The effect of MFC on the pressability and paper properties of TMP and GCC based sheets

Collin Hii, Øyvind W. Gregersen, Gary Chinga-Carrasco, Øyvind Eriksen

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SUMMARY: Different qualities of microfibrillated cellulose (MFC) were blended with thermomechanical pulp (TMP) and ground calcium carbonate (GCC) filler. The addition of MFC reduced the drainage of the pulp suspension but improved strength properties. Wet pressing experiments showed that optimal use of MFC and filler could enhance the strength and optical properties without reducing the solids content after wet pressing. Field-emission scanning electron microscopy (FESEM) revealed that MFC adsorbed onto and contributed to the bonding of the filler particles and fibres. The MFC binds the filler-MFC-fines aggregates to the fibre network and partially filled the pore network. As a result, MFC addition increased the air resistance and internal bonding of the sheet.

ADDRESSES OF THE AUTHORS:
Collin Hii (hii@nt.ntnu.no)
Øyvind W. Gregersen (oyvind.gregersen@chemeng.ntnu.no)
Norwegian University of Science and Technology, NTNU, Trondheim, Norway
Gary Chinga-Carrasco (gary.chinga.carrasco@pfi.no)
Øyvind Eriksen (oyvind.eriksen@pfi.no)
Paper and Fibre Research Institute, PFI, NO-7491 Trondheim, Norway

Corresponding author: Collin Hii

The drive to reduce production cost and increasing the opacity, brightness and surface smoothness has encouraged the use of fillers in papermaking. However, fillers reduce fibre bonding and paper’s strength properties. Addition of fillers to pulp slurry without retention aid increases the filtration resistance of the slurry by plugging of the fibre cake (Springer, Kuchibhotla 1992; Hubbe, Heitmann 1997). The optimal use of retention aid has enabled high filler retention and even filler distribution in the sheet thickness direction (Tanaka et al. 1982). Filler addition has improved dewatering in the wet end (Liimatainen et al 2006). Filler usage also improves optical properties, but reduces strength properties (Mohlin, Olander 1986; Hjelt et al. 2008). Fillers that agglomerate to clusters could resist pressure during wet pressing and maintain the bulk of the sheet (Hjelt et al. 2008). However, filler particles may also form strong agglomerates between fibres and hence prevent fibre-fibre bonds (Hjelt et al. 2008).

Addition of kraft pulp fines counteracts negative effects of fillers on strength properties but affects the drainage of the pulp (Sandgren, Wahren 1960a, 1960b; Htun, Ruvo 1978; Seth 2003; Lin et al. 2007). Fines are inhomogeneous complex particles that passes through a 200 mesh (76 μm) wire in the solid-liquid separation method as stated in TAPPI T261 cm-00. Kraft pulp fines possess high degree of swelling and thus enable the fines to bind well to the fibre structure (Htun, Ruvo 1978). Pre-mixing fillers with fines in a controlled fashion, prior to its addition to the pulp furnish, improves strength and optical properties of the sheet (Lin et al. 2007).

Microfibrillated cellulose

In recent years, studies have shown that microfibrillated cellulose (MFC) can be used as strength enhancer (Iwamoto et al. 2007; Eriksen et al. 2008; Ahola et al. 2008; Subramaniam 2008; Mörseburg, Chinga-Carrasco 2009; Zimmermann et al. 2010; Taipale et al. 2010). Eriksen et al. (2008) found significant tensile index increase at 4% addition of MFC to TMP handsheets independent of the production method. Mörseburg, Chinga-Carrasco (2009) added MFC to clay loaded layered TMP sheets and found that the strength properties improved. Addition of MFC also increased the air resistance (Eriksen et al. 2008; Subramaniam 2008). Taipela et al. (2010) found that the optimal level of MFC addition would maintain or improve strength properties without impeding dewatering. Synergy effects of MFC–filler interactions can counteract the reduction in strength properties from filler addition while improving light scattering (Mörseburg, Chinga-Carrasco 2009).

MFC has been prepared by mechanically disintegrating cellulose fibres through homogenization (Turbak et al. 1983; Herrick et al. 1983; Pääkkö et al. 2007; Syverud et al. 2011), grinding (Iwamoto et al. 2005; Iwamoto et al. 2007; Eriksen et al. 2008; Abe, Yano 2009), fluidization (Taipale et al. 2010) and combined homogenizer treatment and grinding processes (Iwamoto et al. 2005). Others have pre-treated the fibres chemically before a mechanical disintegration (Saito et al. 2006, 2007; Pääkkö et al. 2007; Henriksson, Berglund 2007; Wågberg et al. 2008; Syverud et al. 2011). MFC produced from mechanical disintegration is heterogeneous in size and forms entangled and disordered networks. Mechanically produced MFC, without pre-treatment, is commonly composed of micro- and nano-structural components, e.g. poorly fibrillated fibres, fines and nanofibrils (Chinga-Carrasco, 2011). The nanofibrils have diameters less than 100 nanometres and lengths in the micrometre scale. Increasing the number of passes in the homogenization and fluidization processes produces more nanofibrils. MFC may thus be considered a subset of kraft pulp fines. Taipale et al. (2010) found that increasing the number of passes in a fluidization process produced MFC with more nano-sized fibrils. Enzymatic pre-treatment reduces the fibrils diameter down to below 20 nm range (Pääkkö et al. 2007). MFC produced with TEMPO-mediated oxidation as pre-treatment has exhibited more homogeneous nanofibril diameters (< 10 nm).
and lengths in the micrometre scale (Saito et al. 2006, 2007; Chinga-Carrasco et al. 2011).

**Wet pressing**

Wet pressing has been extensively studied. Wahlström (1960a, 1960b) found that the hydraulic pressure formed during web compression was the main driving force for water removal in the converging part of the press nip. The flow resistance in the wet web generated the hydraulic pressure. The total pressure ($P_T$) can be defined, using the Terzaghi principle (Terzaghi 1943), as the sum of structural ($P_s$) and hydraulic pressure ($P_h$) as in Eq 1. The Terzaghi principle was first applied to papermaking by Campbell (1947).

$$P_T = P_s + P_h$$  \[1\]

Equation [1] is based on force balance. This force balance can be stated as the stress or pressure balance by assuming the inertial effects are not negligible and the area over which the force is acting is equal.

Carlsson et al. (1978) revealed that water within the fibre played an important role in press dewatering. They found that the dewatering of fibre started to occur at 20-25% solid content, thus, as the web compression progressed, the fraction of water pressed out from the fibre wall increased. The water flow through the fibre wall pores has significantly affected the structural pressure when compressing the web in the press nip (Carlsson et al. 1978; Paulapuro 1989). Studies have also shown that the water and fibre surface interaction play a crucial role in press dewatering (Szikla, Paulapuro 1989). Wahlström (1990) redefined the $P_s$ as the sum of pure mechanical pressure that compressed the fibre network, $P_c$ and the pressure required to remove the water from the fibre wall, $P_r$ thus:

$$P_T = P_h + P_c + P_r$$  \[2\]

The pressure components in Eq 2 are varying both in thickness direction and over time during wet pressing.

The swelling of mechanical pulp is largely due to fibrillar fines, internal fibrillation and external fibrillation. (Salmen et al. 1985; Maloney, Paulapuro 1999; Luukko, Maloney 1999). Maloney et al. (1997) postulated that in thermomechanical pulp (TMP) the pore closure after pressing and drying can be due to fines coagulation.

In dynamic wet web compression, increasing the ingoing moisture, sheet basis weight and compression rate increased the hydraulic pressure component in both chemical and mechanical pulp sheets (Szikla 1992). Increase in basis weight will increase the hydraulic pressure in the web during wet pressing and more so in samples with high initial moisture content (Szikla 1992). The roughness of the contacting surface during wet pressing will induce local stress variation and affect the water flow in a compressed fibre network (Vomhoff 1998, I’Anson, Ashworth 2000). In a grammage range relevant to papermaking, the steady state permeability of a web varies considerably with grammage when compressed against a rough permeable surface (Vomhoff 1998). I’Anson, Ashworth (2000) found that at low grammage (30 g/m²), fine contact between the surfaces would enhance press dewatering while coarse contact would improve dewatering for higher grammage (60 g/m²). The high sheet permeability would be needed for large amount of water to escape from high grammage web.

Increasing the web temperature improves water removal in wet pressing. This mechanism is well understood and utilised in papermaking (Wahlström 1960b; Roja, Thorp 1982; Batty et al. 1982; Radwan, Nayar 1982; Saaristo et al. 1984; Busker, Francik 1984; Back 1988; Patterson, Iwamasa 1999). Increasing the web temperature reduces water viscosity and water surface tension and thus reduces hydraulic loads and capillary forces respectively. Heating the web also softens the fibres, which in turn reduces web springback and increases web compressibility; this thus reduces rewetting and structural loads respectively.

The press nip behaviour can be summarized into flow controlled and pressure controlled modes (Wahlström 1969, 1979; Chang, Han 1976; Ceckler et al. 1982; Burns 1992). The flow controlled mode prevails for high moisture sheets with high flow resistance while the opposite holds for pressure controlled mode. Most presses operate in the area between flow control and pressure control mode. The press impulse, integral of pressure over time, promotes dewatering in flow controlled mode. The peak pressure enhances the removal of free water in the pressure controlled mode.

During wet pressing the interaction between viscous flow forces and fibre network can result in uneven compaction of the web in the thickness direction, or stratification (Wick 1982; MacGregor 1983; Burton, Sprague 1987; Szikla, Paulapuro 1989b; Burns et al. 1992). The exit layer densification is caused by the shear force from liquid flow (Chang 1978; Macgregor 1989, 2001; Szikla 1992). The densification of the exit layer will increase the hydraulic pressure in the web and results in a more flow controlled nip.

MFC has a strong water retention property and a high specific surface area. (Herrick et al. 1983; Pääkkö et al. 2007). As a result, MFC addition into a given pulp will reduce dewatering by increasing the hydraulic pressure, $P_h$. Taipale et al. (2010) found that optimum selection of MFC and kraft pulp components and process conditions can enhance the strength properties without negative effects on drainage.

There is a lack of published results on the effects of MFC on the pressability of ground calcium carbonate (GCC) filled TMP sheets. Hence, the purposes of this study are:

1. Determine the optimal MFC addition in 30% filler loaded TMP sheets to yield similar or better pressability as compared to TMP samples with 15% filler content.
2. Determine the effects of MFC and high filler loading, up to 30%, on paper properties.

**Materials and Methods**

**Production of microfibrillated cellulose**

Never-dried Pinus radiata kraft pulp was used to produce the MFC. The pulp fibres were homogenized with a Rannie 15 type 12.56x homogenizer operated at 1000 bar pressure drop. The pulp consistency during homogenization was kept at 0.5% to avoid plugging problems.
Fluffy flocs were seen forming after 500 ml of the furnish mixture was stirred rigorously with a stirrer at 1250 rpm during the addition of the retention chemicals. The fixation aid and flocculant were pre-diluted to 0.5 g/l and 0.25 g/l respectively before addition into the furnish mixture. The time delay between the addition of the fixation aid and flocculant was 30 seconds. The fixation aid and flocculant were pre-diluted to 0.5 g/l and 0.25 g/l respectively before addition into the furnish mixture. The furnish mixture was stirred rigorously with a stirrer at 1250 rpm during the addition of the retention chemicals. Fluffy flocs were seen forming after 500 ml of the furnish mixture was shaken rigorously in a 1 l litre bottle and let to settle in a 1 l graduated cylinder.

All the tests carried out in the experiment followed the standards as illustrated in Table 1. Table 2 shows the TMP pulp coarseness, freeness, water retention values and ash content. The pulp coarseness was measured with FiberMaster. The water retention value (WRV) for all the furnish mixtures were performed by centrifuging the pulp sample for 17 minutes at 4500 rpm and at 23°C.

Hand sheets making
80 g/m² hand sheets for optical properties, strength and physical testing were made using a conventional handsheet former with closed water circulation. In each sample series, 12 sheets were made to ‘fill’ the white water system with fines and filler until the basis weight of the handsheet is stable. The standard for handsheet making with closed water circulation is stated in Table 1. Eight series of handsheets, as illustrated in Table 3, were made to study the effects of MFC and fillers on physical and optical properties. The internal bonding and z-directional strength measurements were carried out using Zwick Roell SMART.PRO material testing machine.

Samples for wet pressing
The 60 g/m² samples for the wet pressing experiment were made using a FiberXpress apparatus (Fig 1). The pulp slurry was dewatered through membrane and felt at 1 MPa for 60 seconds. The membrane used for dewatering the slurry was 50 micrometres thick with 41% open area. The felt was pressurized with 41% open area. The felt placed underneath the membrane gave support and enabled the forming of 60 g/m² samples. The membrane maximised the retention of fines and fillers. Eight series of samples, as depicted in Table 3, were made to study the effects of MFC and fillers on dryness after wet pressing. Samples with 50 mm diameters were formed and conditioned sandwich with wet blotters to maintain dryness close to 20%.

Wet Pressing
A dynamic wet pressing simulator (Fig 2) was used to carry out the pressing experiment. A single sided dewatering set up was used for the experiment. The design details of the dynamic wet pressing simulator were similar to the unit located in the paper laboratory of Aalto University (Saukko 2007). The pressing nip consisted of a top solid metal plate with polished surface and a 90 mm diameter sintered porous bottom plate. A single roll press pulse of 8 ms pulse length was used in the wet pressing experiment. The top and bottom plates had to be in closed position before a stable and repeatable roll pulse could be generated. This inevitably would cause some water to flow from the sample to the porous plate. After pressing, the sample will stick onto the
Table 3. Furnish mixtures and labels.

<table>
<thead>
<tr>
<th>Label</th>
<th>TMP (%)</th>
<th>Ash (%)</th>
<th>MFC (%)</th>
<th>MFC (# of passes)</th>
<th>Drainage (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T100</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>23.9 ±1.0</td>
</tr>
<tr>
<td>F15T85</td>
<td>85</td>
<td>15</td>
<td>0</td>
<td>0</td>
<td>33.2 ±1.4</td>
</tr>
<tr>
<td>5MFC5F15</td>
<td>77.5</td>
<td>17.5</td>
<td>5</td>
<td>5</td>
<td>37.6 ±1.4</td>
</tr>
<tr>
<td>5MFC5F30</td>
<td>66.1</td>
<td>28.9</td>
<td>5</td>
<td>5</td>
<td>51.5 ±3.2</td>
</tr>
<tr>
<td>5MFC2.5F15</td>
<td>82.5</td>
<td>15</td>
<td>2.5</td>
<td>5</td>
<td>53.6 ±4.3</td>
</tr>
<tr>
<td>5MFC2.5F30</td>
<td>64.9</td>
<td>32.6</td>
<td>2.5</td>
<td>5</td>
<td>49.9 ±4.5</td>
</tr>
<tr>
<td>3MFC2.5F15</td>
<td>80.3</td>
<td>17.2</td>
<td>2.5</td>
<td>3</td>
<td>43.6 ±1.2</td>
</tr>
<tr>
<td>3MFC2.5F30</td>
<td>65.5</td>
<td>32</td>
<td>2.5</td>
<td>3</td>
<td>35.0 ±3.5</td>
</tr>
</tbody>
</table>

Table 4. Dewatering data for the different sample series. The maximum pressure is set at 3.5 MPa. The values are reported as average ±1 standard deviation. The labels for the different furnish mixes are explained in Table 3.

<table>
<thead>
<tr>
<th>Label</th>
<th>Ash (%)</th>
<th>WRV (g/g)</th>
<th>Pre solids (%)</th>
<th>Solids (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T100</td>
<td>0.5 ±0.1</td>
<td>1.33 ±0.07</td>
<td>13.9±0.1</td>
<td>33.0±0.3</td>
</tr>
<tr>
<td>F15T85</td>
<td>15.0 ±0.1</td>
<td>0.97 ±0.03</td>
<td>15.5±0.1</td>
<td>36.3±1.2</td>
</tr>
<tr>
<td>5MFC5F15</td>
<td>17.5 ±0.8</td>
<td>0.94 ±0.03</td>
<td>13.9±0.6</td>
<td>36.4±1.4</td>
</tr>
<tr>
<td>5MFC5F30</td>
<td>28.9 ±0.5</td>
<td>0.95 ±0.02</td>
<td>14.7±0.2</td>
<td>36.3±0.2</td>
</tr>
<tr>
<td>5MFC2.5F15</td>
<td>15.0 ±0.1</td>
<td>1.04 ±0.03</td>
<td>14.2±0.4</td>
<td>35.9±0.3</td>
</tr>
<tr>
<td>5MFC2.5F30</td>
<td>32.6 ±0.3</td>
<td>0.90 ±0.02</td>
<td>16.3±0.1</td>
<td>38.9±0.5</td>
</tr>
<tr>
<td>3MFC2.5F15</td>
<td>17.2 ±0.1</td>
<td>1.07 ±0.03</td>
<td>15.7±0.1</td>
<td>33.6±0.8</td>
</tr>
<tr>
<td>3MFC2.5F30</td>
<td>32.0 ±0.2</td>
<td>0.93 ±0.03</td>
<td>18.2±0.5</td>
<td>36.5±1.2</td>
</tr>
</tbody>
</table>

Fig 1. The FiberXpress used to make 60 g/m² samples.

Fig 2. The dynamic wet pressing simulator for the wet pressing experiments. (A) Pressing cones with internal heating element (B) Bottom plate (C) Top plate (D) Distance sensor.

Fig 3. Average solids content (%) after wet pressing vs. pressure (MPa) during wet pressing for 15% filler and 85% TMP mixture pressed at 25°C and 50°C. The error bars are 1 standard deviation.

At around 3.5 MPa the solids content flattened out (Fig 3). The maximum pressure represents the setting with the maximum fraction of flow control until some type of sheet crushing problem is encountered (crush point). Fig 3 shows that the pressing pressure to achieve maximum solids content for F15T85 sample wet pressed at 25°C is at around 3.5 MPa. Thus 3.5 MPa was used as pressure set point for pressing samples at 50°C. As expected, wet pressing with 3.5 MPa at 50°C yields higher solids content, 36%, as compared to the 33.5% at 25°C.

Controlled press pulse

peak pressures at 25°C. The solids content versus pressure response curve was constructed (Fig 3). The pressure at 25°C when the maximum solids content first occurred was used as set point for wet pressing the other sample series at 50°C. The wet pressing at 50°C was done by heating the top and bottom plates to 50°C with heating elements attached inside the pressing cones.

smooth polished surface of the top plate as the nip opened thus minimising rewetting. 20 ms after the nip opened, a vacuum was generated in the bottom plate to clean and dry the porous plate.

To determine the pressure set point where the nip was reasonably flow controlled for wet pressing samples at 50°C, F15T85 samples were wet pressed with 5 different pressures at 25°C. The solids content versus pressure response curve was constructed (Fig 3). The pressure at 25°C when the maximum solids content first occurred was used as set point for wet pressing the other sample series at 50°C. The wet pressing at 50°C was done by heating the top and bottom plates to 50°C with heating elements attached inside the pressing cones.

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As expected, the addition of fixative and retention aid yields an even distribution of filler particles in the sheets thickness direction (Fig 4). The increase in filler content increases the light scattering coefficients as expected (Fig 11). The 5MFC5F30 sample yields a light scattering coefficient of 75 m²/kg, and registers a tensile index of 30 kNm/kg.

Results and discussion

As expected, the addition of fixative and retention aid yields an even distribution of filler particles in the sheets thickness direction (Fig 4).

The drainage time (Table 3) was measured while draining the slurry during handsheet making using a PFI handsheet former. The samples used to measure the WRV (Table 4), the pre solids content (Table 4) and for wet pressing experiment were made by dewatering the furnish mixtures with FiberXPress at 1 MPa for 60 seconds.

Tables 3 and 4 show the following trends:

1. The pure TMP sample yields the shortest drainage time and highest water retention values (WRV).
2. Increasing the ash content reduces the amount of fines and fibres in the sample and thus reduces WRV. This increases both the pre pressing solids content (Pre solids) and the solids content after wet pressing (Solids). The low WRV indicates less swelling water in the sample due to less TMP fraction and therefore easier to dewater during sheet forming and wet pressing.
3. Increasing the fibrillation of the MFC, from 3 pass to 5 pass during homogenization, increases the drainage time.
4. The samples that contain 5% 5 passes MFC and 29% ash can be wet pressed to similar solids content compared to F15T85.

Fig 5 shows that the tensile index and apparently the strain at break decrease with increasing ash content for mixture with 3MFC2.5%. Surprisingly no changes are seen in either tensile index or strain at break in 5MFC5% and no change in strain at break is observed in TMP samples when ash content increases. The samples that contain higher concentration of MFC with higher filler content appear to be more brittle than samples made with 100% TMP.

Fig 6, 7 and 8 show the field emission SEM images for F15T85, 3MFC2.5F30 and 5MFC5F30 samples respectively. The MFC with higher number of passes yields more fibrillated material. Increasing the 5 passes MFC concentration enhances the MFC adsorption onto the filler particles (Fig 8). The increased concentration of more fibrillated MFC (5MFC5%) yields tensile index close to 30 kNm/kg even at 29% ash content (Fig 5).

Increasing the ash content increases the density but reduces the tensile index (Fig 9). The 5MFC5F30 (29% ash content) shows the highest sheet density (490 kg/m²) while the tensile values are still reasonably high, 30 kNm/kg. The higher amount of highly fibrillated, swollen and flexible MFC in the 5MFC5 presumptively fills more crevices between fibre and filler particles. The large coverage caused by an increased amount of nanofibrils adsorsbs more filler particles and bonds them to the fibre network (Fig 8).

As expected, the tensile index for all samples correlated well (r²=85%) with the water retention values (Fig 10), similar to what other researchers have found (Stone et al. 1968; Htun, Ruvo 1978; Scallan, Carles 1972; Scallan 1977).

The increase in filler content increases the light scattering coefficients as expected (Fig 11). The 5MFC5F30 sample yields a light scattering coefficient of 75 m²/kg, and registers a tensile index of 30 kNm/kg.

Sheets with MFC and 15% ash content exhibits higher z-direction strength when compared to TMP sheets with 15% ash content (Fig. 12). 5MFC2.5F30 and 3MFC2.5F30 yields similar z strength as F15T85. 5MFC5F30 samples yields highest z direction strength at 511 kPa.
Fig 5. Tensile index vs. strain at break. The ash content is labelled beside each data point. The error bars are 95% confidence intervals.

Fig 6. FESEM image for the surface of F15T85 samples. The surface images were acquired at low (Left) and high (Right) magnification.

Fig 7. FESEM images for the surface of 3MFC2.5F30 samples. The surface images were acquired at low (Left) and high (Right) magnification.

Fig 8. FESEM images for the surface of 5MFC5F30 samples. The surface images were acquired at low (Left) and high (Right) magnification.
Subramaniam (2008) found that increasing filler and microfines in chemical pulp handsheet improved internal bonding strength. Nanko, Ohsawa (1989) observed that fibrillar fines filled gaps between fibres in wet pressed sheets. The more fibrillated 5MFC5% contains more nanofibrils and larger specific surface area than 3MFC2.5% and 5MFC2.5%. The MFC readily adsorbs onto the fillers and fibres and improves packing and bonding (Fig. 7 and 8) during wet pressing. This increases the z strength of 5MFCF30 as compared to F15T85 (Fig. 12).

**Gurley versus TMP, filler and MFC (Quadratic mixture models)**

The Gurley is modelled with quadratic mixture models in relation to ash content, TMP and MFC (3 and 5 passes) fractions (Table 3).

At 95% confidence level, the addition of MFC is found to significantly increase Gurley or air permeance (P<0.05). The quadratic mixture model showed that the 5 passes MFC fraction has the most positive effect in increasing the Gurley. In mixture that contains 5 pass MFC, the model shows significant effects from interaction components, MFC-ash content (P<0.05) and MFC-TMP (P<0.05). The analysis of variance shows that the error component contributes less than 1% of the detected total variations detected.
Conclusions

Optimal selection of MFC quality and filler content may maintain or improve strength properties without affecting the pressability of the sheet in the wet end. Filler-MFC-fibre interactions simultaneously improve both the light scattering and the strength properties of a given sheet. Increasing both filler and more fibrillated MFC concentration increased the density and the air resistance. MFC readily adsorbs onto the filler particles and fibres thus binding the fillers effectively with the fibre network. MFC produced through higher number of passes, 5 passes in this study, at 5% concentration improved the z direction strength even at 30% filler loading. This study shows the potential use of MFC and filler for engineering sheet structures with optimal properties, i.e. strength, light scattering and air resistance, without impeding dewatering at press.

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