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Durability and combustion values of compacted oat straw biomass with manure binder

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Abstract In this study densified biomass pellets were made from milled oat straw and supplemented with manure to act as the binding agent. Using a single compaction unit, biomass pellets were compressed to 90 MPa. Samples were compressed at a rate of 100 mm/min and mixed to ratios of 0:1, 20:80, 40:60, 60:40, 80:20, and 1:0 manure to oat straw by mass for the density and strength tests. Two characteristics of the biomass pellets were measured for each ratio set: density of a single pellet, pellet strength (durability), while specific energy was measured for 0:1, 40:60. 60:40 and 1:0 ratios. Increasing the manure content from 0 to 40% raised the density of a single pellet from 908 ± 28 kg/m^3 to 1040 ± 29 kg/m^3. The amount of manure present was found to be directly proportionate to the strength/durability of the pellets. Increasing the manure concentration from 0 to 20% caused a 76% increase in strength, and a further 64% increase from 20 to 40%. The pellet samples with manure content of 0 % had the highest specific energy at 15.85 MJ/kg, with the specific energy decreasing linearly to 13.68 MJ/kg as manure content reached 100%. The slope of the trend line was calculated to be -0.0223.

Keywords: biomass, pellet, calorimeter, densification, diametral compression, durability, manure, oat straw, specific energy.

Introduction

In Canada there is approximately 36 Mha of crop land available for agriculture, with more than 85% being located in the Canadian Prairies (Campbell et al. 2002). Between the years of 1994-2003, oat production averaged 4.215 Mt throughout the Prairies (Sokhansanj et al. 2006). In the 2016, there was approximately 11,500 km^2 of seeded oat fields in Canada (Stats Canada, 2016). Inevitably with cereal crop harvest, issues arise due to the residue left over in the form of straw. Although the straw residue is considered a waste product, there are many large uses for straw including fuel, livestock bedding and fodder, and smaller uses such as thatching and basket making.

Over the past decades modifications to crop genetics has allowed for higher yielding crops, while increasing the harvest index, the measured ratio of the mass of harvested grain to the total dry mass of the plant. An increase in the harvest index of a crop would mean a reduction in the total residue left by reallocating the dry matter within the crop towards the harvested grain. The reduction in waste plant material by increasing the harvest index is key in cutting the agricultural waste residue left over after harvest. In 2006, Sokhansanj et al. investigated values used to evaluate the ratio of straw to grain mass, and arrived at a harvest index value of 1.1 for oats. Even with a decrease in residue, questions arise with the use of the remaining residue. This is where oat straw and other agricultural residues have the potential to be used as the main ingredient for biomass pellets that can be used in biofuel production. With the use of agricultural residue, there are logistical challenges due to the need to gather, process and densify the crop residue before it can be used as a viable biofuel (Adapa et al. 2009). The inherent need to transport the densified pellets after they are produced results in the demand for pellets with a high durability value to avoid the production of combustible dust during transportation and handling as well as to reduce the potential of loss of product.

The general objective of this study is to determine if manure can be used as a viable binding agent for pellets made from milled oat straw. The specific objectives are to determine:

1) The effects of manure concentrations on density,
2) The effects of manure concentrations on the strength and durability of pellets and
3) The effects of manure concentration on the specific energy of pellets.

Materials and Methods

Materials

The oat straw used in this study was obtained from the University of Manitoba Glenlea Research Station in Glenlea, Manitoba. The straw was removed from a bale stored in a roofed structure open to ambient temperatures. The straw was harvested in 2016 but the variety of oats are unknown. Once obtained, the oat straw was kept open to inside ambient temperature and conditions until needed. The oat straw was cut in to approximately 2 inch pieces, and approximately 10 g samples were milled using a Stein Laboratory M-1mill (Fred Stein Laboratories Inc., Atchison, KS). Samples of oat straw were milled at 3 and 5 minutes and sieve analysis was done on both sets to determine the median particle size in order to conclude the optimum milling time. Once milled, the oat straw was kept in a sealable plastic bag and stored at ambient conditions to avoid any excess gain or loss of moisture in the sample. A 75 g charge of milled oat straw was sieved according to ASABE 319.4 (2008) for 10 minutes using a W.S. Tyler coarse test sieve shaker (Model RX-812, W.S. Tyler, Mentor, OH) with ASTM E-11 sieves. The ASABE standard calls for a 100 g charge but due to material constraints, a 75 g charge was used. The median particle size was determined for the 3 and 5 minute samples. The 5 minute sample was chosen for its smaller particle size. A table of the sieve sizes used for the particle distribution analysis can be found below in Table 1. Samples of about 2 g of milled oat straw were dried at 103°C for 24 hours according to ASABE 358.3 (2012) to determine moisture content.

Table 1: Sieve sizes used for sieve analysis of oat straw and manure.

US Sieve No.	US Sieve size (mm)
16	1.190
20	0.841
30	0.595
40	0.420
50	0.297
60	0.250
70	0.210
80	0.177
100	0.149
120	0.125
Pan	0.000

Manure was also obtained from the University of Manitoba Glenlea Research Station. Manure was collected by hand from an animal waste storage building located open to outside ambient conditions. The composition of the manure was a combination of solid manure and bedding material. The samples were stored in a sealed bag in a freezer until needed. Before use, the manure was dried for 24 hours at 103°C to eliminate all moisture. The moisture was removed from the sample to avoid inconsistent moisture content when being mixed with oat straw. Samples of about 10 g of manure were milled for 3 minutes using the Stein Laboratory M-1 mill used previously for the oat straw. A 50 g charge of milled manure was sieved according to ASABE 319.4 (2008) for 10 minutes using a W.S. Tyler coarse test sieve shaker. Due to material constraints, a 50 g charge was used instead of 100 g. The sieve sizes used for the analysis are listed in Table 1.

Densification using a single compaction machine

Approximately 1.0 g of the oat straw and manure mixtures were inserted in to a 12.3 mm diameter die and compressed using an Instron universal testing machine (UTM) (Model 3366 Universal Testing Systems, Instron Corp., Norwood, MA) equipped with a 10 kN load cell. Approximately 1.5 g was used for the 100% manure samples to allow for full compaction of the pellets and to avoid damage of the plunger and apparatus. The samples were compressed using a preset load of 8500 N, corresponding to a pressure of 71.5 MPa, and at a rate of 100 mm/min. Inertia of the moving cross head of the test frame resulted in a final pressure of ~90 MPa. The compacted pellet was allowed to relax for 60 s in the die before being removed. The individual pellets were removed from the die by removing the mold base and mounting an open ended apparatus to allow the pellet to be removed by the plunger previously used to make the pellets. Immediately afterwards the pellets were measured for their diameter, and length using electronic calipers and weighed using an electronic scale. Using the diameter, length and mass, the density of each pellet was calculated using Eq. 1.

$$\rho = \frac{mass}{volume} \tag{1}$$

Where ρ is density [kg/m^3], mass is the mass of the pellet [kg] and volume is the volume of the pellet $(\pi \cdot D^2 \cdot t)/4$ [m^3], D is the diameter of the die [m], and t is the thickness of the pellet [m].

Diametral compression test

Several methods have been used to determine the strength, durability and impact resistance of densified materials. ASTM method D440-86 was employed by Li and Liu (2000) for testing the durability of biomass logs. The logs were dropped at standardized heights and the number of smaller pieces after each drop were measured and an impact resistance index was calculated. ASABE Standard S269.5 (2012) uses a tumbling test frame method for determining the durability of densified products. Samples are tumbled in a rotating test frame, and then measured for their change in mass. Lu et al. (2014), Johnson et al. (2014), and Dueck (2016) used a diametral compression test in order to determine the strength of the densified biomass. Due to equipment availability, this study used a diametral compression test as described by Johnson et al. (2014). Pellets were placed between two parallel flat disks, one stationary and one attached to the moving test frame. Using the Instron UTM, the upper disk of the apparatus was lowered at a rate of 1 mm/min until pellet failure. The maximum load was recorded for each sample. Five samples were tested for ratios of 0:1, 20:40, 40:60, 80:20 and 1:0 of manure by mass. Using the diametral compression test allows for the determination of stress at failure using Eq. 2 (Johnson et al. 2014).

$$\sigma_x = \frac{2P}{\pi D t} \tag{2}$$

Where σ_x is the normal stress perpendicular to the loaded diameter [MPa], P is the applied load [N], D is the diameter of densified disc [mm] and t is the thickness of the disc [mm].

Specific Energy

Specific energy of the pellets was determined using an IKA® bomb calorimeter (model C200, IKA® Works Inc., Wilmington, NC) and an IKA® C248 oxygenator. The oxygenator was used to pressurize the vessel to approximately 30 bar oxygen. Prior to testing, the calorimeter was calibrated by burning a benzoic acid pellet. The heat capacity of the bomb calorimeter was calculated by using Eq. 3 (ASTM Standard D5865. 2013).

$$C\Delta T + m_s \cdot u_{RP,0} - U_e = 0 \tag{3}$$

Where C is the heat capacity of the calorimeter [J/K], ΔT is the change in temperature from ignition to completion of the test [K], m_s is the mass of the benzoic acid pellet [kg] (0.500 g), $u_{RP,0}$ is the specific energy of the benzoic acid [J/kg], and U_e is the specific energy of the cotton fuse used for ignition [J]. The values for the specific energy for the benzoic acid and cotton fuse were given as -26.4397x10^6 J/kg and 50 J, respectively. Eq. 3 was used to determine C to be 9888.11 J/K.

Two pellets of about 1.0 gram from the 0:1, 40:60, 60:40, ratio of manure to oat straw samples, and two pellets of about 1.5 g of the 1:0 manure by mass samples were tested. The specific energy of the pellets was calculated by rearranging Eq. 3 and multiplying the right side by -1 to attain Eq. 4.

$$ED = \frac{C\Delta T - U_e}{m_s} \quad (4)$$

Where ED is the specific energy [J/kg].

Results and Discussion

Materials

The milled oat straw samples of 3 and 5 minute milling times had median particle sizes by mass of 0.416 mm and 0.350 mm respectively. The 5 min milling time sample was chosen and the other one discarded. A graph of the particle distribution for the 3 minute, 5 minute and manure can be found below in Figure 1 and Figure 2 respectively. The milled oat straw was found to have a 7.6% moisture content (mc) wet basis (w.b.). The manure sample milled for 3 minutes had a median particle size of 0.210 mm. The particle distribution graph for the manure can be found below in Figure 3. The moisture content of the manure was assumed to 0% after oven drying and storage in sealed bags.

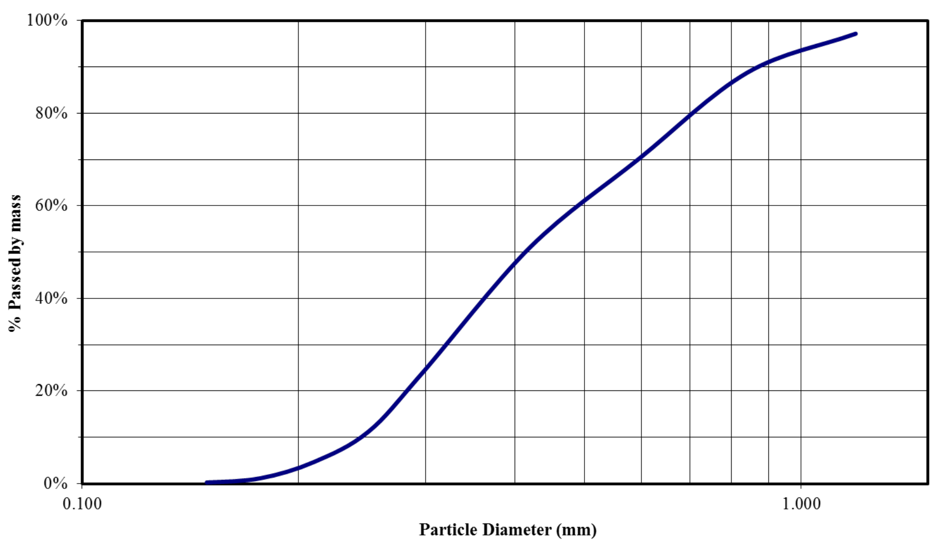

Figure 1 - Particle distribution for oat straw milled for 3 minutes

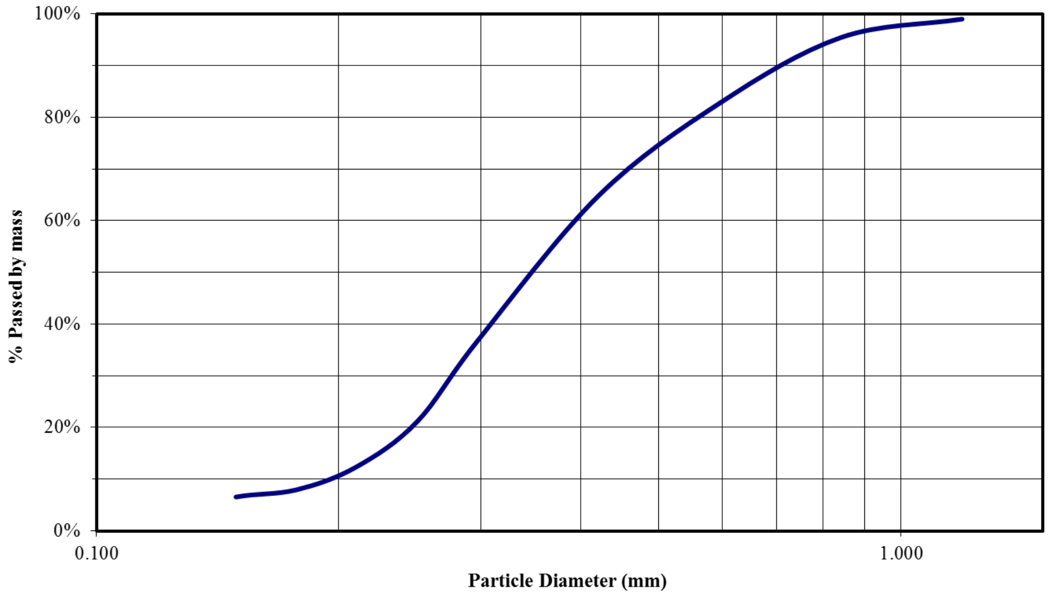

Figure 2 - Particle distribution for oat straw milled for 5 minutes

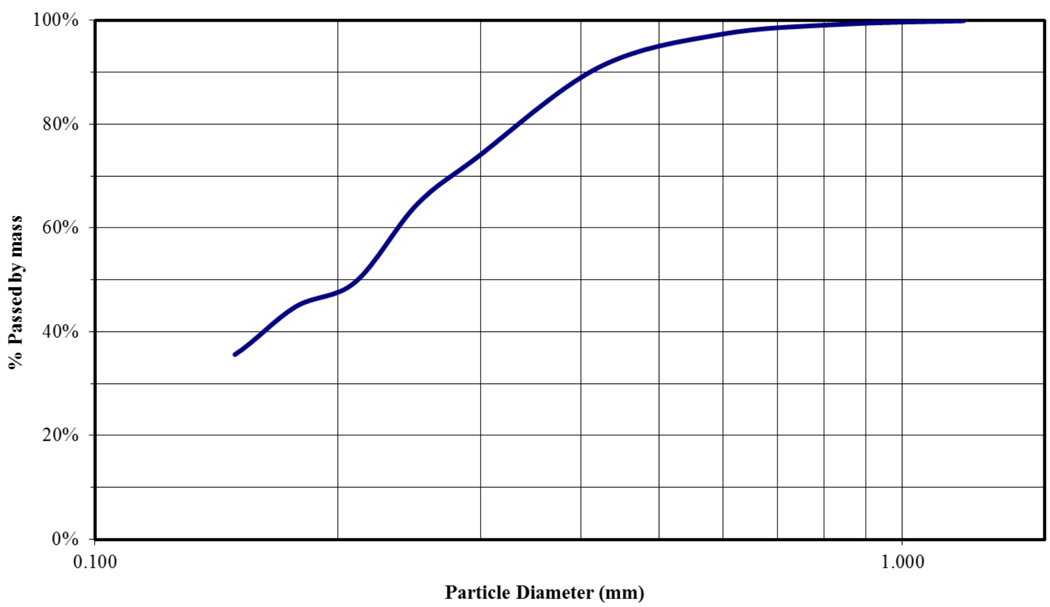

Figure 3 - Particle distribution for manure milled for 3 minutes

Single Pellet Density

It was found that increasing the manure concentration of the pellets resulted in a positive increase in the overall density of the pellets. The pellet density continued to increase from 0 to 100% manure following a linear trendline with high correlation. The rise in density of the pellets is potentially due to the smaller median particle size of the manure, allowing for a more compact pellet compared to pellets with higher oat straw content. The density of the pellets followed a linear equation as defined by graphing software Graph in Eq. 5. (Graph version 4.4.2 Build 543, Ivan Johnson, http://www.padowan.dk).

$$\rho(x) = 2.7084x + 918.5276 \tag{5}$$

Where ρ is the density [kg/m^3], and x is the percent manure content present in the sample.

Figure 1 plots the mean data points for the calculated density values for the sample sets. The error bars are the standard deviation of the population. Eq. 5 provides a mathematical model that can be used to predict the density of the pellet given manure content. Using Eq. 5 we can predict that a percent increase in the manure content, would cause a 2.7 kg/m^3 increase in the density of the pellets. An additional five pellets were tested at 40% manure to get a more representative data set. Discrepancies in data, specifically the 40% manure content samples, could have risen from the procedure in which the oat straw and manure mixture was added to the mold. For each pellet, the mass of manure and oat straw was weighed out before, and the mixture mixed by hand to distribute the particles as evenly as possible. The mixture was then added to the mold using a laboratory spatula. This procedure could have caused an uneven layering of the manure and oat which caused uneven compaction of the pellets.

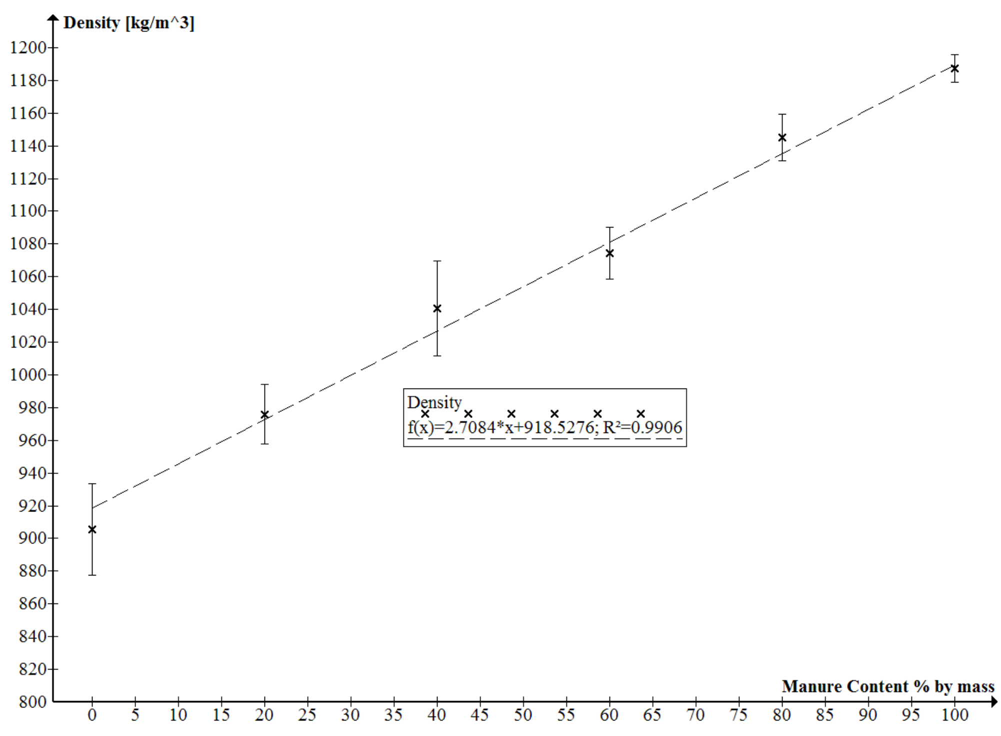

Figure 4: Density of pellets with line of best fit. Vertical bars depict standard deviation for the population.

Dueck et al. (2016) completed similar testing in which biomass pellets were made with oat hull grinds supplemented with microalgae. Pellets were compressed at the same rate of 100 mm/min using a 12.3 mm die. The results of their study showed that density also increased as algae content increased. The density values of pellets made with 0 and 20% microalgae had comparable densities compared to pellets made with 0 and 20% manure. At 40% algae the density was calculated to be 1051 kg/m^3, while pellets with 40% manure content had a calculated density of 1027 kg/m^3.

Diametral Compression Test

The increase of manure content proved to provide a positive impact of the strength of the pellets during diametral compression testing. The mixture of oat straw and manure could be mathematically modelled using an exponential trend line. The exponential mathematical model had a high positive correlation with an R^2 value of 0.9830. In this mathematical model, an increase in manure content causes an increase of the stress at failure. Originally it was observed that the stress at 40% manure content was higher than the stress at 60% manure content therefore an additional five pellets of 40% manure were tested to achieve a more accurate average stress at failure,. Due to the procedure in which the oat straw and manure were added to the single unit pelletizer, it was not possible to achieve a mixture in which all of the manure was evenly distributed throughout the pellet, which could be the cause of a higher standard deviation in the 40 and 60 % sample sets.

The equation for the exponential trend line was defined by graphing software Graph (Graph version 4.4.2 Build 543, Ivan Johnson, http://www.padowan.dk) and can be found in Eq. 6.

$$\sigma(x) = 235.4245 \cdot 1.0227^{x} \tag{6}$$

Where σ is the stress [kPa] and x is the percent content of manure present in the sample.

Figure 5 plots the mean value of the stress calculated from the diametral compression test with the error bars depicting the standard deviation of the population from the mean. The R^2 value for the exponential mathematical model can also be seen in Figure 5.

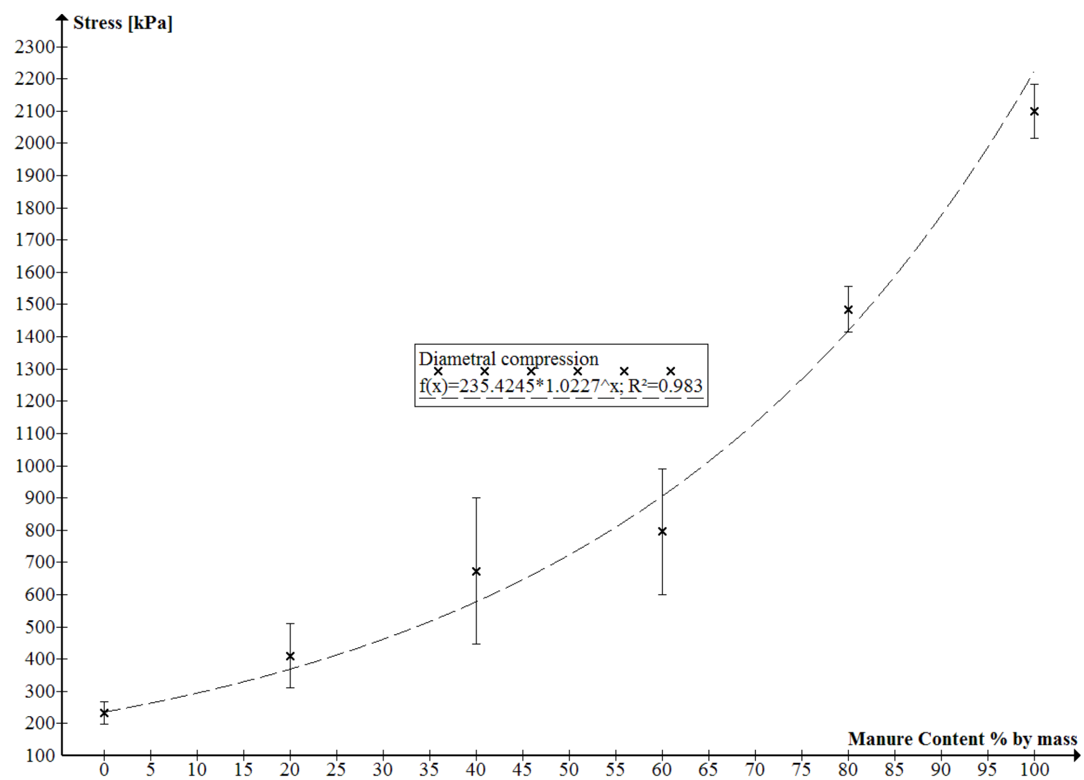

Figure 5: Mean stress at pellet failure with exponential trend line. Vertical bars depict standard deviation of the population.

In comparing the results of this study to Dueck et al. (2016) the stress at failure of pellets with 40% algae compressed at 100 mm/min ranged from about 375 kPa to 625 kPa (varying pellet conditioning). Using Eq. 6, the calculated stress value for pellets with 40% manure was 577 kPa. Further comparison to the results from Dueck et al. (2016), pellets supplemented with manure had higher strength than pellets with microalgae at percentages of 0 and 20%. Dueck et al. (2016) used commercially available oat hull pellets to use as a reference strength of 780 kPa. From the data collected in this study the 60% manure pellets had values higher than the commercially available pellets.

Specific Energy

The mixture of manure and oat straw followed a linear trendline with an R^2 value of 0.9591, with high negative correlation. Increasing the manure concentration caused a linear decrease in specific energy of the pellets with a slope of -0.0223. The highest specific energy was a pellet mixture of 0% manure, while the lowest was the pellet with 100% manure. It is possible to determine the expected specific energy for each concentration of manure using Eq. 7 defined by graphing software Graph (Graph version 4.4.2 Build 543, Ivan Johnson, http://www.padowan.dk). Figure 3 shows the mean value of the samples for the specific energy testing of each ratio set.

$$ED = -0.0223x + 16.0069 \tag{7}$$

Where ED is the specific energy [MJ/kg] and x is the % content of manure present in the sample.

Using Eq. 7, the predicated values for 20, and 80% manure content were calculated as 15.62 and 14.22 MJ/kg respectively. From the data we can conclude that the manure had a lower combustion value than that of pure oat straw. Although increasing the manure content in the pellets increased the strength and density of the pellets, the combustion values decreases as manure was added. There was an overall decrease of 13.7% in the specific energy of the pellets when manure concentration was increased from 0 to 100%.

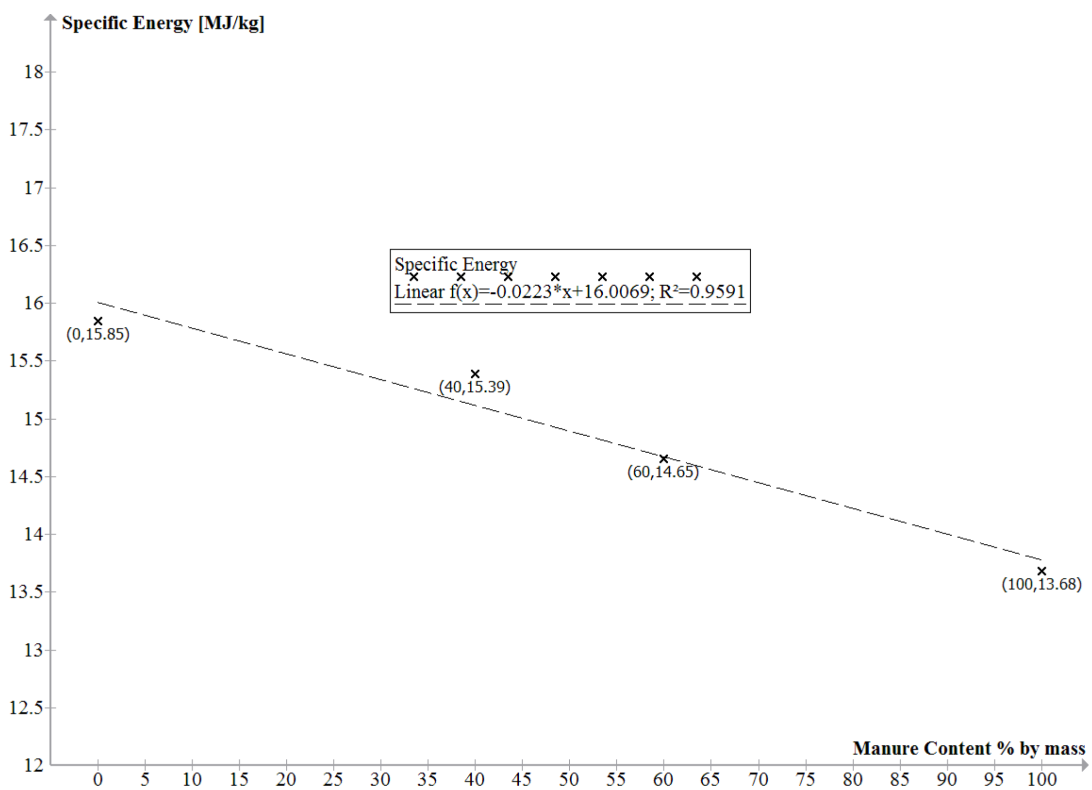

Figure 6: Mean specific energy data points for pellets with linear line of best fit.

Dueck et al (2016) completed identical combustion value testing for their pellets supplement with microalgae. Their data resulted in a positive linear correlation compared to the negative correlation seen in this study. Using their linear trendline equation, it was calculated that pellets with 40% algae, had a specific energy of 18.64 MJ/kg compared to

15.39 MJ/kg for biomass pellets with 40% manure. It is apparent that the microalgae had a higher combustion value because of their higher lipid content compared to the manure used in this study. By comparison of other fuel sources, coal (lignite) has a specific energy values of 14.7 to 19.3 MJ/kg (Deuck et al. 2016), placing the 40 and 60% manure pellets near the lower range of the specific energy of coal.

Conclusions

1) Manure concentration had a positive effect on the density of single pellets. The data followed a linear trendline with a high positive correlation of 0.99. Density of pellets were calculated to rise from 918 to 1190 kg/m^3 when manure content was increased from 0 to 100%.
2) Manure concentration had a positive effect on the strength of the pellets, increasing exponentially with a correlation of 0.98. The strength of pellets had a calculated increase from 235 to 2221 kPa when manure was increased from 0 to 100%.
3) Manure concentration had a negative effect on the specific energy of the pellets, following a linear trendline with a high correlation of 0.96. Pellets made of 0% oat straw had a specific energy of 15.85 MJ/kg while pellets made with 100% manure had a specific energy of 13.68 MJ/kg.

In conclusion, the optimal manure concentration for densified oat straw pellets supplemented with manure be 40 to 60%. Biomass pellets with 40 and 60% manure concentration had comparable density and strength values to commercially available pellets while the specific energy of the pellets was comparable to lower range values of coal (lignite).

Recommendations

It is recommended that the pellets be compressed at a higher pressure (>90 MPa), which would mean that a larger load cell would need to be attached to the UTM. The rate of compression could be increased alongside with the increased pressure to allow for stronger and denser pellets which would allow for higher combustion values for pellets of identical size. It is also recommended that the methodology for which the material and binding agent are mixed together needs to be improved upon to ensure a more uniform sample. It is also recommended that an additional study with larger diameter pellets be conducted to determine the optimal size for biomass pellets that allows for high strength, density and specific energy.

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Appendix A

Table A-1: Raw data for densification of pellets.

Manure	Diameter	Mass	Thickness	Density
%	mm	g	mm	kg/m^3
0	12.3	0.90	8.2	923.69
0	12.3	1.00	9.7	867.62
0	12.3	1.00	9.1	924.82
0	12.3	1.03	9.8	884.53
0	12.3	1.03	9.2	942.21
20	12.3	0.99	8.3	1003.82
20	12.3	1.00	8.8	956.35
20	12.3	0.98	8.5	970.30
20	12.3	1.00	8.5	990.10
20	12.3	0.98	8.6	959.02
40	12.3	0.99	7.7	1082.04
40	12.3	1.00	7.8	1078.96
40	12.3	0.95	7.4	1080.42
40	12.3	1.00	8.2	1026.33
40	12.3	0.99	7.9	1054.65
60	12.3	0.98	7.8	1057.38
60	12.3	0.99	7.7	1082.04
60	12.3	0.99	7.9	1054.65
60	12.3	0.99	7.7	1082.04
60	12.3	0.99	7.6	1096.28
80	12.3	0.98	7.2	1145.50
80	12.3	0.97	7.1	1149.78
80	12.3	0.97	7.1	1149.78
80	12.3	0.97	7.3	1118.28
80	12.3	0.98	7.1	1161.63
100	12.3	1.50	10.5	1202.27
100	12.3	1.48	10.5	1186.24
100	12.3	1.51	10.7	1187.66
100	12.3	1.51	10.8	1176.67
100	12.3	1.49	10.6	1182.99

Table A-2: Raw data of diametric load testing of pellets.

Manure	Diameter	Mass	Thickness	Max Load	Stress
%	mm	g	mm	N	kPa
0	12.3	0.90	8.2	37.653	237.66
0	12.3	1.00	9.7	38.970	207.94
0	12.3	1.00	9.1	47.111	267.95
0	12.3	1.03	9.8	34.277	181.03
0	12.3	1.03	9.2	47.669	268.18
20	12.3	0.99	8.3	96.412	601.21
20	12.3	1.00	8.8	60.639	356.65
20	12.3	0.98	8.5	55.849	340.07
20	12.3	1.00	8.5	69.585	423.71
20	12.3	0.98	8.6	54.850	330.11
40	12.3	0.99	7.7	159.099	1069.43
40	12.3	1.00	7.8	155.124	1029.34
40	12.3	0.95	7.4	131.157	917.35
40	12.3	1.00	8.2	85.269	538.21
40	12.3	0.99	7.9	97.260	637.21
60	12.3	0.98	7.8	84.177	558.56
60	12.3	0.99	7.7	136.215	915.61
60	12.3	0.99	7.9	93.380	611.79
60	12.3	0.99	7.7	119.076	800.40
60	12.3	0.99	7.6	160.215	1091.10
80	12.3	0.98	7.2	200.851	1443.83
80	12.3	0.97	7.1	209.786	1529.30
80	12.3	0.97	7.1	218.176	1590.46
80	12.3	0.97	7.3	194.870	1381.65
80	12.3	0.98	7.1	203.098	1480.55
100	12.3	1.50	10.5	422.684	2083.54
100	12.3	1.48	10.5	412.567	2033.67
100	12.3	1.51	10.7	462.143	2235.46
100	12.3	1.51	10.8	447.291	2143.59
100	12.3	1.49	10.6	409.987	2001.88

Table A-3: Raw data for Specific Energy of pellets.

Manure	**Mass**	**ΔT**	**Specific Energy**
%	g	K	MJ/kg
0	1.00	1.5962	15.78
0	0.97	1.5622	15.92
40	0.97	1.4849	15.14
40	0.99	1.5685	15.64
60	0.99	1.4843	14.83
60	0.97	1.4203	14.48
100	1.49	2.0326	13.49
100	1.46	2.0490	13.88