Lime mud CaCO$_3$ for use as a filler material in papermaking: Impact of its preflocculation with cationic polyacrylamide

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ABSTRACT: Cationic polyacrylamide with different molecular weights were used to preflocculate the lime mud (LM) before it was added to the paper stock for handsheet preparation. The particle sizes, ζ potential, and morphology of the unpreflocculated and resulting preflocculated LM were studied. We found that high-molecular-weight cationic polyacrylamide (H-CPAM) led to larger LM flocs with a more positive ζ potential. The scanning electron microscopy images indicated that the morphological structure of the filler hardly changed during the preflocculation process. The effects of the preflocculation on the filler retention and paper properties were also investigated. The results show that the handsheets filled with preflocculated LM had better hydrophobicity and strength properties compared to handsheets filled with unpreflocculated LM, especially for H-CPAM-preflocculated LM. The paper formation was also improved, and the optical properties nearly remained the same. In addition, the LM preflocculated with H-CPAM had the highest filler retention. At a filler loading of 30%, the filler retention of the H-CPAM-preflocculated LM was higher than 86; it was less than 82.5% in other cases. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 41640.

INTRODUCTION

Lime mud (LM) is a byproduct of the alkali recovery process in the pulp and papermaking industry. Its main component is calcium carbonate (CaCO$_3$). LM coming from the wood-pulping process can be recycled by means of calcination. However, LM coming from a non-wood-pulping process cannot be reused because of the silica problem.[1] The silica content of nonwood LM is usually much higher than that of wood LM. A high silica content not only led to a low calcining efficiency and a high energy consumption, but it also results in silica accumulation. Therefore, some Chinese paper mills have used nonwood LM as a partial or complete substitute for commercial precipitated calcium carbonate (PCC) as a paper filler; this will provide economic and environmental benefits and has vast application prospects.[2–4]

In the papermaking industry, the use of mineral fillers in paper production is a common practice. Many benefits can be achieved as a result of filler addition; these include savings in energy and costs[5–7] and improvements in the optical properties,

dimensional stability, printability, and writability of paper.[7–9] In addition, the use of fillers can confer some unique functions,[10] such as photocatalytic properties,[11–13] flame retardant properties,[14] antibacterial properties,[15] and electrically conductive properties.[16]

However, the addition of fillers also brings some disadvantages, especially at high filler loading levels. The presence of fillers interferes with the fiber–fiber bonding. As a result, the paper strength decreases.[7,17] Other drawbacks associated with the increased filler loading include poor filler retention and decreased sizing efficiency.[5,18] To compensate or alleviate these disadvantages, various methods have been explored and proposed by researchers, such as lumen or cell wall loading,[19,20] fine-fiber complexation,[21,22] fiber modification,[23] filler modification,[5,18,24–32] and filler preflocculation.[33–36]

Filler preflocculation is a technique in which filler particles are aggregated through polymers before they are added to the paper stock. Starch and polyacrylamide are commonly used polymers in filler preflocculation. Compared to the other methods mentioned

Table I. ζ Potentials and Average Particle Sizes of the Unpreflocculated and Preflocculated LM

Sample	ζ potential (mV)	Average particle size (μm)
Unpreflocculated LM	-50.3 ± 1.9	6.49 ± 0.91
LM preflocculated with 0.2% L-CPAM	49.9 ± 1.4	69 ± 2.8
LM preflocculated with 0.2% H-CPAM	88.5 ± 1.7	104.4 ± 3.1

previously, the filler preflocculation technique is more practical and can be readily accomplished in actual production because of its brief process and relatively low cost. This attracts considerable interest from researchers accordingly. Sang et al.[33] preflocculated PCC with cationic starch. The preflocculation was found to provide stronger paper compared to a conventional starch/retention aid addition sequence. Chauhan and Bhardwaj[34] showed that the use of talc, preflocculated with cationic starch, resulted in a relatively higher filler retention, paper strength, and comparable optical properties of paper. Seo et al.[35] found that the use of ground calcium carbonate flocs induced by cationic polyacrylamide (CPAM) increased the tensile strength of the handsheets by 15% without a loss in brightness. Although research on filler preflocculation have been extensively carried out, little literature has been found that has focused on LM; this had different properties from PCC or ground calcium carbonate.[37] Therefore, it is of interest to investigate what effect can be achieved with the preflocculation of LM.

In this study, CPAM with different molecular weights was used to preflocculate the LM suspension before it was added to the paper stock for handsheet preparation. The ζ potential, particle size, and morphology of the unpreflocculated and preflocculated LM were measured. In addition, the effects of the preflocculation of LM with CPAMs of different molecular weights on the filler retention and paper properties were also studied.

EXPERIMENTAL

Materials

The LM, bleached softwood kraft pulp (SWBKP), alkaline peroxide mechanical pulp (APMP), bleached reed kraft pulp (RBKP), cationic starch with a degree of substitution of 0.04, and alkyl ketene dimer (AKD) emulsion with a solid content of 17.46% were all obtained from a paper mill (Hunan,
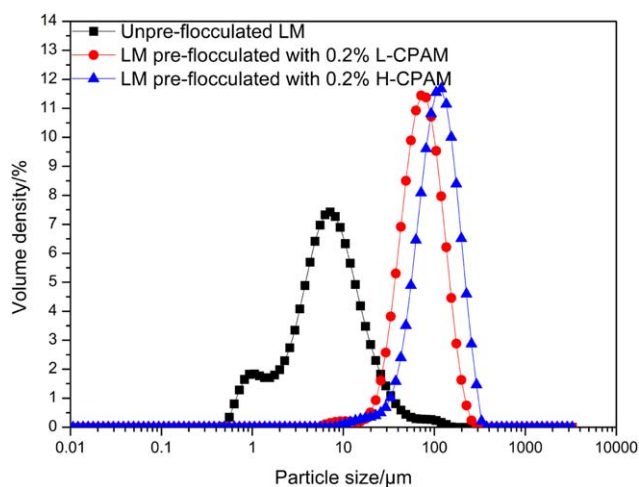

Figure 2. Particle size distributions of the unpreflocculated and preflocculated LM. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

China). The bleached softwood kraft pulp, APMP, and bleached reed kraft pulp were beaten to 40, 58, and 30°SR (where, °SR is the unit of beating degree measured by the Schopper Riegler method, according to GB/T 3332-2004), respectively, with a Mark VI PFI refiner (Norway). The CPAM [high-molecular-weight cationic polyacrylamide (H-CPAM) weight-average molecular weight = 3 million g/mol, charge density = 1.50 mequiv/g, low-molecular-weight cationic polyacrylamide (L-CPAM) weight-average molecular weight = 7 million g/mol, charge density = 1.50 mequiv/g] was supplied by EKA Chemicals Co., Ltd. (Sweden). The starch solution was cooked at 95°C for 30 min at a 1% consistency in advance.

Preflocculation of LM

The LM suspension at a 0.1% concentration was dispersed in a 1000-cm^3 beaker at 500 rpm with a magnetic stirrer. Then, 0.2% (on the basis of the weight of LM) CPAM was added to the LM suspension. The resulting mixture was stirred further for 5 min.

Characterization of the Unpreflocculated and Preflocculated LM

The ζ potential of the unpreflocculated LM and LM preflocculated with CPAMs of different molecular weights were measured by an SZP-06 ζ potential tester (BTG, Germany). The particle size was measured by a Mastersizer 3000 particle size analyzer (Malvern, United Kingdom). The filler suspension was
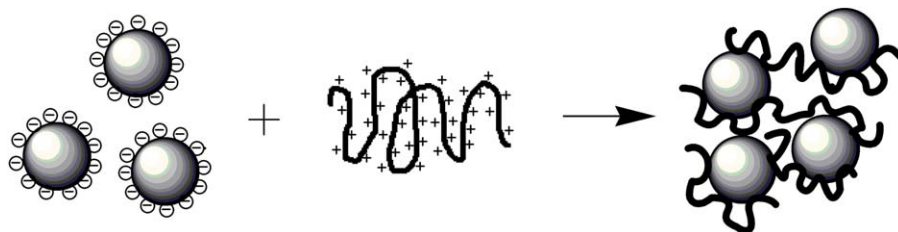

Figure 1. Schematic of the flocculation process between the LM particles and CPAM. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
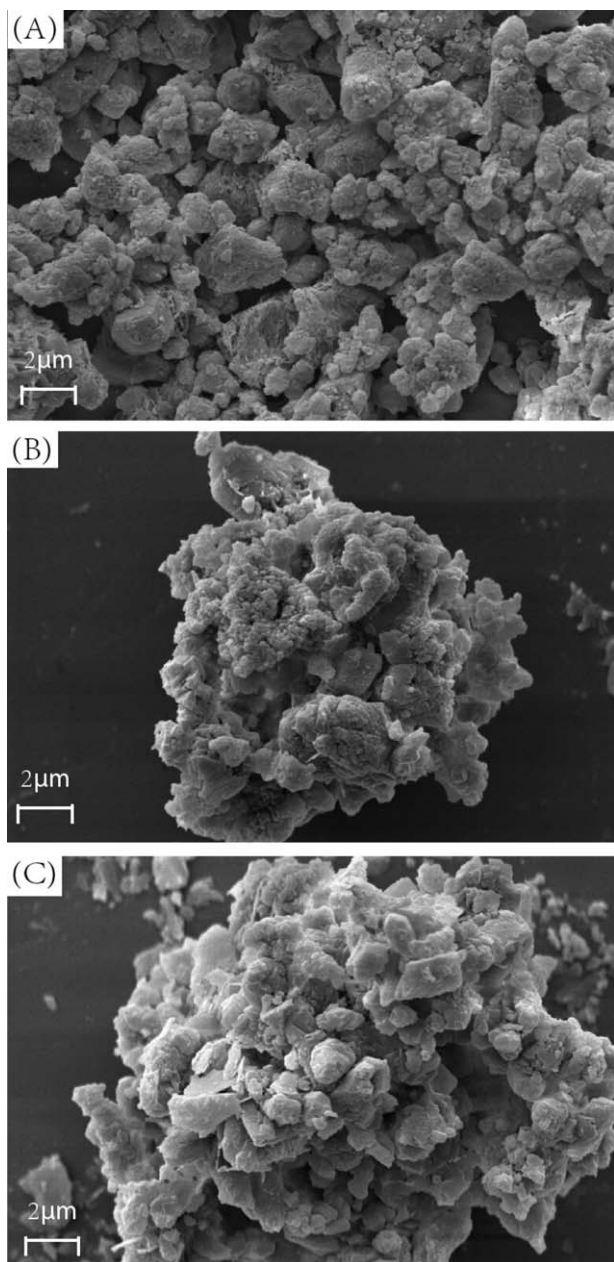
Figure 3. SEM images of the unpreflocculated and preflocculated LM at 5000× magnification: (A) unpreflocculated LM, (B) LM preflocculated with 0.2% L-CPAM, and (C) LM preflocculated with 0.2% H-CPAM.

mechanically stirred at a speed of 3000 rpm in a 1000-cm^3 beaker.

Handsheet Preparation and Testing

Approximately 2 g of dry pulp (SWBKP/APMP/RBKP = 2:3:2) was defibered in 1500 mL of water. For paper filled with preflocculated LM, 0.75% (on the basis of the weight of dry pulp) cationic starch, 0.25% (on the basis of the weight of dry pulp) AKD, and the preflocculated LM slurry was added to the pulp successively and stirred well. For paper filled with unpreflocculated LM, 0.75% (on the basis of the weight of dry pulp) cationic starch, 0.25% (on the basis of the weight of dry pulp) AKD, certain amounts of unpreflocculated LM, and 0.2% (on the basis of the
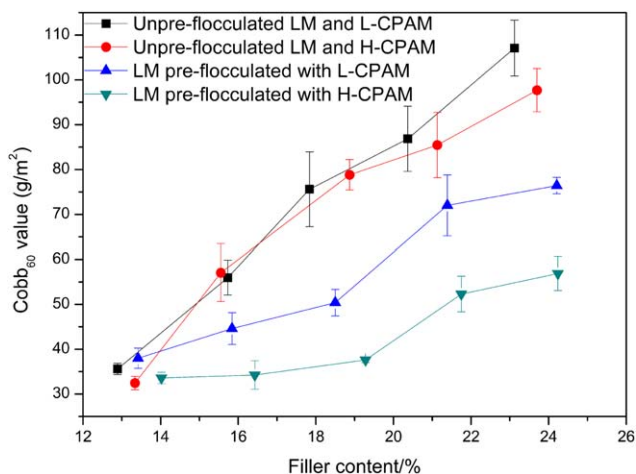

Figure 4. Effect of LM preflocculation with CPAM with different molecular weights on the $Cobb_{60}$ values of paper at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

weight of LM) CPAM were successively added to the pulp and stirred well. Handsheets were prepared by a Rapid-Koethen sheet former (Austria) at 90°C and 1.0 MPa for 10 min. Paper samples were conditioned at 23 and 50% relative humidity for 24 h before testing. The paper properties, including the $Cobb_{60}$ value (where, $Cobb_{60}$ value refers to the mass of the water that is adsorbed by per unit area of paper surface within 60 s), tensile index, burst index, brightness, and opacity, were tested in accordance with GB/T 1540-2002, GB/T 455-2002, GB/T 454-2002, GB/T 7974-2002, and GB/T 1543-2005, respectively. The paper formation index was measured with a two-dimensional laboratory formation sensor (Techpap, France).

We determined the filler content by ashing the filled paper in a muffle furnace (Nabertherm, Germany) according to GB/T 742–2008. The filler retention was calculated with the following formula:
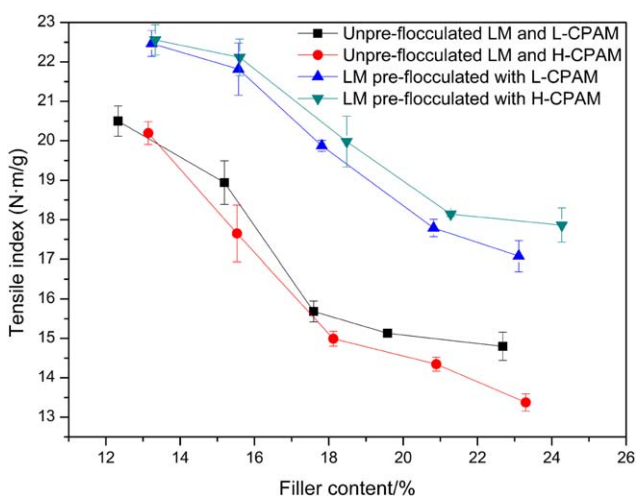

Figure 5. Effect of the LM preflocculation with CPAMs of different molecular weights on the tensile indices of the paper at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
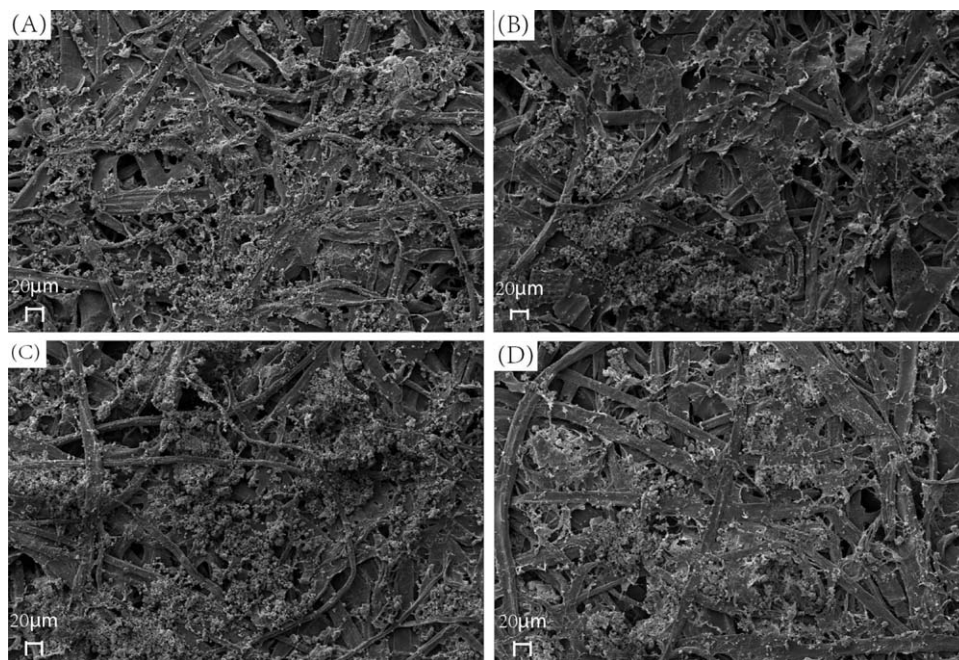
Figure 6. SEM images of the filled paper at 200× magnification: (A) paper filled with unpreflocculated LM and L-CPAM, (B) paper filled with L-CPAM-preflocculated LM, (C) paper filled with unpreflocculated LM and H-CPAM, and (D) paper filled with H-CPAM-preflocculated LM.

$$\text{Filler retention } (\%) = \frac{m_1}{m_2 \times (1-\alpha)} \times 100$$

where m_1 is the weight of ash remained in the paper (g), m_2 is the weight of LM added to the pulp (g), and α is the mass loss fraction of LM (%).

All testings were carried out with three paper samples. A mean value was reported, and the bars shown in the figures represent the standard deviation on either side of the mean value.
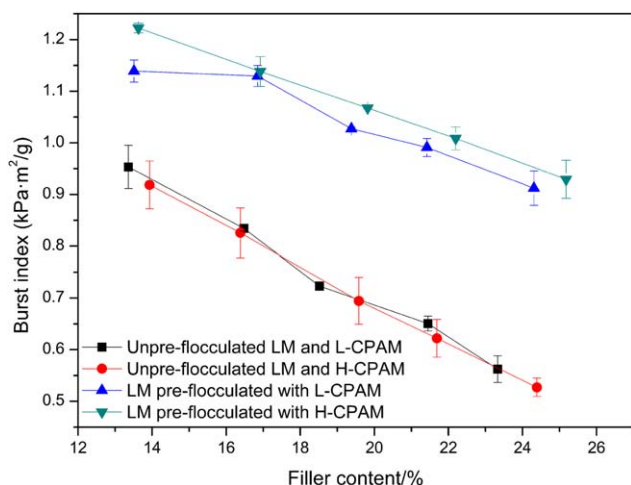
Scanning Electron Microscopy (SEM) Observation

The morphology analysis was performed with an EVO18 scanning electron microscope (Zeiss, Germany) after the spray-gold treatments of the samples.

RESULTS AND DISCUSSION

Characterization of the Unpreflocculated and Preflocculated LM

Table I presents the average particle sizes of the unpreflocculated and preflocculated LM. The results show that the unpreflocculated

Figure 7. Effects of the LM preflocculation with CPAMs of different molecular weights on the burst indices of paper at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
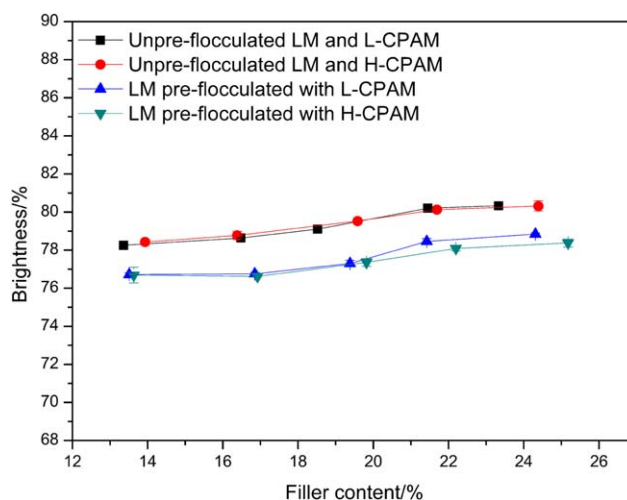

Figure 8. Effects of the LM preflocculation with CPAMs of different molecular weights on the brightness of the paper at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
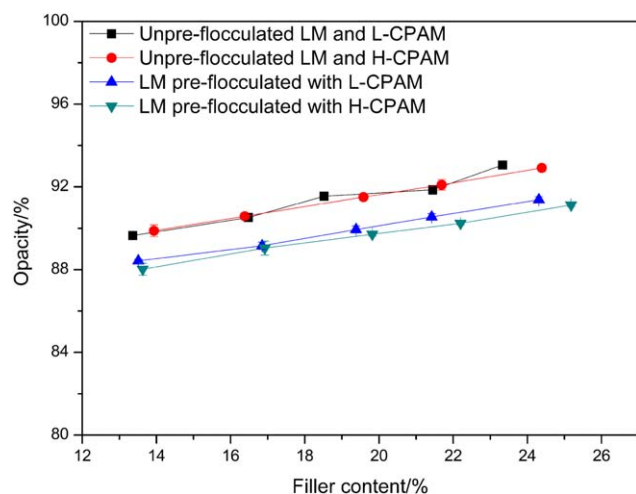
Figure 9. Effects of the LM preflocculation with CPAMs of different molecular weights on the opacity of the paper at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

LM had an average particle size of about 6.49 μm. After preflocculation with L-CPAM and H-CPAM, the average particle sizes of the preflocculated LM increased significantly to around 69 and 104.4 μm, respectively. This could be explained by the schematic of preflocculation as shown in Figure 1. Individual negatively charged filler particles were agglomerated together by positively charged CPAM with a long molecular chain through electrostatic attraction and a bridging function.[38–40] Consequently, large filler flocs were generated. CPAM with a high molecular weight was found to give larger LM flocs, probably because of its longer molecular chain. In addition, both preflocculated LMs had a normal and more narrow particle size distribution than the unpreflocculated LM, as shown in Figure 2.

The ζ potentials of the unpreflocculated and preflocculated LM are also shown in Table I. It was found that the unpreflocculated LM had a ζ potential of about −50.3 mV. After preflocculation with L-CPAM, the ζ potential of the filler particles increased to 49.9 mV, and a higher ζ potential of 88.5 mV was achieved with the introduction of H-CPAM. This was due to the adsorption of positively charged CPAM onto the LM surface during the preflocculation process.

The SEM images of the unpreflocculated and preflocculated LM are shown in Figure 3. It was shown in Figure 3(b,c) that the preflocculated LM mainly consisted of small irregular blocks with a platelike structure. Figure 3(a) shows that the unpreflocculated LM had a similar structure. Therefore, we concluded that the morphological structure of the filler hardly changed during the preflocculation process.

Effect of the Preflocculation of LM on the Hydrophobicity of Paper

Cellulose is hydrophilic with a high surface energy. The porous structure of cellulosic paper acts like a sponge in the presence of liquid. The hydrophobicity of paper was supplied by the sizing process in which a chemical agent [AKD or alkenyl succinic anhydride (ASA)] was used to provide paper and paperboard

with resistance to liquid wetting, penetration, and absorption.[41] The addition of a filler usually has a negative effect on the sizing. It is generally believed that fillers have a larger surface area than plant fibers and adsorbed a portion of sizing agent. As a result, less sizing agent is retained on fibers; this results in a lower sizing efficiency and poorer hydrophobicity of paper.[41–44] The hydrophobicity of paper is usually measured by the $Cobb_{60}$ value. At a constant dosage of sizing agent, the lower the $Cobb_{60}$ value is, the better the hydrophobicity of the paper is. As shown in Figure 4, the $Cobb_{60}$ value of paper filled with preflocculated LM was obviously lower than that of paper filled with unpreflocculated LM at a filler content higher than 14%. In particular, for paper filled with H-CPAM-preflocculated LM, at a filler dosage of 35%, the Cobb value was 52.3 g/m^2 compared to about 85 g/m^2 for unpreflocculated LM-filled paper. This was probably because the preflocculated LM had a higher ζ potential and lower specific surface area than the unpreflocculated LM;[35] it resulted in less adsorption of AKD molecules. Therefore, the sizing efficiency was improved. Compared to the handsheets filled with L-CPAM-preflocculated LM, the handsheets filled with H-CPAM-preflocculated LM had better sizing effects because of the larger flocs and the higher ζ potential produced.

Effect of the Preflocculation of LM on the Paper Strength

Generally, the paper strength is dependent on the fiber strength, the bonding strength between fibers, and the interfiber contact area. Fiber–fiber bonding is primarily attributed to the formation of hydrogen bonds between fibers.[7] Increased filler loading leads to a significant decrease in the paper strength because the filler particles interfere with the fiber–fiber bonding.[25–28] The tensile indices of handsheets filled with unpreflocculated and preflocculated LM at different filler contents are shown in Figure 5. The results indicate that the addition of CPAM-induced preflocculated LM could significantly improve the tensile strength of paper compared to the respective addition of unpreflocculated LM and CPAM into the paper stock. At an 18%
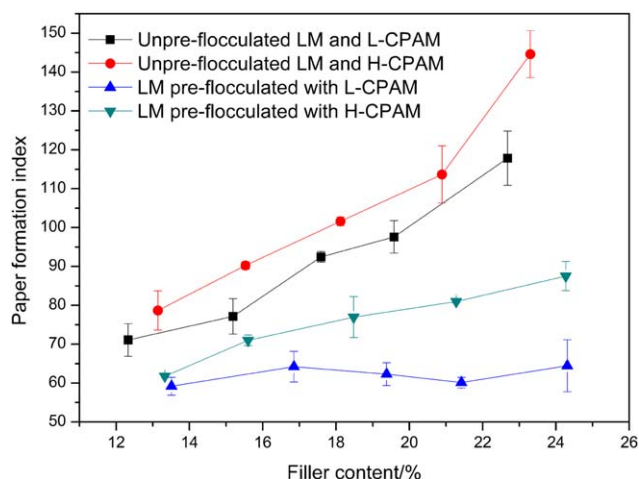

Figure 10. Effects of the LM preflocculation with CPAMs of different molecular weights on the paper formation at different filler contents. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
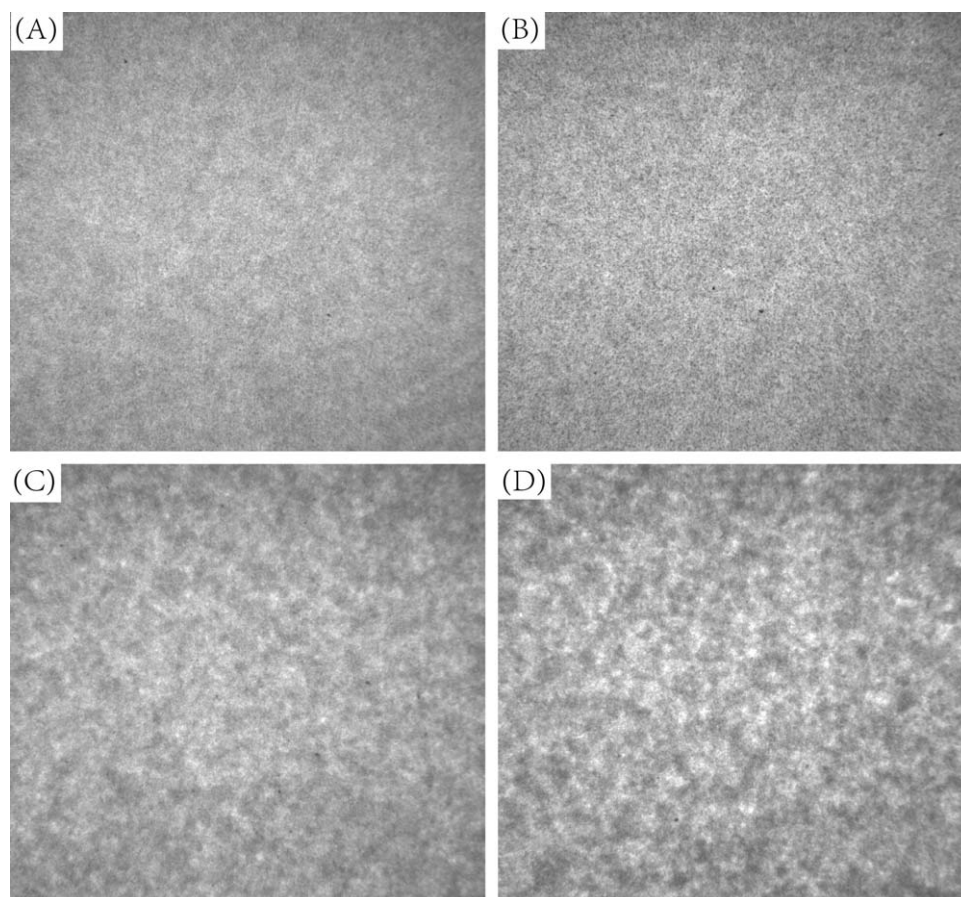
Figure 11. Photographs of the paper filled with unpreflocculated and preflocculated LMs: (A) paper filled with L-CPAM-preflocculated LM, (B) paper filled with H-CPAM-preflocculated LM, (C) paper filled with unpreflocculated LM and L-CPAM, and (D) paper filled with unpreflocculated LM and H-CPAM.

filler content, the tensile strength of the paper with preflocculated LM was approximately 25% higher than that of the paper with unpreflocculated LM. It is worth noting that the size of the filler particles in the handsheet is an important factor in the paper strength. Chauhan et al.[45] and Cao et al.[7] found that the

Figure 12. Filler retention of the unpreflocculated and preflocculated LM.

bigger the filler particles were, the less their negative effect was on the paper strength. After preflocculation, larger and fewer preflocculated LM flocs interfered less with fiber–fiber bonding. Therefore, the paper strength was preserved. H-CPAM was found to give a higher tensile strength, probably because of the larger flocs produced. Figure 6 shows the SEM images of paper filled with unpreflocculated and preflocculated LM. As shown in Figure 6(A,C), the unpreflocculated LM particles had a relatively uniform distribution in the handsheet and tended to be located on the surface of the fibers; this interfered seriously with the fiber–fiber interaction. As shown in Figure 6(C,D), we found that larger and fewer LM flocs appeared clustered and mainly existed in the pore space of the fiber web; this resulted in less interference with the fiber–fiber interaction. Consequently, the paper strength was improved. Similar results were found with respect to the burst index, as shown in Figure 7.

Effect of the Preflocculation of LM on the Optical Properties of Paper

It is widely accepted that the optical properties of paper can be improved with the addition of filler because of its high reflectivity. The preflocculation of the filler particles formed aggregates, so the optical properties were worse than those of paper filled with unpreflocculated filler particles.[7] Figure 8 shows the

brightness of paper filled with unpreflocculated and preflocculated LM. Unexpectedly, we observed that the brightness of paper containing preflocculated LM was just slightly lower than that of the paper containing unpreflocculated LM at the same filler content; this corresponded to about a 1% decrease. The opacity was also investigated and is shown in Figure 9; this was similar to the results of brightness. Therefore, the preflocculation process did not have a strong influence on the paper optical properties. This was probably because the preflocculated LM flocs had a porous structure, and much space existed between the particles. So the loss of the particle ability to refract light was minimized.[34] As a result, the brightness and opacity of paper decreased little after LM preflocculation. Chauhan et al.[34] and Zhao et al.[46] reported similar results.

Effect of the Preflocculation of LM on the Paper Formation

The effect of the preflocculation of LM on the paper formation is shown in Figure 10. The lower the paper formation index was, the better the paper formation was. We found that the handsheets filled with preflocculated LM had better paper formation than the handsheets filled with unpreflocculated LM. This was because the direct addition of CPAM to the stock usually resulted in fiber flocculation and many fiber flocs; this seriously deteriorated the paper formation. H-CPAM had a stronger flocculation ability, so the paper formation was further deteriorated. However, during the filler preflocculation process, a portion of CPAM was previously adsorbed by the filler particles. Thus, there was less chance for fiber flocculation, and the paper formation was improved accordingly. In addition, L-CPAM gave better paper formation when it was used as a filler flocculant; this was attributed to the smaller filler flocs produced. Figure 11 presents the photographs of the handsheets filled with unpreflocculated and preflocculated LM. As shown in Figure 11(C,D), there were obvious differences in the gray level caused by different light transmissions; this indicated the uneven distribution of the fibers in the handsheets. The bright parts indicated the places where the fibers were sparse, and the dark parts indicated the places where the fibers were dense. As shown in Figure 11(B), black spots resulting from the large LM flocs appeared. However, neither case was found in Figure 11(A); this indicated that the fibers dispersed well in the handsheet, and the particle size of the L-CPAM-preflocculated LM was appropriate for filling.

Effect of the Preflocculation of LM on the Filler Retention

The retention of the unpreflocculated and preflocculated LM was investigated and is shown in Figure 12. The results indicate that the H-CPAM-preflocculated LM had the highest filler retention. At a filler loading of 30%, the retention rate of the H-CPAM-preflocculated LM was higher than 86%, and the retention was less than 82.5% in other cases. This was mainly because the H-CPAM-preflocculated LM has the largest particle size of about 100 μm. It was easier for the large-size filler particles to remain on the formation wire through mechanical interception. In addition to the size effect, the enhanced filler retention may also have been relevant to the increased ζ potential of the preflocculated LM because the filler with a higher ζ potential could be retained more easily

by negatively charged fibers.[8,27,29] In addition, we found that the addition of the unpreflocculated LM and L-CPAM gave the lowest filler retention. In addition to the size and potential effects, it might also have been attributed to the weak retention effect of the low-molecular-weight CPAM added to the pulp.

CONCLUSIONS

In this study, the preflocculation of LM with CPAM was proven to be an effective method for improving the paper properties. The particle size and ζ potential analysis showed that the larger LM flocs with positive ζ potential were produced by the preflocculation. The morphological structure of the filler hardly changed during the preflocculation process. With respect to the paper properties, the preflocculation of LM increased the hydrophobicity of the paper, and LM preflocculated with H-CPAM provided paper with better hydrophobicity. At a filler dosage of 30%, the tensile and burst indices of handsheets filled with preflocculated LM were about 25 and 43% higher than those of the unpreflocculated LM-filled handsheets. The optical properties nearly remained the same. The introduction of the preflocculated LM also improved the paper formation compared to the respective addition of the unpreflocculated LM and CPAM, especially for paper filled with L-CPAM-preflocculated LM because of less fiber flocculation and the small flocs produced. In addition, the LM preflocculated with H-CPAM had the highest filler retention because of its increased particle size and ζ potential.

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