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Consolidation and dewatering of a microfibrillated cellulose fiber composite paper in wet pressing

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ABSTRACT

The emerging research field of bio-based nanomaterials has gained a lot of interest recently. One of the most promising new range of materials are the micro and nanofibrillated celluloses, or nanocelluloses that are based on wood or other natural cellulose sources. While the strength increasing potential of nanocelluloses is evident in light of the current knowledge, the related challenges in dewatering are inevitable due to the hydrophilic nature and the large relative surface area of the material. The target of this work was to characterize the dewatering and structural changes of a high filler content (70 wt-% precipitated calcium carbonate) biocomposite containing microfibrillated cellulose in a wet pressing process. Softwood bleached kraft pulp fibers were used as a reinforcement in the composite. Press dewatering performance together with dynamic density measurements were made with a press simulator, and scanning electron microscopy and mercury intrusion porosimetry were used in the structural analysis of the samples. The dewatering of a new type of MFC based composite was shown to be better than traditional softwood based fibers. The high amount of filler in the structure is not contributing to the binding of water and is probably able to provide channels for water removal. Examining the pore structure and distribution of the composite and comparing those with SBKP pulp fibers showed that although the pore structures are completely different, efficient dewatering is possible through flow channels that remain in the MFC composite structure during pressing. Based on the results, it can be concluded that the dewatering of the MFC composite is not limited by the wet pressing operation commonly used in the paper manufacturing industry. Excellent optical properties together with potential ecological and cost savings promote the use of this type of novel composite in future applications.

KEYWORDS: nanocellulose, wet pressing, paper making, dewatering, consolidation, calcium carbonate.

1 Introduction

Cellulose is a polysaccharide consisting of β(1→4) linked D-glucose units with a large range of industrial uses. Natural cellulosic fibers form the back-bone of the paper and packaging industry and, thus, are an extremely important source of sustainable raw material. Cellulosic fibers are built in a hierarchical fashion from cellulose fibrils, with a width range of 3-5 nm. Therefore, cellulose pulp fibers are an excellent source of renewable nanomaterials. In the current generation of natural fiber products, fibers with cross-dimensions of roughly 10-100 µm dominate. However, a growing consensus sees the next generation of fiber products, cellulose nanofibers in the size range of 3-100 nm cross-section, as having a very important role in future natural fiber products. Cellulose nanofibers, commonly known as micro and nanofibrillated celluloses (MFC and NFC) are fibrils or fibril aggregates producible by several different mechanical or chemi-mechanical processes. The first method to produce MFC by mechanically disintegrating pulp fibers was published 30 years ago by Turbak et al. [1]. Since then, various mechanical and chemical treatments have been proposed to produce nanocellulose. These treatments have been described thoroughly in the recent review papers together with the potential uses of nanocelluloses in various applications and composite materials [2-4].

One of the most important uses of cellulose pulp fibers is in paper and board products. Paper is produced by mixing pulp fibers together with other additives, such as pigments, then forming and dewatering a web on a paper machine. Paper machines have evolved over hundreds of years to reach a high level of sophistication. A typical modern paper machine today may have a width over 10 meters, speeds approach 2000 m/min and a production of several hundred thousand tons per year.

Web based products, where nanocellulose composes the major structural component, have many potential advantages over traditional paper products. Because nanofibers are notably smaller than typical pulp fibers, excellent surface- and optical properties can be achieved [5, 6]. Since the surface area is large, the bonding efficiency of the nanofibers leads to good strength properties [7-13]. While numerous laboratory studies have explored the properties of nanocellulose papers and composite materials, comparatively slight attention has been focused on feasible, large scale manufacturing routes.

The manufacture of nanocellulose-based papers faces certain challenges compared to the manufacture of traditional papers. One of the main issues is that the water removal from a web containing nanocellulose is hindered by the relatively large surface area and swelling of the material. While studies have shown water retention values for kraft pulp fibers to be approximately 2 g/g, the water retention value of NFC/MFC produced from the same pulp can be as high as 30 g/g [14]. In order to develop a sensible dewatering strategy, both the furnish composition and chemistry as well as the dewatering processes must be examined.

In ordinary papermaking operations, water is first removed from between the fibers by vacuum, then squeezed out mechanically in wet pressing, and the final water is removed with heat. Since drying is an energy intensive and expensive unit operation, it is important to remove as much of the water as possible by mechanical means. This is also the case for nanocellulose based papers. Large scale, cost effective manufacturing demands that a significant amount of water can be removed from the web by mechanical means. Taipale et al. [15] have shown that selecting optimal retention system would enable the use of nanocelluloses without a significant decrease in drainage. Low amounts of nanocelluloses could also be used in the furnish for low grammage sheets without impairing the dewatering in wet pressing [16]. Furthermore, Hii et al. [17] have

concluded that the optimal use of microfibrillated cellulose and filler could enhance both strength and optical properties without reducing the solids content after wet pressing.

In the present study, the removal of water from a nanocellulose web is examined. The furnish consists of 20% microfibrillated cellulose (MFC), 70% precipitated calcium carbonate (PCC) and 10% reinforcement fiber. In an earlier study, we found that this type of furnish yields desirable physical properties [5], and due to the relatively low price of the PCC, could lead to cost savings in the furnish raw material base. In the present study, our target was to determine whether it is possible to efficiently remove water in wet pressing and to examine the consolidation of the composite paper web compared to the traditional fiber furnish.

2 Materials and methods

2.1 Raw materials

The pulp fibers in the composite furnish were commercially produced softwood bleached kraft pulp (SBKP) which was delivered in the dry form. The SBKP was lightly refined in a conical refiner to a SR^{o} = 18. The length weighted average fiber length of the pulp was 2.24 mm and SR° = 18. MFC was a commercial grade Daicel Celish KY-100G delivered at 10 wt-% solids. Its viscosity at 1.5 wt-% and 10 RPM was $16.1 \cdot 10^{-3}$ Pa·s measured with Brookfield RVDV-II viscometer using a vane spindle V73. PCC (grade FS240) was delivered by Omya AG at 35 wt-%. It was of scalenohedral shape and its weighted mean particle size was 3.97 µm measured with particle size analyzer (Malvern Mastersizer 2000) using a general purpose model. The particle swelling of the raw materials was measured with a solute exclusion method [18, 19].

2.2 Sample preparation

2.2.1 Mixing of components

SBPK fibers and MFC were diluted with deionized water to 1 wt-% prior to mixing. A laboratory mixer (Diaf) was used in the dilution of pulp and a high shear disintegrator in the dilution of MFC in order to achieve a homogeneous suspension. SBPK fibers and MFC were then mixed together, and after adding the PCC, the suspension was mixed and diluted to 0.26 wt-% for sample preparation.

2.2.2 Preparation of SBKP and MFC composite samples

The proportion of the materials in the MFC composite sheets based on dry weight was 10% SBPK, 20% MFC and 70% PCC. Pure SBPK fiber sheets were formed according to standard SCAN-CM 64:00. A modified laboratory sheet forming device was used in the MFC composite preparation. A 50 kPa overpressure was applied for 210 s and a nylon membrane (Sefar Nitex 03-10/2, Sefar AG, Switzerland) was used on top of the standard wire for retaining fine MFC and PCC particles [5]. All samples were adjusted to a moisture ratio of 4 g water / g dry after forming, and 4 circular samples were cut from each sheet for further experiments. For structural analysis before and after pressing, samples were freeze dried at -50 °C and 2.4 Pa. The time span from pressing to sample freezing was 15 s for all samples. Other samples were dried between blotting papers in 570 kPa pressure at 130 °C temperature for 2 min.

2.3 Wet pressing experiments

2.3.1 Press simulator

A universal material testing system (MTS 810) modified for press dewatering studies was used in the experiments. This system consisted of a smooth stainless steel top plate operated with a hydraulic piston and a sintered stainless steel lower plate which enabled water to flow from the

sample. A vacuum of 60 kPa below atmospheric pressure was applied to the dewatering chamber. After the press pulse the sample adhered to the polished top plate. The development of the MTS is described in detail by Saukko [20]. The samples were pressed at varying peak pressures of 2 – 10 MPa with a total duration of 50 ms resulting in press impulses of 50.7 – 250 kPa·s. In modern paper machines, this is comparable to a press nip of 300 mm in length with a production speed of 360 m/min.

2.3.2 Wet pressing efficiency and rewetting

Sample dry weight (m_{dry}) and weight after pressing (m_{out}) were used to calculate the moisture ratio after pressing, $MR_{out} = (m_{out} - m_{dry}) / m_{dry}$. Another characteristic measurement was the minimum moisture ratio during the press impulse, which describes the In order to calculate the minimum moisture ratio during the press pulse, an accurate 3 – point eddy current measurement was utilized to measure the minimum thickness of the sample at compression (b). Using the sample area ($A = 4.54 \cdot 10^{-3}\ m^2$), densities of water ($\rho_{H2O} = 1.0 \cdot 10^6\ g / m^3$), cellulose ($\rho_c = 1.55 \cdot 10^6\ g / m^3$) and PCC ($\rho_{PCC} = 2.71 \cdot 10^6\ g / m^3$), the minimum moisture ratio can be calculated:

$$MR_{min} = \frac{\left[b \cdot A - \frac{m_{dry}}{(0.3 \cdot \rho_C + 0.7 \cdot \rho_{PCC})}\right] \cdot \rho_{H_2O}}{m_{dry}} = \frac{\left[4.54 \cdot 10^{-3} \cdot b - \frac{m_{dry}}{(2.36 \cdot 10^6)}\right] \cdot 10^6}{m_{dry}} \quad (1)$$

The amount of rewetted water (nip rewetting) was calculated as $MR_{NIP} = MR_{out} - MR_{min}$. In the post-nip rewetting tests the peak pressure was first set to 10 MPa and then decreased to 0.4 MPa and held constant for 250 ms. After a total contact time of 300 ms, the top plate was separated from the lower plate together with the sample. Potential increase in the moisture ratio caused by the post-nip rewetting was calculated from moisture ratio after 300 ms contact (MR_{300}) and moisture ratio after 50 ms pulse (MR_{out}), $MR_{PN} = MR_{300} - MR_{out}$. From the wet pressing process

point of view this describes the potential increase in moisture ratio if the contact between the press felt and dewatered material would continue for 250 ms after the actual 50 ms press impulse.

2.3.3 Calibration of the thickness measurement

To be able to accurately measure the sample thickness during pressing, we first used aluminum plates of 40 – 100 µm thickness for calibration. During pressing, the MFC composite furnish material partly penetrated into the sintered bottom plate. This factor was accounted for by measuring the surface topography of the freeze-dried sample that contained an imprint of the sintered plate. From this topography map, an average thickness based on the penetrated volume was estimated mathematically. The sample penetration was not an issue for the SBKP fibers because the fibers are much larger than the pores in the sintered plate.

2.4 Sample characterization

2.4.1 Density measurement

Calculating the density of the samples outside the press simulator was performed by manually measuring the sample thickness. Due to the high compressibility of the samples, two aluminum plates with known thickness (b_{Al}) were placed on each side of a 4 sample stack. A micrometer screw was then used to measure the thickness of the stack (b_{st}) from 4 different points. After weighing the stack of samples (m_s), density was calculated, $\rho = [m_s / (b_{st} - b_{Al}) \cdot A]$.

2.4.2 Mercury intrusion porosimetry

Wet samples from various stages in the dewatering process were freeze dried by immersing them in liquid nitrogen, followed by vacuum drying. This allows one to produce dried samples, suitable for porosimetry measurements, which resemble the structure of the sample in the water saturated state. Pore size distribution (PSD) and sample apparent density were measured with mercury intrusion porosimetry at a 400 MPa maximum pressure.

2.4.3 Scanning electron microscopy

A field emission scanning electron microscope (FE-SEM, Zeiss Sigma) was used in characterizing the morphology of the samples. Prior to their characterization, the samples were sputtered with a gold layer and the acceleration voltage of the microscope was set to 2.5 kV.

2.4.4 Composite paper properties

Tensile strength and Young's modulus of the samples were measured with the MTS – 400/M testing system equipped with a 50 N load cell. The gap length was set to 50 mm and the elongation rate to 12 mm/min. The optical properties were measured with the L&W Elrepho SE 070R Spectrophotometer at 395 nm wavelength.

3 Results and discussion

3.1 Wet pressing water removal and rewetting

The moisture ratio of the sheet as a function of maximum pressure is shown in Figure 1 for the MFC composite sheet compared to a SBKP paper reference. As the figure shows, the press dewatering of MFC composite was excellent. The after-press moisture ratio was approximately 0.1 ml/g lower across the tested peak pressure range when compared with the pure SBKP fibers. At first, the excellent press dewatering of the composite sample seems surprising, since it is a common experience that MFC is extremely hydrophilic and will impede dewatering when added to papermaking furnishes. However, there are some aspects of the composite furnish that help to promote good dewatering. For one thing, the composite furnish is expected to have a larger fraction of interparticle water than the fiber furnish. Wahlström has previously demonstrated that the water fraction inside the fiber wall is a limiting factor in press dewatering [21]. This is because the pores in the cell wall are small, (1-30 nm in diameter) and thus restrict water removal rate.

The swelling of the MFC was 0.6, SBKP 1.1, and PCC 0.0 ml water/g solids measured by solute exclusion. Based on the material proportions, the total intrafiber water for the composite sample was 0.23 ml/g, while for the SBKP sample it was 1.1 ml/g. In other words, the high fraction of non-swelling PCC facilitates good press dewatering. Another important factor in press dewatering is that the permeability of the sample, particularly on the exit layer, must be maintained [22]. Microscopic analysis of the freeze dried samples after pressing showed pores in the exit layer in the range of 1-5 µm (see Figure 2c), which is sufficient for good press dewatering. Clearly, the conditions that lead to good press dewatering in high MFC content composite papers must be studied in more detail. However, this early evidence that effective press dewatering is possible under some conditions is encouraging and indicates industrial production of such papers is not only feasible, but could be even more efficient than traditional paper products.

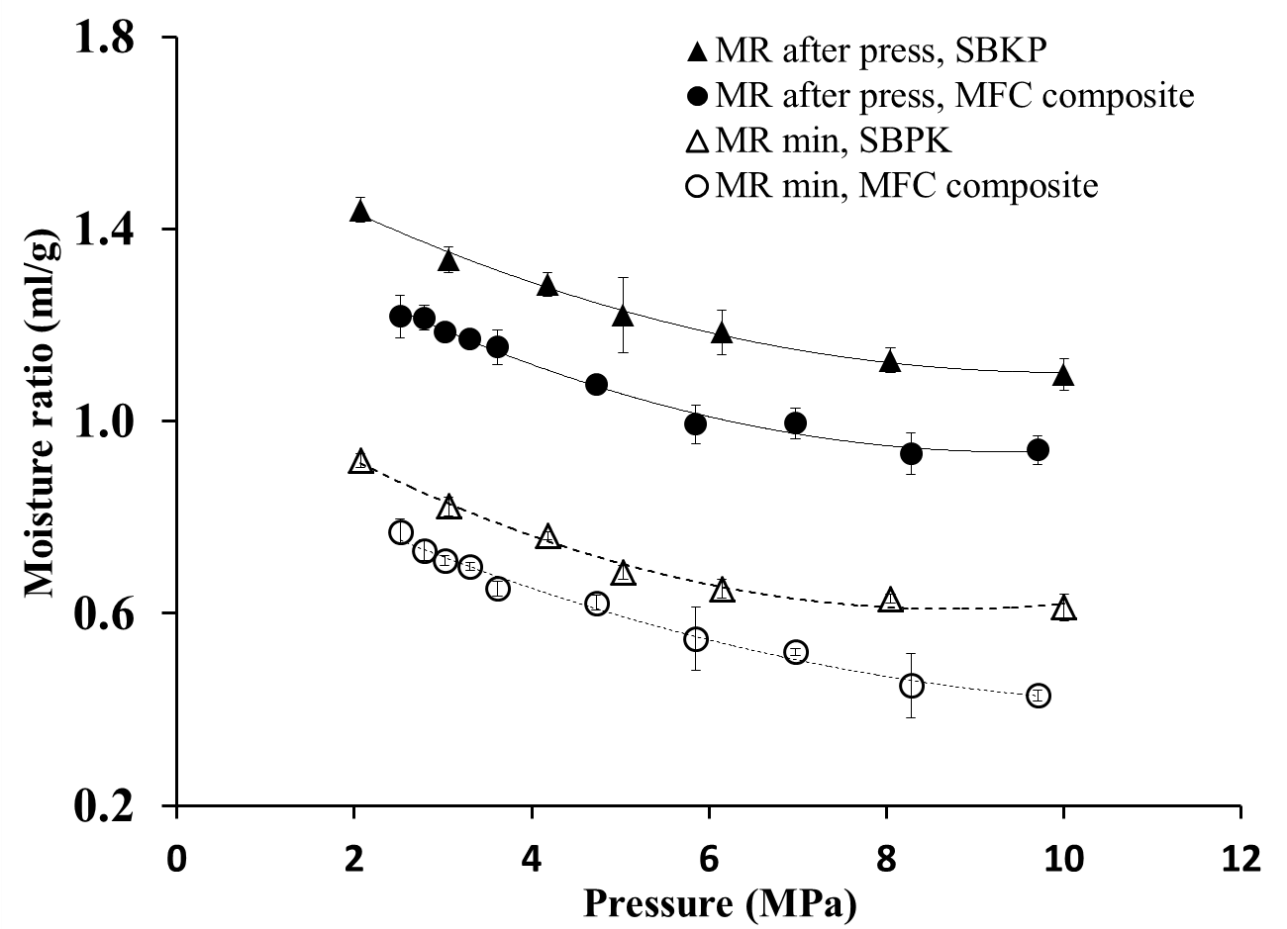

Figure 1.The effect of peak pressure on the moisture of samples after pressing and during maximum compression of the samples. Initial moisture ratio was 4 ml/g. Error bars represent 95% confidence intervals.

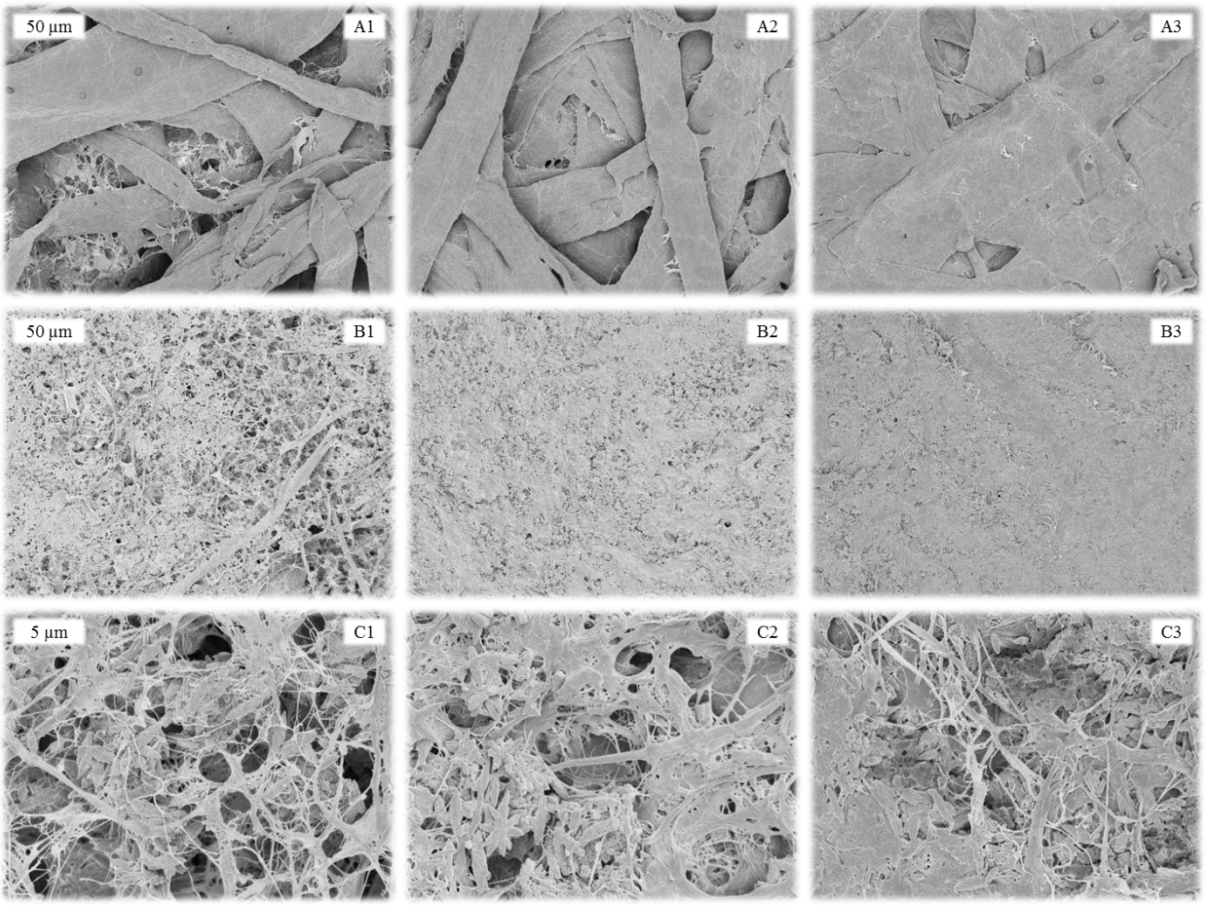

Figure 2. Development of surface morphology of the SBKP fibers (A) and MFC composite (B) before pressing (1), after pressing at 10 MPa (2) and after press drying at 0.57 MPa and 130 °C (3). Higher magnification images of the MFC composite surfaces are presented in C1-C3 which show the packing of PCC and MFC during consolidation.

The minimum MR of the MFC composite during pressing was as low as 0.43 ml/g dry compared with 0.61 ml/g dry of the minimum MR of SBKP fibers. The difference between the minimum MR and the MR after pressing, considered here as the nip rewetting, was approximately the same for both samples across the pressure range (0.51 ml/g for MFC composite and 0.48 ml/g for SBKP fibers). The post nip rewetting showed a slight increase potential in the moisture ratio after wet pressing at 10 MPa (0.16 ml/g for MFC composite and 0.19 ml/g for SBKP fibers). These observations imply that preventing rewetting in wet pressing would result in considerable improvement in dewatering efficiency. Similar observations on nip rewetting were made in an

earlier study performed with low amounts of nano- and microfibrillated celluloses mixed with fibers [16].

3.2 Consolidation and pore size distribution

The consolidation of web throughout the water removal process was examined by measuring the apparent density and the porosity of the freeze dried sheets. After press drying, the MFC composite shows a lower apparent density than the SBKP fibers, whereas in all other consolidation phases (Figure 3) the situation is the opposite. Calculating the porosity of the samples from the mercury porosimetry data shows that the dry MFC composite has a porosity of 0.63 compared to the porosity of 0.36 of the dry SBKP fibers (Figure 4). The porosity of both samples was similar in all other consolidation stages. This implies that the MFC composite has distinctive characteristics in consolidation of the structure during drying. While the SBKP fibers collapse as a result of the drying process, the MFC-filler structure is able to sustain much of its volume.

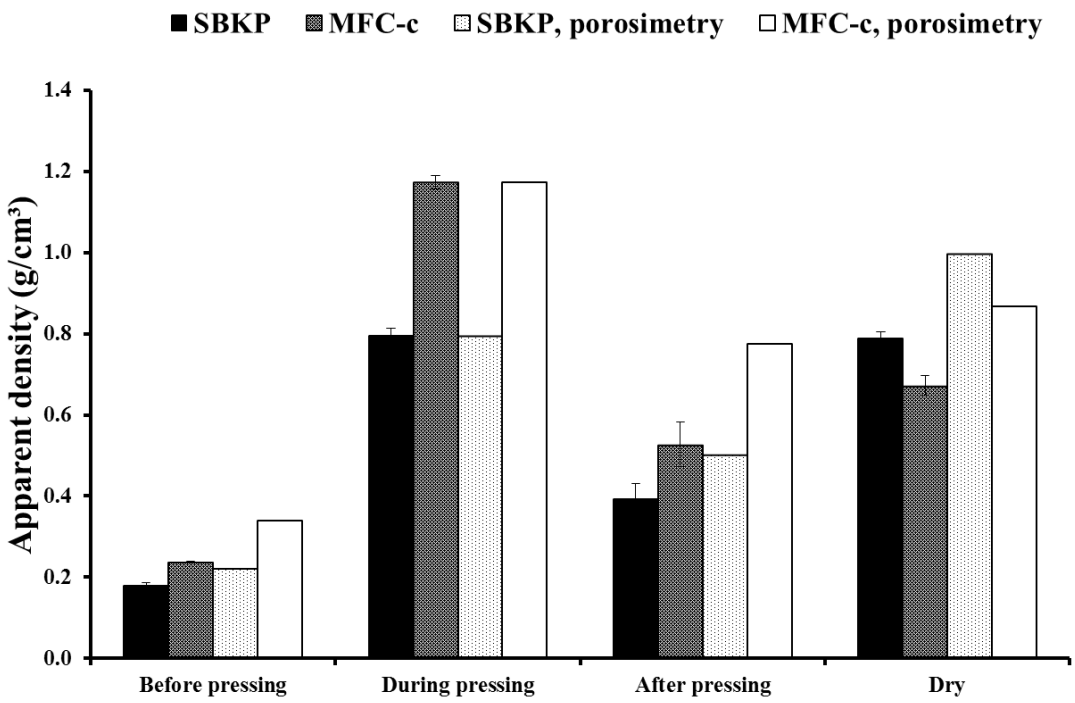

Figure 3. Apparent density (bulk density excluding water) of the samples measured before wet pressing, during the maximum compression in wet pressing at 10 MPa, after wet pressing and after press drying at 0.57 MPa and 130°C. The results are from

physical (dark columns) and from mercury intrusion porosimetry measurements (light columns), except for the density during pressing, which is based on the sheet thickness during the press pulse. Error bars represent 95% confidence intervals.

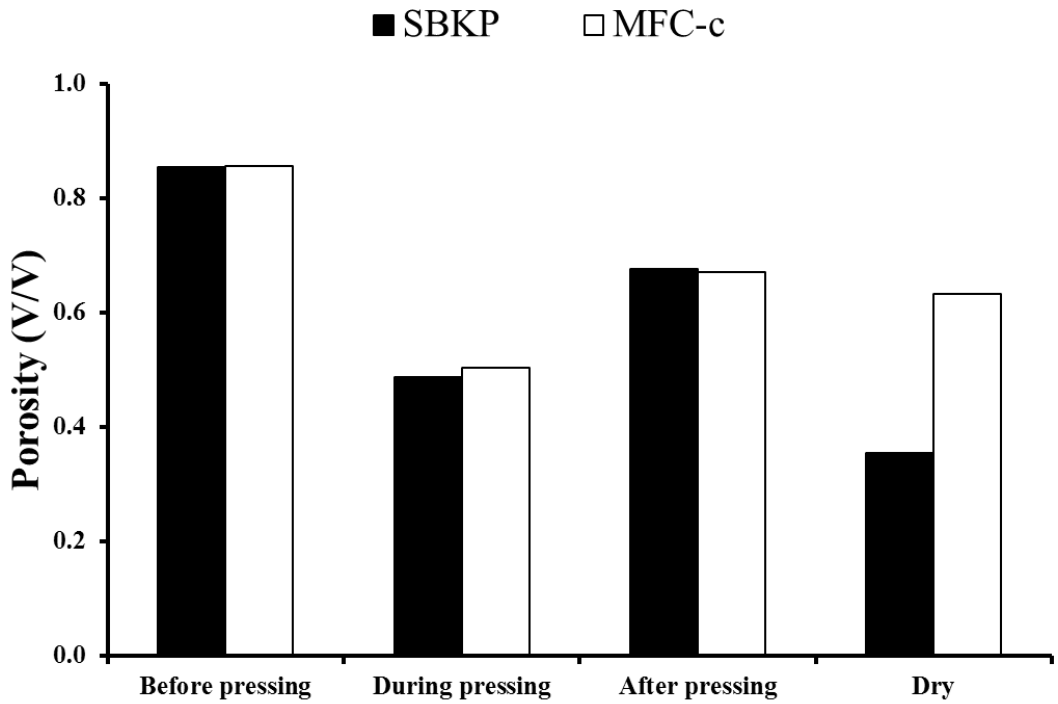

Figure 4. The volumetric porosity of the samples during different consolidation stages measured by mercury intrusion porosimetry, except for porosity during pressing which was based on the thickness during press pulse.

The freeze drying procedure is a useful method to measure the interparticle pores, which is the focus of this study. The small pores within the cellulose samples are not preserved in freeze drying. As the dewatering proceeds in wet pressing, the structure consolidates and the water filled pores are closed. Figure 5 shows the cumulative pore volume of the samples before and after pressing at 10 MPa and after press drying. In the MFC composite, most of the water is distributed in pores smaller than 10 µm in diameter before wet pressing, while in the SBKP fiber network most of the water is located in pores larger than 10 µm in diameter. This is also visualized in the surface morphology SEM-images in Figure 2 (A1 vs B1) which clearly show the difference of the materials. After wet pressing, more water seems to be located in the MFC composite structure, in the pore volume diameter range of 0.1 – 1 µm, than before pressing. This suggests

that the PCC particles form an open structure with voids between them where most of the water is held after pressing (see C2 in Figure 1).

In the MFC composite sample the larger pores are collapsed in wet pressing, reducing the pore volume and also slightly increasing the fraction of small pores under 1 µm. In press drying, the pore size distribution is almost unchanged. This supports our view that for the MFC composite paper, the structured PCC packs to a threshold density which is structurally resilient and resists even the significant consolidation forces imposed by press drying. The fact that the MFC composite furnish resists pore collapse in the later stages of water removal is a significant advantage compared to traditional papers. One reason is that the open structure helps maintain a high evaporation rate and good drying efficiency. Another factor is that the density of the substrate is maintained at a relatively low level, which helps reduce the cost structure of the product.

This contrasts sharply with the consolidation mechanisms of the fiber web sample. In Figure 5b it is observed that after the collapse of the large pores in wet pressing (in this case pores mostly over 10 µm), the sample experiences significant collapse of pores in press drying. For cellulosic materials, the shrinkage and pore collapse accelerates in the later stages of water removal as the bound water from the cellulose surfaces is removed. Enormous surface tension forces, together with a low modulus for wet cellulose, mean that papers containing a large fraction of pulp fibers experience a great deal of shrinkage in water removal. A good review of the pore collapse and consolidation of pulp fibers is given by Weise [23].

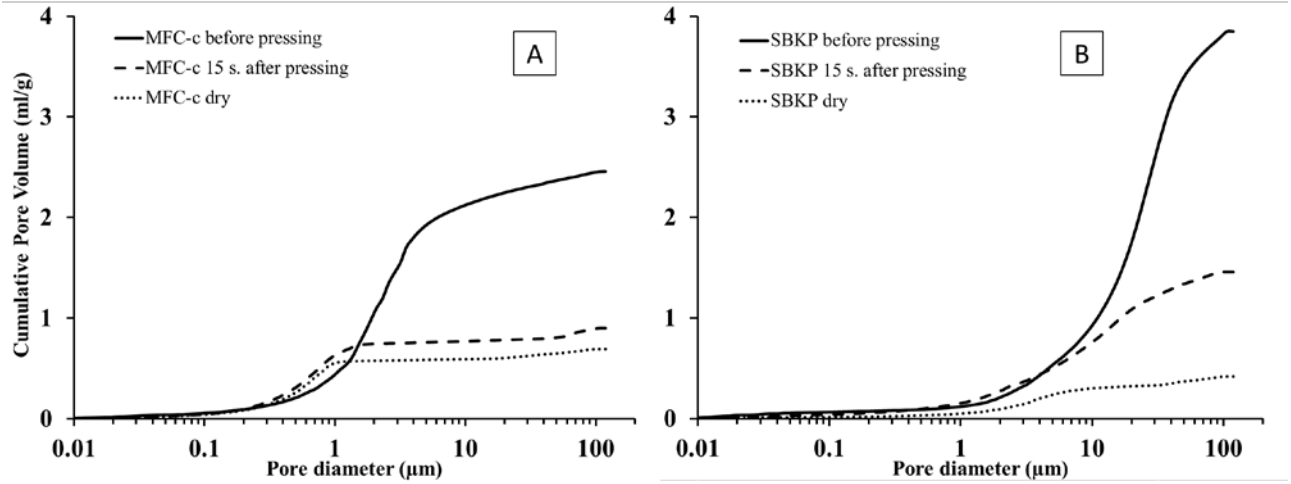

Figure 5. Cumulative pore volumes of the MFC composite (A) and SBKP fibers (B) measured before pressing, 15 s after pressing at 10 MPa and after press drying at 0.57 MPa and 130 °C. Samples taken before and after pressing were freeze dried, thus the pore distributions also correlate with the location of water in the structure.

3.3 Material properties

The physical properties of the composite sample compared to the SBKP fibers are compared in Table 1. The MFC composite has notably lower tensile strength, elastic modulus and strain at failure than the SBKP sample. This can be readily observed from the stress-strain curves in Figure 6. The high PCC content of the MFC sample is the cause of the lower strength properties. In the composite, the MFC partially offsets the debonding effect of the PCC and contributes positively to the sheet strength. This is because its relatively high surface area can effectively form hydrogen bonds with other cellulose surfaces. The MFC forms a percolated network through the PCC particles which helps hold the structure together. It is worth noting, that at 70% PCC content, a paper sample without MFC will have virtually no tensile strength.

The optical properties of the composite paper are excellent. This is partially due to the high PCC content, but is also influence by the presence of MFC. MFC will have a low light scattering when used alone or in conjunction with cellulose fibers. This is because the MFC will consolidate to a nonporous film that does not scatter light effectively. However, the situation is different when MFC is used together with the PCC. In that case, the MFC forms a network together with the PCC

(see Figure 2, c1-c3) and contributes to the overall light scattering. Therefore, this type of composite paper would open a totally new range of potential applications due to very high optical performance, nano and microporous structure, and an attractive cost structure in comparison with traditional paper products.

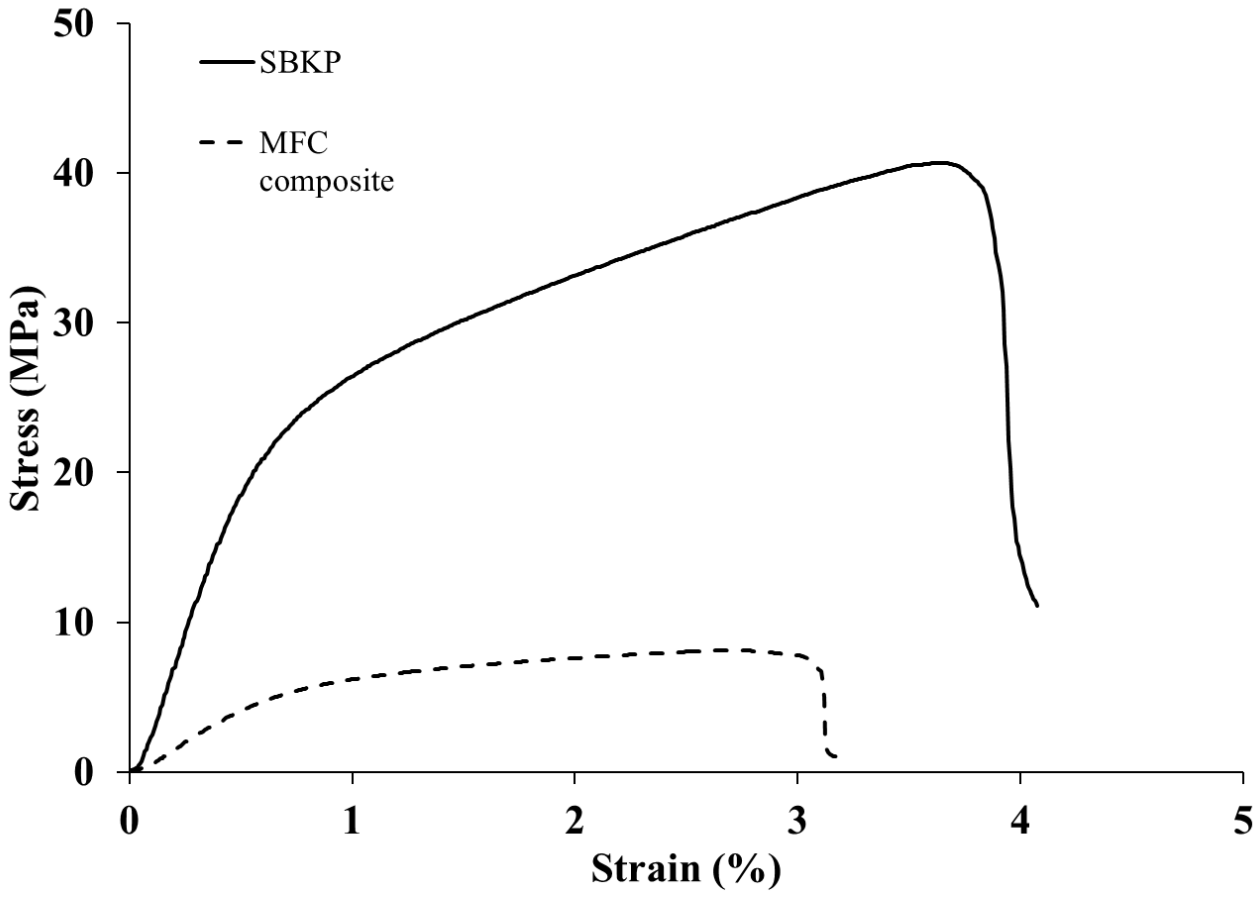

Figure 6. Typical stress vs strain curves of the MFC composite compared with SBKP fibers.

Table 1. Measured material properties of the MFC composite (MFC-c) compared with the SBKP fibers.

	SBKP	MFC-c
Tensile index (Nm/g)	**50.6 ± 1.23**	**12.3 ± 0.84**
Elastic modulus (GPa)	**4.46 ± 0.11**	**1.16 ± 0.12**
Brightness (ISO, %)	**82.5 ± 0.03**	**94.8 ± 0.10**
Opacity (%)	**77.2 ± 0.69**	**97.2 ± 0.14**
Light scattering (m^2/g)	**20.7 ± 0.47**	**173 ± 11.1**
Light absorption (m^2/g)	**0.17 ± 0.01**	**0.13 ± 0.01**

4 Conclusions

The MFC composite paper was shown to have very good dewatering characteristics in dynamic pressing conditions in comparison with a macroscopic pulp fiber based paper. This indicates that wet pressing will not be a limiting factor in the industrial production of the composite paper at least with press impulses up to 250 kPa·s. Wet pressing with higher pressures or shorter press times, which would correspond to higher production rates, might be feasible but were not tested during this work. Evidence from SEM-microscopy and mercury intrusion porosimetry suggest that the low amount of intraparticle water, together with a sufficiently open pore structure during consolidation, were responsible for the good pressing performance. The apparent density and porosity measurements revealed that the MFC composite sample resists z-directional shrinkage in the drying stage, which helps to maintain bulk and high sheet permeability in drying. Based on the SEM-images, MFC was found to form a percolated web throughout the PCC particles. This helps to maintain tensile strength and increases sheet light scattering.

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