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## **Effect of Fuel Additives on Agricultural Straw Pellet Quality**

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**ABSTRACT** An investigation was conducted to determine the effect of different levels of AK2, a fuel additive that reduces ash fusion for agricultural biomass, on the physico-chemical properties of biomass pellets. Three different biomass straws namely, barley, oat and wheat were ground at two hammer mill screen sizes of 0.8 and 1.6 mm. Each ground biomass sample was mixed with three levels of AK2, 0.05, 0.10 and 0.15% by mass and also a blank (no AK2) was set aside for comparison. Pellets were made using single-pelleting unit at a pre-set load of 4400 N corresponding to a pressure of 138.9 MPa. Physical quality of pellets were determined by measuring pellet density, relaxed density, durability and the specific energy required to make a pellet. Pellets having higher durability values (74-88%) were obtained from ground straw at hammer screen size of 0.8 mm and AK2 level of 0.15% compared to other treatments. Carbon, hydrogen, nitrogen and sulfur content of blank pellets and those with 0.15% AK2 at hammer screen size of 0.8 mm were determined. Pellets made with 0.15% AK2 at hammer screen size of 0.8 mm, manufactured by pilot-scale pellet mill, were gasified and the tar content was determined. The tar content of pellets with 0.15% AK2 was significantly lower than blank pellets.

**Keywords:** Biomass, Biofuels, Slag, Foul, Pelleting, Fuel additive.

## INTRODUCTION

Biomass is a renewable source of energy and is carbon neutral, since biofuel helps in diminishing greenhouse gas emission (Sultana et al., 2010). Agricultural biomass, such as wheat, barley, oat and flax straw, has been considered as feedstock for conversion to biofuel, chemicals, electricity and heat. During the last few years, production of biofuel pellets has grown rapidly in North America, China and Europe, especially Sweden (Samuelsson et al., 2009). Canada exports biofuel pellets (wood pellets) to Europe and the biofuel pellet processing industry has expanded in Canada in the last few years. In the Canadian prairies, biofuel pellets may be produced from wheat, barley, oat and flax straw residue which according to estimates may be over 15 Mt (Sokhansanj et al., 2006).

Inherently, biomass has low bulk density, has irregular shape and size which makes it difficult to handle, transport, store and utilize in its original form. Therefore, an efficient solution is to densify low bulk density (40-200 kg/m<sup>3</sup>) biomass straw from loose or bale form to pellet and cubes with higher bulk density (600-800 kg/m<sup>3</sup>) (Kaliyan and Morey, 2009). Since biofuel pellets are transported over long distances and are handled and stored before combustion, durable and stable pellets are desired. Durability and stability of biomass pellets are affected by many factors including feedstock composition and characteristics (starch, protein, fiber, fat, lignin, moisture content and particle size), pre-conditioning processes (steam conditioning/preheating and addition of binders), and parameters for densification (forming pressures, pellet mill and roll press variables) (Serrano et al., 2010).

The resulting biomass pellet is subjected to thermo-chemical conversion process to generate energy. During this process, the organic compounds in biofuel pellet are gasified and usually the inorganic species remain as salt and form ash containing CaO, K<sub>2</sub>CO<sub>3</sub>, MgO, etc. (Mahmoudkhani et al., 2007). Silicon and potassium are the main ash forming elements. Compared to other biomass fuels, herbaceous biomass (cereal straws, grasses, etc.) fuels have high content of chlorine resulting to ash deposition problems during moderate or high thermo-chemical conversion temperatures (Jenkins et al., 1998). Herbaceous biomass also have high amount of alkali metals resulting in slag formation and fouling, which create problem on the burners (Bruun et al., 2010). Nielson and co-workers (2010) reported that the major problem of agricultural (herbaceous) biomass compared to woody materials is their high ash content, the lower ash softening temperature and the higher risk of corrosion and fouling.

The industry collaborator of this project has a patented technology or fuel additive called AK2 that has the potential to reduce slag and clinker formation during thermo-chemical conversion process. However, the effect of adding AK2 on the quality of pellets from agricultural biomass has not been explored, yet. Therefore, the objective of this study was to densify ground barley, oat and wheat straw having various levels of AK2 in a single pelleting to determine its effect on pellet density and durability, and perform ultimate analysis to determine their elemental composition. Pelleting of the optimal mixture of ground biomass and AK2 was conducted in a pilot-scale pellet mill to determine the effect of AK2 additive on the durability of biomass pellets.

## MATERIAL AND METHODS

**Biomass Samples** Barley, oat and wheat straws were obtained in small square bales from a farmer in the Central Butte area of Saskatchewan, Canada in the summer of 2008. All samples were chopped using a chopper equipped with six blades which were mounted at a shearing angle of 14° and rotated at 460 rpm. The chopper was fabricated in the Bioprocessing Lab, Department of Chemical and Biological Engineering, University of Saskatchewan, Canada. The chopped samples were then ground using a hammer mill (Serial no. 6M13688; Glen Mills Inc., Maywood, NJ) using hammer mill screen sizes of 1.6 and 0.8 mm.

**Sample Preparation and Densification in the Single Pelleting Unit** The required amount of water was calculated by mass balance between the original ground sample and the desired sample moisture content of 10%. The sample was re-moistened by adding the required water, mixing it in an air-tight bag. Samples were stored in a cold room at 4°C and mixed every 12 h for at least 72 h to ensure moisture equilibration. The AK2 additive, obtained from Evergreen Biofuels Inc. (Montreal, QC), was mixed with moisture-adjusted straw grinds at 0% as blank, 0.05, 0.10, and 0.15% by mass. Each sample mixture was placed in a bucket with a closed lid and was blended in a rotating cement mixer for about 2 h to provide a uniform distribution of AK2 in the straw grinds. Subsequently, the sample mixtures were stored in air-tight bucket at 4°C.

The ground straw-AK2 samples were pelleted in a single-pelleting unit as shown by Kashaninejad and Tabil (2011) and also used in previous studies (Tabil and Sokhansanj, 1996, 1997; Adapa et al., 2002; Mani et al., 2006; Shaw et al., 2009). The device is composed of a plunger-die assembly having a steel cylinder with internal diameter and length of 6.35 mm and 125 mm, respectively, and a plunger mounted to the upper moving crosshead of Instron testing machine (Model 3360 Dual Column Tabletop Testing Systems, Instron Corp. Norwood, MA) fitted with a 5000 N load cell. The die was wrapped with a heating element maintaining the temperature at 95±1°C to simulate frictional heating in commercial pelleting (Adapa et al., 2006; Mani et al., 2006; Kashaninejad and Tabil, 2011). The cylindrical die sat on a raised base equipped with sliding gate at the bottom. On the base, there is a hole allowing the densified sample to be discharged from the die when the sliding gate is opened. Moisture-adjusted biomass grind-AK2 mixture (0.5-0.6 g) was loaded into the die when the temperature of the pelleting unit was stable (95±1°C). The compressive force was applied to densify the samples using the Instron machine having a pre-set load of 4400 N corresponding to a pressure of 138.9 MPa and crosshead speed of plunger was set at 50 mm/min. When the compression load achieved the pre-set load, the plunger stopped and was retained in place for 60 s for the relaxation stage (Kashaninejad and Tabil, 2011) and also to avoid spring-back of biomass sample being compressed (Mani et al., 2006). The plunger was then moved up to release the compression force, the sliding gate was opened, the plunger moved down after 30 s to eject the pellet through the bottom of die and base. The force-deformation and force-time data during compression and relaxation were logged in the computer. Compression energy was

calculated by integration of the area under the force-displacement curve using the Bluehill software (Version 2.12, Illinois Tool Works, Inc., 2010) and converted to specific energy values in MJ/t by dividing it by the pellet mass. The specific energy calculations did not include the energy consumed for milling and for operating the Instron testing machine. The specific energy was determined in ten replicates.

**Particle Size Analysis, Bulk Density, Ash and Moisture Content** The geometric mean diameter of ground straw samples was determined using ANSI/ASAE standard S319.4 FEB2008 (ASABE, 2008). A Ro-Tap sieve shaker (W.S. Tyler Inc., Mentor, OH) was used for particle size analysis using U.S. sieve numbers 16, 20, 30, 50, 70 and 100 (sieve opening sizes: 1.190, 0.841, 0.595, 0.297, 0.210 and 0.149 mm, respectively). The sieve series selected were based on the range of particles in the samples. The sieves were placed on a Ro-Tap sieve shaker for 10 min sieve shaking time. The geometric mean diameter ( $d_{gw}$ ) and geometric standard deviation ( $S_{gw}$ ) were calculated in three replicates for each ground straw sample.

Bulk density of ground straw samples was determined using a 0.5-L cylindrical container (SWA951, Superior Scale Co. Ltd., Winnipeg, MB) filled using a funnel, with its discharge opening located 55 mm above the top edge of the container. The funnel was removed from top of the container; the container was tapped on a wooden table for approximately 10 times to allow the material to settle down. The container was leveled by rolling a cylindrical stainless steel bar across the container in two perpendicular directions. The container was then weighed. The mass per unit volume gave the bulk density of the biomass grind in  $\text{kg/m}^3$ . The bulk density was determined in three replicates for each sample.

The total ash content was determined in duplicate using AOAC standard method 942.05 (AOAC, 1990), where 2-3 g of sample was burned in furnace at 600°C and the remaining ash was determined. The moisture content of ground straws was determined in duplicate using AACC standard 44-15A (AACC, 2005), where 2-3 g of material was oven-dried at 130°C for 90 min in duplicates. The required amount of water was calculated by mass balance between the original ground sample and the sample with 10% moisture content. The sample was re-moistened by adding required water and mixed in an air-tight bag. Samples were stored in a cold room at 4°C and mixed every 12 h for at least 72 h to ensure moisture equilibration.

**Pellet Density and Relaxed Density** Length, diameter, and mass of newly formed pellets were measured using a digital caliper to calculate the initial pellet density. Each pellet was stored in air-tight bag individually at room temperature. The diameter, length, and mass of pellets were determined again two weeks after compression to calculate the relaxed density ( $\text{kg/m}^3$ ) and determine the stability of the pellets. Pellet density and relaxed density were determined in ten replicates.

**Pellet Durability** Durability of pellets made by the single pelleting unit was measured in ten replicates using the drop test method (Al-Widyan and Al-Jalil, 2001; Khankari et al., 1989; Sah et al., 1980; Shrivastava et al., 1989), where a single pellet was dropped from a height of 1.85 m on a metal plate. The ratio of the weight of the larger portion of the pellet retained intact to the initial weight of pellet was expressed as the percentage durability of the pellet. Durability of pellets made by pilot-scale pellet mill was measured following the ASABE Standard S269.4 DEC1991 (R2007) (ASABE, 2007). Pellets (100 g) were placed in a dust-tight chamber and tumbled for 10 min at 50 rpm. Fine and broken pellets were separated from coarse ones using a sieve with hole opening of 7.93 mm weighed to determined percentage of broken pellets respect to the initial pellet weigh during tumbling, as durability value.

**Elemental Analysis of Biomass Samples** The carbon and hydrogen composition of each dried product was determined by elemental analysis. The ground biomass (4-6 mg) was loaded in tin capsules (12 x 4 x 4 mm) (Isomass Scientific, Calgary, AB) that were subsequently loaded in the CHNS elemental analyzer (Elementar Vario ELIII made by Elementar Americas, Mt. Laurel, NJ). Samples were subjected to combustion and the exhaust gases were quantified by thermal conductivity. The analyzer was calibrated with three blanks, three runins (sulfanilic acid ran as unknowns) and three sulfanilic acid samples (4-6 mg) with an analysis error within  $\pm 2\%$  (Kamburska and Fonda-Umani, 2009).

**Pilot Scale Pelleting** For each biomass grind, an experimental treatment combination made by the single-pelleting unit with the highest durability was selected to make pellets using the pilot-scale pellet mill. The pilot-scale CPM CL-5 pellet mill (California Pellet Mill Co., Crawfordsville, IN) was used for processing of biomass grinds into pellets. The pellet mill consisted of a corrugated roller ( $d = 85.0$  mm) and ring die assembly. The diameter of ring die was 190.5 mm with thickness of 32 mm. The pelleting die had internal diameter of 126.5 mm. The pellet die hole diameter and l/d ratio were 8.0 mm and 4.0, respectively. The rotational speed of the pellet mill was 250 rpm.

The moisture content of biomass grinds (2 kg) was adjusted to 10% and the required amount of AK2 was added and mixed, similar to sample preparation for single-pelleting experiments. The mixture was fed to the pellet mill and passed through the steam conditioner located above the pellet die assembly to be conditioned with steam at 235-250 kPa gage prior to pelleting (Thomas et al., 1997). Since biomass grinds have low bulk density and poor flowability, the pellet mill blocked very often before any consistent pellet production was achieved. Therefore, the injected steam was increased gradually to obtain consistent pellet production through the die. Pellets were cooled down by spreading on a paper sheet at lab ambient temperature. Once cooled, the pellets were stored in plastic bag for further tests.

**Gasification** The experiments were carried out at atmospheric pressure in two-stage fixed bed reactor system. The first stage reactor (10.5 mm ID x 500 mm length) and second stage reactor (10.5 mm ID x 370 mm length) were made of Inconel tubing. First stage reactor was loaded with pre-weighed quantity (1.5-2.0 g) of pellet sample. Silica sand was used to form a 70 mm high packed bed in second stage reactor. The temperature was measured and controlled using K-type thermocouple placed at the heating zone in the furnace and connected to temperature controller (Eurotherm model 2132, USA Eurotherm Controls Inc., Reston, VA). Argon used as the inert carrier gas at flow rate of 44 ml/min. The experimental parameters (750°C and 0.4 ER) were selected based on pre-optimized conditions using biomass in the laboratory. When the second reactor attained the final temperature 750°C, heating of first reactor was started. Both the reactors were heated to the same final temperature at the same heating rate of 25°C/min. Injection of gasifying agent (steam) was started once the first reactor reached to 250°C and collection of product gas was started. The injection of gasifying agent (steam) and collection of product gases were then carried out for the next 60 min. After 60 min, injection of gasifying agent (steam) was stopped and flow of argon continued for cooling the reactors. The volume of gas collected was measured at  $25 \pm 2^\circ\text{C}$  and 1 atm pressure conditions. Gas samples were collected in Tedlar bags and were analyzed by gas chromatography. Char remaining in the gasifier were collected and weighed. Tar was collected in condenser placed in ice bath and gaseous product was collected over solution of sodium chloride (17%). After cooling down the reactor, the system was washed with acetone to remove the

remaining tar. Tar was collected by evaporating acetone using rotary vacuum evaporator and measured for its content.

**Statistical Analysis** The effect of biomass type, hammer mill screen size, and AK2 level on the compaction characteristics of biomass grinds was determined using a completely randomized experimental design with factorial treatment. There were three variable factors, the biomass type (barley, oat, and wheat), the hammer mill screen size (0.8 and 1.6 mm) and AK2 level (0.00, 0.05, 0.10, and 0.15%). Analysis of variance (ANOVA) and comparison of means (Duncan's multiple range test at  $P = 0.05$ ) were performed using the Statistical Analysis System (Version 9.2, SAS Institute Inc., Cary, NC) by the GLM procedure to evaluate the effect of each variable and their interactions.

## RESULTS AND DISCUSSION

**Particle Size and Bulk Density** Table 1 shows geometric mean diameter of samples ranging from 0.29 mm for hammer mill screen of 0.8 mm to 0.45 mm for hammer mill screen of 1.6 mm. There were some variations in geometric mean diameter of samples ground within the same hammer mill screen size. That was related to the variation in moisture content of samples and also difference in mechanical properties of samples (Mani et al., 2006). As the particle size decreased, the bulk density increased (Table 1), which was in agreement with the results of Mani and co-workers (2006). Oat straw grinds had the highest and wheat straw grinds had the lowest bulk density in the corresponding hammer mill screen sizes.

**Pellet Density and Relaxed Density** The effect of biomass type, hammer mill screen size, AK2 level, and the interaction effects of biomass type and hammer mill screen size as well as that of biomass type, hammer screen size and AK2 level were significant ( $P < 0.01$ ) on pellet density and relaxed density (Table 2). The pellet density from oat straw grinds was higher than that from wheat and barley straw grinds (Table 3). The pellet density, in majority of treatments, was higher in hammer mill screen size of 0.8 mm. The pellet density increased as AK2 level increased; the highest density was observed in samples containing 0.15% AK2 and the lowest density was obtained from blank pellets (containing 0% AK2).

The relaxed density of pellets was in the following order wheat > barley > oat. The relaxed density was lower in hammer mill screen size of 0.8 mm. All oat and wheat straw pellets from grinds of 0.8 mm hammer mill screen size expanded in diameter and length and as a result, their density decreased after two weeks. Wheat and barley straw pellets from grinds of 1.6 mm hammer mill screen size showed higher density after two weeks which was in agreement with Kashaninejad and Tabil's (2011) work. This phenomenon was related to the effect of heat on lignin compound during densification. Lignin may have been melted by heat during densification with consequent thermosetting properties having irreversible hardness.

**Durability** The effect of biomass type, hammer mill screen size and their interaction was significant on durability ( $P < 0.01$ ) of pellets made in the single pelleting unit. Oat and barley straw pellets had higher durability than wheat straw pellets. The highest durability was observed in barley and oat straw pellets from grinds of hammer mill screen size of 0.8 mm and AK2 levels of 0.10 and 0.15%, respectively. The AK2 level did not significantly affect the durability of pellets. As a result, biomass grinds from hammer mill screen size of 0.8 mm would be able to make durable pellets.

Table 1. Geometric mean diameter ( $d_{gw}$ )<sup>a</sup> and bulk density<sup>b</sup> of ground straw samples

Straw sample	Hammer mill screen size (mm)	$d_{gw}$ (mm)	Bulk density (kg/m <sup>3</sup> )
Barley	0.8	0.370±0.001	180±2
	1.6	0.456±0.004	155±1
Oat	0.8	0.307±0.008	199±1
	1.6	0.404±0.014	196±4
Wheat	0.8	0.361±0.003	163±8
	1.6	0.452±0.016	154±2

<sup>a</sup> n = 3, Geometric mean diameter ± geometric standard deviation

<sup>b</sup> n = 3, Mean ± standard deviation

Table 2. Effect of biomass type (S), mill screen size (Z) and AK2 level (K) on pellet density, pellet relaxed density, and durability of biomass pellets made in the single pelleting unit

Source of variation	DF	Pellet density		Relaxed density		Durability	
		SS	P-value	SS	P-value	SS	P-value
S	2	44316.26	<0.01	200772.13	<0.01	6731.01	<0.01
Z	1	10307.87	<0.01	291566.95	<0.01	3168.27	<0.01
K	3	22709.94	<0.01	175522.63	<0.01	392.97	0.52
S × Z	2	35283.81	<0.01	167349.14	<0.01	1554.07	0.01
S × K	6	10915.66	0.14	232313.90	<0.01	3593.03	<0.01
Z × K	3	22997.56	<0.01	129597.99	<0.01	994.51	0.13
S × Z × K	6	20326.27	<0.01	209198.97	<0.01	1521.49	0.20
Residuals	216	243239.68	---	299158.37	---	37702.06	---
Total	239	410097.04	---	1705480.08	---	55657.41	---

DF: degrees of freedom, SS: Sum of squares, P: probability

Table 3. Pellet density ( $\rho_p$ ), relaxed density ( $\rho_r$ ), durability, specific energy required for densification (SE), and ash content of pellet samples made at different mill screen sizes (MSS) and AK2 levels (mean  $\pm$  standard deviation)

Sample	MSS (mm)	AK2 level (%)	Peak load (N)	$\rho_p$ (kg/m <sup>3</sup> )	$\rho_r$ (kg/m <sup>3</sup> )	Durability (%)	SE (MJ/t)	Ash (%)
Barley	0.8	0.00	4512 $\pm$ 5	1063 $\pm$ 33 <sup>abcde</sup>	1059 $\pm$ 35 <sup>de</sup>	80 $\pm$ 12 <sup>abc</sup>	29.9 $\pm$ 5.5 <sup>bcde</sup>	3.29 $\pm$ 0.04 <sup>q</sup>
		0.05	4517 $\pm$ 6	1053 $\pm$ 32 <sup>bcdef</sup>	1044 $\pm$ 26 <sup>de</sup>	85 $\pm$ 14 <sup>a</sup>	29.1 $\pm$ 3.2 <sup>cde</sup>	6.12 $\pm$ 0.07 <sup>jk</sup>
		0.10	4519 $\pm$ 2	1080 $\pm$ 21 <sup>abc</sup>	1067 $\pm$ 19 <sup>de</sup>	88 $\pm$ 6 <sup>a</sup>	32.0 $\pm$ 4.3 <sup>bcde</sup>	6.42 $\pm$ 0.03 <sup>f</sup>
		0.15	4566 $\pm$ 5	1040 $\pm$ 28 <sup>def</sup>	1039 $\pm$ 29 <sup>f</sup>	85 $\pm$ 12 <sup>a</sup>	25.4 $\pm$ 6.4 <sup>de</sup>	6.74 $\pm$ 0.07 <sup>d</sup>
	1.6	0.00	4503 $\pm$ 5	971 $\pm$ 30 <sup>g</sup>	981 $\pm$ 38 <sup>de</sup>	62 $\pm$ 19 <sup>ef</sup>	28.0 $\pm$ 4.6 <sup>cde</sup>	5.71 $\pm$ 0.04 <sup>mn</sup>
		0.05	4507 $\pm$ 6	1042 $\pm$ 23 <sup>def</sup>	1048 $\pm$ 33 <sup>a</sup>	78 $\pm$ 10 <sup>abcd</sup>	32.3 $\pm$ 6.3 <sup>bcde</sup>	5.63 $\pm$ 0.01 <sup>no</sup>
		0.10	4518 $\pm$ 6	1036 $\pm$ 34 <sup>def</sup>	1327 $\pm$ 69 <sup>de</sup>	78 $\pm$ 10 <sup>abcd</sup>	33.5 $\pm$ 5.8 <sup>bcd</sup>	5.57 $\pm$ 0.07 <sup>o</sup>
Oat	0.8	0.00	4501 $\pm$ 3	1051 $\pm$ 21 <sup>cdef</sup>	1049 $\pm$ 14 <sup>de</sup>	80 $\pm$ 18 <sup>abc</sup>	30.5 $\pm$ 6.4 <sup>bcde</sup>	3.17 $\pm$ 0.03 <sup>r</sup>
		0.05	4549 $\pm$ 3	1037 $\pm$ 77 <sup>def</sup>	1028 $\pm$ 73 <sup>e</sup>	81 $\pm$ 14 <sup>ab</sup>	34.9 $\pm$ 7.7 <sup>bc</sup>	6.35 $\pm$ 0.05 <sup>fg</sup>
		0.10	4603 $\pm$ 3	1091 $\pm$ 59 <sup>a</sup>	1062 $\pm$ 30 <sup>de</sup>	80 $\pm$ 15 <sup>abc</sup>	28.0 $\pm$ 5.0 <sup>cde</sup>	6.27 $\pm$ 0.01 <sup>gh</sup>
		0.15	4502 $\pm$ 3	1070 $\pm$ 22 <sup>abcd</sup>	1065 $\pm$ 21 <sup>de</sup>	88 $\pm$ 19 <sup>a</sup>	34.4 $\pm$ 5.9 <sup>bc</sup>	6.56 $\pm$ 0.05 <sup>e</sup>
	1.6	0.00	4504 $\pm$ 2	1067 $\pm$ 19 <sup>abcd</sup>	1067 $\pm$ 22 <sup>de</sup>	87 $\pm$ 9 <sup>a</sup>	29.0 $\pm$ 5.6 <sup>cde</sup>	3.86 $\pm$ 0.05 <sup>p</sup>
		0.05	4504 $\pm$ 3	1087 $\pm$ 22 <sup>ab</sup>	1072 $\pm$ 25 <sup>d</sup>	85 $\pm$ 11 <sup>a</sup>	29.0 $\pm$ 4.4 <sup>cde</sup>	6.24 $\pm$ 0.02 <sup>hi</sup>
		0.10	4504 $\pm$ 3	1091 $\pm$ 20 <sup>a</sup>	1075 $\pm$ 19 <sup>d</sup>	83 $\pm$ 11 <sup>ab</sup>	27.9 $\pm$ 4.9 <sup>cde</sup>	6.06 $\pm$ 0.05 <sup>ijkl</sup>
Wheat	0.8	0.00	4506 $\pm$ 5	1064 $\pm$ 9 <sup>abcde</sup>	1060 $\pm$ 12 <sup>de</sup>	83 $\pm$ 14 <sup>ab</sup>	25.7 $\pm$ 2.3 <sup>de</sup>	7.12 $\pm$ 0.06 <sup>c</sup>
		0.05	4506 $\pm$ 3	1053 $\pm$ 19 <sup>bcdef</sup>	1046 $\pm$ 19 <sup>de</sup>	65 $\pm$ 8 <sup>def</sup>	24.8 $\pm$ 0.9 <sup>e</sup>	7.28 $\pm$ 0.02 <sup>b</sup>
		0.10	4507 $\pm$ 4	1062 $\pm$ 13 <sup>abcde</sup>	1059 $\pm$ 14 <sup>de</sup>	79 $\pm$ 13 <sup>abc</sup>	26.3 $\pm$ 4.4 <sup>de</sup>	7.34 $\pm$ 0.04 <sup>b</sup>
		0.15	4510 $\pm$ 3	1057 $\pm$ 24 <sup>abcde</sup>	1063 $\pm$ 21 <sup>de</sup>	74 $\pm$ 10 <sup>abcde</sup>	25.5 $\pm$ 3.3 <sup>de</sup>	7.91 $\pm$ 0.07 <sup>a</sup>
	1.6	0.00	4485 $\pm$ 3	1021 $\pm$ 25 <sup>f</sup>	1191 $\pm$ 59 <sup>bc</sup>	70 $\pm$ 15 <sup>bcde</sup>	43.5 $\pm$ 25.5 <sup>a</sup>	6.67 $\pm$ 0.07 <sup>d</sup>
		0.05	4488 $\pm$ 3	1029 $\pm$ 25 <sup>ef</sup>	1214 $\pm$ 29 <sup>b</sup>	64 $\pm$ 17 <sup>ef</sup>	35.8 $\pm$ 5.6 <sup>bc</sup>	5.98 $\pm$ 0.05 <sup>l</sup>
		0.10	4487 $\pm$ 3	1034 $\pm$ 19 <sup>def</sup>	1219 $\pm$ 44 <sup>b</sup>	67 $\pm$ 13 <sup>cdef</sup>	37.5 $\pm$ 12.1 <sup>ab</sup>	6.15 $\pm$ 0.07 <sup>ij</sup>
		0.15	4487 $\pm$ 3	1037 $\pm$ 32 <sup>def</sup>	1175 $\pm$ 39 <sup>c</sup>	56 $\pm$ 14 <sup>f</sup>	32.8 $\pm$ 6.1 <sup>bcde</sup>	6.06 $\pm$ 0.04 <sup>ijkl</sup>

$\rho_p$  = Pellet density;  $\rho_r$  = Relaxed density; \*Mean values with the same letter are not significantly different at P = 0.05

**Specific Energy for Making Pellet** As shown in Table 4, the effect of screen size was significant ( $P < 0.01$ ) on specific energy. Pellets made from biomass grinds of hammer mill screen size of 1.6 mm required significantly higher specific energy than pellets from 0.8 mm hammer mill screen size. It could be due to the changes in the lignocellulosic components and distribution that happened by grinding. The highest specific energy was observed in wheat at screen size of 1.6 mm with 0.00 and 0.10% AK2 (Table 3). Specific energy did not change in a specific pattern in different samples and by increasing AK2 level.

**Total Ash Content** The effect of biomass type, hammer mill screen size, AK2 level, and their interaction effect was significant ( $P < 0.01$ ) on ash content. The highest ash content was obtained in pellets made from wheat followed by barley and oat straw grinds (Table 3). As AK2 level increased, the total ash content increased. The highest ash content was observed in wheat straw pellets of grinds from 0.8 mm hammer mill screen size and 0.15% AK2 level and the lowest was determined in blank oat straw pellets with 0.00% AK2.

Table 4. Effect of sample type (S), mill screen size (Z) and AK2 level (K) on specific energy required for densification and total ash content of biomass

Source of variation	DF	Specific energy		Total ash content	
		SS	P-value	SS	P-value
S	2	97.84	0.42	15.43	<0.01
Z	1	883.00	<0.01	1.94	<0.01
K	3	28.28	0.92	17.82	<0.01
S × Z	2	2509.55	<0.01	3.80	<0.01
S × K	6	626.24	0.09	12.42	<0.01
Z × K	3	33.18	0.90	7.01	<0.01
S × Z × K	6	1031.63	<0.01	2.51	<0.01
Residuals	216	12163.10	---	0.06	---
Total	239	17372.86	---	60.98	---

DF: degree of freedom, SS: Sum of squares, P: probability

**Elemental Analysis and Durability of Pellets from the Pilot-Scale Pellet Mill** Table 5 presents the chemical composition of blank pellets and pellets containing 0.15% AK2. The analysis was conducted for pellets made from biomass ground by hammer mill of screen size of 0.8 mm. This information is required for running biomass pellets in a gasifier to determine slag and gas formation. In all samples, the carbon, hydrogen, nitrogen, and sulfur contents (except for sulfur content of wheat, which could be attributed to number of replications) in pellets containing AK2 was lower than blank pellets. It was related to composition of AK2. It is likely that AK2 has lower carbon, hydrogen and nitrogen than ground biomass. When AK2 contributed to chemical composition of mixture, the ratio of carbon, hydrogen and nitrogen would be lower than blank samples. Figure 1 shows the biomass pellets with and without AK2 manufactured in the CPM CL-5 pilot-scale pellet mill. Durability of pellets made by the pilot-scale pellet mill is shown in Table 5. Durability of barley pellets increased significantly, 1.7 times, when AK2 was added. Slight increase was also observed in oat pellets containing AK2. However, wheat pellets durability was reduced when AK2 was added, although the reduction was not statistically significant. This trend was similar to that obtained by drop test. The pellet durability test results showed that AK2 did not adversely affect the durability of biomass pellets, and may have even improved durability of barley and oat straw pellets.

**Gasification of Pellets** Table 5 also shows the tar content obtained from gasification of pellets made from biomass ground at hammer mill screen size of 0.8 mm including blank pellets and pellets containing 0.15% AK2. Addition of AK2 resulted in the reduction of tar content compared to the blank pellet as claimed by manufacturer, Evergreen Biofuels Inc. Oat had the highest reduction in tar content (3.8%) followed by wheat (1.6%) and barley (1.2%).

Table 5. Elemental composition (%), and durability of pellets, and tar content of gasified pellets made from the pilot-scale pellet mill using biomass ground at hammer mill screen size of 0.8 mm.

Sample	AK2 level (%)	Carbon	Hydrogen	Nitrogen	Sulfur	Durability* (%)	Tar (%)
Barley	0.00	43.61	6.38	2.62	0.26	57±2 <sup>e</sup>	13.6
	0.15	39.74	5.82	1.13	0.14	97±0 <sup>a</sup>	12.4
Oat	0.00	41.81	6.14	1.54	0.42	71±1 <sup>d</sup>	12.9
	0.15	39.08	5.75	0.65	0.21	78±2 <sup>c</sup>	9.1
Wheat	0.00	40.50	5.95	0.72	0.11	93±2 <sup>b</sup>	11.8
	0.15	39.42	5.80	0.72	0.14	90±1 <sup>b</sup>	10.2

\* n=3, mean ± standard deviation. Mean values with the same letter are not significantly different at P = 0.05

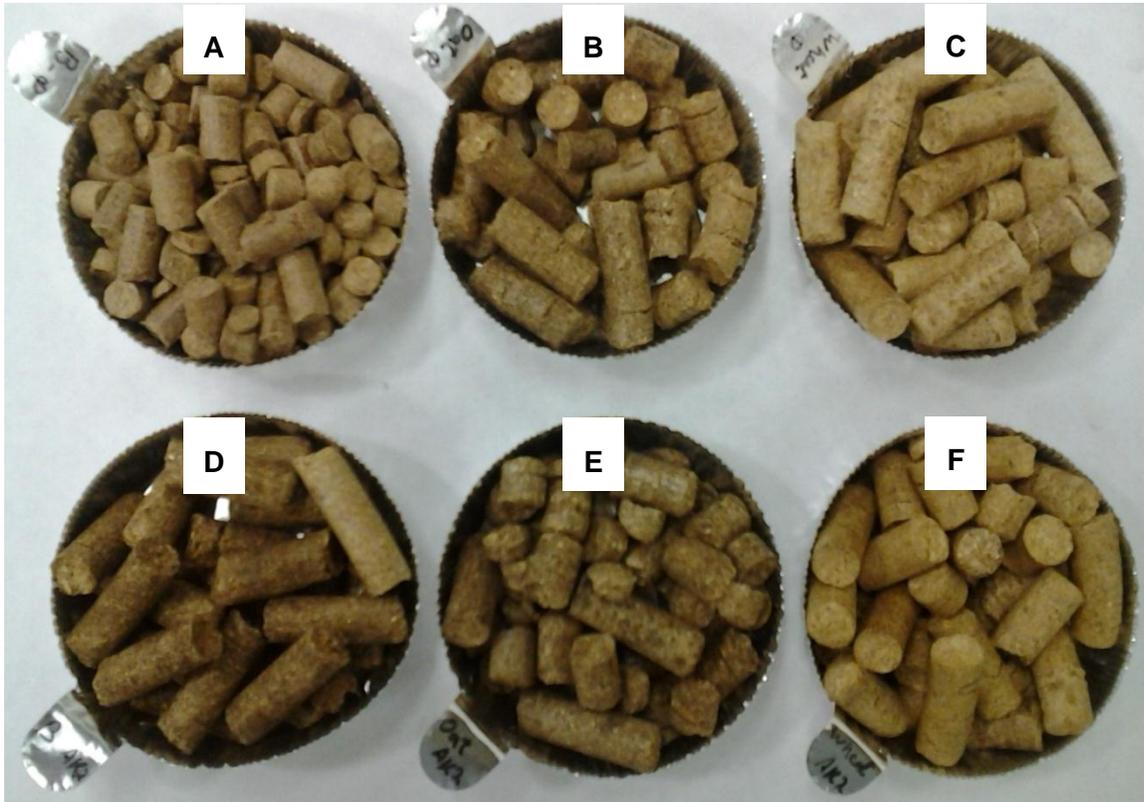


Figure 1. Photograph of pellets manufactured from cereal straw samples at hammer mill screen size of 0.8 mm using the CPM CL-5 pilot-scale pellet mill. **A** - barley straw pellets; **B** - oat straw pellets; **C** - wheat straw pellets; **D** – pellets of barley straw with 0.15% AK2; **E** – pellets of oat straw with 0.15% AK2 ; **F** – pellets of wheat straw with 0.15% AK2

**CONCLUSION** The effect of adding AK2 to ground barley, canola and oat straw to form pellets has been successfully studied. Straw was ground using two hammer mill screen sizes (1.6 and 0.8 mm) and was mixed with AK2 at three levels (0.00, 0.05, 0.10 and 0.15%) prior to pelleting operation. The oat straw pellets showed higher pellet density than barley and wheat straw pellets. Wheat straw had lower durability, measured by drop test, than barley and oat straws. Only screen size had significant effect on specific energy. Pellets from the pilot-scale pellet mill made from grinds of screen size of 0.8 mm and AK2 level of 0.15% were durable. Addition of AK2 at 0.15% has shown to increase the durability of barley and oat straw pellets manufactured by the pilot-scale pellet mill but not for the wheat straw pellets. Through gasification testes, a reduction in tar content in oat, wheat and barley pellets (3.8, 1.6 and 1.2 % resp.) was observed when 0.15% of AK2 was added.

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## REFERENCES

- AACC. 2005. AACC Standard 44-15A - Determination of moisture content by the air-oven method. In *Approved Methods of the American Association of Cereal Chemists*. St. Paul, MN: American Association of Cereal Chemists.
- Adapa, P.K., A. Singh, G. Schoenau and L.G. Tabil. 2006. Pelleting characteristics of fractionated alfalfa grinds - hardness models. *Powder Handling and Processing* 18(5): 294-299.
- Adapa, P.K., L.G. Tabil, G. Schoenau, B. Crerar, B. and S. Sokhansanj. 2002. Compression characteristics of fractionated alfalfa grinds. *Powder Handling and Processing* 14(4): 252-259.
- Al-Widyan, M.I. and H.F. Al-Jalil. 2001. Stress-density relationship and energy requirement of compressed only cake. *Applied Engineering in Agriculture* 17(6): 749-753.
- AOAC. 1990. AOAC Method 942.05 – ash in animal feeds. In *Official Method of Analysis of the Association of Official Analytical Chemists*, 15th ed., Vol. 70. Gaithersburg, MD: Association of Official Analytical Chemists.
- ASABE. 2007. ASAE S269.4 DEC1991 (R2007) - Cubes, Pellets, and Crumbles - Definitions and Methods for Determining Density, Durability, and Moisture. In *ASABE Standards 2007*, 624-626. St. Joseph, MI: American Society of Agricultural and Biological Engineers.
- ASABE. 2008. ANSI/ASAE S319.4 FEB2008 - Method of Determining and Expressing Fineness of Feed Materials by Sieving. In *ASABE Standards 2008*, 1-4. St. Joseph, MI: American Society of Agricultural and Biological Engineers.
- Bruuna, S., J.W. Jensen, J. Magida, J. Lindedama and S.B. Engelsen. 2010. Prediction of the degradability and ash content of wheat straw from different cultivars using near infrared spectroscopy. *Industrial Crops and Products* 31: 321-326.
- Jenkins, B.M., L.L. Baxter, T.R. Miles Jr. and T.R. Miles. 1998. Combustion properties of biomass. *Fuel Processing Technology* 54: 17-46.
- Kaliyan, N. and R.V. Morey. 2009. Factors affecting strength and durability of densified biomass products. *Biomass and Bioenergy* 33: 337-359.
- Kamburska, L. and S. Fonda-Umani. 2009. From seasonal to decadal inter-annual variability of mesozooplankton biomass in the Northern Adriatic Sea (Gulf of Trieste). *Journal of Marine Systems* 78: 490-504.
- Kashaninejad, M. and L.G. Tabil. 2011. Effect of microwavechemical pre-treatment on compression characteristics of biomass grinds. *Biosystem Engineering* 108: 36-45.
- Khankari, K.K., M. Shrivastava, M. and R.V. Morey. 1989. Densification characteristics of rice hulls. ASAE Paper No. 89-6093. St. Joseph, MI: American Society of Agricultural Engineers.
- Mahmoudkhani, M., T. Richards and H. Theliander. 2007. Sustainable use of biofuel by recycling ash to forests: treatment of biofuel ash. *Environmental Science & Technology* 41: 4118-4123.
- Mani, S., L.G. Tabil and S. Sokhansanj. 2006. Effects of compressive force, particle size and moisture content on mechanical properties of biomass pellets from grasses. *Biomass and Bioenergy* 97: 1420-1426.
- Nilsson, D., S. Bernesson and P.A. Hansson. 2011. Pellet production from agricultural raw materials - A systems study. *Biomass and Bioenergy* 35: 679-689.
- Sah, P., B. Singh and U. Agrawal. 1980. Compaction behavior of straw. *Journal of Agricultural Engineering-India* 18(1): 89-96.

- Samuelsson, R., M. Thyrel, M. Sjöström and T.A. Lestander. 2009. Effect of biomaterial characteristics on pelletizing properties and biofuel pellet quality. *Fuel Processing Technology* 90: 1129-1134.
- Serrano, C., E. Monedero, M. Lapuerta and H. Portero. 2011. Effect of moisture content, particle size and pine addition on quality parameters of barley straw pellets. *Fuel Processing Technology* 92: 699-706.
- Shaw, M.D., C. Karunakaran and L.G. Tabil. 2009. Physicochemical characteristics of densified untreated and steam exploded poplar wood and wheat straw grinds. *Biosystems Engineering* 103: 198-207.
- Shrivastava, M., P. Shrivastava and K.K. Khankari. 1989. Densification characteristics of rice husk under cold and hot compression. In *Agricultural Engineering: Proceedings of the 11th International Congress on Agricultural Engineering*, 2441-2443. Dublin, Ireland, 4-8 September. V.A. Dodd and P.M. Grace, eds. Rotterdam, The Netherlands: A.A. Balkema Pub.
- Sokhansanj, S., S. Mani, M. Stumborg, R. Samson and J. Fenton. 2006. Production and distribution of cereal straw on the Canadian Prairies. *Canadian Biosystems Engineering* 48: 3.39-3.46.
- Sultana, A., A. Kumar and D. Harfield. 2010. Development of agri-pellet production cost and optimum size. *Bioresource Technology* 101: 5609-5621.
- Tabil, L.G. and S. Sokhansanj. 1996. Compression and compaction behavior of alfalfa grinds - part 2: Compaction behavior. *Powder Handling and Processing* 8(2): 117-122.
- Tabil, L.G. and S. Sokhansanj. 1997. Bulk properties of alfalfa grind in relation to its compaction characteristics. *Applied Engineering in Agriculture* 13(4): 499-505.
- Thomas, M., D.J. van Zuilichem and A.F.B. van der Poel. 1997. Physical quality of pelleted animal feed. 2. Contribution of processes and its conditions. *Animal Feed Science and Technology* 64(2-4): 173-192.