Powder Technology 378 (2021) 593–601

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Multi-blade milling from log to powder in one step – Experimental design and results

Atanu Kumar Das *, David A. Agar, Sylvia H. Larsson, Tobias Holdo, Dinesh Fernando, Magnus Rudolfsson

Swedish University of Agricultural Sciences, Department of Forest Biomaterials and Technology, SE-901 83 Umeå, Sweden

ARTICLE INFO

Article history:
Received 17 July 2020
Received in revised form 16 September 2020
Accepted 6 October 2020
Available online 09 October 2020

Keywords:
Multi-blade shaft mill (MBSM)
Experimental design
Wood powder
Particle size distribution (PSD)
Specific milling energy

ABSTRACT

This study investigated a new technique for obtaining wood powders from whole logs (*Pinus sylvestris* L.) in a single-step operation. The performance of a prototype multi-blade shaft mill (MBSM) was evaluated using a designed series of experiments including three input parameters, i.e., the moisture content of the log, milling blade speed and log feeding speed, combined with multilinear regression (MLR) analysis. The milling performance was characterised by specific milling energy, particle size distribution and bulk density of powder. For MBSM powders (80 to 95% particles <1.0 mm), the specific milling energy ranged from 99 to 232 kWh t^{-1} DM. The mass per cent of particles <0.5 mm in MBSM powders ranged from 55 to 80% compared to 41% from hammer-milled powders. Powder bulk density varied from 138 to 264 kg m^{-3} DM and the moisture content of the milled log was the only significant ($p < 0.05$) factor affecting the bulk density of resulting powders (dried). MLR models show that the milling energy is inversely proportional to the moisture content, which indicates that moisture influences MBSM milling in a similar way as in the sawing of wood and opposite to that of impact-based mills (i.e. hammer mills).

1. Introduction

Wood is an abundant and widely available renewable material, whose utilisation in industry fulfils political and societal aims for greater sustainable development [1]. In Sweden, the wood supply continues to grow and there is a large supply potential of small-diameter trees from the thinning of young forests [2]. As a structurally complex and multi-purpose raw material, wood has excellent potential in industrial applications and products. Wood powders (i.e. wood particulates with a diameter less than approximately 1 mm) are the starting point for many thermal conversion and biorefining applications including combustion [3], chemical, enzymatic [4] and thermochemical processes for energy and chemical production (e.g. gasification).

Wood is a product of living trees and after falling and delimbing, the stem wood (i.e. log) is a convenient and compact form in which to store wood before utilisation. The original chemical composition, including those compounds of interest in biorefining, are best preserved in the log form with original moisture content or stored underwater [5]. When the time comes for size reduction, there are many technologies for producing wood powders [6] and new ones continue to be developed (e.g. Kobayashi et al. [7]).

Mechanical production of pulp directly from logs is an old technology [8] but outside of wood pulping applications, common industrial size-reduction technologies for producing wood powders are impact-based or knife mills, which use hammers (i.e. hammer mills), swing beaters, discs or impellers to crush wood [6]. These mills create powders from wood chips (with lengths ranging from about 10–70 mm), which are mechanically cut from logs, screened to remove oversized particles and necessarily pre-dried to ease conveying and avoid clogging of screens. Typical particle sizes from the wood chip-hammer mill production route are in the range of 0.2–2.0 mm [9–11]. This strategy is partly based on the ease of handling but the storage of wood as chips also presents its own risks such as the potential for microbial degradation, dry matter losses and unwanted greenhouse gas emissions [12]. From a system perspective, the several steps in this tree-to-powder pathway necessitate intermediate handling operations [9], which prolong the turnaround time, consume monetary resources and influence the emission footprint of wood as a raw material in industrial applications [13].

Powders produced via common chipping and hammer milling have pathway-specific microstructure properties because the mill type [4] and the drying process [14] leave signature modifications on wood cell walls. This affects the particle morphology (i.e. aspect ratio, surface porosity and specific surface area) which contributes to powder bulk properties, the chemical distribution in wood powders and ultimately their potential use in wood-based industries. For example, the chipping, drying and pulping of green wood rapidly accelerate the resin oxidation reactions. For acid sulphite pulping, this is beneficial but in kraft pulping, the loss of extractive content diminishes by-product yields, such as turpentine and tall oil [5]. Green and dry wood also break differently. The

* Corresponding author.
E-mail address: atanu.kumar.das@slu.se (A.K. Das).

https://doi.org/10.1016/j.powtec.2020.10.026

Nomenclature

BD	Bulk density
BS	Blade speed
DM	Dry mass
FS	Feeding speed
FSP	Fibre saturation point
MBSM	Multi-blade shaft mill
MC	Moisture content
PSD	Particle size distribution
e_M	Specific milling energy (kWh t^{-1})
m_h	Mass of the hammer-milled powder (kg)
m_M	Mass of milled log (kg)
P_c	Chipping power (kW)
P_f	Log feeding power (kW)
P_h	Hammer-milling power (kW)
P_m	Log milling power (kW)
Q^2	Goodness of prediction
R^2	Goodness of fit
t_c	Chipping time (s)
t_h	Hammer-milling time (s)
t_M	Log milling time (s)
Y_{BD}	Modelled bulk density (kg m^{-3})
Y_E	Modelled specific milling energy (kWh t^{-1})
Y_{PSD}	Modelled particle size distribution (mass %)

location of fractures within the cell wall is a function of moisture content and affects the quality of the fracture surface; at low moisture content, fractures expose more lignin and at high moisture content, fractures expose more carbohydrates [15]. Such differences in particle morphology have measureable effects on the enzymatic digestibility of sugars in wood powders [4]. Particle shape and particle size distribution (PSD) also contribute to powder combustion behaviour [16], material handling [17] and bulk density [18], which is an important predictor of its feeding properties [19]. What these examples show is that pretreatment methods have high relevance for subsequent conversion and biorefinery processes using wood.

Size reduction of wood requires an energy input to generate the shear, compressional, impact and cutting forces that act to fragment, grind and lacerate wood fibres [6]. From a physiological perspective, size reduction results from the rupture of bonds within the cell walls of wood where moisture content strongly affects hydrogen bonding between organic polymers. As the amount of bound water increases in the cell wall, hydrogen bonding decreases [20,21] from dry wood up to the fibre saturation point (FSP), which is defined as the moisture content at which the onset of shrinkage begins, triggering incremental changes in strength properties compared to green conditions [22]. The FSP is a useful construct but difficult to generalise as it depends on tree species and method of measurement but is approximately 23% moisture content (wet basis) [23]. Water uptake causes a reduction in the strength properties of wood [24] and enhances the breakage required to produce wood powders. Based on this understanding, the energy required to mill near the FSP is lower than that of dry wood. Above the FSP, the presence of free water in cell cavities has no appreciable influence on wood's strength properties [24] but excess water certainly contributes to material behaviour during size reduction [18], shaping with tools and sawing [25]. On a physical basis, water affects density and the response of wood to applied forces [26] through altering the coefficients of friction (static and kinetic) and its large heat capacity and latent heat affect material temperature, a factor of the cutting force.

The most common theories postulate that milling energy is proportional to new surface area generated through the milling process or by the reduction of volume from the starting material [27]. What this means is that there is no free lunch in size reduction – the finer the powder, the greater the energy input. The purpose of size reduction is to produce a finer particle size distribution (PSD). The geometric surface area of a unit mass of powder increases rapidly as particle diameter decreases. Effective surface area also depends on particle morphology and porosity. Fine powders cost more in terms of milling energy but can provide enormous surface areas, which may be beneficial in certain applications (e.g. chemical, thermal and biological conversion).

Also in practice, the specific milling energy is a function of achieved particle size with finer powders requiring more energy [6,10,28]. From experimental studies on both sawing and attrition milling, moisture content, wood density, tree species [29], grain orientation and temperature [30] influence the energy requirements. Specific milling energy in sawing wood is also a function of moisture content. Contrary to attrition milling, the required sawing energy increases as wood becomes more dry below the FSP. In this hygroscopic range of wood, the energy needed to produce the cutting force is greater than above the FSP due to greater wood hardness, fracture toughness and modulus of elasticity [31]. Above the FSP and with a surplus of water, the sawing energy demands decrease further [25]. Therefore, the changes in the physical properties of wood, due to low moisture content, make sawing wood more energy intensive. Yet these same changes assist the destructive forces in the attrition milling wood.

With the resurgence of various wood-based applications in society and wood powder feedstock for bio-based industries, new size reduction technologies have the potential to improve utilisation through supplying better-suited wood powders for refining processes and products. Eliminating multistep pre-processing and thermal treatments could save time and unlock more benefits from the green chemical profile of wood.

The purpose of this study was to evaluate the performance of a novel prototype milling technology for the production of wood powders directly from whole tree stems. The performance analysis was based on characterised wood powder properties, which included the particle size distribution and bulk density as well as the measured specific milling energy in size reduction. Through analysis of the results, the study aimed to determine whether the prototype mill behaved more like a saw or more like attrition-based size-reduction technologies.

2. Materials and methods

2.1. General procedure

In this study, a novel multi-blade shaft mill (MBSM) was utilised to produce fine wood powders from whole pine logs in a single step. A full experimental design was set up for log milling. A hammer-milling procedure was used as a comparison and the performance of the MBSM milling technology was investigated with respect to specific energy consumption, particle size distribution and bulk density for produced powders.

2.2. Milling systems

The multi-blade shaft mill (Klingmill AB, Torshälla, Sweden) consisted of a roller table, a feeder, and two 350 mm wide shafts of packed blades driven by two separate electric motors (Fig. 1). The first shaft had 110 parallel-mounted blades, each having eight teeth with a clearance angle of 18°, a rake angle of 5° and kerf 4.2 mm. The second shaft had 137 parallel-mounted blades, each having 24 teeth with 15° rake angle and 3 mm kerf. Each motor had a rated power of 55 kW and speed of 1480 rpm. A sample collector was set up at the outlet of the mill. It consisted of a vacuum system, a filter, and a plastic bag where the produced powder was collected. According to the manufacturer, the feeder on this prototype can accept log diameters up to 280 mm.

Hammer-milling experiments were performed with a Bühler Hammermill Vertica, Switzerland, which was equipped with a vertical grinding shaft, a 55 kW electric motor of 1480 rpm, with 12 × 4 floating hammers and a 2 mm circular sieve.

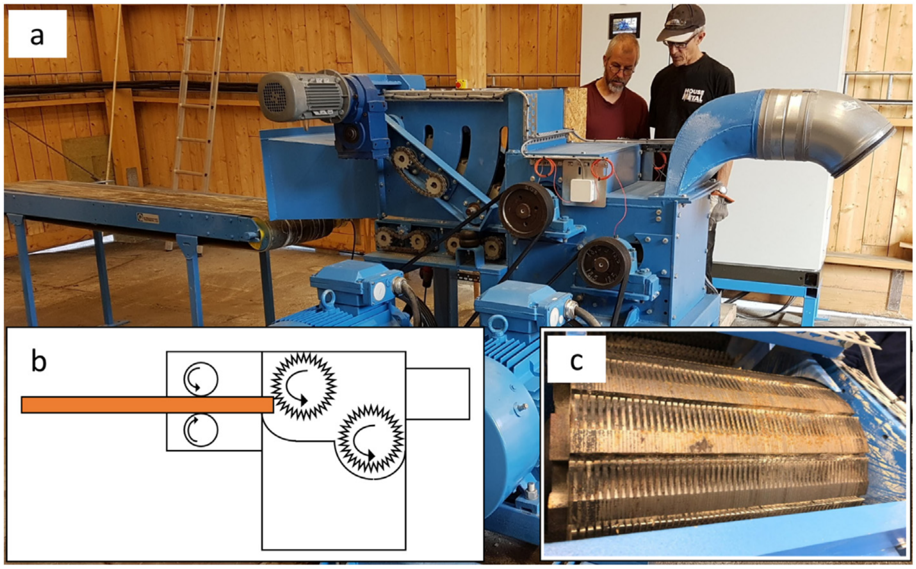

Fig. 1. (a) The prototype multi-blade shaft mill (MBSM) without its housing enclosure. The roller table, feeder and the two shafts connected to the motors are visible. The sample collector is not fitted to the outlet in the photograph. (b) The principle of operation of the MBSM and (c) the multi-blade shaft.

2.3. Experimental design

MSBM log milling was performed according to a full factorial (two-level) experimental screening design with one centre point varying three factors in their corresponding ranges: log moisture content (10 to 50%, wet basis), log feeding speed (1.3 to 2.6 m min^{-1}) and sawblade speed (52 to 72 m s^{-1}). The analysed responses were: the specific milling energy (kWh t^{-1} DM), the wood powder PSD (mass %) and the wood powder bulk density (kg m^{-3} DM). Experiments from each of the experimental design points were replicated three times, bring the total number of MBSM experiments for each response to 27. Settings, obtained factor values and response values are listed in Table S1 (Supplementary). The range of the three experimental factors was predefined by a method of trial and error.

Chipping and hammer milling were carried out at one single setting. The analysed responses were: the combined (chipper and hammer mill) specific milling energy (kWh t^{-1} DM), the wood powder PSD (mass %) and the wood powder bulk density (kg m^{-3} DM).

2.4. Material preparation

Defect-free Scots pine (*Pinus sylvestris* L.) trees were sourced from Vindeln, Sweden in September 2019. The trees, having an age of 22–32 years, were felled, delimbed and delivered next day to the Biomass Technology Centre, Swedish University of Agricultural Sciences, Umeå, Sweden. In total, 47 trees (27 for MBSM and 20 for hammer mill) were selected based on stem straightness and existence of knots. From the selected trees, 1.6 m logs were cut from the butt end aiming to minimise the extent of taper between the bottom and top, which ranged from approximately 10 to 14 cm in diameter. A handsaw was used to avoid chain-oil contamination. The logs were carefully debarked manually with drawknives as to not remove underlying stem wood. Drying was done at 25 °C in a drying cabinet (Elvärmedetaljer, Skurup, Sweden) to minimise changes in wood properties due to temperature. The dried and green logs were kept separate and wrapped in plastic to prevent changes in moisture content prior to experiments. An overview of the experimental procedure is depicted in Fig. 2.

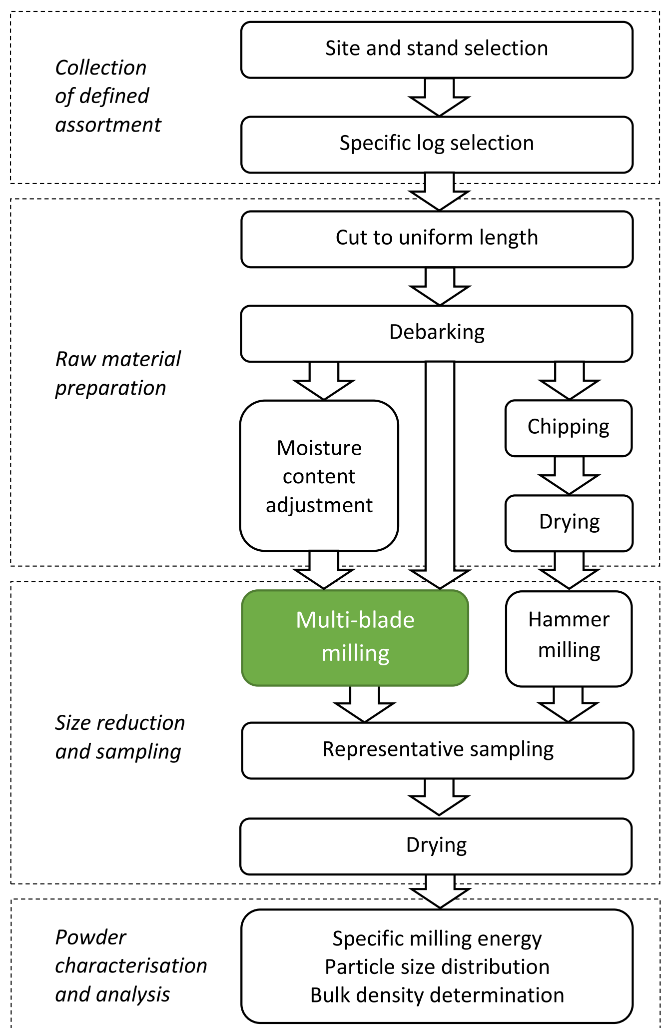

Fig. 2. An overview of the experimental procedure used in the study.

2.5. Milling procedure

The mass and length of the logs were measured and, within each moisture content level, ordered randomly with relation to the experimental design. Before each experiment, a plastic bag was fitted to the sample collector. The logs were then fed centrally via a roller table to the feeding section of the MBSM. During milling, the electric power (kW) and mill setting factors were recorded at 1 Hz frequency with a data acquisition system (INTAB PC-logger 3100). After the experiment, the sample bag was removed, sealed and weighed and the equipment thoroughly cleaned.

For the hammer mill experiment, 20 debarked logs were chipped (Edsbyhuggen, Woxnadalens Energi AB, Sweden) and then dried in an in-house-built flat-bed dryer to reach a moisture content of 7.2% (wet basis). The dry chips were screened (EO554, Fredrik Mogensen AB, Sweden) to obtain a 1.9–16.0 mm chip size range and this fraction was milled in the hammer mill. The electric power (kW) of the chipper and the hammer mill was logged at 1 Hz with a Fluke 1735 Power Logger (Fluke Corporation Everett, WA USA).

2.6. Sampling procedure

The sampling of wood powder from milling experiments was performed according to the standard method (SS-EN ISO 14780:2017 (E)) by systematically coning and quartering. Three samples were used for bulk density determination and one for sieve analysis. All powder samples were dried at 105 °C overnight, within approximately 20 min of milling and stored in sealed plastic bags for further analysis. The moisture content of the logs was measured from the powder moisture content.

2.7. Milling process performance

2.7.1. Specific milling energy

The specific milling energy e_M (kWh t^{-1} DM) for the MBSM milling was calculated according to Eq. (1):

$$e_M = m_M^{-1} \sum_{t_M} (P_f + P_m)\, t_M \tag{1}$$

in which m_M (kg DM) is the dry mass of the milled log, t_M (s) = log milling time, P_f (kW) = log feeding power and P_m (kW) = log milling power.

The specific milling energy e_h (kWh t^{-1} DM) for hammer milling was calculated according to Eq. (2):

$$e_h = m_h^{-1} \left(\sum_{t_c} (P_c \times t_c) + \sum_{t_h} (P_h \times t_h) \right) \tag{2}$$

in which m_h (kg DM) is the dry mass of the hammer-milled wood powder, t_c (s) = the chipping time, P_c (kW) = chipping power, t_h (s) = hammer-milling time and P_h (kW) = hammer-milling power.

2.7.2. Particle size distribution

The particle size distribution (PSD) was determined for the 27 MBSM samples and the hammer-milled samples using sieves following the standard method (EN 15149–2:2010). The sieving was done using a sieve shaker (Analysette 3, Fritsch, Germany) set at 1.5 mm amplitude for 10 min. The used sieves (Cisa, Spain) had standard (ISO-3310.1) sizes of 0.063, 0.125, 0.25, 0.5, 1.0, 1.4, 2.0, 2.8, 3.15, 8.0 and 16.0 mm. The amount of powder sample used in sieving was 0.5–0.7 L. The mass per cent of each size fraction was calculated from the total mass of sieved sample and the retained amount of powder on each sieve. The geometric mean (also known as Sauter) diameter was calculated using the equation from Tannous et al. [32]. Optical images of powders were taken using a stereo-microscope (model M205FA, Leica Microsystems, Germany) and a PLANAPO 0.63× lens at 4.92× magnification.

2.7.3. Powder bulk density

Determination of the bulk density (kg m^{-3} DM) of oven-dried wood powders was performed with a 5.4 L cylindrical vessel according to the standard method (EN 15103:2009).

2.8. Statistical analysis

Multilinear regression (MLR) analysis was utilised to build predictive models for the measured responses from input parameters. The modelling was performed with MODDE Pro-12 software (Umetrics Sartorius, Umeå, Sweden).

3. Results

A total of 27 MBSM experiments were performed at nine different settings according to the experimental design. The measured moisture content (% wet basis) of produced powders had ranges of high: 38–51, medium: 26–31 and low: 11–24 (Table S1).

3.1. Specific milling energy

The MSBM specific milling energy ranged from 99 to 232 kWh t^{-1} DM (Table S1) which is 1.2 to 2.7 times higher than that of the chipping and hammer-milling path (86 kWh t^{-1} DM). The MLR modelled effects and observed versus predicted plots are shown in Fig. 3a. The feeding speed (FS) and blade speed (BS) have significant ($p < 0.05$) negative and positive effects on milling energy, respectively. The moisture content (MC) has a significant ($p < 0.05$) negative effect on milling energy.

Within the range of the design, the specific milling energy was reduced by 62 kWh t^{-1} DM when the FS increased from its lowest to highest value, increased by 38 kWh t^{-1} DM when the BS was increased from its lowest to highest value, decreased by 25 kWh t^{-1} DM when the interaction term FS*BS increased from its lowest to highest value and decreased 18 kWh t^{-1} DM when MC increased from its lowest to highest value. The model has a R^2 value of 0.816 (Table 1), meaning that it can explain 81.6% of the variation in the experimental data. The Q^2 value of 0.733, which being above 0.5 indicates that the predictive ability of the model is good [33].

3.2. Particle size distribution

The cumulative PSDs of MBSM powders are shown in Fig. 4a along with the hammer mill PSD for comparison. The MBSM distributions are averages of the replicates at each experimental design point (Table S2 contains individual PSD data). The mass per cent of particles <0.5 mm in MBSM powders was much greater than in hammer-milled powders; ranging from 55 to 80% compared to 41%. The size specific differences are easily seen in the histogram (Fig. 4b) comparing the finest MBSM powder to that of the hammer mill.

All produced powders from the MBSM had a PSD within 80 to 95% particles <1.0 mm. The observed shape of MBSM powder particles, as seen in optical imaging (Fig. 5), was more spherical than in hammer mill powders. The highest mass per cent <1.0 mm was obtained with high BS and low FS using logs with high MC.

Regarding the model, which has a R^2 value of 0.707 and a Q^2 value of 0.526, the MC and BS factors have significant ($p < 0.05$) effect on the mass per cent of wood particle <1.0 mm (Fig. 3b). Square and interaction terms (MC*MC and FS*BS) also exist in the model. The share of particles <1.0 mm increased by approximately 7% and 6% when the BS and MC increased from their lowest to highest values, respectively.

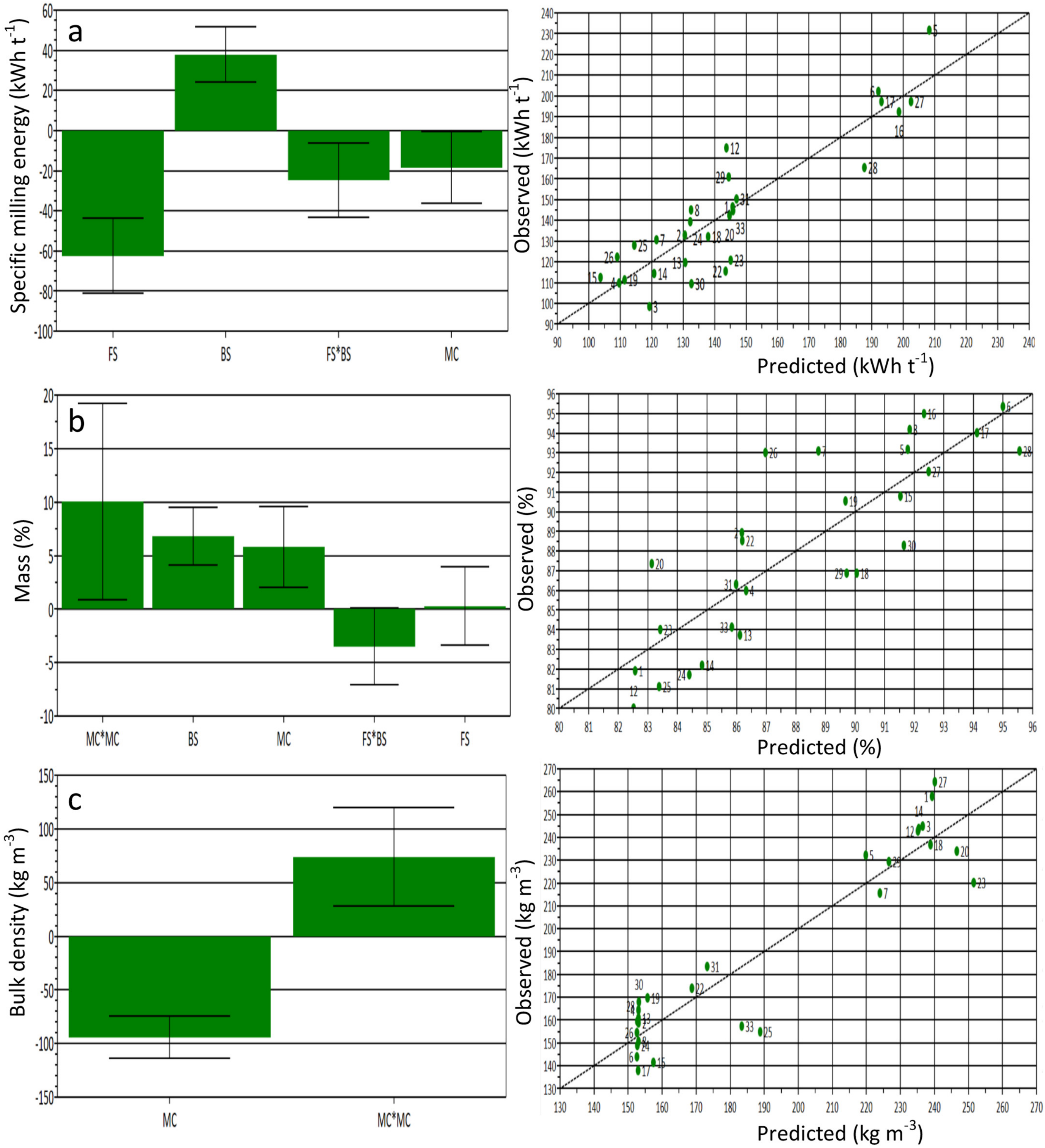

Fig. 3. Effects of scaled and centred factors in the response models for the (a) specific milling energy: Y_E (kWh t^{-1}) = −0.46 MC + 65.72 FS + 5.38 BS – 1.79 FS*BS – 92.16 (b) amount of wood particles <1 mm: Y_{PSD} (mass %) = −0.62 MC + 15.93 FS + 0.84 BS + 0.01 MC*MC – 0.25 FS*BS + 41.56 (c) powder bulk density: Y_{BD} (kg m^{-3}) = −7.98 MC + 0.09 MC*MC + 328.28 when each individual factor is varied from its lowest to its highest value, keeping all other factors at their average values in the design. The error bars indicate 95% level of confidence. Symbols refer to MC = moisture content, BS = blade speed and FS = feeding speed.

3.3. Powder bulk density

The bulk density of the MBSM powder varied from 138 to 264 kg m^{-3} DM (Table S1). According to the model, the MC of the milled log is the only significant ($p < 0.05$) factor affecting the bulk density of resulting dried powders. The bulk density decreases by 94 kg m^{-3} DM when MC increases from its lowest to highest value (Fig. 3c). The square effect of MC (i.e. the term MC*MC) has an opposite effect. The values of R^2 and Q^2 were 0.877 and 0.833 (Table 1), respectively.

Table 1
ANOVA of milling performance based on obtained responses.

Response	Model component					
	N	DF	R^2	Q^2	F	p
Specific milling energy (kWh t^{-1} DM)	28	23	0.816	0.733	25.50	0.00
Particle size distribution (mass %)	27	21	0.707	0.526	10.13	0.00
Bulk density (kg m^{-3})	27	24	0.877	0.833	85.44	0.00

Symbols are defined as: N = number of experiment, DF = degrees of freedom, R^2 = goodness of fit, Q^2 = goodness of prediction, F = ratio of explained to unexplained variance, p = probability of result.

4. Discussion

The influence of the three design factors on the responses (i.e. specific milling energy, wood particles <1.0 mm and bulk density) are shown as contour plots (Fig. 6).

4.1. Specific milling energy

The modelled specific milling energy increases with increasing BS and decreases with FS and the MC (Fig. 6a). The highest milling energy occurs for a BS of 72 m s^{-1} and a FS less than 1.5 m min^{-1}. Moisture content has a significant effect on the milling energy. A physical explanation is that water uptake up to the FSP (i.e. increasing amount of cell wall bound water) reduces hydrogen bonding between wood polymers [20,21] and weakens the strength properties of wood [24]. The result is that the breakage of wood to obtain wood powders is easier with higher MC up to the FSP. Above this level of moisture, the lower milling energy and greater amount of particles <1.0 mm may be due to a lubrication effect from surplus water during milling. The relatively large amount of extractives in pine (thinnings) have been reported to negatively affect the specific milling energy in attrition milling [34]. Without a comparative species in the present study, however, the influence of the extractive content on MBSM milling is not clear.

Based on this observation, the effect of MC in MBSM milling is similar to its influence in the sawing of wood. The lowest MBSM milling energy (99 kWh t^{-1} DM) was measured for powders having the largest PSD (63.5% <1.0 mm) and vice versa for highest milling energy (232 kWh t^{-1} DM) and smallest PSD (93.2% <1.0 mm). Comparison of the calculated geometric mean diameters reflects these differences in PSD (Table S2). While the specific milling energy was higher for the MBSM than for hammer-mill path, the powders produced were also substantially finer, which is what is expected from milling theory [27].

Considering previous studies with similar PSDs, the specific milling energy with the MBSM was comparable to ranges reported in literature. Esteban and Carrasco [9] produced powder 95% <1.0 mm from pine (*Pinus sylvestris L.*) chips using a two-stage hammer mill and a milling energy of 150 kWh t^{-1} DM. Phanphanich and Mani [35] used a knife mill to grind pine chips (21–71 × 15–40 × 1.9–4.9 mm) to geometric mean sized powder 0.71 mm with milling energy 238 kWh t^{-1}. Karinkanta et al. [36,37] milled spruce (*Picea abies*) sawdust with feed size 1–2 mm to obtain 0.02–0.10 mm median sized powder with a milling energy range of 100–380 kWh t^{-1} by oscillatory ball mill. Herein, the input material size (i.e. logs) was much higher than in previous

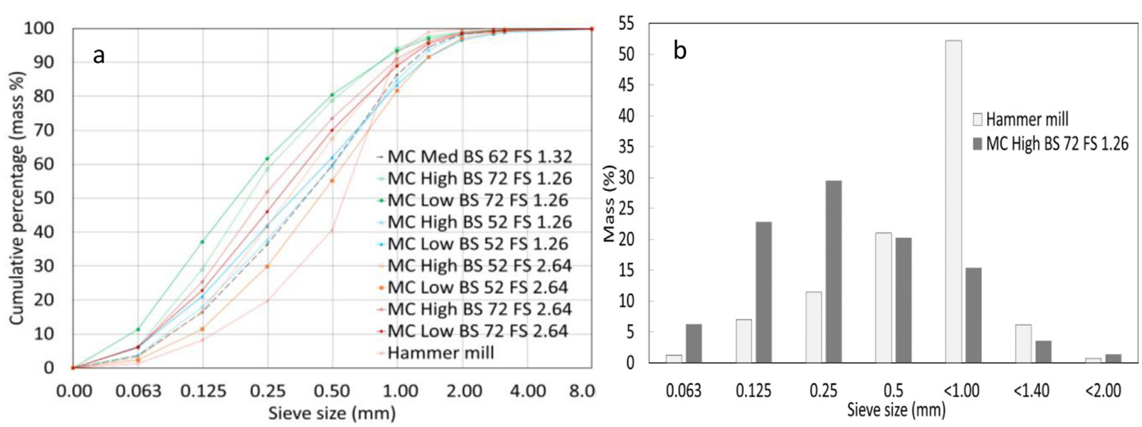

Fig. 4. (a) Cumulative particle size distribution of powders obtained from the multi-blade shaft mill (MBSM) and hammer mill. (b) Specific particle size differences between finest MBSM powder and hammer mill. Label symbols refer to MC = moisture content, Med = medium, BS = blade speed and FS = feeding speed.

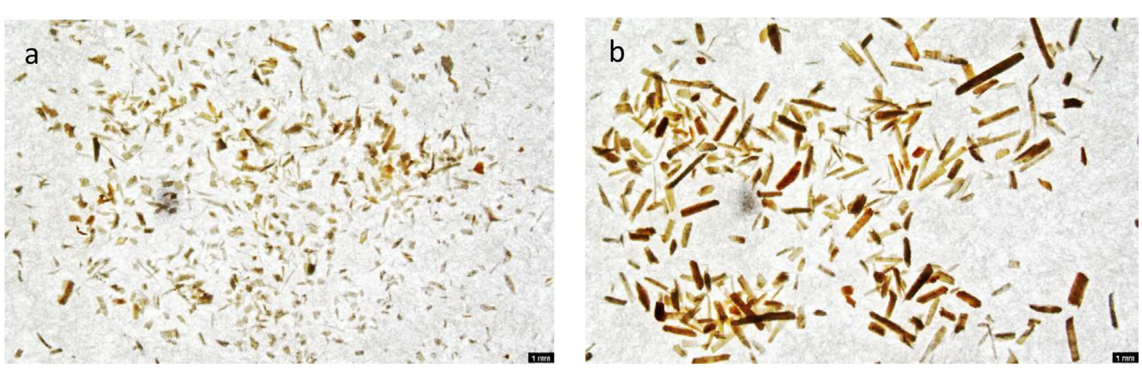

Fig. 5. Optical image of powders passing through 0.5 mm sieve from (a) multi-blade shaft mill and (b) hammer mill. The black line length is 1 mm.

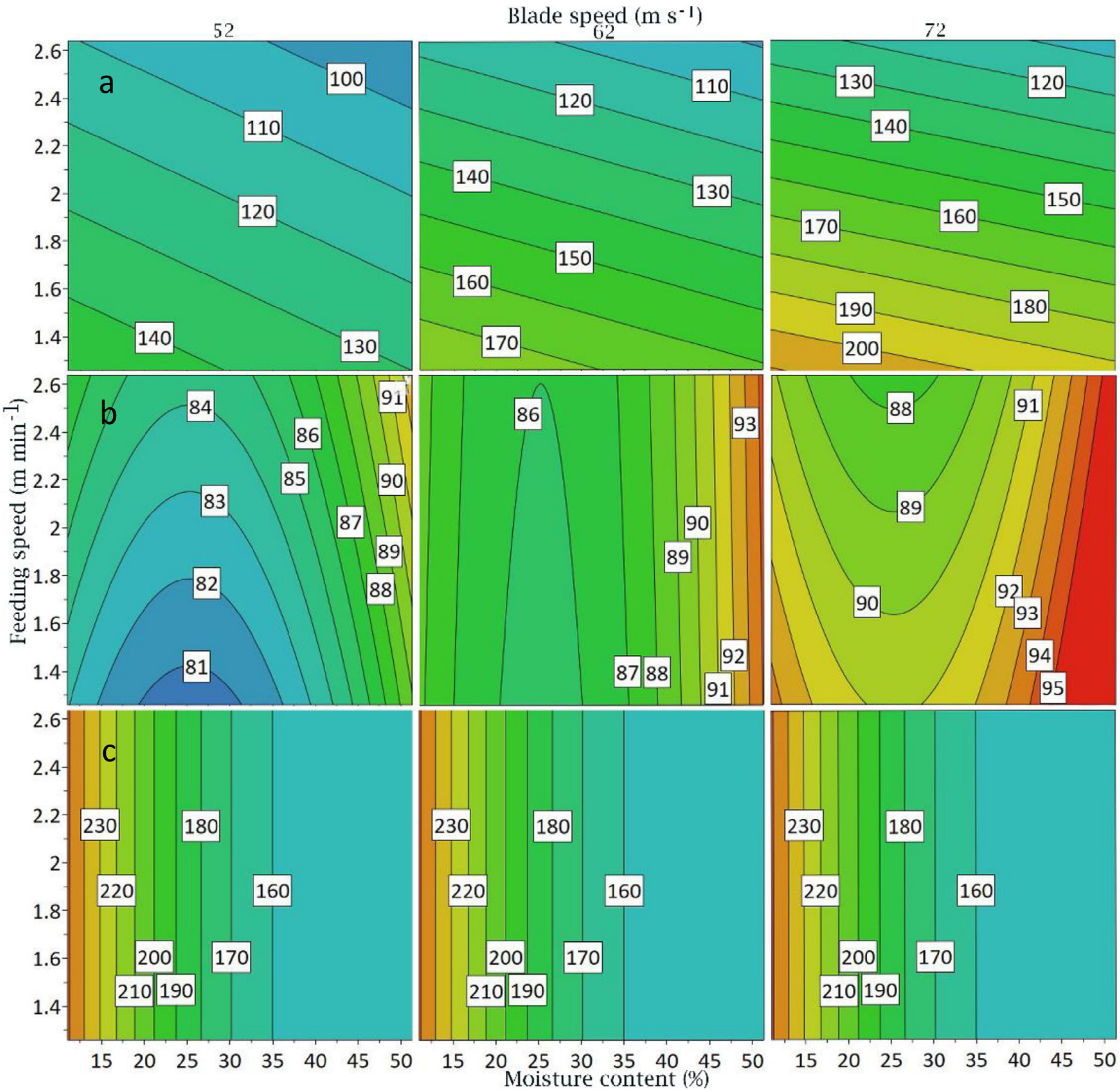

Fig. 6. Contour plots of experimental design space showing the influence of the three experimental design factors (moisture content (%), blade speed (m s^{-1}) and feeding speed (m min^{-1})) on the a) specific milling energy (kWh t^{-1} DM), b) particle size distribution (mass %) and c) bulk density (kg m^{-3} DM) of powders.

studies and the determined fine fraction of MBSM powders was also greater than in earlier work.

As the possibility to mill green wood is one of the key features of the MBSM, it is important to clarify the effect of MC on specific milling energy for other technologies. Attrition milling studies have found that MC dramatically affects milling energy; Liu et al. [38] and Gil et al. [18] have studied the effect using a hammer mill. They have shown that wetter wood required much greater (two to three times) the specific milling energy to produce comparable size wood powders. As indicated by the authors, hammer milling wetter materials makes the outlet screen more susceptible to blockages and this decreases the throughput of the mill (i.e. milling efficiency). Consequently, the mill must operate for longer duration with longer particle residence time and higher specific energy use.

One can conclude that comparing wet and dry materials in attrition mills is not a specific milling energy comparison but rather a comparison of how well the mill functions with moisture content variations. With size reduction using the MBSM, the length of time that the material is in the mill is known with precision and does not differ between wet and dry wood. In this regard, the action of milling with the MBSM seems analogous to sawing timber, only with multiple blades on the shaft working in parallel. This is supported by the influence of FS on milling energy. A higher FS reduces the milling energy of sawing wood because the duration of sawing is less (i.e. there is a higher throughput of material). This has been observed during band sawing of pine wood and it corresponded to a smaller sawdust particle size for lower FS [39]. Within the design space, decreasing FS with the MBSM shows the same result for high BS (Fig. 6a and 3a) but the opposite effect at low BS. Despite this observation, the significant effect of MC in the milling energy model indicates that the MBSM behaves more like sawing than like hammer milling.

4.2. Particle size distribution

Comparing the results, the hammer mill PSD was 92.9% <1.0 mm. With the MBSM, a high MC combined with higher BS increases the percentage of finer wood powders. The percentage of <1.0 mm particles increased with the increasing of MC and BS (Fig. 6b). The highest amount of <1.0 mm particles is achieved with a BS of 72 m s^{-1}, a feeding speed <1.8 m min^{-1} and a moisture content >48%. In general, these PSDs are a shift to finer wood powders compared to the studies in the literature cited above.

The model contains a MC quadratic term (i.e. MC*MC) which can indicate that the aforementioned role of MC in the milling energy is active also indirectly here for the production finer powder (i.e. high MC lowers required milling energy, leading to more fine particles in the PSD and smaller geometric mean diameter) (Fig. 3b). Although FS is a non-significant term in the model, it has a significant interaction with BS (i.e. the term FS*BS).

It was observed that some powder material exits the improvised enclosure of the prototype mill during operation. PSD analysis of leaked powder showed that it had 2–10% higher fines content in the range of 0.25–0.50 mm. Therefore, it can be assumed that further development of the mill enclosure in future prototypes will assist in obtaining even finer PSDs.

4.3. Bulk density

The bulk density was in the range of 138 to 264 kg m^{-3} DM for 63.5 to 95.3% <1.00 mm MBSM pine wood powders while it was 219 kg m^{-3} for 92.9% <1.00 mm hammer-milled powders. BS and FS have no effect on bulk density (Fig. 6c). The highest bulk density occurs at low MC. The bulk density of MBSM and hammer-milled powders was in the range of previous studies (95–381 kg m^{-3} DM) from pine, Douglas fir and poplar wood produced using knife and hammer mills [9,18,32,35,40].

The factors that affect the bulk density of a powder are particle size distribution [28], particle length [28,41–43] and particle density [28]. Hook-shaped particles have an interlocking tendency [44] and larger biomass particles commonly have this shape [45]. Although bulk density is a function of particle size [40] and there is some evidence that finer particles result from the milling of high MC wood, less spherical shapes have more points of contact in the bulk and this worsens particle packing efficiency and decreases bulk density. Vice versa, smaller size particles have shapes that are more spherical and contribute to better packing by filling gaps, leading to higher bulk density [18].

Dimensional changes in wood (i.e. shrinkage and swelling) only occur when the moisture content falls below the FSP [46]. The extent of shrinkage and cracking in wood is a function of the drying temperature [47]. Shrinkage occurs in all three dimensions and can be significant. For example, in wood veneers shrinkage can be 7.6 and 9.9% tangentially and 5.4 and 5.7% radially when dried at 60 and 150 °C, respectively [48]. Drying of green wood to low moisture content then generates hydrostatic tension contributing to cellular collapse, internal checking and washboard depression in wood [49].

The experimental design used logs of low and high moisture content. An important difference in the experimental procedure (Fig. 2) was that low MC logs had their MC level adjusted by partial drying at 25 °C before milling but high MC logs were milled above their FSP so that their powders required more drying than powders from the low MC logs. Powders from the green logs had a higher vapour pressure within the wood matrix during drying at 105 °C [50]. Their powders experienced greater dimensional and chemical compositional changes because they had more water (high vapour pressure) within their cells. Due to this and the fact that drying temperature is also known to alter wood's chemical composition and cellulose crystallinity [48], it is plausible that the different conditions affected the particle shape and density in produced powders (i.e. that particles from high MC logs had shape or density that decreased their packing efficiency). These differences could explain why powders milled from high MC logs had a lower bulk density and why MC is the only significant factor in the model (Fig. 3c).

Referring to the model of bulk density, there is a squared effect of MC (i.e. MC*MC). The screening design in MODDE software does not justify the use of quadratic terms but it can be seen that it fits a physical explanation of fibre saturation in the wood (Fig. 6c). Above the FSP, there is a saturation behaviour of the model, which can be seen as the saturation of the wood itself. Since the extent of shrinkage above FSP does not depend on MC [46] there is no dimensional change at MC above this point.

Further study using imaging techniques will allow more accurate characterisation of particle size, morphology and topochemistry and illuminate how these factors affect the bulk density of MBSM powders. In addition, the relationship between milling factors, milling energy and effective surface area of powders needs further investigation.

Current practice in industry in size reduction technology is reliant on the use of wood chips rather than whole tree stems. Hammer mills have inherent technical limitations for producing wood powders, as they can accept neither all sizes nor all moisture content ranges of chips. Chips storage brings risks of biological degradation, dry-matter losses and undesirable emissions that presenting health risks. Milling with the MBSM prototype bypasses these limitations while at the same time produces powders with much finer particles. The MBSM can mill a wide range of diameters and moisture content logs meaning it can mill green wood directly after felling the tree. It also provides more possibilities to tailor the particle size distribution to the powder application of interest. Smaller particle sizes enhance available surface area, leading to better penetration of chemicals and enzymes in pre-processing for biofuel production. The technology enables wood storage in its preferred green form, thereby better preserving its chemical composition, up until the log is utilised. Moreover, MBSM technology may provide benefits from a supply chain perspective by shortening the tree-to-powder pathway in turnaround time and resource use. Further study is needed to characterise powders (chemical analysis and microstructure) from the MBSM and investigate the milling of other wood species.

5. Conclusions

In this study, wood powders were produced successfully from whole pine logs in a single step using a novel multi-blade shaft mill (MBSM). The specific milling energy of the MBSM was higher than conventional hammer milling but the MBSM produced significantly higher fractions of finer particles. According to the MLR model, the milling energy was inversely proportional to moisture content of wood both above and below the fibre saturation point. This indicates that moisture influences MBSM milling in a similar way as in the sawing of wood and opposite to impact-based milling.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.powtec.2020.10.026.

Funding

This study was funded in part by the Swedish Energy Agency (Project number- 46904-1) and Swedish Bio4Energy Strategic Research Environment.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors would like to thank Gunnar Kalén, for assistance and suggestions during milling and wood powder analysis, and Markus Segerström for his help in tree collection and milling assistance. Borislav Vujadinovic and KlingMill AB are also acknowledged for technical support and assistance. The authors also thank Marta Derba-Maceluch at the Department of Forest Genetics and Plant Physiology for support with optical imaging.

References

[1] United Nations, Transforming our world: the 2030 Agenda for Sustainable Development, sustainabledevelopment.un.org 2015 (pp. 41).

[2] R. Fernandez-Lacruz, F. Di Fulvio, D. Athanassiadis, D. Bergstrom, T. Nordfjell, Distribution, Characteristics and Potential of Biomass-Dense Thinning Forests in Sweden, Silva Fennica, 49, 2015.

[3] S. van Loo, J. Koppejan, The Handbook of Biomass Combustion and Co-Firing, Taylor & Francis Group, London, Routledge, 2012.
[4] J.D. Fougere, M. Lynch, J. Zhao, Y. Zheng, K.C. Li, Impact of mechanical downsizing on the physical structure and enzymatic digestibility of pretreated hardwood, Energy Fuel 28 (2014) 2645–2653.
[5] T.J. McDonough, Wood chemistry - fundamentals and applications – Sjostrom, E, J. Am. Chem. Soc. 105 (1983) 4503.
[6] P. Karinkanta, A. Ammala, M. Illikainen, J. Niinimaki, Fine grinding of wood - overview from wood breakage to applications, Biomass Bioenergy 113 (2018) 31–44.
[7] N. Kobayashi, P. Guilin, J. Kobayashi, S. Hatano, Y. Itaya, S. Mori, A new pulverized biomass utilization technology, Powder Technol. 180 (2008) 272–283.
[8] L.I. Salminen, S. Liukkonen, M.J. Alava, Ground wood Fiber length distributions, Bioresources 9 (2014) 1168–1178.
[9] L.S. Esteban, J.E. Carrasco, Evaluation of different strategies for pulverization of forest biomasses, Powder Technol. 166 (2006) 139–151.
[10] M. Gil, I. Arauzo, Hammer mill operating and biomass physical conditions effects on particle size distribution of solid pulverized biofuels, Fuel Process. Technol. 127 (2014) 80–87.
[11] L. Kratky, T. Jirout, Biomass size reduction Machines for Enhancing Biogas Production, Chem. Eng. Technol. 34 (2011) 391–399.
[12] E. Alakoski, M. Jamsen, D. Agar, E. Tampio, M. Wihersaari, From wood pellets to wood chips, risks of degradation and emissions from the storage of woody biomass - a short review, Renew. Sust. Energ. Rev. 54 (2016) 376–383.
[13] M. Wihersaari, Energy consumption and greenhouse gas emissions from biomass production chains, Energy Convers. Manag. 37 (1996) 1217–1221.
[14] S. Stenstrom, Drying of biofuels from the forest-a review, Dry. Technol. 35 (2017) 1167–1181.
[15] J.X. Jiang, J.W. Wang, X. Zhang, M. Wolcott, Microstructure change in wood cell wall fracture from mechanical pretreatment and its influence on enzymatic hydrolysis, Ind. Crop. Prod. 97 (2017) 498–508.
[16] S. Paulrud, C. Nilsson, The effects of particle characteristics on emissions from burning wood fuel powder, Fuel 83 (2004) 813–821.
[17] S. Paulrud, J.E. Mattsson, C. Nilsson, Particle and handling characteristics of wood fuel powder: effects of different mills, Fuel Process. Technol. 76 (2002) 23–39.
[18] M. Gil, I. Arauzo, E. Teruel, Influence of input biomass conditions and operational parameters on comminution of short-rotation forestry poplar and corn Stover using neural networks, Energy Fuel 27 (2013) 2649–2659.
[19] J. Falk, R.J. Berry, M. Brostrom, S.H. Larsson, Mass flow and variability in screw feeding of biomass powders - relations to particle and bulk properties, Powder Technol. 276 (2015) 80–88.
[20] D.W. Green, J.E. Winandy, D.E. Kretschmann, Mechanical Properties of Wood, Wood Handbook Chapter 4, Forest Products Laboratory, USDA Forest Service, USA, 1999.
[21] R.M. Rowell, How the environment affects lumbar design, in: D.E.A.G. Lyon (Ed.), Forest Products Laboratory ReportUSDA, Forest Service, 1980.
[22] W.W. Barkas, Fibre saturation point of wood [9], Nature 135 (1935) 545.
[23] S. Glass, S.L. Zelinka, Moisture relations and physical properties of wood, Wood Handbook—Wood as an Engineering Material, US Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, WI, USA, 2010.
[24] J.E. Winandy, R.M. Rowell, Chemistry of wood strength, Handbook of Wood Chemistry and Wood Composites Chapter 11, CRC Press, N.W. Corporate Blvd., Boca Raton, Florida 334312005, 2000.
[25] T. Morita, K. Banshoya, T. Tsutsumoto, Y. Murase, Corrosive-wear characteristics of diamond-coated cemented carbide tools, J. Wood Sci. 45 (1999) 463–469.
[26] L. Cristovao, M. Ekevad, A. Gronlund, Industrial sawing of Pinus sylvestris L.: power consumption, Bioresources 8 (2013) 6044–6053.
[27] V.R. Voller, A note on energy-size reduction relationships in comminution, Powder Technol. 36 (1983) 281–286.
[28] H. Rezaei, C.J. Lim, A. Lau, S. Sokhansanj, Size, shape and flow characterization of ground wood chip and ground wood pellet particles, Powder Technol. 301 (2016) 737–746.
[29] V. Repellin, A. Govin, M. Rolland, R. Guyonnet, Energy requirement for fine grinding of torrefied wood, Biomass Bioenergy 34 (2010) 923–930.
[30] V. Nasir, J. Cool, A review on wood machining: characterization, optimization, and monitoring of the sawing process, Wood Mater. Sci. Eng. 15 (2020) 1–16.
[31] J. Nordstrom, J. Bergstrom, Wear testing of saw teeth in timber cutting, Wear 250 (2001) 19–27.
[32] K. Tannous, P.S. Lam, S. Sokhansanj, J.R. Grace, Physical properties for flow characterization of ground biomass from Douglas fir wood, Part. Sci. Technol. 31 (2013) 291–300.
[33] L. Eriksson, E. Johansson, N. Kettaneh-Wold, C. Wikström, S. Wold, Design of Experiments: Principles and Applications, Umeå Learnways AB, Stockholm, 2008.
[34] D. Fernando, J. Hafren, J. Gustafsson, G. Daniel, Micromorphology and topochemistry of extractives in scots pine and Norway spruce thermomechanical pulps: a cytochemical approach, J. Wood Sci. 54 (2008) 134–142.
[35] M. Phanphanich, S. Mani, Impact of torrefaction on the grindability and fuel characteristics of forest biomass, Bioresour. Technol. 102 (2011) 1246–1253.
[36] P. Karinkanta, M. Illikainen, J. Niinimaki, Impact-based pulverisation of dried and screened Norway spruce (*Picea abies*) sawdust in an oscillatory ball mill, Powder Technol. 233 (2013) 286–294.
[37] P. Karinkanta, M. Illikainen, J. Niinimaki, Pulverisation of dried and screened Norway spruce (*Picea abies*) sawdust in an air classifier mill, Biomass Bioenergy 44 (2012) 96–106.
[38] Y. Liu, J. Wang, J.C. Barth, K.R. Welsch, V. McIntyre, M.P. Wolcott, Effects of multistage milling method on the energy consumption of comminuting forest residuals, Ind. Crop. Prod. 145 (2020) 111955.
[39] M. Bariska, Z. Pasztory, The optimum log feed speed with bandsaw, Eur. J. Wood Wood Prod. 73 (2015) 245–250.
[40] Z. Miao, T.E. Grift, A.C. Hansen, K.C. Ting, Energy requirement for comminution of biomass in relation to particle physical properties, Ind. Crop. Prod. 33 (2011) 504–513.
[41] N. Chevanan, A.R. Womac, V.S. Bitra, S. Sokhansanj, Effect of particle size distribution on loose-filled and tapped densities of selected biomass after knife mill size reduction, Appl. Eng. Agric. 27 (2011) 631–644.
[42] P.S. Lam, S. Sokhansanj, X. Bi, C.J. Lim, L.J. Naimi, M. Hoque, S. Mani, A.R. Womac, X.P. Ye, S. Narayan, Bulk density of wet and dry wheat straw and switchgrass particles, Appl. Eng. Agric. 24 (2008) 351–358.
[43] R.P. Zou, X.Y. Lin, A.B. Yu, P. Wong, Packing of cylindrical particles with a length distribution, J. Am. Ceram. Soc. 80 (1997) 646–652.
[44] J.E. Mattsson, P.D. Kofman, Method and apparatus for measuring the tendency of solid biofuels to bridge over openings, Biomass Bioenergy 22 (2002) 179–185.
[45] M. Gil, D. Schott, I. Arauzo, E. Teruel, Handling behavior of two milled biomass: SRF poplar and corn Stover, Fuel Process. Technol. 112 (2013) 76–85.
[46] P. Bajpai, Wood and Fiber fundamentals, Biermann's handbook of pulp and paper, 3rd edition, volume 1: raw material and pulp making, chapter 2, Amsterdam, Netherlands, Elsevier (2018) 19–74.
[47] H.J. Chai, C. Xu, J. Li, F.X. Kong, Y.C. Cai, Effects of pretreatment with saturated wet air and steaming on the high-frequency vacuum drying characteristics of wood, Bioresources 14 (2019) 9601–9610.
[48] Y.H. Shen, Z.Z. Gao, X.F. Hou, Z.Y. Chen, J.Y. Jiang, J. Sun, Spectral and thermal analysis of Eucalyptus wood drying at different temperature and methods, Dry. Technol. 38 (2020) 313–320.
[49] B.S.W. Dawson, H. Pearson, M.O. Kimberley, B. Davy, A.R. Dickson, Effect of supercritical CO_2 treatment and kiln drying on collapse in Eucalyptus nitens wood, Eur. J. Wood Wood Prod. 78 (2020) 209–217.
[50] M. Hadjiski, N. Deliiski, N. Tumbarkova, Mathematical description of the latent heat of water vaporization in capillary porous materials, Informa. Technol. Control 1 (2019) 2–8.