

Effect of Pre-flocculation of Lime Mud CaCO₃ Filler on AKD Sizing Efficiency

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Lime mud (LM), whose main component is calcium carbonate, is a by-product in the pulp and papermaking industry. For non-wood pulping processes, large amounts of LM are produced every year, and a portion of them has been used as a paper filler. However, LM significantly lowers alkyl ketene dimer (AKD) sizing efficiency when added to paper. In this study, LM slurry was first pre-flocculated using some commonly used wet-end additives; then, the pre-flocculated LM was added to the pulp for handsheet preparation. The effect of LM pre-flocculation on AKD sizing efficiency was investigated by means of Cobb₆₀ value, contact angle, and sizing reversion. Finally, the AKD adsorption, particle size, BET surface area, BJH pore volume, and zeta potential of native and pre-flocculated LM were measured. It was found that handsheets filled with pre-flocculated LM had lower Cobb₆₀ values and larger contact angles compared to handsheets filled with native LM. The sizing reversion was also alleviated to a certain extent. This was probably because the BET surface area and BJH pore volume of pre-flocculated LM were lower than native LM and the zeta potential was higher than native LM, which led to a much less adsorption of AKD. Thus, AKD sizing efficiency was significantly improved.

Keywords: Lime mud; Sizing efficiency; Adsorption

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INTRODUCTION

Lime mud (LM) is a by-product of the alkali recovery process in the pulp and papermaking industry. There are two kinds of LM due to different pulping raw materials. LM coming from wood materials can be calcined and recirculated into the causticizing process, while LM from non-wood materials such as bamboo, reed, or wheat straw cannot be recycled because of a serious silica problem. The silica content of non-wood LM is usually around 10%. However, to recycle LM by means of calcination, the silica content is required to be lower than 5%. High silica content leads to high energy consumption and low calcining efficiency (Zhang 2007). In China, non-wood materials account for a large proportion of pulping raw materials. Thus, large amounts of non-wood LM are produced every year and become solid waste. How to deal with these solid wastes without damaging the environment has become a challenge for papermakers. Considering its lower cost, and the fact that the main component of LM is amorphous calcium carbonate (CaCO₃, Fig. 1(a)), LM has been used in some Chinese paper mills as a partial or complete substitute for commercial precipitated calcium carbonate (PCC) as paper filler.

From pilot scale tests (Wang 2008; Pan *et al.* 2009) and laboratory experiments (He *et al.* 2014), it was confirmed that LM can be partially used as a paper filler. However, some problems hinder its practical application. The most serious one is that LM can severely lower alkyl ketene dimer (AKD) sizing efficiency compared to conventional PCC. Wang *et al.* (2014) investigated this effect and found that irregular LM (irregular in shape) had higher Brunauer, Emmett, Teller (BET) surface area and Barrett-Joyner-Halenda (BJH) cumulative desorption pore volume, resulting in higher amounts of AKD adsorption and less AKD remaining on fibers.

Pre-flocculation is a technique that aggregates filler particles by means of one or more kinds of polyelectrolytes before adding them to pulp in the papermaking process. Studies on filler flocculation kinetics have been carried out by many research teams (Gaudreault *et al.* 2009; Peng and Garnier 2012). Sang *et al.* (2012) investigated the dynamics of the flocculation of PCC with two commercial cationic tapioca starches of different charge densities. They found that larger and more compact flocs were produced by high charge density starches. Nasser *et al.* (2013) showed that at optimum polymer concentrations, the kaolinite floc sizes were larger and the settling rates were greater in the presence of anionic polyacrylamide (APAM) than cationic polyacrylamide (CPAM) at the same molecular weight (3 to 4 million g/mol) and charge density (35%). This is because the electrostatic attractions between positively charged CPAM and negatively charged kaolinite produced strong and less compressible floc structures. In the case of APAM, the interactions appeared to be weakened as a consequence of electrostatic repulsion between the negatively charged APAM and negatively charged kaolinite, which led to large open-structure flocs having less resistance to compression loads.

Filler pre-flocculation can lead to larger aggregates (Fig. 1(b,c)), which facilitates filler retention in the handsheet (Sang *et al.* 2012). Moreover, the strength of paper can also be improved by filler pre-flocculation due to the reduction of the apparent surface area of filler and less disruption of inter-fiber bonding (Yan *et al.* 2005; Zhao *et al.* 2005; Petersson 2011), especially for highly filled paper. Chauhan and Bhardwaj (2013) showed that when the talc filler was pre-flocculated by 0.4% cationic starch, it increased the first pass ash retention by 2.86% at a 35% filler addition level. The increase in tensile and burst index of paper filled with 0.8% starch pre-flocculated talc at 24% ash level was about 11.2% and 23%, respectively. A dual polymer system was also employed to pre-flocculate PCC, and it was found that the mechanical strength of the sheets increased significantly at a constant filler dosage due to the larger filler flocs (Cheng and Gray 2009).

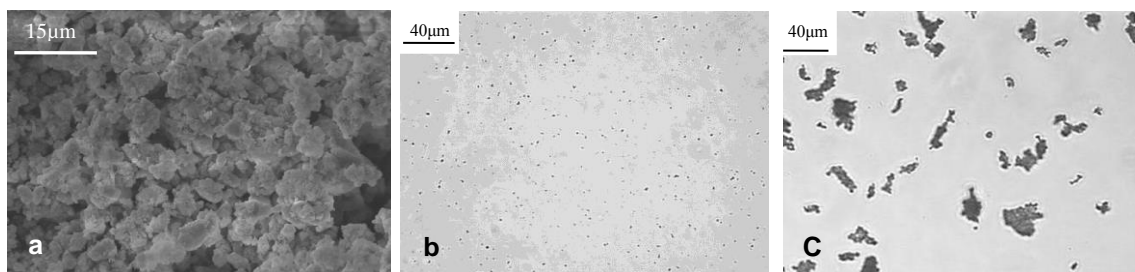


Fig. 1. SEM image of LM at 3000x magnification (a) and microscope images of filler before (b) and after (c) flocculation

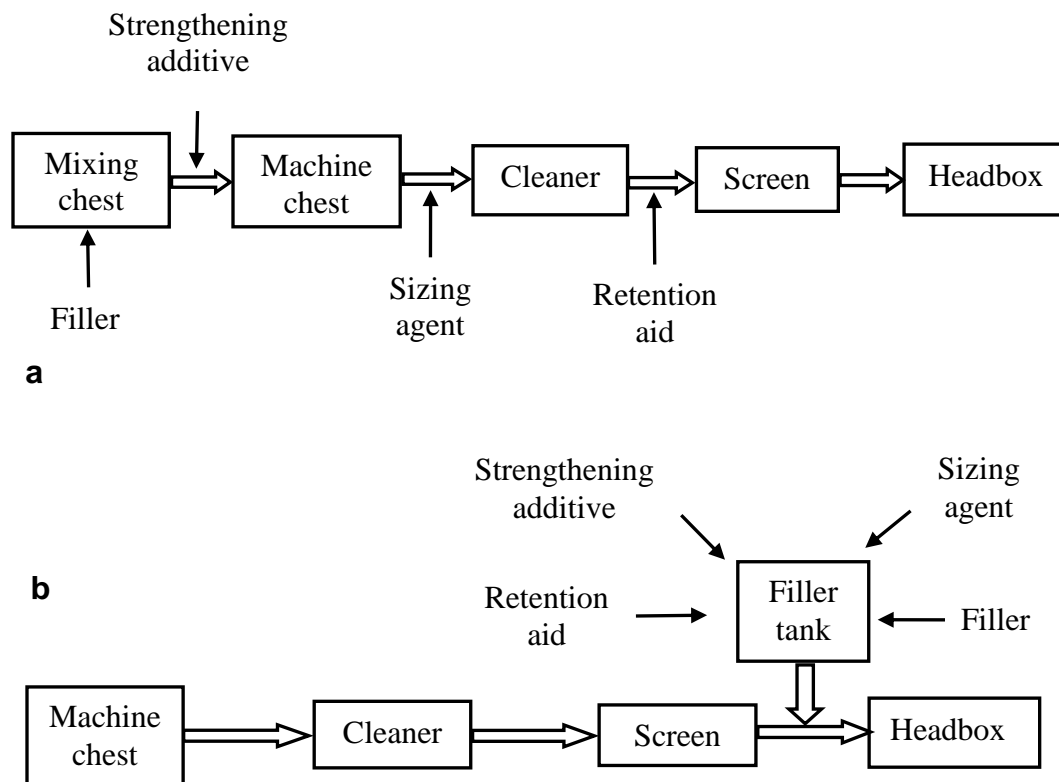


Fig. 2. Traditional (a) and pre-flocculation (b) production process; the chemicals in Fig. 2(b) were added sequentially

In this study, a novel and simple pre-flocculation process was employed. Specifically speaking, the LM was pre-flocculated by several commonly used wet-end additives, including a retention aid, dry-strength additive, and sizing agent, before it was added to the pulp for handsheet preparation, which was different from the traditional production process that the filler and other functional additives were added at different positions in the approach flow system (Fig. 2). Consequently, the pre-flocculation process had the following advantages: increased filler retention; simplified approach flow system; short response time for process changes; reduced filler loss in cleaner and screener; and reduced abrasion of units in the paper machine (Matula 2008; Pan *et al.* 2012). However, its influence on paper sizing efficiency is still unknown. The objective of this study was to evaluate the effect of LM pre-flocculation on AKD sizing efficiency. The hydrophobicity, contact angle, and sizing reversion of handsheets filled with native and pre-flocculated LM were compared. In addition, the AKD adsorption capacity, particle size, BET surface area, BJH pore volume, and zeta potential of native and pre-flocculated LM were also measured.

EXPERIMENTAL

Materials

The LM (charge density = -5.75 mmol/kg), softwood bleached kraft pulp (SWBKP), alkaline peroxide mechanical pulp (APMP), reed bleached kraft pulp (RBKP), CPAM (average molecular weight ≈ 8 MDa, charge density = 1.186 meq/g), cationic

starch (degree of substitution = 0.04), and AKD (solid content = 17.46%, dispersed by lignosulfonate, average particle size = 0.89 μm) were obtained from a paper mill (Hunan, China). The starch solution (1%) was cooked at 95 °C for 30 min in advance. The LM was used as received without washing.

Methods

Pre-flocculation of LM

The LM suspension of 0.1% concentration was dispersed in a beaker at 600 rpm by a magnetic stirrer. Then, 0.04% CPAM, 0.75% cationic starch, and 0.25% AKD were added to the LM slurry successively. The resulting mixture was further stirred for 10 min. The dosage of all additives was based on the weight of dry pulp.

Characteristics of LM and pre-flocculated LM

The particle sizes of LM and pre-flocculated LM were determined by a Mastersizer 3000 particle size analyzer (Malvern, UK) without ultrasonic dispersion. The BET surface area and BJH desorption pore volume were determined by nitrogen adsorption using a Micromeritics ASAP 2020 adsorption instrument (USA). The zeta potential of native and pre-flocculated LM were measured by a SZP-06 zeta potential tester (BTG, Germany).

Adsorption of AKD on native and pre-flocculated LM

Various amounts of AKD were added to the filler suspension containing 0.1 g of LM or pre-flocculated LM. Then, the volume of the mixture was adjusted to 50 mL by adding deionized water and stirred for 10 min. Finally, the mixture was centrifuged for 10 min at 3000 rpm. The supernatant liquor was used to determine the absorbance at 238 nm with a Hach DR 5000 ultraviolet spectrophotometer (USA). The content of AKD in the supernatant liquor was calculated according to the standard curve. The AKD absorbed by LM or pre-flocculated LM was equal to the difference between the loading dosage of AKD and the content of AKD in the supernatant liquor.

Handsheets preparation and testing

Approximately 2 g of dry pulp (SWBKP: APMP: RBKP = 2: 3: 2) was defibered in 1500 mL of water. For pre-flocculated LM, a pre-flocculated LM slurry including LM and other additives was poured into the pulp and stirred. For native LM, certain amounts of LM, 0.75% cationic starch, 0.25% AKD, and 0.04% CPAM were added to the pulp successively and stirred. Handsheets were prepared with a Rapid-Köthen sheet former (Austria) at 90 °C and 1.0 MPa for 10 min according to GB/T 24326-2009. The dosage of all additives was based on the weight of dry pulp. The filler loading levels were 13.5%, 19%, 25%, 31.5%, and 39% based on the weight of dry pulp. Paper samples were conditioned at (23±1)°C and (50±2)% relative humidity for 24 h prior to testing.

The Cobb₆₀ value of paper was measured using a Cobb sizing tester (China) as per GB/T 1540-2002 and carried out in quadruplicate. The error bars shown in the following figures represent the standard deviation of the four measured values on either side of the mean. The contact angle of handsheets was measured by a Dataphysics OCA 20 video-based contact angle measuring device (Germany). The ash content was determined in a muffle furnace (Nabertherm, German) at 525 °C according to GB/T 22877-2008.

RESULTS AND DISCUSSION

Effect of Pre-flocculation of LM on Hydrophobicity of Handsheet

It is widely known that the presence of PCC in paper deteriorates AKD sizing efficiency. However, LM has a more negative effect on AKD sizing efficiency than commercial PCC. Wang *et al.* (2014) showed that at a filler dosage of 30%, the $Cobb_{60}$ value of paper filled with commercial PCC was 24.3 g/m^2 while the $Cobb_{60}$ values of paper filled with LM from three paper mills were 70.2, 86.1, and 123.4 g/m^2 , respectively. The lower the $Cobb_{60}$ value, the better the hydrophobicity of the handsheet and the higher the AKD sizing efficiency. Figure 3 shows the $Cobb_{60}$ values of paper filled with native and pre-flocculated LM at different ash contents. The AKD was added at a constant dosage of 0.25%. As expected, with an increase in ash content from about 10% to 23%, the $Cobb_{60}$ value of paper filled with native LM increased by more than three times; however, the $Cobb_{60}$ value of paper filled with pre-flocculated LM increased only slightly. This indicated that filler loading had less of a negative effect on AKD sizing efficiency in pre-flocculated LM-filled paper than in native LM-filled paper. Moreover, the $Cobb_{60}$ value of paper filled with pre-flocculated LM was lower than that of paper filled with native LM at an ash content within 10% to 23%. Especially at filler loading levels of 31.5% and 39% (corresponding to ash contents of around 20% and 23%), the $Cobb_{60}$ values of paper filled with native LM were 92.0 and 114.1 g/m^2 , respectively, while the $Cobb_{60}$ values of paper filled with pre-flocculated LM were 27.3 and 34.2 g/m^2 , decreasing by 70.3% and 70%, respectively. Thus, the lower $Cobb_{60}$ values for pre-flocculated LM-filled paper demonstrated an improvement in the hydrophobicity of paper and AKD sizing efficiency.

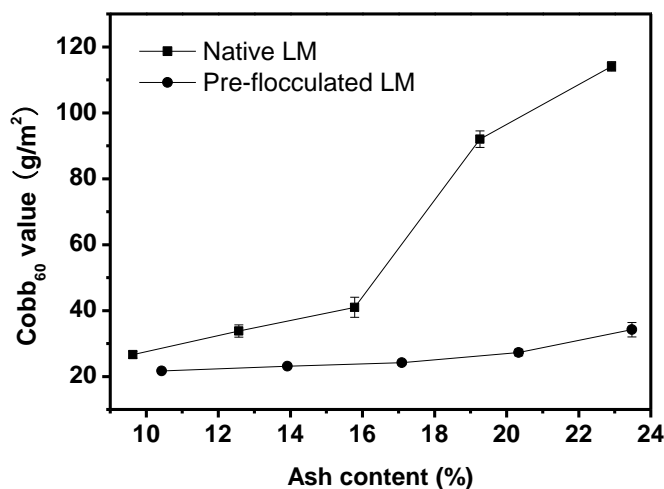


Fig. 3. Effect of pre-flocculation of LM on $Cobb_{60}$ value of paper at different ash contents

Other researchers also worked on improving the AKD sizing efficiency of paper filled with LM. Hu *et al.* (2012) modified LM with a starch-sodium stearate complex and found that after modification the sizing degree of paper increased by 17.4% and 45.5% at filler loading levels of 30% and 38%. Compared to their method, the pre-flocculation technology adopted in this study had a better effect, lower cost, simplified operability, and more space for application in actual production.

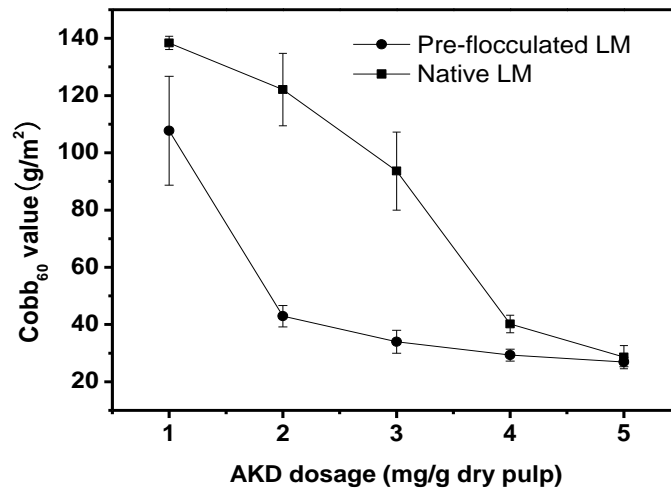


Fig. 4. Effect of pre-flocculation of LM on Cobb₆₀ value of paper at different AKD dosages

An increase in AKD dosage will also lead to a decrease in the Cobb₆₀ value of paper. As shown in Fig. 4, when filler was added at a constant level of 30.5%, the Cobb₆₀ value of paper filled with pre-flocculated LM was lower than that of paper filled with native LM for a given AKD dosage lower than 0.5%. When the AKD dosage was around 0.2%, the Cobb values of native and pre-flocculated LM-filled paper were 122.1 and 42.9 g/m², respectively. The latter is 64.9% lower than the former. Results from the two figures confirmed that the pre-flocculation of LM improved the AKD sizing efficiency compared to native LM.

Effect of Pre-flocculation of LM on Contact Angle of Handsheets

In addition to the Cobb₆₀ value, the contact angle at the water and paper interface is another parameter used to characterize the hydrophobicity of paper and sizing efficiency. A larger contact angle signifies a higher sizing efficiency. The change in contact angle with time shows the stability of the water resistance ability of the paper, which was shown in Fig. 5 over a total of 60 s. When filler was added at dosages of 13.5% and 19%, the contact angle of paper filled with pre-flocculated LM remained nearly constant, while that of paper filled with native LM decreased slightly. At higher filler loading levels of 25% and 31.5%, the contact angle decreased slightly for pre-flocculated LM-filled paper, but that of native LM-filled paper decreased to 0° within 37 and 20 s, respectively. When the filler loading level was 39%, the contact angle of paper filled with pre-flocculated LM was in the range of 100° to 11.8° over 60 s, compared to 100.9° to 0° over 9 s in the case of paper filled with native LM.

Figure 6 shows pictures of contact angle of a water droplet on paper taken after 30 s of penetration time. It could be seen that the water droplet on 25%, 31.5%, and 39% pre-flocculated LM-filled paper penetrated partly, while the water droplet on 25%, 31.5%, and 39% native LM-filled paper almost fully penetrated into the paper. These results further proved that the pre-flocculation of LM improved the AKD sizing efficiency compared to native LM, especially for highly filled paper, in accordance with the conclusion drawn from the Cobb₆₀ value.

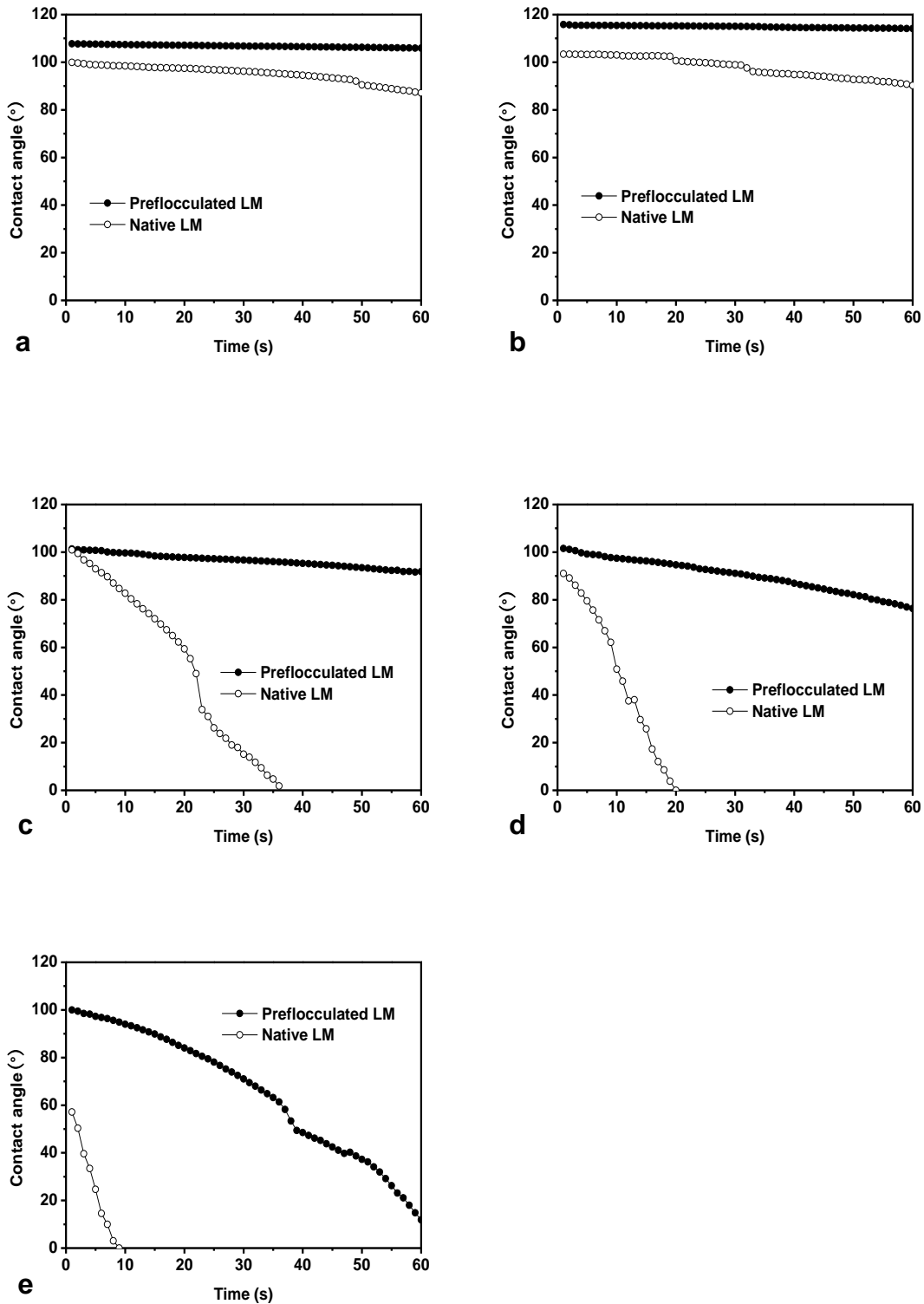


Fig. 5. Effect of pre-flocculation of LM on contact angle as a function of time at different filler loading levels (a) 13.5%; (b) 19%; (c) 25%; (d) 31.5%; and (e) 39%

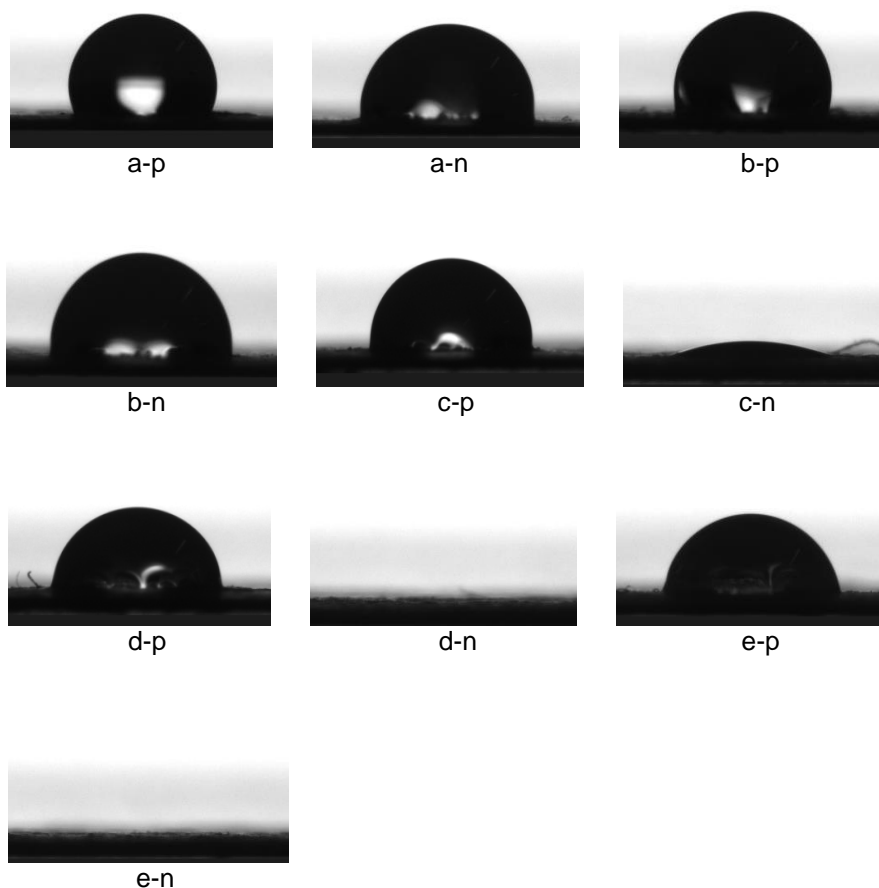


Fig. 6. Contact angle of paper at different filler loading levels after 30 s of penetration time (a-p) 13.5% pre-flocculated LM; (a-n) 13.5% native LM; (b-p) 19% pre-flocculated LM; (b-n) 19% native LM; (c-p) 25% pre-flocculated LM; (c-n) 25% native LM; (d-p) 31.5% pre-flocculated LM; (d-n) 31.5% native LM; (e-p) 39% pre-flocculated LM; and (e-n) 39% native LM

Effect of Pre-flocculation of LM on Sizing Reversion

It is well known that paper filled with CaCO_3 , especially for PCC, tends to lose water repellency to some extent over time after sizing. This phenomenon is called sizing reversion or fugitive sizing. Moyers (1991) used CaCO_3 in AKD sizing process and reported that the portion of the AKD that coated the CaCO_3 particles gave only a temporary sizing effect because the resulting calcium beta keto salt was converted to ketone easily by hydrolysis, which almost did not contribute to the hydrophobicity of paper. Bartz *et al.* (1994) considered that the unreacted AKD molecules tended to migrate into the pore volume of CaCO_3 and resulted in sizing reversion. Colasurdo and Thorn (1992) showed that the adsorption ability and alkalinity of filler had an influence on sizing reversion because the residual alkali contained in the filler could promote AKD hydrolysis. In order to prevent sizing reversion to a certain extent in actual production, the appropriate AKD dosage was within the range of 0.12% and 0.25% and the filler loading level had better be no more than 30% based on the weight of dry pulp (Liu *et al.* 2012). In this study, the effect of pre-flocculation of LM on sizing reversion was also investigated and shown in Fig. 7.

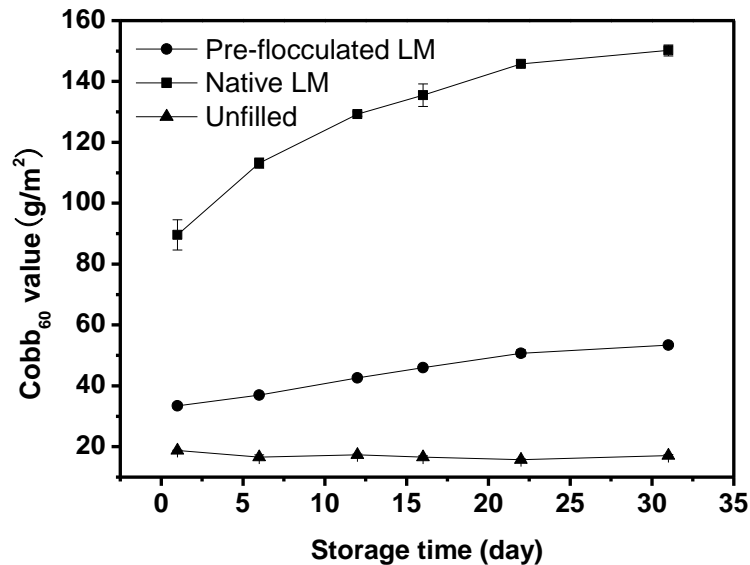


Fig. 7. Effect of pre-flocculation of LM on sizing reversion at a filler loading level of 31.5%

It was found that the Cobb₆₀ value of unfilled paper remained almost the same over one month, while that of paper filled with LM increased obviously. With respect to paper filled with native LM, the Cobb₆₀ value increased rapidly from 89.6 to 145.8 g/m² over 24 days, corresponding to a sizing loss of 62.7%. However, when it turned to paper filled with pre-flocculated LM, the Cobb₆₀ value varied from 33.5 to 50.7 g/m², increasing by 51.3%. In addition, the Cobb value of the latter was always about 60% lower than that of the former during the whole storage period. Therefore, it could be concluded that the pre-flocculation of LM can alleviate the sizing reversion to a certain extent.

AKD Adsorption of Native and Pre-flocculated LM

Table 1. Characterization of Native and Pre-flocculated LM (without AKD)

Fillers	Average particle size (μm)	Total BET surface area (m ² /g)	BJH pore volume (cm ³ /g)	Zeta potential (mV)
Native LM	5.68	7.531	5.720	-99.5
1# Pre-flocculated LM	89.1	0.532	1.719	218.6
2# Pre-flocculated LM	70.6	2.578	3.100	160.1
3# Pre-flocculated LM	60.7	2.745	3.029	72.7
4# Pre-flocculated LM	53.6	3.217	3.403	54.4
5# Pre-flocculated LM	50.2	3.344	3.497	-80.5

Many scholars have studied the effect of filler on AKD sizing efficiency. It is generally believed that filler has a larger surface area than plant fibers and adsorbs a portion of sizing agent. As a consequence, less sizing agent is retained on fibers, resulting in a lower sizing efficiency (Lindström and Larsson 2008; Bartz *et al.* 2014). Wang *et al.* (2014) prepared several kinds of LM with different structures as paper filler and found that the greater amount of AKD adsorbed by LM, the lower the AKD sizing efficiency will be. He *et al.* (2014) also considered that reducing the amount of AKD absorbed by fillers will help to improve AKD efficiency in filled paper. Therefore, the amount of AKD adsorbed by native and pre-flocculated LM was measured, and results are shown in Fig. 8. For distinction and comparison, 13.5%, 19%, 25%, 31.5%, and 39% LM pre-flocculated by 0.04% CPAM and 0.75% cationic starch were named 1#, 2#, 3#, 4#, and 5# pre-flocculated LM, respectively. It was found that the amount of AKD absorbed by native LM was 3 to 4 times as much as the amount of AKD absorbed by pre-flocculated LM. These results likely explain why the LM pre-flocculation can significantly improve the AKD sizing efficiency. Because less AKD adsorbed by fillers means that more AKD remains on fibers at a constant dosage of AKD and contributes more to effective sizing.

The properties of fillers have an important influence on AKD adsorption. For further study, the properties of native and pre-flocculated LM were compared. As shown in Table 1, the pre-flocculated LM had larger particle size, lower BET surface area, and BJH pore volume than native LM. Filler particles with lower surface area result in less AKD adsorption (Lindström and Larsson 2008; Wang *et al.* 2014). Also, a portion of AKD molecules adsorbed by the filler can migrate into the pore structure of LM during the drying process (Bartz *et al.* 1994). The lower BJH pore volume means that there will be less capacity to accommodate AKD.

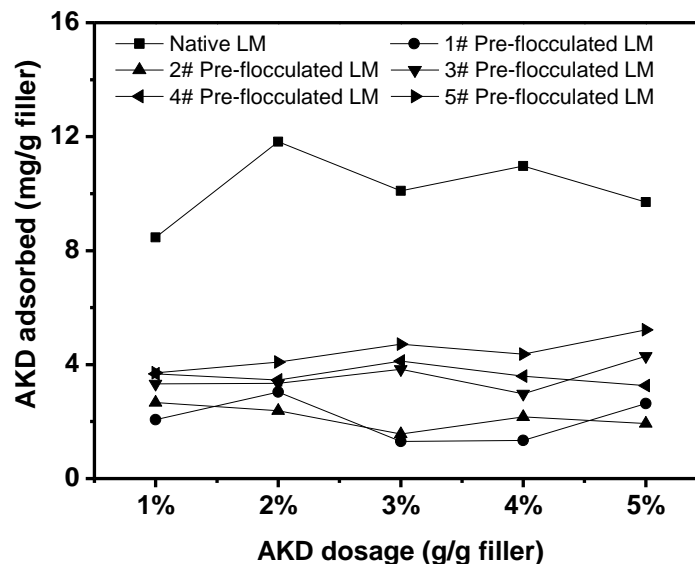


Fig. 8. Effect of pre-flocculation of LM on AKD adsorption

Besides the morphology effect, the AKD adsorption may also be affected by the zeta potential of filler particles. Table 1 also shows the zeta potential of native and pre-flocculated LM. It was found that the zeta potential of pre-flocculated LM was higher

than that of native LM and increased drastically with the decrease of filler dosage. This is due to the adsorption of positively charged additives onto the surface of LM during the pre-flocculation process. Increased zeta potential will also lead to less AKD adsorption by charge adsorption. As a result, the AKD sizing efficiency is significantly improved.

CONCLUSIONS

1. The Cobb₆₀ value decreased and the contact angle increased for paper filled with pre-flocculated LM compared to paper filled with native LM. The difference was much bigger in highly filled paper. The pre-flocculation of LM significantly improved the AKD sizing effect and alleviated the sizing reversion to a certain extent in comparison with native LM
2. Pre-flocculated LM had larger particle size, lower BET surface area, lower BJH pore volume, and higher zeta potential than native LM, resulting in much less adsorption amount of AKD. So that the AKD sizing efficiency was significantly improved.

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