Dewatering of Softwood Kraft Pulp with Additives of Microfibrillated Cellulose and Dialcohol Cellulose

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The addition of nano- and micro-fibrillated cellulose to conventional softwood Kraft pulps can enhance the product performance by increasing the strength properties and enabling the use of less raw material for a given product performance. However, dewatering is a major problem when implementing these materials to conventional paper grades because of their high water retention capacity. This study investigated how vacuum dewatering is affected by different types of additives. The hypothesis was that different types of pulp additions behave differently during a process like vacuum suction, even when the different additions have the same water retention value. One reference pulp and three additives were used in a laboratory-scaled experimental study of high vacuum suction box dewatering. The results suggested that there was a linear relationship between the water retention value and how much water that could be removed with vacuum dewatering. However, the linear relationship was dependent upon the pulp type and the additives. Additions of micro-fibrillated cellulose and dialcohol cellulose to the stock led to dewatering behaviors that suggested their addition in existing full-scale production plants can be accomplished without a major redesign of the wire or high vacuum section.

Keywords: Vacuum dewatering; Dewatering; Microfibrillated cellulose; Dialcohol cellulose; Papermaking; Strength additives; Retention aids; Drainage; Water retention value

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INTRODUCTION

One of the key strategies to reaching sustainable development is using bio-based, renewable, and biodegradable products, where their development meets existing requirements without depriving future generations the ability to fulfil their needs (Brundtland 1987). Therefore, it is important to further develop paper products, both new and existing designs, as well as other forest-based material products. To reach the goals of sustainable development by utilizing a forest-based bio-economy, paper products must be developed with both process and raw material efficiency. New and existing processes should be as energy efficient as possible, and products should be optimized regarding raw material use by choosing the most suitable material and using an appropriate amount.

Introducing materials, such as nano-fibrillated cellulose (NFC) and micro-fibrillated cellulose (MFC) that are often referred to as cellulose nanofibrils (CNF) and microfibrils (CMF), as additives to regular pulps has the potential to enhance product performance by increasing strength properties, which enables the use of less raw material per amount of material produced. Reports of enhanced strengths *via* MFC, NFC, and fines additives can be found in previous literature (Ahola *et al*. 2008; Eriksen *et al*. 2008; Taipale *et al*. 2010; Lindqvist *et al*. 2011; Hii *et al*. 2012; González *et al*. 2013; Hellström *et al*. 2014; Merayo *et al*. 2017). Adding these materials to the pulp has been found to enhance the paper product strength by providing more fibre-fibre bonds and a larger bonded area (Taipale *et al*. 2010; Lindqvist *et al*. 2011). The additives interacted with the pulp fibers in different ways. Ahola *et al*. (2008) reported that the addition of CNF can increase both the dry- and wet-strength, but only when it attaches in one of two possible ways. Eriksen *et al*. (2008) evaluated the strength increase of two different MFCs. Both increased the strength, and the difference was explained by different particle sizes that could give different mechanisms for adhesion and thereby enhance strength. The adhesion mechanism and particle size of the additives is important for the strength increase, although Eriksen *et al*. (2008) or Ahola *et al*. (2008) did not mention the dewatering aspects. However, the additives that bring strength enhancing effects often decrease the dewatering capacity of the pulp (Taipale *et al*. 2010; Lindqvist *et al*. 2011; Hii *et al*. 2012; González *et al*. 2013; Rantanen and Maloney 2013; Hellström *et al*. 2014; Brodin *et al*. 2014; Koponen *et al*. 2015). Koponen et al. (2016) showed that the rigidity of fibers was connected to dewatering capacity.

Studies have shown that MFC additives under certain circumstances can result in an unchanged dewatering capacity depending on the attaching mechanisms (Taipale *et al*. 2010; Hii *et al*. 2012; Koponen *et al*. 2015; Bousfield *et al*. 2017). Hubbe (2002) and Merayo *et al*. (2017) showed that the retention aid can interact with fines and CNF to make the drainage faster in certain concentrations.

When introducing for example MFC to conventional papermaking processes, dewatering becomes a major issue due to high water retention capabilities of MFC. Alternative process design could be a solution to the dewatering issues. Beneventi *et al*. (2015) described a spray deposition process of concentrated MFC on fabric.

Pulp fibers can be altered by different chemical reactions to change properties. As far back as the 1960s, chemical modifications of this kind were tested according to Larsson *et al*. (2014). Aracri *et al*. (2012) introduced aldehyde- and carboxyl-groups to the pulp fibers through the addition of the enzyme laccase and 2,2,6,6- tetramethylpiperidine-1-oxyl (Laccase-TEMPO). The aldehyde-groups were most prominent and increased the number of fiber-fiber bonds, which increased the tensile strength in both the wet and dry conditions. Larsson *et al*. (2014) developed another modified fiber through the oxidation of the C2-C3 bonds followed by the reduction of the aldehydes, and the product of this modification was named dialcohol cellulose (DAC). Sheets made with DAC fibers have high density and become more transparent and more ductile compared to non-treated fibers. The DAC properties depended on the degree of oxidization, which is the percentage of C2-C3 bonds that are affected by the modification.

Larsson and Wågberg (2016) further developed DAC through a combination of adding another oxidation set and hot pressing, and then showed both the transparency and oxygen barrier properties of the sheets. The properties of transparency, gas barriers, strength, and ductility are similar to products made from fossil-based raw materials. The renewable, biodegradable properties of cellulose-based materials, such as DAC, combined with the possibilities of these new properties are a step in the right direction regarding sustainable development. There is interest in using this type of cellulose material in combination with more traditional fibers, for example as part of the layered structure of paperboard (Jonasson and Neagu 2018). Minimal experimental data has been published on dewatering of stock containing DAC. Larsson and Wågberg (2016) mentioned that dewatering and processability in a paper machine are presumably good.

The aim of the study was to investigate how vacuum dewatering is affected by different types of additives, regarding moisture ratio and air volume. The hypothesis was that different types of pulp additions behave differently during a process such as vacuum suction, even when the different additions have the same water retention value.

EXPERIMENTAL

Materials

One reference pulp and three different additives were used in the study of high vacuum suction box dewatering at a laboratory scale. The reference pulp was a commercial beaten bleached softwood kraft pulp (BillerudKorsnäs Gruvön, Grums, Sweden), typically used together with the additives. The beaten reference pulp was taken after the refiners of a paper machine and therefore contained fines. The three additives were two MFCs (MFC2; BillerudKorsnäs Frövi, Frövi, Sweden) and one DAC (BillerudKorsnäs Gruvön, Grums, Sweden). MFC1 was provided under a non-disclosure agreement, and the origin therefore is not mentioned, but it was characterized in the same way as MFC2.

The pulp compositions are shown in Table 1. The DAC had an oxidization degree of 24%, which corresponds to approximately 3 mmol aldehyde/g fiber (Larsson *et al*. 2014). MFC1 and MFC2 had a total charge of 36 μeq/g and 70.1 μeq/g, respectively (SCAN-CM 65:02 2002). The two types of MFC were mixed with the reference pulp to 2%, 6%, and 10%. The MFC additives were for enhancing strength and the percentages used allowed the sheets to still behave like regular paper. Previous experiments (Eriksen *et al*. 2008; Taipale *et al*. 2010; Hellström *et al*. 2014) used approximately 5% MFC. The addition of more than 10% MFC to the pulp causes too long of a dewatering time. However, DAC consists of modified fibers, where 100% DAC gives a paper sheet with long but flexible fibers. This brings the possibility of testing a range between 0% and 100% in the beaten reference pulp.

Table 1. Pulp Composition

Denotation	Pulp Composition
Ref B	Beaten, bleached softwood Kraft pulp
D-10	90% Ref B + 10% DAC
D-33	67% Ref B + 33% DAC
D-66	34% Ref B + 66% DAC
D-100	100% DAC
M1-2	98% Ref B + 2% MFC1
M1-6	94% Ref B + 6% MFC1
M1-10	90% Ref B + 10% MFC1
M2-2	98% Ref B + 2% MFC2
M2-6	94% Ref B + 6% MFC2
M2-10	90% Ref B + 10% MFC2

All pulps from Table 1 were prepared to a consistency of 0.2%, and the water retention value and drainage (°SR) were measured using ISO 23714 (2014) and ISO 5267-1 (1999), respectively. Water retention value is defined as the ratio between the weight of a sample before and after oven-drying, after a specified centrifugation (ISO 23714, 2014).

Retention aid for MFC

All pulps including MFC were used together, during vacuum dewatering, with 0.1% of the dry-weight of the retention aid, cationic polyacrylamide (C-PAM). The C-PAM (Core Shell TM 74553, Nalco, Naperville, IL, USA) was added to the stock and the mix was stirred vigorously for 30 s before sheet forming or the dewatering resistance measurement. The drainage and water retention value measurements were performed both with and without the retention aid to investigate the impact on the dewatering mechanism of the retention aid. Hubbe (2002) and Merayo *et al*. (2017) showed that retention aid, fines, and CNF interact with each other and a certain optimal dosage can increase the drainability.

Methods

Laboratory scale vacuum dewatering

All pulps were tested in the laboratory suction box (Karlstad University, Karlstad, Sweden) described by Granevald *et al*. (2004). A schematic picture of the laboratory suction box, redrawn from Granevald *et al*. (2004), is shown in Fig. 1.

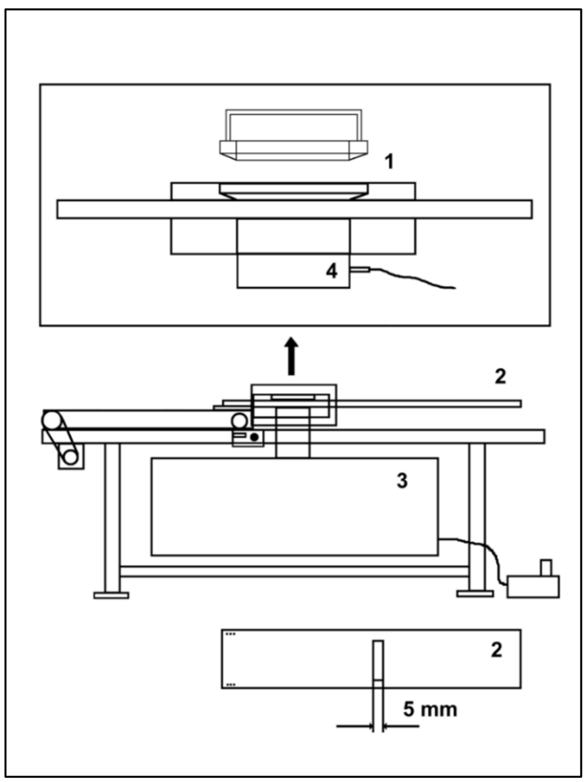

Fig. 1. Schematic picture of the laboratory suction box. The machine includes: (1) sample frame, (2) moveable plate with 188x5-mm rectangular opening, (3) 300-dm^3 vacuum tank, and (4) a transducer logging the pressure directly underneath the sample. The vacuum zone has a diameter of 188 mm.

For all tests, a sheet was formed directly onto a forming fabric in a handsheet former. The sheets had a diameter of 18.4 cm. The sheets and the fabric were transferred by hand to the sample frame of the laboratory suction box. There were no differences in sheet forming between the samples; thus they varied in moisture ratios at the start of vacuum dewatering. A vacuum level of 40 kPa was used for all of the measurements. Dryness measurements according to ISO 638 (2008) were performed before suction box dewatering and after five different dwell times (1 ms, 2.5 ms, 5 ms, 10 ms, and 20 ms) obtained with different plate velocities on the machine. All of the dryness measurements were recorded after complete rewetting because the time to remove the sheet from the forming fabric was larger than the time for full rewetting, as reported by Sjöstrand *et al.* (2015). The moisture ratio was calculated for all samples. The moisture ratio is defined as the ratio between the weight of water in the sample and the weight of dry mass. Triplets of each measurement point were made.

The pressure was logged with a transducer (Amtele AB, Stockholm, Sweden) located directly underneath the sample during the vacuum dewatering. The air volume that passed through the sample was calculated using Eq. 1,

$$V_{\mathrm{air}} = \frac{V_{\mathrm{tank}}}{P_{\mathrm{atm}}} * \Delta P, \tag{1}$$

where V_{air} is the air volume through the sample (dm^3), V_{tank} is the volume of the vacuum tank (dm^3), P_{atm} is the atmospheric pressure (kPa), and ΔP is the measured pressure increase in the tank (kPa).

Three sheets each of M1-2, M1-6, M1-10, D-10, D-33, D-66, D-100 were prepared in a Rapid Köthen machine (RL-ASF-A; Rycobel, Deerlijk, Belgien) according to ISO 5269-2 (2004). C-PAM was used as before with the MFC. Two sheets from each of these were used for pore size distribution measurements, and one sheet was used for image analysis *via* a scanning electron microscope (SEM; JSM-6460 LV; JEOL, Peabody, MA, USA). The pore size distribution was determined using capillary flow porometry (Jena and Gupta 2010). The capillary flow porometer (iPore; Porous Materials Inc., Ithaca, NY, USA) measured the flow rate of air through a dry and wet sample at different pressures. Then, a flow distribution was reported, which was assumed proportional to the number density of pores of different radii.

RESULTS AND DISCUSSION

The water retention values (WRV) and drainage measurements (°SR) for all pulps are shown in Figs. 2 and 3, respectively. The stocks containing DAC (D-10 through D-100) and MFC1 (M1-2 through M1-10) had lower drainability and higher water retention values than the reference. In contrast, the addition of MFC2 (M2-2 through M2-10) only resulted in minor changes.

Additions of the retention aid to the stock led to a substantial increase in drainability and a considerable decrease in water retention (except for WRV of Ref B and M2-2, the latter due to a high deviation between measurements) as illustrated in Fig. 3. The increase in drainability was expected and has been shown before by Hubbe (2002) and Merayo *et al*. (2017).

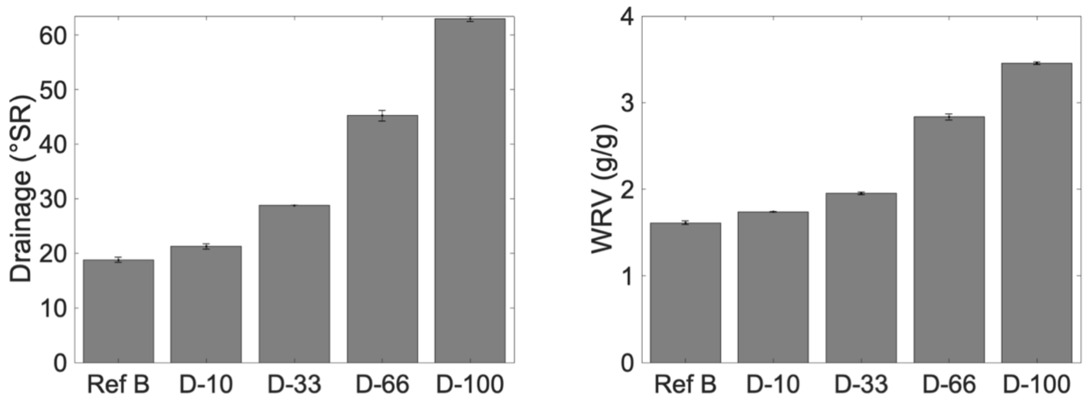

Fig. 2. Drainage (°SR) and water retention value (g/g) for reference pulp and different percentages of DAC

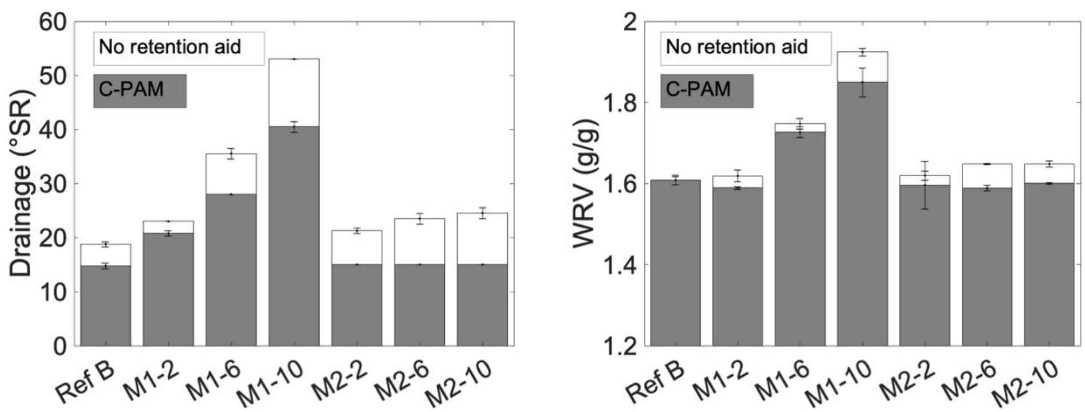

Fig. 3. Drainage (°SR) and water retention value (g/g) for reference pulp and different percentages of MFC1 and MFC2, with and without retention aid (C-PAM)

Hubbe and Heitmann (2007) wrote about the choke point mechanism, where fine materials without a retention aid block pores between fibers, which leads to decreased drainabililty. Hubbe and Heitmann also explained that the presence of retention aids can diminish the choke point mechanism when the retention aid binds the fine material onto the fibers and decreases the blocking of interfiber pores. The retention aid (C-PAM) in this study increased the bonding of MFC to fiber surfaces and thereby increased drainability by leaving more pores open for water flow. However, the changes in water retention were not expected. The water retention was not believed to be dependent upon the network morphology because MFC should be able to hold equal amounts of water regardless of location in the sheet. By changing the network morphology with a retention aid, the WRV was affected as well as the drainage (Fig. 3).

The WRV was calculated from the mass of a sample before and after oven-drying, after a specified centrifugation, according to ISO 23714 (2014). One explanation for the WRV being dependent upon the presence of the retention aid could be that the sample mass before centrifugation was lower with the retention aid due to increased drainability, which in turn, lowered the sample mass after centrifugation. The mass after oven-drying did not depend on the presence of the retention aid because bone dry weight did not depend on whether or not the MFC was attached to the fiber surface. When the sample mass before oven-drying was lowered, but the sample mass afterwards was unchanged, the water retention value was lower. Evidence of this behavior, *i.e.*, different network morphology due to the presence of retention aid, is not provided in this study. This would be interesting to investigate further for vacuum dewatering. Related results describing how dewatering is affected by different conditions have been reported in previous research (Taipale *et al*. 2010; Hii *et al*. 2012; Koponen *et al*. 2015; Bousfield *et al*. 2017).

Results from the vacuum suction box included the moisture ratio (g/g) and air volume (dm^3) depending on the vacuum dwell time (Fig. 4). The data are included for Ref B because it was used in all mixtures.

As shown in Fig. 4, the dewatering curves of the M1-10 additives indicated that the outgoing moisture ratio was higher and the dewatering rate was slower than for the DAC additives and references pulp. In addition, M1-10 also had the highest initial water content. This was also observed in the longer time before air started to flow through the M1-10 sheet. The moisture ratio dependence on dwell time showed that MFC2 additives had approximately the same dewatering rate and outgoing moisture ratio as Ref B. The hand-sheet forming procedure that resulted in different ingoing moisture for the samples where important, as it made it possible to draw conclusions about the forming section of a paper machine, not only a single vacuum suction box.

For high contents of DAC (D-66, D-100), dewatering was slower than for Ref B even though the initial moisture content was similar regardless of the DAC content. The slower dewatering for DAC was also reflected by the slower onset of air permeation through the sheet (D-33, D-66, and D-100). Koponen *et al.* (2016) show results of how obtainable moisture ratio during the initial dewatering on the forming section depends on the compressibility of the sheet. High compressibility gives less efficient dewatering. Looking at DAC fibers, this is true for WRV measurements and vacuum dewatering. Higher percentages of DAC added gives lower obtainable moisture ratio.

The results depicted in Fig. 4 showed that no air passed through the sheet for the first millisecond for all pulps. This was expected because the pore structure of the sheet was initially filled with water, but the phenomenon has not been shown in previous studies. Air started to flow through the sheet as the largest pores were evacuated by the vacuum suction. This air flow quickly reached a steady state and later became constant enough for the air volume to increase almost linearly to dwell time. The dwell time was different for different pulps when the air flow became nearly constant. The linear behavior started after approximately 1 ms for Ref B and MFC. It took approximately 5 ms before the linear behavior started for high contents of MFC1 (M1-6, M1-10) and DAC (D-33, D-66, and D-100).

The air flow depended on the pore structure during the vacuum dewatering, which is shown as the slope of the air volume measurement in Fig. 4. Previous research (Stenström and Nilsson 2015) suggests that the only water capable of being removed with vacuum dewatering lies between the fibers. This means that the lumen and fiber walls are still filled with water and gives the sheets a porous structure that will correspond with the achievable air flow during vacuum dewatering. The sheets made from DAC fibers showed low air volumes flowing through the sheets. This suggested that the sheets made from DAC started to lose their porosity as early as in the vacuum system.

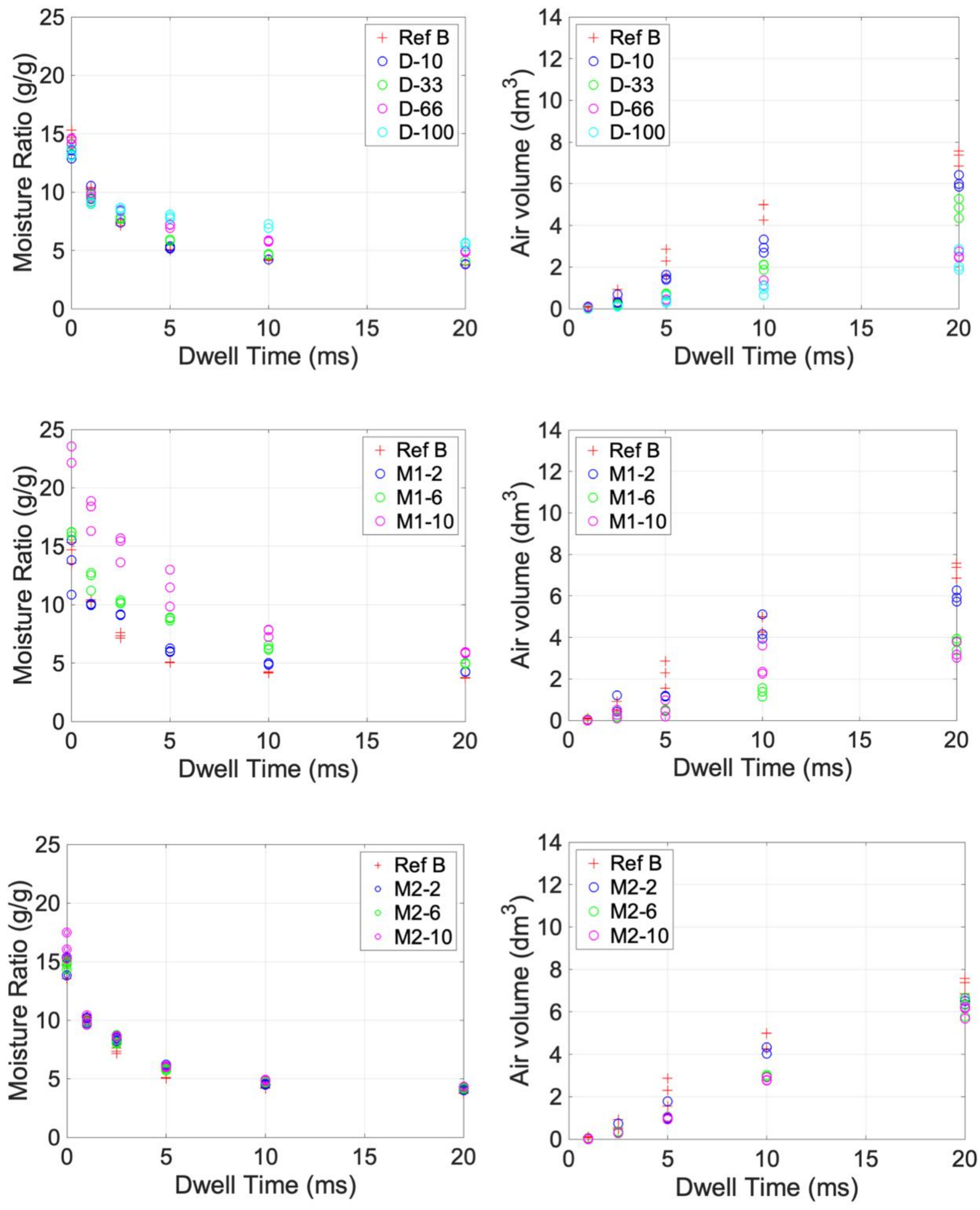

Fig. 4. Moisture ratio (g/g) and air volume (dm^3) depending on vacuum dewatering dwell time (ms) for all pulps

Because fibers are flexible, their lumens must have collapsed before vacuum dewatering. Additionally, high MFC1 concentrations had low air volumes flowing through the sheets due to large amounts of small-scale materials lodged between the fibers where air is supposed to flow through.

Moisture ratio curves in Fig. 4 suggested that both MFC and DAC additions could be used in existing vacuum systems of full-scale production plants because there was not a large deviation relative to the beaten reference pulp. However, there could be problems with adding more than 6% of this particular MFC1 in a paper machine because the curve for M1-10 was much slower in the initial vacuum dewatering. To introduce the additives in full-scale production, investigations of pressing and drying are needed as well.

Figure 5 shows the obtained moisture ratio after 20 ms dwell time in the vacuum suction box compared with the corresponding individual water retention values.

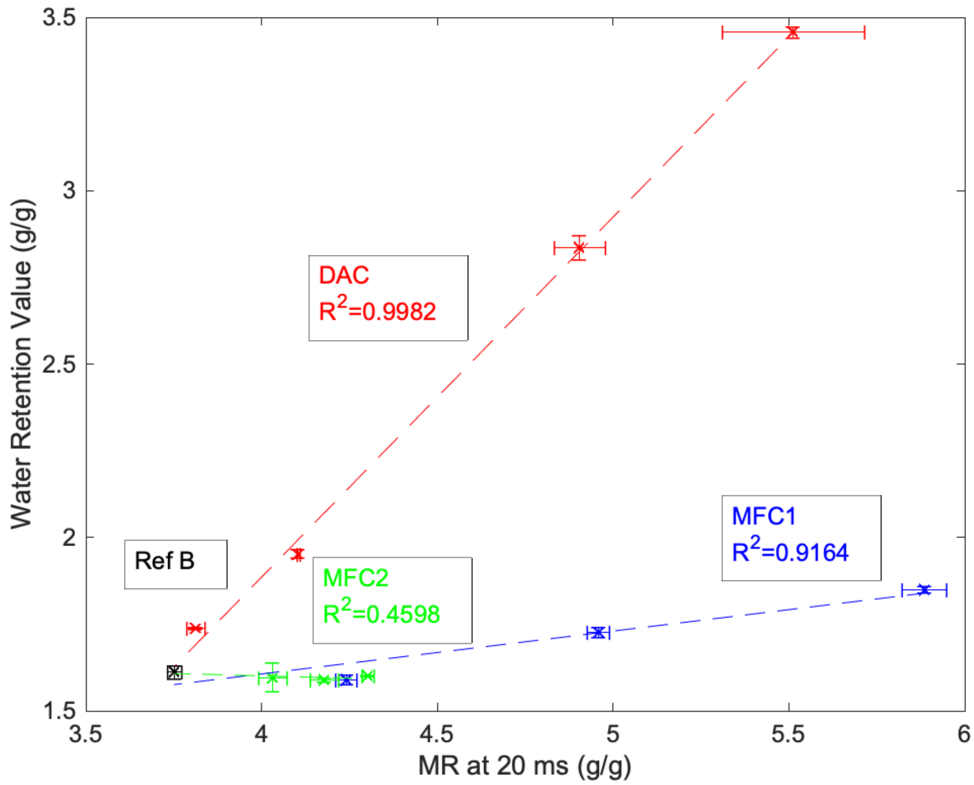

Fig. 5. Obtained moisture ratio (MR) after 20 ms vacuum dwell time with a 40 kPa vacuum level for all pulp compositions compared to WRV

Figure 5 shows a linear behavior of the outgoing moisture ratio based on WRV. This can be used when determining the possibilities of using DAC and MFC as additives in full-scale production. Measuring WRV determines whether the vacuum dewatering section of the paper machine can dewater specific additives. However, the slope must be determined for different pulps and additives.

Figure 5 also shows that the different pulp additives follow different linear behaviors. Both MFC additives followed the same line while DAC followed another. This meant that the same WRV value did not give the same outgoing moisture ratio, which in turn meant that the dewatering mechanisms between WRV and the laboratory vacuum suction box were not the same for these pulp additives. Therefore, experimenting with different pulps with high values for °SR and WRV in more process-like conditions, such as dewatering equipment used in this study, can reduce the cost of pilot or full-scale trials while still avoiding ranking the pulps only in terms of their WRV as this study shows that conclusions made solely based on WRV can be misleading.

The R^2-value for MFC2, calculated with MATLAB (MathWorks, Inc., version 2018b, Natick, MA, USA), was low, which indicated that the linear behavior observed for MFC1 and DAC was not present for all kinds of additions. The additives MFC2 had similar values for both WRV and moisture ratio that could explain their poor linearity. Additions of larger percentages of MFC2 could give a more linear behavior with a higher R^2-value.

The mechanisms of dewatering with a vacuum are suggested not to be the same as those of dewatering during WRV testing. The data in Fig. 5 show that the moisture ratio after vacuum dewatering was more similar to the WRV for samples containing DAC fibers than for samples containing MFC. One explanation for this could have been the lack of water content in fiber lumen for D-100. Larsson *et al*. (2014) showed higher densities for DAC hand sheets with a higher degree of oxidization. A denser sheet yields more collapsed fibers and less lumen water holding capacity. Determining the WRV would likely involve removing the water from fiber lumen for non-collapsed ordinary fibers, while vacuum dewatering does not (Stenström and Nilsson 2015). Because the DAC fibers were collapsed, they could have lost small amounts of available lumen water for both vacuum dewatering and WRV measurements. This could explain the different slopes of Fig. 5.

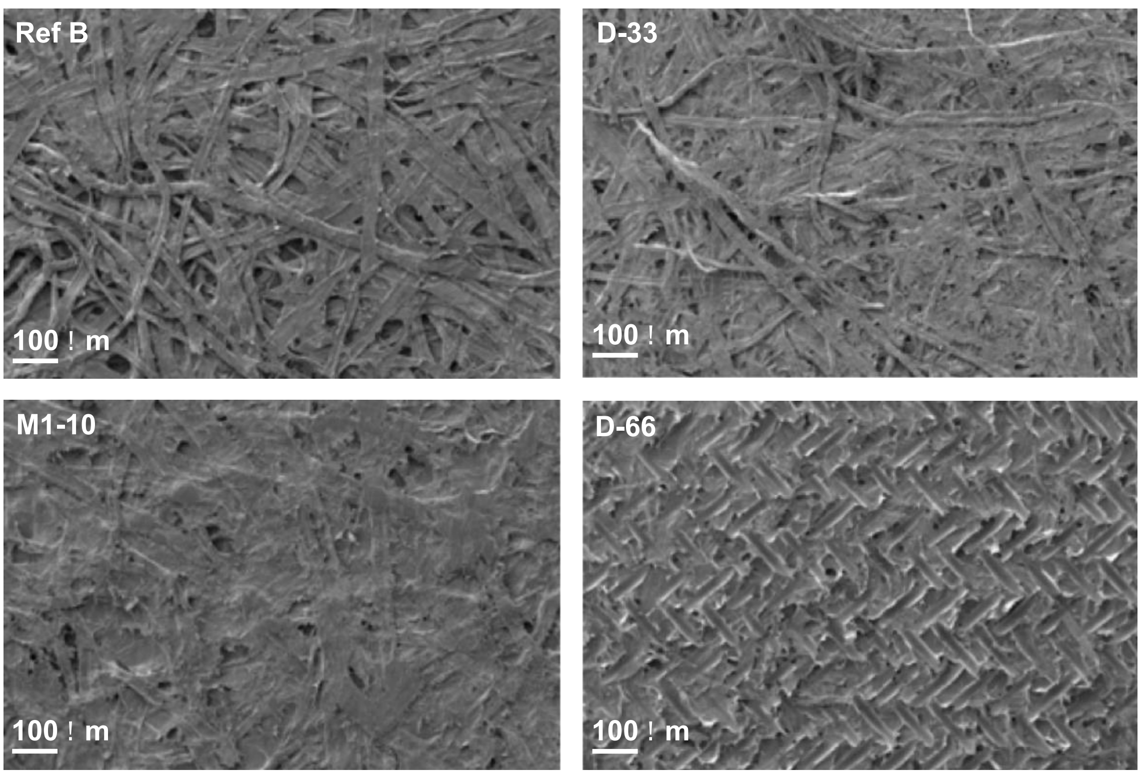

Fig. 6. SEM pictures of sheets of beaten reference pulp, and selected MFC1 and DAC; note the clear wire markings for DAC 66%

Through an analysis of SEM pictures, a difference between MFC and DAC was observed. The low percentages of DAC did not differ much from the pictures of the reference pulps.

The pictures of MFC1 showed many materials between fibers. They also illustrated that the MFC was successfully retained, which was also suggested by the results of the dewatering and air volume measurements. The SEM pictures showed the difference between the MFC and DAC where MFC blocked large pores between regular fibers, which was connected to the results from the air volume measurements. The DAC made the whole sheet extremely flexible, and this can be seen by the clear wire mark for D-66 (Fig. 6).

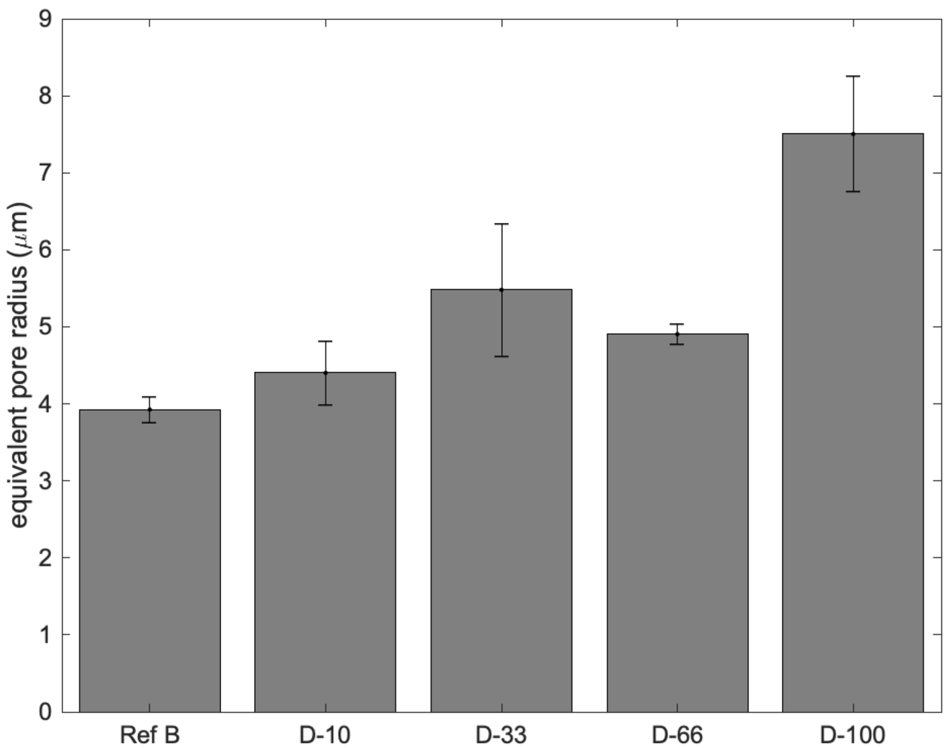

Fig. 7. Equivalent pore radii for reference beaten and DAC

The pore size distribution measurements (Fig. 7) for DAC were more open than the reference pulp pores. This indicated that the DAC behaved differently when wet than when dry because the DAC was more closed than the Ref B after vacuum dewatering. Measurements could not be performed for the MFC sheets because of their closed structures. The wet DAC sheet had a more closed structure, as the fibers were extremely flexible and the fibers themselves held a lot of water. This gave less efficient vacuum dewatering for high percentages of DAC compared to the reference pulp. When the DAC sheet was dry, the structure could open, which would explain the high porosity shown in the pore size distribution measurements.

CONCLUSIONS

1. There was a linear relationship between the water retention value and the achievable moisture ratio of vacuum dewatering for the tested pulps and additives. The slopes of

the linear relationships were dependent upon the additive. For water retention value around 2, the sheets prepared with the finest MFC additive were almost 50% more wet after vacuum dewatering compared with DAC additives.

2. Up to 75% lower air volumes were recorded at vacuum dewatering for select additives compared with the reference. There were also several milliseconds before the large pores between fibers were evacuated enough to allow air to penetrate the structure.
3. Additions of MFC and DAC had dewatering behaviors that suggested their potential for use in existing full-scale production plants for strength enhancement. Depending on the characteristics of MFC, precautions must be taken regarding the amount of MFC used. More specifically, in our case, addition of M1-10 would probably cause runnability issues depending on the incomplete dewatering after 20 ms.
4. Retention aids and additives can interact to change dewatering behavior. This study showed increasing drainability and decreased water retention values with the addition of a retention aid. It is important to be aware of that the type and amount of retention aid will affect the possibilities of industrial up-scaling.

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