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CONDITIONING AND DEWATERING OF PULP AND PAPER SLUDGE

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ABSTRACT

The dewaterability of pulp and paper sludges is usualy poor. In this study, biological sludge samples from two pulp and paper mills were conditioned with three cationic polyelectrolytes that have similar molecular weights and different charge densities. Cationic polyelectrolyte of high charge density could efficiently enhance sludge dewaterability and markedly reduce the residual moisture content in the dewatered cake. On the other hand, the introduction of seawater to pulp and paper sludge would increase the demand of the cationic polyelectrolyte.

INTRODUCTION

The high suspended solid concentration in wastewater from pulp and paper mill includes a great portion of hardly biodegradable organic com-

pounds, such as cellulose and lignin (Ghosh and Taylor, 1999). The employment of conventional activated sludge processes to treat these wastewaters is commonly accompanied by the generation of a vast amount of extracellular polymers (ECPs), hence inducing frequent sludge bulking (Halttunen, 1994; Paice et al., 1996; Van Ginkel et al., 1999). On the other hand, the rich fiber content also attracts wide research interests for the sake of recovering these fibers from the pulp and paper sludge (Ozturk et al., 1992; Eroglu and Saatci, 1993; Jackson and Line, 1997).

The generation of primary sludge consists mainly of cellulose, hemicellulose, lignin and other components of wood furnish, and it may additionally contain process chemicals (such as fillings) and bark. Secondary sludge is comprised of biomass, cellulose fibers and lignin (Jokela et al., 1997). Conditioning is always required to achieve satisfactory dewaterability. The extracellular polymers (ECPs) contents in pulp and paper sludge are noted to be higher than those originated from other sources. As a result, sludge dewaterability might be deteriorated and the dosage for chemical conditioners is increased (Pere et al., 1993). The success of conditioning and dewatering of pulp and paper sludge depends upon the interactions between conditioner and the sludge particles. However, related information is still largely lacking in the available literature.

This work employed three cationic polyelectrolytes, which are of similar molecular weights but of different charge densities, for conditioning the waste activated sludge from two pulp and paper mills. We evaluated the dewaterability of the sludge subject to chemical conditioning and the residual moisture in the dewatered cake.

EXPERIMENTAL

Sludge Samples

Activated sludge samples were taken from two pulp and paper mills: Chung Hwa Pulp Corporation, Hualien, Taiwan (Sludge I), and Yung-Feng-Yu Pulp Corporation, Taitung, Taiwan (Sludge II), whose main products are both pulp and paper. For sludge I, the wastewater treatment plant is a typical anaerobic/aerobic system that treats $6.5 \times 10^4\,m^3$ of wastewater daily. Lime (200 mg/l) and seawater (3%, basis of wastewater) are drawn into primary sedimentation tank to improve settleability of suspended solids. Then the wastewater is treated by anaerobic/aerobic process. For sludge II, the wastewater treatment plant is a typical activated sludge system that treats $2.2 \times 10^4\,m^3$ of wastewater daily. Both sludge

samples were taken from the recycled activated sludge. Table 1 lists some details of the wastewater treatment plants.

Suspension solid (SS) determinations were performed in accordance with the procedures outlined in *Standard Methods* (APHA, 1995). The dry solid density, ρ_s, was determined by a pycnometer (Pycnometer, AccuPyc 1330). The chemical oxygen demand (COD) of sludge was measured with direct reading spectrophotometer (DR/2000, HACH). Value of pH was measured by immersing the electrode (Jenco, 6209) 2 cm below the sludge/air interface. ECPs measurement includes three steps, i.e., washing (or unwashing), extraction, and quantification (Poxon and Darby, 1997). In the washing step, a sludge sample of 50 ml was centrifuged at 591 *g* and 4°C for 10 min, and the soluble polymer in supernatant was removed. The sludge pellets were resuspended in deionized water to original volume. In the extraction step, ECPs extraction was carried out at 80°C for 1 h (Morgan et al., 1990). Afterwards, sludge was centrifuged at 14 800 *g* and 4°C for 30 min. The supernatant was poured into a 150-ml beaker, and ethanol was added in 2 : 1 (ethanol : supernatant) volumetric ratio to the beaker, and kept refrigerated overnight for precipitation of ECPs to proceed. The precipitated ECPs were recovered by centrifugation at 17 700 *g* and 4°C for 30 min. In the quantification step, the supernatant was discarded, ECPs in the bottom of the bottle were washed into the evaporating dish, and dried at 36 ± 1°C for quantitative determination (Sanin and Vesilind, 1994). In the unwashing step, a sludge sample of 50 ml was also centrifuged at 591 *g* and 4°C for 10 min, except that the soluble polymer was not removed. The following steps for extraction and quantification were performed per previous procedures.

Table 1. Typical Operating Conditions of the Two Pulp and Paper Wastewater Treatment Plants

	Mill (I)	Mill (II)
Amount of wastewater influent (m^3/day)	6.5×10^4	2.2×10^4
Chemicals added in primary settler	3% seawater and 200 mg/l caustic lime	None
COD of effluent from activated sludge process (mg/l)	120	150
Amount of biological sludge from secondary settler (ton/day)	900	200

The zeta potential was measured using a laser electrophoresis meter (Zeta Master, Malvern). To obtain suitable dilute sample, the flocs formed from conditioned sludge were diluted with supernatant. The dilute solution was ultrasonicated for 30 s to break flocs into small particles (Yu and Somasundaran, 1993).

For sludge I, floc sizes were measured from digital images. The method used was a modification of the procedure proposed by Wu et al. (1998). A glass cylinder (6 cm in diameter and 50 cm in height), sectioned on a side with an attaching plane view glass, was used for the floc size measurement. A CCD-camera was used to record the images of flocs. Two lenses, a close-up lens (Moritex, Japan) and a zoom lens (Tokina, 12.5 mm ~ 75 mm f/1.8), were equipped for the CCD-camera. The close-up lens was used to measure the flocs of size less than 100 μm. The zoom lens was used to measure the floc size larger than 100 μm. A parallel plate was installed in the centerline of the cylinder. Samples of flocs were taken using a wide mouth dropper to prevent damage to flocs. About 20 flocs were allowed to settle in the parallel plate, then the images were recorded. More than 500 flocs were measured in each sample. A software package (Matrox, Inspector V2.2) was used to calculate the projected area (A) in the images, and the equivalent diameter (ED) was calculated by $ED = (4A/\pi)^{0.5}$. For sludge II, floc sizes were analyzed using a particle sizer (L230, Coulter), whose size detection ranges from 0.3 to 2000 μm. Floc samples were diluted before analysis to obtain particle counts within the limits of the analyzer.

Sludge Conditioning and Test

Three cationic polyelectrolytes (Diafloc), KP-201C, KP-156T and KP-108, were provided by Dives Trading Co. Ltd., Taiwan. Charge density was determined with titration of potassium poly(vinyl) sulfate (PVSK), and toludin blue was used as an indicator (Ueno and Kina, 1985). Table 2 lists the polyelectrolyte properties. They have comparable molecular weight ($4–6 \times 10^6$) but different charge density. Stock solution (2000 mg/dm^3) of polyelectrolyte was prepared and aged for at least 24 h before use.

A conventional jar-stirring device was used to mix the sludge. Sludge samples of 500 ml in 1000-ml beaker each were used in the experiments. Polyelectrolyte of the pre-calculated dose was added and mixed at 100 rpm for 30 s. After that, the samples were mixed at 45 rpm for 90 s.

A constant head piston press (Triton Electronics Ltd., type 147) was employed for all tests. A schematic of the experimental setup can be found elsewhere (Chang et al., 1997). The sludge was placed in a stainless steel

Table 2. Properties of Cationic Polyelectrolytes

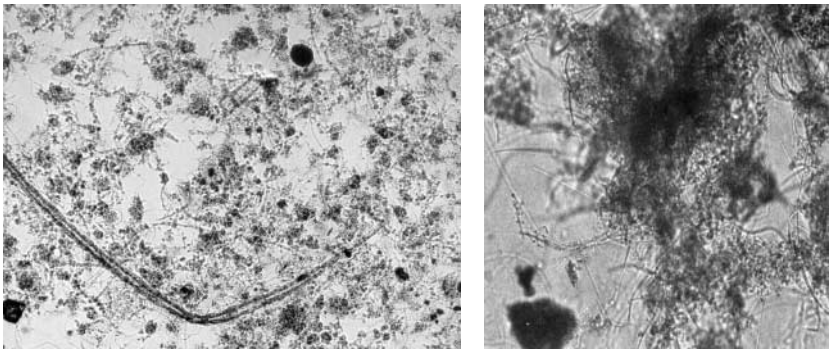
	KP-201C	KP-156T	KP-108
Molecular weight	4×10^6	5×10^6	6×10^6
Charge density (meq/g)	5.6	1.3	0.53
Price (US$/kg)	7.4	5.5	5.2

cylinder of diameter 7.6 cm and of height 20 cm equipped with a free piston. The cylinder is coated with chrome with one end connected to a port where the hydraulic pressure of 20 MPa was exerted. This high pressure pushes the free piston to squeeze water out of the sludge. An electronic balance connected to a personal computer automatically recorded the evolution of filtrate weight with time. The residual moisture content of filter cake was determined via weighing and drying. This portion of moisture should include some physically adsorbed and other chemically bounded water contents (Lee and Hsu, 1995). With the filtrate weight versus time data and the true solid density data, the time evolution of cake porosity can be subsequently obtained. For each sample, three independent tests were conducted to check the reproducibility.

RESULTS AND DISCUSSIONS

Sludge Characteristics

Figure 1 depicts the microscopic photographs of sludge flocs (100×), which were taken with the assistance of Projection Microscopes 4013 (Projectina) and Polaroid camera. It is found that some filamentous bacteria form rigid backbones for the floc, to which other microorganisms attach like flesh. Table 3 compares the measured sludge characteristics. Previous measurements of ECPs have used washed samples, thereby excluding soluble polymers (Sanin and Vesilind, 1994; Dignac et al., 1998). The difference of extracted ECPs between washed and unwashed samples was considered as soluble polymer. Notably, although both sludges are from pulp and paper mills, the binding ECPs in sludge I were less than sludge II. However, the soluble ECPs in sludge I were nearly twice those of sludge II. The floc size of sludge I (35 μm) is much smaller than that of sludge II (233 μm). Moreover, the solid density of the former is lower than that of the latter. The smaller size and lower density of flocs of sludge I may be due to the presence of excess



(a) (b)

Figure 1. Microscopic photographs of sludge flocs (100×), a) sludge I, b) sludge II.

Table 3. Characteristics of the Pulp and Paper Sludges

	Sludge I	Sludge II
pH	6.9	6.9
Solid weight percent (%)	0.83	1.22
ρ_S (kg/m^3)	1380	1820
Particle size (μm)	35	233
Zeta potential (mV)	−25.7	−28.3
Washed ECPs (mg/g DS)	36.4	45.1
Unwashed ECPs (mg/g DS)	69.7	58.7

sodium ions brought about by seawater used in the primary clarifier. The divalent metal ions of flocs are replaced by sodium ions that resulted in the release of bound ECPs to liquid phase which decreases floc density (Higgins and Novak, 1997). Additionally, a vast amount of soluble ECPs was found. Despite these differences, both sludges exhibit a similar zeta potential.

Sludge Conditioning

Figure 2 depicts the measurements of zeta potential for the original and the flocculated sludges. The KP-201C, which exhibits the highest charge density among the three conditioners, would neutralize the surface charge of sludge I (Figure 2a) and sludge II (Figure 2b) at a dose of 15 and 5 kg/ton

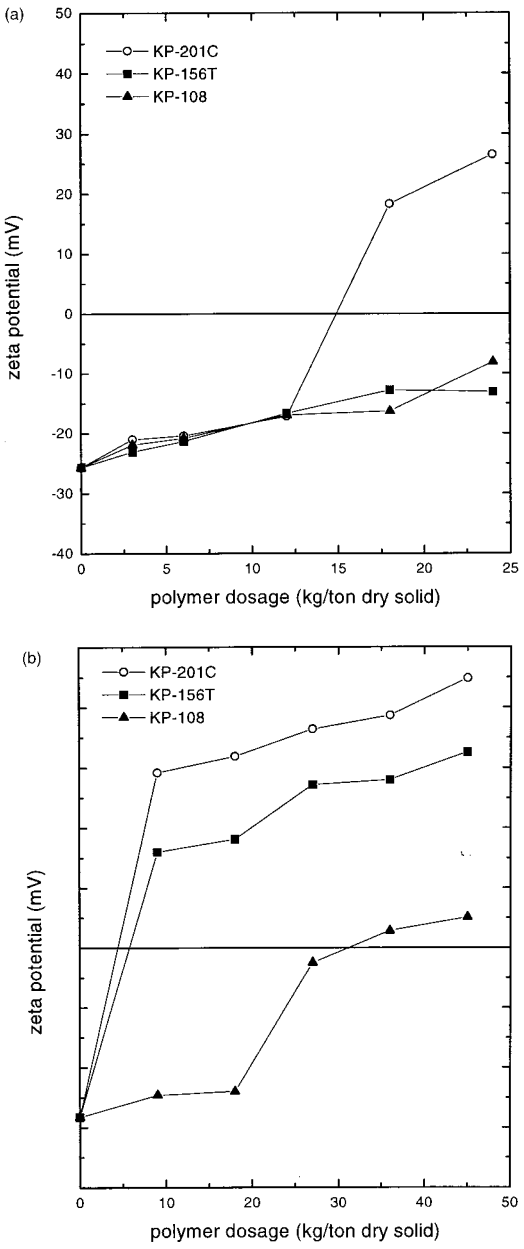


Figure 2. Zeta potential of flocculated sludges at different polyelectrolyte dose, a) sludge I, b) sludge II.

DS, respectively. Meanwhile, within the experimental range, the KP-156T (of medium charge density) and KP-108 (lowest charge density) could only neutralize the surface charge of sludge II at doses of 5–7, and 30–35 kg/ton DS, respectively; but no charge reversal was found for sludge I. This confirms the finding that the dose required to neutralize particle surfaces decreased with increasing charge density of polyelectrolyte (Lee and Gregory, 1991). Again, the utilization of seawater in the primary clarifier might cause changes in surface characteristics of sludge I. The higher amount of soluble ECPs resulted in higher dose of polymer needed to neutralize surface charge of sludge particles.

Figure 3 depicts the results of floc size measurements. Floc size increased markedly on the addition of polyelectrolyte. When sludge I was conditioned with KP-201C, floc size was too large to measure by the video recording as it exceeds 6 kg/ton DS. The increase in floc sizes follows the sequence: KP-201C > KP-156T > KP-108. The application of KP-201C or KP-156T would yield larger flocs in sludge II. However, the polymer with lowest charge density (KP-108) could only slightly enlarge the constituent flocs. Bajpai (1997) proposed that polyelectrolytes might take rod-like or cylindrical conformations at extremely high charge density. At low charge density or in the absence of any charge, they often take a more spherical conformation. The stretched conformation of KP-201C provided a better interparticle bridging that resulted in larger floc size. In sludge II, the floc strength of these large flocs might become relatively weak. The appearance of the maximum floc size (580 μm) of KP-201C flocculated sludge might be due to the compensation between floc growth and the floc breakage effects in the flow chamber of the sizer. The measurements for KP-156T sludge also reveal a large data scattering. Regardless of the possible role of polyelectrolyte, most large flocs would settle to the bottom of container within one to two minutes and form a loose cake because all sludge flocs after flocculation would exceed 280 μm in size.

Sludge Dewatering

Figures 4 and 5 illustrate the compression dewatering curves for the original and flocculated sludges. Each curve consists of two stages: the filtration stage (stage F) and consolidation stage (stage C). At an applied load of 20 MPa, sludge I needs 2000 s to reach a moisture content of 10 kg/kg DS, while that for sludge II is 3900 s. Restated, the former exhibits a better dewaterability than does the latter. However, the application of the polyelectrolyte could enhance the dewaterability of sludges, with the degrees

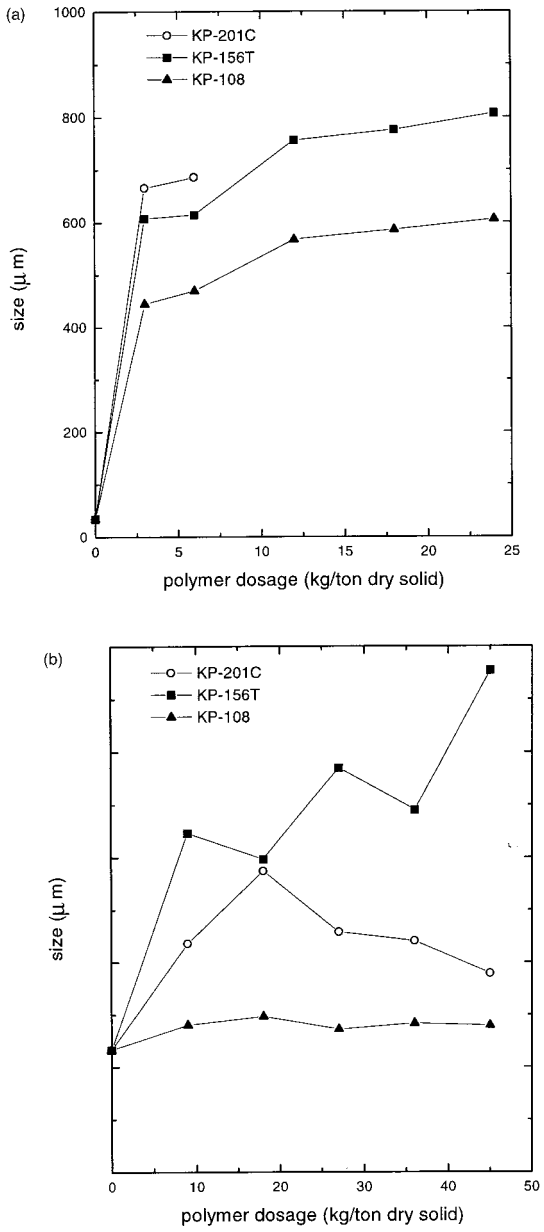


Figure 3. Floc size of flocculated flocs at different polyelectrolyte dose, a) sludge I, b) sludge II.

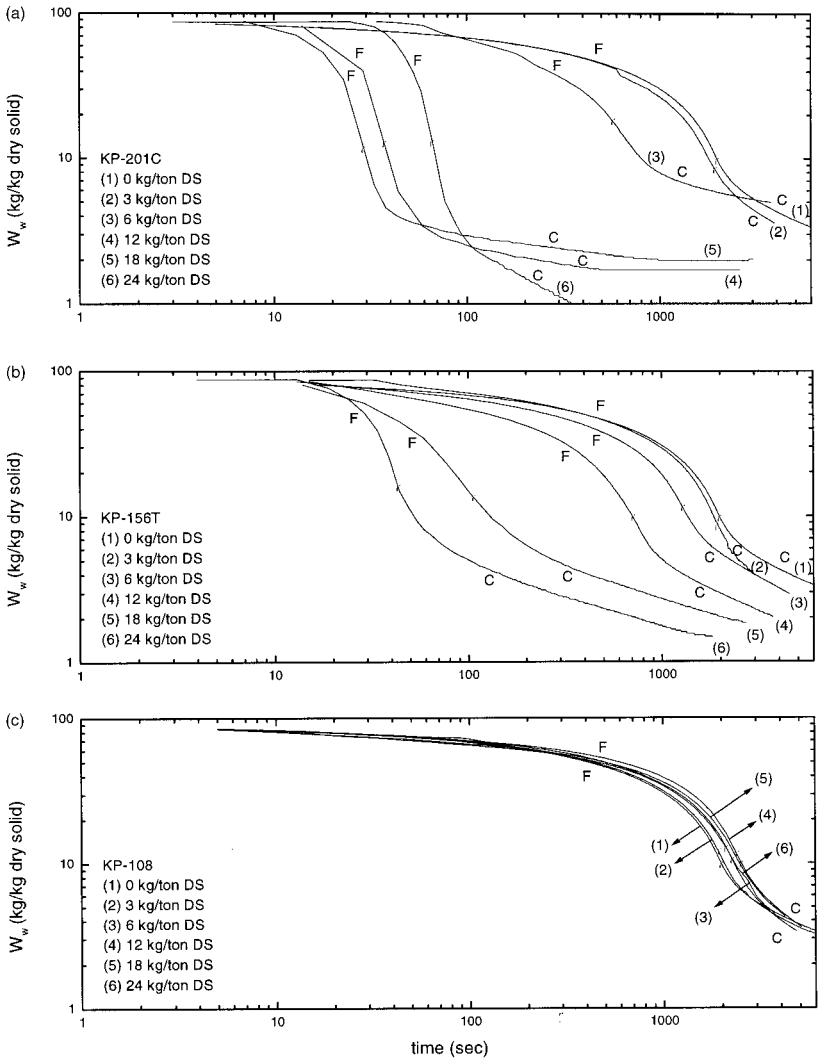


Figure 4. The overall dewatering curves of sludge I respectively flocculated with: (a) KP-201C; (b) KP-156T; (c) KP-108.

of enhancement increases with increasing charge density of the polyelectrolytes. For example, the sludge conditioned by KP-201C always has the best dewaterability for both sludges, and KP-108 could not significantly improve their dewaterability. Furthermore, as noted in Figures 4 and 5 when the

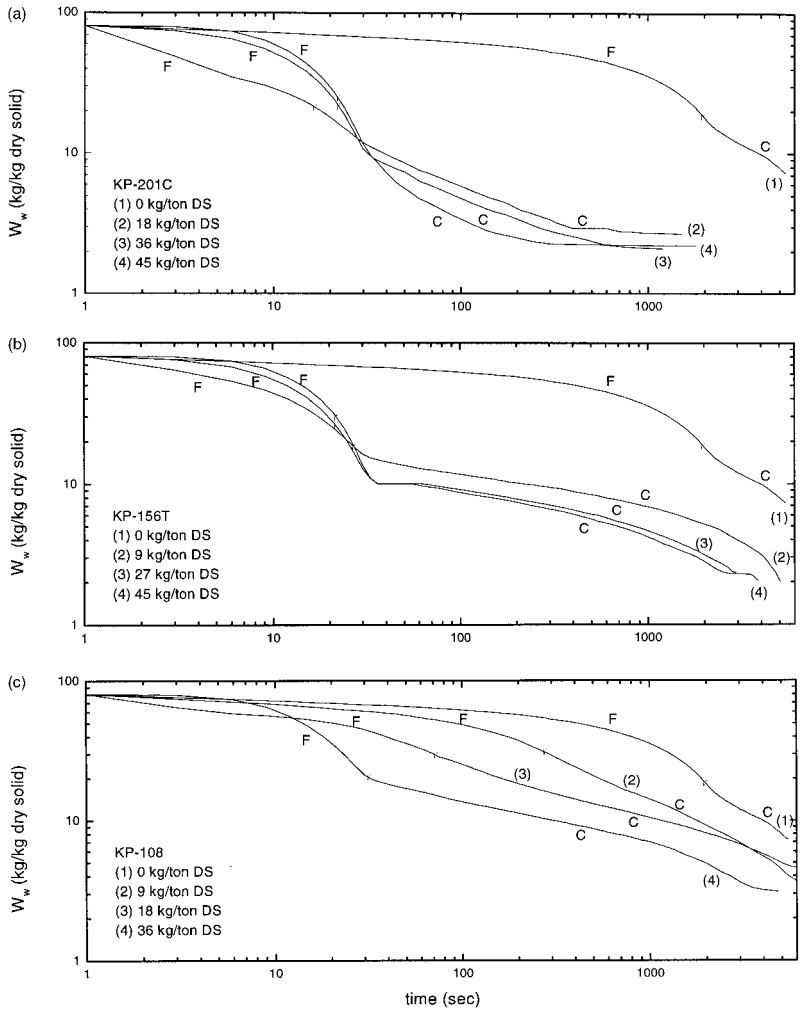


Figure 5. The overall dewatering curves of sludge II respectively flocculated with: (a) KP-201C; (b) KP-156T; (c) KP-108.

filtration stage was enhanced, the following consolidation stage became retarded instead. One also could observe "rebound" of the dewatering curve at polyelectrolyte dosage somewhat higher than the corresponding charge neutralization dosage. For example, the dewatering curves for

sludge I shifted to the left with increasing polyelectrolyte dose of 18 kg/ton DS, then moved back at higher doses. Such a dose is herein regarded as "optimal" which is slightly higher than the charge neutralization dose (ca. 13–15 kg/ton DS). Similar pattern could be noted for sludge II, but the "rebound" occurred at 9 kg/ton DS, which is much higher than the corresponding charge neutralization point (5 kg/ton DS). These findings, together with the fact that the employed polyelectrolytes are of a similar molecular weight, suggest that charge neutralization might not be the sole mechanism during the pulp and paper sludge conditioning. Other mechanisms such as bridging may also be involved.

The filtration and expression stages were distinguished using the graphic method proposed by Shirato et al. (1970), which were respectively indicated as symbols F and C in Figures 4 and 5. The corresponding average resistance to filtration (SRF) data for the filtration stage could be obtained according to the procedures proposed by Leu (1981), and are depicted in Figure 6. The original sludges exhibit an SRF value ranging from 2 to 3×10^{15} m/kg. Restated, both pulp and paper sludges could be categorized as "very difficult to filter" sludges. The SRF for sludge flocculated with KP-201C or with KP-156T could be reduced by more than an order of magnitude when subject to their optimal doses. Such a marked reduction suggests the benefit of polyelectrolyte conditioning. In the overdosing regime the increase in resistance is also noticeable. The control of the applied dose is hence very important for conditioning these sludges. It is noted that, although less efficient when compared with the other two polyelectrolytes, KP-108 could still reduce the cake resistance for sludge II at a higher dosage. Nevertheless, corresponding to the dewatering curves for sludge I depicted in Figure 4, the SRF is not affected by the presence of KP-108.

The consolidation stages (stage C in Figures 4 and 5) were differentiated from the filtration stage and redrawn in Figures 7 and 8, in which U_c is the consolidation ratio. Although the presence of polyelectrolyte still helps in enhancing the efficiency of consolidation process, the dependence of polyelectrolyte dose, however, is very different from that observed in filtration stages (Figures 4 and 5). First, the shift in consolidation curves showed the "rebound" characteristic for sludge I, which was not observed for sludge II. Second, although the KP-201C still yielded the fastest consolidation, the polymer with lowest charge density, KP-108, could be as efficient for improving consolidation efficiency as that with KP-156T.

The three-stage rheological model proposed by Chang and Lee (1998) is applied herein to describe the cake response under constant pressure compaction. The $\ln(1-U_c)$ versus consolidation time relation, if linear in

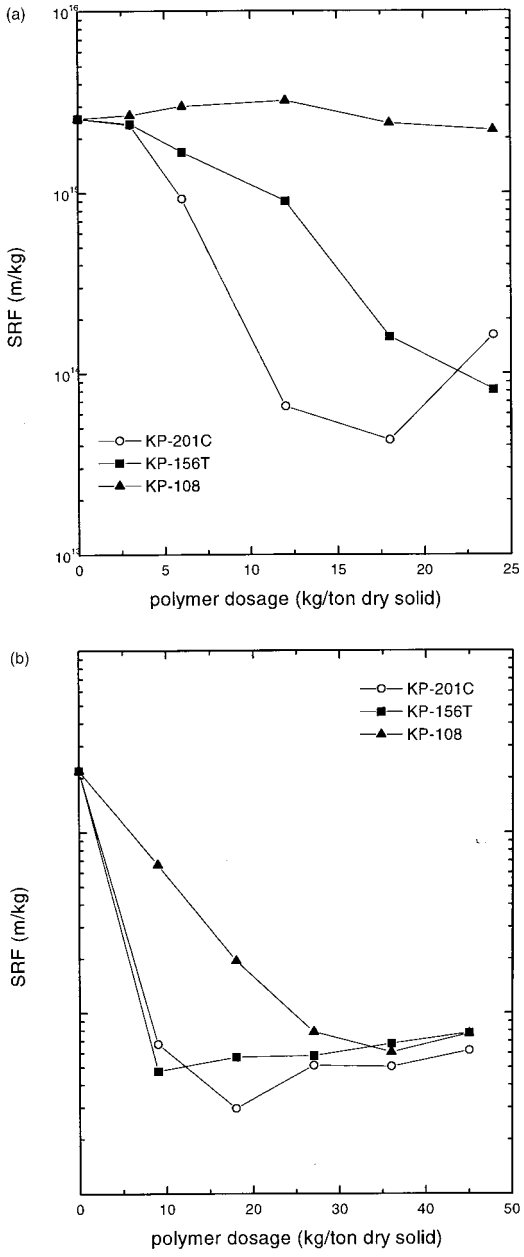


Figure 6. The specific resistance to filtration of filter cake at different polyelectrolyte dose, a) sludge I, b) sludge II.

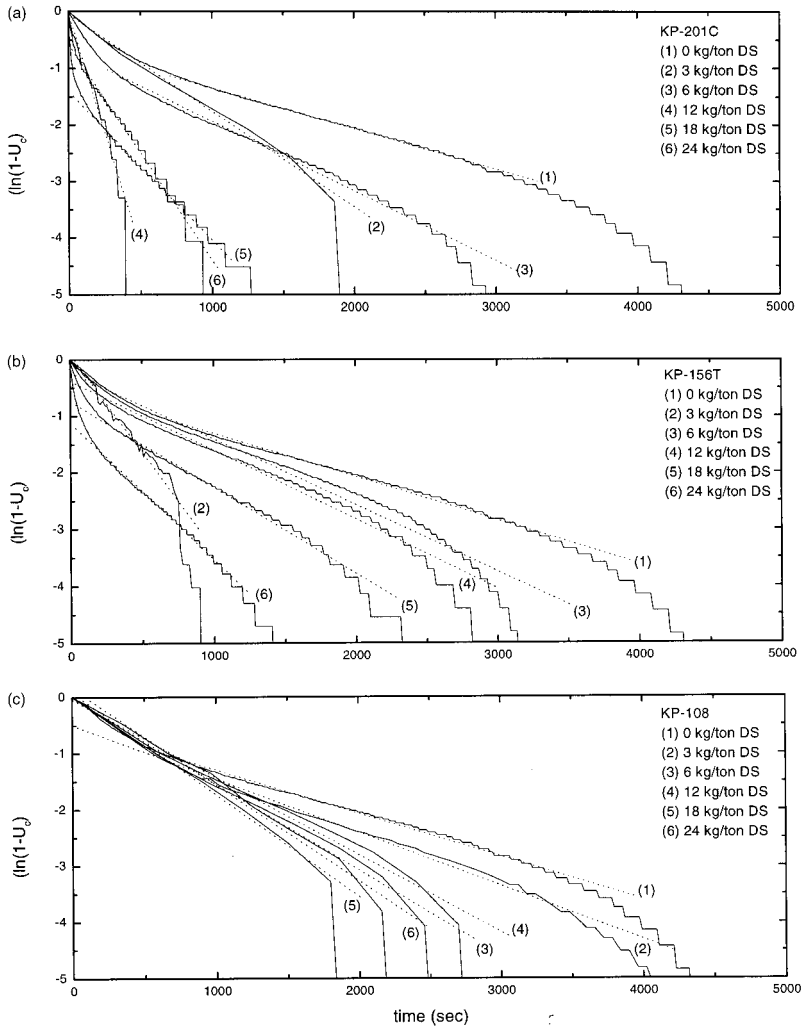


Figure 7. Expression curves of sludge I respectively flocculated with: (a) KP-201C; (b) KP-156T; (c) KP-108.

character, could be used to estimate the rheological parameters η and B, where η denotes the creep factor between constituent particles and B, the fraction occupied by the so-called "secondary consolidation stage" (Shirato et al., 1974). The dash lines in Figures 7 and 8 demonstrate these linear

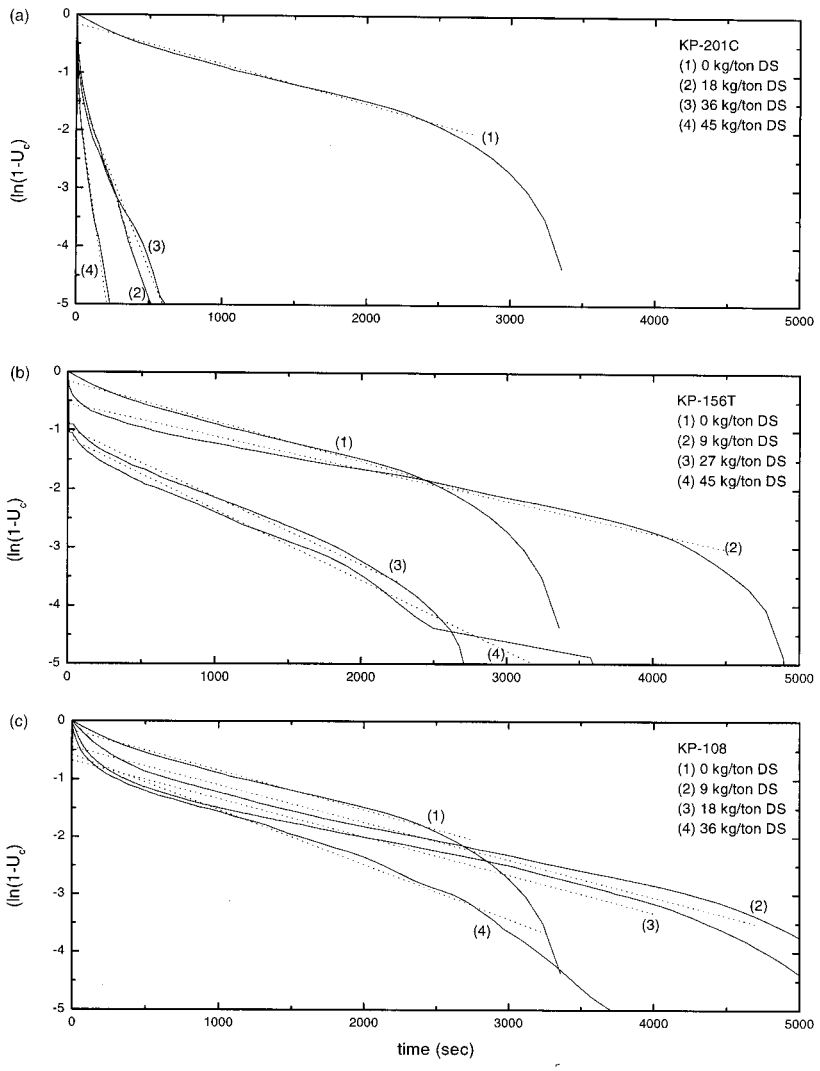


Figure 8. Expression curves of sludge II respectively flocculated with: (a) KP-201C; (b) KP-156T; (c) KP-108.

relations. Figures 9 and 10 depict the parameters B and η, respectively. A cake of smaller B value denotes a weak intra-aggregate strength, while a large η refers to the lesser inter-aggregate interactions. A cake of small B and/or large η is favorable to consolidation dewatering.

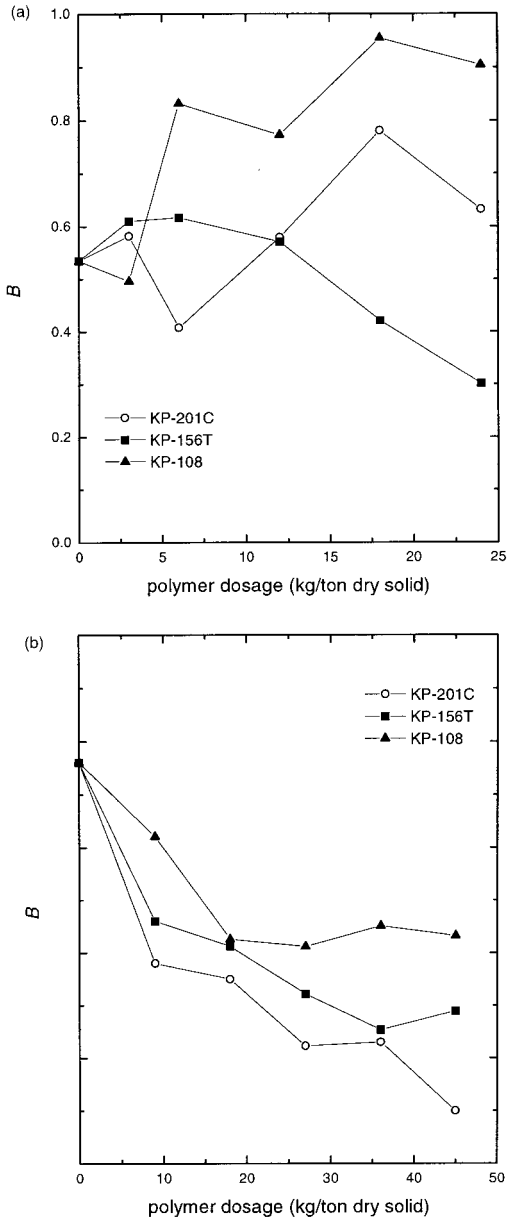


Figure 9. Secondary consolidation ratio at different polyelectrolyte dose.

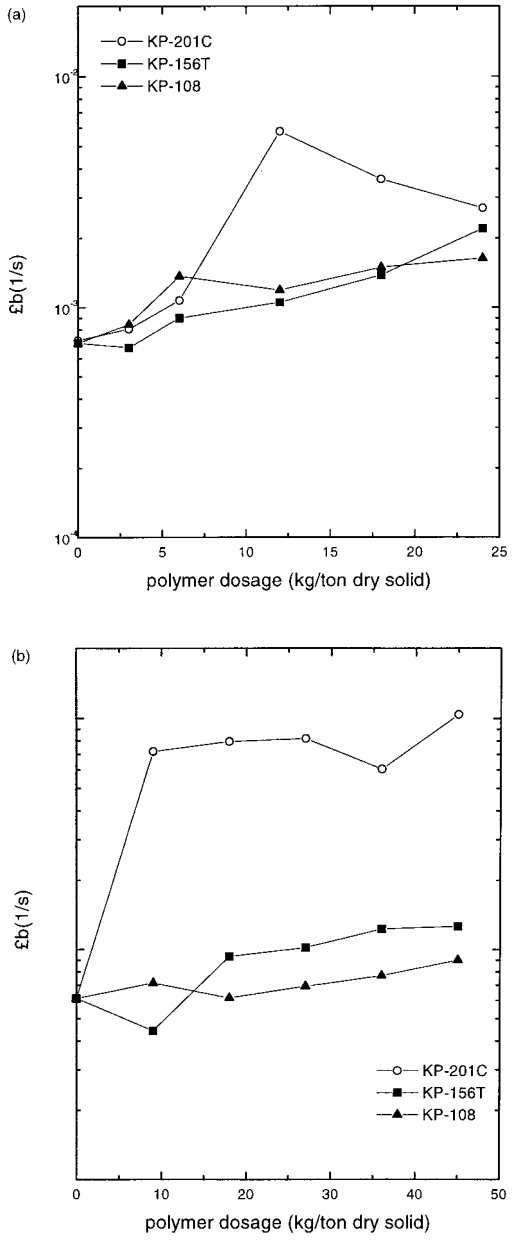


Figure 10. Creeping factors of constituent particles at different polyelectrolyte dose.

As shown in Figure 9, the original sludge I and sludge II exhibit a B value of 0.53 and 0.76, respectively, indicating that the cake structure of the former is easier to collapse than is the latter. Flocculation changes the cake rigidity. For sludge I, although with large data scattering, conditioning with KP-108 would lead to a more rigid cake. The sludge conditioned with KP-156T would on the contrary lead to a softer cake. For sludge II, on the other hand, B value decreases with the polyelectrolyte dose. Restated, the flocculation would always yield a cake of lower strength to resist the applied load. There is no correlation to the charge neutralization points noted in Figure 2.

The creep factor illustrated in Figure 10 shows a different trend. The original sludge I and sludge II exhibit η values of 7.3×10^{-4} and $6.1 \times 10^{-4}\,s^{-1}$, respectively, indicating that the constituent particles in the cake of sludge I would creep more easily than those in sludge II. This result, together with the smaller B value demonstrated in Figure 9, suggests a better consolidation efficiency for the sludge I when compared with sludge II. Conditioning with KP-201C would significantly facilitate the relative motion among the particles in consolidated cake. The best mobility closely correlates with the corresponding charge neutralization points (13 and 5 kg/ton DS for sludge I and II, respectively). Chang et al. (1997) also found that the particle creeping becomes the easiest when the suspension is conditioned to the charge neutralization point. These authors speculate that such a phenomenon is attributed to the elimination of electrostatic repulsive force when particles creep. However, the conditioning with KP-156T could not significantly increase the η value at the charge neutralization point. Therefore, the electrostatic effect could not be generally applied to interpret the conditioning effects of the present pulp and paper sludge.

A dry filter cake is beneficial for final disposal of sludge. Figure 11 depicts the bound water content at the end of consolidation ($W_{W,EXP}$). The large data scattering indicates the relatively large error bar for such a measurement. The bound water content for original sludge I is merely 40% of that of original sludge II. Therefore, although the sludge volume generated for sludge I is greater than that from the sludge II, however, the dewatered cake volume needed to be disposed of for the latter would be larger than the former. The presence of polyelectrolyte would not markedly alter the bound water content for sludge I. On the other hand, the conditioning would markedly deplete the moisture from the sludge cake for sludge II. Hence, the application of polyelectrolyte to sludge II could not only promote the mechanical dewatering efficiency, but also reduce the final cake volume to be disposed of.

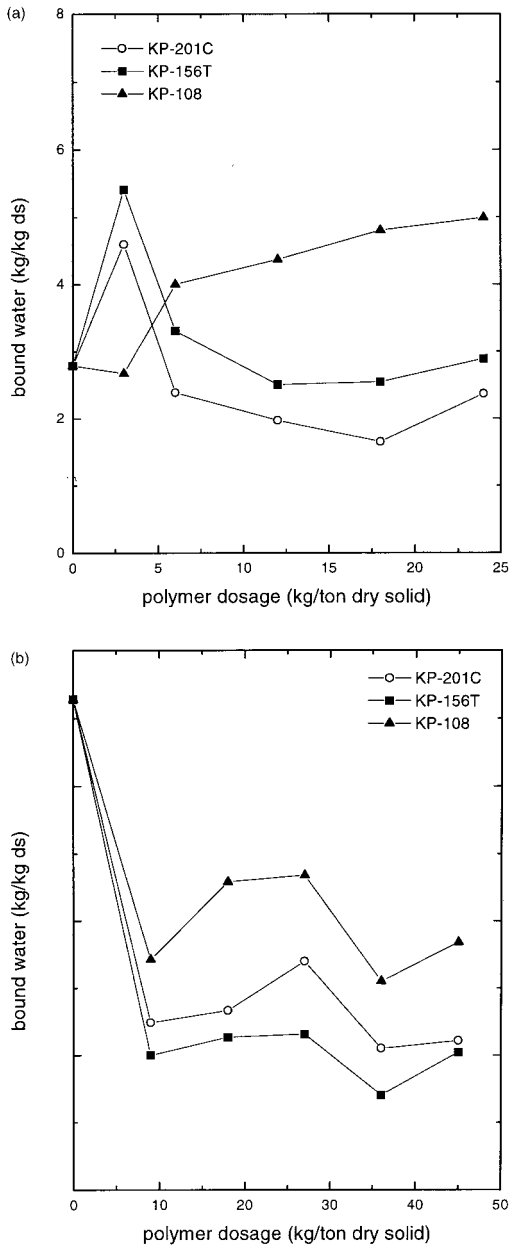


Figure 11. The residual moisture at the end of expression test at different polyelectrolyte dose.

CONCLUSION

Pulp and paper sludges contain a vast amount of extracellular polymers (ECPs) and have a poor dewaterability. Three cationic polyelectrolytes of similar molecular weight but very different charge density were tested. Filtration followed by consolidation tests were conducted to characterize sludge dewaterability. Cationic polyelectrolyte conditioning could efficiently enhance sludge dewaterability and markedly reduce the residual moisture content in the dewatered cake. Rheological model was used for interpreting the consolidation data. To condition the sludge with the conditioner of the highest charge density would lead to rigid filter cake. On the other hand, to apply seawater to pulp and paper sludge would yield an increased demand of the cationic polyelectrolyte.

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