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Cationic Polystyrene-Based Paper Sizing Agents

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CATIONIC POLYSTYRENE-BASED PAPER SIZING AGENTS

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ABSTRACT

A series of cationic polystyrene copolymeric sizing agents were synthesized and characterized. The sizing agents could be divided into two categories: cationic polymeric microparticle (CPMP) prepared by emulsion polymerization, and cationic polymeric microaggregate (CPMA) polymerized in ethanol then dispersed in water. The effects of particle size, crosslinking degree, and cationic monomer ratio on the sizing efficiency were studied. Results from this study suggest that these two types of copolymers can be used not only as a sizing agent, but also a dry strength agent and retention aid. Potential advantages of these copolymers as paper sizing agents include that (1) they can directly adsorb onto negatively charged fibers and fines in a broad pH range from 1 to 11; (2) they can uniformly distribute and defuse on fiber surfaces if the particle size of latex is less than 100 nm; (3) no retention aid is needed; and (4) they do not hydrolyze in water. Compared to previously reported cationic polymeric sizing agents, our copolymers demonstrated to be more effective.

INTRODUCTION

Currently, alkyl ketene dimers (AKD), alkenyl succinic anhydride (ASA), and rosin

sizing are the most common sizing products used in papermaking. However, it is well recognized that rosin sizing is very difficult to use in alkaline papermaking [1,2]. On the other hand, although AKD and ASA have been commonly used in alkaline papermaking, there are still some problems, such as hydrolysis of ASA and AKD in water [2], sizing reversion on PCC surface [3-5], and slow sizing development of AKD [1,2].

Polymeric cationic internal sizing agents, such as cationic polystyrene latexes [6-8], polystyrene acrylic acid ester [9], and cationic vinyl polymers [10] have been developed recently. One of the advantages of these copolymer sizing agents is that no retention aid is needed during the sizing development. Furthermore, the cationic polymeric sizing agent will reduce the total cationic charge demand of a pulp furnish. In addition to the paper sizing development, paper strength and optical properties improvements were also observed in previous studies [6-8]. However, a large amount of cationic polymeric sizing agent (5% based on dry fibers) is required in order to obtain a reasonable sizing degree [6].

This paper describes two types of polymeric sizing agents which are prepared by emulsion polymerization in water and bulk polymerization in ethanol. The emulsion polymerization leads to a cationic microparticle, and the bulk polymerization carried out in ethanol gives an ethanol-soluble, but water-dispersible, cationic polymer. The polymerization of the cationic sizing agents, and the effect of the copolymer geometry, chemical composition, and processing conditions were examined. The sizing agents described here can also be used as paper strength agents and filler and fines retention aids.

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MATERIALS AND METHODS

Materials

Styrene (Aldrich) was distilled under vacuum to remove inhibitors before polymerization. Vinyl benzyl trimethyl ammonium chloride (VBTMA) was used as cationic monomer, and was prepared in a 25%-aqueous solution using chloromethyl styrene and trimethyl amine. Divinylbenzene (DVB, Aldrich) was used as a crosslinker. The initiator, 2,2'-azobis(N,N'-dimethyleneisobutylamidine)dihydro-chloride (VA-044), was provided by Wako Pure Chemicals. Cationic surfactant, cesyltrimethyl-ammonium was used as an emulsifier for emulsion and microemulsion polymerization.

Once-dried bleached kraft softwood (BKS) pulp was used in PCC retention test. However, for the sizing test, the fines in BKS pulp were removed by extensive washing followed by filtration using a 200-mesh screen (Tappi Test Method T261). The absence of colloidal and soluble polymers in the pulp was confirmed by colloidal titration of the pulp supernatant. Charge density of long fiber was 1.23 eq/g measured by colloid titration.

Precipitated calcium carbonate (PCC, Specialty Minerals Inc.) was a scalenohedral calcite filler product (ALBACAR HO®) with an average particle size of 1.3 μm and specific surface area of 12 m^2/g .

Preparation of cationic polymeric microparticle sizing agent (CPMP)

The cationic polymeric microparticles with various particle sizes were prepared using a monomer-in-water emulsion or microemulsion. The preparation conditions and the physical properties of CPMP are given in Table 1. No cationic monomer was used in

CPMP preparation and the positive charges of the microparticles were introduced by cationic initiator and cationic surfactant. A typical example of polymerization (for sample C) is described as follows: a double-wall glass reactor equipped with mechanical stirrer was filled with 150 ml of deionized water, 108 mmol of styrene, and 8.2 mmol of cationic surfactant. The solution was flashed with nitrogen for 30 minutes and temperature-regulated at 60°C to remove oxygen in the solution. After temperature stabilization, 0.7 mmol of VA-044 in 5 ml of deionized was injected into the reactor. The polymerization was carried out under nitrogen at 60°C and 400 rpm for 4 hours. The cationic polymeric microparticles were centrifuged and washed twice under 60,000 rpm (Beckman L-80 ultracentrifuge) for 120 minutes to remove cationic surfactant.

Preparation of cationic polymeric microaggregate (CPMA)

The polymers that used for preparation of cationic polymeric microaggregates were synthesized in ethanol. The chemical structure of this type of copolymer is schematically shown in Figure 1. The cationic monomer, vinyl benzyl trimethyl ammonium chloride (VBTMA), which was used for copolymerization was prepared in a 25% aqueous solution using equimolar amount of chloromethyl styrene and trimethylamine at room temperature. The polymerization conditions and the physical properties of CPMA are given in Table 2. No cationic surfactant was used in the polymerization of CPMA. A typical example of polymerization (for sample H, Table 2) is described as follows: a double-wall glass reactor equipped with mechanical stirrer was filled with 150 ml ethanol, 180 mmol of styrene, and 12 mmol of VBTMA solution. The solution was flashed with nitrogen for 30 minutes and temperature-regulated at 60°C to remove oxygen in the solution. After stabilization of temperature, 0.7 mmol of VA-044 in 5 ml of deionized was injected. The polymerization was carried out under nitrogen

for 4 hours with gentle agitation. The cationic polymeric microaggregates could be prepared by adding above polymer into water. The size and the stability of the aggregates strongly depend on the copolymer charge density and concentration, which will be further discussed latter.

Microparticle size and zeta potential measurement

The particle sizes of CPMP and CPMA in water was determined by Doppler Electrophoretic Light Scattering (Zetasizer 2c, Malvern). The electrophoretic mobility was measured using a Zetasizer 2c (Malvern) at a stationary position, and the zeta potential was calculated by computer using the Smoluchowski equation.

Sizing development and handsheet preparation

The sized fibers were made by the reaction of long fibers with varying amounts of sizing agents, including CPMP and CPMA in ~5% fiber consistency without adding any retention aid. After one minute of adding sizing agent, the fibers with sizing agents were transferred into a British Sheet Machine and handsheets were made according to Tappi Test Method T205 om-88. Handsheets were dried at room temperature and cured at 120 °C.

Physical property measurements of paper sheet

Z-tensile strength of sized paper handsheets was measured using Tappi Method UM 581. The sizing efficiency was evaluated by measuring the time requirement of a water drop remaining on the handsheet (Tappi T432 om-87). The contact angle of a water drop against a handsheet was measured using a FTA contact angle analyzer.

Adsorption isotherm

The adsorption of CPMP and CPMA on the fibers were measured at pH 6.5 and room temperature. The adsorption measurements were carried out only on long fiber fraction. Five grams of fibers in one liter deionized water were mixed with required CPMP or CPMA for one hour. The CPMP concentration in supernatant was determined by 250 nm UV adsorption using a concentration calibration curve, and the CPMA concentration was measured by colloid titration.

RESULTS AND DISCUSSION

CPMP and CPMA properties

Generally speaking, the particle size of cationic polymeric microparticle (CPMP) prepared by emulsion polymerization is a function of the concentrations of surfactant and cationic monomer. It can be seen from Table 1 that the particle size decreases with increasing surfactant concentration, but increases with increasing cationic monomer concentration. The decrease in the microparticle size with the increase in surfactant has been well described in the literature [11]. The increase in the particle size with the increase in cationic monomer concentration is a result of the swelling of the particles. As the charge content of microparticle increases, more water can penetrate into the core of the particles and associate with cationically charged repeat units in the polymer chains, resulting in the swelling of the particles. The conversion of the polymerization of sample E was 87% after a 4-hour polymerization (measured by polymer weight).

The cationic polymeric microaggregate (CPMA) prepared in ethanol could be either ethanol-soluble or insoluble, depending on the charge ratio, polymerization degree, concentration, etc. Although the solubility of the copolymer in ethanol has not been systematically examined in this study, it was found that the ethanol-soluble

cationic polymer could be obtained if the content of charged repeat unit in the copolymer is higher than ~10%. If the charge density is less than 10%, the copolymers precipitate or form aggregates in ethanol.

The solubility of CPMA copolymers in water was also strongly dependent on the charge density. When the charge density is less than ~5%, the copolymers precipitate in water, and if the copolymer charge density is between ~5 and ~15%, they form large aggregates in water. However, when these large aggregates were heated to 80 °C for more than 30 minutes, or stirred at room temperature for few days, these large aggregates gradually form micelle-like particles which are thermodynamically stable in water. The particle size strongly depends on both the polymer charge density and concentration. It was found that stable micelle-like aggregates can be formed only at low concentration. It was believed that the micelle-like aggregates are stabilized by the strong electrostatic repulsion force between the particles. When the cationic repeat unit is higher than 30%, the copolymers are fully soluble in water.

It is believed that the sizing development by the cationic polymeric microparticle or microaggregates (either directly from emulsion polymerization or from dispersion of ethanol-soluble cationic polymer in water) is a function of charge density, particle size, and crosslinking degree. Obviously, the higher the cationicity, the stronger the attractive force between the polymer and fibers, which may lead to a high adsorption of copolymer on fiber surface. However, a strong attractive force between polymer and fiber surface may not mean an effective sizing because a highly charged copolymer will also increase the hydrophilicity of the fiber surface, resulting in a decrease in sizing efficiency.

It has been well known that an effective sizing agent must diffuse on the fiber surface to form a uniform monolayer or thin film. Among other parameters, the particle size of adsorbed polymer aggregates is one of the important factors controlling the polymer diffuse rate. This means that not only the charge density of the copolymer but also the particle size will affect the sizing efficiency. The effects of both the charge density and the particle size on the paper sizing efficiency were investigated in this study.

Sizing development by CPMP

The sizing development of CPMP on BSK fibers was first examined using sample D and the results are given in Table 3.

It can be seen that the sizing was developed when the additive amount is more than 0.4%. At the addition amount of 0.8%, water remained as a stable drop on the surface of handsheets more than 10 minutes, which indicates an effective sizing. The sizing efficiency was also examined by directly measuring the contact angle of the water drop on the handsheets and those results are also given in Table 3.

It is interesting to compare our results with previous results. In a previous study reported by Inoue et al. [6], an effective paper sizing using polystyrene-based latex was seen at high polymer addition (5%). However, only 0.8% of our CPMP led to a high sizing degree. It should be noted that there are many differences between the polymers reported by Inoue et al. and those prepared in this study. For examples, their latexes contain hydroxyl groups, but our CPMP does not; the particle size of their latex is larger than ours; the charge density of two latexes are different; and different cationic monomers were used in the respective polymer preparations. All of these differences may significantly affect the sizing efficiency.

The diffusion of the copolymer on fiber surfaces is an important step in sizing development, and this will be significantly affected by the crosslinking degree and glass transition temperature of the copolymers. If the diffusion of the particles on fiber surface is one of the control steps, the crosslinked CPMP should not lead to an effective paper sizing. Table 4 shows the results obtained by cross-linked sample E (39 nm) and non-crosslinked sample D (36nm). Although the particle size of these two samples was almost the same, crosslinked CPMP did not show any sizing improvement. This result indicates that the cross-linked polymer particles can only deposit but not diffuse on fiber surfaces. Therefore, crosslinked microparticles cannot be used as an effective sizing agent. This result also suggests that an effective sizing development by CPMP is not a result of the close parked microparticles on the fiber surface, but is a result of the formation of thin film of copolymer during the curing process. Other evidence for this mechanism is that unheated handsheets did not show any sizing development even after CPMP adsorbed onto the fibers.

Table 4 also shows particle size effect on the sizing efficiency. For the CPMPs without crosslinker, it was found that an effective sizing can only be obtained by the particles that have a diameter of 36 nm. Although some other factors may also affect the result, we believe that the spreading of polystyrene particles on the fiber surface during the curing process is important, which is a function of latex particle size. If the particle size of the latex is large, the polymer cannot spread fully as a monolayer on the cellulose fiber. As a result, the sizing efficiency will decrease. Comparing our results with those previously reported by Inoue et al., it may be suggested that the particle size of the cationic polystyrene latex is a critical factor in controlling sizing efficiency.

Sizing development by CPMA

The mechanism of sizing development by CPMA is shown in Figure 2. As mentioned before, CPMA will form micelle-like aggregates in water if the content of cationic repeat unit is in the range of 5 - 15%. Theoretically, these particles should behave similarly to that of CPMP when they are used as particle sizing agents. However, there is a significant advantage to using CPMA instead of CPMP because very small particles of CPMA can be prepared using a surfactant-free polymerization technique.

Table 5 shows the effect of sample G on the sizing of handsheets after curing at 120 °C. It can be seen that sizing developed at a relatively low additive amount when CPMA was used compared to CPMP. Two possible reasons may cause this difference. First, the CPMP was prepared by emulsion polymerization in the presence of cationic surfactant. Therefore, the residual surfactant (even after washing) may adsorb onto the fiber surface resulting in a decrease in hydrophobicity of fibers. In contrast to the CPMP, CPMA was prepared in ethanol without adding surfactant and this negative effect was eliminated. Second, it was found that the CPMA formed even smaller particles (micelle-like) than CPMP in water at low concentrations. As discussed before, small particles will distribute more uniformly on a fiber surface and diffuse more easily than large particles, which may lead to an more effective paper sizing.

The ratio of the cationic repeat units in the copolymer is important. It was found that if the content of the cationic repeat unit is too low, the copolymers form large aggregates in water. These large particles cannot be uniformly distributed on the fiber surface; therefore, they cannot form a monolayer on the fiber surface during the curing process. When charge density increases, the copolymer particles can be stabilized by strong electrostatic force to form thermodynamic stable micelle-like particles in water. However, if the

charge density is too high, the copolymer will be soluble in water and no sizing can be developed by this hydrophilic copolymer. This suggests that there is an optimum charge ratio for maximum sizing efficiency. The effect of the cationic charge ratio on the sizing efficiency is shown in Table 6.

Adsorption isotherm of CPMP and CPMA on fiber surfaces

Figure 3 shows adsorption isotherm of CPMP (36nm) and CPMA (5% cationic monomer addition) on SBK fibers. It can be seen that CPMA adsorbed completely on the fiber surfaces at the addition amount less than 1% (based on dry fiber weight). However, CPMP adsorption was relatively low (less than 50%). Although CPMP is also a good sizing agent, to further increase adsorption amount and sizing efficiency by optimize the particle size and charge density is desirable.

Paper strength development

It has been reported that some polystyrene-based latexes can improve both the paper sizing and paper tensile strength [6]. It was found in this study that both CPMP and CPMA can be used as paper strength additives. Figure 4 shows that the improvement of paper z-direction strength is significant even at a relatively low polymer addition (0.8% based on dry fibers). It was also noted that CPMA is more effective than CPMP for z-direction strength improvement, which is consistent with the results of sizing development.

PCC retention improvement

It was reported in our previous studies that CPMP is a very effective flocculant and retention aid for PCC and TiO_2 when it was used in combination with a low-charge-density, high-molecular-weight cationic polymer

[12,13]. Because the CPMA particles have a structure similar to CPMP in water, it is reasonable to expect that CPMA can also be used as an effective retention aid for PCC suspension in the presence of cationic polyacrylamide (CPAM).

Figure 5 shows PCC first-pass retention obtained by the addition of CPMA in the model furnish of 0.4 % of washed SBKP and 0.1 % of anionic PAM pretreated PCC. It can be seen that the PCC first-pass retention can be increased from 24 to 41% when CPMA was used alone. However, when a dual system of CPMA-CPAM was used, (CPMA was added first), more than 90% of PCC first-pass retention was obtained. This is consistent with our previous study using CPMP. The mechanism of PCC retention in a dual system of CPMP-CPAM was suggested in our previous publications [12,13]. The results of PCC retention obtained in this study indicated that an effective cationic microparticle retention aid can be prepared in ethanol or other organic solvent in the absence of surfactant. This will be a more economic and environmental-friendly method for cationic polymeric microparticle preparation compared to emulsion polymerization in the presence of surfactant.

CONCLUSIONS

- (1) CPMP and CPMA copolymers are potentially effective sizing and retention agents.
- (2) Significant improvement on Z-direction strength of handsheet was observed.
- (3) The particle size is an important factor affecting sizing efficiency.
- (4) Hydrophobic and hydrophilic balance of CPMA will significantly affect paper sizing efficiency.

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Table 1. Preparation of cationic polymeric microparticles.

Chemicals	A	B	C	D	E
Styrene (mmol)	108	108	108	108	108
Cationic surfactant (mmol)	0	2.7	3.2	8.2	8.2
Divinylbenzene (mmol)	0	0	0	0	4.3
Water (ml)	150	150	150	150	150
VA-044 (mmol)	0.7	0.7	0.7	0.7	0.7
Particle size (nm)	178	91	55	36	39
Zeta potential (mV)	-	-	53	-	56
Conversion (%)	-	-	-	-	87

Table 2. Preparation and physical properties of dispersible cationic polymer (CPMA).

Sample	F	G	H	I
Styrene (mmol)	108	108	108	108
Cationic monomer VBTMA (mmol)	0	5.7	12	19
Ethanol (ml)	150	150	150	150
VA-044 (mmol)	0.7	0.7	0.7	0.7
Cationic monomer ratio (%)	0	5	10	15
Apparent charge density* ($\mu\text{eq/g}$)	148	375	609	750
Conversion (%)	52	35	49	60

*: Obtained by colloid titration in water. However, because the polymer forms micelle-like aggregates, the charges inside the particle core may not be measured during the titration.

Table 3. Effect of CPMP (sample D) on the time of the water drop remaining on the handsheet surface and the contact angle of water drop on the handsheet surface.

CPMP addition (% of dry fiber weight)	Time of a water drop remaining on the handsheet surface (second)	Contact angle (degree)
0	0.5	0
0.1	0.5	0
0.2	0.5	0
0.4	104	76
0.8	>600	86.8

Table 4. Effect of particle size and crosslinker on the time of a water drop remaining on the handsheet surface. Amount of CPMP addition: 0.8% based on dry fiber weight.

Sample	Time of a water drop remaining on the handsheet before curing (second)	Time of a water drop remaining on the handsheet after curing (second)
Sample D (36 nm, non-crosslinked)	0.5	>600
Sample E (39 nm, crosslinked)	0.5	0.5
Sample C (55 nm, non-crosslinked)	0.5	0.5
Sample B (91 nm, non-crosslinked)	0.5	0.5
Sample A (178 nm, non-crosslinked)	0.5	0.5

Table 5. Effect of CPMA (sample G) on the time of a water drop remaining on the handsheet surface.

CPMA addition (% of dry fiber weight)	Time of a water drop remaining on the handsheet surface (second)
0	0.5
0.1	0.5
0.2	0.5
0.4	>600
0.8	>600

Table 6. Cationic monomer ratio effect on the time of a water remaining on the handsheet surface. CPMA addition: 0.8% based on dry fiber.

Sample of CPMA	Cationic monomer addition during the polymerization (%)	Time of a water drop remaining on the handsheet (second)
F*	0	0.5
G	5	>600
H	10	0.5
I	15	0.5

*: Sample F could not form stable dispersion in water.

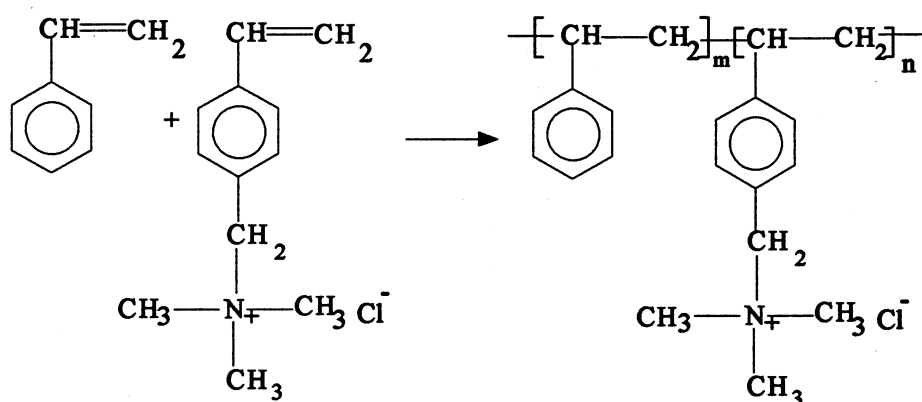


Figure 1. The chemical structure of cationic polymeric microaggregate (CPMA).

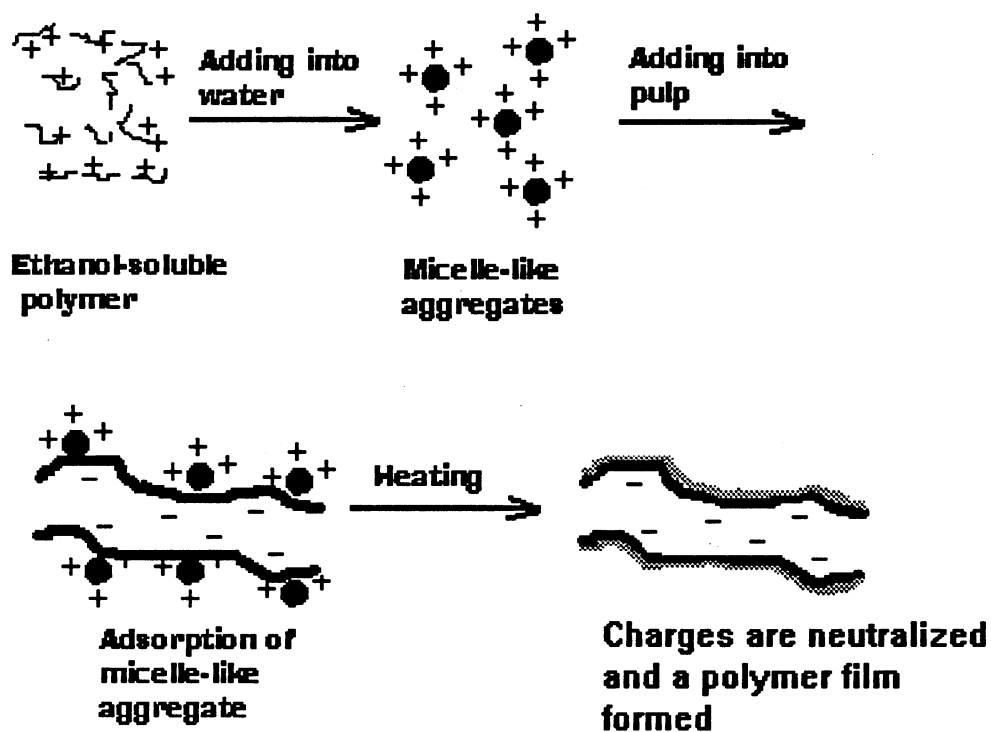


Figure 2. Mechanism of paper sizing by CPMA.

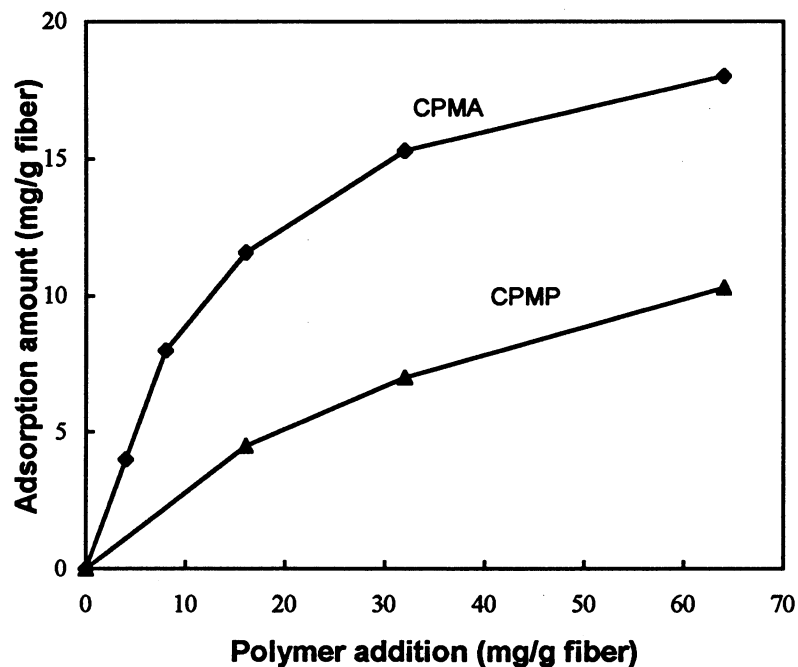


Figure 3. Adsorption isotherm of CPMP (sample D) and CPMA (sample G) on SBK fiber surface. Only clear long fibers were used.

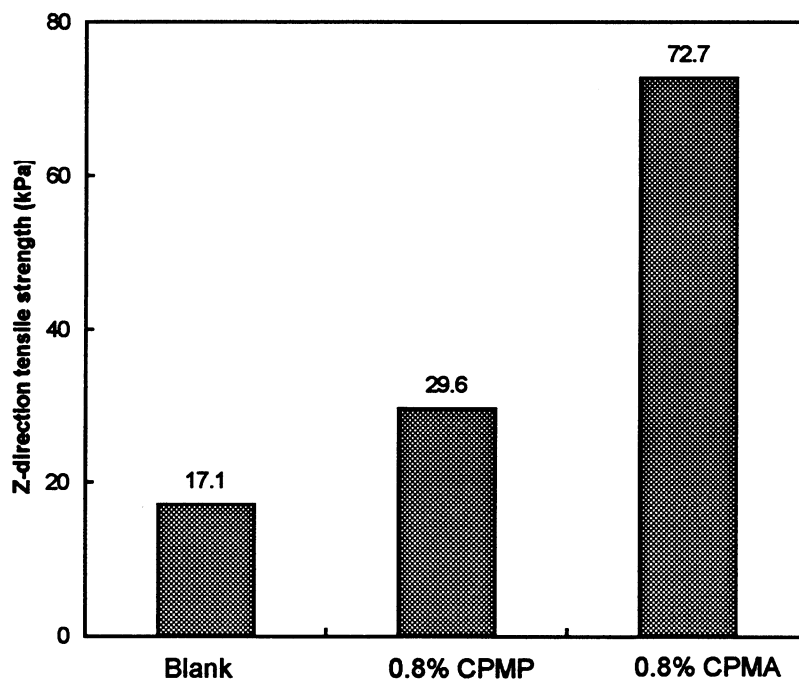


Figure 4. The effect of CPMP (sample D) and CPMA (sample G) on Z-direction tensile strength of handsheet.

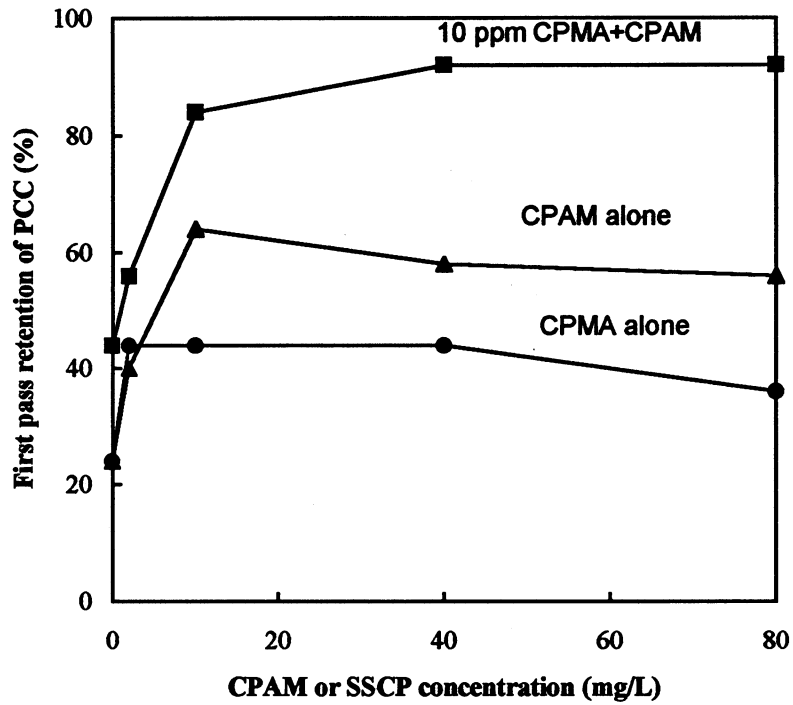


Figure 5. First-pass retention of PCC in SBK pulp. CPMA: sample G; CPAM: high molecular weight, medium-charged cationic polyamide; fiber consistency: 0.4%; filler: anionic PAM pretreated PCC (20% based on dry fiber).

