Analysis of Factors Affecting *Miscanthus* Pellet Production and Pellet Quality using Response Surface Methodology

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The *Miscanthus sacchariflorus* strain Goedae-Uksae 1 has recently been developed as an energy crop, because of its rapid growth, ease of culture, and large size. In this study, *Miscanthus* pellets were investigated for further practical use of renewable resources. The pellets were produced on a pilot scale and their characteristics and quality were investigated. Moisture content, density of raw materials, and die ratio (L/D) were determined to be the main factors affecting *Miscanthus* pelletizing. Thus, a central composite design using response surface methodology (RSM) was applied to optimize conditions for standard grade *Miscanthus* pellet production. The optimal values predicted by the model equation were confirmed by the experimental data. The optimum ranges of parameters for pellet production were identified as moisture content, 20 to 25%; density of raw materials, 240 to 300 kg/m^3; and die ratio, 4.5:1 to 5.0:1. Under these conditions, *Miscanthus* pellets were produced meeting the standards of qualities, such as size, bulk density, durability, moisture content.

Keywords: Solid fuels; Lignocellulosic pellets; Miscanthus pellets

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INTRODUCTION

The search for alternative fuels due to recent oil crises and environmental concerns has raised interest in lignocellulosic biomass utilization for solid and liquid biofuels (Kumar *et al.* 2008; Lynd *et al.* 2006; Rubin 2008). Pellets, one of type of biomass fuel, have been increasingly produced in recent years due to their relative ease of use, with expanding biomass boiler markets mainly in Europe and North America (Sikkema *et al.* 2010). High-quality pellets should be dry (moisture content less than 10%), hard, and durable with little ash remaining after combustion (Tumuluru *et al.* 2010). Wood pellets are the most commonly used globally (Pirraglia *et al.* 2010). However, recent studies have evaluated production of agripellets or lignocellulosic pellets from a variety of bio-wastes, such as herbaceous, fruit, and blended biomass, depending on agricultural availability (Adapa *et al.* 2009; Gilbert *et al.* 2009; Salema and Ani 2012; Serrano *et al.* 2011). Most studies have focused on process parameters related to pelletization of the biomass and associated impacts on pellets quality. Generally, these parameters include particle size, moisture content, steam conditioning, pelletizing pressure/speed, pellet mill operating conditions, and pelletizing temperature (Kaliyan and Morey. 2009).

In South Korea wood pellets are mostly used in greenhouse heating, and they are highly dependent on imports rather than local production (Goh *et al.* 2013). In 2013 the total production of wood pellets was 0.05 million tonne (metric), while total imports was 0.48 million tonne (Korea Forest Research Institute). The pellet market is expected to grow rapidly along with government's renewable energy portfolio standards in 2012 (IEA Bioenergy update, 2014; available at http://dx.doi.org/10.1016/S0961-9534(14)00041-5). The main challenge in utilization of biomass fuels would be a stable supply of raw materials (Erb *et al.* 2012). Thus, *Miscanthus sacchariflorus* Goedae-Uksae 1, "giant *Miscanthus* in Korean" was developed as energy crop and is now being cultivated in some parts of Korea. The main characteristic of Goedae-Uksae 1 is massive yield, 30 ton/ha (Moon *et al.* 2010). Indeed, *Miscanthus* has been considered a key energy crop since 1980 due to its economic and environmental benefits (Brosse *et al.* 2012; Chung and Kim 2012; Lewandowski *et al.* 2000). Recently, Lehmann *et al.* (2012) demonstrated that addition of *Miscanthus* to wood pellets improved their quality as well as reducing costs. However, there is little information on production of high-quality pellets using pure *Miscanthus* because of its unique mechanical and chemical properties that differ from those of woody biomass (Kallis *et al.* 2013).

Therefore, the aim of this study was to overcome the import dependency of wood pellets and evaluate the feasibility of *Miscanthus* pellets production in Korea, as we examine pelletizing factors suitable for production of high quality *Miscanthus* pellets through practical and theoretical data analysis.

MATERIALS AND METHODS

Materials

Samples of *Miscanthus sacchariflorus* Goedae-Uksae 1 (An *et al.* 2013) were harvested from Sancheong, Kyoengsangnam-do, Korea in November 2011 and packed in a round bale with a diameter of 1.2 m. Two tonnes of material was transported to a pilot-scale plant at the Bioenergy Crop Research Center in Muan, Korea where the pelletizing experiments were performed. The samples were then chopped to a similar length (~5 cm) using a tub grinder (Tomotech Ltd., Seoul, Korea). Moisture content of the chopped samples was measured at approximately 7.5%. The samples were divided into 5 groups depending on moisture content by adding 2.5 to 22.5 % of water for the pelletizing experiments (Table 1). A 50 kg sub-batch of each group was prepared for the optimization for pelletizing of *Miscanthus*. Pine sawdust was purchased from a local market in Muan, Korea.

Table 1. Coded Values for Each Variable of the Central Composite Design

Factors	Coded levels				
Moisture (%)	10	15	20	25	30
Density of raw materials (kg/m^3)	220	240	260	280	300
Die ratio (L:D*)	4.00:1	4.25:1	4.50:1	4.75:1	5.00:1

*L, length of die; D, diameter of die

Pellet Production

Figure 1 illustrates the pelletizing process at a pilot scale. The chopped samples were finely ground using a 20-hp hammer mill with 3-mm screens (Sunbrand Industrial, Inc., Chun-nam, Korea). Pelletizing experiments were continuously conducted using a 25-hp ring flat die pellet mill (Sunbrand Industrial, Inc.). Five flat dies that differed in the size of cylindrical holes were used to optimize pelletizing conditions; 24, 25.5, 27, 28.5, and 30.0 of length with 6 mm of diameter. Thus, the ratio between the length (*L*) and the diameter (*D*) of the press die were from 4.0:1 to 5.0:1 (Table 1). Meanwhile, the die for pre-compaction was 24.0 × 8.0 mm (3.0:1, *L*:*D*) at size, and pre-compaction was controlled by feed rate (40 to 80 kg/h), resulting in 240 to 280 kg/m^3. The die temperature was measured approximately at 105 °C during the pelletizing. Produced pellets were cooled down at room temperature at pilot plant. As a positive control to verify our pelletizing processes, pine sawdust was pelletized prior to the *Miscanthus* experiments, because wood pellets were comparatively well known for its pelletizing conditions.

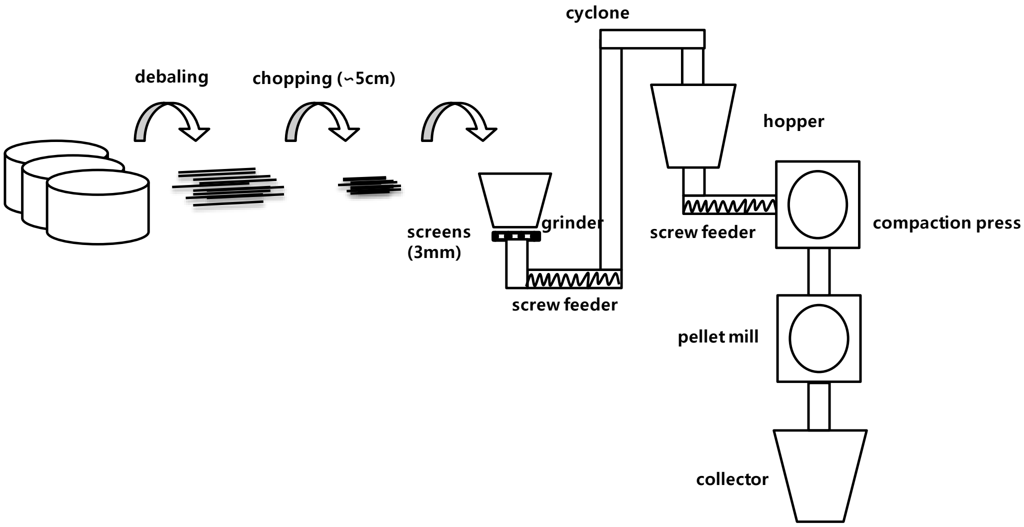

Fig. 1. Schematic of the *Miscanthus* pelletizing process

Response Surface Methodology and Statistical Analysis

Response surface methodology (RSM) is a collection of mathematical and statistical techniques for empirical model building in order to optimize the independent variables through a series of tests (Steinberg and Bursztyn 2010). Observations made throughout *Miscanthus* pellet experiment suggested that density of *Miscanthus* should be a main factor in pelletizing as compared with that of saw dust. In addition, moisture contents and die ratio (*L*/*D*) were determined important factors because pelletizing with a various combination of moisture and die size was unsuccessful. Other factors, such as particle size, stream conditioning, and pelletizing temperature were held constant throughout the study, although they are understood to have significant effects on pelletizing. Bulk density and durability were treated as the response variables affected by the three independent variables in this study, because densification of biomass and its durability would be main purposes of pellet production. Experimental ranges and levels of the independent variables, moisture content (X_1), density of raw materials (X_2), and die ratio (X_3), are shown in Table 1. Twenty set of independent experiments was conducted for the response surface designs and regression of the acquired data was conducted using Design Expert® v. 8.1.

Physical and Chemical Analysis

To determine whether the produced pellets met the standards for quality of pellets, physical and chemical analyses were conducted (Tumuluru *et al.* 2010). The moisture content of the samples was measured with a moisture analyzer (HR83 halogen moisture analyzer; Mettler-Toledo; Swiss). Bulk density of produced pellets was measured using a 5-L cube. Durability was estimated using a modified tumbling method (DIN EN 15210-1) with a tumbler (Sunbrand Industrial, Inc.), representing percent durability. Briefly, 50 g of pellets were placed in a tumbling can and tumbled at 50 rpm for 10 min. Then, the tumbled pellets were sieved through 1.5 mm. The chemical composition of the *Miscanthus* biomass and pellets was analyzed using an elemental analyzer (TruSpec CHN; Leco Corp., St. Joseph, MI, USA), ion chromatography (881-compact-IC-Pro, Metrohm, Swiss), and a sulfur analyzer (SC-432DR; Leco Corp.). Electricity consumed during the process was estimated with a wattmeter. The higher heating value (HHV) of the pellets was measured using a calorimeter (6320EF; Parr Instrument Co., Moline, IL, USA). Lignin analysis was performed according to NREL/TP-510-42618 (Sluiter *et al.* 2008), and hardness was measured using micro hardness analyzer (TA.XTplus Texture Analyser, Surrey, U.K.). Raw, chopped, ground, and pelletized *Miscanthus* for physical chemical analysis included their own moisture content. Elemental analysis of pellet was conducted under dry basis.

Scanning Electron Microscopy (SEM)

To observe the structural changes that are important factors of bulk density and durability, SEM analysis was conducted (TM-1000; Hitachi, Tokyo, Japan). Samples were mounted on aluminum sample stubs and observed under a vacuum at an acceleration voltage of 15 kV.

RESULTS AND DISCUSSION

Effect of Pre-compaction on *Miscanthus* pelletizing

Prior to *Miscanthus* biomass pelletizing, pine sawdust pelletizing was conducted to determine whether the experimental processes (Fig. 1) were valid. Pelletizing conditions were 3 mm particles, 12.2 ± 0.15% moisture, 255.4 ± 6.51 kg/m^3 density of particles, 4.5:1 die ratio (L:D). Finally, feed rate was constantly regulated at 130 g/min. In results, the sawdust was successfully densified as pellet, and their characteristics were 656.1 ± 11.0 kg/m^3 of bulk density, 7.7 ± 0.65% moisture, and 98.1 ± 0.22% durability. Thus, the present experimental processes could be verified by the conditions reported previously in sawdust pelletizing (Kaliyan and Morey 2009; Wilson 2010). Next, the processes were applied to produce *Miscanthus* pellets. However, *Miscanthus* biomass pellets did not form under the conditions given above. Considering the factors potentially affecting *Miscanthus* pelletizing compared to the sawdust, the density of the raw materials was considered the main requisite (Larsson and Rudolfsson 2012). Thus, the traits of raw materials were compared between *Miscanthus* and the pine sawdust biomass such as weight, volume, and density (Fig. 2). The cortex is a main component of *Miscanthus*, and it occupied 66.0 ± 1.19% in total weight and 59.8 ± 1.38 % in total volume of *Miscanthus* (Fig. 2a). The density of cortex was comparatively lower (158.1 ± 11.37 kg/m^3), whereas the node had the highest density (319.6 ± 11.37 kg/m^3) in *Miscanthus*, but it only occupied less than 15% of the total weight and volume of

Miscanthus. Therefore, the mean of particle density of *Miscanthus* was 175.0 ± 8.00 kg/m^3, representing 33.5% lower than that of sawdust, 267.0 ± 5.00 kg/m^3. By comparison, the bulk density of lignocellulosic biomass is typically 40-200 kg/m^3 (Adapa *et al.* 2009; Mani *et al.* 2003).

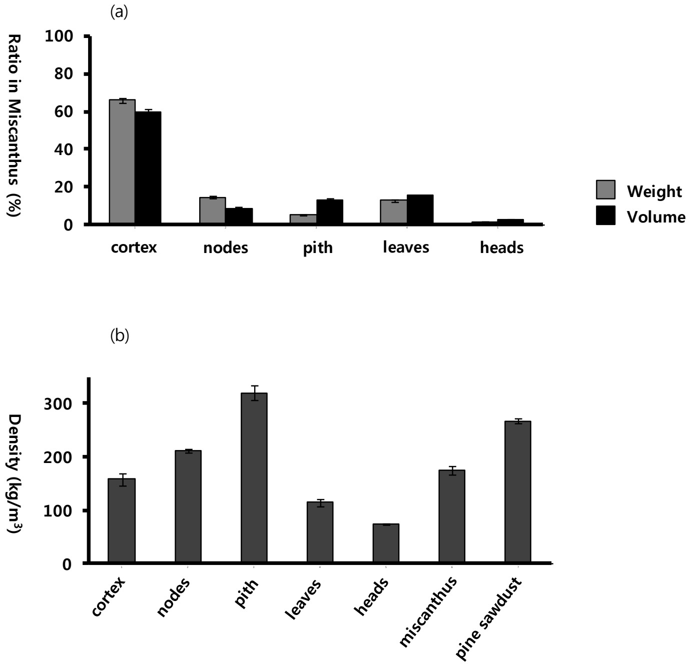

Fig. 2. Density comparison between *Miscanthus* and pine saw dust biomass: (a) Ratio of each tissue in *Miscanthus*; and (b) Density of *Miscanthus* and pine sawdust biomass. Bars represent standard deviation from five independent measurements

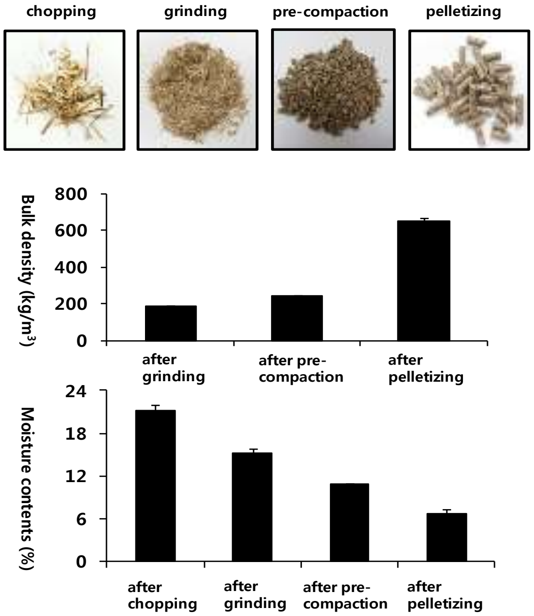

Fig. 3. *Miscanthus* pellet by increasing bulk density. Bars represent standard deviation from three independent measurements

Larsson *et al.* (2008) suggested pre-compaction as a means of producing pellets with low-density materials. A compression die (24.0 × 8.0 mm, L:D, 3:1) was therefore applied for pre-compaction in our experiment, in advance to *Miscanthus* pellet production. As feed rate increased from 40 to 80 kg/h in pre-compaction, 240 to 280 kg/m^3 of particle density of *Miscanthus* was achieved. Following the pre-compaction, *Miscanthus* pellets could be produced under the conditions stated earlier (Fig. 3). However, pellets were not produced at a density less than 200 kg/m^3. The density of the raw biomass might be positively correlated with pellet formation to some degree. For this, further studies might be required to analyze the pellet formation conditions, such as particle size, steam conditioning, pelletizing pressure/speed and pelletizing temperature, and binders, together with different density. The lignin content (24.1 ± 0.33%) in *Miscanthus* Goedae-Uksae 1 seems not to have affected pellet formation after pre-compaction. Lower lignin contents along with low density have been reported to cause weak bonding between particles in lignocellulosic pellet formation, and thereby lower durability (Mani *et al.* 2006). To investigate the pelletizing conditions for pellet bulk density and durability further conditions were investigated.

Optimization of Parameters for *Miscanthus* Pelletizing

Additional factors affecting production of pellets were investigated beyond the density of raw materials. In previous studies, particle size (or hammer mill screen size) was set variously at 1.58 to 6.35 mm, resulting in 37.0 to 97.4% durability depending on the feed biomass type (Mani *et al.* 2006). Although finer particles resulted in higher pellet durability, fine grinding is often undesirable due to increased costs. In the present study, the particle size of *Miscanthus* was fixed at less than 3 mm based on the pine sawdust pelletizing experiment. In addition, die temperature, steam conditioning, and additives are factors that potentially improve durability by increasing bonding between the particles (Gilbert *et al.* 2009). The die temperature was measured at 105 °C in the stationary stage when pellets were stably produced. The temperature probably induced lignin softening and promoted the binding process in pellets (Tabil and Sokhansanj 1996). The moisture content of chopped *Miscanthus* was regulated at 20 to 25% by adding water, and no steaming and additives were introduced during pelletizing. The produced pellets were allowed to solidify and strengthen at the temperature of the pilot plant (<20 °C) without a cooling system.

Moisture content, density of the raw material, and die ratio (L/D) were regarded as important factors in the pilot trials, because these largely influenced the feasibility of pellet formation. For instance, a combination of 15% moisture content with either lower density (180 kg/m^3) or smaller/larger die ratios (L/D of 3.75:1 or 5.25:1) resulted in no pellet formation, blocking the pellet mill die, and/or lower durability of the pellets. The three variables were therefore applied to RSM for further analysis. The response by three variables was defined as bulk density and durability. The central composite design (CCD) for a series of run is shown in Table 2. Analysis of variance (ANOVA) results indicating the effects of the variables are summarized in Table 3. Finally, the following ternary quadratic equation was obtained:

$$Y_{\text{bulk density}} = 626.3 + 20\ X_1 + 11.2\ X_2 + 7.5\ X_3 - 36\ X_1 - 8.4\ X_1X_3$$

where X_1 is the moisture content (%), X_2 is the density of raw materials (kg/m^3), and X_3 is the die ratio (L/D).

Table 2. Central Composite Design Matrix and the Results for the Measured Responses

Exp #	Factors			Measured response	
	Moisture content (%)	Density of raw materials (Kg/m^3)	Die ratio (D/L)	Bulk density (Kg/m^3)	Durability (%)
1	15	240	4.25:1	589	97.2
2	25	240	4.25:1	608	96.0
3	15	280	4.25:1	594	97.4
4	25	280	4.25:1	617	97.0
5	15	240	4.75:1	611	95.4
6	25	240	4.75:1	581	97.3
7	15	280	4.75:1	620	96.9
8	25	280	4.75:1	625	97.3
9	10	260	4.50:1	391	35.9
10	30	260	4.50:1	543	94.7
11	20	220	4.50:1	568	96.7
12	20	300	4.50:1	624	97.4
13	20	260	4.00:1	599	94.3
14	20	260	5.00:1	644	94.9
15	20	260	4.50:1	622	96.4
16	20	260	4.50:1	627	97.0
17	20	260	4.50:1	617	96.9
18	20	260	4.50:1	622	96.9
19	20	260	4.50:1	622	97.1
20	20	260	4.50:1	626	97.3

Table 3. Regression Analysis of a Predictive Model for Optimization of the Bulk Density of *Miscanthus* Pellets

Factor	Coefficient estimated	Standard error	F-value	P-value
Intercept	626.3	7.355	12.27	0.0001*
X_1	20.0	6.781	8.70	0.0105
X_2	11.2	6.781	2.73	0.1207
X_3	7.448	6.781	1.20	0.2906
X_1 X_3	-36.0	5.200	47.92	< 0.0001*

R^2 = 0.8141, Adj. R^2 = 0.7478, CV=4.54%.

The ANOVA results showed that the quadratic model was reasonable (p = 0.0001). However, the R^2 and adjusted R^2 were 81.1% and 74.8%, respectively. The R^2 indicated somewhat lower reliability of the observed values. Moisture content was highly correlated with the bulk density, as shown in a previous study (Kaliyan and Morey 2009). For example, lowest and highest moisture content of 10% and 30% resulted in very much lower bulk density and comparatively lower durability (Table 2). Two-factor interaction between the moisture content and die ratio (L/D) produced the lowest p-values (p < 0.0001), identifying these two factors as the most significant parameters for bulk density after insignificant coefficients were removed from the model by backward elimination (Table 3). The relationships between the die ratio and moisture content are visualized in Fig. 5, showing optimal moisture content of 20 to 25% and a die ratio of 4.5:1 to 5.0:1.

Although the density is very important factor, it did not significantly affect *Miscanthus* bulk density in this experiment. This might be why the ranges of density were so narrow (220 to 300 kg/m^3), which all might be proper for pelletizing (Fig. 4). Therefore, the density of the particles was decided to be an important factor for pellet formation, because the pellet could be formed at density values between 220 and 300 kg/m^3. The bulk density of the produced pellets was nearly constant at more than 600 kg/m^3 except when the density of the raw materials was lowest at 220 kg/m^3. The moisture content of the pellets was on average less than 8.3% and their durability was more than 97.5% (Fig. 4). Meanwhile, when bulk density was satisfied with the standard (620 kg/m^3), so was durability. Thus, we omitted a second response, durability, in order to avoid confusion.

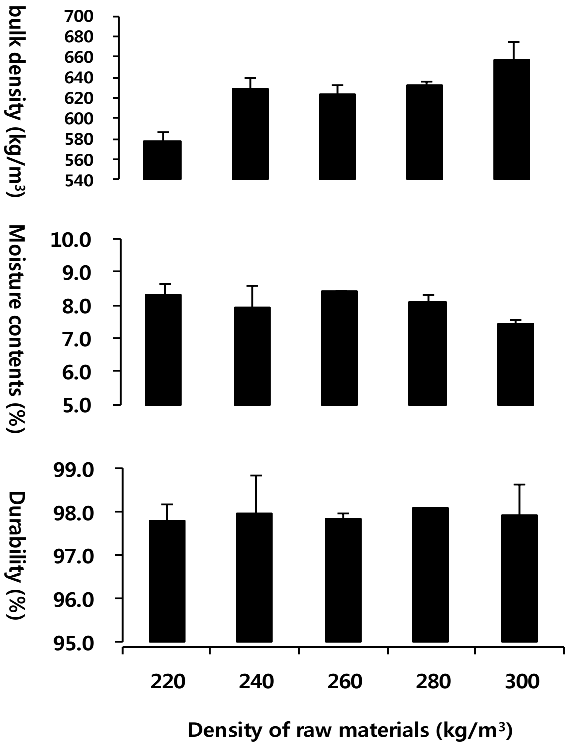

Fig. 4. Physical properties of the *Miscanthus* pellets at different the density of particles. Bars represent standard deviation from five independent measurements.

Verification of *Miscanthus* pelletizing based on predicted optimal values is shown in supplementary Table A1 (see appendix). The best theoretical conditions were 20% moisture content, 280 kg/m^3 density of raw materials, and 5.0:1 (L/D) die ratio for maximal bulk density. Meanwhile, the verified pelletizable ranges were moisture content 20 to 25%, density of raw materials 240 to 300 kg/m^3, and die ratio of 4.5:1 to 5.0:1. Stable wood pellets were generally formed over a range of moisture contents of 8 to 12% (Wilson 2010), while the moisture content for *Miscanthus* pellets ranged from 20 to 25% in this study. Pellets were not properly produced at less than 10% and more than 30% moisture content, which may represent a threshold (up to 30%). A similar result was observed in wafers of alfalfa hay, whose moisture content was 25% and above (Reece 1966). Indeed, it has been shown that the durability and strength of pellets increase with increasing moisture content until a threshold concentration is reached (Larsson and

Rudolfsson 2012). Meanwhile, the die ratio declined roughly with an increase in the moisture content up to 20% for maximal bulk density of pellet. In sum, it might be concluded that the density of the raw materials was the main factor affecting the feasibility of pellet formation, while the die ratio and moisture content were significant factors determining pellet bulk density and durability.

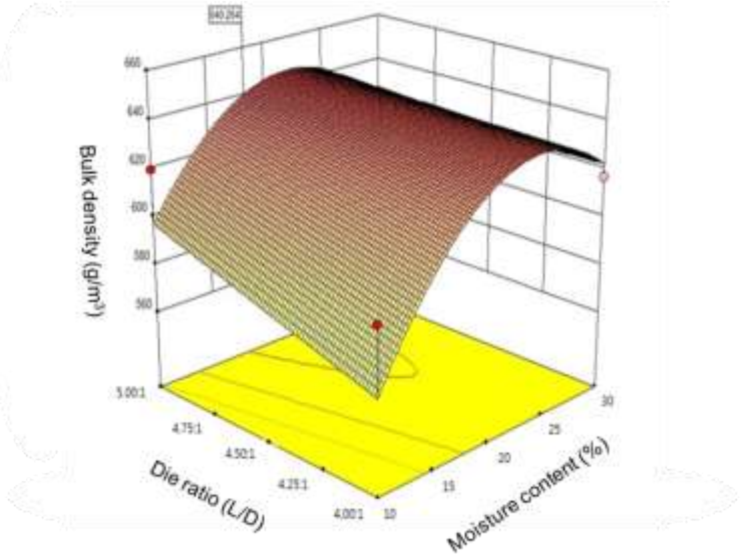

Fig. 5. Effect of die ratio and moisture content on bulk density of the pellets

Pellet Quality Evaluation

The physicochemical and mechanical properties of the *Miscanthus* pellets are summarized in Table 4. Pellets were produced under the 20% moisture content, 280 kg/m^3 density of raw materials, and 5.0:1 (L/D) die ratio. Bulk density was measured at 624 ± 9 kg/m^3. The net loss of biomass after pelletizing was less than 2.5%. The mean durability was 97.5%, and hardness was measured at 458.3 ± 22.6 N/mm^2. The ash content was slightly higher (2.2%) than that of wood due to the nature of *Miscanthus*. The carbon content of the pellets was significantly lower than that of wood pellets (generally 50%) (Carroll and Finnan 2012). This may reflect a somewhat lower higher heating value (HHV). Sulfur and chlorine, known as corrosive elements for boilers, were measured at 0.1% and 0.12%, respectively. These figures seemed to originate from the nature of *Miscanthus*. It was reported that a limited factor of herbaceous biomass for solid fuel is higher sulfur and chlorine (Monti *et al.* 2008). *Miscanthus* contains approximately 0.15% of sulfur and 0.13% of chlorine, while woody biomass contains 0.04% and 0.02%, respectively (Vassilev *et al.* 2010). This limitation could be overcome by developing of suitable boiler for *Miscanthus* pellet.

Overall, the *Miscanthus* pellets were fit for the 2nd grade standard (Korea forest service) in terms of diameter (6~8 mm), length (< 32 mm), bulk density (600 kg/m^3), durability, and moisture content (< 10%). However, lower calorific value should be increased in further study (*e.g.* wood or charcoal addition).

Table 4. Physical and Chemical Characteristics of the Pure *Miscanthus* Pellets

Pelletizing (%)	Size		Bulk density (kg/m^3)	Durability (%)	Moisture contents (%)
	Diameter (mm)	Length (mm)			
99.4±0.2	20.3±2.7	5.5±0.7	624±9	97.8±0.2	8.4±0.06

Carbon (%)	Hydrogen (%)	Nitrogen (%)	Oxygen (%)	Sulfur (%)	Chlorine (%)	Ash (%)	HHV[1] (Kcal/kg)
44.0	5.8	0.3	50.0	0.1	0.12	2.2±0.2	4,025±6.38

[1] HHV, higher heating value.

Scanning Electron Microscopy (SEM) Analysis

To observe pelletizing performance indirectly, SEM analysis was performed before and after pelletizing. As shown in Figure 6, chopped samples of 5 cm length were observed to consist of a composite structure of individual fibers, and the ground samples were comparatively relaxed in structure, probably due to milling. On the other hand, the surface of the pellet was tightly compacted, and the fibers seem to be destroyed. Thus, *Miscanthus* pelletizing was shown to be effective in producing dense and physically stable pellets.

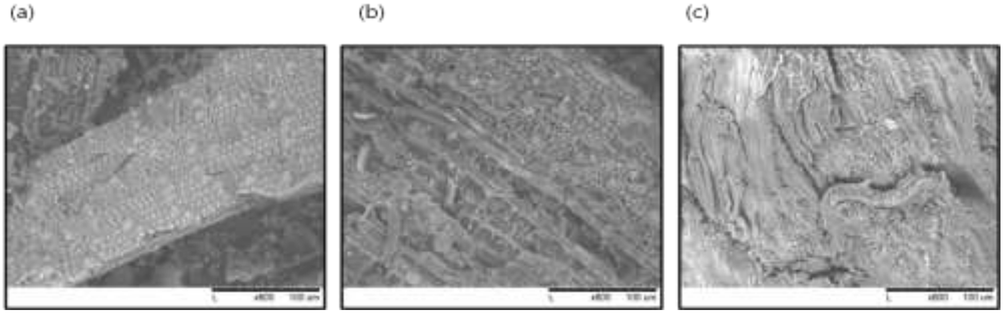

Fig. 6. SEM analysis (x800) of *Miscanthus* before and after pelletizing; cross-section views of (a) chopped, (b) ground, and (c) pelletized samples

CONCLUSIONS

1. Optimization of pelletizing factors was analyzed by using response surface methodology (RSM). The density of the raw materials (240 to 300 kg/m^3) was found to be a key factor for *Miscanthus* pellet formation, while the die ratio (L/D, 4.5:1 to 5.0:1) and moisture content (20% to 25%) important parameters, influencing the bulk density and durability of the pellets.

2. Overall, the *Miscanthus* pellets were fit for the 2nd grade standard (Korea) in terms of pellets size, bulk density, durability, moisture content, ash content and chemical composition. However, further investigation needs in order to overcome the lower calorific value and higher ash contents along with combustion test.

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Supplementary Table 1. Verification of *Miscanthus* Pelletizing Based on Predicted Optimal Values

Moisture content (%)	Density of Raw materials (%)	Die ratio (L/D)	Bulk density of pellet (kg/m^3)
20	220	4.75:1	601
20	220	4.75:1	608
20	220	5.00:1	616
20	240	4.25:1	605
20	240	4.50:1	612
20	240	4.75:1	620
20	240	5.00:1	627
20	260	4.00:1	608
20	260	4.25:1	616
20	260	4.50:1	623
20	260	4.75:1	631
20	280	5.00:1	638
20	280	4.00:1	620
20	280	4.25:1	627
20	280	4.50:1	635
20	280	4.75:1	642
20	280	5.00:1	650
20	300	4.00:1	621
20	300	4.25:1	638
20	300	4.50:1	646
20	300	4.75:1	653
20	300	5.00:1	661
25	240	5.00:1	603
25	260	4.75:1	606
25	260	5.00:1	614
25	280	4.25:1	603
25	280	4.50:1	610
25	280	4.75:1	618
25	280	5.00:1	625
25	300	4.00:1	606
25	300	4.25:1	614
25	300	4.50:1	621
25	300	4.75:1	629
25	300	5.00:1	636