An approach to produce nano-ligno-cellulose from mechanical pulp fine materials

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KEYWORDS: Mechanical Pulp, Fractionation, Fines, Fibre length-weighted distribution, Nano-ligno-cellulose

SUMMARY: Mechanical pulping, also called high-yield pulping processes, are pulping systems where a great deal of effort is taken with regards to the fractionation in screens and cleaners as well as to optimize process conditions to refine the rejected fractions. The fraction that is rejected for further treatment can vary from 10 to 50% depending on process strategy and final product which can be from printing paper, writing paper, paperboard middle layer and tissue. In practice, it is common that approximately 10% of the pulp fibres and also a large part of the fines fraction have properties that are unsatisfactory in relation to the final products. Part of the less useful fines fraction could instead be used to produce nano-ligno-cellulose (NLC) of high value either in the main product or used for completely different purposes.

In order to study the potential of this concept, treatment of thermo-mechanical pulp (TMP) fines fractions were studied by means of homogenization. It seems possible to homogenize fine particles of thermo-mechanical pulp (1% w/v) to NLC. A corresponding fines fraction from bleached kraft pulp (BKP) was tested as a reference at 0.5% w/v concentration. This fines (BKP) fraction was very difficult to homogenize at a higher concentration (1% w/v). An explanation for this could be that the BKP fines have much higher cellulose content and lower charge level compared to the fines fraction of the hemicellulose and lignin-rich TMP. Fibre length-weighted distribution plays a vital role with respect to both pressure fluctuations and clogging during treatment in the homogenizing equipment.

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Wood pulp is a biodegradable biopolymer and mostly consists of three chemical components; cellulose, hemicelluloses and lignin. Cellulose is the most highly abundant renewable biodegradable biopolymer on earth, it is tough, fibrous and water-insoluble. The molecular framework of cellulose is held together by a repeating unit of 1-4 - β-glucopyranose. The elementary fibrils of cellulose are held together by intermolecular and intramolecular hydrogen bonds (Sjöström 1993; O'Sullivan 1997) which provide the molecule with an inherent high strength property.

It is because of this unique high strength property that many "schools of thought" are looking for environmentally and economically feasible methods in relation to producing microfibrillated cellulose (MFC) or nano-ligno-cellulose (NLC) from diverse sources.

The concept of MFC was initially highlighted by Turbak et al. (1983) and Herrick et al. (1983). However, the development work regarding MFC then accelerated until the late 1990s and was then reignited in the early 2000s and over the past 10-13 years there has been tremendous progress within this research area. MFC could be obtained from several plant and wood sources, as well as bacteria, and this has been elucidated rather well within the reviewed documents of Klemm et al. (2011) and Eichhorn et al. (2010).

The TEMPO (2, 2, 6, 6-tetramethylpiperidine-1-oxyl radical)-mediated oxidized nanofibrillated cellulose has a high aspect ratio (length/diameter) in the range of 110 to 550, i.e. with an average length of 2.2 μm and a diameter of 4 to 20 nm (Ishii et al. 2011) and also with a large specific surface area of about 482 m^2/g (Sehaqui et al. 2011).

Turbak et al. (1983) attested that pre-cut fibres in the length range of 0.6-0.7 mm facilitates fibre treatment in the homogenizer. Alternative techniques that have been routinely used in fibre size reduction include; PFI milling (Henriksson et al. 2007), Escher Wyss refining (Pääkkö et al. 2007), valley beater refining (Spence et al. 2010a; 2010b). It is known that fibre beating enhances disintegration and prevents fibre clogging in relation to the homogenizing equipment (Spence et al. 2011; Iotti et al. 2011; Henriksson et al. 2007; Pääkkö et al. 2007; Andresen et al. 2006; Herrick et al. 1983).

Today, it is possible to produce MFC using a conical refiner with a control gap size of less than 0.1 mm (Björkqvist et al. 2012) and a method has also been patented to produce cellulose nanofilaments using high consistency and/or low intensity refining (Hua et al. 2012). Ankerfors (2012) and Klemm et al. (2011) noticed that the high energy consumption and constant clogging tendency of the homogenizer are the two major obstacles that have proved to be unfavourable for the industrial production of MFC.

Many researchers have been relentless in their efforts to fibrillate fibres into fibrils using chemical (or enzymatic) pre-treatment methods combined with mechanical disintegration (Zimmermann et al. 2006; Saito et al. 2007; Henriksson et al. 2007; Stelte, Sanadi 2009). As reported by Klemm et al. (2011), microfibrillated cellulose has been given a multitude of names including nanofibrillated cellulose (NFC), nanocellulose, nano-fibres, nanofibrils and microfibrils.

Mechanical pulping also called high-yield pulping processes are pulping systems where a great deal of effort is taken with regards to the fractionation in screens and cleaners as well as to optimize process conditions to refine the rejected fractions. The fraction that is rejected for further treatment can vary from 10 to 50% depending

on process strategy and final product which can be from printing paper, writing paper, paperboard middle layer and tissue. In practice, it is common that approximately 10% of the pulp fibres and also a large part of the fines fraction have properties that are unsatisfactory in relation to the final products. Part of the less useful fines fraction could instead be used to produce nano-ligno-cellulose (NLC) of high value either in the main product or used for completely different purposes. In order to study the potential of this concept, treatment of thermo-mechanical pulp (TMP) fines fractions were studied by means of homogenization.

In this article we have designated the name nano-ligno-cellulose (NLC) which of course could be questioned. The reason for the choice of name is motivated by our aim to utilize the knowledge regarding wood polymers related research in the mechanical pulping area. In addition, to understand how to separate fibre materials into micro-fibrils with width lower than 200 nm. More specifically we chose to utilize the ionic polymer and softening aspect of the amorphous wood polymer, hemicellulose and lignin so as to enhance delamination of the fibrillar structure during homogenization. The specific feature with the homogenizer used in this study is the possibility to implement a high temperature treatment of up to approximately 140°C and, by this means, to affect the softening of lignin. It has been reported that both temperature and chemical treatment affect lignin softening (Höglund, Bodin 1976; Norgren 2008; Andersson 2010).

High-yield pulps consist of both fines and fibre materials, the fines are noted for their rather good light scattering properties and thus their high opacity, good formation, improved smoothness, and outstanding printability (Giertz 1973). Fines are seen as particles that pass through a 200-mesh screen of a Bauer-McNett (BMcN) classifier or a Britt Dynamic Drainage Jar (BDDJ) (Seth 2003). It is worth noting that fines consist of a wide range of particles; flakes or lamellae, thread-like bands or fibrils, tori from bordered pits, parenchyma cells and ray cells (Sundberg, Holmbom 2004; Kangas, Kleen 2004; Seth 2003; Mosbye et al. 2002; Westermark, Capretti1988). Seth (2003) highlighted that the most unique properties of fines as opposed to those of fibres is in relation to their the large surface area per unit mass and their higher amounts of anionic charged groups. It has been noted that ray cells have poor bonding properties in pulps (Westermark, Capretti 1988).

This article is part of the "FORE" (forest as a resource) project at Mid Sweden University; the project investigates the means of utilizing low quality fibres in a mechanical pulp and paper mill in order to produce new products and/or improve the product quality of traditional products within these industries (Björkqvist et al. 2010).

The main objective of this paper is to develop means of producing NLC from mechanical pulp fines using fractionation for the purpose of separating coarse and longer fibres from fine particles. BKP is used as the reference pulp. This simple approach could easily be applied on a larger scale and there is a high possibility of integrating such a concept in an already existing mechanical pulp mill thereby reducing business risks and investment costs. In addition, to the best of our knowledge, very few articles have described the equipment used in producing nanocellulose or nano-ligno-cellulose and , in this regard, we have provided an explanation of ; the functionality of our homogenizing equipment at FSCN/Mid Sweden University.

Materials and Methods

Pulps

Thermo-mechanical pulp (TMP) with Canadian Standard Freeness (CSF) value of about 65 ml and dry solid content 34.4% was obtained from the SCA pulp mill at Ortviken, Sundsvall, Sweden.

Bleached kraft pulp (BKP) made from 75% pine and 25% spruce (softwood), ISO brightness of 89.3% and a dry solid content of 30.5% taken from the SCA Östrand pulp mill, Sundsvall, Sweden.

Fractionation techniques

Bauer-McNett Classifier (BMcN): Fractionation of the never-dried TMP was performed using the Bauer-McNett Classifier (BMcN). The following mesh screens were used: 16-mesh, 30-mesh, 50-mesh, 100-mesh and 200-mesh screens. The BMcN classification was performed according to standard.

Britt Dynamic Drainage Jar (BDDJ): In this project we have used a self-designed BDDJ with a capacity of 8 litres. Pulps were disintegrated at 30,000 number of revolutions and diluted using 8 litres of tap water. The diluted pulp suspension was poured onto a BDDJ 30-mesh wire equipped with an agitator (400-450 rpm) fixed at the centre and the valve opened for the filtrate to be collected (flow rate = 1.4 l/min). The fines content (filtrate) is collected by filtration using a filter paper. The amount of fines obtained is then calculated by drying and weighing the sample collected.

Homogenization

GEA Niro Soavi homogenizer (ARIETE, Model: NS2006H, Serial Number: 8755, 2010, Parma-Italy) was used to produce the microcellulose suspension. The homogenizer has a single-stage valve system and the pressure could be adjusted manually using the handwheel. It has a minimum feed pressure in the range of 7-8 bars. The machine was further designed at Mid Sweden University. A new feeding pump (Nemo-pump type NM011BY02S12B, Stockholm, Sweden), heat exchanger (Piovan heat exchanger, Model: TH06, Santa Maria di Sala (VE)-Italy, 2011), piping system (inlet and outlet) and the drainage water pipe were connected to the homogenizing equipment. A thermo-regulator was adapted to the homogenizer and this thermal-regulator uses diathermic oil (Mobiltherm 605) for heating the pipes connected to the homogenizer. The homogenizing system plus the feeding pump is recognized as a closed-loop system (*Fig 1*) with a programmable controller which has a constant feeding pressure of 7 bars.

The main motor power of the homogenizer is 3 kW while the motor power of the feeding pump is 0.75 kW. The flow rate of the suspension in the homogenizer is 1.4 l/min with an estimated 18 passes per trial. The

Fig 1. Homogenizer designed for microcellulose suspension.

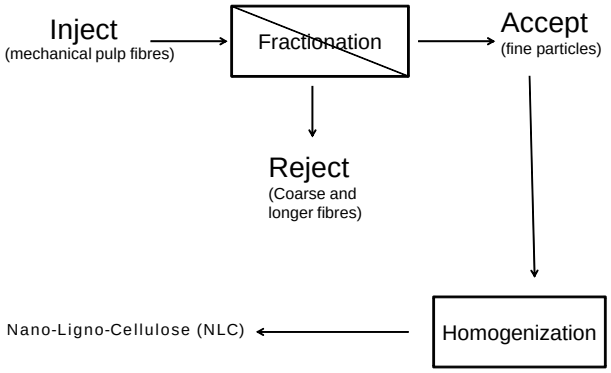

Fig 2. Schematic approach to producing nano-ligno-cellulose.

homogenizing system is made up of a positive-displacement piston pump and a delivery spring-operated valve. Mechanical pulp fine particle suspensions are fed axially into the valve channel and these materials are subjected to harsh treatment in the valve system and the homogenized product is sent through the outlet.

In this paper, the trials have generally been performed at 23^{O}C and at an approximate 1% pulp consistency. However, it has been noticed that there is significant increase in temperature of up to about 50^{O}C during homogenization. When studying the rheological behaviour of TMP-NLC (0.66%, 0.33%, and 0.165%) the samples were homogenized at both 140^{O}C and 23^{O}C. The homogenization pressure was usually set within the 200-300 bars range, but it was sometimes difficult to maintain a constant pressure due to the clogging of the fine particles in the homogenizing system. An average of approximately 5 litres of pulp suspension was used almost in the majority of the investigations. The homogenizing equipment has a maximum working pressure of 600 bars; and the pressure is detected by means of the small manometer on the compression head of the machine (*Fig 1*).

Fig 2 shows a schematic view of our approach to producing nano-ligno-cellulose from mechanical pulp fine materials. A Britt Dynamic Drainage Jar 30-mesh screen and a Bauer McNett Classifier were used as the fractionation equipments.

Fractionation was achieved using BMcN and BDDJ; particles that passed the fractionation equipment are referred to as fines while those retained on the mesh wire are regarded as coarse and longer fibres. The fines are used as feed material in the homogenizing equipment to produce nano-ligno-cellulose.

FibreLab Analyzer

The fibre length-weighted distribution, width and cell wall thickness (CWT) of the fibres and fines fractions were characterized using the Kajaani FiberLab instrument.

Light Microscopy

The micrographs of TMP and BKP fines were recorded on a Leica DMRX Research Microscope using Differential Interference Contrast Technique (DIC). The samples were diluted to very low concentrations and placed on mica substrate for imaging.

Atomic Force Microscopy (AFM)

A Silicon wafer was used as an attachment surface for the NLC. We used p-DADMAC (poly-diallydimethyl-ammonium chloride) as glue to attach the NLC onto the wafer. The wafer was left to dry in air and images of the NLC materials were observed by AFM and SEM. More details of the sample preparation method can be read in Henriksson et al. (2007). The surfaces of the specimens were imaged with a tapping-mode AFM Dimension 3100 (Nanoscope IIIa, VeecoInstruments, Santa-Barbara, CA). A Super sharpened (SS) tip and low frequency were used in the imaging. The height image of the NLC was recorded at 23°C and at 50% relative humidity.

Scanning Electron Microscopy (SEM)

SEM micrographs were taken using ZEISS EVO50 equipped with a Backscattered Electrons (BSE) detector. The working conditions were as follows: acceleration voltage was 15 kV; pressure in the sample chamber was 0.7 mbar and working distance approximately 10 mm. Au/Pd (Gold/Palladium) coating was used for the analyzed samples. Sample preparation was performed as explained in the light microscopy section.

Rheological Properties

The rheological properties of NLC suspensions were characterized on a Physica MCR-300 (Physica Messtechnik GmbH, Stuttgart, Germany) with a concentric-cylinder (CC27) rheometer series adapted to a US 200 software. The concentric cylinder (CC27) has a diameter of 26.67 mm and a concentricity of 1 µm. The flow curves of the TMP-NLC were determined using the following concentration; 0.66%, 0.33%, and 0.165% and the samples were homogenized at both 140^{O}C and 23^{O}C. unless otherwise stated, all the rheological investigations were carried out at 23^{o}C.

Results and Discussion

The TMP was divided into the following sequence; BMcN R16, BMcN R30, BMcN R50, BMcN R100, BMcN R200 and BMcN P200. Table 1 shows the percentages of the different fibre fractions obtained from the TMP BMcN classification.

Table 2 shows the different fractions obtained from the BDDJ fractionation, and in this case both TMP and BKP were used. It is important to stress that our aim is not to compare the two fractionation techniques i.e. BMcN and BDDJ. The former was used for the pre-study and performed according to the standard procedure whereas; the latter was not conducted using the standard procedure. The fibre length-weight distribution graphs can be viewed in *Figs 3a-3j*. Though the fibre length-weighted distribution of the samples were studied by the Kajaani FiberLab instrument, it is also worth noting that the instrument is not suitable for measuring very thin and shorter fibres, i.e. fibres less than 0.2 mm in length. Further, the measurement in *Fig 3e* is not a clear representation of the fibre length- weighted distribution for the TMP-NLC samples because the analyzer is not sensitive for particles less than 0.2 mm and majority of the TMP-NLC samples could be under the insensitive zone (less than 0.2 mm).

A comprehensive knowledge of pulp fractionation is available in Ämmäla (2001). A preliminary study of fibre length-weighted distributions (*Figs 3a-3j*) was conducted using both BMcN and BDDJ Classifiers. After critically analyzing the fibre length-weighted distributions, it is possible to suggest that the fibre length-weighted distribution plays a vital role with respect to pressure fluctuations and clogging during treatment in the homogenizing equipment.

Pulp fractions made up of coarse and longer fibres were not possible to treat in the homogenizer due to fibre clogging and pressure fluctuations (*Figs 3a, 3b, 3f, 3g, 3h and 3i*). BKP-BDDJ30/P (*Fig 3c*) sample was able to pass through the homogenizer without clogging at a very low concentration (less than 0.5%).

Fine fractions from the BMcN R200 mesh screen proved to be easier to homogenize than for those of the BMcN R100. Fibre fractions from the BMcN R16, BMcN R30 and BMcN R50 were considered as both coarse and longer fibre particles and these posed serious clogging problems. The BDDJ fractionation was implemented because it can easily be upscaled, making it simple to obtain a huge quantity of fine materials i.e. using a self- designed BDDJ with a capacity of 8 litres. *Fig 4* shows the fibre length distribution of the TMP – whole pulp (WP), TMP fines from the BDDJ 30-mesh screen, BKP-whole pulp (WP) and BKP fines from BDDJ 30-mesh wire.

In this project, the fine materials are considered to be within the particle length range of 0.00-1.20 mm, and it is evident that TMP-BDDJ30 is the pulp fraction with the highest amount of fine particles (*Fig 4*). It can also be clearly seen in *Fig 4* that BKP-WP contained a very small amount of fine fraction whereas TMP-WP contained a considerable amount of fine particles as compared to the BKP-WP. The results from the fiberlab analysis show that the fibre clogging begins when the fibre length is beyond 1.20 mm.

It seems possible to homogenize fine particles of thermo-mechanical pulp (1% w/v) to NLC. A corresponding fines fraction from bleached kraft pulp (BKP) was tested as a reference at 0.5% w/v concentration. This fines (BKP) fraction was very difficult to homogenize at a higher concentration (1% w/v). An explanation for this could be that the BKP fines have much higher cellulose content and lower charge level compared to the fines fraction of the hemicellulose and lignin-rich TMP.

After performing fractionation and, with the samples taken for homogenization, it was noticed that the fine particles with a fibre length distribution within the range 0.00-1.20 mm (*Fig 3c, 3d* and *3j*) were easier to homogenize and , additionally, posed no serious clogging threats, while clogging was observed for fibres whose lengths were beyond 1.20 mm. The fibre width and cell wall thickness (CWT) were also considered as being factors that could cause clogging in the homogenizing equipment. BKP-BDDJ30 and TMP-BDDJ30 have the narrowest fibre width as shown in table 3 while the BKP-WP has the largest fibre width. The fibre CWT was also relatively even for the BKP-BDDJ30 and TMP-BDDJ30 fractions, as seen in Table 3.

Table 1. Bauer-McNett Classification of TMP

Pulp Fraction	%
BMcN R16	21.3
BMcN R30	19.6
BMcN R50	16.6
BMcN R100	11.9
BMcN R200	6.4
BMcN P200	24.2

10g of pulp sample as feed material
R = retained, P= passed

Table 2. Britt Dynamic Drainage Jar classification of pulps

Pulp fraction	%
BDDJ-30 (P) - TMP	23.3
BDDJ-30 (R) - TMP	76.7
BDDJ-30 (P) - BKP	3
BDDJ-30 (R) - BKP	97

30g of pulp sample as feed material
R = retained, P= passed

Table 3. Fibre width and fibre cell wall thickness (CWT) distribution of whole pulp (WP) and pulp fractions

Pulp type	Fibre width (μm)	Fibre CWT (μm)
TMP - WP	26	5
TMP - BDDJ30	21	4
BKP - WP	29	6
BKP - BDDJ30	20	4

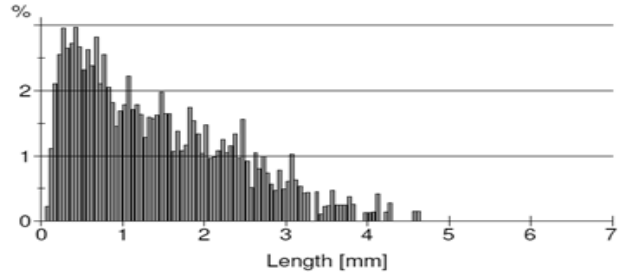

Fig 3a. TMP-whole pulp, sample consists of coarse and longer fibre length distribution.

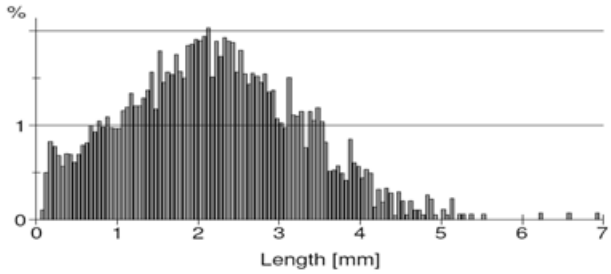

Fig 3b. BKP-whole pulp sample is also made up of coarse and longer fibres.

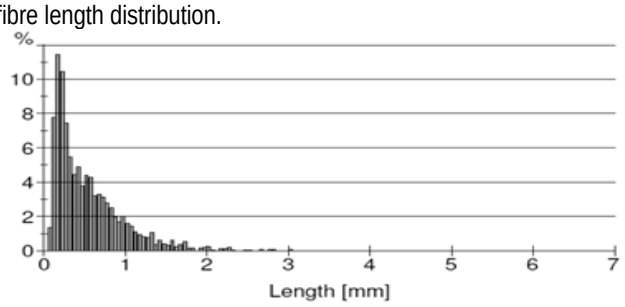

Fig 3c. BKP-BDDJ30/P. This sample shows fibres that passed through the 30 mesh screen of a BDDJ.

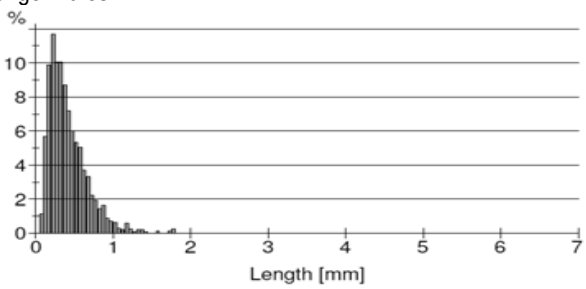

Fig 3d. TMP-BDDJ30/P consists of shorter fibre fractions and can be homogenized without clogging.

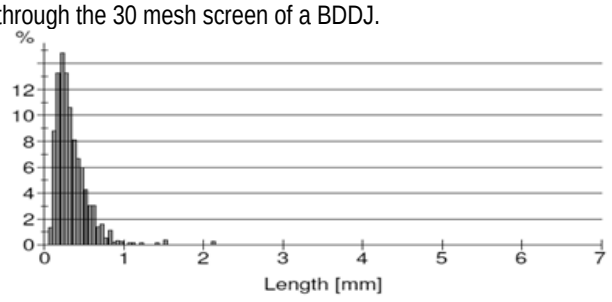

Fig 3e. TMP-NLC.The sample was homogenized at room temperature with approximately 18 passes, at 1% concentration. The homogenizing pressure was in the range 200-300 bars.

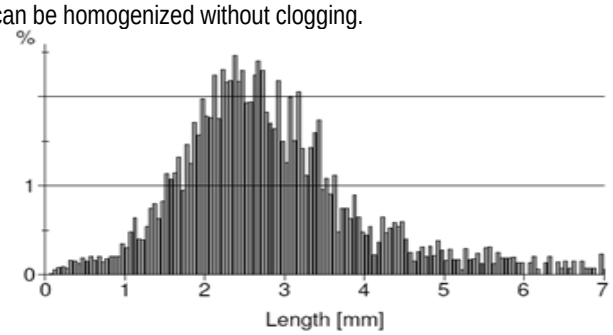

Fig 3f. TMP-BMcN R16, sample consists of coarse and longer fibres, and cannot be treated directly in the homogenizer due to fibre clogging and pressure fluctuations.

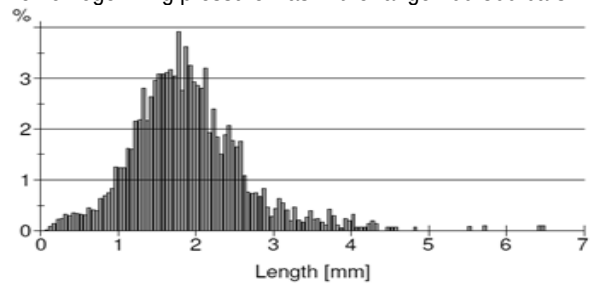

Fig 3g. TMP-BMcN R30, sample consists of coarse and longer fibres.

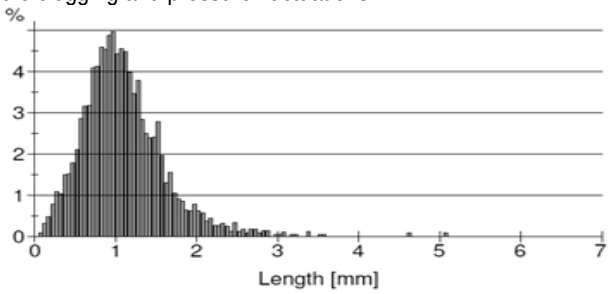

Fig 3h. TMP-BMcN R50. This sample is made up of longer fibre fractions.

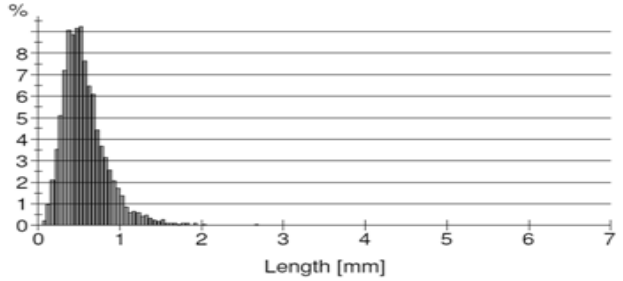

Fig 3i. TMP-BMcN R100 This sample is made up of thin and short fibre fractions.

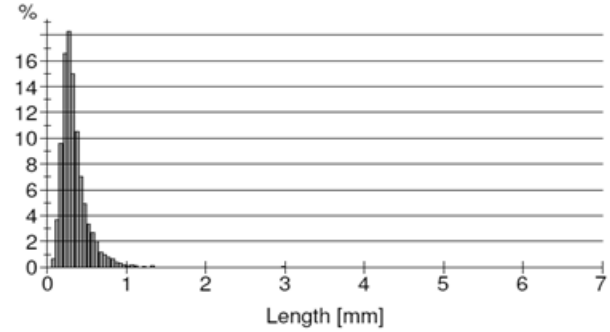

Fig 3j. TMP-BMcN R200. This sample is made up of very thin and short fibre fractions.

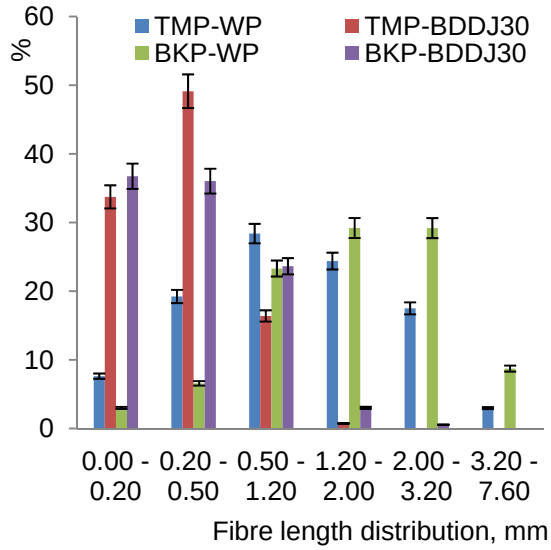

Fig 4. Fibre length distribution of TMP and BKP.

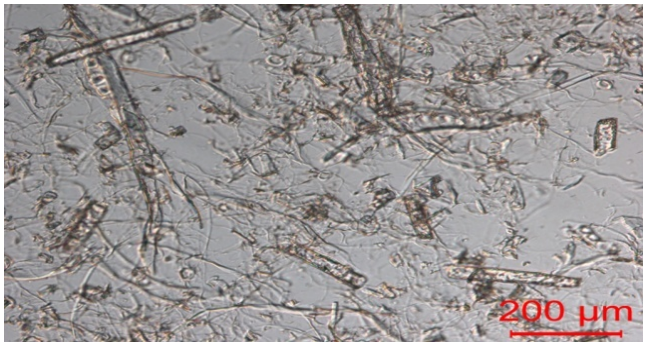

Fig 5. Light microscopy image of TMP fines using BDDJ-30. (The image indicates broken parenchyma cells and fibrillar-fines).

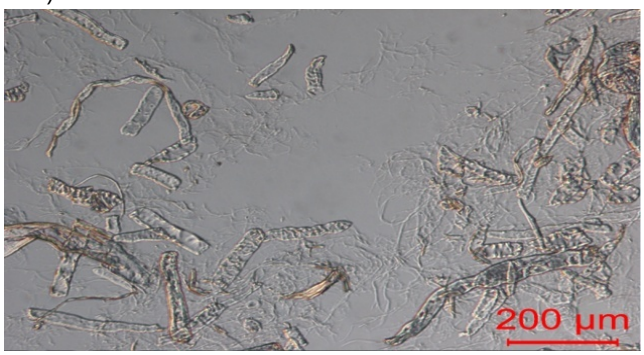

Fig 6. Light microscopy image of BKP fines using BDDJ-30 (The image reveals unchattered rectangular-like ray cells and ribbon-like fine

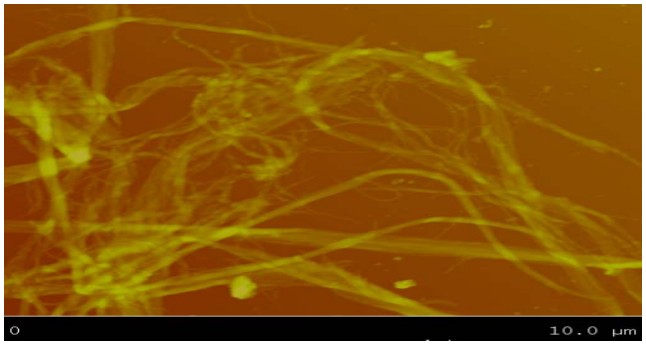

Fig 7. AFM image of TMP-NLC (height image).

Fine particle micrographs of TMP and BKP are seen in *Fig 5 and 6*. Similar studies of fines from TMP and kraft pulp have been reported by Krogerus et al. (2002). In addition, micrographs of ray cells and parenchyma cells could be seen on Westermark and Capretti (1988) paper. The TMP-NLC sample was scanned in tapping mode at a scan rate of 0.502Hz using a super-sharpened (SS) tip. It is observed that the fibrils are bound together as a fibril bundle, *Fig 7*, and it was hypothesized that this might be due to the presence of hemicellulose and lignin. Zimmermann et al. (2006) have also reported images of MFC using AFM. The fibril diameter of the TMP-NLC sample after homogenization is approximately 80-115 nm (*Fig 7*).

The images in *Fig 8* and *9* show the fibrillar structures of the TMP-fines and TMP-NLC respectively. The SEM micrograph in *Fig 9* shows how the fines in *Fig 8* have been delaminated into smaller particles after homogenization. The fibril diameter of TMP-NLC as viewed on AFM is in the range of 80-115 nm. One of the difficulties faced when using thermo-mechanical pulp fines in producing nanocellulose or NLC is that the fine particles contain a high amount of lignin, which acts as a gluing substance to the cellulose fibrils, thus preventing the individualization of the fines into fibrils (*Fig 7*). The SEM image in *Fig 8* reveals the fibril dimension before homogenization, with fibril diameter of approximately 19 µm. The SEM micrograph also reveals the fibril dimension after homogenization, with fibril diameter of approximately 110 nm (*Fig 9*).

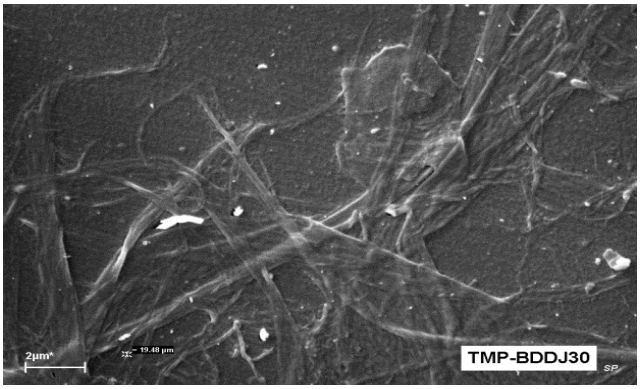

Fig 8. SEM micrograph of TMP fine particles obtained from BDDJ 30-mesh screen.

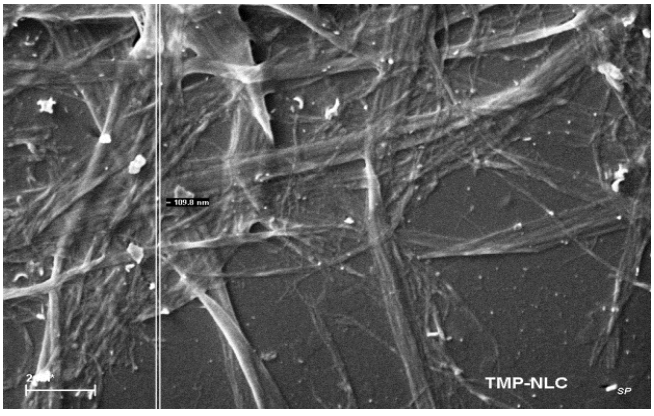

Fig 9. SEM Micrograph of TMP-NLC particles obtained after homogenization (140^{O}C).

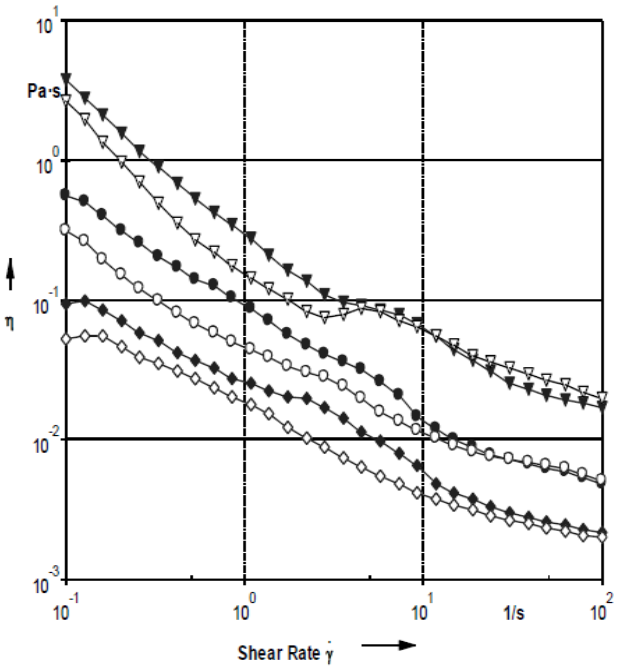

Fig 10. Rheological properties of TMP-NLC (0.66% TMP-NLC at 23°C, 0.66% TMP-NLC at 140°C 0.33% TMP-NLC at 23°C, 0.33% TMP-NLC at 140°C 0.165% TMP-NLC at 23°C, 0.165% TMP-NLC at 140°C).

When studying the rheological behaviour of TMP-NLC (0.66%, 0.33%, and 0.165%) the samples were homogenized at both 23°C and 140°C. From the flow curve presented in *Fig 10*, it is observed that the shear viscosity (Pa.s) of all the suspensions decreases with an increasing shear rate (s^{-1}), thus the TMP-NLC exhibits a shear thinning behaviour at a concentration as low as 0.165%. Tatsumi et al. (2002) highlighted that fibre suspensions at a low concentration above 0.3% concentration displayed a non-Newtonian behaviour meanwhile Xu et al. (2009) observed a shear-thinning behaviour for galactoglucomannans at a concentration higher than 0.5%. The flow curve in *Fig 10* shows the shear rate range between 0.1 and 100 s^{-1}.

The flow curves in *Fig 10* shows concentration (0.66%, 0.33% and 0.165%) dependency of TMP-NLC suspension homogenized at both 23°C and 140°C ; with the 0.66% TMP-NLC at 23°C having a highest viscosity. TMP-NLC suspensions (0.66%, 0.33% and 0.165%) homogenized at both 23°C and 140°C vary with the low shear rate. The curve in *Fig 10* is quite similar to that of Pääkkö et al. (2007) at 0.66% and 0.33% concentrations. However, their concentrations were 0.5% and at 0.25% and , in fact, our results have a similar appearance. The major difference between our investigation and that of Pääkkö et al. (2007) is that their experiment was run at a higher shear rate of between 0 and 1000 s^{-1} which has been expanded to a greater extent for industrial applications. Pääkkö et al. (2007) also studied the dynamic rheological behaviour of MFC in a concentration range of 0.125-5.9% w/w, with G' in the range of 1.5 Pa to 10^5 Pa. It can be viewed in *Fig 10* that the viscosity of the TMP-NLC samples homogenized at 23°C and 140°C are somewhat similar and the slight difference that does exist between the two samples at shear rate 0.1 to 100 s^{-1} is unable to be explained, maybe it might be due slight difference in consistency in both samples. The shear viscosity decreases with an increasing shear rate for the different TMP-NLC concentrations.

Conclusions

After carefully fractionating the various pulp fibres and after the fines had been further treated in the homogenizing equipment, the following conclusions were drawn;

- Fine fractions from mechanical pulp (TMP) could be homogenized to NLC.
- We could also fibrillate fines materials from BKP (c.a. 0.5% w/v), but it was difficult to homogenize BKP at a higher concentration (c.a. 1% w/v).
- Fibre length-weighted distribution plays a key role with respect to pressure fluctuations and plugging in the homogenizer.
- AFM image and SEM micrograph revealed the fibrillar structure of NLC.
- TMP-NLC exhibit shear-thinning behaviour.

Future work

In the next phase of our investigation, the intention is to verify the difference between the final products i.e. TMP-NLC and BKP-NLC by evaluating the mechanical properties (z-strength, tensile index, tear index and burst index) of Rapid Köthen handsheets blended with nano-ligno-cellulose.

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