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Effects of Water-Soluble Inorganic Salts and Organic Materials on the Performance of Different Polymer Retention Aids

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EFFECTS OF WATER-SOLUBLE INORGANIC SALTS AND ORGANIC MATERIALS ON THE PERFORMANCE OF DIFFERENT POLYMER RETENTION AIDS

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ABSTRACT

One of the current trends in papermaking is to fully close mill water streams. The mill water closure will result in a significant increase of inorganic salts and organic anionic trashes in papermaking white water, which, in turn, affects the retention aid performance. The possible effects of the contaminants, including inorganic salts and organic anionic materials, on the performance of different retention systems were examined in this study. Four retention systems, including a single cationic polyacrylamide (CPAM) system, a dual cationic polymer system of CPAM and polyamine (PAE), an anionic microparticle system (CPAM + bentonite), and a nonionic retention aid of polyethylene oxide (PEO) in combination with phenol-formaldehyde resin (PFR), were investigated. The results showed that the inorganic salts and organic anionic trashes will significantly affect the performance of cationic polymer and microparticle retention systems, but will have less effect on the retention efficiency of the PEO/PFR system. The results presented in this study suggest that, from a filler retention efficiency point of view, the nonionic PEO system is better than cationic polymers for the furnishes containing high inorganic salts and anionic trashes.

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INTRODUCTION

The paper machine white water system contains a large variety of dissolved and colloidal dispersed inorganic and organic substances. Some of these substances may be introduced via the fresh water supply, but the majority originate from raw materials and pulping and papermaking additives such as sizing agents, surfactants, defoamers, dyes, and retention and drainage aids. As paper mills close up their water streams, these dissolved and suspended solids will be accumulated in the process water, resulting in a significant increase in their concentrations. The presence of these substances will cause severe production difficulties such as increased deposits, foaming, biological activity, corrosion, decreased retention and web formation, paper strength, etc. In addition to water closure, the increased use of recycled fibers is another source of contamination of pulp furnishes.

The buildup of inorganic and organic materials in a closed white water system has been examined and discussed in previous studies [1-10]. Typical ions that build up in a closed white water system are sodium, aluminum, calcium, magnesium, various heavy metals, chloride, sulfate and phosphate [7]. Most of these small ions have poor retention in the paper sheets and they will cycle-up quickly in the white water, which is solely based on the degree of closure. Precipitation may eventually occur for low solubility elements such as calcium, which will seriously interfere with the papermaking operation [2,7].

The high inorganic content may affect the structure and the configuration of polymer retention aid in a solution. Because of the salting-out effect, high concentrations of dissolved electrolytes can result in a collapse of the polymer chain and reduce the polymer's bridging capability. However, the increase in the inorganic concentration may also enhance the filler and fines retention because the ions, mainly divalent and trivalent, will reduce the electrostatic double layer thickness. Therefore, the effect of ionic strength on the filler and fines retention may be complicated, depending on the polymer system used in papermaking.

Another factor that may significantly affect the water quality and fine particle retention is the increased use of precipitated calcium carbonate (PCC). Because of the high solubility of PCC under acid conditions, this specific filler can only be used under neutral or slightly alkaline conditions. The increase in pH alters many issues in the papermaking wet-end chemistry. For example, the total cationic demand will increase because of the significant increase in the charge density of wood fibers and fines. The fibers will uncoil and the fibrils will extend outwards resulting in a decrease in water drainage rate [11]. The cationic retention additives may also be sensitive to the pH, e.g., higher pH reduces cationic charge of primary, secondary, and tertiary amines significantly, resulting in a decrease in the efficiency of polymers as papermaking retention aids. Although quaternary amines maintain their cationic charge characteristics over the entire pH range, the increased anionic charge density of wood fibers and total cationic demand in alkaline conditions may also result in a decrease in the retention efficiency when these polymers are used. Therefore, the traditionally used retention systems may not be an optimized system for current papermaking.

Many studies regarding the effect of water closure on the papermaking process have been done previously. Webb [3] discussed the impact of water quality on the wet-end chemistry, including both freshwater quality and recycled water. His study was focused on the influence of inorganic electrolyte on the adsorption of cationic starch on the solid suspension; less attention was paid to other polymer systems. A series of recently published papers discussed the effect of water closure on the final paper products and the wet-end operation [4-8]. However, no discussion regarding the polymer retention aid performance was given in these studies. Pelton et al. [12,13] studied the retention of fines and fillers in mechanical pulp using different retention aids. They concluded that anionic trashes will significantly reduce the efficiency of cationic retention aids, but have less effect on the nonionic retention system of PEO/PFR. However, they did not study the effect of inorganic electrolytes on the performance of different retention aids. Although the effect of inorganic electrolytes on the polymer adsorption, configuration and flocculation of solid suspension by different polymers have also been extensively studied previously [14-17], the effect of these electrolytes on the retention of fillers, particularly PCC, in wood pulps has not been well studied. Most recently, Allen et al. published an excellent paper [10] that discussed the influence of water closure on the performance of cationic, microparticle and nonionic polyethylene oxide systems as retention aids for fines retention in a TMP newsprint furnish. They concluded that the increase of contaminants in white water will negatively affect the performance of cationic polymers as a retention aid, but will not significantly affect the PEO/PFR system. This suggests that a nonionic retention aid is superior to cationic polymers in a highly contaminated white water system. Although important information was given in Allen's paper, it is not known if this conclusion can be applied to other conditions, such as high pH and different furnishes. Furthermore, it is also interesting to know if the filler retention, particularly for widely used PCC filler, can also follow the same trend. In this paper, the PCC retention in a mixture of ground food and chemical pulps as a function of dissolved and colloidal substance concentration was studied. Obviously, an understanding of the effects of inorganic and organic electrolytes on the performance of ionic and nonionic polymer retention aids will help papermakers in selecting an effective retention system for their papermaking furnishes, particularly when mill water is closed and a high recycled fiber level is used.

EXPERIMENTALS

Materials

The mechanical pulps were black spruce and balsam fir-based hydrosulfide-bleached pulp (brightness 66-67%) and pressurized ground wood (PGW) pulp. The pulps were diluted with deionized water and hot-disintegrated. The chemical pulp was refined to 470 mL Canadian freeness level by a laboratory Valley beater. The ground wood and chemical pulps were mixed in a ratio of 5:2. The pH of the pulps was adjusted by sodium hydroxide and hydrochloride. **Table 1** shows the properties of the mechanical and chemical pulps as well as the pulp mixture. All experimental data presented in this paper were obtained from a mixture of ground wood and chemical pulps at a ratio of 5:2.

PCC (30.4% solids in water) was a scalenohedral calcite filler product with an average particle size of 1.3 μ m and specific surface area of 12 m²/g (BET measurement).

The properties of four retention systems used in this study were given as follows:

1. A high molecular weight and low charge density cationic polyacrylamide (PC8713, Betz Dearborn Product) was used as a single polymer system. The polymer powder was diluted to a concentration of 0.1%, and agitated at 600 rpm stirring rate for 60 minutes before use.

2. The anionic microparticle system consists of CPAM (the same chemical used as a single component polymer system) and bentonite (Baroid Mineral & Chemicals). The bentonite was diluted to 5 g/L concentration by distilled water before use. The CPAM was added to pulp first, then followed by bentonite. The fixed ratio of CPAM to bentonite of 1:5 was used.

3. A moderate molecular weight and high charge density polyamine (PC1279, Betz Dearborn Product) was used as "coagulant", which was added before cationic retention aid (CPAM, the same chemical used as a single component polymer system) in a dual polymer retention system. PAE is a polymer aqueous solution with 50% active concentration and it was diluted to 2 g/L before use. The addition ratio of PAE to CPAM was 2:1.

4. The nonionic dual component retention system includes polyethylene oxide and a low molecular weight phenol-formaldehyde resin (PFR, Borden Chemicals). The PEO had an average molecular weight of 8 million (M_w). Water-soluble PFR had a solid content of 46% and molecular weight of 13000. The PEO solution was prepared by adding 0.1% of PEO to distilled water and agitating for 3 hours at 600 rpm stirring rate. The addition ratio of PFR to PRO was 2:1. The PEO was added first, followed by PFR.

The dosages of retention aids were calculated based on the total stock volume and presented as mg/L. The corresponding dosage of 0.5, 1, 2, 3, 4, 6 and 8 mg/L in pulp correspond to 0.0178, 0.356, 0.721, 1.068, 1.424, 2.131, and 2.848 lb/tone of dry solids (pulp and fillers), respectively.

The white water closure was simulated by adding different amounts of inorganic salts and organic polymers to pulp suspensions. Sodium chloride and sodium sulfate powders were used as inorganic electrolytes. Dry xylan powder, separated from birch wood, was dissolved in distilled water. The lignin is Indulin-C product. The total anionic charge (cationic demand) of the pulp was determined by colloidal titration using poly(diallyldimethylammonium chloride) (polyDADMAC, Nalco Chemicals) and polyvinyl sulfate potassium (PVSK, Nalco Chemicals). Both direct titration and back titration were used. The properties of white water are given in **Table 2**.

It should be noted that a nonlinear relationship between organic material addition and the cationic demand was observed (see **Table 2** column 4). The possible reason for this is because of the adsorption of organic materials onto the surface and lumens of fibers and fines.

Methods and procedures

The Dynamic Drainage Jar (DDJ) with a 100-mesh screen (150 μ m holes) was used to measure the first-pass retention (FPR) of PCC on a fiber web. The agitation rate was 1000 rpm. The FPR of PCC was calculated from the difference between the filler contents in the original pulp suspension and the filtrate from DDJ, respectively.

A pulp suspension containing 500 mL 0.4% fibers was added into the DDJ under 1000 rpm stirring rate. The distance between stirrer and screen was adjusted to 0.5 cm. After the addition of PCC (0.875 g), the total solid consistency in the furnish was 0.57%. The furnish was agitated for 20 seconds before the required retention polymers were added. For a dual retention system, the first component was added 7 seconds and the second component 5 seconds before the first-passed white water was collected.

During the white water collection, the first 10 mL of white water was drawn away, then a solution containing 100 mL white water was collected. The collected white water from DDJ was adjusted to a pH < 3 by adding HCl at room temperature and standing for 5 min to dissolve the

PCC particles, then ~ 1 mL ammonium hydroxide solution was added to the solution bringing the pH up to ~ 9.5 . The calcium concentration in the collected white water was analyzed by potential titration using EDTA (sodium tetra ethylenediamine tetraacetate) salt as a titrant. The end-point of titration was measured using a voltmeter, which was equipped with a calcium-selective electrode and a glass pH reference electrode.

RESULTS AND DISCUSSION

Retention in original white water conditions

The results of the first-pass retention (FPR) of PCC obtained from original pulp (without adding any inorganic and organic compounds) by four different retention systems are illustrated in **Figure 1**. It can be seen that the best first-pass retention of PCC in this condition was obtained with the PEO/PFR system. Very high retention can be achieved with only a moderate amount of PEO addition. For the cationic polymer related retention systems, the dual polymer system of CPAM/PAE was more effective compared to the single CPAM system, which suggests that the pre-addition of highly charged cationic coagulant enhances the filler retention. The improved retention with the addition of PAE may be attributed to the neutralization of particle surface charge which leads to a decrease in the colloidal stability of fillers in pulp, and also the neutralization of anionic trashes that will reduce the effective activity of CPAM. The bentonite-based microparticle retention system was the least effective retention system, which is even poorer than a single CPAM system.

The effects of inorganic salts on the performance of different retention aids

The effects of inorganic salts on the different retention systems were examined by adding different inorganic electrolytes in pulp suspensions. Typical results for 3000 mg/L of Na₂SO₄ (3930 μ S/cm) are given in **Figure 2**. Compared to the results shown in **Figure 1**, it can be seen that the presence of sodium sulfate in the white water has a positive effect on the retention of PCC with the PEO/PFR dual component system, but the three cationic polyacrylamide-based

retention systems lost their efficiency. Thus, the retention performance of the PEO-based system was superior in comparison to the other systems at high conductivity conditions.

The retention tests were also performed in different salt concentration levels (1000 mg/L, 2000 mg/L and 3000 mg/L of Na₂SO₄) with practical polymer dosage according to the literature (For the dual cationic system the addition levels of PAE and CPAM are 2 mg/L and 1 mg/L, respectively) [11]. In cases of the single polymer system and bentonite-based microparticle system the CPAM addition level of 2 mg/L was chosen. The results of the first-pass retention of PCC as a function of Na₂SO₄ concentration are given in Figure 3. As expected, the PCC retention decreased as the concentration of Na₂SO₄ was increased for single and dual cationic systems as well as the microparticle system. However, increase continued in the PCC first-pass retention as the increase in the salt concentration was observed for nonionic PEO system.

To clarify the results of the inorganic salt effect on the PCC retention with PEO system, the retention tests were further performed in the presence of different concentrations of Na_2SO_4 in the whole polymer addition range and the results are demonstrated in **Figure 4**. It can be seen that when PEO/PFR was used as a retention system, the addition of Na_2SO_4 improved FPR of PCC consistently.

The decrease in the first-pass retention of PCC by adding inorganic salts for CPAM-based retention systems (single and dual CPAM systems as well as microparticle system) may be attributed to the salting-out effect of inorganic electrolytes. It has been well known that the polymer configuration will change from an extended structure to a coil-up structure when the conductivity of solution is increased. Because the retention of PCC by CPAM is based on bridging mechanism, the longer the polymer chain, the higher the retention is expected. Therefore, the formation of coil-up structure of CPAM in a high inorganic electrolyte solution will significantly reduce the polymer chain length, resulting in a reduction of the forming of effective bridges between PCC particles and fibers. The retention efficiency of the microparticle system is also decreased due to the decrease in the polymer chain length.

Although the mechanism of the PEO/PFR retention system is not clear and several mechanisms have been suggested [18-21], all of these suggested mechanisms indicate that the chain length and the configuration of PEO is very important. Because PEO is a nonionic polymer, the salting-out effect of inorganic electrolytes is less significant for the PEO compared to ionic polymers. Therefore, the performance of this nonionic polymer as a retention aid will not be significantly impacted. Furthermore, the addition of electrolyte will significantly reduce the electrostatic stability of PCC and fines by depressing the diffusion double layer surrounding the solid suspension, which leads to a more effective retention when the PEO/PFR retention system is used.

The high ionic strength seems to reduce the retention PCC more effectively for dual cationic polymer systems than single polymer systems. This may be because the inorganic salt will affect not only the configuration of high-molecular-weight CPAM, but also reduce the efficiency of coagulant of PAE. As coagulant of PAE coiled-up in a high conductive solution, the coagulant efficiency decreased. Therefore, the total retention efficiency of this dual cationic polymer system decreases dramatically as the salt concentration is increased.

The effects of organic materials on the performance of different retention aids

The effects of dissolved and colloidal organic materials (lignin 25 mg/L, xylan 120 mg/L) on the performance of different retention aids were studied and the results are shown in Figures 5 and 6. It can be seen that at the same anionic trash level and polymer concentration, the PEO/PFR system gives the best retention. Figure 5 also indicates that the dual cationic retention system (CPAM + PAE) is better than single CPAM, which suggests that the total cationic demand is very important for the retention when a cationic polymer is used. It has been known that the best level of zeta potential is from -5 to -10 mV for a functional cationic retention aid [22]. To achieve this optimized zeta potential, the addition of cationic coagulant prior to the CPAM is necessary if the concentration of anionic trash is high. Figure 6 shows that at fixed polymer addition levels, the increase in anionic trash levels resulted in a slight decrease in the PCC first-pass retention when PEO/PFR was used. One of the possible reasons for this small drop in the

PCC first-pass retention may be attributed to the increase in the charge density and the stability of PCC particles when anionic trash is adsorbed onto the filler surface. Compared to the nonionic PEO system, the first-pass retention of PCC with cationic polymer-based systems decreases considerably with the increase in the concentration of organic contaminants. On average, the first-pass retention of PCC decreases about 41.7% for the single CPAM system and 32.1% for the bentonite-based microparticle system as cationic demand increases from 2.5 μ eq/L (original water conditions) to 8.7 μ eq/L (lignin 25 mg/L, xylan 120 mg/L). In contrast to the cationic retention aids, no neutralization reaction between nonionic PEO and anionic trashes is expected. As a result, the anionic trashes should have no significant effect on neither the polymer structure nor the retention performance. Therefore, a higher retention of PCC can be obtained with this dual retention system than CPAM-based retention systems. This has been confirmed by the experimental results shown in **Figure 6**.

The PCC first-pass retention as a function of anionic trashes at different PEO/PFR addition levels is summarized in **Figure 7**. Once again, PEO showed its unique properties of being rather insensitive to dissolved and colloidal materials present in the pulp suspension.

Retention of PCC in simulated white water closure conditions

The first-pass retention of PCC in a model "closed white water" system was examined. The closed conditions were simulated by adding the following contaminants in white water: 200 mg/L of NaCl, 3000 mg/L of Na₂SO₄, 25 mg/L lignin, and 240 mg/L of xylan. The conductivity of the system was 4820 μ S/cm and cationic demand 9.2 μ eq/L. The results obtained with different retention systems are given in **Figure 8**. Compared to the results obtained in the original white water (see **Figure 1**), the FPR of PCC in simulated white water dropped sharply for all of the retention systems studied except the PEO/PFR system. It has been mentioned previously that the PCC retention increased as the inorganic salt concentration increased, but slightly decreased as the concentration of organic trashes increased. As a co-effect of inorganic and organic contaminants in the water, the retention of PCC particles with PEO is even higher than in original white water conditions, and it is much higher than that obtained with any CPAM related retention systems.

CONCLUSIONS

The laboratory experimental results indicated that cationic polymer systems (including single and dual systems) and microparticle systems (betonite + CPAM) are not effective retention systems for PCC at high inorganic and organic electrolyte furnishes. The main reason for the decrease in the retention performance of cationic polymer in a high inorganic electrolyte solution may be attributed to the salting-out effect of cationic polymer, which resulted in a transition of the polymer configuration from extended structure to a shrinking coil structure. However, the charge neutralization between cationic retention aid and anionic trashes seems to be the dominating factor for the decrease in the retention efficiency for the furnishes containing high organic trashes. The inorganic electrolytes have a positive effect on the PCC retention when the PEO/PFR is used because the inorganic salt will reduce the particle stability by depressing the diffusion dabble layer but without significantly affecting the polymer properties. The organic anionic trashes will slightly reduce the PCC retention if the PEO/PFR system is used. The small negative effect of this system may be mainly attributed to the increased particle stability due to the adsorption of negatively charged substances onto filler and fiber surfaces. It can be concluded that even though the highmolecular-weight PEO is more expensive than CPAM, PEO can still be a cost-effective retention system, particularly in a furnish with high anionic trashes and inorganic salts such as a fully closed white-water system. The main conclusions from this paper support previous findings [10].

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FIGURE CAPTIONS

Figure 1. First-pass retention of PCC in the original water conditions.

Figure 2. First-pass retention of PCC in 3000 mg/L Na₂SO₄ solution.

Figure 3. First-pass retention of PCC as a function of Na_2SO_4 concentration obtained with different retention aids.

Figure 4. The FPR of PCC with PEO-based dual component retention system in the presence of different concentrations of Na₂SO₄. The ratio of PEO to PFR is 1:2.

Figure 5. First-pass retention of PCC as a function of CPAM or PEO concentration in the presence of 25 mg/L lignin and 120 mg/L xylan.

Figure 6. First-pass retention of PCC as a function of organic anionic trash concentration with different retention systems.

Figure 7. First-pass retention of PCC with PEO-based dual component system in the presence of different concentrations of organic contaminants.

Figure 8. First-pass retention of PCC in the model closed white water.

	PGW	Chemical	Mixture
		pulp	
CSF (mL)	50	470	85
pН	5.5 (mill)	7	7.8 (adjusted)
	6.4 (after dilution)		
Consistency (%)	3% (after disintegration)	1.5% (after	0.4%
		refining)	
Fines (%) -100 mesh	35.1%	_	28%
-200 mesh	28.3%		15%
Zeta potential of fines	_	_	-11.7 (filtrate)
(mV)			

 Table 1. Properties of the mechanical and chemical pulp.

 Table 2. The properties of white water.

Effect of inorganic salt on the conductivity		Effect of organic material on the cationic	
		demand	
Concentration	Conductivity	Concentration	Cationic demand
Original white water	108 µS/cm	Original white water	2.5 µeq/L
Na ₂ SO ₄ (1000 mg/L)	1570 μS/cm	Lignin (12.5 mg/L)	5.4 µeq/L
Na ₂ SO ₄ (2000 mg/L)	2890 μS/cm	Lignin (25 mg/L)	6.5 µeq/L
Na ₂ SO ₄ (3000 mg/L)	3930 µS/cm	Xylan (120 mg/L)	6.3 µeq/L
NaCl (200 mg/L) +	4820 μS/cm	Lignin (25 mg/L) +	8.7 µeq/L
Na ₂ SO ₄ (3000 mg/L)		Xylan (120 mg/L)	
		Lignin (25 mg/L) +	9.2 µeq/L
		Xylan (240 mg/L)	



Figure 1. First-pass retention of PCC in the original water conditions.



Figure 2. First-pass retention of PCC in 3000 mg/L Na₂SO₄ solution.



Figure 3. First-pass retention of PCC as a function of Na_2SO_4 concentration obtained with different retention aids.



Figure 4. The FPR of PCC with PEO-based dual component retention system in the presence of different concentrations of Na_2SO_4 . The ratio of PEO to PFR is 1:2.



Figure 5. First-pass retention of PCC as a function of CPAM or PEO concentration in the presence of 25 mg/L lignin and 120 mg/L xylan.



Figure 6. First-pass retention of PCC as a function of organic anionic trash concentration with different retention systems.



Figure 7. First-pass retention of PCC with PEO-based dual component system in the presence of different concentrations of organic contaminants.



Figure 8. First-pass retention of PCC in the model closed white water.