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Increased sugar yield from pre-milled Douglas-fir forest residuals with lower energy consumption by using planetary ball milling

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ABSTRACT

Impact of planetary ball milling on pre-milled wood fiber was studied to improve efficiency of energy consumption for bioconversion using post-harvest forest residuals. Crystalline cellulose decreased from 40.73% to 11.70% by ball milling. Crystallinity index of ball milled wood samples had a negative correlation with glucose yield ($r = -0.97$, $p < .01$), xylose/mannose ($r = -0.96$, $p < .01$), and a positive correlation with median particle size ($r = 0.77$, $p < .01$). Range of glucose yield and xylose/mannose yield for ball milled samples was found to be 24.45–59.67% and from 11.92% to 23.82%, respectively. Morphological changes of the lignocellulosic biomass were observed; the compact fiber bundles of the forest residuals were cleaved to smaller particles with lower aspect ratio with increasing intensity of ball milling. The required energy ranged from 0.50 to 2.15 kWh/kg for 7–30 min of milling respectively.

1. Introduction

[B]Consumption of fossil fuels which are the major energy resource in the world is growing due to the developing economic and industrial areas all over the world with increasing demands of energy (Sun and Cheng, 2002).

Lignocellulosic biomass such as wood residuals, grass, wheat straw, corn stover, and barley hull is an abundant, low-cost and practical alternative source of fossil fuels in comparison to starch, and oil-based biomass (Haghighi Mood et al., 2013; Shi et al., 2015; Sun and Cheng, 2002; Yoo et al., 2011).

Lignocellulosic biomass possesses cellulose, hemicellulose, and lignin, as major components and the bioconversion of lignocellulosic biomass is more difficult than other biomass resources (Himmel et al., 2007). Therefore, pretreatment is an essential step for lignocellulosic biomass to gain higher digestible cellulose for enzymatic hydrolysis. Lignin and hemicellulose interconnected with cellulose have to be detached from cellulose for better cellulose accessibility for the enzymes through pretreatment (Haghighi Mood et al., 2013; Sun and Cheng, 2002). Various pretreatment methods for lignocellulosic resources have been developed including biological, physical, chemical, physico-chemical and combination methods (Shi et al., 2015). Pretreatment methods if used must be economical for the overall biofuel processing and practically feasible in increasing sugar yield (Yoo et al., 2011).

Douglas-fir is an abundant byproduct of the logging and lumbering industry. Significant research work has been reported on the conversion of the Douglas-fir wood with various pretreatments for biofuels. (Biswas et al., 2015; Du et al., 2017; Jiang et al., 2017a; Liu et al., 2016; Mais et al., 2002; Zhu et al., 2015). Majority of the research has been focused on chemical pretreatments of lignocellulosic biomass such as acid treatment (with sulfuric acid), alkaline treatment, organosolv and other treatments (Haghighi Mood et al., 2013; Sun and Cheng, 2002). However, these pretreatments can generate inhibitors as byproducts of the chemical reactions and need to be removed from the treated biomass for the next steps.

Comminution is considered as an important pretreatment because this can decrease transportation cost of feedstock as well as storage fee on the pulverized feedstock (Barakat et al., 2014). Further, this can help reduce the hydrolysis time, as the smaller particle sizes of the biomass can provide better access to the enzymes. Hammer milling is broadly used for pulverization of biomass due to its high size reduction ratio and easy adjustment of the particle size range (Kratky and Jirout, 2011). If size reduction process is divided into two stages, hammer mill can belong to coarse grinding (Naimi et al., 2006). Air classifier mill divides the feed material into a coarse and a fine fraction by using air, which results in saving energy, avoiding over-grinding, enhancing

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[B] M7: Ball milling for 7 min; BM8: Ball milling for 8 min; BM10: Ball milling for 10 min; BM20: Ball milling for 20 min; BM30: Ball milling for 30 min.

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quality of product and unit capacity (Galk et al., 1999; Wang and Forssberg, 2007).

Ball milling which is a mechanical pretreatment is one of the common means of comminution to reduce biomass crystallinity as well as particle size for increasing surface area (Du et al., 2017; Gao et al., 2017; Gong et al., 2016; Khan et al., 2016; Lee et al., 2017; Vaidya et al., 2016). These factors lead to a decreased degree of polymerization, leading to a reduction of hydrolysis time and increase in sugar yield as well as reducing energy inputs (Lin et al., 2010; Mais et al., 2002). Lee et al. (2017) reported that planetary mill pretreatment effectively decreased particle sizes and crystallinity of corn stover. However, ball milling processes demand high energy consumption, which has to be approached by pragmatic means (Inoue et al., 2008).

There are many published reports on different milling techniques for increasing sugar-conversion yield and reducing particle size and crystallinity of lignocellulosic biomass (Barakat et al., 2014; Haghighi Mood et al., 2013; Sun and Cheng, 2002; Vaidya et al., 2016). However, there is a lack of information on the multiple step pulverization of lignocellulosic biomass, with specific consideration given to energy consumption. Hammer mill might be useful to grind the biomass into coarse particles followed by air classifier mill as intermediate milling process of the coarse fraction. Air classifier mill would prevent over-grinding, leading to energy savings. These two preliminary milling processes could help reduce the total energy consumption of the overall milling process. Ball milling is an energy-intensive process in mechanical comminution, although it's efficiency in decreasing crystallinity of cellulose may be effective as fine milling process (Zakaria et al., 2014). Thus, it is ideal to use preliminary milling processes before ball milling (Shi et al., 2015).

Therefore, the objective of this study was to develop a multi-step milling processing that is a combination of hammer milling and air classifier milling as preprocessing steps followed by optimizing the ball milling processes. The overall goal of this study was to develop an efficient combination milling process with minimal total energy input and higher sugar yields.

2. Materials and methods

2.1. Raw materials

Douglas-fir forest residuals were obtained from the Weyerhaeuser Co. (Seattle, WA, USA), from southwest Oregon. The residuals were pulverized through two pre-processing steps, using i) hammer mill (EMF-24,115-TFA, Bliss Industries, Ponca City, OK, USA) with 45.72 mm screen for a coarse milling followed by ii) air classifier mill (Mikro-ACM® Model 15, Hosokawa Micron Powder Systems, Summit, NJ, USA) as an intermediate milling step, before being subjected to ball milling process.

2.2. Ball milling

Ball milling was performed using a planetary ball mill (PQ-N20, Across International, Livingston, NJ, USA) equipped with four, 5 L capacity jars made of stainless steel. Sample (250 g) was loaded into each jar with three different sized stainless-steel balls. Each jar contained balls with diameters 6 mm, 10 mm, and 20 mm in quantities of 4501, 1011, and 15, respectively. Rotation speed and the rotation ratio were kept constant at 270 rpm and 1: − 2, respectively. Different milling times of 7, 8, 10, 20 and 30 min were used. Energy consumption (kWh/kg) was measured as accumulated active energy recorded by a three-phase power data logger (1735, Fluke, Everett, WA, USA).

2.3. Particle size analysis

Volumetric particle size distribution was measured using a laser diffraction particle size analyzer combined with Hydro LV wet dispersion unit (Mastersizer 3000, Malven Instruments Ltd, Worcestershire, UK) with a detection range of 0.01–3500 μm. The weighed sample, 50–150 mg (w.b), was dispersed in 10 ml of distilled water by a sonifier (Digital Sonifier 250, Branson Ultrasonic Corp., Danbury, CT, USA) with 20% of amplitude for 90 s and then loaded into the particle size analyzer for analysis. D90%, D50%, and D10% were utilized to analyze the particle size distribution (Jiang et al., 2017b).

2.4. X-ray diffraction analysis

Crystallinity index of the samples was measured using an X-ray diffraction (XRD) instrument (MiniFlex 600, Rigaku Americas Corp., The Woodlands, TX, USA). Milled wood particles placed on standard holder were scanned over the range of 2θ from 10 to 40° at a rate of 2 degree/min using Cu Kα radiation produced at 15 mA and 40 kV. The crystallinity was calculated by the Segal et al. (1959) method.

$$CrI\,(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

where, I_{002} is the main peak of diffraction intensity, and I_{am} is the minimum intensity between the main and secondary peaks.

2.5. Enzymatic hydrolysis, sugar yield and energy efficiency

Sugar yield was determined using the Laboratory Analytical Procedures (LAP) as reported by National Renewable Energy Laboratory (NREL) (Jiang et al., 2017a; Selig et al., 2008; Sluiter et al., 2011). Enzymatic hydrolysis was conducted at 5% solids content (1 g of wood residues per 20 ml solution) with a combination of Cellic CTec2 and HTec2 (Novozymes, Franklinton, NC, USA). 1 g sample (d.b.) was loaded into a 50 ml flask with 17.8 ml of 0.1 M sodium citrate buffer (pH 4.8), and 0.2 ml of 2% sodium azide was added. Following this, 1 ml of diluted enzyme solution containing 5% of CTec and 0.5% of HTec based on the dry wood was added into the flask. Flasks were then incubated in a shake incubator (GyromaxTM 747, Amerex Instruments Inc., Lafayette, CA, USA) with 180 rpm rotation at 50 °C for 72 h. After enzymatic hydrolysis, enzymes were deactivated by boiling for 15 min, and the hydrolysates were separated by using a centrifuge (AccuSpin Micro 17, Fisher Scientific, Pittsburgh, PA, USA) at 17,000*g* for 5 min. Supernatant from the centrifugation was diluted and then used for measuring sugar with an ion-chromatography (ICS-3000, Dionex Corp., Sunnyvale, CA, USA). Energy efficiency of the pretreatment was calculated by using he method reported by Zhu and Pan (2010). The amount of glucose was divided by the energy consumption.

$$\eta_{pretreatment}\ (kg\ glucose/kWh) = \frac{The\ amount\ of\ monomeric\ glucose\ (kg\ glucose/kg\ biomass)}{Energy\ consumption\,(kWh/kg\ biomass)}$$

2.6. Scanning electron microscopy (SEM)

Scanning electron microscope (FEI Quanta 200F, FEI Corp., Hillsboro, OR, USA) with a 20 kV accelerating voltage, was used to study the microstructure of the samples. Samples were mounted on cylindrical sample stubs and held with a conductive carbon tape and loaded into the SEM for observation. Images of the samples were captured at various locations of the sample at 200×, 500×, and 1000× magnifications.

2.7. Statistical analysis

Pearson correlation coefficient analysis was performed between product responses. Analysis of variance (ANOVA) followed by Fisher's LSD test was performed to compare the mean values concerning properties of ball-milled forest residuals on different ball milling time.

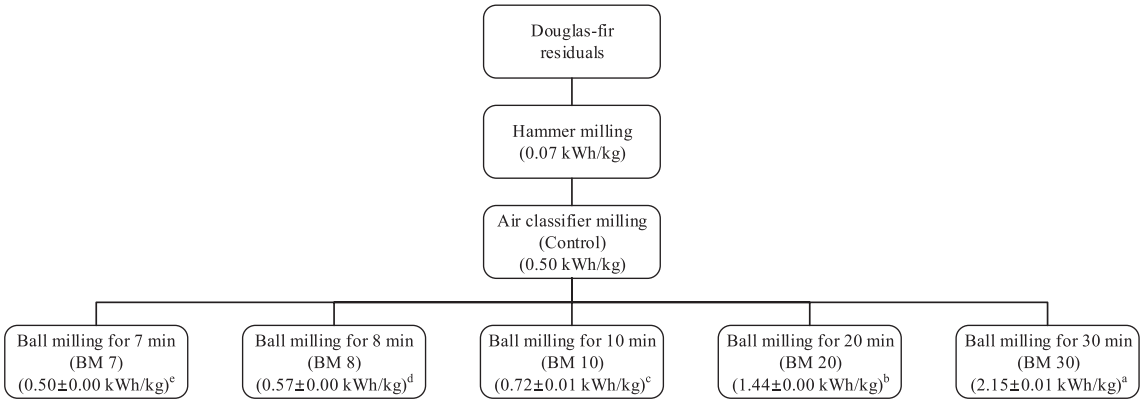

Fig. 1. Process flow diagram and energy consumption of each milling process set used. Different alphabet superscripts indicates that means of energy input for ball milling were significantly different ($p < .05$).

Pearson correlation coefficient and Fisher's LSD test were performed using Statistical Product and Service Solutions (IBM SPSS Statistics 22.0, IBM Corporation, NY, USA).

3. Results and discussions

3.1. Milling processes

Milling experiments were conducted following the steps shown in Fig. 1. The first pre-processing step of hammer milling was used for energy efficient coarse milling of the forest residuals (with an energy input of 0.07 kWh/kg) and the second step of air classifier milling was applied for an intermediate micronization (with an energy input of 0.50 kWh/kg) of the samples. This combination was a result of our preliminary testing. The sample pulverized through these two preliminary processes was used as the control sample for the rest of the experiments.

The control sample was subjected to ball milling process as described before. Energy consumption of ball milling significantly increased with increasing milling time. Energy consumption for a ball milling with different milling times studied ranged from 0.50 ± 0.00 kWh/kg (for 7 min milling time) to 2.15 ± 0.01 kWh/kg (for 30 min milling time).

3.2. Particle size of ball milled wood powder

Particle size of control sample on D90%, D50% and D10% was 416.00 ± 5.20, 121.33 ± 0.58 and 20.32 ± 0.10 μm respectively (Table 1). After ball milling, the particle size of the ball milled sample on D90%, and D10% significantly decreased to 41.67 ± 0.15 and 3.10 ± 0.01 μm respectively (Table 1). Energy consumption associated with milling time showed a negative correlation with the median particle size ($r = -0.74$, $p < 0.01$) (Table 2). Median particle size of milled samples significantly decreased from 41.23 ± 0.15 to 14.87 ± 0.06 μm with an increase in ball milling time since the impact time by the balls increased, resulting in more destruction of the recalcitrant wood structure. Median particle sizes of BM10, BM20, and BM30 were 28.10 ± 0.00, 16.83 ± 0.06, and 14.87 ± 0.06 μm respectively (Table 1). The difference in average median particle size between BM10 and BM20 was 11.27 μm, corresponding to a decrease of 40.11%. On the other hand, the difference in the size between BM20 and BM30 was 1.97 μm with a decrease of 11.65%. This suggests that after 20 min of ball milling treatment, the efficiency of size reduction of the ball milling process reduced.

A previous study also showed similar results for oil palm biomass. Size reduction of oil palm empty fruit bunch and oil palm frond fiber were dramatically decreased after 50 min and 10 min of ball milling, respectively (Zakaria et al., 2014). Yuan et al. (2016) also overserved a gradual decrease in particle sizes of micronized Camphorwood sawdust,

Table 2
Correlation matrix for ball milled forest residuals.

Parameter	Energy consumption	Crystallinity index	Sugar yield		Median particle size
			Glucose	Xylose/ Mannose	
Energy consumption	1	…	…	…	…
Crystallinity index	−0.980**	1	…	…	…
Glucose yield	0.996**	−0.972**	1	…	…
Xylose/ Mannose yield	0.979**	−0.958**	0.977**	1	…
Median particle size	−0.743**	0.770**	−0.725**	−0.840**	1

** Indicate significant at $p < .01$ (2-tailed).

Table 1
Energy efficiency and particle size distribution on D10, D50 and D90% of ball milled residuals.

Sample	Particle size (μm)			Energy consumption (kWh/kg)	Energy efficiency (kg glucose/kWh)
	D10%	D50%	D90%		
Control	20.30 ± 0.10[a]	121.33 ± 0.58[a]	416.00 ± 5.20[a]		
BM7	6.78 ± 0.02[b]	41.23 ± 0.15[b]	144.33 ± 0.58[b]	0.50	0.229
BM8	6.04 ± 0.03[c]	35.33 ± 0.12[c]	122.00 ± 1.00[c]	0.57	0.208
BM10	5.17 ± 0.01[d]	28.10 ± 0.00[d]	91.07 ± 0.38[d]	0.72	0.170
BM20	3.48 ± 0.01[e]	16.83 ± 0.06[e]	46.27 ± 0.32[e]	1.44	0.141
BM30	3.10 ± 0.01[f]	14.87 ± 0.06[f]	41.67 ± 0.15[f]	2.15	0.129

Means within the same column followed by different letters are significantly different ($P < .05$).

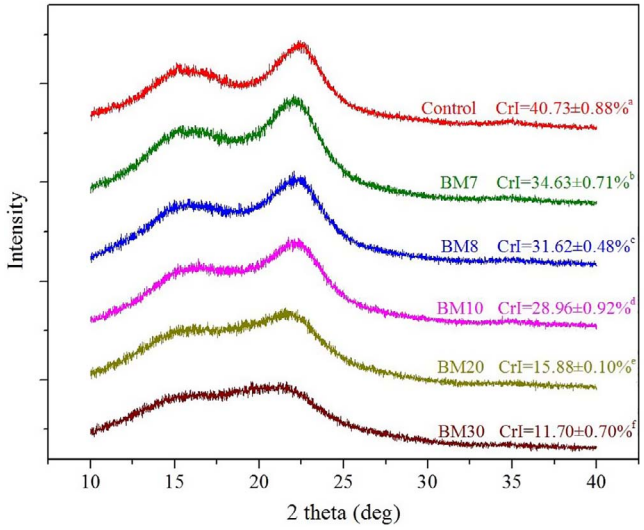

Fig. 2. Effect of ball milling time on X-ray diffraction of wood residuals. Different alphabet superscripts indicates that means of crystallinity index were significantly different ($p < .05$).

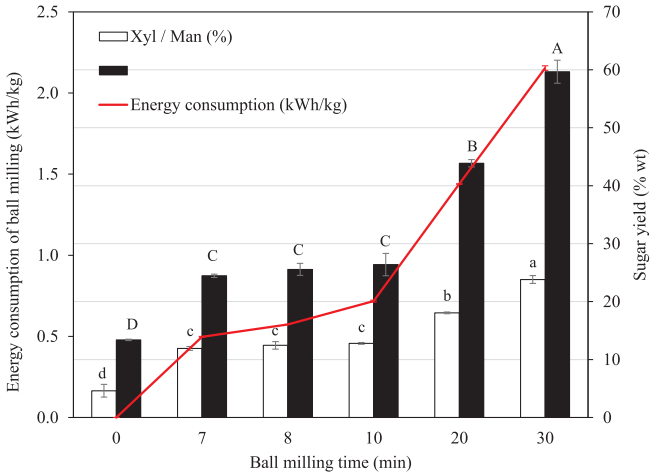

Fig. 3. Sugar yield of Douglas-fir forest residuals at various ball milling time with different energy consumption. Different letter above the bars indicate values significantly differs at $p < .05$ level. Capital letter (A, B, C, and D) indicates compared means of glucose yields (% wt) and small letter (a, b, c, and d) indicates compared means of Xyl/Man (% wt).

but the particle size did not decrease after 2 h of milling time with stirring ball mill. Although, they did not report the energy consumption for the milling studies. Therefore, these results suggest that the efficient milling time for particle size reduction may vary depending on milling processes, materials, and initial particle sizes.

3.3. Crystallinity index

Change in crystallinity index with different ball milling times is shown in Fig. 2. Ball milling process for bioconversion has been reported as an effective method that can increase amorphous cellulose regions which are not aligned parallelly. This results in providing better access to cellulose for hydrolysis (Barakat et al., 2014; Lin et al., 2010). Crystallinity index of control was 40.73 ± 0.88% while the range of crystallinity of ball milled samples ranged from 34.63 ± 0.71 to 11.70 ± 0.70%. With the increase in the milling time from 7 to 30 min, the degree of crystallinity index decreased by 71.27% compared to control. Crystallinity index of the forest residuals was significantly influenced by ball milling time (Fig. 2). The slope of crystalline peaks of ball milled wood was significantly decreased by increasing the milling time. This results from the amorphous regions being gradually enlarged at the expense of the crystalline regions with loss of the distinguishable peak (Da Silva et al., 2010). After 20 min of ball milling time, the crystallinity index was critically affected, showing a broad peak with a significantly higher sugar yield compared to BM7, BM8, and BM10. The ball milling time is directly proportional to the energy input. For extensive modification of the crystalline regions in cellulose, higher impact energy is required (Yu and Wu, 2011; Yuan et al., 2016; Zhao et al., 2006). Thus, crystallinity index had a positive correlation with median particle size ($r = 0.77$, $p < .01$) which was highly correlated with energy consumption ($r = -0.74$, $p < .01$). These results indicate developments of amorphous cellulose structure from crystalline cellulose structure. The impact energy causes the breakdown of the high-ordered crystalline cellulose structure that is made of the chains of cellulose with strong intermolecular and intramolecular hydrogen bonds, resulting in a decrease in the crystallinity index after ball milling.

3.4. Sugar yield and morphology

Fig. 3 shows the effect of ball milling time on glucose and xylose/mannose yield with corresponding energy consumption. Sugar yield of glucose and xylose/mannose were significantly different depending on ball milling time except for 7, 8, and 10 min treatments. The energy consumption had a high correlation with glucose yield ($r = 0.99$, $p < .01$) and xylose/mannose yield ($r = 0.98$, $p < .01$), which means that the energy input into the recalcitrant forest residuals during processes is critical to produce digestible substrates for enzymatic hydrolysis. Since a high amorphous cellulose region led to an improvement of accessibility for enzymes to attack cellulose amenably, the sugar yield of glucose and xylose/mannose showed a highly negative correlation with crystallinity index ($r = -0.97$ for glucose and $r = -0.96$ for xylose/mannose).

Residuals milled for a longer a time had greater surface area, which resulted in the positive effect on the accessibility of cellulose by the enzymes. Therefore, median particles sizes had a negative correlation with glucose ($r = -0.73$, $p < 0.01$) and xylose/mannose ($r = -0.84$, $p < 0.01$). This is because the separation of linkages by milling between cellulose, hemicellulose, and lignin improved the subsequent enzymatic hydrolysis of the residuals with increasing milling time.

By comparison, Du et al. (2017) reported that the glucose yield for Douglas-fir increased from 23.7 to 78.3% by increasing ball milling pretreatment time from 80 to 120 min. According to Hideno et al. (2009), energy consumption for pretreatment of rice straw using ball milling for 5–60 min was 9.0–108 MJ/kg (approx. 2.5–30 kWh/kg) with 52.2–89.4% glucose yield respectively. Kim et al. (2013) also stated that 2080 MJ/kg energy (approx. 577.78 kWh/kg) for 0.26–0.29 g/g biomass for glucose yield was required to pretreat rice straw with planetary wet-ball milling when the planetary mill was operated for 8 h. In our study, the range of glucose yield and xylose/mannose yield ranged from 13.39 ± 0.13 to 59.67 ± 1.98% for glucose yield, and from 4.61 ± 1.10 to 23.82 ± 0.69% for xylose/mannose yield respectively. The energy consumption increased from 0.50 to 2.15 kWh/kg by increasing ball milling time from 7 to 30 min. The most energy efficient milling time was BM7 showing 0.229 kg glucose/kWh and the energy efficiency decreased from 0.229 to 0.129 kg glucose/kWh by increasing ball milling time. In comparison with other results, Barakat et al. (2013) and Licari et al. (2016) reported that the energy efficiency was determined as 0.078–0.011 kg glucose/kWh with 5–60 min of ball milling time for rice straw, and 0.011–0.007 kg glucose/kWh with 24–72 h of ball milling time for sugarcane bagasse respectively.

This suggests that the multi-step process may reduce energy requirement for milling processes to improve enzymatic hydrolysis significantly compared to single step milling process.

Different particle morphology of final products was observed using scanning electron microscopy (SEM). By increasing the ball milling

time, greater destruction of the structures of fiber was observed and particles size decreased, losing the characteristic of longitudinal arrangements in the control sample. This random destruction and rupture increased contact surface area for subsequent enzymatic hydrolysis, increasing sugar yield, and amorphous cellulose regions.

Control, BM7, BM8 and BM10 samples showed a long compact structure compared to BM20 and BM30 samples, resulting in relatively low sugar yields at BM7, BM8 and BM10. This suggests strong networks between cellulose, hemicellulose and lignin abundantly exist at BM7, BM8, and BM10 compared to BM20 and BM30. Wood has a property of strongly forming its structure with longitudinal arrangements, and splitting along the longitudinal direction. This causes the comparatively long shape with larger aspect ratio of particles when the wood is pulverized through milling process (Abdullah and Wu, 2009; Jiang et al., 2017b). Fiber located between balls is broken apart into fiber fragments by ball-on-ball collision. Impact energy of the balls leads to crushing of the intact biomass, reducing particle sizes and increasing the amorphous regions of cellulose (Vaidya et al., 2016). BM7 and BM8 samples, the longitudinal separation characteristic was observed. The macrofibril bundles were damaged and were split as a result of ball milling. Jiang et al. (2017a,b) also observed that the dislocation and separation of an individual fiber or fiber bundles at middle lamella regions through SEM micrographs for lower moisture samples for Douglas-fir. When the ball milling time increased the longitudinal arrangement was destroyed by the strong mechanical impact energy from balls, resulting in irregular rounder shape fibers. This resulted in enhanced accessibility of the recalcitrant woody fiber structure for the enzyme to attack the cellulose and hemicellulose, leading to an increase of sugar yield.

4. Conclusions

Combination of milling processes including planetary ball milling was shown to be a potential pretreatment for lignocellulosic biomass for energy efficient conversion into sugars through enzymatic hydrolysis. Preprocessing the biomass with coarse milling can help bring the biomass to a form that can be more easily disrupted by the impact energy in ball milling. Further research is needed to optimize the efficiencies of the different combination milling steps and developing correlations with the raw material characteristics. This can help the industry to arrive at optimum combination milling processes that can result in higher sugar yields with overall low energy inputs.

The scanning electron micrograph images are available as a Supplementary information (Fig. S1).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.biortech.2017.11.103.

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