



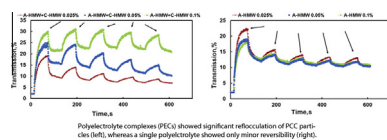
Effect of charge balance and dosage of polyelectrolyte complexes on the shear resistance of mineral floc strength and reversibility



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GRAPHICAL ABSTRACT



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ABSTRACT

We evaluated the effect of polyelectrolyte complexes (PEC) with varying balance of charges on the flocculation of precipitated calcium carbonate (PCC) particles. PECs composed of polyacrylamides carrying opposite charges (A-PAM and C-PAM) were investigated in terms of PCC floc shear resistance and re-flocculation effects. Light transmission was used in real time to monitor the dynamics of flocculation under shear fields. Compared to the single polyelectrolytes, PECs greatly enhanced particle re-flocculation while minor differences in shear resistance were observed. Shear resistance and re-flocculation depended strongly on the molecular weight and charge ratio of the PEC components. In order to achieve floc stability and re-flocculation conditions a minimum concentration of charge-asymmetric PEC should be applied.

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1. Introduction

Mineral particles or fillers are of great interest to improve fiber product quality and to reduce raw material costs in papermaking. Polymeric flocculants are commonly used to retain fillers, with characteristic small, colloidal size on the papermaking forming fabric during water removal by hydrodynamic forces and suction. Typically, long chain polymers (flocculants) are used to adsorb on suspended particles and to bridge them via loops and tails that extend from their surfaces. The polymers that most efficiently form bridges between adjacent particles have a high molecular mass (several million Dalton), moderate charge density and/or have a branched architecture. In order to further improve filler retention, several kinds of retention systems have been developed in recent years, mainly by combining two or more polymers.

Retention aids have a great influence on flocculation phenomena in the wet end of papermaking machines. The respective addition points should be selected carefully according to several factors affecting fiber and filler flocculation efficiency and formation [1,2]. Paper machines contain sections that expose the fiber aqueous suspension to high shear, such as in the dilution or fan pump or in the pressure screens [3]. The high shear forces in such units tend to break down flocs formed after polyelectrolyte addition. This is due to the disruption of the attachment points on particle surfaces or by breaking the covalent bonds within the bridging polymer chains [1]. Therefore, floc strength is important to ensure high fine and filler first pass retention [4]. Retention aids that flocculate by the so-called electrostatic patching mechanism tend to break apart easily when subjected to shear. This is in contrast to flocs formed by bridging, which under same condition are usually stronger and have a loose, large hydrodynamic volume with temporal resistance to shear (sustained high shear can break them apart, though).

Single component, high mass flocculants may allow particles to disperse irreversibly if a given level of shear is maintained [5–8].

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However, the shear stress can be reduced drastically in the paper-making forming section, which may lead to particle re-flocculation. Blanco et al. [9,10] found that reversible flocculation or re-flocculation is only possible at low polymer dosages.

By using combinations of different additives, re-flocculation ability can be increased significantly. Swerin et al. [11] reported on sequential treatments consisting of cationic starch and colloidal silica while Hubbe [12,13] found that microparticle treatments produced the highest level of reflocculation. Yukselen et al. [14] studied the breakage and re-flocculation of clay flocs. They reported that compared to single polyelectrolytes, a combination of high charge density polydiallyldimethylammonium chloride (poly-DADMAC) and high molecular mass anionic polyacrylamide (A-PAM) forms strong, reversible flocs. Recently, Deng and Yoon [6] studied the re-flocculation of clay suspensions under shear with dual systems including poly-DADMAC with A-PAM, poly-DADMAC with cationic PAM (C-PAM) and C-PAM with microparticles. They observed that these systems displayed a higher re-flocculation ability compared to the single polymers.

Several techniques are available to monitor flocculation in real-time, such as the photometric dispersion analyzer (PDA), which is a flow-through turbidity device, and the focused beam reflectance measurement (FBRM), which is based on the backscattered light flux from particles. These techniques have been used to monitor flocculation with different chemical systems and under various shear conditions [9,10,14–16]. In this study, experiments were carried out with a Turbiscan Online optical analyzer [17], which measured in real time both transmitted and backscattered light flux from the dispersions.

For dual retention systems such as C-PAM and colloidal silica, re-flocculation has a strong positive impact on dewatering due to a more open paper web structure. Relevant to this study is our previous finding indicating that compared to single component polymer systems a more efficient filler flocculation takes place with pre-mixed polyelectrolyte complexes (PEC) [18]. It was also shown that the efficiency of flocculation with PEC significantly depended on the properties of its components (charge density, molecular mass and mixing ratio). Earlier we demonstrated that dewatering of fiber suspensions can also be improved by using PEC [19]. Overall, the general goal of this effort was to further elucidate the flocculation mechanism of PEC systems, especially in relation to the extent of re-flocculation. Thus, we evaluated the shear resistance and re-flocculation phenomena of flocs formed with pre-mixed PECs and the effects of PEC composition, dosage and shear cycles. These are subjects that to our knowledge have been reported only to a limit extent in the literature.

2. Materials and methods

2.1. PCC and polyacrylamides

Experiments were carried out with dried precipitated calcium carbonate (PCC, mean particle size of ca. 2 μm , specific surface area of ca. 10 m^2/g , according to the manufacturer), which was provided by Specialty Minerals Inc., USA. PCC was dispersed to a dry solids content of 20–30 wt% in distilled water in the absence of any dispersing agents. For the experiments the PCC dispersions were diluted to a solids content of 0.1 w-% in 1 mmol/l NaHCO_3 and 9 mmol/l NaCl. The pH of the dispersions was adjusted with 0.1 M HCl to 8.5. Under these conditions the ζ -potential of PCC was ca. +15 mV (positively charged).

Cationic and anionic polyacrylamides were obtained from Kemira Pulp and Paper, Finland. Three cationic and one anionic polyacrylamides with different molar masses were used. See Table 1 for nomenclature, molecular weight (from manufacturer)

Table 1
Polyacrylamides of low charge density used as PCC flocculant.

Sample code	Description	MW $\times 10^{-6}$	Charge density (meq/g)
A-HMW	Anionic, high molecular weight	7.0	−1.6
C-HMW	Cationic, high molecular weight	7.0	1.2
C-MMW	Cationic, middle molecular weight	2.0	1.4
C-LMW	Cationic, low molecular weight	0.5	1.4

and measured charge density (polyelectrolyte titration). Electrolytes (NaCl and NaHCO_3) were of analytical grade. Deionized and distilled water were used in all experiments.

2.2. Polyelectrolyte complexes (PEC)

Four polyelectrolyte complex systems were studied (Table 2). Cationic and anionic polyacrylamides were mixed together for asymmetric charge ratios (anionic:cationic) of 3:1, 2:1, 1:2. According to the measurements reported previously [18], the PECs of 3:1 and 2:1 ratios were anionic while that of 1:2 was cationic. Before complex formation the A-PAM and C-PAM stock solutions (1 g/l each and prepared one day before experiments) were diluted to a concentration of 0.25 g/l and the salt concentration was adjusted to 1 mmol/l NaHCO_3 and 9 mmol/l NaCl (pH with buffer was 8.5). PECs were then prepared by mixing these solutions by using a magnetic stirrer (500 rpm, 1 h) resulting in a final PEC concentration of 0.25 g/l.

2.3. Flocculation tests

The flocculation of PCC was studied with a Turbiscan online optical analyzer (Formulation, France). In this device a near-infrared focused LED (wavelength 850 nm) illuminates the scattering medium flowing through a cylindrical glass tube. Both the transmitted (detection angle 0°) and the backscattered (detection angle 135°) light flux are measured. Detailed information on this method is presented elsewhere [17].

For the experiments the Turbiscan online was built into a circulation system similar to the one presented previously [18]. The sample vessel was equipped with baffles in order to prevent vortex formation. The sample size was 600 ml and it was continuously pumped (ca. 10 cm^3/s flow rate), through the measurement cell (mixing speed 500 rpm) before starting the experiment. The solids content of the suspension was 0.1% and the salt concentration 1 mmol/l NaHCO_3 and 9 mmol/l NaCl. The data points were measured several times, however, the profiles reported correspond to single runs as averaging is impractical. We note that hundreds of test points have been collected with the Turbiscan online unit for the studied systems and the results have a high confidence level. A standard deviation of less than 5% can be cited.

Table 2
Composition (as anionic:cationic polyelectrolyte ratio) of PECs. The 3:1 and 2:1 systems were net anionic and the 1:2 ratio PEC was net cationic.

Polyelectrolyte	A-HMW: high MW
C-HMW: high MW	3:1, 2:1, 1:2
C-MMW: middle MW	3:1, 2:1, 1:2
C-LMW: low MW	3:1, 2:1, 1:2

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