreated of canola straw grind at moisture content of 12% (wb).

Physical properties	Untreated	Microwave pretreated	
		1% NaOH	1% $Ca(OH)_2$
Bulk density (kg/m^3)	151.30	160.35	153.68
Particle density (kg/m^3)	1290.73	1367.33	1342.26

Table 3. Effect of microwave pretreatment on pellet density and specific energy required for densification of canola straw grind.

Biomass	Applied load (N)	Pellet density (kg/m^3)	Specific energy (MJ/t) for compression	Specific energy (MJ/t) for extrusion	Total specific energy (MJ/t)
Untreated	1000	748.96	13.63	2.40	16.03
	2000	874.59	19.93	3.55	23.48
	3000	951.31	23.87	3.77	27.64
	4000	992.12	28.05	3.99	32.04
Microwave/NaOH (1%)	1000	1124.63	12.13	7.83	20.06
	2000	1254.91	16.29	8.83	25.12
	3000	1306.68	19.60	8.37	27.97
	4000	1335.47	23.37	10.24	33.61
Microwave/$Ca(OH)_2$ (1%)	1000	963.36	13.48	1.61	15.09
	2000	1062.99	15.41	2.04	17.45
	3000	1112.04	21.01	1.74	22.75
	4000	1167.58	25.93	2.29	28.22

Table 4. Effect of microwave pretreatment on fracture load and tensile strength of canola straw grind pellets

Biomass	Applied load (N)	Tablet thickness (mm)	Tablet diameter (mm)	Fracture load (N)	Tensile strength (MPa)
Untreated	1000	2.79	6.47	3.12	0.11
	2000	2.90	6.49	8.24	0.28
	3000	3.19	6.52	13.38	0.41
	4000	2.93	6.55	18.74	0.62
Microwave/NaOH (1%)	1000	2.41	6.54	48.00	1.95
	2000	2.47	6.55	66.25	2.60
	3000	2.40	6.54	96.50	3.95
	4000	2.40	6.55	109.20	4.47
Microwave/$Ca(OH)_2$ (1%)	1000	2.42	6.53	16.70	0.69
	2000	2.47	6.49	19.89	0.79
	3000	2.39	6.54	26.15	1.07
	4000	2.38	6.52	31.25	1.28