THE USE OF TWIN-SCREW EXTRUSION IN PROCESSING OF WOOD: THE EFFECT OF PROCESSING PARAMETERS AND PRETREATMENT

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In this study the effect of processing parameters on different types of wood raw material in extrusion was examined. The study consisted of two parts: the first part was to break and separate individual fibers from wood chips during the extrusion process; in the second part the effect of chemical pre-treatment and screw elements on wood raw material was evaluated. Statistical analysis was performed to evaluate the most important factors affecting wood particle size in extrusion. The statistical analysis showed that the screw speed is the main factor affecting wood fiber length in twin-screw extrusion of wood chips. This study showed that a twin-screw extruder can be used to separate individual fibers from wood chips, and the separated fibers have higher aspect ratios than the wood flour particles typically used in wood-polymer composites. When more fibrous and chemically softened wood raw material was used, fibers with even higher aspect ratios were obtained.

Keywords: Wood; Twin-screw extrusion; Processing parameters; Statistical analysis

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INTRODUCTION

Wood-polymer composites (WPC) are typically described as materials that combine the properties of wood and thermoplastic materials. The biggest market for WPCs at the moment is outdoor building materials, but automotive parts and furniture are also manufactured from WPCs (Clemons 2002). When compared with inorganic and synthetic fillers and reinforcements traditionally used in polymer composites, wood is considered a low-cost and more environmentally friendly raw material.

Most of the commercially manufactured WPCs today are made by compounding wood flour consisting of small wood particles with polyolefin polymer, e.g. polyethylene or polypropylene as matrix (Caulfield et al. 2005). Wood flour is produced from the wood residue of various wood processors by size reduction with different kinds of mills and size classification of the pulverized wood by screening (Clemons and Caulfield 2005). The particles in wood flour actually consist of fiber bundles, not individual fibers, and therefore the aspect ratio (length-to-diameter ratio) of wood flour is quite low, typically between 1 and 5 (Clemons 2008). Because of the low aspect ratio the reinforcing potential of wood flour is limited, and wood flour acts mainly as filler material in composites. The aspect ratio of a softwood fiber, for example, can be 100 (Smook 1999) and earlier studies have shown that wood fibers with a higher aspect ratio have better reinforcing capability in wood-polymer composites (Stark 1999; Stark and Rowlands 2003). However, fibers are seldom used in manufacturing of commercial WPCs because feeding and metering of low-bulk-density fibers is difficult and they are also more expensive than wood flour (Caulfield et al. 2005; Clemons and Caulfield 2005). Problems with poor dispersion of longer wood fibers in composites have also been reported (Klason et al. 1984; Rozman et al. 1998).

WPC research has strongly concentrated on enhancing the interfacial adhesion between the wood and polymer (Dalväg et al. 1985; Maldas et al. 1989; Raj et al. 1989; Felix and Gatenholm 1991; Coutinho et al. 1997; Ichazo et al. 2001), and the effect on wood characteristics is less frequently reported. The effect of wood particle size and aspect ratio on composite properties has been studied to some extent (Stark and Berger 1997; Stark and Rowlands 2003; Bledzki and Faruk 2003; Chen et al. 2006; Migneault et al. 2009; Bouafif et al. 2009), but only a few studies can be found where the effect of extrusion compounding on the dimensional properties of wood fibers and particles is examined (Bledzki et al. 2005; Le Baillif and Oksman 2009; Bouafif et al. 2010; Schirp and Stender 2010), even though twin-screw extrusion is one of the main processing methods used in the manufacture of WPC.

In twin-screw extrusion the material is subjected to shear forces, and the amount of shear force depends greatly on the used screw configuration. Shear forces could also be used to reduce the particle size and to separate fibers with a higher aspect ratio from the wood particles if suitable processing conditions can be found. Thus, the wood raw material would not need as much pre-processing as the commonly used wood flour requires before it can be compounded with plastic in an extruder. If larger, undried wood particles can be used as raw material in WPCs, use of cheaper wood residues in composite manufacturing without pre-processing could be possible.

In the current study the effect of extrusion parameters on different types of wood raw material was investigated. The study was made as a preliminary study with the goal of using wood chips as raw material in one-step extrusion of WPCs. If wood processing can be done in the same step as the compounding of WPC, then energy-consuming processing steps could be omitted from the composite manufacturing process. The main goal in this work was to produce wood particles/fibers with as high aspect ratio as possible. Basically, the study consisted of two parts. In the first part, the possibility to break down and separate individual fibers from wood chips in a co-rotating twin-screw extruder was examined. Statistical analysis was performed to evaluate the most important factors affecting wood particle size in extrusion. The effects of raw material moisture content, screw configuration, and screw speed on the aspect ratio of wood particles were evaluated using multiple linear regression analysis. In the second part of the study the effect of chemical raw material pre-treatment and reverse screw elements (RSE) on more fibrous wood raw material was evaluated.

The dimensional properties of the extruded wood fibers and particles were measured using optical fiber analysis. Scanning electron microscopy was also used to study the morphology of the samples. To avoid laborious matrix removal, all the experiments were carried out without addition of matrix polymer in the extruder. Therefore the extrusion conditions used in the study were more severe for the wood raw material than they would be with addition of polymer.

EXPERIMENTAL

Materials

Spruce (*Picea abies*) wood chips (Fig. 1a) from Finland were used as raw material in the first part of the study. The average wood particle size was of 16.2 mm × 3.3 mm. Wood chip particle size was determined by measuring the length and the width of 100 particles manually. Wood chips were stored frozen until use. Both untreated (defrosted) wood chips and wood chips pre-treated in boiling water for 4 minutes immediately before extrusion were used. The purpose of the pre-treatment was to soften the wood chips and thus avoid crushing them into small particles, thereby facilitating the separation of individual fibers. The initial dry matter content (before freezing) of the wood chips was *ca.* 51 wt%. After defrosting, the dry matter content of the non-treated wood chips was 57 wt%, and the dry matter content of the pre-treated wood chips was 30 to 35 wt%.

In the second part of the study mechanically pre-crushed spruce (*Picea abies,* from Finland) (Fig. 1b) was used as a raw material. This raw material was more fibrous than wood chips. The pre-crushed spruce was used as such (untreated) and as chemically pre-treated. The pre-treatment, a mild sulphonation was performed to achieve more softened raw material. The sulphonation was done at 90°C and at pH 9 for 210 minutes. The raw material was diluted to 10% consistency and 5% sodium sulphite, Na_2SO_3, was used in the treatment. The dry matter content of the untreated, pre-crushed spruce (referred as PC-U) was 46.3 wt%, and the dry matter content of sulphonated pre-crushed spruce (referred as PC-S) was 36.3 wt%.

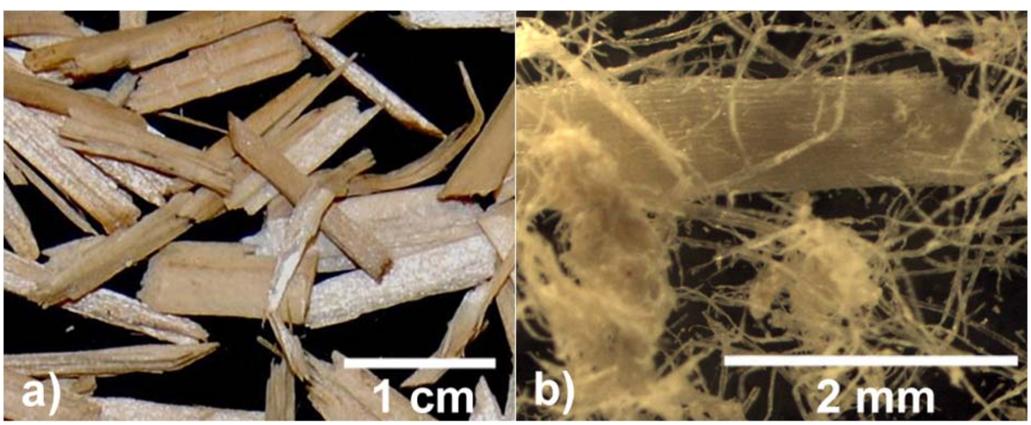

Fig. 1. a) Spruce wood chips used in this study; b) Pre-crushed spruce used in this study

Extrusion

A co-rotating twin-screw extruder (Coperion W&P ZSK-18 MEGALab, Stuttgart, Germany) without the extrusion die was used in the experiments. The screw length to diameter ratio (L/D) of the extruder was 40 with a screw diameter of 18 mm. The extruder consisted of seven (7) zones, and wood was fed into the first zone. Feeding of the wood chips was done manually, since the feeding equipment was not suitable for the materials used in the study. The extruder screws were fed as full as possible to maintain the filling degree as consistent as possible in all experiments.

The extruder screws had an elemental structure, and the screw elements used in this study were conveying elements, two types of kneading elements, and reverse screw elements. Conveying elements are used to take the material into the extruder and transport it downstream through the processing sections. Kneading elements are typically used for melting, dispersion, and mixing processes. Kneading elements with wide discs have a dispersive mixing effect while narrow kneading discs are more distributive. The reverse screw elements are conveying the material in the contrary direction and will resist the material flow effectively (Sämann 2008).

Screening of the important factors

The aim of the first part of this study was to determine the most important factors affecting wood aspect ratio in a co-rotating twin-screw extruder. MODDE 8 (Umetrics) statistical software and multiple linear regression analysis (MLR) were used to analyze the experimental data. A confidence level of 95% was used in the regression analysis. The experiments were performed according to a non-replicated 2^3 full-factorial design with three centre points. The total number of experimental runs was 11, and they were performed in randomized order. Experimental design is presented in Table 1. The factors studied were 1) rotational speed of the screws, 2) screw configuration, and 3) raw material treatment. The aspect ratio of wood particles after extrusion was used as a response.

Table 1. Experimental Design

Exp. no.	Run order	Raw material	Speed	Configuration*
1	11	non-treated	100 rpm	A
2	2	non-treated	500 rpm	A
3	10	non-treated	100 rpm	B
4	3	non-treated	500 rpm	B
5	1	pre-treated	100 rpm	A
6	6	pre-treated	500 rpm	A
7	8	pre-treated	100 rpm	B
8	4	pre-treated	500 rpm	B
9	5	non-treated	300 rpm	A
10	7	non-treated	300 rpm	A
11	9	non-treated	300 rpm	A

*A: screw configuration with WKE, B: screw configuration with WKE and RSE.

The rotational speeds used were 100, 300, and 500 rpm. The speed 300 rpm functioned as a centre point in the design. The rotational speeds were chosen as extreme as possible, but there were some limitations when selecting suitable levels. 100 rpm was chosen as the lowest rotational speed to keep the processing times reasonable, and 500 rpm was chosen as highest rotational speed to avoid exceeding the maximum motor load when using the screw configuration with reverse screw element. The two different screw configurations used are presented in Fig. 2. Configuration A consisted of conveying elements and one wide kneading element (WKE), and configuration B consisted of conveying elements with one wide kneading element (WKE) followed by reverse screw element (RSE). The screw elements were chosen so that configuration A would be gentler than B. Pre-runs made with different screw configurations also showed that using of wide kneading element in front of RSE was necessary to avoid motor overload of the extruder when using wood chips as raw material. The raw material used was either non-treated or pre-treated wood chips. To avoid excessive drying or burning of the wood in the extruder, the barrel temperature was set to 80°C in all experimental runs.

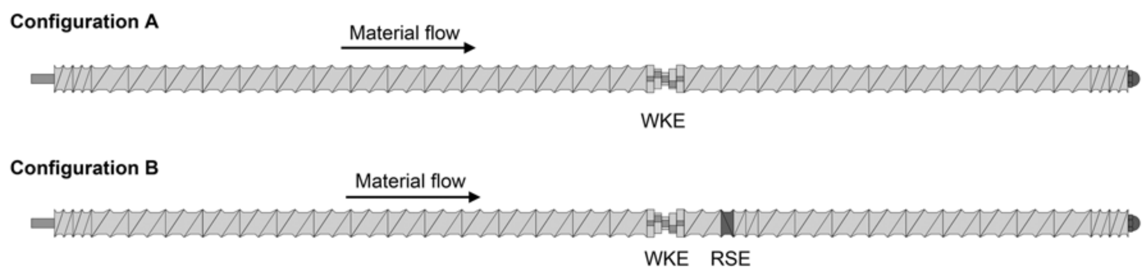

Fig. 2. Screw configurations A and B. WKE = wide kneading element; RSE = reverse screw element

Effect of raw material and screw configuration

Because the pre-treatment of the wood chips in the first part of the study did not increase the wood fiber aspect ratio, it was decided to use a more fibrous raw material with and without chemical pre-treatment in the second part of the study. Due to the change in raw material, reverse screw elements, RSE, could now be used as the first screw element. Three different screw configurations were used in the experiments (Fig. 3). Configuration C had one reverse screw element (RSE), configuration D had two RSEs, and configuration E had two RSEs with a thin kneading element between them (TKE) in addition to conveying elements (Fig. 3). The temperature of the extruder barrel was set to 70°C to avoid unwanted drying of the raw material. Screw speed in all experimental runs was 500 rpm. 500 rpm was chosen based on the results from the first part of the study.

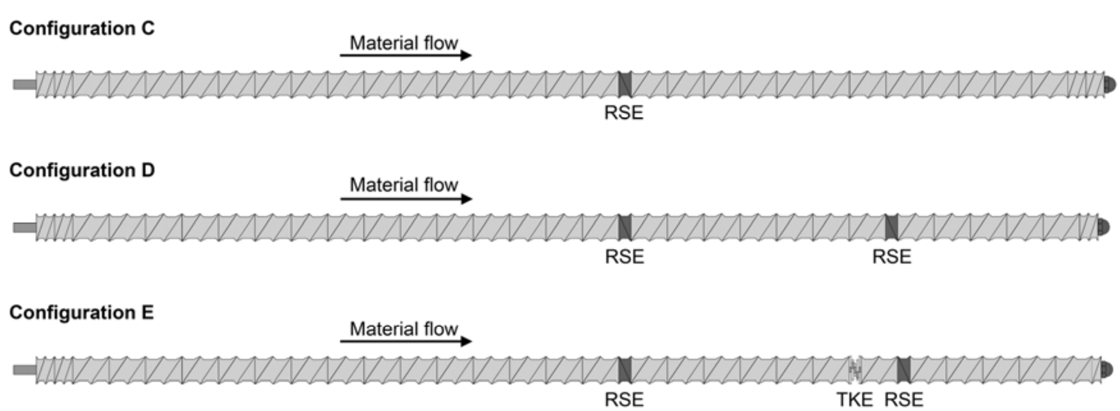

Fig. 3. Screw configurations C, D and E. RSE = reverse screw element, TKE = thin kneading element.

Characterization

The average fiber length (length weighted) and average fiber width used to calculate the aspect ratio (l/d) were measured using a FiberLab optical fiber analyzer (Metso Automation, Kajaani, Finland). Morphology of selected samples was studied using a field emission scanning electron microscope, FESEM (Zeiss ULTRA Plus, Carl Zeiss SMT AG, Jena, Germany) and scanning electron microscope, SEM (Jeol JSM-6400, Jeol Ltd., Tokyo, Japan). The samples for scanning electron microscopy were freeze dried and coated with platinum (FESEM) or gold (SEM) before examination.

In the experiments in which wood chips were used as raw material the samples were fractionated into four different size categories before measuring the fiber lengths. The fractionation was done using a tube flow fractionator (Metso Automation, Kajaani, Finland). The purpose of the fractionation was to remove the first fraction containing the largest wood particles (shives) from the sample, thereby preventing blockage of the FiberLab analyzer. The average fiber lengths were then measured by combining the fractions 2, 3 and 4 from the tube flow fractionator into one sample. The tube flow fractionation method is described in more detail in the following publications: Krogerus et al. (2003) and Laitinen et al. (2006). The shive content of the samples in which wood chips were used as raw material were measured according to TAPPI T275 standard (Somerville method, 0.10 slots).

In addition, a Bauer McNett classifier was used to divide samples into fractions R14, R28, R48, and R200 before examination with SEM in the second part of the study. The R14 fraction is typically considered as the shive fraction, R28 as the long fiber fraction, R48 as the short fiber fraction and R200 as the fines fraction.

RESULTS AND DISCUSSION

Screening of the Important Factors

The average fiber length (length weighted), fiber width, aspect ratios (l/d) calculated from the length and width measurements, as well as the shive contents of the samples are presented in Table 2.

Table 2. Average Fiber Length, Average Width, and Aspect Ratio of Samples

Exp.	Raw material	Speed, rpm	Screw configuration	Length, L(l), mm	Width, μm	Aspect ratio, l/d	Shives, %
1	non-treated	100	A	0.65	31.1	20.9	38.5
2	non-treated	500	A	0.68	31.2	21.8	47.9
3	non-treated	100	B	0.55	30.6	18.0	6.9
4	non-treated	500	B	0.68	30.9	22.0	9.3
5	pre-treated	100	A	0.59	29.9	19.7	33.1
6	pre-treated	500	A	0.67	29.4	22.8	47.6
7	pre-treated	100	B	0.55	28.9	19.0	5.4
8	pre-treated	500	B	0.67	27.8	24.1	13.2
9	non-treated	300	A	0.64	30.7	20.8	43.7
10	non-treated	300	A	0.67	30.3	22.1	44.1
11	non-treated	300	A	0.63	30.5	20.7	46.2

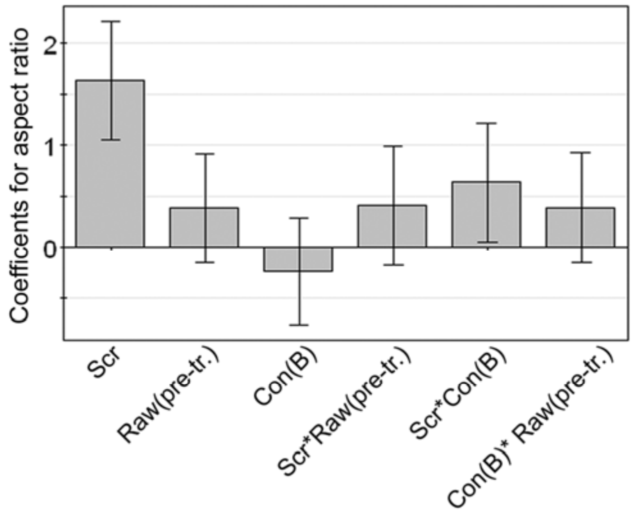

Fig. 4. Scaled and centered regression coefficients for aspect ratio before model reduction

The statistically significant factors affecting the aspect ratio of wood raw material in twin-screw extrusion were estimated using MODDE 8 statistical software and multiple linear regression analysis (MLR). Figure 4 shows the coefficient plot for the aspect ratio before reducing the insignificant factors from the model. The insignificant factors were removed from the model, and the results from the regression analysis for the reduced model are presented in Tables 3 and 4. Table 3 shows the regression coefficients for the significant (α = 0.05) factors affecting the aspect ratio of wood raw material in a co-rotating twin-screw extruder together with the R^2, R^2(adj.), and Q^2 parameters measuring the goodness of fit of the model. The values of these parameters presented in Table 3 indicate that the model fit the data moderately. According to the MLR analysis, screw speed (Scr) had the largest effect on aspect ratio, and it was also the only significant factor with a 95% confidence level affecting the aspect ratio. The final model for aspect ratio can be written as:

$$\textit{Aspect ratio}, l/d = 18.6256 + 0.0082 \cdot \textit{screw speed (rpm)} \tag{1}$$

The analysis of variance, ANOVA, for the fitted model is presented in Table 4 showing the P-value of 0.399 indicating that there was no lack of fit.

Table 3. The Unscaled regression coefficients for the fitted model for aspect ratio

Response	Constant	Scr	Con	Raw	Scr×Con	Scr×Raw	Con×Raw
Aspect ratio	18.6256	0.0082	-	-	-	-	-
	R^2 =0.704, R^2(adj.) =0.671, Q^2= 0.536						
	Scr = screw speed, Con = configuration, Raw = raw material treatment.						

From Table 3 it can be seen that the shive content of the samples made using configuration B were clearly lower in comparison with samples made with configuration A. Therefore it seems that the use of RSE in the screw configuration subjected the material to more shear, thus reducing the shive content of the samples. According to the statistical analysis and the results presented in Table 3, the raw material pre-treatment did not have much effect either on the aspect ratio or the shive content of the samples. Therefore it was thought that the pre-treatment was insufficient.

Table 4. Analysis of Variance (ANOVA) for the Fitted Model for Aspect Ratio

Response	Source	DF	SS	MS	F-value	P-value
Aspect ratio	Regression	1	21.45	21.45	21.44	0.001
	Lack of Fit	7	7.79	1.11	1.82	0.399
	Residual	9	9.01	1.00		
	Pure Error	2	1.22	0.61		
	Total	11	4919.33	447.21		
	DF = degrees of freedom, SS = sum of squares, MS = mean squares.					

FESEM pictures from experiments No. 2, 4, 6 and 8 are presented in Fig. 5. These samples were chosen for the analysis because they had the longest fiber lengths and highest aspect ratios according to the analysis. The screw speed in these four experiments was 500 rpm. From Fig. 5 it can be seen that the samples in which screw configuration A (configuration with wide kneading element, WKE) was used (exp. 2 and 6) contained more coarse material (more shives) in comparison with the samples in which the screw configuration B was used (exp. 4 and 8). However, only minor differences can be seen when comparing the non-treated and pre-treated samples made with same screw configurations (exp. 2 and 6, exp. 4 and 8).

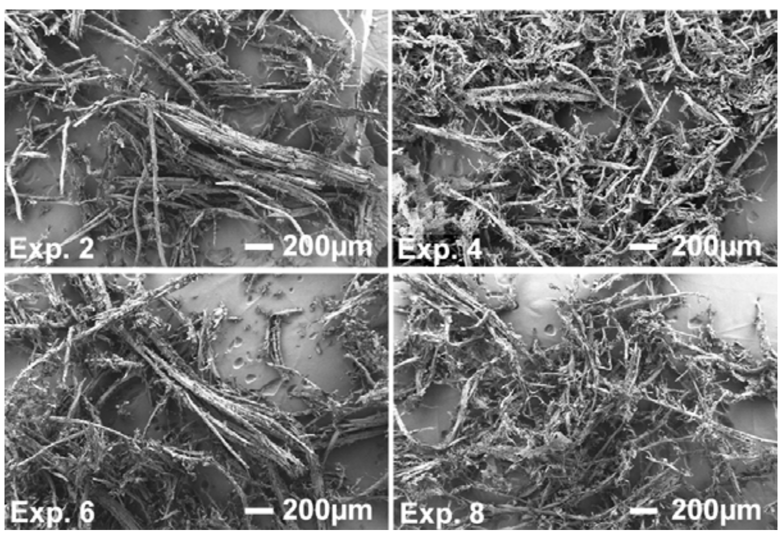

Fig. 5. Scanning electron microscopy images of experiments No. 2, 4, 6 and 8.

Even though the regression analysis showed that the screw configuration did not have a significant effect on aspect ratio and it had only a very small effect on fiber length, it was thought that the real effect of screw configuration was not clearly seen in this experiment. The wide kneading element, WKE, was used in both screw configurations as the first element to avoid motor overload, and it was suspected that the results actually show that the reverse screw element (RSE) had a small fiber shortening effect after the wood had first gone through the wide kneading element. Thus, it is possible that the kneading element was responsible of the extensive fiber shortening.

Effect of Raw Material Type and Reverse Screw Elements

The results from the average fiber length and fiber width measurements together with the calculated aspect ratios are presented in Table 5. In these experiments the pre-crushed, more fibrous wood raw material was used.

Table 5. Fiber Properties of the Pre-Crushed Spruce after Extrusion

Exp.	Configuration	Length, L(l), mm	Width, μm	Aspect ratio, l/d
PC-U1	C	1.21	27.3	44.3
PC-U2	D	0.82	25.0	32.8
PC-U3	E	0.85	25.5	33.3
PC-S1	C	1.59	30.0	53.0
PC-S2	D	1.20	27.9	43.0
PC-S3	E	1.24	27.8	44.6
	PC-U = pre-crushed, untreated spruce. PC-S = pre-crushed, sulphonated spruce.			

Based on the results obtained from the first part of this study, three different screw configurations were used in the experiments. The highest aspect ratios were achieved using screw configuration C (samples PC-U1 and PC-S1) with one reverse screw element. The screw configurations with two reverse screw elements (configuration D and E) were most likely too harsh; therefore, the aspect ratios were lower in the samples made with these. As expected, the sulphonation pre-treatment affected the aspect ratio of the extruded fibers positively. This indicates that a proper softening of the wood raw material is very important to gain high aspect ratio fibers in extrusion of wood raw material.

In Fig. 6 the SEM images of R14, R28, R48 and R200 mass fractions of two of the samples (PC-U1 and PC-S1) are shown. The samples were divided into fractions to achieve a better understanding of the morphology of the fibers. The images in Fig. 6 show that both of the samples contain individual fibers and only a small amount of shives. When comparing the images taken from the different fiber fractions, especially R14 fractions of the samples, it seems that PC-U1 contains coarser material than the PC-S1 sample (Fig. 6).

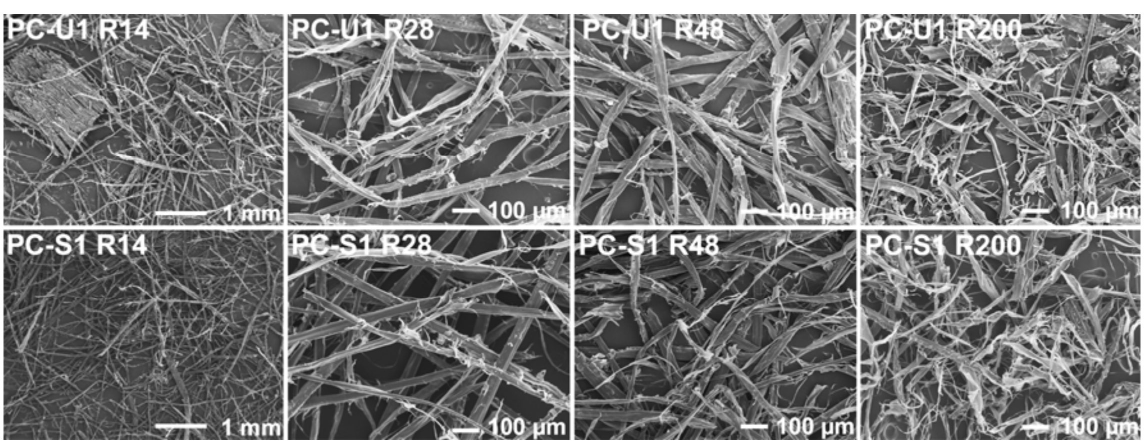

Fig. 6. Scanning electron microscopy images of R14, R28, R48, and R200 fractions of samples PC-U1 & PC-S1. PC-U = untreated pre-crushed spruce, PC-S = sulphonated pre-crushed spruce

CONCLUSIONS

This study shows that a twin-screw extruder can be used to separate individual fibers from wood chips. The separated fibers have clearly higher aspect ratios than the wood flour particles typically used in wood-polymer composites. When using more fibrous and chemically softened wood raw material, fibers with even higher aspect ratios were obtained. According to the statistical analysis, the screw speed was the main factor affecting wood fiber aspect ratio in twin-screw extrusion of wood chips. It was also observed that the screw configuration can affect the fiber properties and the reverse screw elements reduced the shive content in the samples.

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Article submitted: July 21, 2011; Peer review completed: August 31, 2011; Revised version received and accepted: September 22, 2011; Published: September 24, 2011.