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Effect of Fixing Agent Dosage on the Mechanism of Colloidal Substances Retention onto Pulp

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Three polyamine fixing agents with increasing molecular weights (m.w.), PA-Lw, PA-Mw, and PA-Hw, were used to treat a deinked pulp at three different levels of chemical dosage. The objective was to elucidate whether the retention mechanism of colloidal substances (CS) onto fibers by a fixing agent is different when the dosage is different. The results show that, for the polyamine with the lowest molecular weight (PA-Lw), it performed in the colloidal fixation mode over a wide range of dosage, but re-dispersion of CS took place in the pulp when its dosage was increased to a level high enough but still beneath the charge reversal point. For the polyamine with the highest m.w. (PA-Hw), CS re-dispersion was not observed over the whole dosage range, but a small part of the colloidal agglomeration coexisted with colloidal fixation even when the dosage was very low. For the polyamine with the middle m.w. (PA-Mw), both CS re-dispersion and colloidal agglomeration were observed. This study showed that if one wants to determine the dosage of a fixing agent during CS control better, both CS removal ratio and CS agglomeration behavior should be considered.

Keywords: Fixing agent; Polyamine; Dissolved and colloidal substances; Agglomeration; Fixation; FBRM

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INTRODUCTION

Due to the increasing use of recovered fibres and high yield pulps, and the adoption of tightly closed whitewater recirculating systems in the wet-end of a modern papermaking system, high concentrations of dissolved and colloidal substances (DCS) are often accumulated in white water, and these may cause severe pitch and stickies deposit problems. Polymeric chemicals, called fixing agents or fixatives and usually having high cationic charges and relatively low molecular weights, are often used to neutralize the negative charges of the DCS components and “fix” them onto fibers. As a result, the DCSs are removed out of the system as a part of the paper product, and deposit problems are greatly alleviated. Typical fixing agents include the polyamines (PAs), polyvinylamines (PVams), polydimethyldiallylammonium chlorides (PDADMACs), polyethyleneimines (PEIs), and specially-made highly cationic starches (Neimo 1993; Allen and Filion 1996; Esser *et al.* 2001; Yu *et al.* 2003; Maher *et al.* 2007; Miao *et al.* 2013). However, the term “fixing agent” or “fixation” has actually not been well defined. One simple example is when a fixing agent is dosed to a pulp and interacts with colloidal substances, it remains unclear whether the colloidal particles are fixed onto the fiber in a form where they retain their original single particle state, *i.e.*, the so-called “colloidal fixation" mechanism, or if they are coagulated first into bigger agglomerates before being retained by the fiber mat during dewatering, *i.e.*, the so-called "colloidal agglomeration" mechanism that is associated with "mechanical trapping". Mechanical trapping requires that the agglomerate be big enough to be trapped by the fiber mat. When a fixing agent is used, colloidal fixation is desired but colloidal agglomeration is not, in that many CS agglomerates are still small enough to pass through the wet webs during sheet formation. These agglomerates suspended in whitewater have a much higher tendency to deposit onto surfaces of the equipment, which deteriorates the paper machine runnability and paper product quality (Leduc *et al.* 2005; Chen *et al.* 2007; McLean *et al.* 2009; Zabihian *et al.* 2012; Pruszynski 2009; Wagberg *et al.* 2007; Mosbye *et al.* 2003; Kekkonen *et al.* 2001). Therefore, for a successful fixing agent program, it is important to fix the colloidal particles onto wood fibers before they are agglomerated with each other. To achieve this objective, choosing a proper fixing agent and using it at proper dosage are critically important.

In one of our previous works, five polyamine fixing agents with different molecular weights and slightly different charge densities were used to treat a deinked pulp, and the fixation mechanism was probed with the help of a technique called focused beam reflectance measurement (FBRM). The results showed that polyamines with smaller molecular weight tend more to do colloidal fixation, while polyamines with higher molecular weight tend to do more extensive colloidal agglomeration (Wang *et al.* 2014). However, the investigations were done with the dosages of the polyamines fixed at a reasonable dosage (0.22% of dry chemical to dry pulp). Later, it was realized that for a certain fixing agent, its mechanism in interacting CS particles might be different at different dosages. The work was continued in the present study; however, to simplify the process, this time only three different polyamines were used, at three different dosage levels.

EXPERIMENTAL

Materials

The polyamine fixing agents were synthesized in a laboratory according to known methods (Joo *et al.* 2003). The solids contents of the polyamine solutions were determined by drying the samples in a dry oven at 105 °C to constant weight. The molecular weights of the polymers were indirectly expressed as intrinsic viscosity which was measured with a 4-0.55/16 Ubbelohde viscometer in a 30 °C water bath, with the polymers being dissolved in a 1 M NaCl solution. The charge density of the polyamines were determined according to polyelectrolyte titration with a standard 0.001 N sodium polyethylene sulphonate (PES-Na) solution, using a charge analysis system (CAS) by AFG Analytic GMBH in Germany.

The pulp used in this study was a deinked one sampled from a newsprint line from Zhengda Papermaking Co. Ltd., in the Shangdong Province of China. The pulp consistency was determined according to TAPPI Standard T240 om-02. The charge demand of the whole pulp suspension was analyzed by the back titration method, *i.e.*, a portion of the pulp was overcharged with 0.001 N PolyDADMAC and the excess cationic charge in the filtrate was titrated back to a zero output of the CAS device mentioned above, using 0.001 N PES-Na standard solutions. To determine the separate contributions of anionicity by colloidal substances (CSs) and dissolved substances (DSs), firstly, the

DIP raw suspension stood overnight, and then the supernatant was decanted as DCS water. Then, the DCS water was filtered through a 0.22-μm membrane disc filter, and the filtrate was regarded as DS water. The cationic demands of the DCS and DS water were measured using the CAS instrument mentioned above, and the differential of cationic demands between the DCS and DS was calculated as that contributed by the CSs.

Applying Fixing Agents to the Deinked Pulp

The three polyamines were diluted to 5 g/L solutions. Then, to each 100 g of DIP slurry, a predetermined aliquot of polyamine solution was dosed into the slurry, and quick stirring followed for 45 seconds. The mixtures were then filtered through a 200-mesh filter cloth, and the filtrates were collected for further FBRM, cationic demand, and turbidity analyses, but for turbidity measurement, the samples were diluted 10 folds so that they were within the measurable range.

Measurement of Filtrate Properties

For FBRM measurements, a S400 focused beam reflectance meter (Mettler-Toledo, USA) was used for the measurement. The FBRM probe was inserted into a 100-mL beaker containing 50 mL filtrate. The scanning speed was set at 2 m/s, and the scanning interval was 2 seconds. Stable data were collected for 2 min while the filtrate was continuously agitated at 250 rpm, by a computer connected with the FBRM and installed with a copy of iC-FBRMTM 4.2 software.

The cationic demands of the filtrates were measured with colloidal titration method using the CAS instrument mentioned above, and the filtrate turbidity values were detected by LP 2000-11 turbidimeter made by Hanna Inc., Italy.

RESULTS AND DISCUSSION

Determining the Dosages of Fixing Agent to Pulp

During the syntheses, different amounts of cross-linking agent were used to adjust the molecular weights and branching degree of the fixing agents. As a result, PA-Lw was a linear product, while PA-Mw and PA-Hw were branched ones. The properties of the fixing agents are shown in Table 1, where the molecular weights of the polyamines, indirectly expressed as intrinsic viscosities, were in the order of PA-Lw < PA-Mw < PA-Hw (We deliberately denoted L as “Low”, M as “middle” and H as “High”). For charge density, PA-Lw was a little lower than the other two, while PA-Mw and PA-Hw were statistically same.

Table 1. Main Properties of the Fixing Agents

Polyamines	Cross-linking agent* (%)	Solids content (%)	Intrinsic viscosity (mL/g)	Charge density (meq/g)
PA-Lw	0	52.76	4.52	5.93±0.05
PA-Mw	2.1	52.63	13.00	6.26±0.04
PA-Hw	3.8	52.50	49.00	6.21±0.02
*The molar percentage of the cross-linking agent to that of the total monomers (including the cross-linking agent itself) used in the synthesis.				

Then the properties of the deinked pulp were determined. The results are shown in Table 2. It was found that the pulp consistency was 3.8%, and for this pulp slurry, its charge demand was 2.170 meq/L. The DCS water listed in the table consisted of supernatant solution obtained after overnight sedimentation of the pulp slurry. Its charge demand was 0.498 meq/L. Therefore, only 23% of the total anionicity was contributed by the DCS, and the remaining 77% was contributed by other solids such as fibers and fines. Additionally, the cationic demand of DS water was determined to be 0.374 meq/L. Therefore, it can be deducted that for the DCS water, 75% of the anionicity was contributed by the DS components, while only 25% was contributed by the CS components.

Table 2. Main Properties of the Deinked Pulp

Consistency (%)	Cationic demand (meq/L)			
	Whole pulp suspension	DCS	DS	CS
3.8	2.17	0.498	0.374	0.124

With the above information and the charge densities of the fixing agents in Table 1, theoretical demands of the polyamines to neutralize the total anionicity of the filtrate or the whole pulp suspension can be calculated (for convenience, they are named as first and second neutralization level respectively). The results are shown in Table 3, where it can be seen that for PA-Lw, the first and second neutralization level were 0.21% d/d and 0.93% d/d respectively. For both PA-Mw and PA-Hw, the values were 0.20% d/d and 0.88% d/d respectively.

Table 3. Calculated Dosages of Polyamines for Fully Neutralization of the Pulp Filtrate (First neutralization level) and the Whole Pulp Suspension (Second Neutralization Level)

	PA-Lw	PA-Mw	PA-Hw
First Neutralization Level * (% d/d)	0.21	0.20	0.20
Second Neutralization Level (% d/d)	0.93	0.88	0.88

*Here it is supposed that all of the charge of a fixing agent is consumed on neutralizing the negative charges of the pulp filtrate but not those of the fiber.

With the above data, the dosage levels of fixing agents were determined arbitrarily to be 0.13%, 0.39%, and 0.65%, all based on dry chemical to dry pulp (hereafter expressed as % d/d). Among them, 0.13% d/d was lower than the first neutralization level in Table 3; 0.39% d/d and 0.65% d/d were higher than the first but lower than the second neutralization level.

Effect of Polyamine Dosages on CS Removal and CS Agglomeration Detected by FBRM

As described in the experimental section, the pulp was treated with a polyamine fixing agent and then the water phase was separated with a 200-mesh wire. The numbers and size distributions of the particles in the filtrate were measured by FBRM. The methodology of FBRM is based on a highly focused laser beam scanning across particles in a suspension at a fixed speed. Light is backscattered into the probe whenever the focused laser beam crosses a particle or particle aggregate. The duration time of this backscattered light pulse is measured, and the chord length (CL) is calculated as the multiple of duration time and the velocity of the focused laser beam. Each measured chord length is considered a count. Each second, thousands of chords are measured. As a result, information about size distribution and the numbers (counts) of the particles in the suspension are obtained (Blanco *et al.* 2002a,b; Lumpe *et al.* 2001; Negro *et al.* 2006).

The changes of particle counts according to different fixing agent treatments at three different amounts are presented in Figs. 1 and 2. The difference is that in Fig. 1, the Y-axis, *i.e.*, the counts of particles are not weighted, but in Fig. 2 the Y-axis is square-weighted.

For better understanding on the square-weighting calculation, Eq. 1 is given as follows,

$$\mathbf{Sqr_i} = \frac{\mathbf{CL_i^2}}{\frac{\sum_{j=1}^{N} \mathbf{CL_j^2}}{\mathbf{N}}} \times \mathbf{n_i} \tag{1}$$

where $\mathrm{Sqr_i}$ is the square-weighted count of particle *i*; $\mathrm{CL_i}$ is un-weighted chord length of particles *i*; $\mathrm{CL_j}$ is the un-weighted chord length of particles *j*; and N is the number of channel groups or chord length groups that the FBRM takes for statistics, it is set as 100 in the software of the instrument; and $\mathrm{n_i}$ is unweighted counts of particles *i*.

As can be understood from the above equation, by giving more weight to larger particles, the square-weighting method can be very useful in detecting the contribution of CS agglomerates more clearly.

In Fig. 1 it can be seen that for all filtrate samples, including the blank one (the filtrate from the original pulp without treatment by any fixing agent), most of the particles had chord lengths smaller than 10 μm; while fewer between 10 and 50 μm, and almost none above 50 μm.

At the dosage of 0.13% d/d, it was seen that all the three polyamines reduced the particle numbers sharply, compared to the blank curve. Interestingly, at this dosage, the shapes of the curves of the polyamine treated samples were basically the same as that of the blank, indicating that the CS particles remaining in the filtrate were mostly not agglomerated. This also means that the CS particles retained onto pulp were not agglomerated; therefore, the CS removal mechanism was mainly “colloidal fixation”.

Then, with the dosage being increased, different phenomena can be seen for the different polyamines. For PA-Lw in Fig. 1a, increasing the dosage to 0.39% d/d further reduced the total particle counts to some extent, but at the higher dosage of 0.65% d/d, an unexpected curve reversion was seen, in that the particle counts at this dosage were at the same level as that in the blank sample. The phenomenon indicates that the CS was re-dispersed into the pulp water phase with this treatment. For PA-Mw in Fig. 1b, at the dosage of 0.39% d/d, the curve became lower in height but wider in width, and its center was shifted to the right side, which clearly indicates that CS agglomeration had happened; however, when the dosage was increased further to 0.65% d/d, although “curve-shifting” was still seen, “particle re-distribution” was also seen, since the curve became higher than that by 0.39% d/d. For PA-Hw in Fig. 1c, only curve-shifting could be seen, indicating the main mechanism was colloidal agglomeration.

The square-weighting method aims to observe more clearly the contribution of big particles, including particles agglomerates. The results are shown in Fig. 2. Compared to Fig. 1a where no CS agglomeration was observed, slight CS agglomeration can be seen in Fig. 2a. Then for Fig. 2b and Fig. 2c, more obvious curve-shifting was seen. In more detail, at the dosage of 0.39% d/d, PA-Mw resulted in more particles with chord length larger than 20 μm and PA-Hw resulted in more particles with chord length larger than 30 μm.

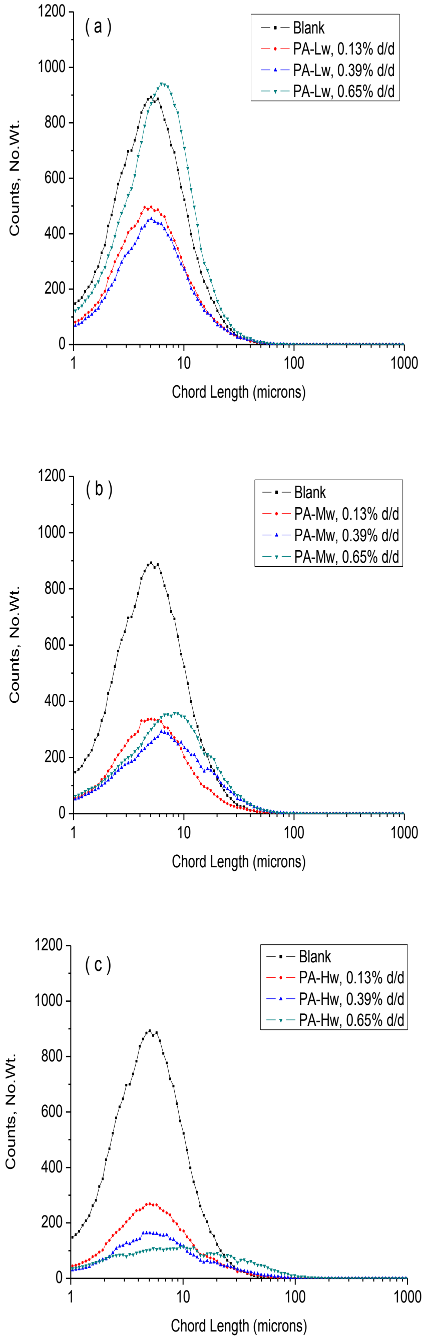

Fig. 1. Change in size distribution of the particles in the filtrates separated from pulps treated with three polyamine fixing agents at different dosages (Note: Values of particle counts were not weighted.)

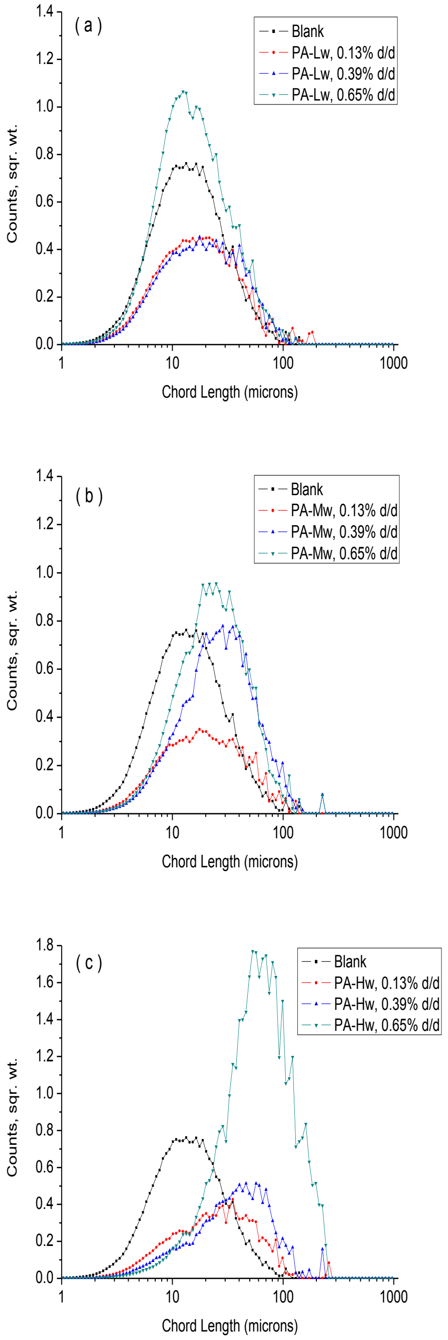

Fig. 2. Change in size distribution of the particles in the filtrates separated from pulps treated with three fixing agents at different dosages (Note: Values of particle counts were square-weighted.)

The particle size increment indicates the agglomeration occurrence at the treatment of 0.39% d/d and 0.65% d/d; however, the agglomerates induced by PA-Hw were much larger than those induced by PA-Mw.

In general, it can also be concluded that the three polyamines were all effective at reducing the CS contents, but since there were fewer particles in the filtrate separated from the pulp treated with the higher molecular-weighted polyamines over the whole dosage range, it can be concluded that polyamine with higher molecular weight had better efficacy in reducing the CS numbers.

Effect of Polyamine Dosages on CS Removal and CS Agglomeration Detected by Turbidity and Charge Demand Measurements

Traditionally, charge demand, turbidity, and chemical oxygen demand (COD) measurements have been used frequently for assessing DCS removal efficiency. From previous works, it is known that COD reduction by fixing agent treatment is generally low (Ravnjak *et al.* 2003; Lee *et al.* 2005; Wang *et al.* 2006), so only charge demand and turbidity measurements were used in this study.

From the above discussion, it is known that the phenomenon of CS re-dispersion happened when PA-Lw and PA-Mw were used at the high dosage of 0.65% d/d. It has been reported that re-dispersion of colloidal particles occurs when the charge of the particles are reversed. At that time, the colloidal system then becomes stable again, and its turbidity increases (Rojas and Hubbe 2005). The result of filtrate turbidity according to different fixing agent treatment is shown in Fig. 3, where the reversion phenomenon is also apparent at the dosage of 0.65% d/d for PA-Lw; however, it is not clearly visible for PA-Mw at 0.65% d/d, where the turbidity levels off compared to the value at 0.39% d/d. It has been observed that FBRM can supply much more information than turbidity measurements can. Turbidity results can also be misleading. For example, it is seen that for PA-Lw, the turbidity at 0.65% d/d was still lower than that of 0.13% d/d; therefore one may conclude that the effect of 0.65% d/d is better than 0.13% d/d. However, FBRM shows different information.

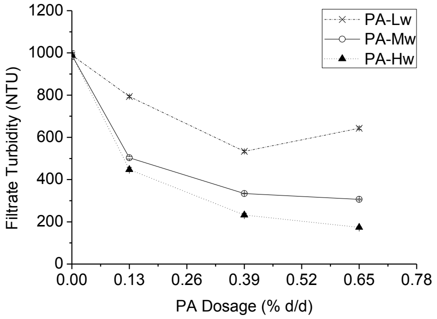

Fig. 3. Turbidity of the filtrates separated from pulps treated with different dosages of three polyamines

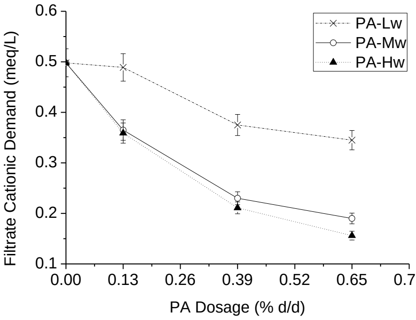

Fig. 4. Charge demands of filtrates separated from pulps treated with different dosages of three polyamines

The results of cationic demand analysis for the filtrate are shown in Fig. 4. It is apparent that the cationic demands of the filtrates decreased with the increased dosage of polyamines, and increasing the molecular weight increased the effect of cationic demand reduction. Polyamine PA-Hw, with its molecular weight being the highest, had the greatest effect in reducing the cationic demand. But even at its highest dosage level of

0.65% d/d, only 68.67% of the charge was neutralized, which is far from charge reversal. From the above discussion, it can be seen that re-distribution of CS particles can occur even though the charge neutralization point has not been attained. The phenomenon is puzzling, and very little explanation can be found in literature except that mentioned in a review, which says that there are two situations in which a polyelectrolyte will reliably contribute to the stabilization of a suspension: First, if the molecular mass of the polyelectrolyte is too low for it to act as an effective bridging agent (*e.g.*, $<<10^6$ g/mole), then the dispersing effect are expected to dominate. Second, if a sufficient amount of hydrophilic polyelectrolyte has been added to cover essentially all of the surfaces, then a highly stabilized system can be achieved, regardless of the sign of charge or other attributes (Hubbe *et al.* 2012). Here, a model was temporarily proposed by us as follows:

Point 1: When a large amount of fixing agent (high dosage) is dosed into the system, a part of fixing agent will be adsorbed onto fiber and the others will interact with the DCS. Due to the fact that for the DCS components in our pulp slurry system, around 80% of the negative charge is contributed by DS, while only 20% of the negative charge is contributed by CS, it is very possible that DS will form complexes with the fixing agent quickly, meanwhile, the CS is very possible to be nearly fully neutralized by the fixing agent.

Point 2: The DS-fixing agent complex can cover the surface of the CS particles, no matter whether the latter is fully or partly neutralized.

Point 3: Due to the fact that the negative charge of DS is high, it is not fully neutralized by the fixing agent, thus the CS particles stabilized by DS-fixing agent complexes are well dispersed in the negative DS water.

Point 4: Most of the DS-fixing agent complexes are insoluble in water and have certain particle sizes (Kekkonen *et al.* 2001, 2002; Lofton *et al.* 2005); thus after the re-distribution, the new CS particle sizes are larger and more numerous, as can be seen typically from Fig. 2(a).

Point 5: For a fixing agent with lower molecular weight, such as PA-Lw, it reacts more quickly with DS components, *i.e.*, the formation of DS-fixing agent complex is quicker, thus it is more easily re-distributed over the CS particles. On the other hand, a fixing agent of higher molecular weight, such as PA-Hw, reacts more easily with CS particles, causing CS agglomeration.

The DS-fixing agent complexes covered colloidal particles are much like an egg white surrounding an egg yolk; therefore the mechanism can be called the “egg model”.

CONCLUSIONS

1. For a certain polyamine fixing agent, using it at different dosages will cause different functioning mechanisms with respect to its interaction with colloidal substances. No matter what the molecular weight is, generally when the dosage is low, the colloidal fixation mechanism, *i.e.*, colloidal substances being fixed onto fibers in their unagglomerated, single-particle state, overwhelms colloidal agglomeration, *i.e.*, colloidal particles being coagulated into bigger agglomerates.
2. For a fixing agent whose molecular weight is low enough, such as the PA-Lw demonstrated in this study, it performs in the colloidal fixation mode over a wide range of dosages, but when used at a high enough dosage, it can re-disperse colloidal

particles in the pulp system, even if the dosage is below the point at which the negativity of DCS has been fully neutralized. This phenomenon of “CS redispersion before charge neutralization” can be explained by the formation of DS-fixing agent complexes followed by covering CS particles and stabilizing them.

3. Polyamines of higher molecular weight have stronger effects in reducing the number of unattached CS particles; however, they tend to result in more severe colloidal agglomeration. Also increasing their dosage generally increases more colloidal agglomeration. For a fixing agent whose molecular weight is high enough, such as the PA-Hw that was considered in this study, there is less tendency to cause colloidal re-dispersion in comparison to a fixing agent with much lower molecular weight, such as PA-Lw mentioned above; this is possibly because the higher-mass cationic polymer interacts more easily with CS particles than with DS components to form complexes that can stabilize CS particles.
4. For a fixing agent of moderate molecular weight, such as the PA-Mw considered in this study, all the phenomena related to colloidal fixation, colloidal agglomeration, and CS redispersion can be seen, *i.e.*, its performance is between that of its lower and higher molecular-weighted products, suggesting that the proposed concepts are valid.
5. The study suggests that for better determining the dosage of a fixing agent, both CS removal ratio and CS agglomeration behaviour should be considered. Developing better CS control program may also be possible through the combined use of a low molecular-weighted and a high molecular-weighted fixing agent. The fixing agent of relatively low molecular weight may favour forming complexes with DS, and the high molecular-weighted fixing agent may favour fixing or retaining colloidal substances before they are covered by the complexes and re-stabilized.

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