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Review

Basic effects of pulp refining on fiber properties—A review

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ABSTRACT

The requirement for high quality pulps which are widely used in paper industries has increased the demand for pulp refining (beating) process. Pulp refining is a promising approach to improve the pulp quality by changing the fiber characteristics. The diversity of research on the effect of refining on fiber properties which is due to the different pulp sources, pulp consistency and refining equipment has interested us to provide a review on the studies over the last decade. In this article, the influence of pulp refining on structural properties i.e., fibrillations, fine formation, fiber length, fiber curl, crystallinity and distribution of surface chemical compositions is reviewed. The effect of pulp refining on electrokinetic properties of fiber e.g., surface and total charges of pulps is discussed. In addition, an overview of different refining theories, refiners as well as some tests for assessing the pulp refining is presented.

Contents

Abbreviations: C, C-factor; CSF, Canadian Standard Freeness; CTMP, chemithermomechanical pulp; FSP, fiber saturation point; FTIR, Fourier transform IR spectroscopy; HC, high consistency; LC, low consistency; MFA, microfibrillar angle; ML, middle lamella; NMR, nuclear magnetic resonance; P, primary wall; SEM, scanning electron microscopy; SR, Schopper Riegler; TMP, thermomechanical pulp; ToF-SIMS, time-of-flight secondary ion mass spectrometry; XPS, X-ray photoelectron spectroscopy; XRD, X-ray diffraction.

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Nomenclatures

Symbols

BEL	bar edge length (km)
CEL	cutting edge length (km/rev)
C_f	pulp consistency (%)
D	depth of grooves (m)
G	width of grooves (m)
IL	impact length of the bars (m)
l_A	length of fiber (m)
n_b	number of rotor and stator bars on circle $2\pi r$ in refiner
P_{net}	net power (kW)
P_{noload}	no-load power (kW)
P_{tot}	total power (kW)
$R1$	inner radius of refining zone (m)
$R2$	outer radius of refining zone (m)
SEL	specific edge load (W s/m)
SRE	specific refining energy (KW h/t)
SSL	specific surface load (W s/m^2)
w	coarseness of fiber (mg/m)
W_r	width of rotor (m)
W_{st}	width of stator (m)
α	average intersecting angle (deg)
ρ	density (kg/m^3)
ϕ	bar angle (deg)
ω	rotational speed (rpm)

1. Introduction

Modification of the pulp or fiber quality to improve the paper features is one of the most significant scientific challenges in the paper industries. Pulp consists of cellulose fibers which come from wood and non-wood plants, and it is the major raw material in papermaking. The main sources for wood pulps are softwood (e.g., spruce, pine) and hardwood (e.g., eucalyptus, aspen) trees, and for non-wood are crops and agriculture residues (e.g., cotton, kenaf). Chemical compositions and structural properties of wood (hardwood and softwood) and non-wood species along with some remarks are presented in Table 1.

The cell wall structure (Fig. 1) of different species is generally composed of cellulose, hemicelluloses and lignin. Cellulose is a polysaccharide consisting of glucose units (Pokhrel, 2010). The cellulose molecule with several chains organized into elementary fibrils, which are the narrowest fibrils (diameter 3.5 nm). Each elementary fibril can consist of as high as 40 cellulose chains. The aggregation of elementary fibrils forms the microfibrils having diameters between 10 and 35 nm (Chinga-Carrasco, 2011; Sixta, 2008). Finally, the macrofibrils are other units which are shaped by the microfibrils aggregations (Fig. 1b) (Abe & Yano, 2009; Donaldson, 2007). Macrofibrils are twisted around the cell wall axis and introduce the term microfibrillar angle (MFA), (Fig. 1a) (Barnett & Bonham, 2004; Pulkkinen, 2010; Wathen, 2006). A smaller fibril angle is beneficial for paper strength (Blomstedt, 2007; Courchene, Peter, & Litvay, 2006). Cellulose has crystalline structure while hemicellulose has amorphous structure (definition of crystallinity is discussed in Section 4.6). Hemicellulose surrounds cellulose microfibrils. Hemicellulose has a lower strength than cellulose and can be easily hydrolyzed. It is a polymer of neutral polysaccharides present in the plant cell wall matrix and can be divided into xylans, mannans, β-glucans with mixed linkages, and xyloglucans. It can be seen in Table 1, for the cases of the hardwood and softwood, the amount of hemicelluloses are more or less same. However, there are significant differences between the types of hemicelluloses in the hardwood and softwood species. Detailed information about the above mentioned polysaccharides and amount of them in different species can be found in literatures (Ebringerova, Hromadkova, & Heinze, 2005; Sixta, 2008). The third component of the cell wall is lignin. Lignin acts as glue and binds the different layers of cell wall (Sutton, Joss, & Crossely, 2000). Lignin is a hydrophobic substance and can be removed by chemical pulping and bleaching. Low amount of lignin in the raw material makes it a good candidate for paper making. All of these components are present in the different layers of cell wall. The cell wall can be divided into different layers: middle lamella (ML), primary cell wall, secondary cell wall and lumen (Fig. 1a; Sjöström, 1993). ML acts as a cementing substance between the cells and has highest concentration of lignin. (Shafiei Sabet, 2013). The middle ML surrounds the primary wall (P). P layer is thin and flexible. It consists mainly of hemicelluloses and lignin and a loose aggregation of microfibrils which are oriented randomly in this layer. Cellulose chains are twisted along the axis of glucan chains and are held by hydrogen bonds between the chains (Sixta, 2008; Thomas et al., 2013). Secondary wall is located between the P and lumen. It sometimes consists of three distinct layers: S1, S2 and S3 (Bergander & Salmén, 2002; Meier, 1962). Most of fiber mass belongs to the S2 layer, the MFA in the S2 is 10–30° while the S1 layer has a high microfibril angle (50–70°). The S3 layer is thin with fairly horizontal microfibrils (MFA is 70–90°) (Blomstedt, 2007). The last layer in the cell wall is lumen (w) which is the hollow core and can hold the moisture (water or water vapor). Fibres with large lumen tend to flatten to ribons during pulping which result in good strength properties.

To produce the pulp from the raw materials, different pulping processes can be performed on the chips or small parts which have been produced by the chipping of timber or other parts of plant. Depending on the pulping processes the wood pulps are categorized as mechanical pulp, chemical pulp (e.g., kraft, sulfite pulps) and chemithermomechanical pulp (CTMP). Mechanical pulps are produced from raw material by the application of mechanical energy. The mechanical pulps have good print quality. Thermomechanical pulps (TMP) are one of the popular types of mechanical pulps which are produced by processing the wood chips using the high temperature steam and mechanical refining. Chemical pulps are almost pure celluloses which are produced by heat and chemical treatment. During the treatment of raw material with chemicals, a large amount of lignin is extracted from the material. The chemical pulps can be classified to kraft and sulfite pulps. Kraft pulp or sulfate pulp, is obtained by the treatment of the chips with a mixture of sodium hydroxide and sodium sulfide and sulfite pulp is formed during the pulping process by using the various salts of sulfurous acid. CTMPs are produced by the combination of chemical and mechanical treatments. They need less mechanical energy,

Table 1
Chemical composition and structural properties of typical wood and non-wood species.

Properties	Wood		Non-wood			Remark
	Softwood	Hardwood	Wheat straw	Corn stalks	Bagasse	
Chemical composition						
Cellulose	40–45[a]	43–47[a]	30[a]	35[a]	40[a]	Fiber length is approximately 3 mm for softwood and 1 mm for hardwood. Most of the softwoods are softer than hardwoods. Softwood fibers are more flexible than hardwood fibers. In the case of non-wood, the average ratio of fiber length ranges from 1 mm to 30 mm. It depends on plant species and the plant part from which the fiber is derived. Non-woods have lower lignin content (compared to wood), so can be pulped in less time compared to woods.
Hemicellulose	25–29[a]	25–35[a]	50[a]	25[a]	30[a]	
Lignin	25–31[a]	16–24[a]	15[a]	35[a]	20[a]	
Extract	1–5[a]	2–8[a]	5[a]	5[a]	10[a]	
Structural properties						
Slenderness ratio (fiber length/fiber diameter)	95–12[b]	55–75[b]	86.76[c]	54.32[d]	70[e]	
Flexibility ratio (fiber lumen diameter/fiber diameter)×100	75[b]	55–70[b]	71.76[c]	44.03[d]	29[e]	

Data have been presented by [a] (Sixta, 2008), [b] (Smook, 1992), [c] (Singh, Dutt, & Tyagi, 2010), [d] (Usta, Kirci, & Eroglu, 1990) and [e] (Agnihotri, Dutt, & Tyagi, 2010).

and the chemical treatment is performed with lower temperature and shorter time. The CTMPs have good strength. Among different types of pulps, the chemical pulps have the highest share in pulp productions. For instance, in Europe, kraft pulps account for 65% of total pulp production (Abdul Khalil, Bhat, & Ireana Yusra, 2012).

A variety in pulp sources have made a demand for having a fundamental process for improvement of the fiber quality that can be applied on all types of pulps. One of the processes that is conducted in the stock preparation is so-called "pulp refining" or "beating". Pulp refining or beating could be described as a mechanical treatment of the pulp by using the special equipment (refiner). Nowadays the term "refining" is more common than "beating" in the studies because of the appearance of different refiners which are widely used in the pulp industries. However both phrases have same meaning. The term "refining" is adopted in this study to keep the consistency in the text.

In the refining process, the fibers are under compression and shear forces which are causing several changes in specifications of fibers. Dependent on the initial fiber properties, pulp consistency (weight in grams of oven-dry fiber in 100 g of pulp–water mixture) and refiner specification, the changes in fiber result in higher bonding (Mohlin, Miller, Mohlin, & Miller, 1995).

This review focuses on the effects of pulp refining on the structural and electrokinetic properties of the fibers which are critical in

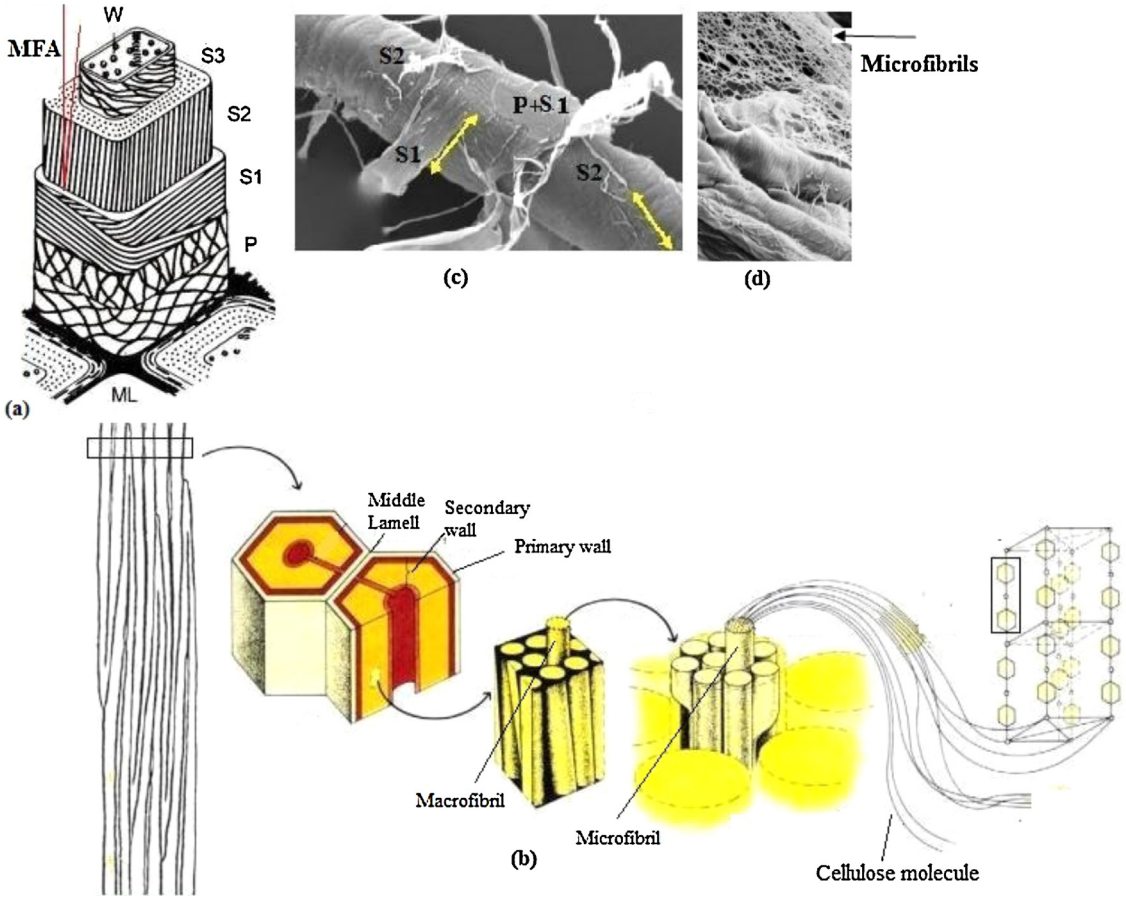


Fig. 1. Fibre unit structure: (a) schematic of cell wall layers (middle lamella (ML), primary wall (P), secondry wal (S1, S2, S3) and lumen (W); (b) fibrillar structure of cell wall; (c) scanning microscopic image of cell wall layers; and (d) scanning microscopic image of microfibrils (Chinga-Carrasco, 2011; Fardim, Liebert & Heinze, 2013; Fellers & Norman, 1998; Page, 1989a,b; Sixta, 2008).

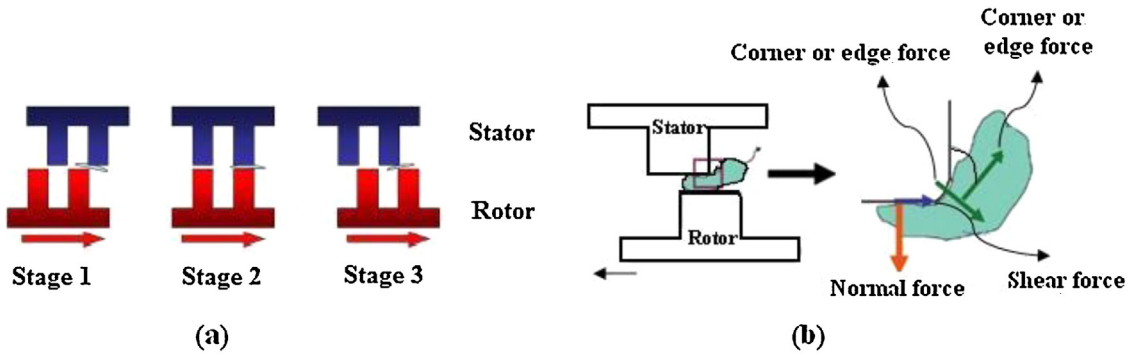


Fig. 2. (a) Refining mechanism (Nugroho, 2012). (b) Forces acting during refining (Cuberos-Martinez & Park, 2012).

the paper industries. In addition, a summary of different theories, refiners (laboratory and industrial refiners) and also measurement methods suggested for monitoring the refining are presented. The effects of refining on paper properties are out of scope of the present study, but it has been referred where it is related to the fiber properties.

2. Theories of refining

This section is presented to provide a common basis of the refining theories. The goal of refining theory is to predict changes in pulp properties from known refining conditions. Prior to discuss about theories, mechanism of refining is explained in this section.

In the refiners, fibers are treated between two parallel grooved plates; stator and rotor. The three dominant refining stages are shown in Fig. 2(a). The first one is pick-up stage fibers are accumulated and trapped between the edges of bars. In the next step, the trapped fibers are compressed by the surfaces of the moving and stationary bars. In the final stage, the fibers are affected by the shear forces. In this stage the fibers also hit the bars on the surface to edge and again edge to edge. During the bar crossing, two different forces act on the fibers, one of them due to the contact fibers to bars and another one due to the contact fiber to fiber (Lumiainen, 2000; Nugroho, 2012; Technology, 2001).

2.1. Specific edge load (SEL) theory

The SEL theory is a well known and a most reliable theory in papermaking industries (Genco, 1999; Karlström, Eriksson, Sikter, & Gustavsson, 2008; Kerekes, 2011; Mohlin, 2002; Olejnik, 2013). The SEL is the amount of effective energy spent per unit edge length of bar crossing and rigorously is presented as "machine intensity", meaning it represents the energy expenditure at bar crossings without reference about the pattern of energy distribution (Kerekes & Senger, 2006; Kerekes, 2011). SEL can be obtained from Eq. (1): (W s/m or J/m)

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

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. It is noticeable that this power has no contribution in pulp refining, and it is only consumed to overcome the losses, including loss due to shaft and bearings friction (mechanical losses), required energy to rotate the refiner plate in pulp suspension close to the stationary plate (hydraulic) and required energy consumed by refiner when pumping pulp suspension from inlet to outlet of refiner (pumping). Different studies have been reported to explore the no-load power in LC refining both experimentally and computationally (Dietemann & Roux, 2005; Rajabi Nasab, 2013).

As can be seen in Eq. (1) another parameter contributed in the SEL is cutting edge length (CEL) which is calculated by Eq. (2).

$$\mathrm{CEL} = \mathrm{BEL} \times \omega \tag{2}$$

where BEL is bar edge length and ω is rotational speed. Plate designs are typically characterized by BEL and BEL/CEL is given to paper makers by suppliers of refiner plates.

Typical SEL for softwood and hardwood is 1.5–3 J/m and 0.2–1.0 J/m respectively (Nugroho, 2012). The SEL theory works quite well with coarse fillings when bars are wider than the length of the fiber flocs (Lumiainen, 2000).

One parameter that contributes in the study of different theories is specific refining energy (SRE). SRE is used for calculating how much energy is given from the refiner to the fibers, and can be obtained by dividing the net power by the fiber mass flow rate. Typical SRE for chemical pulps is between 80 and 250 Kw h/t (Heymer, 2009; Loijas, 2010). The higher the SRE, the more refining is happening to the fibers. Nugroho (2012) found that at constant refiner speed, flow rate, and pulp consistency, the specific refining energy and refining intensity are influenced by net power only, so it makes a linear correlation between specific refining energy and refining intensity. In this case, the specific refining energy is proportional to refining intensity.

2.2. Specific surface load (SSL) theory

However SEL is widely used theory in industries, it is quite simple. As an example, in this theory, the width of bars has not been considered. To eliminate this lacking, Lumiainen (1990) proposed the SSL theory which consider the impact length of the bars (IL) in the SEL theory (Loijas, 2010). SSL (W s/m^2 or J/m^2) is calculated by Eq. (3).

$$\mathrm{SSL} = \frac{\mathrm{SEL}}{\mathrm{IL}} \tag{3}$$

IL can be calculated from the width of rotor (W_{r}) and stator bars (W_{st}), and from the average intersecting angle α of the rotor and stator bars.

$$\mathrm{IL} = \frac{W_{\mathrm{r}} + W_{\mathrm{st}}}{2} \times \frac{1}{\cos(\alpha/2)} \tag{4}$$

The SSL theory seems to work quite well when bars are so narrow. If bars are much narrower than the fiber floc, then they heavily cut the fibers (Lumiainen, 2000).

2.3. C-factor theory

Several scientists have described the SRE into two parameters which are the number of impacts (N), and the intensity of each

impact (I), on pulp (Kerekes, 1990; Lewis & Danforth, 1962; Stevens, 1981). To provide a relationship among the net power and fiber mass flow rate with N and I, Kerekes (1990) has proposed the C-factor theory (Eq. (5)).

$$\mathrm{SRE} = \frac{P_{\mathrm{net}}}{\dot{m}} = N.I = \frac{C}{\dot{m}}\frac{P_{\mathrm{net}}}{C} \tag{5}$$

where C is described as the capacity of a refiner to inflict impacts on fibers. C has been given for different refiners (disk, conical and PFI mil) as a function of fiber length, coarseness (mass per unit length), plate geometry, rotation speed and pulp consistency (Heymer, 2009; Kerekes, 1990; Kerekes, Soszynski, & Doo, 2005). For example C for a disk refiner is calculated by Eq. (6):

$$C = \frac{8\pi^2 GD\rho C_{\mathrm{F}} l_{\mathrm{A}} n_{\mathrm{b}}^3 \omega(1+2\,\tan\,\phi)(R_2^3 - R_1^3)}{3w(l_{\mathrm{A}} + D)} \tag{6}$$

variables has been defined in the nomenclature.

Although, C is a more precise theory than SLE and SSL, it is not yet a widely used theory in the papermaking. One important factor that makes some difficulties in use of C especially in the industries, is the measurement of the initial pulp dimensions (length and coarseness). Another factor, refers to the basic effects of refining, as during refining, the length of the fiber is subjected to the change, so the C is not constant (Batchelor, Kjell-Arve, & Ouellet, 1999; Olejnik, 2013).

2.4. New concept in refining theories:

By reviewing the above mentioned theories, (SEL, SSl and C) it is found that all of them are energy-based intensities and energy is the commonly used variable in the paper mills. However, it is stated that force and not energy is responsible to change the fiber properties during refining. In the new concept, the attempts are focused to use force in characterizing refining action (Kerekes, 2010; Kerekes & Senger, 2006). To understand such theories, detailed knowledge of the applied forces, the fiber distribution in refiner gaps, and the amount of fibers, is needed (Heymer, 2009). In the pulp refining, the typical gap is 0.1–0.2 mm which is as big as the thickness of several single fibers. Therefore fibers are trapped between the stator and rotor bars as the flocs and the forces act on the flocs rather than single fibers (Andersson, 2011; Batchelor, Lundin, & Fardim, 2006). The forces which are transmitted to the flocs are: normal force, shear force and corner or edge force (Fig. 2(b)). The normal force is due to the compression of the floc between bars, shear force is produced by the movement of bar surface on the floc and corner force appears at the bar edge (Batchelor, Martinez, Kerekes, & Ouellet, 1997; Martinez & Kerekes, 1994; Roux, Bloch, & Nortier, 2007). To estimate the amount of flocs trapped between bars, Batchelor et al. (2006) conducted some experiments. He proposed that the number of fibers trapped under any segment of bar will determine the point at which the net refining power begins to rise and the net refining power is fitted with a negative exponential function.

Kerekes (2011) proposed a force based theory that the refining intensity is based on forces on bars which are linked to the forces on fibers through fiber distribution over bars and gap size. It should be noted that in Kerekes theory, the forces are predicted on the individual fibers. To develop such concept, more studies are needed.

3. Summary of refiners and control tests used in the stock preparation

In order to aid the future discussions, the present section provides an overview of different types of refiners, and tests applied on pulps for the stock preparation.

3.1. Different pulp refining machines

Refiners can be categorized into laboratory and industrial refines. Table 2 provides a summary of refiners along with the quantitative information about the different refiners which have been used in published research works since 2000, based upon the search in the Scopus website.

3.1.1. Laboratory refiners

In the laboratory scale, PFI Mill, Valley Beater, Jokro mill and pilot-refiners are used for refining the small pulp samples where the mechanisms of refining are different.

3.1.1.1. PFI mill. As illustrated in Table 2, the PFI mill is the most commonly used laboratory refiner. It is a high energy and low intensity refining device (Kerekes et al., 2005). Using this refiner, the pulps are refining between a stainless steel roll with bars and a rotating disk with a smooth bed where pulps are distributed over the disk wall uniformly. Both elements rotate in the same direction. However they have different speed. The refining specification is reported either in “revolution”, or “PFI count” which is 10 times of revolution.

3.1.1.2. Valley beater. The Valley beater is another refiner that has a different shape in comparison to PFI mill, which consists of the roll bars and bedplate. Comparing to the PFI mill, the bigger amount of samples and longer time for refining is needed in the Valley beater (Yasumura, DAlmeida, & Park, 2012). The valley beater increases the fiber cutting and the fine formation, whereas PFI mill tends to increase the internal fibrillation and swelling, comparatively (Garcia et al., 2002; Jones, Venditti, Park, Jameel, & Koo, 2013; Park, Venditti, Jameel, & Pawlak, 2006).

After each run in the refining devices, the refining degree is assessed by the Canadian Standard Freeness test (CSF). Detail of the test is incorporated in this section. Regarding the calibration requirements, it is recommended for the valley beater that the refining should not continue when pulp reach at 250 CSF.

3.1.1.3. Jokro mill. The Jokro mill is not very common to the researchers in comparison to other laboratory devices. Jokro mill same as PFI mill consists of a refining roll with several bars within a container, where the groove dimensions in Jokor mill are smaller than PFI mill, especially in groove depth. The time is regulated for controlling the refining.

3.1.1.4. Small-scale pilot refiner. The small-scale pilot refiners are used to simulate the industrial scale refining. They have different types of geometries same as the industrial refiners. Details of different geometries are presented in Section 3.1.2.

3.1.2. Industrial refiners

The refiners with different types of geometries such as disk, conical and cylindrical are used in the industry. They consist of rotor and stator, which could have various patterns of plates with different grooves and bar dimensions. Nowadays, the manufacturers are proposing new refiners by considering the industrial demands.

One of the key objectives in developing the new refiners is the reduction of energy consumption. However, many suppliers have stated that their refiner can lead to more energy savings, this claim needs further investigation to know how much energy could be saved in comparison to the existing ways such as using of one large refiner instead of numerous small refiners.

Another parameter is homogeneity. Reduction in the amount of untreated fibers is an important challenge in fiber refining. Although different concepts such as multiple disk refining, low consistency (LC) and stable gap refining could slightly overcome this

Table 2
Summary of different refiners.

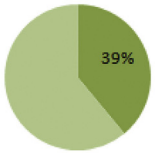
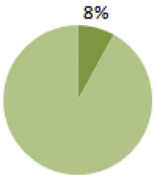
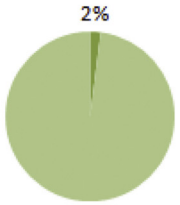
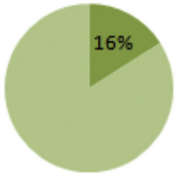
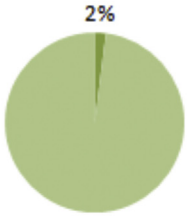
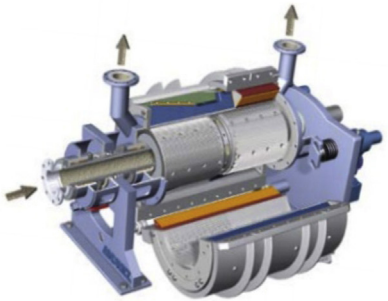
Type of refiner	Share of each refiner in the scientific literatures in Scopus since 2000	example	configuration	specification	Reference
PFI mill		-		The external diameter of roll is 201 mm. It has 33 bar, each 50 mm long and 5mm wide. Initial pulp consistency is 10% and 30g oven dry pulp sample can be refined per run.	(TAPPI, 2008)
Valley beater		-		5.2.1 A dimensioned drawing of the 0.7-kg (1.5-lb) beater is shown in Fig. 1. The bedplate and roll shall be made of Type 410 heat treatable stainless steel (chromium content, 11.5 to 13%) or its equivalent. The diameter of the roll with flybars inserted (32 in number) shall be 193.8 mm (7 5/8 in.). The thickness of each flybar shall be 4.8 mm (3/16 in.) and the width of the roll shall be 152.4 mm (6 in.). The Brinell hardness of the flybars shall be 350 to 400 and bedplate bars 325 to 375. 5.2.2 The bedplate assembly shall consist of 7 bars, each 3.2 mm (1/8 in.) thick, 15.9 mm (5/8 in.) high, and spaced 2.4 mm (3/32 in.) apart. These shall be bent into a V-shaped form, having an angle of 5° with the roll axis, with the apex of the V pointing in the direction of movement of stock over the plate. The grooves between the bedplate bars shall be filled with strips of kiln-dried white oak. The projected length of the bedplate assembly shall be 158.7 mm (6 1/4 in.) and its projected width 42.8 mm (1 11/16 in.) and shall be ground in to conform to the bars in the roll. The bedplate bar assembly shall be securely mounted in the bedplate with a low melting-point bismuth alloy, to prevent heat damage to the wooden spacers.	(TAPPI, 2001)

Table 2 (*Continued*)

Jocro mill		-		The external diameter of roll is 89.15 mm. It has 35 bar, each 60mm long and 20mm wide. Initial pulp consistency is 6% and 96g oven dry pulp sample can be refined per run.	(DIN54360, 2004)
Conical refiner		OptiFiner Pro, by Metso		Motor size is 1200KW. It has 60% energy saving at 23 SR when compare to old refiners. It is compact and easy to instal. pulp consistency is between 3-6%	(Huhtanen & Partanen, 2013)
Cylindrical refiners		Papillon refiner, by Andritz		It has several models such as CC 380, CC450 and CC 600. The specifications for CC600 are as below: diameter of refining area is 600 mm. Motor size is 2000 kW. Idle load is 160 kW.	(Sixta, 2008)

issue, homogeneity is still an aspect which is reflected in the design of new refiners.

Capacity is another point. It can be restricted by the available net power as well as the amount of volumetric flow passed through the refiner. In fact, enhancement of the capacity along with the improvement of the fiber quality during refining is an important view considered in the design of refiners.

3.1.2.1. Disk refiners. Generally, the disk refiner is a low cost machine. It can be classified into two groups; the single disk refiner and the multiple disk refiners. Among the multiple disk refiners, the double disk refiners e.g., TwinFlo refiners (Table 2) are more popular in the paper mills. It is generally known that the double disks can improve the homogeneity and save energy more than the single disk refiners. However, recently it has been claimed that a new 42″ single disk refiner named "PURE" has 25% reduction in power more than the conventional parallel 42" double disk refiner (Demler, Beder-Miller, & Aldridge, 2012).

Disk refiners have been widely used by several authors for understanding the forces and mechanisms in LC refining (in LC refining, the pulp consistency is 3–6%; Heymer, Olson, & Kerekes, 2011; Luukkonen, 2011; Martinez & Kerekes, 1994; Rajabi Nasab, 2013). Furthermore, disk refiners have been used for manufacturing the microfibrillated and nanofibrillated cellulose (Nakagaito & Yano, 2004; Pääkkö et al., 2007; Siró & Plackett, 2010; Tonoli et al., 2012). In this case, refining process can be applied independently or combined with different downsizing mechanisms such as homogenizing and grinding (Afra, Yousefi, Hadilam, & Nishino, 2013; Rebouillat & Pla, 2013; Stelte & Sanadi, 2009). Compared with combined processes, the energy consumption of refining process is higher because of the large amount of passages (Abdul Khalil et al., 2014). It has been reported that the nanofibrillated cellulose from short staple cotton fibers could be produced after 30 passage of refining by using a disk refiner (Karande, Bharimalla, Hadge, Mhaske, & Vigneshwaran, 2011).

3.1.2.2. Conical refiners. Several types of conical refiners with different cone angles such as Jordan-type, Claflin-type and Conflo-type have been introduced. Conical refiners have higher capacity compared to the disk refiners (Andersson, 2011)

One of the newest of conical refiners is OptiFiner Pro refiner (Table 2). It can replace two traditional refiners. Energy saving, homogeneity improvement and capacity increase in this type of refiner are based on this subject where a different method of feeding the stock into the refining zone is introduced. In this technique, the stock can be spread to the bars uniformly and could be placed in the refining zone directly (Huhtanen & Partanen, 2013).

3.1.2.3. Cylindrical refiners. The cylindrical refiners are not popular like disk or conical refiners, but they are gradually attracting some attentions because of their ability to increase the homogeneity. Rene, Ulrich, and Wolfgang (2006) have reported that the degree of fiber swelling in the cylindrical refiner is to some extent, higher than double disk and conical refiners. They also noted that the cylindrical refiner could treat the pulps more uniformly and decreases the percentages of fibers remain untreated.

"Papillon" is one of the newest types of cylindrical refiners (Table 2). Due to the cylindrical shape, it has less pumping action than disk or conical refiners; also it has one gap, so the gap control could be done more accurately. On the other hand, it is a costly machine and the industry is still evaluating the economic investments, however, it is claimed that it has very low no load power, and this leads to the saving of energy.

3.2. Control tests in the stock preparation

One of the most commonly used methods to monitor the occurred changes during the stock preparation, is the measurement of the amount of water in a pulp suspension which could pass through a mesh screen, and is so called "freeness".

Since the value of freeness is found to be a function of fiber fibrillation and fines formation (Polan, 1993), the freeness test can be used as an indicator of refining effects (Bhardwaj, Hoang, & Nguyen, 2007a). For instance, in some cases, degree of fiber swelling can be specified as a function of freeness (Rene et al., 2006). In other words, generally, the fiber and paper properties could be presented based on freeness (Helmerius, von Walter, Rova, Berglund, & Hodge, 2010).

To explain how refining affects the freeness, it is possible to get help from available theories regarding the release of water during paper manufacturing. As mentioned, refining causes the swelling and hence the fibers become flexible. In this situation, fibers entangled firmly together and make a web during draining in the freeness test. Furthermore, refining creates the fines that are not attached to the fibers (refer to the secondary fines in Section 4.3); these fines move freely and finally get stuck in the pores between fibers, which mean blocking the water flow path and slowing the drainage (Fig. 3), (Hubbe & Heitmann, 2007; Paradis, Genco, Bousfield, Hassler, & Wildfong, 2002). Therefore, it is concluded that refining reduces the drainability (Beg & Pickering, 2008; Gao, Huang, Rajbhandari, Li, & Zhou, 2009) which is not attractive in the paper making.

The methods used for measuring the freeness include the off-line and on-line procedures are briefly described here.

3.2.1. Off-line measurements

3.2.1.1. CSF test. The widely used method to measure the freeness especially in the laboratory scale has been so-called CSF. To carry out freeness test, a sample of suspension with disintegrated pulps (3 g of pulp in 1 L of water) is poured in the chamber and passes though the (funnel) mesh. The water is drained from the side and the bottom orifices and the collected discharged volume by side orifice is the freeness value. The higher CSF value means higher and faster draining. To delaminate the effect of chemical ions present in the water and to gain the accurate results it is recommended to use the distilled or de-ionized water during the test.

The CSF value measured in the laboratory can be correlated with the pulp consistency and temperature according to the tables presented in TAPPI (1999). It can be found from the TAPPI tables, for example, at 400 CSF; around 10% change in CSF value could be caused by the $\pm 5\,^{\circ}C$ change in temperature or by the consistency variation of $\pm 0.06\%$ in comparison to the reference temperature $20\,^{\circ}C$ and consistency 0.3%.

Usually the unrefined, unbleached softwood kraft pulps have the CSF values as high as 750 ml while after refining, the typical values of Freeness for kraft pulps drop down in the range of 600–250 ml which is strongly dependant on the initial freeness value (Genco, 1999; Technology, 2001). Reduction in freeness for both wood and non-wood pulps has been reported by different authors (Bhardwaj et al., 2007a; Lumiainen, 2000; Nugroho, 2012). For example, it has been reported that during refining of organosolv wheat straw, the freeness decreases disproportionately due to the presence of weak fiber which appears from the fiber dehydration (Tschirner, Ramaswamy, & Goel, 2002).

Reduction in freeness due to PFI revolution for hardwood is more than softwood pulp. It means to reach a specific freeness, softwood pulps show more resistance to refining than hardwood and non-wood pulps which means they need more PFI revolution and more

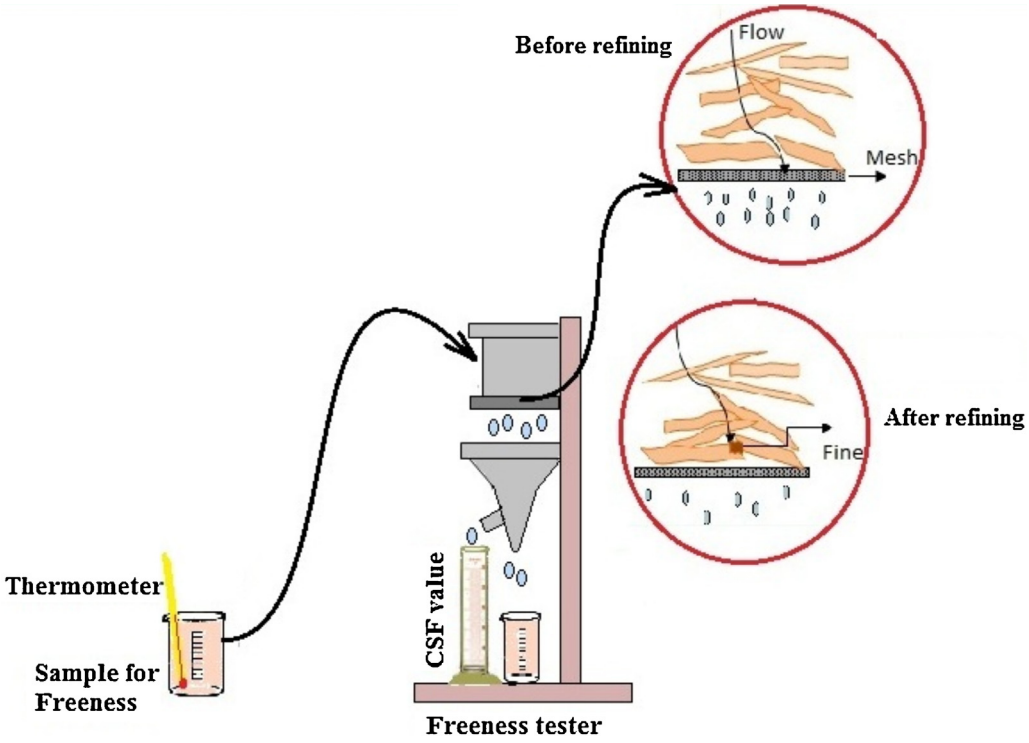


Fig. 3. Schematic diagram of measuring the CSF value and the mechanisms affecting the freeness.

refining time as well (Banavath, Bhardwaj, & Ray, 2011; Bhardwaj et al., 2007a), this might have linked with the amount of lignin in the pulps. As presented in Table 1, softwood pulps have a higher amount of lignin than hardwood pulps. It has been reported that at the same refining revolution, the pulps with fewer Kappa number exhibit the lower freeness degree (Gulsoy & Tufek, 2013; Rosli, Mazlan, & Law, 2011).

Energy consumption and operating cost are several important aspects that are interested in the study of refining and freeness. More refining would be happened due to the higher SRE and this means higher reduction in freeness (Nugroho, 2012; Seth, 1999).

The high consistency (HC) refining (in HC refining the consistency is 20–40%) causes a higher drop in freeness for pulps in comparison to (LC) refining (Miles & Karnis, 1991) so with the increase in consistency the less energy is needed to achieve the specific freeness (Miles & Karnis, 1991). In case of different pulps, mixing the softwood with hardwood pulps can decreases the required SRE for a specific Freeness (Nugroho, 2012). In addition, Nugroho (2012) said, by reducing the gap between plates, the freeness and SRE could be reduced.

3.2.1.2. The Schopper Riegler (SR) test. SR test is the oldest drainability test which could be used for information about conformability but in principle this is similar to the CSF test. It is to be noted that the samples in this method have lower concentrations, and a reverse scale is used for measuring of the water drainage. Both the used apparatus in these tests have similar design, although in SR apparatus, the wire screen is used instead of calibrated screen plate. In contrast to the freeness, refining increases the SR value and higher SR means the lower discharge. The measuring scales are between 0 and 100 SR. Many literatures used the SR value to characterize the pulps (Table 3; González, Alcalá, Arbat, Vilaseca, & Mutjè, 2013; Lanson, Karademir, & Sampson, 2006; Mutjé, Pèlach, Vilaseca, García, & Jiménez, 2005; Zeng, Retulainen, Heinemann, & Fu, 2012)

3.2.2. On-line measurements

Various factors such as temperature, pulp consistency, pH and amount of additives affect the freeness measurements, thereby a fast response to freeness variations and reduction of the source of stock changes lead to uniformity in the freeness, less load on the refiner and thus reduction in energy costs (Hojjatie & Coffin, 1998).

The widely used principle in the on-line analyzers is filtration in which water is extracted from a pulp suspension through a screen, and its drainage rate through a fiber mat is measured. An example of such analyzers is DRT-5200. The DTR-5200 provides two standard analog output signals, one of the signals shows the drainage time and another one shows the temperature in the chamber. The system consists of a mixing chamber which is connected from the top and below, to a measuring glass through a screen plate, and a manometer respectively. Meanwhile the system has two electrodes with different levels to measure the drainage time. The operation of the DTR-5200 can be divided into four steps (Fig. 4). In the first step (sampling), the sample is taken out from the process line using the piston pressed by the air cylinder and is collected in the mixing chamber. The extracted sample is diluted to consistency 0.2–0.5%, to avoid the flocculation in the chamber. In the second step (formation), the sample is drained from the mixing chamber to the screen plate (due to the pressure differential formed in the manometer), and a mat is formed on the screen. In the third phase (measuring) the drained water from the fiber mat is diverted to the measuring glass and the rising time from the lower to the upper electrode is measured as a drainage time. Finally (cleaning step), the system is cleaned by the water and is prepared for the next cycle (Hojjatie & Coffin, 1998; Proulx, 2000).

Other available analyzers are Drainac IIIB, KOEI, DTR-5500 and Intelligent drainage analyzer. The specification of these analyzers is enlisted in Table 4. Clearly, the measurements in on-line sensors are faster and accurate than laboratory devices. Usually measuring the freeness by using the on-line sensors takes only a few minutes. In these sensors, the drainage time is used as an index of pulp freeness, and mostly dependent on the device, could convert to CSF or SR.

Table 3
Summary of Schopper Riegler (SR) values with various refining degrees.

Reference	Pulp	Refining counter	Refining degree	SR value
González et al. (2013)	Kraft liner/fluting pulp produced from recycle paper	PFI mill revolutions	0	34
			500	42
			1000	48
			1500	57
	Rapeseed chemithermomechanical pulp (CTMP)	PFI mill revolutions	0	52
			250	55
			500	61
			1000	65
			1500	68
	Rapeseed CTMP with 1.5% nanofibrillated cellulose (NFC)	PFI mill revolutions	0	52
			500	66
	Rapeseed CTMP with 3% NFC	PFI mill revolutions	0	52
			250	66
Zeng et al. (2012)	Bleached pine kraft pulp	Valley beater (time, min)	0	14
			15	18
			30	28
			45	52
			60	79
Lanson et al. (2006)	Commercial unbleached Kraft pine pulp	PFI mill revolutions	0	14
			56,000	41
			72,000	63
Garcia et al. (2002)	Industrial Eucalyptus globulus bleached kraft pulp (wet pulp)	PFI mill revolutions	0	17
			3000	30
			4500	40
			6000	50

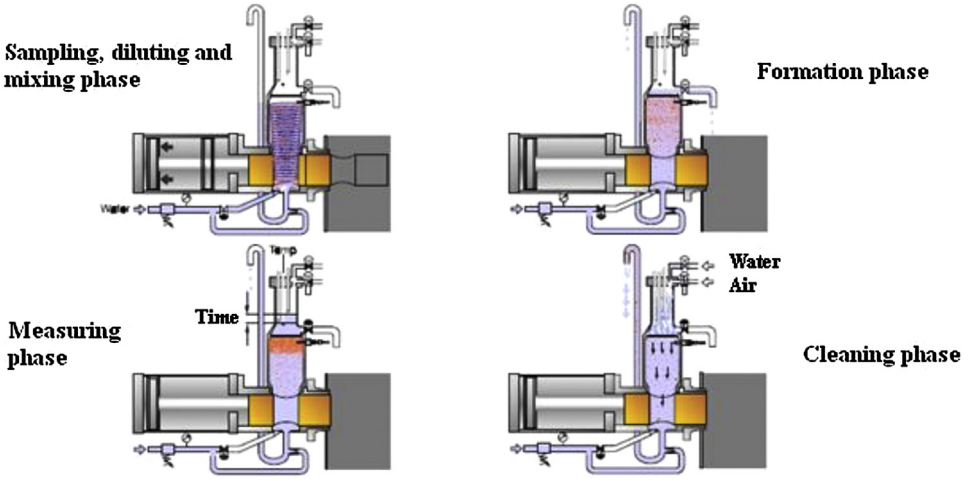


Fig. 4. Measuring principle of the DTR-5200 (Proulx, 2000).

4. Changes happen to structural properties of fiber during pulp refining

Major effects of refining of fibers that result in changes in fiber structure are categorized by many researchers (Loijas, 2010; Oksanen, Pere, Buchert, & Viikari, 1997; Page, 1989b; Rene et al., 2006). These are listed as: fibrillation (external fibrillation, internal fibrillation (swelling)), fines formation, fiber shortening and fiber straightening. Except these fundamental variations, we discuss the crystallinity and redistribution of surface chemical compositions.

4.1. Internal fibrillation

The first effect of refining on fiber layer is internal fibrillation. This is delamination of the P and S1 layers, caused by the cyclic compression action of forces inside the refiner (Haavisto, Koskenhely, & Paulapuro, 2008; Nugroho, 2012).

Internal fibrillation has been extensively studied, because it is believed by several investigators, to be the most important effect of refining (Ingmanson & Thode, 1959; Kang, 2007). As mentioned earlier, higher internal fibrillation could be achieved by using the PFI

Table 4
Specification of on line freeness analyzers.

Analyzer model	Quality measured	Freeness range	Consistency (%)	Measuring interval (s)	Reference
Drainac™ IIIB	Time 2–3 sample/min typical	0-800CSF, 10-90SR	0.5–6	60–120	Hojjatie and Coffin (1998)
Intelligent drainage analyzer	Time	25-800CSF	1.5–6	60	Optest (2005)
DTR-5500	Time	15-750 CSF 14-90 SR	1–6	120	Molter (2011)
KOEI	Volume of filtrate	0-800 CSF	1–6	210–300	Hojjatie and Coffin (1998)

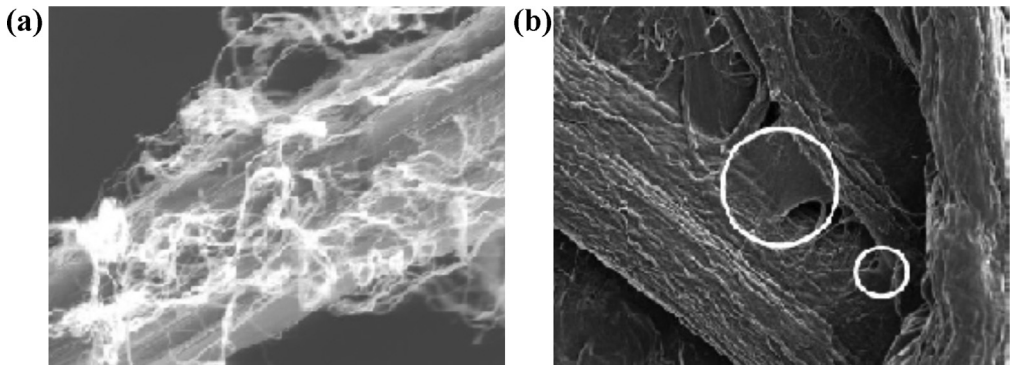


Fig. 5. (a) Example of external fibrillation of once-dried bleached kraft softwood pulp refined for 90 min in a valley beater (Kang, 2007). (b) SEM image of the refined pulps with gel-like material links (Mou, Iamazaki, et al., 2013).

mill in comparison to other laboratory refiners (Jones et al., 2013; Kerekes et al., 2005).

The breakage of inner bonds which are between cellulosic fibrils, between fibrils and hemicellulose, between cellulose and lignin, and between hemicellulose and lignin causes the pore structure inside the cell wall to expand and swelling (Maloney & Paulapuro, 1999). Actually, swelling is an evidence of internal fibrillation during refining (Hubbe, Venditti, & Rojas, 2007) and can be evaluated by the fiber saturation point (FSP) test (Joutsimo, 2004). This test was developed by Stone and Scallan (1968). In the FSP method, there is a combination of a partly high concentration fiber suspension and dextran polymer with high molecular mass and with adequate molecular size that could not be penetrated into the cell wall pores (Pulkkinen, 2010). The higher the FSP values, the more difficult the water drain from the sample. Softwood pulps usually have the smaller increase in FSP due to refining than hardwood pulps. Hardwood and softwood pulps swell inwardly and this behavior is confirmed by the decrease in the lumen size (Mossello et al., 2010). A different behavior of swelling in bamboo pulp as a non-wood pulp, in comparison to wood pulp was reported by Wai, Nanko, and Murakami (1985). They claimed, the bamboo swells toward the outside of cell wall, and this is due to the small size of lumen in bamboo. Furthermore, they emphasized that the internal fibrillation dispreads rapidly in bamboo than wood pulps.

Internal fibrillation also makes the fiber more flexible or conformable (Genco, 1999). It can be speculated that the layers which are delaminated during refining are major restraints against swelling due to their hydrophobic nature and the high fibril angle of the S1 layer (Hartman, 1984). Loosening of the fiber wall or reducing the bending stiffness of the fiber wall due to a decrease in the effective E-modulus (a tendency of the fiber, to be deformed elastically, fiber with a high E-modulus show high tensile strength) occur in result of internal fibrillation (Lammi & Heikkurinen, 1997). Flexibility is a key factor as it governs the most physical and optical properties of pulp and paper, including paper formation and paper strength (Fernando, Muhić, Engstrand, & Daniel, 2011; Paavilainen, 1993; Peng & Johansson, 1996; Petit-Conil, Cochaux, & De Choudens, 1994). The flexible and collapsible fibers give more close contact which lead to strong bonding (Forsström, Torgnysdotter, & Wågberg, 2005; Lumiainen, 1990; Rusu, Mörseburg, Gregersen, Yamakawa, & Liukkonen, 2011). Rusu et al. (2011) showed that fiber bendability increases with the internal fibrillation, and the spruce has a higher increase in flexibility than pine.

4.2. External fibrillation

Pilling off the fibrils from the fiber surface is associated with exposing of the S2 layer which is defined as an external fibrillation (Page, 1989b); this phenomenon can be observed in the microscopic images as the fibrils are still attached to the fiber wall (Fig. 5(a)). Sometimes these un-removed layers are sources of roughness enhancement (Fardim & Duran, 2003). The most significant effect of external fibrillation is increasing of the specific surface area of fibrils (Clark, 1969; Nugroho, 2012). During the fibrillation process, some hydrophilic compounds from the cell wall are released which produce the gel-like layers (Fig. 5(b)). These gelatinous layers improve the fiber–fiber bonding which can appear as a film after drying (Mou, Iamazaki, Zhan, Orblin, & Fardim, 2013b).

However it is claimed that external fibrillation is the main reason for improving the bonding (Clark, 1969), the role of external fibrillation on pulp and paper properties is a controversial subject. In comparison with internal fibrillation, the external fibrillation has been subjected to less interest because during refining it is associated with internal fibrillation and fine formation and these simultaneous changes made it difficult to judge the role of external fibrillation. It is reported that by using a special refiner (ultra-fine friction grinder) the external fibrillation could be monitored independently (Kang, 2006a, 2006b) although several researches on external fibrillation accomplished through conventional refiners (El-Sharkawy, 2008; Haavisto et al., 2008; Molin & Daniel, 2004). The specifications and conditions of mechanical treatment in ultra-fine friction grinder and related illustrations are mentioned in Table 5. The pulp passing through the gap between the stone plates is subjected to compressive and shear forces. Kang (2006a, 2006b) found that the monitoring of the change in FSP values, with different gap clearances in the recommended refiner, could bring about a unique condition to survey the influence of external fibrillation on pulp and paper characterization. FSP increases dramatically with the decrease of the gap clearance, whereas at a larger gap the FSP reaches a plateau, so internal fibrillation remains constant while the external fibrillation continues to increase (Table 5). Therefore this plateau behavior could be used to recognize the role of external fibrillation.

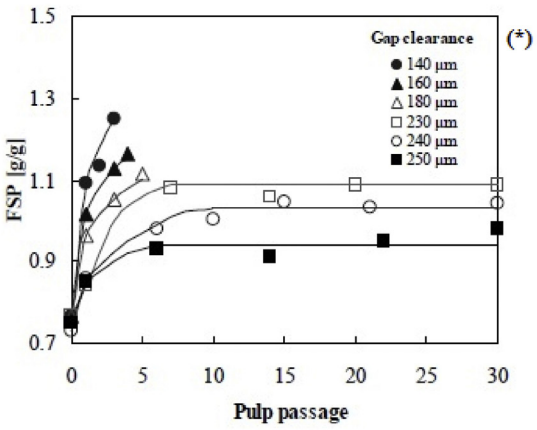
More external fibrillation could be produced by using the high cutting speed fillings than lower cutting speed fillings in refiners because at high cutting speed fillings, the fibers have more chance to pass the filling grooves than trapped between the bars (Haavisto et al., 2008).

4.3. Fine formation

Two kinds of fines are considered present in pulp that are: primary fines and secondary fines. Primary fines are present in unbeaten pulp and come from ray cells and parenchyma cells (Ferreira, Matos, & Figueiredo, 1999; Wistara & Young, 1999) while secondary fines are produced during the refining, as a result of external fibrillation or fiber shortening (Hartman, 1984). These

Table 5
Specification and condition of mechanical treatment in Ultra-fine friction grinder (Kang, 2007).

Illustration	specifications	Condition for treatment
	Consists of two grinding stones : a lower rotating one and an upper stationary one. The surface of the outer periphery of the stone is rather flat and the tapered inner part is grooved, which allows the gap to be narrowest at the outlet of the grinder and widest at the inlet or inner part	3-6 % initial consistency. Rotating speed 900 & 1500 rpm.



(*) Diagram of FSP versus pulp passage for different gap clearances, obtained using ultra-fine friction grinder, Once-dried bleached kraft pulp with 6% consistency treated at a rotating speed of 1500 rpm (Kang, 2007)

fines consist of fragments of primary and secondary walls with size less than 0.3 mm (Heymer, 2009; Pattara, 2012). Usually a 200 mesh (75 μm) screen of a classifier is used to remove fines (Ferreira et al., 1999). Fines have high surface area and can improve the fibers bonding, though, they have negative effect on drainage time (Wistara & Young, 1999). Increase of refining time or shear rate means an increase of the amount of fines (González et al., 2012; Page, 1985; Zeng et al., 2012). The primary fines content of chemical pulp is typically less than 2%, but refining can increase the total fines content to 15%. Banavath et al. (2011) observed that the refining increases the fine content of wheat straw significantly followed by the hardwood and bagasse pulp.

It is noticeable that there is a significant difference in the amount of produced fines, between the refined never dried pulps (virgin pulps) and the refined recycled pulps (re-wet pulps which are produced by the disintegrating of the socked paper sheets in the water). The refined recycled pulps formed fewer fines than refined never dried pulps and more reduction in fines content occurs with the more rounds of recycling (Chen, Wan, Zhang, Ma, & Wang, 2012; Wistara & Young, 1999). Laivins and Scallan (1996) also found that PFI mill refining of dried and re-wetted (first recycling) chemical pulps recovers the swelling capacity of the fibers and fines, and secondary fines have more swelling than the primary fibers. It is stated that swelling of the fines increases, as more energy is applied (Luukko & Maloney, 1999; Luukko & Paulapuro, 1999).

Different behaviors of fine formation based on different consistencies have been reported by authors. An increase in fine content has been observed by increasing the specific energy consumption of LC refining (Mou, Iamazaki, et al., 2013). HC refining causes fewer fines formation while LC refining tends to produce more fines (Kang, 2006a), however Wistara and Young (1999) have reported the different results. They showed that there is no significant decrease in the production of fines, %P (fibers with the length in the range of 0.00–0.20 mm) by refining of never dried pulps at 5% versus 10% consistency, although higher consistency refining (30%) results in a higher amount of the fines in comparison with the refining results, at the lower consistencies (5% and 10%). The effect of gap clearance on fines formation by ultra-fine friction grinder has been reported by Kang (2006a). He has reported that the generation of fines is reduced by increasing the gap. He showed that mechanism of fines formation in the grinder seems to be different in a Valley beater.

4.4. Fibre shortening

Another change in fiber quality is a reduction in fiber length (Kerekes & Olson, 2003) which usually is an undesirable impact during the refining. Fibre length can be estimated by using the statistical average lengths such as numerical or arithmetic, length-weighted and weight-weighted.

Breaking happens when the strain on a fiber is great enough. The mechanical effects of refining will cause transverse subdivision of the fibers. Accordingly, fibers may be cut by direct shearing forces of the passing refiner bars, or they may fail when pulled from a network of other fibers (Kerekes, 1990).

It was described above that fines generation also occurs in refining because of excess force during external fibrillation. Since there is a relationship between fiber cutting and fine generation, so accurate measurement of the fiber length changes during refining is difficult (Batchelor et al., 1999). Practically, reduction of the fiber

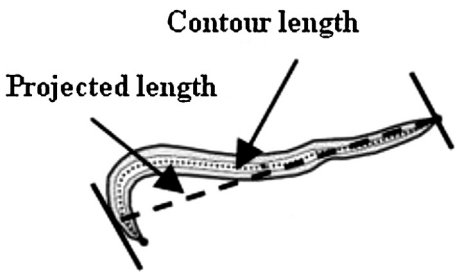


Fig. 6. Definition of contour length and projected length (Sixta, 2008).

length was calculated based on the length weighted average before and after refining. It is notable that the changes in the length-weighted fiber length did not completely reflect the changes in the number of long fibers per unit mass, due to this fact that the measured average fiber length is affected by the generation of fines during refining (Batchelor et al., 1999). Chen et al. (2012), reported that the fiber weight length of first cycle and second cycle of refined eucalyptus pulps cellulose reduced by 8% and 8.16%, respectively, in comparison to the first cycle and second cycle unrefined pulps, and showed that at consistency of 6%, refining has no significant effect on fiber cutting. Sjoberg & Hoglund (2007) pre-treated the fibers with LC and then applied the high consistency refining and observed that HC refining did not significantly decrease fiber length of bleached softwood Kraft fibers.

In some rare applications, the reduction of fiber length is a desired effect to improve formation, by decreasing the crowding number (Stoere, Nazhad, & Kerekes, 2001). The shortening of fibers improves sheet formation considerably due to decrease in the crowding number which leads to the lowering of flocculation tendency (Kerekes et al., 2005; Zeng et al., 2012) and smaller sizes of flocs formation (Chen et al., 2012; Ramezani & Nazhad, 2005), thus, contributing to paper uniformity and smoothness. Details of the relationship between the fiber length and the paper properties have been studied in many literatures (Pulkkinen, 2007; Richter et al., 1996; Seth & Page, 1988).

Various results of refining influence on the fiber shortening, by using the different refiners have been reported. Zeng et al. (2012) treated the bleached pine kraft pulp with a conventional Vally beater and showed that the fiber length first increased due to fiber straightening and then decreased gradually with refining.

Ramezani and Nazhad (2005) showed PFI refining has no considerable effect on fibers shortening after treating pulps with different refining degrees (1000–12,000 revolutions). Similar results have been reported by Jones et al. (2013). Beg and Pickering (2008) refined the fiber by using a Sprout-Waldron disc refiner with a SEL of 0.89 W s/m and indicated that fiber length decreases with increased refining time. The average fiber length for virgin fiber was 2.36 mm with a fiber distribution from 0.2 to 5.8 mm and after 8 min, the fiber length was reduced to 1.84.

4.5. Fibre straightening

Chemical pulp fibers are initially curly and refining causes the fiber straightening (Mohlin & Alfredson, 1990; Page, 1985). Shape factor and curl index are two common parameters to determine the curl index. Page, Jordan and Barbe (1985) introduced the curl index (Eq. (7)) as the relationship between the fiber "contour length" and the "projected length" (Fig. 6). Shape factor, is defined as the ratio between the shortest distance between the fiber ends and the true (contour) fiber length and given as a percentage (Zeng et al., 2012). There also exist a relationship between shape factor and curl index as indicated by Eqs. (8) and (9).

$$\text{Jordan's and Page's "Curl Index"} = \left(\frac{\text{fibre contour length}}{\text{projected length}} - 1\right) \quad (7)$$

$$\text{Shape factor} = \frac{1}{\text{Curl index} + 1} \times 100 \quad (8)$$

$$\text{Curl index} = \frac{1 - \text{Shape factor}}{\text{Shape factor}} \quad (9)$$

A curl index of zero indicates that no curl is present, however in practice initial fiber is not straight and finding a fiber with zero curl index is difficult due to this fact that during the pulp processing, the pulp is subjected to the varieties of mechanical and chemical treatments which introduce the curl to the fiber. The nature of the distributions reveals that about 600 fibers must be counted to get a mean with acceptable error. Curl affects the drainage resistance of most pulps. A reduction in curl index results in a reduction in freeness value (Page et al., 1985). The average curl index for straight fiber and curly fiber is 0.1 and 0.2 respectively. Fibre straightening plays an important role in the paper properties. Straightening of fibers improves the load carrying ability as well as the stress distribution in the fiber network and mostly increase the elastic modulus and tensile strength of the paper (Gärd, 2002; Haavisto et al., 2008; Hartler, 1995). The term of curl can be classified as "reversible deformation" as it could be removed by refining (Mohlin, Dahlbom, & Hornatowska, 1996; Mohlin & Alfredson, 1990).

The curl is generally reduced upon LC refining (Page et al., 1985) while the HC refining increases the curl because of the stresses transferred between fibers (Hartler, 1995; Wistara & Young, 1999) and this is often a disadvantage, as it changes the properties of paper, for instance, the tensile strength is reduced.

The analysis of the fiber straightening in the valley beater, commercial refiner, PFI mill (Page et al., 1985; Seth, 2006) and an ultra-fine friction grinder (Kang, 2006a,b) showed that the curl index decreases with the enhancement of refining and the fibers treated in the Valley beater in comparison to the ultra-fine friction grinder, are more straightened under compressive and shear forces. It has been shown that the amount of fiber curl is decreased sharply at the initial refining with valley beater and later decreased more gently (Zeng et al., 2012). Haavisto et al. (2008) examined the effect of different plates on the fiber straightening of two types of fibers (softwood pulp and mixture of hardwood and softwood pulp). He presented that at the same refining condition, the conical filling has more influence on the straightening of pulps than wide bar disk filling and this effect is more significant in case of mixture of softwood and hardwood pulps. Furthermore, in the study of refining influence on fiber morphology of soda pulp derived from oil palm empty fruit bunches, it has been stated that with the increase of the refining degree, the fiber curl index have the largest decrease among other fiber morphologies (Rushdan, 2003).

Mohlin (2002) applied the refining gap as a parameter indicating control of changes in fiber curl and reported that the fiber curl promotes at a critical level of about 0.3 mm of the refiner gap.

4.6. Crystallinity of cellulose

Change in the crystallinity during refining is another hypothesis that has been considered in some studies. Although the supermolecular structure of cellulose consists of two domains; crystalline and amorphous regions, presence of long chains and large amount of hydrogen bonds formed between chains due to the hydroxyl groups, increase the cellulose tendency to have a crystalline structure. The ratio of crystalline to amorphous domains is so-called "degree of crystallinity" (Sixta, 2008). The degree of crystallinity of cellulose is one of the most important crystalline structure parameters. Generally, an increase in crystallinity brings about an increase in tensile strength and stiffness and a decrease in chemical reaction (Chen et al., 2012; Leitner, Seyfriedsberger, &

Kandelbauer, 2013; Tschirner, Barsness, & Keeler, 2007; Yuan et al., 2013). Another parameter that has been affected by crystallinity is swelling. As the water does not penetrate into the crystalline reign, the absorption of water by the cell wall will be decreased by an increase in crystallinity. In other words, an increase in the crystallinity results in a decrease in swelling of fiber (Kongdee, Bechtold, Burtscher, & Scheinecker, 2004; Wan, Yang, Ma, & Wang, 2011). It should be noted that crystallinity of pulps usually used in papermaking is approximately 60–70% (Kočar et al., 2004).

The crystallinity could be determined by various methods such as X-ray diffraction (XRD), nuclear magnetic resonance spectroscopy (NMR), and Fourier transform IR spectroscopy (FTIR) (Chen, Wang, Wan, & Ma, 2010; Lian, You, Huang, & Li, 2012; Liu, Yu, & Huang, 2005; Pulkkinen, 2010; Thygesen, Oddershede, Lilholt, Thomsen, & Ståhl, 2005).

Chen et al. (2012) reported that in the initial phase of refining, the amorphous regions of cellulose are influenced by the forces and the crystallinity of cellulose increases while in the later phase of refining, the crystalline regions of cellulose are subjected to the force, thereby the crystallinity decreases (Ibrahem, Yousef, & El-Meadawy, 1989). It has been reported that a decrease of crystallinity for high degree of fibrillated fiber (nanofibrillated fiber) which refined in several passages (Karande et al., 2011; Tonoli et al., 2012). Karande et al. (2011) analyzed the XRD spectra and showed a decline in the crystal peaks of the cotton fibers, with further refining. They indicated that the degree of crystallinity decreases from 81.28% in five passes to 65.93% in 30 passes.

An increase in crystallinity in the prior stages of refining specially has been reported for unbleached pulps. Unbleached pulps contain lignin and hemicelluloses (amorphous structure). As a result of refining, these materials can be removed partly which result in an increase in crystallinity (Leitner et al., 2013). In Leinter's study, the data has been presented only after 2000 revolution PFI mill and there is no data available regarding the further refining for monitoring the trend of the crystallinity.

To sum up, it can be stated that crystallinity is sensitive to degree of refining, and increase or decrease in refining time or severity will change the results. To confirm the claims regarding the effect of refining on the crystallinity, more studies and experiments are needed to be done by researchers.

4.7. Redistribution of surface chemical compositions

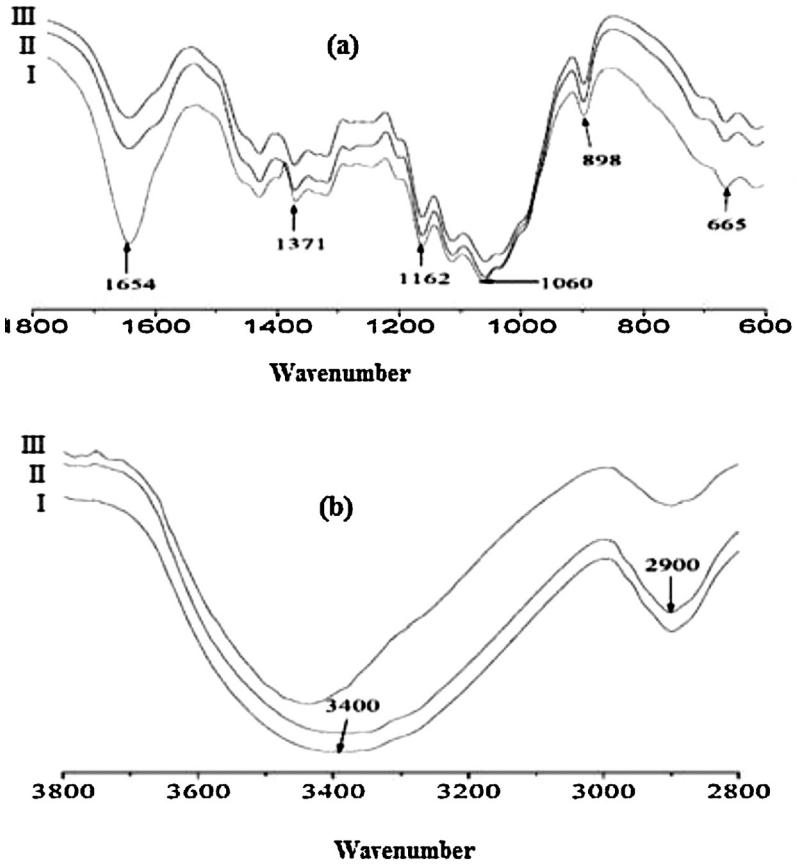
It is stated that refining can release and expose the chemical compositions existence in the fiber wall pores. This theory has recently attracted some interests to investigate the effect of refining on surface chemical compositions. The results showed that during refining, the functional groups have no strong change while slight variations occur in the distribution of surface chemical compositions. Different equipment is used to find the data of chemical composition such as scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), time-of-flight secondary ion mass spectrometry (ToF-SIMS), and FTIR (Fardim & Duran, 2003; Mou, Iamazaki, et al., 2013). The study of the structure assignments corresponding to the absorption bands (Fig. 7(c)) from FTIR, shows that the functional groups remain essentially constant during refining because refining is just a mechanical treatment (Fig. 7(a) and (b)) (Chen, Wan, Dong, & Ma, 2013; Chen et al., 2012; Yuan et al., 2013). However, there are some absorption peaks which are slightly shifted to the different wave numbers. An example of shifted absorption bands can be found in the study presented by Yuan et al. (2013), which reported a change in absorbance of the hydrogen bond strength. Moreover, Fardim and Duran (2003) have presented the ToF-SIMS spectra of refined and unrefined samples and have noted the same components in both cases while the peak intensities are not similar. It was reported that the distribution of the chemical composition of fiber surfaces changes during refining, and it leads to a higher exposure of carbohydrates. The study of high-resolution XPS spectra confirms the variations in the C1s peak. C1s peak consists of four groups of carbon atoms; C1 (C—C or C—H), C2 (C—O), C3 (O—C—O or C═O) and C4 (O═C—O) (Freire et al., 2006). The C1 and C2 components are affected by the amount of surface lignin and carbohydrates respectively (Liu et al., 2012). Refining leads to an increase in the C2 and a decrease in the C1 (Fardim & Duran, 2003). Also an increase in C4 (correspond to the magnitude of carboxyl groups) during refining has been reported (Mou, Li, et al., 2013). The results obtained from ToF-SIMS are in agreement with the XPS results. The ToF-SIMS results postulate that during refining, surface coverage by carbohydrates increases possibility due to the internal fibrillation. Moreover, it has been seen that surface coverage by lignin decreases by refining (Fardim & Duran, 2003) due to opening up of the cellulose during external fibrillation (Mou, Li, et al., 2013). However, an increase in surface coverage by lignin has been reported by Mou, Iamazaki, et al. (2013), who compared the surface chemical composition of the two different samples of pulps, one sample contains the pulp with fines (whole pulp) and another one contains the fines removed pulp (fiber fraction). He showed that the surface coverage by lignin increases in the fiber fraction samples by refining. It can be explained that during kraft pulping, the hemicelluloses are deposited on the primary cell wall therefore the fines created during refining are composed of these deposited hemicelluloses. In conclusion, removal of the fines from samples can expose the lignin more. In addition, it has been reported that the surface coverage by extractives increases with refining where the internal fibrillation, and distribution and re-deposition of the hydrophobic compounds (most of fatty acids) on the cellulosic surfaces are performed (Mou, Li, et al., 2013; Mou, Iamazaki, et al., 2013).

5. Changes happen to the electrokinetic properties of fiber during pulp refining

Wood fibers develop a negative surface charge when suspended in water (Aloulou, Boufi, Belgacem, & Gandini, 2004; Bhardwaj, Duong, & Nguyen, 2004; Horvath & Lindstrom, 2007; Sarrazin, Beneventi, Chaussy, Vurth, & Stephan, 2009). The level of anionic charge on the pulp supplied from the pulp mill is typically –20 to –25 mV depending on the level of carboxyl acid groups present in the pulp which in dependent on the source of fibers, and some chemical treatments applied in the pulping process (Banavath et al., 2011; Genco, 1999). For example, hardwood pulps have a higher surface charge and total charge density than softwood pulps.

Two mechanisms: (i) presence of ionizable groups in the fiber cellulose such as carboxylic acids, sulfonic acidic, and functional groups in lignin, (ii) adsorption of suitable ions, have been introduced by researchers as a responsible for surface charge development (Banavath et al., 2011; Bhardwaj et al., 2007a; Bhardwaj, Hoang, & Nguyen, 2007b; Bhardwaj, Kumar, & Bajpai, 2004; Carrasco, Mutje, & Pelach, 1996; Chen, Wang, & Lucia, 2004; Stratton & Swanson, 1981). However, Goulet (1989) reported about the pH limit of 10.5, which is below this level, a less number of hydroxyl groups of wood fibers could be ionized.

Numerous studies have been conducted on the effect of refining on electrokinetic properties of fibers (Bhardwaj, Duong, et al., 2004; Bhardwaj, Kumar, et al., 2004; Carrasco et al., 1996; Herrington & Petzold, 1992; Horvath & Lindstrom, 2007; Penniman, 1992). Refining increases the surface charge while it has no considerable influence on the total charge.



(c)

Wavenumber (cm^{-1})	Assignment
3350	Intra- molecular OH stretching vibration, related to rupture of cellulose hydrogen bonds.
2900	CH, CH2 and intermolecular OH stretching vibration
1637	Absorbed water and oxygen-containing group
1432	HCH and OCH in- plane bending vibration
1372	Cellulose and hemicelluloses CH bending vibration
1163	Cellulose and hemicelluloses C-O-C stretching vibration
1058-1060	Cellulose and hemicelluloses C=O stretching vibration
898	Amorphous cellulose

Fig. 7. (a) and (b) FTIR spectra of unbleached eucalyptus pulp in different beating times: (I) 5 min, (II) 15 min and (III) 25 min, (c) the assignment of FTIR absorption bands (Yuan et al., 2013).

It is widely known that charge groups affect the fiber swelling, fiber flexibility and conformability, wet-end chemistry, retention of cationic papermaking additives, flocculation and mechanical properties of the paper such as tensile index (Grignon & Scallan, 1980; Joutsimo, 2004; Laine, Buchert, Viikari, & Stenius, 1996; Lindström, 1989; Lyytikäinen, Saukkonen, Kajanto, & Kayhko, 2010; Zhang, Sjögren, Engstrand, & Htun, 1994), thus the measurement of the charges is a subject of interest. To obtain the charges, some methods based on titration are recommended, such as polyelectrolyte titration, conductometric titration and potentiometric titration (Bhardwaj et al., 2007a; Cui, Pelton, & Ketelson, 2008; Horvath, 2006; Hubbe & Chen, 2004). In polyelectrolyte titration, use of a cationic polymer of high molecular weight is suggested. Using a polymer with low molecular mass is not recommended because the cellulosic fibers have a porous structure and the low molecular mass of the polymer can penetrate through the internal layer of fibers, thus losing much of its ability to affect the electrokinetic measurements. It is worth noting that the previous findings show that there is no significant difference between results obtained from conductometric titration and potentiometric titration (Bhardwaj et al., 2004a; Fardim et al., 2002). Apart from the size and molecular weight of additives, the presence of fines could affect the charges. The magnitude of surface charge for fines is more than the fibers due to the different accessibility level of charged groups in the fibers and fines (Lyytikäinen et al., 2010).

As explained earlier, there are two mechanisms occurring during the refining which contribute in changing the cationic demand. There is an interesting question that how these mechanisms can contribute during refining. The facile answer is that increase in interactions between ionizable groups and cationic chemicals due to the fiber fibrillation during refining generally lead to the presence of ionic exchange as a first mechanism. In the next stage of refining, carboxylic groups get surrounded by water molecules, so their activity as well as ionic exchange decreases. With further refining, due to fibrillation and fines formation, the surface area increases. As a result of this development, adsorption as a second mechanism contributes to enhance the cationic demand of suspension (Carrasco et al., 1996). It is noticeable that during refining, change in the ionic exchange is not significant and is independent from refining degree (Fig. 8; Mutjé et al., 2006). As illustrated in Fig. 8, there is a difference of behavior between different types of pulps. This behavior could be explained by considering the amount of fines as well as magnitude of carboxyl group's density, in the pulps.

Monitoring of changes in the behavior of zeta potential has revealed that the refined pulps with no chemical cationic, result higher magnitude of zeta potential than unrefined pulps, and once cationic chemical is added, the refined pulp will show a very large cationic demand. Also, there is an almost linear correlation between zeta potential and cationic demand (Bhardwaj, Kumar, et al., 2004; Miyanishi, 1995). Similar conclusions have been reported by Sarrazin et al. (2009). Bhardwaj et al. (2007a) also presented the linear relationship between the surface charge and freeness. They used the pine kraft pulp and two types of eucalyptus pulps and reported that in the same freeness, the surface charge for pine kraft pulp is higher than eucalyptus pulps; however, it has the lowest charge ratio. Furthermore, in another study, it has been found similar trend between the fiber surface charge and refining level for various types of wood and non wood pulps (Banavath et al., 2011). The study showed bagasse has the highest change of surface charge based on the increase in the freeness while bamboo has the lowest change among the pulps. Table 6 presents the charge values for different pulps, evaluated by different authors.

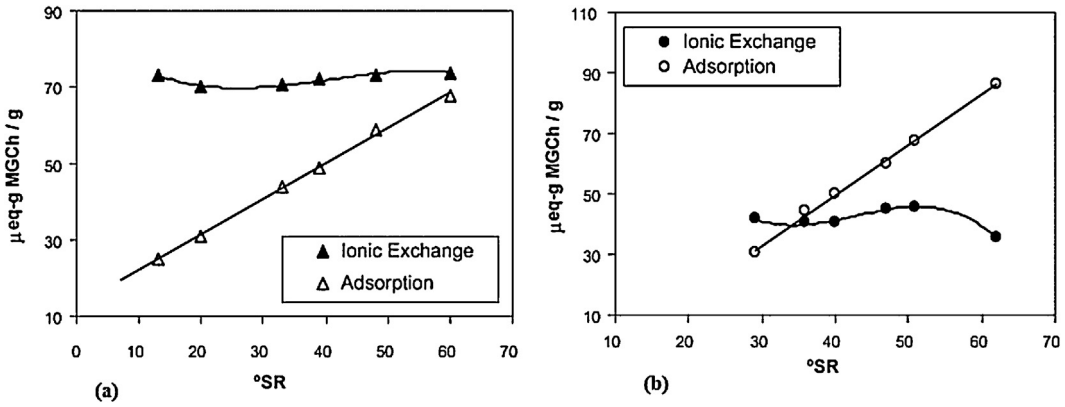


Fig. 8. Contribution of the ionic exchange and the adsorption to the polymer fixation as a function of freeness (SR): (a) eucalyptus pulp and (b) olive wood organosolv pulp (Mutjé et al., 2006).

Table 6
Summary of charges values reported by different authors.

pulp	Refining degree (CSF)	Method	Surface charge	Total charge	Charge ratio (Surface charge/Total charge)	Reference
Softwood	690	– Surface charge determined by titrations with poly-DADMAC	10.89[b]	68.6[b]	0.16	Banavath et al. (2011)
	610	– Total fiber charge determined by conductometric titrations.	12.05[b]	66.9[b]	0.18	
	500		13.65[b]	71.8[b]	0.19	
	460		14.23[b]	64.6[b]	0.22	
	330		16.11[b]	67.1[b]	0.24	
Hardwood	670		13.38[b]	70.4[b]	0.19	Banavath et al. (2011)
	540		16.56[b]	78.8[b]	0.21	
	460		18.56[b]	77.3[b]	0.24	
	370		20.73[b]	76.7[b]	0.27	
	290		22.69[b]	73.1[b]	0.31	
Bamboo	710		23.53[b]	130.7[b]	0.18	Banavath et al. (2011)
	560		25.55[b]	134.4[b]	0.19	
	440		27.17[b]	135.9[b]	0.20	
	340		28.52[b]	135.8[b]	0.21	
Bagasse	430		10.65[b]	96.8[b]	0.11	Banavath et al. (2011)
	390		14.35[b]	95.6[b]	0.15	
	340		18.97[b]	105.3[b]	0.18	
	270		25.45[b]	97.8[b]	0.26	
	225		29.61[b]	92.5[b]	0.32	
Wheat straw	400		33.98[b]	147.7[b]	0.23	Banavath et al. (2011)
	320		38.49[b]	160.3[b]	0.24	
	260		41.86[b]	149.5[b]	0.28	
Fines-free bleached eucalyptus	409	– Based on 3000 revolutions in a PFI mill		81[c]		Liimatainen (2009)
Bleached eucalyptus (unrefined)	496	– Total anionic groups as measured by methylene blue sorption at pH 8.0		79[c]		
Bleached pine. Bleached pine (unrefined)	571			55[c]		
	657			47[c]		
Kraft pine	715	– Refined in a Valley beater	58.5[c]	179.8[c]	0.33	Bhardwaj et al. (2007a)
	400	– Surface charge determined by titrations with poly-DADMAC	81.1[c]	179.8[c]	0.45	
NSSC eucalyptus	585		45.6[c]	77.8[c]	0.59	Bhardwaj et al. (2007a)
	400		53.0[c]	77.8[c]	0.68	
Kraft eucalyptus	410		63.3[c]	116.0[c]	0.55	Bhardwaj et al. (2007a)
Bleached spruce Kraft pulp	0[a]	– Surface charge determined by titrations with poly-DADMAC, Mw 9.2·105 Da	1.7[b]	37[b]	0.046	Horvath (2006)
	1000[a]		2.6[b]	37[b]	0.070	
	5000[a]		4.9[b]	38[b]	0.129	
	10000[a]		6.0[b]	40[b]	0.150	

[a] Unit of refining degree is number of revolutions (rev) in PFI mill.
[b] Unit of charge is µeq/g.
[c] Unit of charge is mmol/kg.

6. Summary

Pulp refining (beating) is a mechanical treatment of fibers in the stock preparation for improvement of the fiber quality. By considering the research results, cited throughout this article, it can be concluded that refining can affect the fiber structure and its properties through some simultaneous changes. Although, these changes are not quite desirable and have either advantages or disadvantages in papermaking, the profitable gain from refining is more than its adverse effects. For example, fine formation improves the bonding while decreases the drainage time.

Apart from the structural changes, study on the change in electrokinetic properties of fibers covers a wide range of interest in research. The charged groups have influence for instance on

fiber flexibility, retention of chemical additives and other properties. Comparing different titration methods showed that refining increases the surface charges while it has insignificant influence on the total charges.

Finally, it can be pointed out that the related investigations are widely varied due to several sources of fibers such as wood and non wood pulps, fiber consistencies and various refiner devices with different specifications. Nowadays, many efforts are given on introducing the new refiners to cope with some issues regarding the homogeneity, energy saving and capacity. However, it is declared by the suppliers that the new refiners have unique specifications which lead to treat the pulps more efficiently and save the energy, these claims still need to be verified by more evidences.

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