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Fibre characteristics of hemp decortication using a planetary mono mill

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Abstract. The hemp industry is an expanding sector of the Canadian economy that presents a positive change for the environment. Hemp fibre is a renewable resource which has the market potential for producing new, and replacing existing, composite products. A hemp decortication experiment was conducted using a Fritsch Planetary Mono Mill. Varying treatments, including three grinding durations (2, 4, and 6 min) and seven grinding speeds (100, 150, 200, 250, 300, 350, and

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400 rpm) were studied using retted hemp feedstock cut to a length of 40 mm. Fibre yield, core yield, chaff, and effectiveness of milling were determined following decortication. Results showed that milling duration and speed impacted the yields and effectiveness. Higher grinding speeds and longer grinding durations allowed for a greater separation of fibre bundles and resulted in greater fibre yields. Extreme combinations of grinding speed and duration showed high levels of chaff production. Low grinding speeds and short grinding durations resulted in a poor separation of fibre bundles, and fibre lengths remained similar to the original 40 mm feedstock lengths.

Keywords. *hemp, decortication, planetary mono mill, fibre characteristics, grinding speed, duration.* .

INTRODUCTION

Hemp, (*Cannabis sativa*), is a temperate climate plant grown mostly in Europe, Russia, and Canada. Several different varieties exist, all of which vary in physiological and anatomical aspects. In April of 1998, hemp was legalized for industrial cultivation in Canada. The legalization of hemp allowed for its use as a natural fibre. High quality hemp fibre may be cottonized and used in the textile industry, composite industry, or other industrial fibre uses (Kostuik et al. 2007). Natural fibre composites have been used to replace glass mostly in non-structural applications, such as automobile components (Wambua et al. 2003) as well as building materials. Biocomposites offer numerous advantages as opposed to traditional composites, such as light weight and high tensile strength.

Increasing interest in hemp fibre has been occurring due to properties such as low density, acceptable specific strength, good thermal insulation properties, reduced tool wear, reduced dermal and respiratory irritation, renewable resource, recyclability, and biodegradability (Park et al. 2006; Li et al. 2000; Wong et al. 2004; Van de Weyenberg et al. 2003; Gassan 2002; Baiardo 2004). Hemp fibre is comparable to glass fibres in high volume applications, such as tensile and modulus strength, and durability (Williams and Wool 2000). In a study conducted by Wambua et al. (2003),

hemp fibre showed a tensile strength of approximately 52 MPa. The high tensile strength makes hemp fibre an acceptable choice for biocomposite material.

Decortication, the process of removing the outer fibrous layer from the inner core layer, can be achieved through the use of a number of different machines, such as hammer mills and crushing rollers. Hammer mills are energy intensive machines that use impact and shear forces for decortication. The energy requirement of a hammer mill is affected by factors such as; screen size of the hammer mill, moisture content, bulk and particle density of hemp material, and feed rate (Yu et al. 2006; Lopo 2002; Shi et al. 2003). Baker (2009) observed an increase in power requirement for larger mean chop sizes of retted hemp feedstock. Another method used for decortication involves a cutterhead. Gratton and Chen (2004) used a modified cutterhead with three knives and 38 x 38 mm angle steel scutching bars to decorticate hemp stalk. A maximum fibre yield of 61% with a purity of 52% was observed. Gratton and Chen (2004) observed that approximately 45% of the fibre contained in the hemp feedstock was lost into the chaff outlet. In general, hammer mill and cutterhead methods generate low fibre purity (Gratton and Chen 2004) and do not allow for an entire separation of fibre from core (Fürll and Hempel 2000; Gratton and Chen 2004). Another method of decortication involves crushing rollers. The flour milling industry widely uses crushing rollers to grind wheat into flour (Fang et al. 1997). Crushing rollers utilize fluted, pinned, and flat rollers to crush and separated hemp feedstock. Roller speed and roll gap are two critical parameters for hemp decortication. If roll speed is too slow then poor decortication is observed (Hobson et al. 2001). Roll gap affects hemp particle size and distribution, and energy requirement. Small roll gaps have a higher energy requirement due to more efficient grinding (Fang et al. 1997).

Decortication may also be achieved through the use of a ball mill. Ball mills have a wide range of research applications. Fukazawa et al. (1982) used a ball mill to investigate the efficiency

of enzyme digestion of ball milled aspen wood. Ball mills have also been utilized for medical research to prepare a dry powder vaccine formulation containing whole inactive influenza virus, and a mucoadhesive compound for nasal delivery (Garmise et al. 2006). Ball mills have also been used in the materials research industry. Périgo et al. (2007) used a planetary ball mill to produce sintered PrFeB magnets. The milling process is a mechanical process in which energy is released at the point of collision between grinding balls (Prasad et al. 2005). Material is placed into a specific size and type of grinding bowl along with an appropriate size, type, and number of grinding balls. The ball mill operates in a circular motion, and when the mill rotates centrifugal forces cause the balls to travel upward along the wall of the grinding bowl. The faster the mill rotates, the farther the balls are carried up inside the mill (Prasad et al. 2005).

In summary, some research has been performed on the decortication of hemp using a hammer mill, roller mill, and cutterhead, but no research has been done on the decortication of hemp using a ball mill. The objectives of this study was to investigate the performance of a ball mill for the decortication of 40 mm cut retted hemp under different grinding durations and grinding speeds. The performance indicators measured included fibre yield and effectiveness of grinding.

MATERIALS AND METHODS

Description of feedstock

In this study, field retted and baled USO 31 hemp feedstock was used (Fig. 1). Samples were collected at Baker Farms, located in Dauphin, MB. The moisture content of the feedstock was 9.7%, as measured by the oven-dry method at 60°C for 72 h. Plants were harvested according to common farming practices in Manitoba. The hemp crop was swathed using a mower-conditioner in the second week of August 2007, and left to ret in the field for approximately six weeks prior to

being baled. Bales were stacked and stored outside. Samples for the ball mill experiment were handpicked from a randomly selected bale (Fig. 1) in July 2008, and bagged in poly-woven grain bags to be transported to the laboratory at the University of Manitoba.



Fig. 1. Retted USO 31 hemp feedstock.

Ball mill machine

A lab-scale Planetary Mono Ball Mill (Fig. 2a) (Model Pulverisette 6, Fritsch, Idar-Oberstein, Germany) was used to decorticate hemp stock. The mill was securely fastened to a level surface during decortication. The control panel of the mill allowed for the selection of rotational speeds in the range of 100 to 650 rpm in 10 rpm intervals, and grinding durations from 1 min to 99 h. Inside the mill housing, there was a grinding bowl holder and a disc which supported the grinding bowl (Fig. 2b). The grinding bowl (Fig. 2c) had agate-liner inside and the net volume of the bowl was 250 mL. The grinding media were fifteen 20 mm diameter agate balls (Fig. 2d). In a grinding operation, the grinding bowl, filled with the grinding balls, was clamped onto the bowl holder. The grinding bowl rotates about its own axis and at the same time rotates with the support disc in the opposite direction (Fig. 3a). Thus, the centrifugal forces alternately act in the same and

opposite directions (Fritsch 2005). This results in the grinding balls running along the inner wall of the grinding bowl (Fig. 3b) causing frictional and impact effects on the material being ground.



(a)



(b)



(c)



(d)

Fig 2. Decortication equipment; (a) housing of the planetary mono ball mill; (b) inside the housing; (c) grinding bowl; and (d) agate grinding balls.

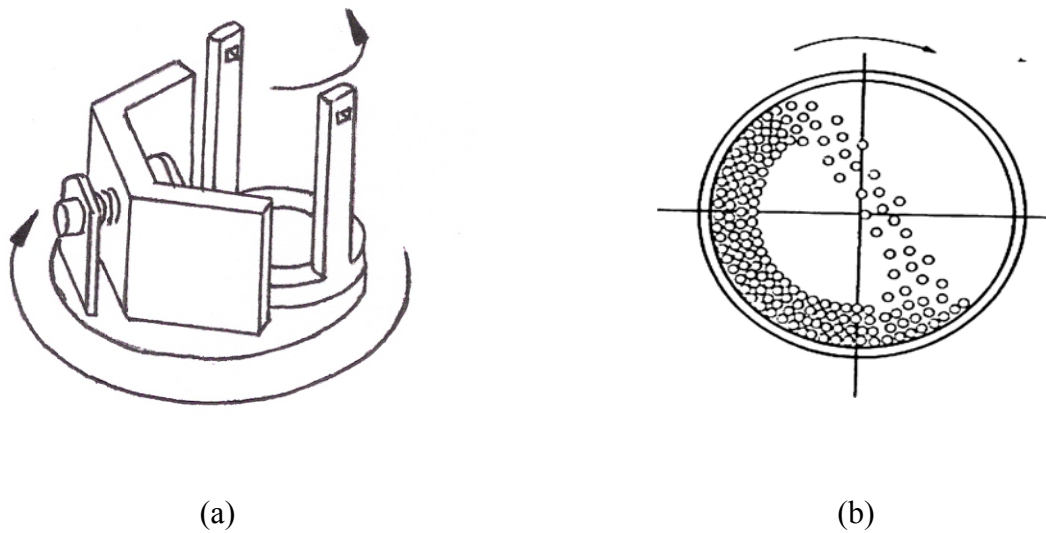


Fig. 3. Diagram showing the working principle of the planetary mono ball mill; (a) opposite rotational directions of grinding bowl holder and support disc; and (b) top view of balls running along inner wall of grinding bowl (Fritsch 2005; Prasad 2005).

Experimental design

A randomized complete block design was used for the hemp decortication experiment. Two experimental factors were used: seven rotational grinding speeds (100, 150, 200, 250, 300, 350, and 400 rpm) and three grinding durations (2, 4, and 6 min). Factorial combinations of grinding speed with grinding duration gave 21 treatments. These speeds and durations were selected based on preliminary trials using different speeds and durations to determine an appropriate range of speeds and grinding durations. Results from preliminary trials showed that grinding durations greater than 6 min, in combination with the grinding speed greater than 400 rpm, produced entirely chaff (fine particulate matter). A total of four replications were performed. Thus, a total of 84 tests (7 rpm grinding speeds x 3 grinding durations x 4 replications) were performed.

Test procedure

Hemp feedstock was cut to a length of 40 mm (Fig. 4a) using a heavy-duty paper cutter. A length of 40 mm was chosen as it was the most appropriate length for the production of biocomposites. Three gram samples were selected based on the ball mill capacity for hemp feedstock. Sample masses were weighed using a Mars electronic scale (Model MS200, North York, Canada). The 3 g sample was placed on top of the grinding balls in the bowl (Fig. 4b), and the bowl was clamped into the bowl holder. The bowl was securely fastened and the lid was closed, milling commenced for the appropriate grinding duration and grinding speed. Once milling stopped, the ground sample was then collected and placed in small plastic bags. The grinding bowl, grinding balls, and ring seal were cleaned between milling tests.



(a)



(b)

Fig. 4. Hemp feedstock (a) 40 mm cut lengths; and (b) loaded in grinding bowl prior to milling.

Measurements

Ground samples were separated using a stainless steel sieve shaker (Retsch AS200, Hann, Germany) (Fig. 5). The two screen opening sizes used were: 20 and 5 mm. Thus, the sample was separated into three lengths: Long (≥ 20 mm); Short (5-20 mm); and Chaff (< 5 mm). Following sieving, samples from each length distribution were separated into three products: fibre (the outer fibrous layer of the plant), fibre bound to core (the effectiveness in which the inner woody core layer remains attached to the outer fibrous layer), and core (the inner woody layer of the plant). Each fraction was weighed using a Mars digital scale (Model MS200, North York, Canada). The mass of each fraction was recorded. The designations of the mass fractions measured are summarized in Table 1.



Fig. 5. Retsch sieve shaker: screens and collecting pan, used for sizing.

Table 1. Summary of mass fractions of hemp products.

Output	Mass fraction		
	Long	Short	Chaff
Fibre	m_{Lf}	m_{Sf}	
Fibre/core	$m_{Lf/c}$	$m_{Sf/c}$	
Core	m_{Lc}	m_{Sc}	
Total	m_{Lt}	m_{St}	m_{chaff}

Fibre was the desired end-product of the decortications process while core was the by-product. Fibre bound to core was the undesired product. In practice, if fibre bound to core go to the fibre stream, it will decrease the purity of the fibre; if fibre bound core go to the core stream, it will decrease the fibre yield or increase the fibre loss. The following parameters were derived from those fibre fractions listed in Table 1 to evaluate the performance of the ball mill and the milling process.

Fibre yield Fibre yield was defined as the mass of fibre after milling divided by the mass of feedstock. Long fibres (>20 mm) were expected to have more applications than Short fibres (5-20 mm). Thus, yields for Long and Short fibres were determined separately, using the following equations:

$$Y_{Lf} = \frac{m_{Lf}}{M} 100 \quad (1a)$$

$$Y_{Sf} = \frac{m_{Sf}}{M} 100 \quad (1b)$$

$$Y_f = \frac{m_{Lf} + m_{Sf}}{M} 100 \quad (1c)$$

where:

Y_f , Y_{Lf} , and Y_{sf} = yield for total, long, and short fibre (%), respectively,
 m_{Lf} and m_{sf} = mass of long and short fibre (g), respectively, and
 M = mass of feedstock sample (3g).

Core yield Core yield was defined as the mass of core after milling divided by the mass of feedstock. As cores were the by-product, Long cores (>20 mm) and Short cores (5-20 mm) were expected to have similar applications. Thus, yields for Long and Short cores were combined to the total core yield:

$$Y_c = \frac{m_{LC} + m_{SC}}{M} 100 \quad (2)$$

Where:

Y_c = core yield (%),
 m_{LC} = mass of Long core (g),
 m_{SC} = mass of Sort core (g), and
 M = mass of feedstock sample (3 g).

Chaff Chaff was defined as those particles smaller than 5 mm in length. It was determined using the following equation:

$$Ch = \frac{m_{chaff}}{M} 100 \quad (3)$$

Where:

Ch = percentage of chaff (%),
 m_{chaff} = mass of chaff (g), and
 M = mass of feedstock sample (3 g).

Effectiveness To assess the effectiveness of the decortication process, the reduction in the amount of fibre bound to core was determined. This parameter shows how much fibre was detached from the core for a given grinding speed and duration. The effectiveness of decortication was defined as:

$$e_L = \frac{M - m_{Lf/c}}{M} 100 \quad (4a)$$

$$e_S = \frac{M - m_{Sf/c}}{M} 100 \quad (4b)$$

$$e_T = \frac{M - m_{Lf/c} - m_{Sf/c}}{M} 100 \quad (4c)$$

Where:

e_L , e_S , and e_T = effectiveness of decortication for long, short, and total (%), respectively,

$m_{Lf/c}$ and $m_{Sf/c}$ = mass of long and short fibre bound core (g), respectively, and,

M = mass of feedstock sample (3 g).

Data analysis Analysis of variance (ANOVA) was performed on fibre yield results for each length distribution for the varying treatments with Statistical Analysis Software (SAS/STAT V9.13) to detect any significant differences. Statistical Analysis Software macro procedure “pdmix800.sas” was used in combination with LSD test to detect differences among treatment means at a significance level of 0.05.

RESULTS AND DISCUSSION

The results of the ANOVA test showed an interaction result for total fibre, long fibre yield, and chaff, but no interaction for short fibre yield, core yield, or effectiveness (Table 2). Following significant interactions, simple effects were presented to observe the differences among the means. In cases where no significant interactions were present, main effects were presented.

Table 2. Analysis of variance (ANOVA) for ball mill.

Variables	Source					
	Duration		Speed		Duration*Speed	
	F	Pr>F	F	Pr>F	F	Pr>F
<i>Fibre yield</i>						
Long fibre	5.17	0.0085*	10.58	<0.0001*	10.85	<0.0001*
Short fibre	4.28	0.0183*	2.97	0.0131*	0.90	0.5506
Total fibre	7.70	0.0011*	12.93	<0.0001*	3.41	0.0230*
<i>Core yield</i>	2.29	0.1100	7.24	<0.0001*	1.27	0.2602
<i>Chaff</i>	11.45	<0.0001*	55.73	<0.0001*	3.85	0.0002*
<i>Effectiveness</i>						
Long	0.61	0.5451	50.11	<0.0001*	1.07	0.3988
Short	0.28	0.7581	2.46	0.0340*	0.61	0.8237
Total	0.35	0.7073	63.59	<0.0001*	1.20	0.3013

* Interactions are present for the source (P<0.05)

Fibre yield

The effects of grinding speed and duration for short fibre yield can be found in figures 6a and 6b. The effect of grinding duration resulted in a decrease of short fibre yield after 2 min. Results indicated that with grinding speeds of 400 rpm, fibre bundles were broken down into more individual fibres, thus, decreasing the short fibre yield. These results agreed well with an early study done by Wang and Chen (2008), who observed that with longer grinding durations fibre becomes more refined.

Grinding speeds and grinding durations affected the total and long fibre yield results. Results from total and long fibre yield showed no significant differences among grinding speeds for the 2 min duration among all levels of grinding speed (Figs. 7a and 7b). However, significant

differences were detected for the 4 and 6 min durations. Results showed that grinding speeds greater than 250 rpm caused a decrease in both long and total fibre yield. These results are in agreement with Prasad et al. (2005) who observed that with intense milling some fibre bundles are opened up and become powder.

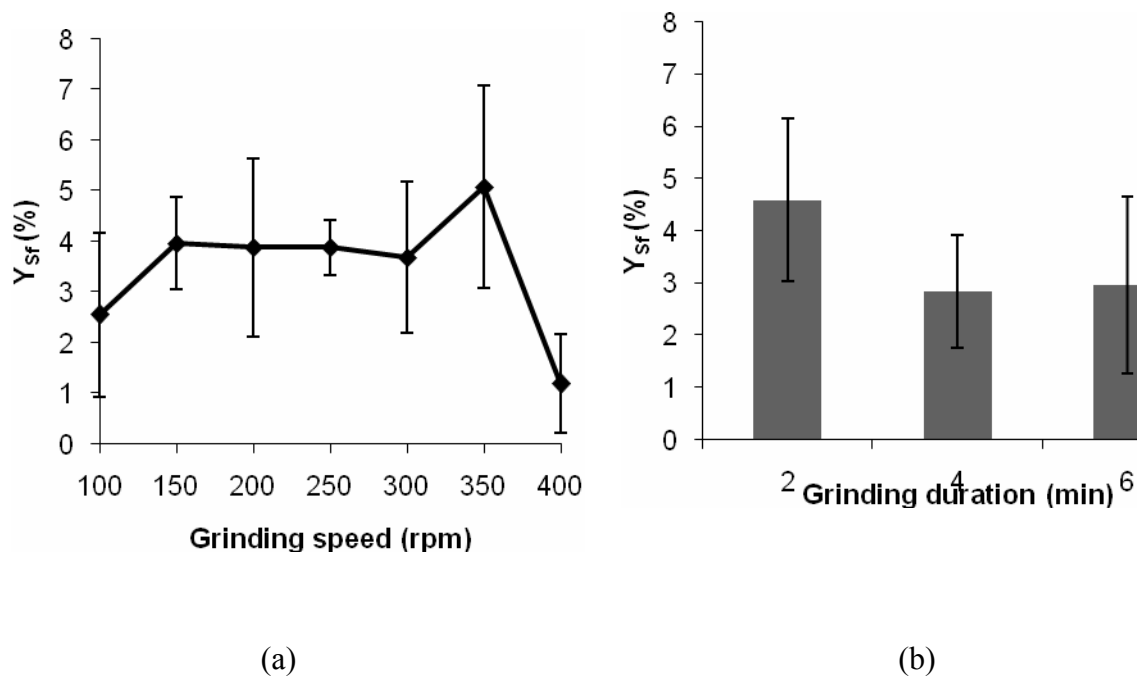
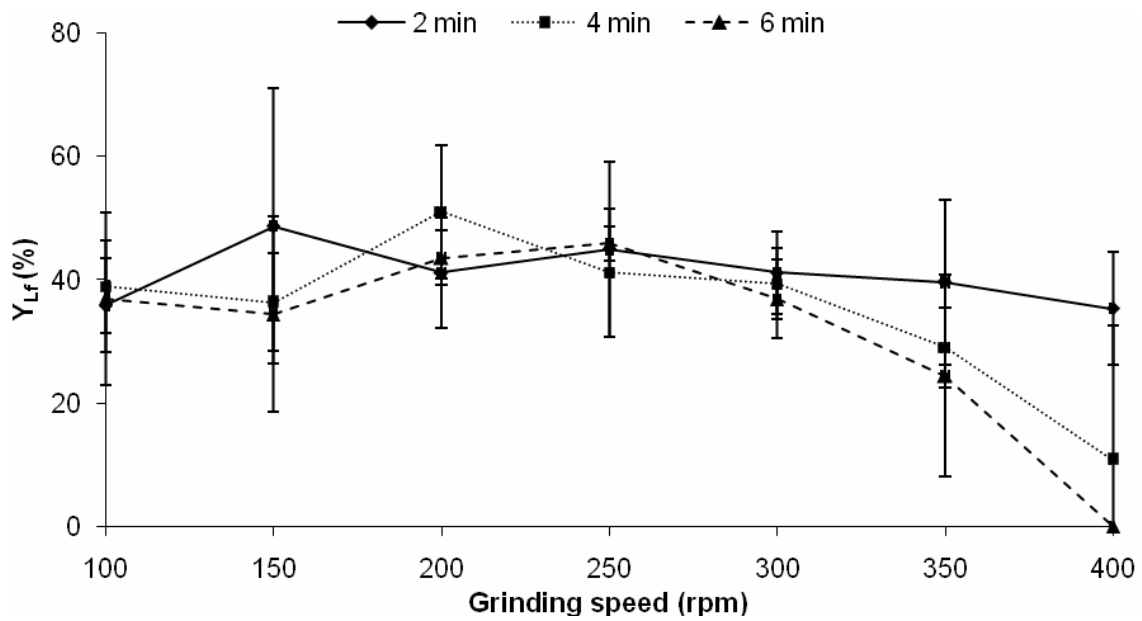
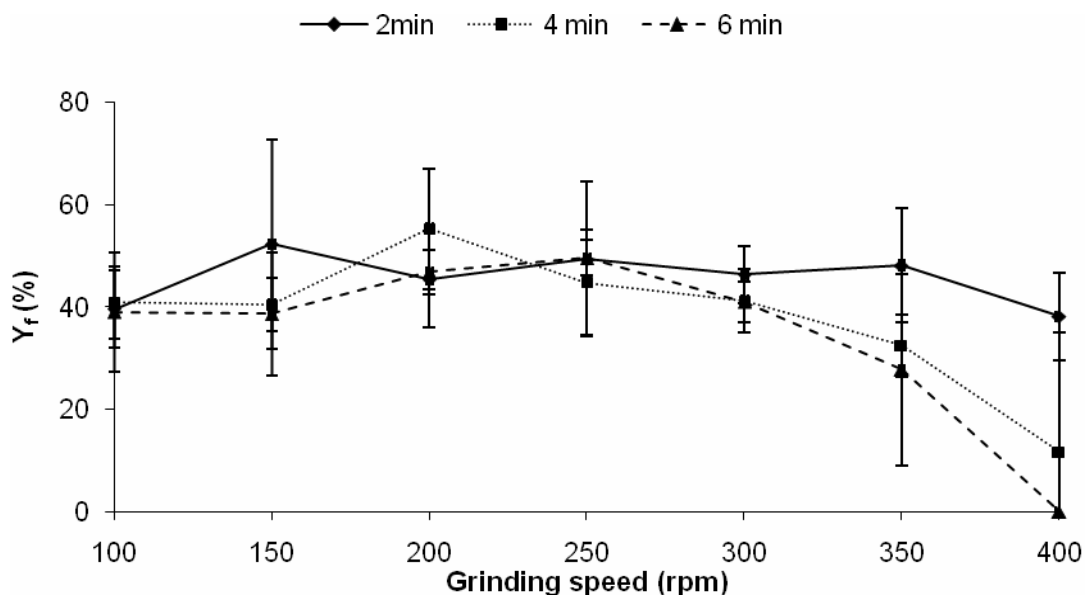


Fig. 6. Short fibre yield (Y_{Sf}); (a) for different grinding speed; and (b) grinding durations.



(a)



(b)

Figs. 7. Fibre yield results of grinding durations of 2, 4, and 6 min for (a) long fibre yield (Y_{Lf}) fibre; and (b) total fibre yield (Y_f).

Core yield

Core, the inner woody portion of hemp stocks, is classified as a by-product of hemp decortication. Results from the ANOVA (Table 2) showed no interaction result and only grinding speed had a significant effect on the total amount of core produced. A grinding speed of 200 rpm produced the greatest amount of core (Fig. 8a). Results showed that, for both grinding speed and grinding duration, the total core amount decreased with greater grinding speeds and longer grinding durations (Figs. 8a and 8b). With the faster speeds and longer durations, core fractions were reduced in size and then lost to waste. Results agree well with a study done by Hobson et al. (2001), who observed that with vigorous processing, core was reduced in size and ultimately lost to the waste fraction.

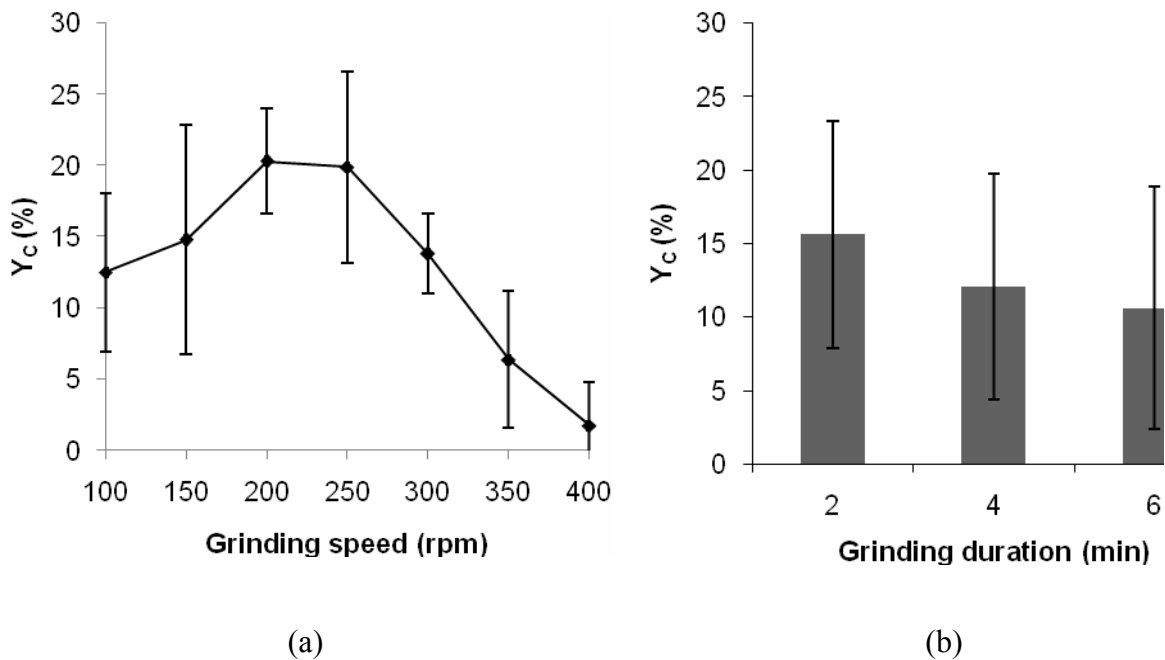


Fig. 8. Core yield (Y_c) results due to (a) grinding speeds; and (b) grinding durations.

Chaff

Material less than 5 mm in length, and dust fragments, was classified as chaff since it was not an acceptable length for biocomposite applications. The ANOVA (Table 2) showed that the effects of grinding speed and grinding duration on waste was significant. Results showed that with increasing grinding speed and grinding duration, the amount of chaff produced also increased (Fig. 9). The low moisture content of the samples (9.7%) may have contributed to the ease of decortication with extreme grinding combinations of speed and duration. These results agreed well with a study by Kymäläinen et al. (2001), who observed that a greater amount of core was due to the degree of retting; the greater the degree of retting the weaker the bonds between fibre and core. The grinding speed of 400 rpm and grinding duration of 6 min resulted in the greatest amount of chaff production. For the 2 min grinding duration, there was no significant difference in chaff production among the grinding speeds until 300 rpm. Speeds slower than 300 rpm did not produce enough momentum in 2 min to decorticate the sample.

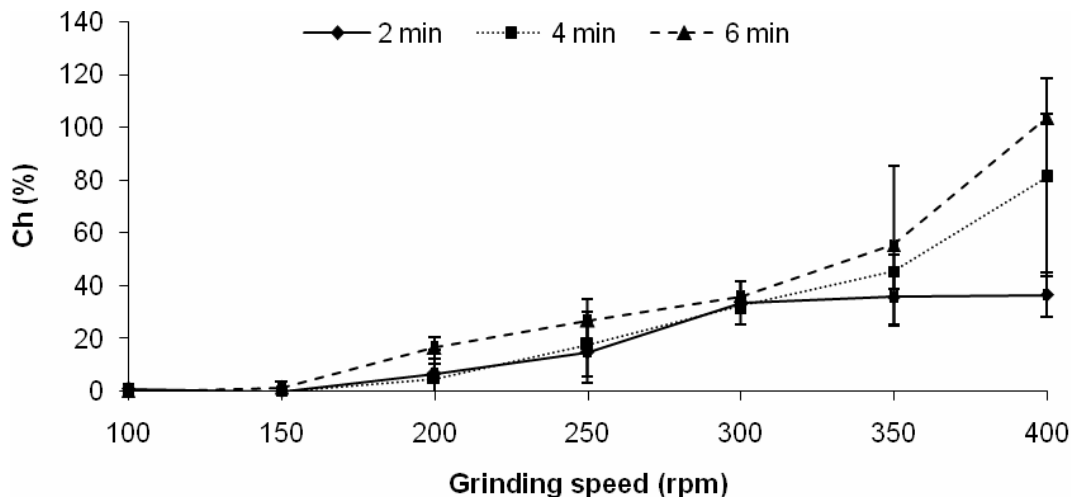


Fig. 9. Effects of grinding speed and grinding durations of 2, 4, and 6 min on chaff production.

Effectiveness

The effect of grinding speed and grinding duration were not significant within the range of this study for long, short, and total effectiveness (Table 2). The effects of grinding duration are presented in figures 10. The longest grinding duration (6 min) resulted in the greatest amount of long and total fibre effectiveness. For short effectiveness, 4 min grinding duration resulted in the greatest effectiveness (Fig. 10b). The range of grinding duration was not great enough to show a significant difference in the effectiveness of the ball mill for decortication. Due to the high degree of retting of the samples, the hemp stock may have already been broken down into more individual fibres. In an experiment done by Hepworth et al. (2000), it was noted that, in retted and decorticated hemp, the hemp is broken down from strips of tissues to bundles and individual cells.

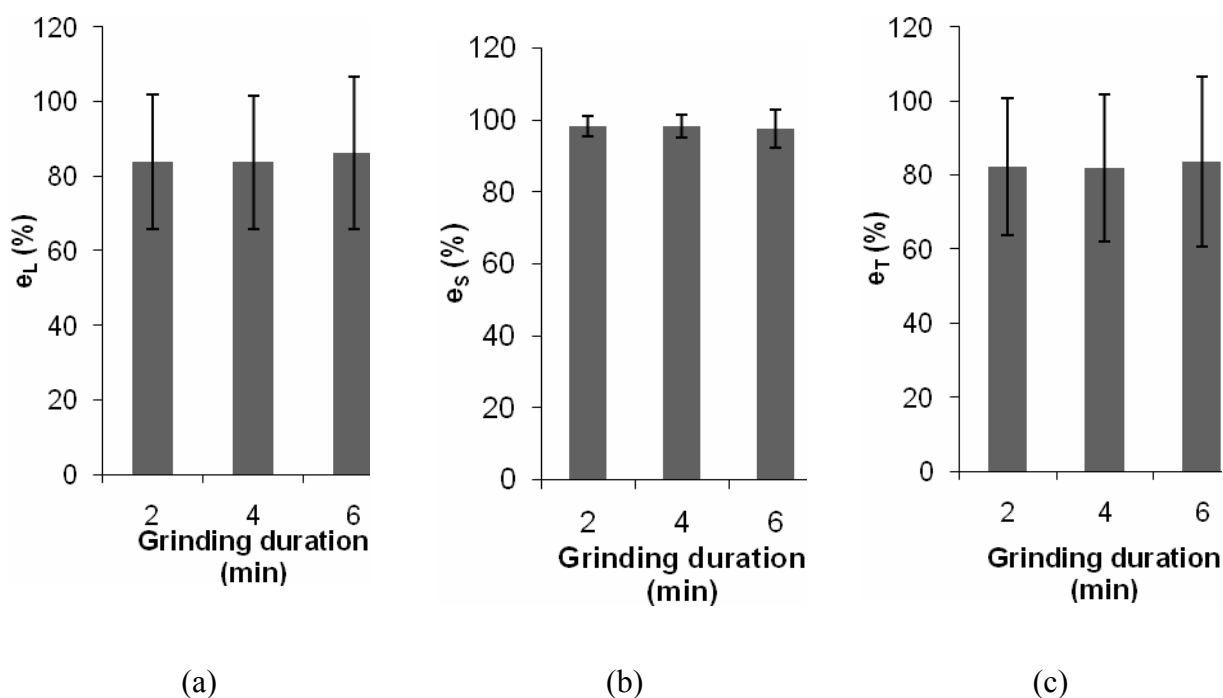
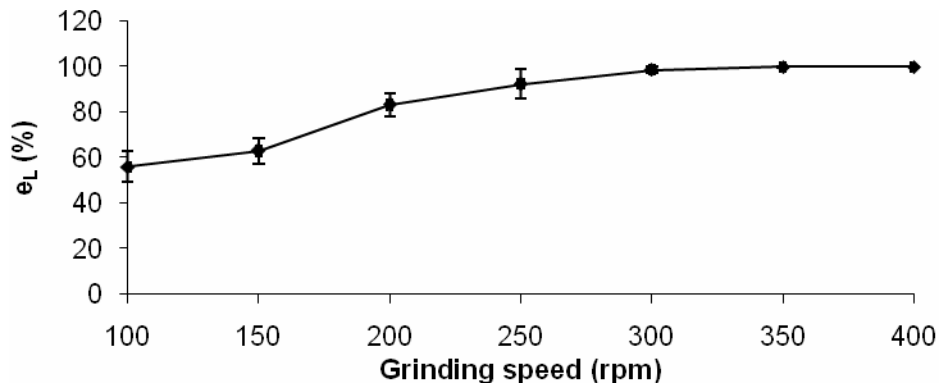
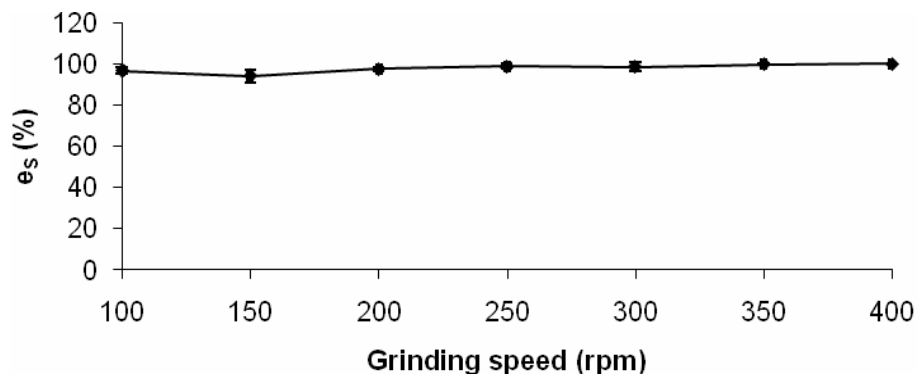


Fig. 10. Effectiveness results due to grinding duration for (a) long (e_L); (b) short (e_S); and (c) total (e_T).

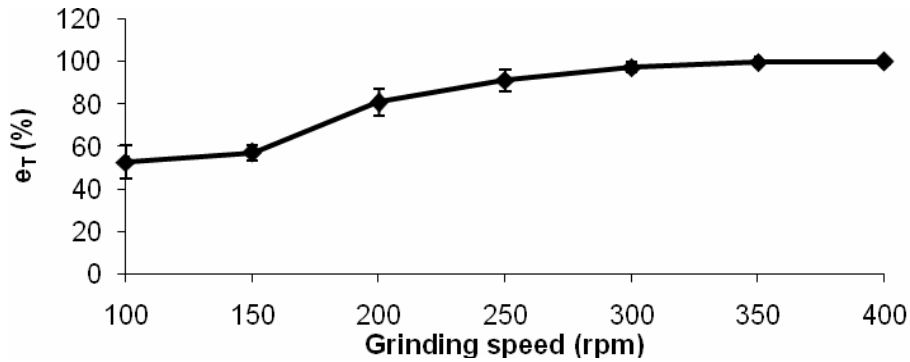
Grinding speed effects on effectiveness were significant for long, short, and total fibre effectiveness (Table 2). Results indicated that with increasing grinding speed the long, short, and total effectiveness increased (Figs. 11). With faster grinding speeds more momentum was generated to break apart the fibre bound to core in the original samples, and produce more pure fibre and pure core final products. These results are in agreement with Schwanninger et al. (2004) who observed that intensive ball milling resulted in a greater percentage of fine materials in the sample.



(a)



(b)



(c)

Fig. 11. Effectiveness results due to grinding speed for (a) long (e_L); (b) short (e_S); and (c) total (e_T).

CONCLUSIONS

Grinding speed and duration have an effect on fibre yield, core yield, chaff, and effectiveness. An interaction between the two parameters was observed for long and total fibre yield, but not for short fibre yield. Grinding speeds greater than 250 rpm resulted in a decrease in long and total fibre yield. The optimal condition for the greatest production of long fibre was 200 rpm for 4 min (51.00%). Short fibre yield decreased after 2 min grinding duration from 4.59% to 2.85% and 2.96% for 4 and 6 min, respectively.

Core yield was greatest for a grinding speed of 200 rpm (20.31%) and significantly decreased with increasing speeds. For the extreme speed of 400 rpm, core yield was ultimately lost to the chaff fraction. An interaction was observed for chaff production. Results indicated that with an increase in grinding speed and duration, chaff production also increase.

No interaction was observed for long, short, or total effectiveness. Grinding durations longer than 2 min resulted in the greatest effectiveness. Grinding speed and effectiveness showed a direct relationship; as speed increased so did long, short, and total effectiveness.

These conclusions are limited on account that the study was carried out with a hemp stock which had very low moisture content (9.7%) and only three grinding durations. Further research is required to confirm the above conclusions.

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