

# The Effect of the Refining Intensity on the Progress of Internal Fibrillation and Shortening of Cellulose Fibers

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The utility properties of paper are dependent on the modification of the structure of the cellulose fibers, which is achieved *via* refining. The most important outcomes of the refining process are changes in the internal fibrillation and the shortening of the cellulose fibers. There are numerous opinions published in literature describing the relationship of these parameters and their impact on the final paper properties. These publications have been primarily based on the results of measurements conducted using insufficiently precise methods and simple speculations. The authors of this work decided to determine the effect of the refining intensity on the progress of internal fibrillation and shortening of cellulose fibers and the interrelation between these effects. Refining was performed with a laboratory Hollander beater, which was able to apply different refining loads. Utilizing additional control equipment, the specific edge load was also calculated. Finally, the impact of the refining effects (fibrillation and shortening) on the final properties of the paper were investigated.

*Keywords: Refining; Internal fibrillation; Shortening of fibers; Energy consumption; Mechanical resistance properties of paper*

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## INTRODUCTION

Refining is the traditional name for the mechanical treatment of fibrous paper pulps suspended in water. The conditions of this process, which result in modification of the properties of the pulps and have a strong impact on the final properties of the paper, are determined by the unit energy consumption (Lumiainen 2000; Laine *et al.* 2004; Bajpai 2005; El-Sharkawy *et al.* 2008; Kerekes 2010, 2014; Gharehkhani *et al.* 2015). Due to the dynamically growing production of paper products, which is currently above 400 million tons a year, both the optimization of utility paper properties and the simultaneous minimizing of the total unit energy consumption are very important from both a scientific and practical point of view (Garside 2019).

Paper properties related to its utilization are dependent on the properties of the fibrous paper pulps as well as on the changes in the structure of the fibers caused by refining (Higgins and De Young 1961; Mohlin 1975; Ebeling 1980; Przybysz 2003; Ferritsius *et al.* 2016). The most important of these changes are as follows: (1) the enhanced internal fibrillation of cellulose fibers, and (2) the shortening of the fibers (Giertz 1957; Stone *et al.* 1968; Fahey 1970; Page *et al.* 1985; Iwasaki and Naito 1996; Maloney 1998; Ferreira *et al.* 1999; Seth 2003; Kang and Paulapuro 2006; Htun and DeRuvo 2015; Mayr 2017; Olejnik *et al.* 2017). Therefore, changes in these two parameters are considered the

principal outcomes of the refining process. There is also external fibrillation, which is described in scientific literature as an additional refining effect. However, this effect is related to the generation of fines in the process (Ebeling 1980; Przybysz 2003; Bajpai 2005; El-Sharkawy *et al.* 2008). Due to the fact that the presented research was focused on the changes in fiber structure, the external fibrillation was not a point of interest.

The increase in internal fibrillation results from the cleavage of the hydrogen bonds between the structural elements of the cellulose fibers (the micro- and macro-fibrils) by water molecules (Hietanen and Ebeling 1990; Retulainen 1997; Wang *et al.* 2007; Kekäläinen *et al.* 2012; Lindqvist *et al.* 2012). This in turn enables the penetration of the interior of the refined cellulose fibers *via* water molecules that entered cavities less than 50 nm in size (Klemm *et al.* 1998; Andersson *et al.* 2003; Gupta and Chatterjee 2003; Lundin 2008; Botková *et al.* 2013). This results in a rise in swelling and increased deformability of the fibers (Page and De Grâce 1967; Kibblewhite 1977; Luukko *et al.* 1997; Joutsimo and Asikainen 2013; Heymer *et al.* 2018). The increased deformability of the refined fibers causes an increase in both the compactness of the structure and the contact surface area between the fibers in the newly manufactured paper web (Mohlin *et al.* 1996; Joutsimo *et al.* 2005; Seth 2005; Zeng *et al.* 2012; Vishtal and Retulainen 2014).

Numerous bonds, including hydrogen bonds (whose overall strength affects the static resistance properties of paper), are formed between the cellulose fibers during the process of paper web consolidation, during the drying phase (Laivins and Scallan 1994; Maloney *et al.* 1997; Norman 2009; Przybysz *et al.* 2016). Since the overall strength of the bonds between the cellulose fibers in paper is a function of the specific energies of the hydrogen bonds, van der Waals interactions, Coulomb interactions, mechanical mutual blocking, mutual diffusion, and the surface area of molecular contact between the fibers, an increased internal fibrillation improves the static resistance properties of paper (Ingmanson and Thode 1959; Scallan and Grignon 1979; Lindström *et al.* 2005; Hubbe 2006; Hirn *et al.* 2013; Persson *et al.* 2013; Hirn and Schennach 2015).

The studies aiming at the development of an objective and simple method for measuring the deformability caused by the swelling of refined fibers have been conducted for many years (Stone and Scallan 1967; Przybysz and Czechowski 1985; Berthold and Salmén 1997; Wang *et al.* 2003; Forsstrom *et al.* 2005; Hui *et al.* 2009). Currently, the increase in internal fibrillation of refined fibers is usually determined on the basis of the growth of swelling, which is characterized by the Water Retention Value (WRV) (Scallan and Carles 1972; Luukko *et al.* 1999; Maloney *et al.* 1999; Bäckström *et al.* 2008; Cheng *et al.* 2010). Some researchers prefer the FSP (Fiber Saturation Point), in which the results are not substantially different from the WRV, but the measurement of FSP is much more complicated than determining the WRV (Maloney *et al.* 1999).

The shortening of the cellulose fibers is the second important effect of refining on paper pulp (Steenberg *et al.* 1963; Corte and Agg 1980; Batchelor *et al.* 1997; Kerekes and Olson 2003; Olson *et al.* 2003). This phenomenon has a direct negative impact on the dynamic paper properties, which includes the tear resistance (Kane 1959; Hartman 1984). In practice, the shortening of the cellulose fibers is either caused by direct interactions between the refining elements and the fibers or *via* extremely high velocity gradients inside the fibers suspended in the refining zone (Beadle 1908; Page 1989; Kerekes and Senger 2006; Karlström and Eriksson 2014). The progress in this phenomenon is measured as a decrease in the average length of the refined fibers (Biermann 1996; Nordström and Hermansson 2017). To eliminate the influence of fine fragments of cellulose fibers on the results and error of measurements, the progress of shortening is typically determined basing

on parameters such as the average length-weighted length or the average weight-weighted length of the fibers (Batchelor *et al.* 1999; Bajpai 2017).

The shortening of the cellulose fibers during refining is inseparably associated with the other results of this process, *e.g.*, the growth of internal fibrillation, and it cannot be eliminated (Wahren 1983; Loijas 2010; Cuberos-Martinez and Park 2012). However, certain technological approaches enabling the enhancement of one refining effect with the simultaneous minimizing of another effect have been reported (Page 1985; Mohlin and Miller 1995; Seth 2006; Wang 2006; Koskenhely *et al.* 2007; Lundin *et al.* 2008; Heymer 2009; Harirforoush *et al.* 2018; Kerekes and Meltzer 2018).

According to literature dedicated to papermaking, a low intensity refining process promotes the growth of internal fibrillation and reduces the shortening of the cellulose fibers (Scallan and Carles 1972; Wahren 1983). This refining technology has been called the lubricant or grease refining process. According to the opinions of Stone *et al.* (1968) and Corte and Agg (1980), an intensive refining process causes advanced shortening of the cellulose fibers and limits their internal fibrillation. However, it has to be emphasized that these opinions are primarily based on speculations, which were not supported by results that measured both the internal fibrillation and the length the fibers were shortened or conducted using sufficiently objective methods.

The principal objective of this study was to determine the effect of the refining intensity on the profile of changes on the internal fibrillation and length of the cellulose fibers, as well as an estimation of the impact of these two parameters on the basic static and dynamic paper properties.

## EXPERIMENTAL

### Characteristics of the Cellulose Pulp

Industrial air-dried bleached pine kraft pulp in the form of sheets was used in the study. Analysis of the chemical composition of the pulp included quantification of the extractive, lignin, cellulose, hemicellulose, and ash content. The lignin content was defined *via* gravimetric methodology in compliance with the TAPPI T222 standard (2002) after the removal of the extractives, according to the TAPPI T204 standard (2007). The holocellulose content was determined according to TAPPI T249 (1985). Cellulose was quantified as alpha cellulose, according to TAPPI T203 (1999). The hemicellulose content was calculated by determining the difference between the holocellulose and cellulose contents. Ash content was determined *via* gravimetric methodology in compliance with TAPPI standard T211 (1993).

The average degree of polymerization of the cellulose content in the pulp was determined *via* viscometric methodology, which was compliant with ISO standard 5351 (2010).

The degree of crystallinity of the cellulose was determined using a multi-tasking X'Pert PRO MPD polycrystalline diffractometer (Malvern Panalytical Ltd, Malvern, United Kingdom) where CuK $\alpha$  radiation was applied. The measurements were carried out with a 10° to 40° range of 2 $\theta$  angles. A continuous scan was used (step 0.0167°) and the one-step measurement time was 50 sec. The WAXFIT program (version 4.0, The University of Bielsko-Biala, Bielsko-Biala, Poland) was used to calculations and reduce data.

The results of the chemical composition analysis, cellulose polymerization, and crystallinity degree of the raw pine materials are presented in Table 1 (as means and standard deviation of triplicate assays).

**Table 1.** Chemical Composition of the Industrial Bleached Pine Kraft Pulp

Parameter	Value
Cellulose (%)	93.6 (0.4)
Hemicelluloses (%)	5.8 (0.3)
Lignin (%)	Less than 0.1%
Extractives (%)	Less than 0.1%
Ash (%)	0.6 (0.1)
Degree of polymerization of cellulose	931 (18)
Degree of cellulose crystallinity	83.3 (n/a)

Note: Standard deviations are shown in brackets.

Before the refining process, the solid substance content was determined (5 measurements), according to ISO standard 638 (2008).

Parameters of the pulp performed after slushing and before refining were as follows: (1) the Schopper-Riegler freeness was measured using a Schopper-Riegler apparatus (Danex, Katowice, Poland), according to PN-EN ISO standard 5267-1 (2002); (2) the water retention value (WRV), the ratio of water to weight of bone dry sample, was measured according to ISO 23714 (2014); and (3) the fiber dimensions and fines content were measured according to ISO standard 16065-2 (2014) using a Morfi Compact Black Edition apparatus (Techpap, Grenoble, France). The results of these measurements are shown in in Table 2.

**Table 2.** Pulp Parameters before the Refining Process

Parameter	Value
Solid substance content (%)	96.0 (0.1)
Freeness (°SR)	12 (1)
Water retention value (%)	85.6 (0.5)
Mean length-weighted length (µm)	1956 (6)
Fibre width (µm)	28.3 (0.2)
Coarseness (mg/m)	0.1487 (0.0021)
Kinked fibre content (%)	46.87 (0.52)
Mean fibre curl index (%)	11.79 (0.21)
Macro-fibrillation index (%)	0.289 (0.011)
Broken fibre content (%)	34.64 (0.38)
Fine content (% in area)	2.241 (0.028)
Fine content (% in length)	13.76 (0.32)

Note: Standard deviations are shown in brackets.

### Preparing of Pulp Samples

The industrial cellulose pulp had to be prepared under the appropriate conditions before the refining experiments. Pulp samples (360 g bone dry) were soaked in demineralized water for 24 h before refining. Then the samples were torn into approximately 2 cm x 2 cm pieces, which were subjected to slushing using a laboratory Hollander (Valley) beater, according to TAPPI standard T200 (2001). The Valley beater was additionally equipped with an ND10 sensor (Lumel, Zielona Góra, Poland), which was

used as a wattmeter to measure the motor power and no-load power and a SM2 sensor (Lumel, Zielona Góra, Poland), which was used to measure the rotational speed of the Valley beater. All the sensors were connected to a DT312 panel (DT Research, New Taipei City, Taiwan). Data acquisition was performed using Lumel Process software (version 1.1, Lumel, Zielona Góra, Poland).

### Refinement of the Paper Pulp

Refinement of the paper pulp was conducted using a laboratory Hollander (Valley) beater, according to TAPPI standard T200 (2001), at a pulp concentration of 1.57%. A Valley beater was chosen for this experiment because its load was easily modified and there were 7 standard weights used to exert load.

The intensity of the refining process was changed *via* modification of the load of the Valley beater, by means of changing the pressure of its knife edge on the rotor using special weights. The specific edge load (SEL) was calculated according to (Eq. 1) presented by Gharekhani *et al.* (2015),

$$SEL = \frac{P_{net}}{CEL}, \quad P_{net} = P_{tot} - P_{noload} \quad (1)$$

where  $P_{tot}$  is the total power consumed,  $P_{net}$  is the net power (kW) consumed to change the pulp properties.  $P_{noload}$  is the initial power or no-load power, which is defined as the power needed to rotate the rotor in the refiner.

The length of the cutting edge (CEL) of the tested Valley beater was 34.14 m/rev (BEL) multiplied by number of revolutions per second measured by an SM2 sensor. The parameters of the seven independent series of measurements (weight and load) were presented in Table 3.

**Table 3.** Parameters of Refining Process

Weight (g)	Load (N)	SEL (J/m)
1650	31.5	0.984
2750	52.5	1.254
4400	83.9	2.284
5500	104.9	2.998
7150	136.4	3.884
8250	157.4	4.395
9900	188.8	5.412

### Characterization of the Fibrous Pulp

Each refined paper pulp sample, which was obtained under the conditions presented above, was characterized in terms of the freeness, water retention value (WRV), and the average length-weighted fiber length. The freeness was characterized according to ISO standard 5267-1 (2002), with 5 independent measurements, 3 of which gave the same result. The WRV was characterized according to ISO standard 23714 (2014), with the arithmetic mean calculated from 4 measurements (the relative error was less than 0.8%). The average length-weighted fiber length was characterized according to ISO standard 16065-2 (2014), with the arithmetic mean calculated from 3 measurements (the relative error was less than 1%). The results of these measurements are shown in Figs. 1, 3, 4, and 5.

## Characterization of the Paper

The characterization of the paper was conducted using laboratory paper handsheets that were produced from the refined pulp samples, using a standard laboratory apparatus for paper handsheet formation (RKFD-03, Danex, Katowice, Poland), according to PN-EN ISO standard 5269-2 (2007). The grammage of each paper sheet was  $80 \pm 2 \text{ g/m}^2$  according to ISO standard 536 (2012). Before the characterization, each paper sheet was acclimated at  $23 \text{ }^\circ\text{C}$  and 50% relative air humidity, according to ISO standard 187 (1990).

Each paper sheet was characterized in terms of: (1) breaking length, in which the arithmetic mean of at least 12 measurements (the relative error was less than 2.7%) were measured with a Zwick Roell Z005 TN ProLine tensile testing machine (Zwick Roell, Ulm, Germany), according to PN-EN ISO standard 1924-2 (2010); and (2) tear resistance, in which the arithmetic mean of at least 12 measurements (the relative error was less than 4.5%) were measured with a Lorentzen and Wettre Tear Tester (Kista, Sweden), according to ISO standard 1974 (2012).

## RESULTS AND DISCUSSION

In papermaking, the freeness of the paper fibrous pulps is a commonly applied parameter, which allows for the characterization of the paper (Hallan and Barkas 1953). This parameter was based on a conventional determination of the dewatering properties of the paper pulp, but its values are not expressed in terms of SI units ( $^\circ\text{SR}$ ) (Hallan and Barkas 1953). Because this parameter is very popular, its dependence on the refining intensity was determined in this study (Fig. 1).

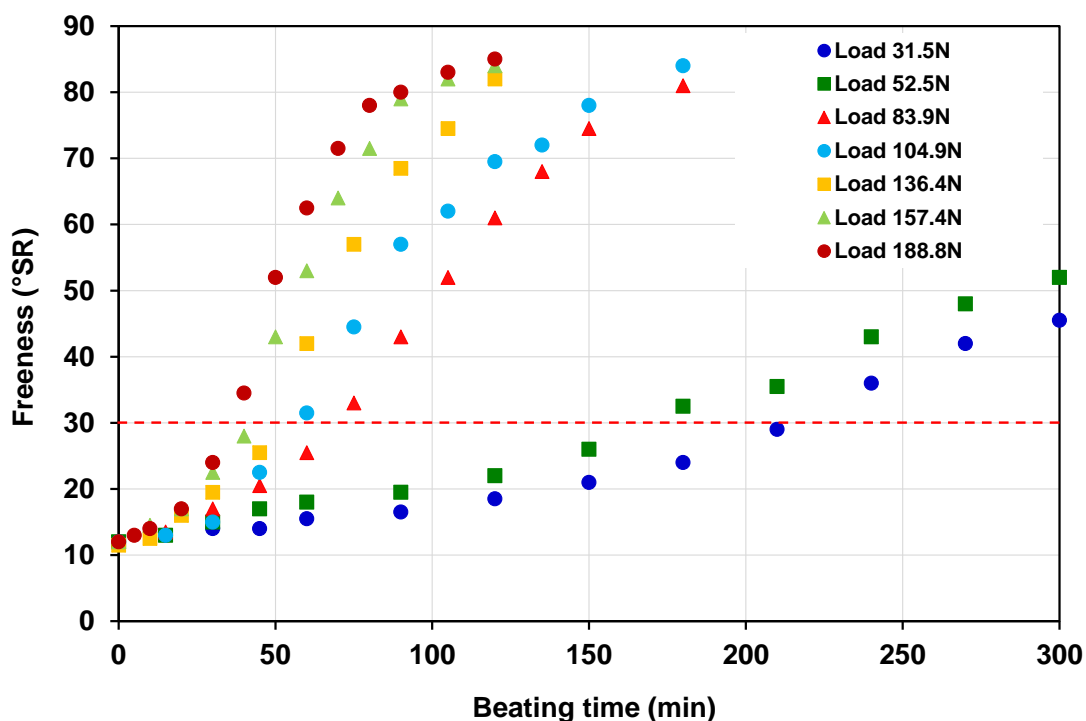
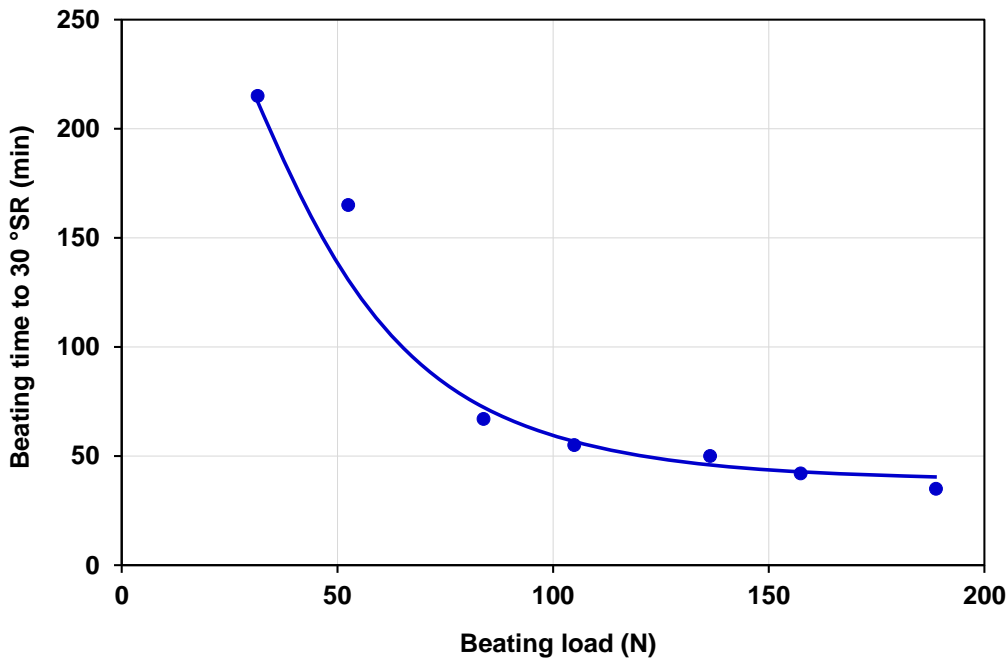


Fig. 1. The effect of the load of the laboratory Valley beater on the changes in the freeness of the cellulose pulp over the beating time.

The data presented in Fig. 1 provide evidence that an increase in the refining intensity accelerated the growth in the degrees Schopper-Riegler ( $^{\circ}\text{SR}$ ) of the cellulose pulp. It is generally accepted that paper produced from pulp with its majority characterized with a freeness corresponding to approximately  $30^{\circ}\text{SR}$  has the optimal utility properties, so this value was the point of reference in this study. A value of  $30^{\circ}\text{SR}$  was achieved at greater than 200 min when the refining intensity was the lowest (1650 g) and at approximately 40 min (approximately 5-fold shorter time) when the intensity was the highest (9900 g). Interestingly, the increase in  $^{\circ}\text{SR}$  was not directly proportional to the load of the Valley beater (Fig. 2).



**Fig. 2.** The effect of the load of the Valley beater on the beating time required to achieve a value of  $30^{\circ}\text{SR}$ .

The data presented in Fig. 2 demonstrated that as the load was increased to approximately 4400 g (83.9 N), the beating time to achieve a value of  $30^{\circ}\text{SR}$  decreased at a moderate rate. When the load was above this value (4400 g) the process stabilized and the beating time to achieve a value of  $30^{\circ}\text{SR}$  decreased at a slower rate. This in turn demonstrated that dependent on the refining intensity, the refining process could be divided into two ranges, which have different mechanisms.

The range of low refining intensities (for this study this included loads up to approximately 4400 g or 83.9 N) may be termed the non-effective range because of the long beating times, which indicates only a fraction of energy is used for changing the fibers. When the load was increased, the energy efficiency rapidly increased and the process achieved the second, efficient phase. Therefore, it may be concluded that the low refining intensity was insufficient to cause the appropriate changes in the structure of the fibers subjected to refining.

At sufficiently high loads, the refining process was efficient, and an increased refining intensity caused more advanced and permanent changes in the structure of the fibers. This in turn provided evidence of the increased energy efficiency of the process.

To verify these conclusions, the effect of the refining intensity on the changes to the two basic parameters, *i.e.*, internal fibrillation and the shortening of fibers, was determined. The dependence of the WRV and the average length-weighted fiber length on the load of the Valley beater are presented in the Figs. 3 and 4.

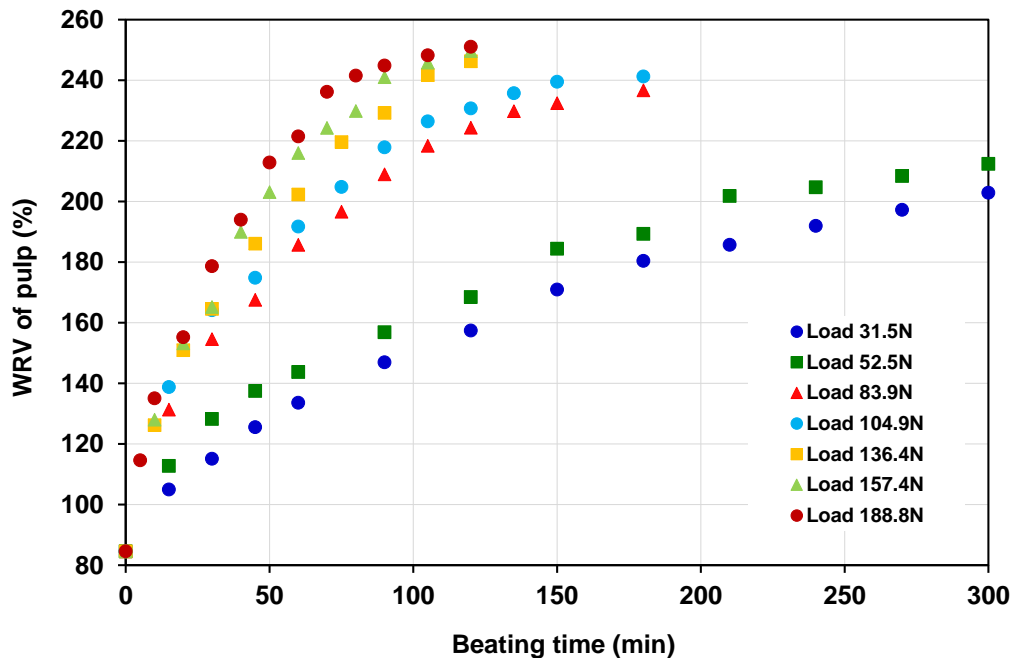


Fig. 3. The effect of the load of the laboratory Valley beater on the profile of changes in the WRV of the cellulose pulp over the beating time

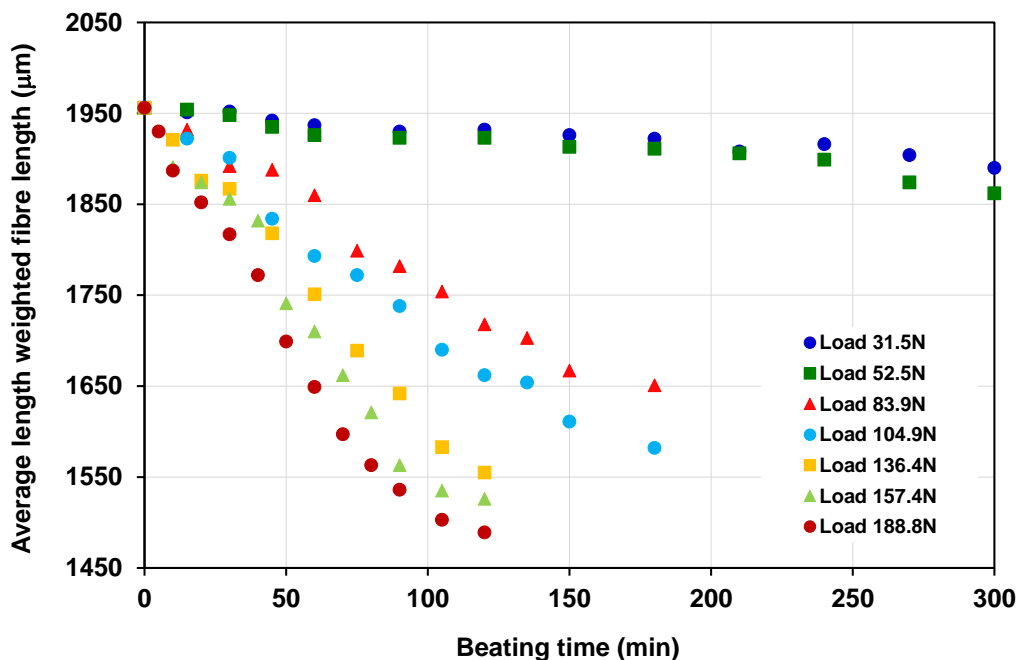


Fig. 4. The effect of the load of the laboratory Valley beater on the profile of changes in the average weighted fiber length of the cellulose pulp over the beating time

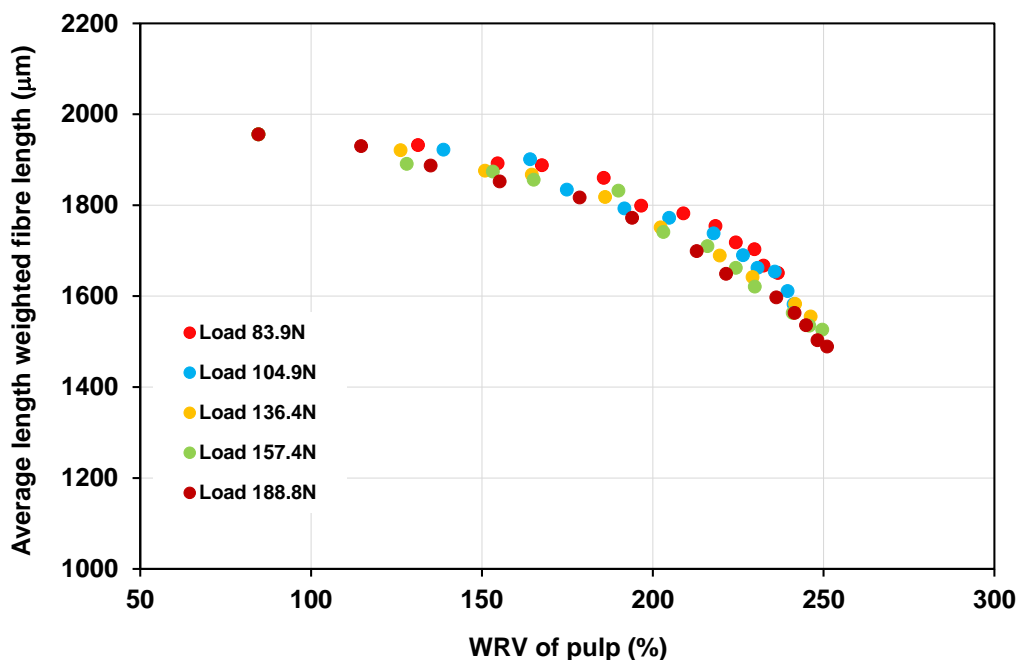


The data presented in Figs. 3 and 4 demonstrated that an increased load for the Valley beater accelerated the increase of the WRV and accelerated the decrease of the average length-weighted fiber length.

The results of these measurements confirmed the earlier conclusions, which stated that the refining intensities that corresponded to loads less than 4400 g (83.9 N) were not able to cause sufficient changes in the structure of the cellulose fibers, in terms of both the internal fibrillation and the fiber length. Furthermore, these results confirmed that the conclusion related to the two refining intensity ranges was correct. This meant that the process was not efficient at a low intensity and was efficient at a sufficiently high (greater than 4400 g loading) refining intensity.

In addition, the relationship between the internal fibrillation and the fiber length was very important. Typically, the most desired outcomes of the refining process are an increase in the internal fibrillation, an increase in the WRV, and a limited decrease in fiber length, which was measured as a decrease in the average weighted fiber length (Godlewska and Rawicka 2003; Olejnik *et al.* 2016). This enabled the paper to obtain high static resistance properties (which included the breaking length) and to minimize the natural decrease of the dynamic resistance properties (which included the tear resistance).

The relationship between the average length weighted fiber length and the WRV, for the loads applied in this study, was plotted to select the efficient range of the refining intensity (Fig. 5).

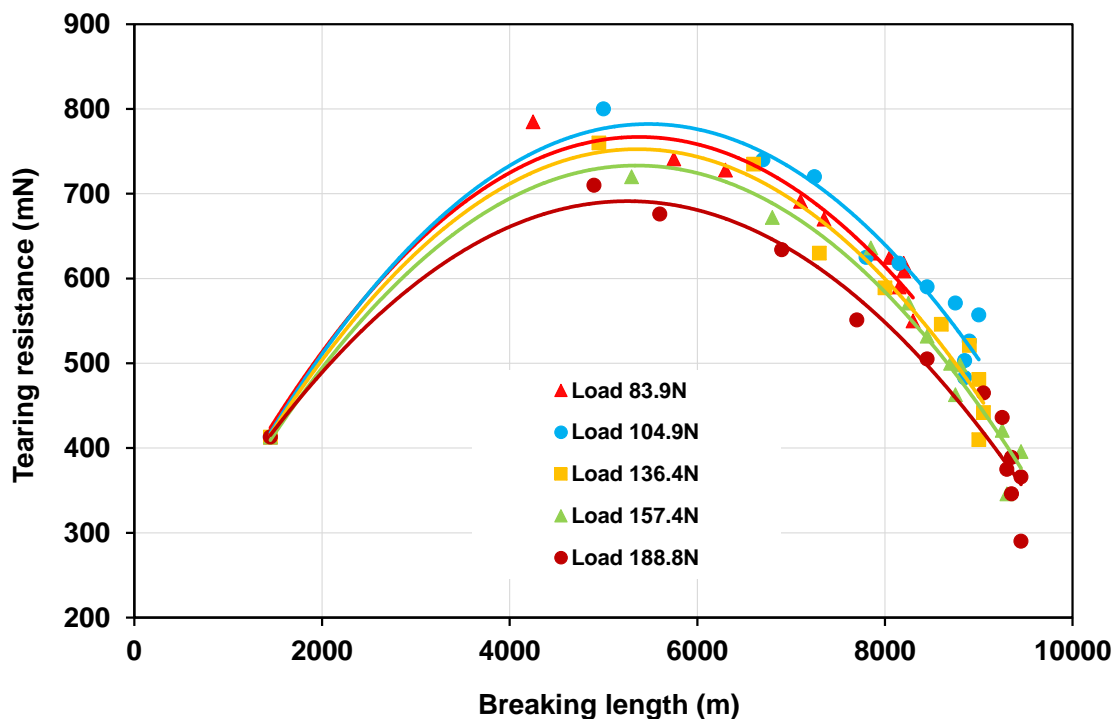


**Fig. 5.** The impact of the load of the laboratory Valley beater on the relationship between the WRV and the average weighted fiber length

The data shown in Fig. 5 provides evidence that for the cellulose pulp used in the study, the refining intensity had a relatively weak impact on the interplay between the progress of internal fibrillation and the progress of fiber shortening. This was due to the fact that for approximately the entire WRV range (80 to 200%), the deviation of the average fiber length from the general trend line was not greater than 100 µm (0.1 mm).

However, the analysis of this relationship demonstrated that for a WRV of up to approximately 200%, the rate of the decrease in the average length weighted fiber length was relatively low. In this range, the growth in internal fibrillation was faster than the shortening of the cellulose fibers. When the WRV were greater than 200%, the shortening of the fiber length was faster than the growth of the WRV. Noteworthy, the cellulose pulp used in this study achieved a critical WRV at a freeness of approximately 30 °SR. According to the empirical data obtained under industrial conditions, this freeness value is optimal for manufacturing of the majority of paper products.

The existence of the interplay between the basic parameters of cellulose pulp, irrespective of the refining intensity, suggested that the properties of the paper produced from this pulp would be similar. To verify this assumption, the authors plotted the relationship between the tear resistance and the breaking length for the applied values of the load (Fig. 6).



**Fig. 6.** The effect of the load of the laboratory Valley beater during the refining process of cellulose pulp on the relationship between the values of self-tearing and the tear resistance of the paper produced from the pulp

The data presented in Fig. 6 demonstrates that paper sheets produced from the pulp refined at different intensities were characterized by the similar interplay between the tear resistance values and the breaking length values. This was due to the fact that for the whole range of changes in the breaking length values, the deviation of the values of the tear resistance from the general trend line was not greater than 100 mN. Furthermore, the error of the tear resistance measurements of the paper sheets was approximately 20 mN.

## CONCLUSIONS

1. Two refining intensity ranges were observed in this study. With a load less than 3000 g, the tested softwood cellulose fibers did not undergo modification (very weak swelling and limited shortening). With a load greater than 2750 g (52.5 N), the increase in the load caused enhanced swelling as well as the simultaneous shortening of the fiber length.
2. Irrespective of the refining intensity, there was a close interplay between the progress of the internal fibrillation and the shortening of the fibers for the tested pulp. There was no refining load weight that made it possible to limit the fiber shortening while enhancing only the fibrillation.
3. At water retention value (WRV) greater than 200% (a freeness corresponding to degrees of Schopper-Riegler of approximately 30°SR), an increase in the WRV of the cellulose pulp was associated with an enhanced decrease in the average length weighted fiber length.
4. To reduce the total energy consumption, the refining process for cellulose pulp using a Valley beater should be conducted with a load greater than 2750 g (approximately 52.5 N). This was the minimal load that ensured the effective internal fibrillation of the tested cellulose fibers.

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