

Effect of Refining on the Properties of Fibres from Young Scots (*Pinus sylvestris*) and Lodgepole Pines (*Pinus contorta*)

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Abstract

Pulp refining is a mechanical treatment of fibres using special equipment to initiate changes in structural and electro-kinetic characteristics, which lead to the improvement of the fibre quality. The nature and intensity of the fibre changes depend on several factors, including wood species. The objective of this study was to assess the impact of pine wood species to their processability during pulp refining. Two pine wood species, lodgepole (*Pinus contorta*) and Scots (*Pinus sylvestris*) pines, were mechanically treated and differences in fibre properties between pines were compared and estimated. A strong and significant correlation between the time of mechanical treatment (PFI mill beating for 5, 10 and 15 min..) of pulp (Shopper-Riegler number and water retention value) and mechanical, optical, and surface paper properties were determined. Higher refining grade and better water retention value and mechanical properties of paper were recognized in the case of lodgepole pine. Also, paper of beaten lodgepole pine fibres had better air permeability and surface smoothness. Light scattering and ISO brightness were higher for Scots pine; there was no difference between species in light absorption.

Key words: Fibre refining; Paper properties; Paper optics; Water retention; Air permeability.

Introduction

Modification of the pulp or fibre quality to improve paper features is one of the most significant scientific challenges in the paper industry (Gharehkhani et al. 2015), and fibre deformations are proven to have a significant effect on the fibre strength and sheet properties (Zeng et al. 2012). Pulp refining or beating is a mechanical treatment of fibres using special equipment to initiate changes in structural and electro-kinetic characteristics, which lead to the improvement of the fibre quality and result in higher bonding in the fibre. The industry testifies (Ebeling 2000) that mechanical treatment increases the inter-fibre bonding and is the key component of quality for appropriate pulp production.

The nature and intensity of fibre changes depend on several factors, including the wood species (Joutsimo and Robertsen 2004, 2005, Joutsimo et al. 2005); even a difference among softwood fibres is found (Robertsen and Joutsimo 2005). External fibrillation, internal fibrillation (swelling), fines formation, fibre shortening, and fibre straightening are the properties that are changing in the refining process. Fibrillation is explained as a peeling-off mechanism –

the primary wall and secondary S₁-layer of fibre are peeled off, and the secondary S₂-layer is exposed to inter-fibre bond (Afra et al. 2013). This delamination is caused by the cyclic compression action of the forces inside the refiner. Gharehkhani (2015) also reports a differentiation in crystallinity and redistribution of surface chemical compositions.

Recent statistics of the European pulp and paper industry (CEPI 2014) report indicates 72.8% of softwood consumption in the total amount of virgin fibres and 41.1% of pine consumption in total amount of softwood fibres. Therefore, kraft pulp was chosen for attaining the target, considering that this is the main processing method for wood fibres, providing 65% of total pulp production (Gharehkhani et al. 2015) in Europe. Pine is the main wood species in Latvia, and it accounted for 34% of the total wood volume in 2014 (SFS 2014). To increase the source of fast-growing and quality wood, the introduction of lodgepole pine (*Pinus contorta*) began in the 1980s, following the tendencies in other Nordic countries (Elfving et al. 2001, Knight et al. 2001). Wood chips from Latvia are exported to pulp producing countries, mostly to Scandinavia. The quality and properties of fibres are an important factor for both wood pulp sales man-

agement and for developing a solution to the most efficient use of local renewable resources.

Investigations show that lodgepole pine produces 36% more wood than Scots pine (*Pinus sylvestris*) during a 25-year period. Productivity, stem and branch quality, and resistance to biotic and abiotic factors have been studied for the Latvian case (Jansons et al. 2009a,b, Sisenis et al. 2012); wood chemical content has been characterized and fibre properties, important for paper production, have been estimated for lodgepole pine in comparison to Scots pine (Sable et al. 2012a,b). However, the effect of mechanical pre-treatment of fibres of those tree species has not been analyzed and compared. Therefore, the objective of the present study was to explore and compare the properties of mechanically treated fibres of lodgepole and Scots pines to estimate their processability in obtaining pulp and its products.

Materials and Methods

Tree samples of pines at the age of 24 to 25 years were collected in 2009 and 2010 in an experimental site in the central part of Latvia. In total, 93 Scots pine sample trees and 26 lodgepole pine sample trees from three provenances from Canada were selected. More detailed sample procurement of trees is described in a previous investigation (Sable et al. 2012a).

Fibres were obtained through Kraft pulping in a 2-L laboratory digester at 170°C, process parameters: 57.4 g/L active alkali as NaOH, sulfidity 29.8%, and liquor to wood ratio 4.5 L/kg. Samples for refining and testing were selected randomly from all matrixes of Scots and lodgepole pine fibres.

Mechanical treatment of the fibres was performed according to ISO 5264-2: 2011 in a PFI MILL (IDM Test, Italy) for 5, 10, and 15 min. (energy consumption 0.07 ± 0.00 , 0.14 ± 0.00 , 0.21 ± 0.01 kWh or 7105 ± 37 , 14176 ± 69 and 21342 ± 73 revolutions). Shopper-Riegler number ($^{\circ}$ SR) was determined according to ISO 5267-1: 1999 with a Shopper-Riegler freeness tester (PTI, Austria); water retention value (WRV) was obtained according to ISO 23714: 2014, using a centrifuge T23 (MLW, Germany). Paper handsheets (5 replicates for every sample) were prepared according to ISO 5269-2: 2004 with a Rapid Köthen paper machine (PTI, Austria), and their thickness was measured according to ISO 534: 2011 with a micrometer F16502 (Frank-PTI, Austria). Samples for mechanical testing were prepared with a strip cutter and tested according to ISO 1924-2: 2008 and ISO 2758:2014, using a Tensile Tester Vertical F81838 and Burst Tester for Paper (both from Frank-PTI, Austria). Air permeability of paper handsheets was measured according to ISO 5636-3: 2013, using an air permeability tester (Lorentzen and Wettre, Sweden), and surface roughness was obtained following ISO 8791-2:2013, using a Bendsten Tester (Lorentzen and Wettre, Sweden). Brightness was measured according to ISO 2470-1: 2009, and light absorption and scattering were done following ISO 9416: 2009. All optical measurements were done with an Elrepho spectrophotometer

(Lorentzen and Wettre, Sweden). The ANOVA procedure was used for the determination of the factor (time of refining) effect on the inspected properties.

Results

Fibres of Scots pine demonstrated (Figure 1, 2) a higher $^{\circ}$ SR after 5 and 10 min. of refining (6% and 27%, respectively). After 15 min., the difference between the species was 22%, although the statistical procedure found it not significant ($P > 0.05$). With a similar amount of energy used for refining, lodgepole and Scots pine fibres reached 42 $^{\circ}$ SR and 54 $^{\circ}$ SR, respectively. Significantly ($P < 0.05$) higher WRV were observed for Lodgepole pine than Scots pine: 1.24 and 1.06, respectively. Results of WRV measurements showed a significant change in the value after every step of mechanical treatment for both species, except Scots pine in the time interval of 5 to 10 min. Water retention dynamically increased until 10 min. of treatment, but remained unchanged or even decreased, when the time of refining reached 15 min. Comparing the species, the results become almost same after refining, with a slight predominance of lodgepole pine.

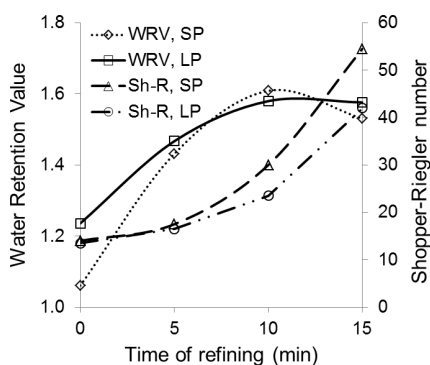


Figure 1. Effect of refining time on the Shopper-Riegler number and WRV of Scots and lodgepole pine fibers (LP – lodgepole pine, SP – Scots pine)

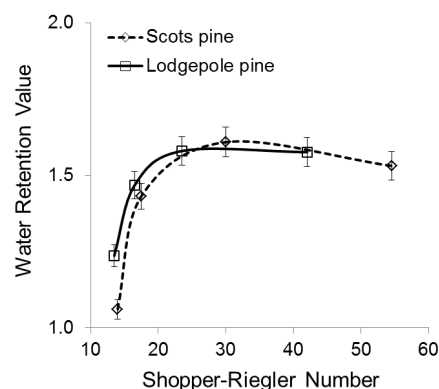


Figure 2. Effect of the Shopper-Riegler number on the WRV of Scots and lodgepole pine fibers (average values \pm standard deviation)

The changes in burst and tensile indices, tensile energy absorption, and breaking length of refined fibres are listed in Table 1. All parameters increased with an increase in refining time for both Scots pine and lodgepole pine. In our study, the starting number of burst index was lower in the case of lodgepole pine, but increased significantly after 5 to 15 min. of refining time and became significantly higher ($P < 0.05$) than in the case of Scots pine at the same refining time. Tensile index results also exhibited the same trend originally, but the lodgepole pine exceeded the Scots pine by more than 14% after 15 min. of refining.

Table 1. Effect of refining time on the mechanical properties of paper handsheets made from Scots and Lodgepole pine fibres (average \pm standard deviation)

Species	Refining Time (min.)	Burst Index (kPa m ² g ⁻¹)	Tensile Index (N·m g ⁻¹)	Tensile Energy Absorption (J m ⁻¹)	Breaking Length (km)
Scots pine	0	1.3 \pm 0.0	27.8 \pm 1.2	17.3 \pm 3.4	2.8 \pm 0.1
	5	4.7 \pm 0.2	70.8 \pm 2.9	94.0 \pm 12.7	7.2 \pm 0.3
	10	5.5 \pm 0.1	75.3 \pm 0.8	133.3 \pm 6.0	7.7 \pm 0.1
	15	6.1 \pm 0.3	81.2 \pm 4.4	147.6 \pm 19.3	8.3 \pm 0.5
Lodgepole pine	0	0.9 \pm 0.0	26.0 \pm 0.8	11.8 \pm 1.7	2.7 \pm 0.1
	5	5.5 \pm 0.1	76.9 \pm 0.5	127.7 \pm 12.2	7.8 \pm 0.0
	10	6.3 \pm 0.1	85.6 \pm 0.7	167.5 \pm 13.6	8.7 \pm 0.1
	15	6.9 \pm 0.1	92.8 \pm 5.3	166.8 \pm 16.6	9.5 \pm 0.5

The results showed a noticeable increase in tensile energy absorption already after 5 min. of fibre refining (five times in the case of Scots pine and more than 10 times in the case of lodgepole pine compared to untreated fibres), with a continuous increase after 10 and 15 min. refining.

The results showed a decrease in air permeability with an increase in the refining time (Figure 3). The results from the initial fibres and those after 5 and 10 min. of refining gave almost equal numbers, comparing the two pines. A statistically significant difference ($P < 0.05$) appeared after 15 min. of refining, 3.2 s in the case of Scots pine *versus* 1.9 s (40% less) in the case of lodgepole pine.

Our study showed the improvement of surface smoothness (Figure 3) with an increase in the refining time of fibres.

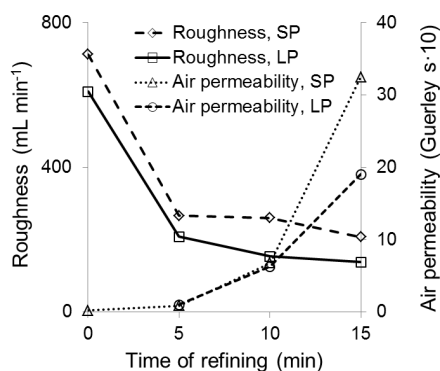


Figure 3. Effect of refining time on the air permeability and surface roughness of paper handsheets made of Scots and lodgepole pine fibers

The parameter changed significantly ($P < 0.05$) with refining in the case of both species, except for the differences in Scots pine for 5 and 10 min. of refining. Roughness was lower ($P < 0.05$) in all stages of the refining process in the case of lodgepole pine.

Our study revealed (Figure 4) decreases in light scattering as a result of 5 min. of refining of 35% and 43% in the case of Scots pine and lodgepole pine, respectively. Further reduction was rather equable, 5% to 8% of the value per each next 5 min. of refining. The tendency of changes in light scattering was similar for both species, but differences in values were statistically significant ($P < 0.05$), but Scots pine had higher results. The changes in light absorption with increasing refining time were statistically insignificant.

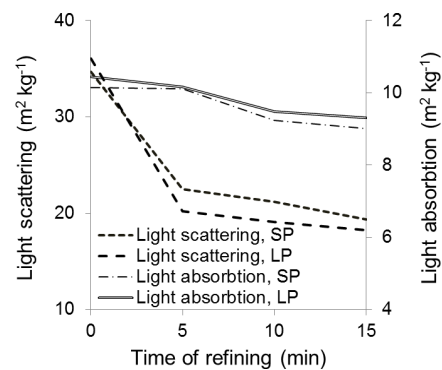


Figure 4. Effect of refining time on light scattering and absorption of paper handsheets, made of Scots (SP) and lodgepole (LP) pine fibers

Brightness – A similar trend was observed for both species, but Scots pine had a higher ($P < 0.05$) value at every step of treatment. A larger decrease in brightness was seen after 5 min. of refining, *i.e.* 18% and 24% for Scots pine and lodgepole pine, respectively, but further changes were more uniform.

Time of mechanical treatment of fibres on their properties, the time of refining had a good Pearson *correlation* with all the properties tested, *i.e.* 0.58 °SR, 0.83 with the WRV, 0.85 with air permeability, -0.82 with roughness, 0.89 with burst index, 0.87 with tensile index, 0.88 with tensile energy absorption, 0.87 with breaking length, -0.71 with brightness, -0.85 with light scattering, and -0.9 with light absorption (significance < 0.05 in all cases).

Discussion and Conclusions

One of the most commonly used methods to monitor the changes in fibres during mechanical treatment is the measurement of the amount of water in a pulp suspension that could pass through a mesh screen (Gharehkhani et al. 2015), the so-called “freeness” or “beating degree,” which is presented as the °SR in this study according to the selected methodology. °SR numbers were found to be the same as that for the initial pulp without mechanical treatment.

However, the numbers gradually increased for both species after refining (Figure 1, 2). According to Gharekhani et al. (2015), the value of °SR was found to be the function of fibre fibrillation and fines formation; so the increase of °SR in our results proved the presence of these processes, suggesting that fibres of Scots pine were easier to process.

The larger number of WRV indicated a better water holding ability in the pulping process, when paper or another pulp product was made. Gharekhani (2015) reported of the dependence of the refining effect on the amount of lignin in the pulps, but in the case of Scots and lodgepole pine, there are no differences in the WRV measured (Sable et al. 2012a). Joutsimo and Giacomozzi (2015) also claim differences in the water holding ability caused by structural changes of fibres, like micropore closure or creation of new mesopores and macropores as a result of delamination, or a combination of both. The study indicated the different behaviour of pine fibres as a reaction to refining, and allowed using less energy for the refining process in the case of Scots pine to reach appropriate properties. A literature review by Gharekhani et al (2015) stated that the energy consumption and operating costs are aspects that are of interest in the study of refining and freeness.

However, when the optimum properties were achieved, the role of mechanical force on fibre fibrillation gradually decreased with a further increase in the refining time. Mechanical treatment cannot further provide the fibre fibrillation and only increases the energy consumption (Chen et al. 2012). This connection is readily apparent (Figure 2) in the results of our study; when 35 to 40 °SR was achieved, the WRV started to slightly decrease.

Refining of fibres caused internal and external fibrillation, thereby increasing the binding ability and the mechanical properties of the product of fibre network.

Our study discovered that the species responded to mechanical treatment differently. Results (Table 1) are in accordance with earlier comparative studies of pines, which showed lodgepole pine wood to be more suitable for high-quality paper production than that of Scots pine. Statistically significant differences were found in a number of fibre quality traits: fibre coarseness, Runkel index, and fibre width-length ratio, as well as slightly higher strength properties (Sable et al. 2012 a, b).

The tensile strength of a paper sample depends on the ability of the fibre network to absorb tensile energy. The predominance of the tensile strength of lodgepole pine fibres testified to their ability to build a homogenous and strong fibre network. The same tendency of the predominance of lodgepole pine was observed in the results of breaking length, which is the calculated limiting length of a strip of paper, beyond which it would break off by its own weight.

The improvement in mechanical properties is also based on the chemical changes of fibres through refining, e.g. more hydroxyl groups are fractionated out, which results in a

higher bonding ability of the fibre-fibre hydrogen bonds (Chen et al. 2012).

Figure 3 demonstrates how the air permeability of paper handsheets depends on the refining time of fibres. Air permeability is measured in Gurley second, a unit describing the number of seconds required for 100 cubic centimetres of air to pass through 6.45 square centimetres of a given material: the air permeability decreases with increasing value. As a result of fibrillation, the fibres formed a denser and less porous material, and air flow was disturbed through it. It should be noted that the higher or lower number of this parameter could not be defined as “good” or “poor” because of the wide range of the possible applications of the fibre product. The results of air permeability testify to the wide applicability of lodgepole pine fibres, and they form a strong and breathable product at the same time.

The roughness of the paper surface is an important factor for its printability. A smoother surface ensures a good print quality and less ink consumption. The difference in lodgepole pine can be caused by a dissimilar fibrillation process, where the formation of fine fibres results in tighter compatibility of the fibres.

The presence and amount of fine fibres also affect the optical properties because a community of individuals' forms defines the total optical behaviour of a material. Light scattering decreases with refining time because of the increase of contact area of fibres, and light absorption depends on the fibre colour (Pauler 2012). The mechanical treatment of pine fibres caused a decrease in the measured paper brightness (Figure 5), although it was not darker visually. The interpretation of results gives information about the fibre demand for chemicals in the bleaching process. Scots pine may be less demanding, but the need for bleaching increases with the total refining time.

Evaluating the present study, we concluded that there is effect of mechanical treatment (refining) for various amounts of time on Scots and lodgepole pine fibres, and suggests putting less energy into the refining process in the case of Scots pine to reach appropriate refining degree (Shopper-Riegler number) and water retention value. Pre-

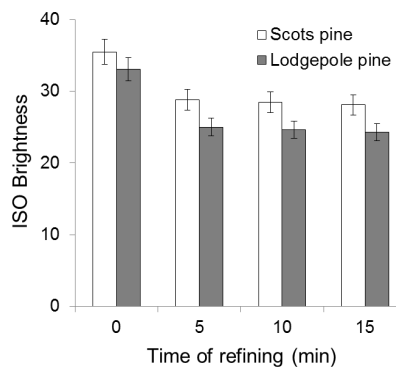


Figure 5. Effect of refining time on the ISO brightness of paper handsheets made of Scots and lodgepole pine fibers

dominance of lodgepole pine in the mechanical properties of handsheets was detected after refining. Fibres of lodgepole pine testified to their ability to build a homogeneous and strong fibre network. Air permeability of lodgepole pine was significantly higher after 15 min. of mechanical treatment. The roughness of the paper surface of lodgepole pine was lower ($p < 0.05$) at all stages of the refining process compared to Scots pine. The tendency of changes in optical properties was similar for both species, but differences in values were statistically significant ($p < 0.05$). Scots pine had higher results with respect to light scattering and ISO brightness. A strong ($r^2 = 0.8$ to 0.9), and significant ($p < 0.05$) correlation of the time of mechanical treatment and all investigated properties was detected.

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