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The Influence of Super Masscolloider and Lab valley beater on the Morphology of Sapwood Pulp Fibres

   

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ABSTRACT

The major focus of pulp refining process is to allow effective delamination of cell wall and lumen collapse, thus breaking down internal and external structure of the fibrils, which helps in creating bonding between the fibres and thus increasing the tensile strength of the handsheet. Refining also leads to size reduction of the fibres. The objective of this research work is to investigate the effect of refining process on pulp fibres using Lab valley beater (LVB) and Super Masscolloider (SMC) separately, in order to attain micro/nanocellulose fibres to prepare biodegradable nonwoven fabric filters for capturing particulate matter below 2.5 µm. Subabul sapwood pulp obtained after Kraft cooking process (165 °C, 3 h, 1:4=W: L) is subjected to LVB refining process (pulp was run for 20 mins at 600 rpm with a consistency of 12.5 g/l) and SMC refining process. Unlike in LVB, in SMC, different clearance values of 0.5, 0.1, 0.01 units were used, at each unit refining was carried out for 20 min at 1000 rpm with varying consistencies to maintain a smooth flow between the grinders. Characterization of refined pulps is carried out using optical microscopy to compare length, diameter and aspect ratio of pulp fibres obtained from LVB and SMC using Box-and-Whiskers plots.

The studies indicate that clearance between grinders in SMC greatly affects the morphology of the fibres i.e a significant decrease in fibre diameter with decrease in clearance value was observed. At zero clearance (0.01 unit), pulp fibre with diameter of 4.5 ± 1.7 µm were found. The pulp fibres from SMC have exhibited high length and low diameter in comparison to LVB. The angle and arrangement of the grooves in SMC have helped in maintaining the desired length of the fibres, which is important for sheet strength. Hence it is recommended to refine the pulp using LVB initially for 20 mins followed by further size reduction using 0.5, 0.1 and 0.01 clearance in SMC to attain micro/nanofibres with required aspect ratio, which is optimal for strength and opacity properties of the nonwoven sheet.

KeyWords: Refining, Micro/nano cellulose fibres, Lab valley beater (LVB), Super Masscollider (SMC), Optical microscopy, Fibre aspect ratio

Introduction

Cellulose/Nanocellulose Fibres

Cellulose is the most abundant organic polymer on earth with degree of polymerization values (weight average) reaching up to 3500 in case of wood and it requires a temperature of 320°C and pressure of 25 MPa to become amorphous in water (1). The presence of ß-D-linkages in cellulose, induces the chains to remain in extended forms (1). As a consequence, cellulose molecules fit snugly together over long segments, thus giving rise to powerful associative forces that eventually results in great strength (as its microfibrils are smaller in diameter i.e 5-30 nm wide and oriented in fibre direction) of native cellulose materials and in products made from cellulose (2). Biodegradability, renewability, high thermal stability, high strength and extraordinary stiffness are some of the desirable properties of cellulose (3). The chemical modification of cellulose hydroxyl groups has facilitated the expansion of its applications. Different chemical and mechanical treatments of native cellulosic materials have shown to be efficient in producing nanometer or micrometer sized fibres (4-5). Nanocellulose have been gaining attention in the recent times as it is a renewable substitute for synthetic polymers. High mechanical properties and high specific surface area are some of the features that give nanocellulose a great potential for use in green packaging applications. Nanocellulose can be in the form of cellulose nanocrystals, nanofibres, micro fibrillated cellulose, and bacterial nanocellulose (6).Cellulose nanofibres (CNF) have high aspect ratio (L/D) i.e diameter in the nanometers range (10 to 850nm) with a wide range of length in micrometers(7-9).The high L/D ratio of cellulose nanofibres gives it a low percolation strength thus forming a high rigid network(10).

Refining

CNF's are mostly obtained by disintegration or refining of cellulose fibres by mechanical treatments (11-12). The objective of refining is to modify the pulp fibre cell walls in an optimal manner to meet the demands of a particular paper making furnish i.e. collapsibility. Mechanical refining alters the properties of pulp fibre in three mechanisms: compression for internal fibrillation of fibres, shearing for fibrillation of fibres in the exterior surface and finally cutting which reduces the length of the fibres (13). The shearing action also delaminates the cell wall, collapses the lumen, loosens the fibres and makes them flexible(14).Various mechanical treatments that are used for refining are Lab Valley Beater, PFI Mill, Super Masscolloider, Microfluidiser and High Pressure Homogenizer. Studies involving extraction of CNF through mechanical grinding have been substantiated by various researchers (15-18). The morphology and the degree of fibrillation were characterized to evaluate the performance aspects of the produced cellulose microfibrils. The studies concluded that refining through micro grinding is very effective in producing mechanical shearing and compression motions which induce external and internal fibrillation thus resulting in nano fibrils. Furthermore, the grinding process has shown a potential to break up the fibre cell wall into homogeneous microfibres(15).

Refining using Lab Valley Beater (LVB)

This equipment(Fig1) works on the principle of shear forces for rupturing the cell wall by passing the slurry through the stationary blade (beater plate) and rotating blades (beater roll) which are made of acid proof steel. The gap between the beater plate and the beater roll is reduced by increasing the weights (4.5kg + 1kg) on the pan so that very fine pulp is attained. The clearance between the two blades becomes zero by putting the maximum weight which considerably decreases the size of the fibres. As the motor rotates, the V-belt drive induces agitation/ vibration, due to which the pulp slurry moves forward in the beater basket (due to slight sloping) and passes through the gap between the beater roll and beater plate. It should be noted that beater plate fixed to the basket can move up and down i.e controlling the clearance between rotating blade and stationary blade, when weight is added. Wet pulp undergoes shearing action due to which cell wall is disintegrated, lumen is collapsed and also slight change in colour is observed due to the removal of small quantities of lignin (dark brown slurry). A decrease in length of the fibres have been observed at the zero clearance which can be optimized by loading the lever.

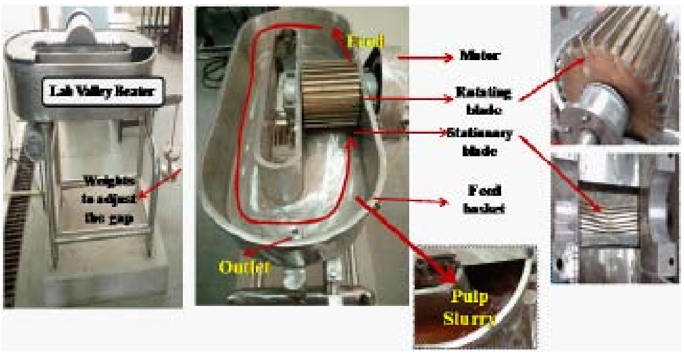

Fig 1: Lab Valley Beater. Front view (left), top view (middle) and beater plate-stationary and beater roll-rotating.

Refining using Super Masscolloider (SMC)

One of the most used ultrafine grinder is Super Masscolloider (Fig 2 a) which has two grinders. The upper grinder (Fig 2c) is fixed in its position, while the bottom one is adjustable (vertically and rotating, Fig 2b) while grinding is carried out. Both are made from composites consisting of SiC and Al_2O3 which makes it non porous and antibacterial. The grooves (Fig. 2d) present on the grinders are solely responsible for obtaining nanofibres instead of nanoparticles. The fibres undergo shearing action which breaks the cell wall and partly induces fibrillation which breaks down the hydrogen bonds (6).The characteristics of the resultant material is highly affected by the time of operation and number of cycles (10). The clearance value between the two grinders can be adjusted using the adjusting knob (Fig. 2a) which helps in reducing the diameter of fibres from micrometres to nanometres. The reduction of diameter to nanometres size occurs at zero clearance level. The grinding of the slurry charged through the hopper happens at the grinding zone, which is at the edge of the grinders (Fig 2 d), due to shearing force and high pressure in the slit and a sudden pressure drop when it leaves the grinding zone. As the clearance between the two grinders is reduced, the size of the fibres reduces and the viscosity of slurry increases. This increase in viscosity affects the smooth flow of slurry through the grooves and there will be deposition of fibres on the lower rotating grinder, which is avoided by adding extra water after each clearance value to dilute the slurry so that it flows smoothly. Adding more water after each clearance also ensures that the size reduction is only due to interaction between the grinders and the fibre and no fibre-fibre interaction is there. Multiple passes through the grinder can be a problem for the required product as it leads to severe fibrillation which reduces the strength, degree of polymerisation and crystallinity (19) of the fibres.

Fig 2: Super Masscolloider set up, (b). Lower rotating grinder with leftover slurry, (c). Upper stationary grinder, (d). Grooves for smooth flow of slurry and the actual grinding zone in a Super Masscolloider.

MATERIALS AND METHODS

Kraft Cooking Process: The raw material, Subabul wood (Leucaena leucocephala) used in this study was obtained from Kandukoor area, Ranga Reddy Dist, Hyderabad, India (Fig 3). The log selected was 7-8 years of age. Sapwood was separated from heartwood using a wood turning machine and was cut down to chips of 2 cm x 2 cm x 2.5-3 mm dimensions with the help of a chisel. The wood chips were dried at 103.5 °C in a hot air oven (Osworld OOG-90) to remove the bound moisture. The chips (sapwood) were subjected to pretreatment at 120 °C for 1 h in an autoclave (Metalab MSI-41) to soften the lignin present in the middle lamella and to remove the organic extractives (20). The delignification process (Kraft process) was carried out in a Rotary pulp digester (UEC-2015, Saharanpur, India) at a cooking temperature of 165^{o}C for 3 h with wood to liquor ratio of 1:4, 28 % sulphidity (Na_2S and NaOH expressed as Na2O) and 20% active alkali (as Na2O) (Fig. 3). After 3 h, the pulp was separated from the black liquor and it was thoroughly washed with deionized water and dried at 103.5 °C in a hot air oven for 12 h prior to refining process. The pulp refining was carried out in Lab valley beater - LVB (GEC-P40313-A) and Super Masscolloider- SMC (MKCA6-2J) separately.

LVB: 100 g of pulp was soaked overnight in 3 liters of demineralized water. Then 5 litres of water is added i.e ~100 g of pulp with ~8 litres of water (12.5 g/L) is fed into the beater basket from top. The refining was started by loading the lever with 1 kg weight followed by loading the lever with 4.5 kg of weight while pulp slurry was running in the beater. By loading the lever with 5.5 kg of weights, the clearance between the beater roll and beater plate is reduced from 10 mm to 0.1 mm (21). Here the minimum gap of 0.1 mm is considered as zero clearance. It should be noted that clearance should be reduced (adding of 4.5 Kg weight) while beater is running with wet slurry. Beating process was run for ~ 20 minutes at 600 rpm with a consistency of 12.5 g/L before the pulp slurry is discharged from the outlet drain (Fig.1).

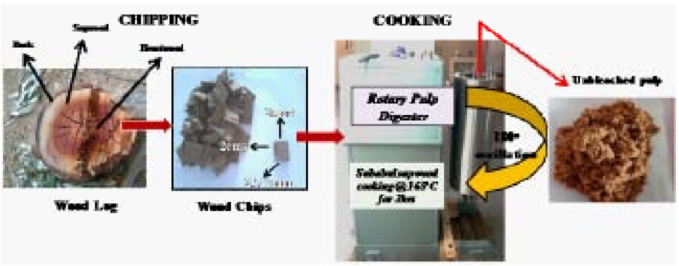

Fig 3:Kraft process of obtaining sapwood pulp from wood chips using Rotary pulp digester. Note: Digester is shown without head.

SMC: Similar to LVB, 100 g of pulp was considered for the study. The pulp was soaked overnight in 3 liters of demineralized water. The SMC was run at different clearance values (1, 0.5, 0.3, 0.2, 0.1, 0.01 units) and at each clearance it was run for 20 minutes at 1000rpm. Unlike 12.5 g/L of uniform consistency in LVB, here varying consistencies (Table 1) were used to maintain a smooth flow between the two grinders.The refining using Super Masscolloider was started using 100g of pulp in 5L of water (20g/l) at 1-unit clearance. Water was added after each reduction in the clearance unit to maintain the smooth flow, but the slurry with 50g of pulp in 6L (at 0.3 clearance) of water was not able to pass through the grooves in the grinder (Fig 4) which also increased the load on the grinder and stopped running. At this consistency level, the slurry was divided into two equal parts and the consistency was reduced to 25g in 8L of water after many trials so that the slurry flowed freely through the grooves in the grinders. The final consistency of the pulp was 25g of pulp in 14L of water i.e 1.78g/L wherein smooth flow of slurry was observed at 0.01 clearance (zero clearance). It should be noted that clearance between two grinders should be reduced while SMC is running with wet slurry.

Table 1:
Different consistencies of slurry used in Super Masscolloider (SMC)

Clearance between two grinders (units)	Pulp weight (g)	Volume of Water (L)	Pulp consistency (g/L)
1	100	5	20.0
0.5	50	6	8.33
0.3	25	8	3.12
0.2	25	10	2.50
0.1	25	12	2.08
0.01	25	14	1.78

Fig 4:Deposition or piling-up of micro cellulose fibres on the lower rotating grinder at 0.3 units of clearance due to the high consistency of pulp (8.33 g/L). This piling-up was not observed when consistency of pulp reduced to 3.12 g/L.

RESULTS AND DISCUSSION

Characterization of refined pulp was carried out to compare the effect of beating/refining process on the morphological characteristics of the fibres. The length and diameter of the fibres were measured through stereomicroscopy (8X-56 X) and optical microscopy (50X -500X) respectively. Resulting data was plotted using Box-and-Whiskers plot. Here stereomicroscope was used in reflectance mode and it has high working distance. Opticalmicroscope was used in transmittance mode and it has less working distance.

(a) Microscopic Analysis

This analysis reveals the morphology of different pulp fibres obtained after different levels of refining. A small quantity of wet pulp slurry was poured on a clean glass slide and the excess water was removed using blotting paper and the pulp was allowed to dry for 24 hrs. Imaging of individual pulp fibres was carried out using a microscope to measure length (L) and diameter (D). It is important to dilute the slurry before pouring on glass slide, so that network of single pulp fibres can be seen after drying.

The optical microscopic images of (Fig 5) refined pulp fibres after SMC refining process clearly indicates the presence of different types of cells i.e libriform (thick and long fibres, Fig 5 a), vessel (thick and very short fibres, Fig 5 a) and tracheids (thin and long fibres, Fig 5 b). Length, diameter and aspect ratio of micro cellulose fibres obtained after each clearance in SMC are compared. The idea is to optimize the clearance wherein high aspect ratio can be attained. For making a fabric or sheet, aspect ratio plays an important role than fibre diameter. So an attempt has been made to find optimum L/D ratio of micro cellulose fibres.

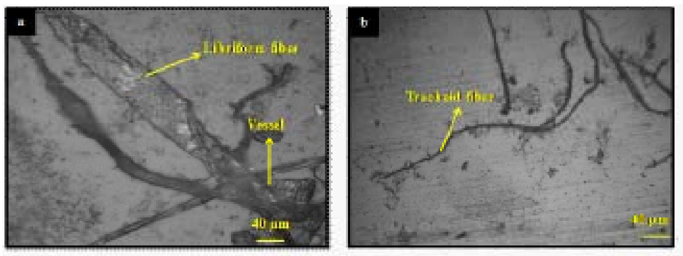

Fig 5:Optical microscopic image of fibres (transmittance mode) obtained from SMC showing different types of fibres at 0.01 clearance.

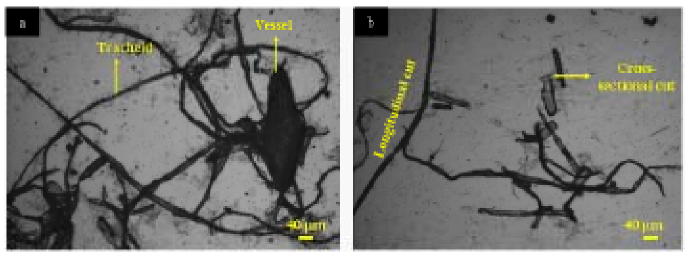

Fig 6:Optical microscopic image of fibres obtained from SMC at a) 0.1 clearance, b) 0.5 clearance.

Fig 6 shows the optical images of the fibres obtained after SMC refining. Fibres of different sizes (5 - 10µm (6 a) and 12 – 20 µm (6 b)) can be seen from the images. Microfibrillation from the fibres, twists, kinks and cross-sectional cuts can be observed from the images which could be the result of shearing forces acting on pulp fibres as a result of SMC refining.

From the stereomicroscopic images (Fig7) of refined pulp fibres after LVB and SMC refining process, a clusters of very short fibres

(100-400 µm of length) along with bigger fibres were observed, which indicates non-uniformity of pulp fibres. The presence of these short fibres may be due to the zero clearance between the two plates in LVB or between two grinders in SMC. The shear, compression and impact forces on the fibres developed due to the narrow gap between blades (LVB) or between the grinders, cuts the fibres in both transverse and longitudinal directions. Note the length measurements of micro cellulose fibres in Fig.7.

Fig. 8 shows the image of fibres after LVB refining with varying length, 500-1200µm (a) and 400-900µm (b). Fibres with wide range length obtained from the refining process can be due to the heterogeneity of the pulp fibres. Fig 9 shows the image of fibres obtained from SMC. From the image, the length of the fibres varies from 900-1400µm (a) at 0.1 clearance and 800-1100µm (b) at 0.01 clearance. The length of the fibres obtained from SMC is higher as compared to LVB and does not change much with decrease in clearance values.

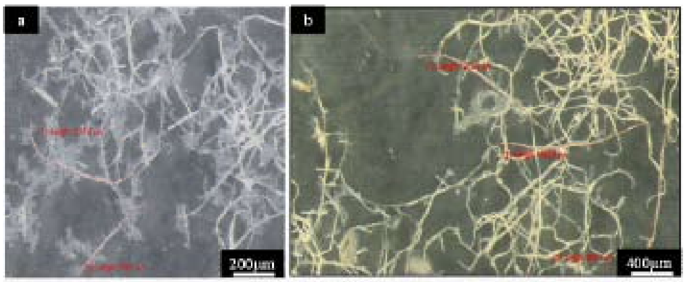

Fig 7:Stereo Microscope image of fibre from LVB (a) and SMC – 0.01 clearance (b) showing clusters of short fibres.

(b) Measurement of Length (L), Diameter (D) and L/D Ratio

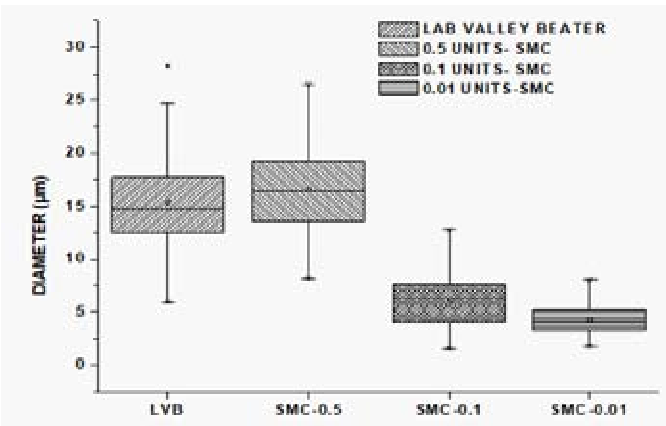

Fig 10:Box-and-whiskers Plot comparing the diameter of the fibres obtained after LVB and SMC.

Fig10 compares the diameter of the fibres from LVB and SMC. The fibres from SMC refining revealed less diameter than LVB, whereas the length of the fibres obtained from SMC is higher when compared to LVB (Fig11). There is a steep decrease in the diameter of the fibres in SMC after 0.5-unit clearance but while comparing the length of the fibres, the decrease is gradual and less. SMC refining was able to maintain the length of the fibres due to the arrangement and angle of the grooves on the grinders(as shown in Fig.2d).

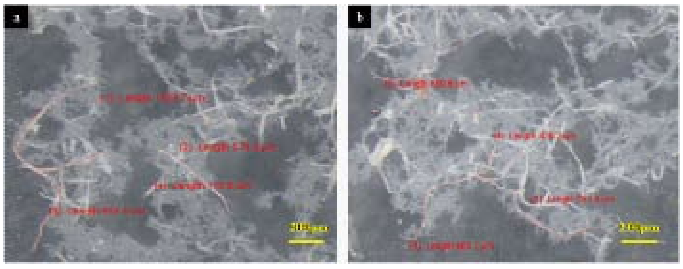

Fig 8:Stereo microscope image of fibres after LVB showing varying lengths of the fibres.

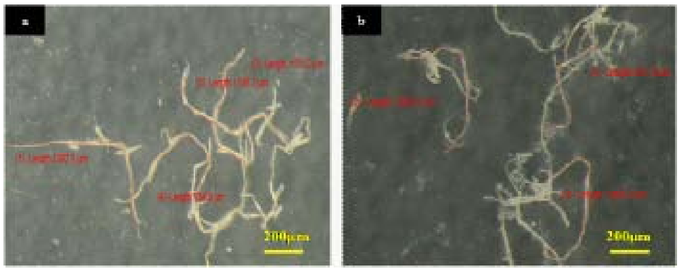

Fig 9: Stereo microscope image of fibres after SMC.
a) after 0.1-unit clearance, b) after 0.01-unit clearance

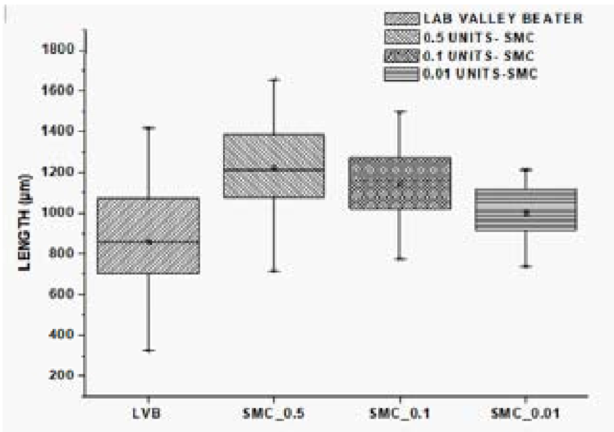

Fig 11:Box-and-whiskers Plot comparing the length of the fibres obtained after LVB and SMC

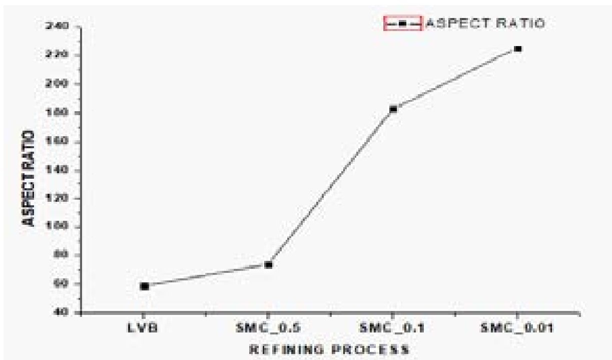

Fig 12:The aspect ratio (L/D)of fibres after LVB and SMC refining processes.

Fig12 compares the aspect ratio of the fibres after refining using Lab Valley Beater (LVB) and Super Masscolloider (SMC). The aspect ratio of the fibres from LVB is very low (58.8). Using a Super Masscolloider, we were able to obtain high aspect ratio as there was not much decrease in the length of the fibres and the diameter was reduced considerably. High aspect ratio is the most important parameter in determining the tensile and tear strength of the sheet and it also facilitates the entanglement of the fibres.

Table 2: The length and diameter of the fibres after refining using LVB and SMC

REFINING	LENGTH (μm)		DIAMETER (μm)	
Lab Valley Beater	868 ± 246	CV: 28.5	14.7 ± 3.1	CV: 21.1
Super Masscolloider (0.5 Units)	1250 ± 194	CV:15.6	16.9 ± 4.4	CV: 26.0
Super Masscolloider (0.1 Units)	1141.5 ± 155	CV: 13.6	6.3 ± 2.2	CV: 34.9
Super Masscolloider (0.01 Units)	1004.78 ± 124	CV: 12.3	4.5 ± 1.7`	CV: 37.8

Table 2 compares the length and diameter of the fibres after the refining processes. The coefficient of variance (CV) depicts huge variability of the data from the mean point. The coefficient of variance for some of the data is greater than 30% i.e fibres with a wide range of diameters is available as we go for less clearance values in Super Masscolloider. This condition is beneficial in paper making as it will enhance inter fibre bonding. This improves the opaqueness of the handsheet which is an important parameter in determining the quality of the nonwoven fabric.

CONCLUSIONS

From the studies it can be concluded that refining instrument and clearance between the upper and lower units in the grinding process greatly affects the morphology of the fibres. Refining using Lab valley beater-LVB with the maximum weight i.e. minimum clearance between the beater roll and beater plate resulted short lengths and higher diameter compared to Super masscolloider-SMC refining which resulted fibres of more lengths with a little compromise on diameter at 0.5 clearance. So it can be concluded that pretreatment can be carried out (20 minutes, 600 rpm) using LVB to reduce the size of the fibres before using SMC, because SMC is energy intensive process. But to obtain micro/nanocellulose fibres with required aspect ratio, further size reduction using 0.5, 0.3, 0.1 and 0.01 clearance values of SMC should be carried out. Due to the presence of grooves in SMC grinders, pulp fibres with high aspect ratio (>200) were obtained which could be beneficial for making high strength nonwoven fabrics filters for capturing particulate matter below 2.5 μm.

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