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# Microwave-assisted alkali pretreatment and densification of canola straw and oat hull

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**ABSTRACT** *The effect of microwave-assisted alkali pretreatment on lignocellulosic biomass of canola straw and oat hull was investigated. The ground canola straw and oat hull were immersed in distilled water, sodium hydroxide and potassium hydroxide solutions at two concentrations (0.75 and 1.5% w/v) and exposed to microwave radiation at power level 713 W and three residence times (6, 12 and 18 min). Bulk and particle densities of ground biomass samples were determined. Alkaline-microwave pretreated and untreated samples were subjected to single pelleting test in an Instron universal machine, preset to load and compressed at 4000 N. The measured parameters, pellet density, tensile strength and dimensional stability were evaluated and the results showed that the microwave-assisted alkali pretreated pellets had a significantly higher density and tensile strength compared to the samples pretreated by microwave alone and untreated samples.*

**Keywords:** Densification, microwave pretreatment, canola straw, oat hull, pellet quality

**INTRODUCTION** The world relies on fossil fuels for its energy usage and the sources of these fossil fuels are from coal, oil and natural gas. Any event that threatens their availability affects the cost of supply such as being experienced in petroleum supply (Nomanbhay et al. 2013). However, the negative impact of fossil fuels on the environment is the increasing problem of greenhouse gas emissions. These emissions to the environment have attracted global interest in searching for alternatives, non-petroleum based sources of energy (Alvira et al. 2010; Nomanbhay et al. 2013). These renewable energy sources include solar energy, biomass, wind, hydroelectric and other sources which are more environmental friendly (Balat et al. 2008).

According to Alvira et al. (2010) and Balat et al. (2008), fuel ethanol can be produced from renewable biomass such as sugar, starch or lignocellulosic materials. The lignocellulosic materials from agricultural residues are interesting alternative. This is because they are second generation feedstock, less expensive than conventional agricultural feedstocks, available worldwide, do not compete with food crops, renewable and a good source of raw materials for developing bio-based products and bio-chemicals such as bioethanol, biodiesel (Demirbas et al. 2009; Smith 2013). Lignocellulosic materials include agricultural residues and by-products such as canola straw, wheat straw, rice straw, oats straw, corn stover, corn fiber, oat hull, rice hull, etc (Mosier et al. 2005). According to Sanchez and Cardona (2008), annual production of lignocellulosic biomass residue was estimated in $1 \times 10^{10}$ MT word wide. In Canada, an estimated of average agricultural residue generated for over 10 year period (2001 – 2010) was 82. 35 million (dry Mg/yr) and Saskatchewan recorded highest (17.38 million dry Mg/yr) (Li et al. 2012). These agricultural residues and by-products can be used for conversion into bioethanol.

Canola and oat are major crops grown in Canada. Canola an oilseed has estimated crop production as 15,555.1 million metric tonnes (mmt) and Saskatchewan production is estimated at 8.9 mmt. While oat production is estimated 2,907.5 mmt and Saskatchewan (1.6 mmt), Manitoba and Alberta are the major producers in Canada (Sask. Seed Statistics Canada 2014).

The pretreatment of lignocellulose material from agricultural residue is a key step for efficient utilization of biomass for ethanol production. Pretreatment helps in the breakdown of cell walls and internal tissues of the lignocellulosic biomass through biochemical conversion processes involving disruption and disintegration of recalcitrant structures in order to open channels for enzymatic reactions processes in the material (Mosier et al. 2005; Agbor et al. 2011; Quintero et al. 2011). An effective pretreatment technique is needed to liberate the cellulose from lignin, reduce cellulose crystallinity and to increase cellulose porosity (Zhu et al. 2006; Zhao et al. 2008; Nomanbhay et al. 2013). Various pretreatment methods have been developed, but the choice of pretreatment technology for a particular raw material is influenced by many factors such as enzymatic hydrolysis step and enzymes used (Alvira et al. 2010). Such pretreatment methods include; alkali and microwave-assisted pretreatment dilute acid, steam explosion, ammonia fiber explosion (AFEX), lime treatment and organic solvent treatments. Also, combination of these methods has been studied and still ongoing. (Alvira et al. 2010).

Microwave pretreatment method is a physico-chemical process involving thermal and non-thermal effects. Microwave has gained application in research studies because of its easy operation, high heating efficiency, reduction of process energy requirements, selective heating, etc. The early discoveries of microwave pretreatment on lignocellulosic biomass was reported by Ooshima et al. (1984) and Azuma et al. (1984) (Hu and Wen 2008; Xu 2015) and since then, the technology has shown an efficient applications in various ways (Gong et al. 2010; Keshwani and Cheng 2010; Quitain et al. 2013). Microwave-assisted alkali separates lignocellulosic biomass components by disruption of biomass structure, reduction in crystallinity of cellulose, improve formation of fermentable sugars and reduce the degradation of carbohydrates (Sun and Cheng 2002). The pretreatment process is carried out by immersing the biomass in alkaline concentration and exposing the slurry to microwave radiation for varying residence time (Keshwani 2009). Research

studies reported that alkaline reagents (sodium hydroxide) are the most effective and suitable for microwave-assisted pretreatment (Zhu et al. 2006; Alvira et al. 2010). Kashaninejad and Tabil (2011) investigated on the effect of microwave pretreatment on densification of wheat straw using dilute NaOH and $Ca(OH)_2$. The results indicated that the density and tensile strength of microwave alkali pretreated pellets were significantly higher than the untreated samples.

Biomass feedstock is bulky, loose and difficult to utilize as a fuel. The biomass has high moisture content, irregular shape and size, and low bulk density. All these factors make it difficult to handle, transport, store and utilize the biomass feedstock in its original form (Adapa et al. 2013). Some agricultural straws can be turned into forage by ensiling or made into pellets for energy applications. Pelletizing of biomass is a primary means to achieve densification (Veal 2010). Densification of biomass, such as pelletizing or briquetting increases bulk density, improves handling and storage characteristics, enhances volumetric calorific value, reduces transportation cost, improves combustion process control with coal, gasification and pyrolysis, increases uniformity of physical properties (shape and size), clean and stable pellets for environmentally friendly fuel production (Jenkins et al. 2011; Kashaninejad and Tabil 2011). Cellulose, lignin, hemicellulose, extractives and non-extractives are components of lignocellulose biomass.

However, it was observed from the research studies that there is knowledge gap in the application of microwave-assisted alkali pretreatment and densification on canola straw and oat hull. Therefore, the objective of this research was to investigate the effect of microwave-assisted alkali pretreatment on the densification characteristics of canola straw and oat hull.

## MATERIALS AND METHODS

### Sample preparation

Two agricultural residues (canola straw and oat hull) were used in this study. The canola straw was collected from Black soil zone, Saskatchewan (52.78$^o$N, 108.30$^o$W) and oat hull was sourced from (Richardson Mill) Martinsville, Saskatchewan (52.29$^o$N, 106$^o$W). The canola straw was 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 and 3.2 mm. The oat hull was cleaned using an aspirator cleaning machine (Carter-Day Company N.E Minneapolis, MN) to remove some oat kernel remaining after initial cleaning by the producers. The cleaned oat hull was ground using the same hammer mill and screen opening sizes. A dust collector including a cyclone system was used to collect the ground samples and reduced the dust during operation. The moisture contents of samples as-received and ground were determined using ASABE Standard S358.2 (ASABE, 2006) in three replicates. Also, ground samples particle size analysis was measured and determined using ASABE S319 (ASABE, 2008).

### Bulk and particle density analysis

The bulk densities of treated and untreated ground samples were determined and calculated using the mass and volume of a standard cylindrical steel container with 0.5 L volume (SWA951, Superior Scale Co. Ltd., Winnipeg, MB). The sample passed through a funnel and filled the 0.5 L volume container. A thin steel rod was used to roll across the top of the container in a steady pattern motion and weighed. The particle densities of the treated and untreated ground samples were determined. Ground canola straw and oat hull of known mass were placed in the gas multi-pycnometer (QuantaChrome, Boynton Beach, FL) and the volume of the sample determined. Thereafter, the particle densities were calculated by mass per unit volume of the samples. The procedure was done in five replicates for both bulk and particle densities.

## Particle size analysis

The particle size analysis of the ground samples was determined before microwave-assisted alkali pretreatment and densification. The geometric mean particle diameter of ground sample canola straw and oat hull was determined using ASAE Standard S319 (ASABE, 2012). The geometric mean diameter ($d_{gw}$) of the sample and geometric standard deviation of particle diameter ($S_{gw}$) were calculated using the standard mentioned (Mani et al. 2006; Adapa et al. 2009 and 2011).

## Microwave pretreatment

Microwave (MW) treatments were carried out using a domestic microwave oven (Model NNC980W, Panasonic Canada Ltd, Mississauga, ON, Canada) with an operating frequency of 2450 MHZ and variable power from 220 to 1100 W. 20 g of ground biomass sample (canola straw and oat hull) was immersed in 180 g of various alkaline solutions of 0, 0.75 and 1.5% (w/v) NaOH and 0, 0.75 and 1.5% (w/v) KOH. The mixture was placed in a 600 ml beaker and placed at the center of rotating ceramic plate inside the microwave oven for treatment at a fixed power of 713 W. The mixture was exposed to three levels of residence time 6, 12 and 18 min. After the treatments, the moisture content of each sample was determined and the sample maintained at appropriate moisture level of 12% (w.b.) for densification process and stored in Ziploc bag.

## Ash content

Ash content is a measure of mineral content and extractable in biomass (Iroba et al.2013). The ash contents of canola straw and oat hull were determined based on National Renewable Energy Laboratory standard (Sluiter et al. 2008). 2.0 ± 0.2 g of the oven dried microwave alkali treated and untreated samples were weighed into the tared dried crucible. The weighed crucible and sample were placed in a muffle furnace (Model F-A1T30, Thermolyne Sybron Corp., Dubuque, IA) and allowed to stay overnight at 575 – 600$^{o}$C. The sample was removed placed in an oven of temperature 105$^{o}$C for 20 – 30 min before placed in a desiccator to cool. The crucible and the ash were weighed. The ash content was calculated as the percentage of residue remaining after drying and each sample was replicated three times.

## Densification

The microwave-assisted alkali pretreated and untreated samples were compressed and pelleted in a single pelleting unit consisting of a plunger-cylindrical die connected to a computer that interprets and records the force-displacement data (Fig. 1). The plunger was connected to the Instron universal machine (NVLAP Lab Code 200301-0, Norwood, MA) in which the upper moving crosshead provided the load necessary to compress the biomass samples. About 0.5 – 0.8 g of selected pretreated and untreated biomass samples was loaded into the die cylinder. The temperature adjusted at about 95$^{o}$C and loads pre set compressed the samples. A 5000 N load cell fitted Instron universal machine was used and the pre set load compressed the samples at 4000 N. The plunger compressed the biomass sample using a crosshead speed of 50 mm/min. Once the pre set load was achieved, the plunger was stopped and held in position for 60 s to avoid spring back effect of biomass (Mani et al. 2006; Kashaninejad and Tabil 2011). Ten pellets each were produced from pretreated and untreated biomass samples, and force-deformation data at compression and the force-time data at stress relaxation were recorded in the computer. The physical characteristics of the densified pellets such as: pellet density, dimensional stability, and tensile strength tests were measured to evaluate the effect of the treatment combination of the various factors.

## Pellet density and dimensional stability

The pelleted samples height, diameter, and mass of microwave-assisted alkali pretreated and untreated were measured using digital calipers to calculate the volume and pellet density of the

samples. After two weeks, the diameter, height, and mass of the pelleted samples were measured to calculate the dimensional stability of the pellets. The change in density after two weeks was used to evaluate the dimensional stability and the pellets were stored in Ziploc plastic bags at room temperature at both stages for further analysis.

### Tensile strength test

The diametral compression test as reported by Tabil and Sokhansanj (1997) and Kashaninejad et al (2011) was used to determine the tensile strength of microwave-assisted alkali pretreated and untreated canola straw and oat hull pellets. The pellets were cut diametrally into specimens of thickness about 2.5 mm using laser cutting machine. The single cut pellet was placed at the middle of padded platen fasten on Instron machine (Fig. 2) and compressed by upper plunger until failure occurred. The Instron was fitted with a 5000 N load cell and the samples were compressed at a crosshead speed of 1 mm/min. The specimen fractured cracking into halves and failure occurred along the axis. Thirteen replicates were made for each sample. The fracture force was recorded and the tensile strength calculated as:

$$\delta_x = \frac{2F}{\pi d l} \qquad (1)$$

where $\delta_x$ is tensile strength (horizontal) stress (Pa); F is load at fracture (N); d is specimen diameter (m) and l is specimen thickness (m).

### Statistical analysis

Response Surface Methodology (RSM) is a statistical technique for designing experiments, building models, evaluating effects of factors which extract the maximal information with the minimal number of runs (Yue et al. 2008; Ma et al. 2009). In order to statistically study the effect of microwave treatment and alkali solution, User-Defined Design (UDD) was applied via analysis of variance (ANOVA) to investigate the effect of microwave heating time and alkali concentration on compaction of canola straw and oat hull. The range and levels of variables determined are shown in Table 1 and a polynomial quadratic equation was fitted to evaluate the effect of each independent variable against the responses.

Table 1.Code levels for independent variables used in the UDD and actual factor levels corresponding to coded factor levels

| Independent variable | Actual factor level at coded factor levels | | |
|---|---|---|---|
| | -1 | 0 | 1 |
| Alkali conc. (%) | 0 | 0.75 | 1.5 |
| MW time (min) | 6 | 12 | 18 |

## RESULTS AND DISCUSSION

### Physical properties

Table 2 shows the physical properties of ground canola straw and oat hulls. The geometric mean particle diameter of canola straw was slightly smaller than that of oat hull samples. The ash content was higher in canola straw samples compared to oat hull samples. These might be variation in moisture content of the different residue materials and difference in mechanical properties. The canola straw 1.6 mm screen size was the finest among other screen sizes. Also, the oat hull 1.6 mm sample recorded highest in bulk and particle densities 331.32 and 1440.51 kg/m$^3$ respectively.

It was observed that as the screen size was increased in openings, the lower were the bulk and particle densities.

Table 3 and 4 are the physical properties of microwave-assisted alkali pretreated canola straw and oat hull. It was observed that samples pretreated with microwave alone showed lower bulk and particle densities 108.10 kg/m$^3$ and 982.42 kg/m$^3$ than the untreated samples. Increasing the time and alkali concentration affected the bulk density of microwave-alkali pretreated canola straw and oat hull. The analysis of variance of the data shows that microwave heating time and alkali concentration significantly affected the bulk density of microwave-alkali pretreated canola straw and microwave heating time had significant effect on the bulk density of microwave-alkali pretreated oat hull. Similarly, increasing the alkali concentration increased the particle density for microwave-alkali pretreated canola straw and oat hull except at 3.2 mm 0.75% NaOH. The microwave heating time did not show significant effect on particle density for microwave-alkali pretreated oat hull and canola straw. These significant in the pretreated samples was as a result of microwave pretreatment which causes swelling of material and increases internal surface area of lignocellulosic structure (Kashaninejad and Tabil 2011). Canola straw and oat hull pretreated by microwave-assisted alkali showed higher bulk and particle densities than untreated samples. Kashaninejad and Tabil (2011) reported that this is a result of increased depolymerized components and ash content of pretreated samples. In addition, samples pretreated with microwave/NaOH had higher bulk and particle densities than samples pretreated with microwave/KOH.

## Pellet density

Table 5 and 6 show the effect of microwave-alkali pretreatments on pellet density, dimensional stability and tensile strength for canola straw and oat hull pellets. The microwave-assisted alkali pretreated samples showed the highest pellet density (canola straw 1392.21 kg/m$^3$ and oat hulls 1292.59 kg/m$^3$) compared to microwave alone and untreated samples. Increasing the alkali concentration increased the pellet density of the samples. Increasing the microwave heating time decreased the pellet density of canola straw samples with treatments of 1.6 mm/0, 1.5% NaOH, 0.75 and 1.5% KOH and 3.2 mm/ 0 and 0.75%; for oat hull, the microwave heating time increased for samples with treatments of 1.6 mm/1.5% NaOH, 0.75 and 1.5% KOH and decreased in samples treated with 3.2mm/0.75% KOH. Analysis of variance of the data showed that alkali concentration significantly affected canola straw and oat hull pellet density. Microwave heating time had significant effect for samples with treatments of 3.2 mm NaOH and KOH for canola straw pellets and oat hull pellets with treatment of 1.6 mm KOH. In addition, microwave/NaOH pretreatment was more effective at the initial heating time for 0.75% alkali concentration in increasing the initial density of the pellets while microwave/KOH pretreatment was more effective at1.5% alkali concentration in increasing the initial pellet density.

## Dimensional stability of samples pellets

Table 5 and 6 also show the effect of microwave/alkali pretreatments on dimensional stability of canola straw and oat hull pellets. Samples pretreated with microwave-assisted alkali have the highest dimensional stability than microwave alone and untreated samples. In canola straw, microwave-assisted alkali pretreated had the highest dimensional stability in 1.6 mm 1.5% NaOH at 12 min (0.99%) and oat hull in 3.2 mm 1.5% NaOH at 6 min (0.68%). This is because samples released the binding agent (lignin) which increased the adhesion within the particles, activated the intermolecular bonds within the contact area of the samples and in addition enhanced the mechanical interlocking of the particles (Iroba et al. 2014). The data indicated that dimensional stability of canola straw pellets increased with alkali concentration in 1.6 mm screen size except samples treated for 12 min with 1.5% NaOH; stability decreased in both 3.2 mm screen size samples. Oat hull pellet stability decreased with increasing alkali concentration. Lower microwave heating time resulted in higher stability of the canola straw pellets for treatment combination of: 1.6

mm/0 and 0.75% NaOH, and 3.2 mm 1.5% NaOH and 1.5% KOH and in oat hulls pellets 1.6 mm/ 0, 0.75 and 1.5% KOH, and 3.2 mm/ 0, 0.75 and 1.5% NaOH and KOH. Iroba et al. (2014) and Tabil (1996) reported that when biomass is heated, the lignin becomes soft, melts and exhibits thermosetting binder resin properties to produce pellets with higher density and dimensional stability. Analysis of variance shows that alkali concentration and microwave heating time significantly affected the dimensional stability of the canola straw pellets in 3.2 mm NaOH and KOH and oat hull pellets except in 1.6 mm NaOH, time was not significant.

## Tensile strength of pellets

Table 5 and 6 also show the tensile strength and the fracture load values of the pellets produced from microwave-assisted alkali pretreated, microwave alone and untreated canola straw and oat hull. The observed data indicates that alkali concentration and microwave heating time are important factors and process condition for the physical characteristics of the pellets. Microwave-assisted-alkali pretreated pellet samples showed highest tensile strength (canola straw 5.22 MPa at 1.6 mm 1.5% NaOH 6 min and oat hull 3.36 MPa at 1.6 mm 1.5% NaOH 18 min). Increasing the alkali concentration increased the tensile strength of canola straw and oat hull pellets. This means that biomass samples pretreated with microwave-assisted alkali has the ability to disintegrate the structure of lignocellulosic materials involved in particle binding (Kashaninejad and Tabil 2011). Increasing the microwave heating time reduces the tensile strength of canola straw pellets and increases the strength of oat hull pellets. The analysis of variance shows that alkali concentration significantly affected the tensile strength of canola straw pellets and oat hull pellets at 1.6 and 3.2 mm NaOH. Microwave heating time significantly affected tensile strength of canola straw pellets except in 3.2 mm NaOH and oat hull pellets, the microwave heating time had significant effect only in 3.2 mm NaOH.

## CONCLUSIONS

Based on the data collected from this study, the following conclusions are made:

1. Microwave-assisted pretreated samples had significant effect on the ground and pelleted samples compared to microwave alone (water) and untreated samples.

2. After the pretreatment of the canola straw and oat hull samples by microwave-assisted alkali, the ash content increased with increasing alkaline concentration and microwave heating time.

3. The ground samples pretreated by microwave-assisted alkali had significantly higher bulk and particle densities than microwave alone and untreated samples; samples pretreated with microwave/NaOH had higher bulk and particle densities than samples pretreated with microwave/KOH.

4. Microwave-assisted alkali pretreatment increases pellet density, dimensional stability and tensile strength over that of pellets from microwave alone (water) and untreated samples.

5. Increasing the alkali concentration and reducing the microwave heating time resulted in high tensile strength of canola straw pellets while the tensile strength of oat hull pellets was increased by increasing the alkali concentration and the microwave heating time.

6. Microwave/NaOH is more efficient than microwave/KOH on canola straw and oat hull samples.

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## APPENDIX A

Table 2. Physical properties of ground canola straw and oat hull

| Samples | Moisture content as received (% wb) | Hammer mill screen size (mm) | Moisture content (% wb) | $d_{gw}$ (mm) | Sgw (mm) | Ash content (%) | Bulk density ($kg/m^3$) | Particle density ($kg/m^3$) |
|---|---|---|---|---|---|---|---|---|
| Canola straw | 9.08 | 1.6 | 7.64 | 0.348 | 0.280 | 6.47 | 168.14 | 1305.53 |
| | | 3.2 | 8.28 | 0.520 | 0.498 | 6.66 | 141.16 | 1220.41 |
| Oat hulls | 9.72 | 1.6 | 6.96 | 0.370 | 0.217 | 5.31 | 331.32 | 1440.51 |
| | | 3.2 | 7.7 | 0.547 | 0.284 | 5.65 | 285.10 | 1391.01 |

Geometric mean diameter = $d_{gw}$ Geometric standard deviation = $S_{gw}$

Table 3. Physical properties of MW/alkali pretreated canola straw samples.

| Treatment method | | Ash content (%) | | | Bulk density ($kg/m^3$) | | | Particle density ($kg/m^3$) | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 |
| CS 1.6 mm NaOH | 0 | 5.16 | 5.33 | 5.48 | 122.43 | 134.57 | 137.72 | 1262.91 | 1206.60 | 1124.45 |
| | 0.75 | 15.50 | 14.83 | 14.33 | 149.41 | 171.54 | 183.15 | 1514.18 | 1423.10 | 1303.16 |
| | 1.5 | 20.13 | 22.17 | 22.96 | 160.22 | 194.72 | 260.11 | 1572.56 | 1472.80 | 1358.89 |
| CS 1.6 mm KOH | 0.75 | 12.83 | 12.67 | 12.33 | 137.79 | 154.45 | 157.00 | 1389.39 | 1428.87 | 1134.54 |
| | 1.5 | 19.33 | 19.67 | 19.83 | 145.33 | 173.98 | 200.99 | 1411.14 | 1496.22 | 1343.62 |
| CS 3.2 mm NaOH | 0 | 5.17 | 5.33 | 5.50 | 108.10 | 116.40 | 126.96 | 1033.48 | 1045.16 | 982.42 |
| | 0.75 | 15.17 | 14.67 | 14.33 | 131.71 | 148.11 | 170.48 | 1324.92 | 1423.39 | 1229.76 |
| | 1.5 | 21.17 | 22.33 | 22.50 | 153.09 | 183.61 | 247.76 | 1462.90 | 1466.03 | 1297.84 |
| CS 3.2 mm KOH | 0.75 | 13.17 | 13.00 | 12.67 | 114.86 | 133.13 | 137.40 | 1285.89 | 1335.35 | 1043.47 |
| | 1.5 | 20.17 | 20.50 | 20.67 | 123.82 | 164.70 | 190.07 | 1429.45 | 1511.66 | 1281.60 |

Table 4. Physical properties of MW/alkali pretreated oat hull samples.

| Treatment method | | Ash content (%) | | | Bulk density (kg/m$^3$) | | | Particle density (kg/m$^3$) | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 |
| OH 1.6 mm NaOH | 0 | 4.67 | 4.83 | 5.00 | 258.28 | 264.84 | 321.27 | 1427.75 | 1430.20 | 1410.10 |
| | 0.75 | 8.50 | 9.67 | 9.83 | 235.95 | 270.10 | 334.46 | 1465.14 | 1502.91 | 1502.89 |
| | 1.5 | 15.17 | 15.83 | 16.17 | 280.39 | 329.28 | 353.11 | 1544.32 | 1557.82 | 1548.69 |
| OH 1.6 mm KOH | 0.75 | 7.00 | 7.17 | 7.83 | 243.14 | 276.28 | 298.96 | 1447.42 | 1451.40 | 1464.83 |
| | 1.5 | 13.00 | 13.17 | 13.50 | 247.12 | 290.12 | 339.04 | 1498.86 | 1546.26 | 1523.19 |
| OH 3.2 mm NaOH | 0 | 4.50 | 4.67 | 5.33 | 207.07 | 206.46 | 240.53 | 1373.74 | 1361.42 | 1394.83 |
| | 0.75 | 8.83 | 9.00 | 9.50 | 207.31 | 238.94 | 253.98 | 1324.92 | 1423.39 | 1229.76 |
| | 1.5 | 15.00 | 15.67 | 16.00 | 236.56 | 336.55 | 283.27 | 1533.35 | 1559.22 | 1548.87 |
| OH 3.2 mm KOH | 0.75 | 7.17 | 7.50 | 8.00 | 209.05 | 217.53 | 244.91 | 1456.67 | 1464.51 | 1457.28 |
| | 1.5 | 13.33 | 13.67 | 13.83 | 221.49 | 257.88 | 258.75 | 1507.52 | 1541.46 | 1530.87 |

Table 5. Effect of MW/Alkali pretreatments on pellet density, dimensional stability and tensile strength for canola straw pellets.

| Treatment method | | Pellets density | | | Dimensional stability | | | Tensile strength | | | Fracture load | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Untreated | 1.6 mm | 1030.87 | | | 5.40 | | | 0.26 | | | 6.65 | | |
| | 3.2 mm | 1060.82 | | | 6.41 | | | 0.62 | | | 15.95 | | |
| Alkali conc./MW time | | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 |
| CS 1.6 mm NaOH | 0 | 1066.17 | 1037.10 | 1021.65 | 2.67 | 3.83 | 4.52 | 0.72 | 0.74 | 0.56 | 18.55 | 19.06 | 14.38 |
| | 0.75 | 1286.59 | 1309.35 | 1248.24 | 1.67 | 2.76 | 2.79 | 4.71 | 2.66 | 1.79 | 120.15 | 68.4 | 45.99 |
| | 1.5 | 1319.21 | 1327.98 | 1370.27 | 2.14 | 0.99 | 2.80 | 5.22 | 3.44 | 2.31 | 133.72 | 88.37 | 59.32 |
| CS 1.6 mm KOH | 0.75 | 1243.01 | 1195.28 | 1160.16 | 2.24 | 1.26 | 3.64 | 2.67 | 1.90 | 0.85 | 68.33 | 48.89 | 21.87 |
| | 1.5 | 1392.21 | 1339.64 | 1324.43 | 4.11 | 2.97 | 3.22 | 3.75 | 2.58 | 2.11 | 95.77 | 66.44 | 54.31 |
| CS 3.2 mm NaOH | 0 | 1089.17 | 1086.86 | 1029.82 | 3.98 | 3.79 | 5.54 | 1.19 | 1.04 | 0.81 | 30.56 | 26.94 | 21.16 |
| | 0.75 | 1324.75 | 1283.60 | 1277.29 | 3.04 | 2.68 | 3.86 | 4.85 | 2.53 | 1.69 | 123.93 | 65.04 | 43.5 |
| | 1.5 | 1351.61 | 1345.57 | 1388.30 | 1.32 | 1.57 | 3.01 | 4.20 | 4.11 | 2.59 | 107.5 | 105.49 | 66.53 |
| CS 3.2 mm KOH | 0.75 | 1201.33 | 1220.50 | 1176.32 | 2.30 | 2.29 | 4.28 | 2.07 | 1.73 | 1.41 | 53.39 | 44.6 | 36.4 |
| | 1.5 | 1382.62 | 1344.09 | 1355.93 | 1.53 | 1.62 | 3.67 | 5.16 | 3.19 | 2.89 | 132.84 | 82.06 | 74.29 |

Table 6. Effect of MW/Alkali pretreatments on pellet density, dimensional stability and tensile strength for oat hull pellets.

| Treatment method | | Pellets density | | | Dimensional stability | | | Tensile strength | | | Fracture load | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Untreated | 1.6 mm | 1031.23 | | | 14.64 | | | 0.04 | | | 0.93 | | |
| | 3.2 mm | 1087.74 | | | 7.93 | | | 0.39 | | | 10.08 | | |
| Alkali conc./MW time | | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 | 6 | 12 | 18 |
| OH 1.6 mm NaOH | 0 | 989.14 | 1029.53 | 1028.72 | 6.05 | 10.46 | 10.60 | 0.14 | 0.04 | 0.30 | 3.57 | 1.07 | 7.90 |
| | 0.75 | 1238.12 | 1209.12 | 1221.99 | 3.62 | 1.74 | 7.19 | 1.34 | 1.58 | 1.33 | 34.22 | 40.43 | 34.4 |
| | 1.5 | 1198.89 | 1286.52 | 1292.59 | 1.35 | 1.18 | 6.36 | 1.19 | 1.96 | 3.36 | 30.70 | 50.27 | 87.31 |
| OH 1.6 mm KOH | 0.75 | 1123.85 | 1164.37 | 1166.59 | 2.90 | 4.70 | 6.84 | 0.57 | 0.82 | 0.73 | 14.63 | 21.00 | 18.97 |
| | 1.5 | 1185.69 | 1220.52 | 1290.75 | 2.15 | 2.57 | 5.06 | 0.63 | 0.83 | 1.43 | 16.19 | 21.32 | 36.79 |
| OH 3.2 mm NaOH | 0 | 1045.82 | 1018.03 | 1066.38 | 4.46 | 10.10 | 12.19 | 0.25 | 0.27 | 0.45 | 6.52 | 7.10 | 12.07 |
| | 0.75 | 1205.73 | 1198.83 | 1219.29 | 3.56 | 3.71 | 7.74 | 1.23 | 1.17 | 1.91 | 31.76 | 30.41 | 49.85 |
| | 1.5 | 1218.86 | 1321.34 | 1274.09 | 0.68 | 2.58 | 6.96 | 1.28 | 2.27 | 2.61 | 32.95 | 58.55 | 68.45 |
| OH 3.2 mm KOH | 0.75 | 1073.31 | 1160.83 | 1143.75 | 2.24 | 7.14 | 8.49 | 0.46 | 0.87 | 0.90 | 12.06 | 22.67 | 23.65 |
| | 1.5 | 1212.34 | 1248.13 | 1210.94 | 1.65 | 4.62 | 6.85 | 0.99 | 1.08 | 1.17 | 25.54 | 28.06 | 30.65 |

## APPENDIX B

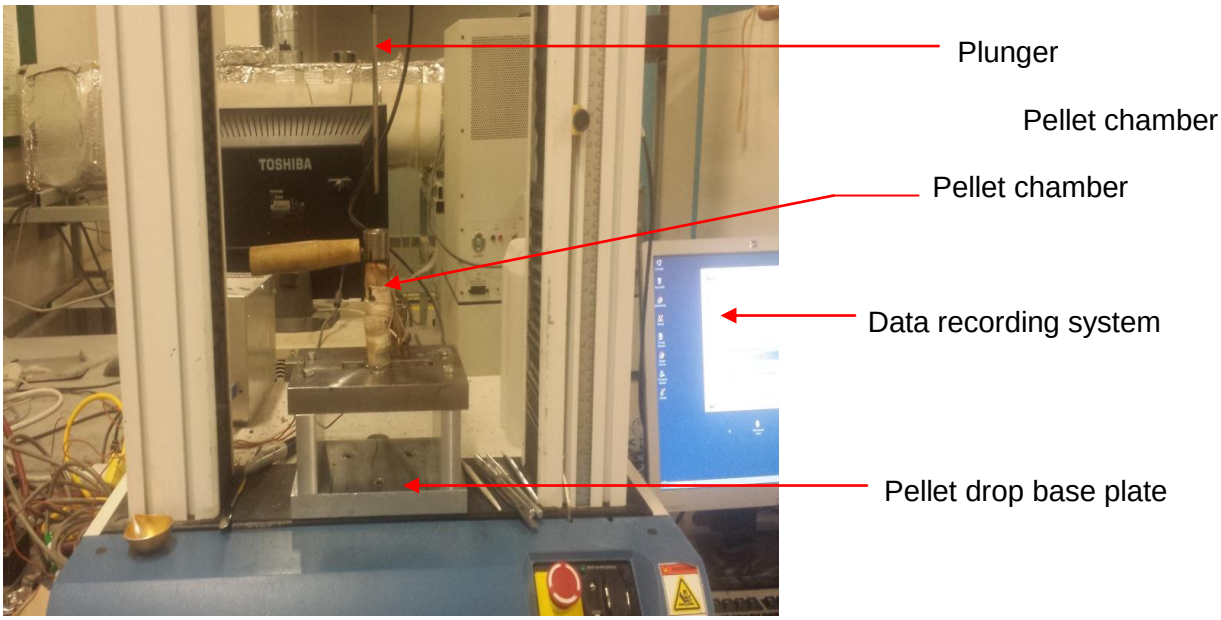


Figure 1. Single-pelleter connected to Instron universal machine and a data recording unit.

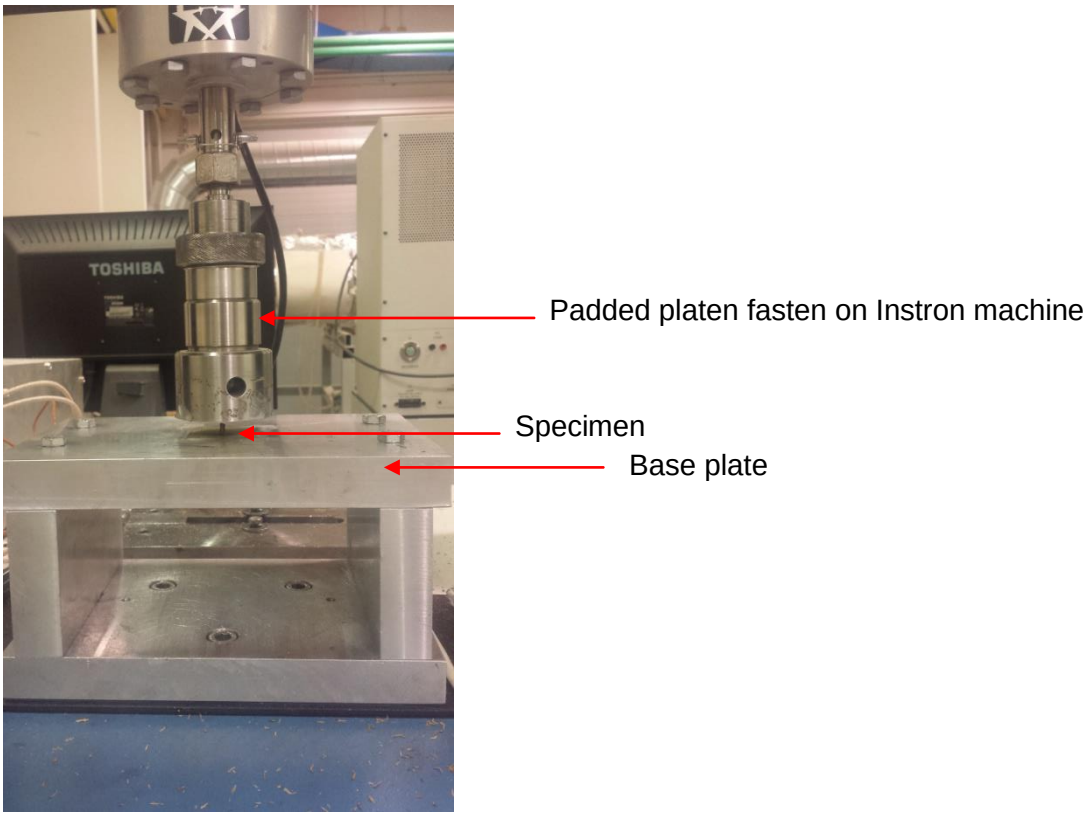


Figure 2. Instron machine fixed with padded platen used for tensile strength testing.