

THE EFFECT OF CHARGE AND CHEMICAL STRUCTURE OF CATIONIC SURFACTANTS ON LASER TONER AGGLOMERATION UNDER ALKALINE PULPING CONDITIONS

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Laboratory-scale agglomeration experiments followed by image analysis were used to evaluate the effectiveness of different cationic surfactants on the 1-octadecanol agglomeration of a negatively charged laser toner. Various types of surfactants with different geometric structures were investigated. It was found that this toner became agglomerated under neutral pulping conditions, but it did not agglomerate under alkaline conditions at all. A small amount of the cationic surfactant compensated for the agglomeration disruption caused by the negative surface charge of the toner and made this toner agglomerate very well. These cationic surfactants consist of a chemical structure of C12 to C18 saturated alkyl hydrophobic chains. The positive charge of these surfactants played the major role in alleviating agglomeration disruption. Additionally, an extra phenol group on these surfactants contributed only minor advantages for toner agglomeration in the presence of 1-octadecanol. The best co-agglomeration performance occurred within a very narrow range of similar total positive charge densities based on the total toner weight. It was also found that this positive charge effect could not be applied to the chemical compounds of high molecular weight polymeric materials.

Keywords: Laser toner; 1-octadecanol; Cationic surfactants; Agglomeration; Surface charge

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INTRODUCTION

The importance of fiber recycling and usage of recycled fiber have become crucial concepts in the paper industry in many countries where there is a shortage of natural wood resources. For example, China used more than 60% of the country's recycled paper to generate new paper products in the year 2010 (China Paper Association 2011). Among these classes of recycled papers, "mixed office waste" plays an important role as recovered fiber for high quality printing and writing grade papers, because it has high brightness and a sufficiently large proportion of long fiber. However, one main problem with using mixed office waste is that it contains thermoplastic-based inks that are printed and fused on the paper. Most toner for this type of paper is difficult to handle with conventional deinking methods of washing, flotation, centrifugal cleaning, and screening (Wayne et al. 1991).

A more efficient approach is to agglomerate the toner into larger particles or more dense particles with suitable chemicals, heat treatment, and mechanical mixing. The

agglomeration of the particles can increase the efficiency of their removal in the fine screen and centrifugal cleaners, yielding a clean, high quality deinked pulp (Borchardt et al. 1997; Olson et al. 1993; Snyder et al. 1994).

1-octadecanol is known to be a very effective agglomerating agent that acts well under neutral to alkali conditions, relative to the performance of other agglomeration-inducing chemicals. It also helps in separating the toner from the surface of fibers, thus improving flotation efficiency (Chang et al. 1996; Chen et al. 1997). However, the presence of cationic starch has been found to prevent the agglomeration of some types of toner when only 1-octadecanol is used as the agglomerating agent (Zheng et al. 2001). The cationic starch also has a negative agglomerating effect for Apple laser toner by agglomerating oil, but the reason for this disruptive effect is unclear (Snyder et al. 1994). It was proposed that the cationic starch is absorbed onto the toner surface when the toner carries a negative charge, and a positively charged surfactant CTAC (cetyl-trimethyl ammonium chloride) could eliminate this problem in a model system. However, the proposed solution did not work in the case of commercial printing paper (Chen et al. 2004).

A co-agglomeration system was proposed by using non-ionic surfactants with a phenolic structure and an HLB (hydrophilic-lipophilic balance) value of about 10 or below together with 1-octadecanol to combat the negative effect of cationic starch during agglomeration of toner printed on commercial printing paper (Welf et al. 2001). However, that report used neutral pulping conditions, and the effect of alkaline pH was not discussed. Patents issued to Richmann and Letscher indicate that the use of surfactants having an HLB of 10 or less, sometimes in conjunction with an aliphatic hydrocarbon with a backbone of 8-12 carbon atoms, could effectively agglomerate the toner printed on waste papers (Richmann et al. 1992, 2001). These patents did not show the toner surface charge, and the charge effect was not discussed. However, they did report that under alkaline and medium consistency pulping conditions, the toner of mixed office waste will agglomerate and form ink particles with density greater than 1.0, which can be effectively removed by forward cleaners (Olson et al. 1993).

Most research has shown no disadvantage of toner ink agglomeration under alkaline pulping conditions, which are known to promote fiber swelling and fiber surface ink detachment. Previous laboratory research found that some xerographically copied toner ink carrying a negative charge could not be agglomerated by 1-octadecanol under weakly alkaline pulping conditions. The addition of a small amount of cationic surfactant would help the 1-octadecanol agglomeration of these toners in both alkaline and neutral conditions (Wang et al. 2011). More recent research found that the same alkaline disruption phenomenon occurred with some laser toner agglomeration. Addition of cationic surfactants would also eliminate the negative effect to agglomeration.

The objectives of the present research were to evaluate the effectiveness of charge characteristics and geometric structure for different cationic surfactants on the agglomeration of 1-octadecanol under alkaline pulping conditions. Additionally, the goal was to find the most suitable cationic surfactants that would make the 1-octadecanol agglomeration perform normally in this cationic co-agglomeration system.

EXPERIMENTAL

Materials

The printed paper used was a commercial product called “GOLD BALL”, made by APP Co., China. The laser-printed paper was printed on the same paper by using HP-1010 LaserJet printer with the toner made by the same company. This toner carried a slightly negative charge. All other chemicals were purchased locally. Detailed information on these materials is listed in Table 1.

Table 1. List of Materials

Materials	Names (or grades)	Source
Paper	APP copy paper, 70 g/m ² , AKD sized, dual purpose for xerographic, laser, bubble jet and offset printing	APP Co., China
Toner	Cartridge Q2612A for HP-1010 LaserJet printer	Hewlett-Packard Company, CA, USA
1-octadecanol	Pure chemical	Shanghai Jiu Yi Chemical Reagent Co.
NaOH	Sodium hydroxide, purity > 96%	Nanjing Chemical Reagent Co.
CTAB	Cetyl trimethyl ammonium bromide, cationic surfactant, pure chemical; Molecular weight: 364.46	Shanghai Ling Feng Chemical Reagent Co.
SDBAC	Stearyl dimethyl benzyl ammonium chloride, cationic surfactant, pure chemical; Molecular weight: 424.15	Shanghai Jin wei Chemical Co., Ltd.
LDBAC	Lauryl dimethyl benzyl ammonium chloride, cationic surfactant, pure chemical; Molecular weight: 339.99	Shanghai Jin Wei Chemical Co., Ltd.
DMDAAC	Diallyldimethyl ammonium chloride, commercial grade; Monomer; Molecular weight: 161.67; Charge density: 6.19 mmol/g	Kemira Yixing Co., Ltd
P-DMDAAC	Poly- Diallyldimethyl ammonium chloride, commercial grade; Polymer; DP: 5000; Charge density: 7.52 mmol/g	Kemira Yixing Co., Ltd
CPAM	Cationic Polyacrylamide, commercial grade; Molecular weight: 5 to 6 million; Charge density: 1.56 mmol/g	Ciba,Co.,Switzerland
HCA	Poly-diallyldimethyl ammonium chloride, cationic standard titrant	BTG Mütek GmbH
PVSK	Potassium sulfate ester of polyvinyl alcohol, anionic standard titrant	BTG Mütek GmbH

Pulping

Laser-printed paper was torn to 1 cm x 1 cm pieces before pulping. A homemade 1.0 L stainless steel pulper with a screw type rotor driven by a variable speed motor was used for pulping and agglomeration. Before pulping, 465 mL of distilled water was added to the pulper and heated to 70 °C by partially submerging the pulper in a water bath maintained at a little bit higher than 70 °C. 1-octadecanol (0.6 g, 2% based on OD paper), different amounts of surfactants, with or without NaOH, were added and mixed at 300

rpm for three minutes to ensure that the 1-octadecanol was molten. To the pulper, 30 OD grams of the printed paper was added and disintegrated at 813 rpm for 15 minutes. After 15 minutes, the rotor speed was reduced to 443 rpm for 45 minutes for toner agglomeration. After pulping, the pulp slurry was transferred to a plastic bag and cooled down in tap water. Six handsheets were made according to TAPPI Standard Method T205 OM-8. The handsheets were air dried for 24 hours and evaluated by image analysis system with a Canon LiDE100 Scanner. The software used was Autospec V4.0 Image Analysis System (State Key Laboratory of Pulp and Paper Engineering; South China University of Technology).

Screening

After pulping, the pulp slurry was transferred to a plastic bag and cooled down in tap water. The pulp slurry was then transferred to a 0.15 mm slot screen (Somerville Shive-Content Analysator, Model no. 40170, PTI, Austria) for fiber and ink particle separation. The screening took approximately 7 minutes to finish when there was no fiber on top of the screen plate. The screening accepts were collected by a two layer cloth bag with very tiny holes to make sure there was no loss of any fine ink particle. Handsheet preparation and residual ink evaluation followed the same procedures as that used in the pulping process.

Surface Charge of the Toner

The toner was printed directly onto poly-acetate plastic overhead projector film by the laser printer. The printed toner was then scraped and collected from the surface of this plastic film by a stainless steel perpendicular. The collected toner was then screened to sizes between 50 and 100 meshes. The toner (0.1 g) was added to a 150 mL glass beaker with 40 mL of distilled water, and the pH value was adjusted to 12 with 0.0082 mol/L of NaOH. The beaker was then put onto an electric heater with automatic temperature control and mixed by a speed controlled Teflon rotor for 60 minutes at 70°C. Cationic polyelectrolyte (0.001 mol/L HCA, 5 mL) was added after the reactants had been cooled down to room temperature, and they were allowed to further react for 45 minutes in order to the charge of the toner which was neutralized completely by the cationic titration reagent. After the reaction, the filtrate was separated from the slurry using a 200 mesh ceramic filter. The filtrate was then back titrated using an anionic polyelectrolyte titrant (PVSK) to determine the surface charge of the toner. The end point was determined by a particle charge detector (PCD-03 Mütek, BTG).

RESULTS AND DISCUSSION

1-Octadecanol Agglomeration in Different Pulping Conditions

Agglomeration performance of 1-octadecanol under both neutral and alkaline conditions was evaluated by pulping the laser printed papers. The results are shown in Table 2.

Table 2. Performance of 1-octadecanol Agglomeration

NaOH (%)	pH (After pulping)	1-octadecanol (%)	NPM (number/m ²)	PPM (mm ² /m ²)	Average particle size (mm ²)
0	7.9	0	373,000	20,500	0.055
		2.0	27,600	5,000	0.180
0.5	10.9	0	503,000	26,000	0.052
		2.0	224,400	9,000	0.040

It is clear from Table 2 that the number of ink particles per square meter (NPM) was reduced by more than 90% (from 373,000 to 27,600) and the average ink particle size increased by three times, from 0.055 mm² to 0.180 mm², after agglomeration with 1-octadecanol without adding any NaOH. These data indicate that this toner ink agglomerated with 1-octadecanol under neutral pulping conditions, and the results are consistent with earlier studies (Chang et al. 1996; Chen et al. 1997; Wang et al. 2011). When alkali was added, the toner ink performance was totally different. The NPM value increased from 373,000 to 503,000 without adding any 1-octadecanol, which meant that the toner ink was further dispersed by alkali. When 2% of 1-octadecanol was added, the reduction of NPM value was only 55% (from 503,000 to 224,400), and the average particle size was even reduced from 0.052 mm² to 0.040 mm². These data demonstrate that this toner was still highly dispersed and could not be agglomerated by 1-octadecanol under alkaline conditions, which was consistent with earlier findings on xerographic toners (Wang et al. 2011).

Effect of Alkali

Although it was reported that 1-octadecanol agglomeration was not affected by a pH change during the pulping stage (Chang et al. 1996; Chen et al. 1997), it has been found that pH has a great influence on agglomeration for some negatively charged xerographic toners in recent research (Wang et al. 2011). In this study, it was found that the laser toner that carried a negative charge demonstrated a similar phenomenon, and the results are shown in Figs. 1 and 2.

As can be seen in Fig. 1, the NPM after pulping dramatically increased from 27,000 to 220,000, along with the increment of sodium hydroxide for this toner. The result of average ink particle size after pulping is shown in Fig. 2. The average ink particle size of the toner was reduced from 0.18 mm² to 0.04 mm² when the alkali dosage exceeded 0.5%. From these data, it can be concluded that this toner, rather than agglomerating, was strongly affected by alkali and did not agglomerate at all. Furthermore, visual observation clearly showed that not only the particle size but also the particle shape was different before and after the addition of alkali. Before alkali addition, the agglomerated ink particles were spherical, whereas after alkali addition the toner ink particles were flat. As reported, these differences in size and shape can greatly affect the screen efficiency (Borchardt et al. 1997).

In order to further clarify the agglomeration effect, all pulped slurry was passed through a slot screen with a 0.15 mm slot. The results before and after screening are shown in Figs. 1 and 2. As can be seen from Fig. 1, the NPM was reduced from 27,000 to 6,600 after screening, which is about a 75% reduction from the neutral condition. When 0.25% NaOH was added, the ink removal rate was reduced to 55%. When alkaline

dosage exceeded 0.5%, only a very small amount of the reacted toner was screened out, and the screening efficiency was very poor.

These effects were also reflected as a change in average ink particle size. In Fig. 2, it is clear that the toners with an average particle size greater than 0.04 mm^2 can be screened out. Since the neutral pulping condition agglomerated well and generated much bigger toner particles, the screening efficiency was high. The efficiency became lower with the addition of alkali. After reaching 0.5% sodium hydroxide addition, the toner was highly dispersed, and almost nothing was screened out.

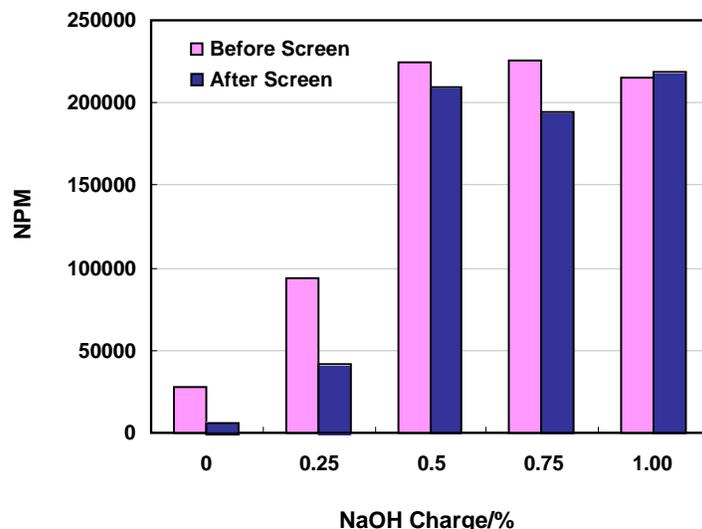


Fig. 1. The effect of sodium hydroxide on 2.0% 1-octadecanol agglomeration

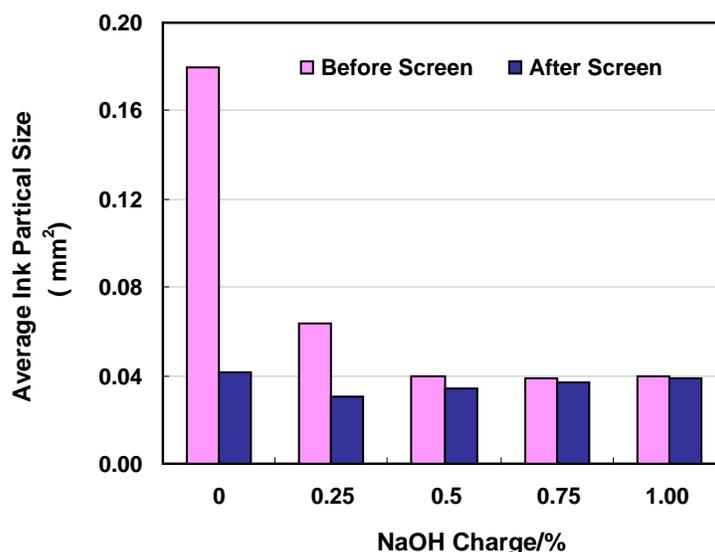


Fig. 2. The effect of sodium hydroxide on 2.0% 1-octadecanol agglomeration

Surface Charge of Toners

It was reported that positively charged polymeric paper additives might affect toner agglomeration if that toner carried a negative charge (Chen et al. 2004). It has also been found that some anionic groups are generated on the surface of xerographic toner

under alkaline pulping conditions, which would affect the 1-octadecanol agglomeration (Wang et al. 2011). In this study, the surface charge of the laser toner displayed the same behavior, which was also influenced by alkali. The results are shown in Table 3.

As shown in Table 3, the toner exhibited a slightly negative charge of -0.0022 ± 0.0002 mEq/g at room temperature without any treatment. When the toner was treated by 70 °C water with a pH of 7, the surface charge of the toner changed slightly from -0.0022 ± 0.0002 mEq/g to -0.0014 ± 0.0002 mEq/g. The negative surface charge of the toner became weaker compared to that at room temperature. It was suspected that a very tiny proportion of the negative surface sites was neutralized by the dissociated proton from the hot water during the 70 °C reaction, and as a result there was less cationic polyelectrolyte titrant (HCA) consumed when the slurry was cooled down to room temperature. Nevertheless, the change in the negative value was very small. When the toner was treated by 70 °C hot alkali water with a pH of 12, the surface charge of toner was dramatically changed from -0.0014 ± 0.001 mEq/g to -0.0068 ± 0.0015 mEq/g, which is a much stronger negative charge. These results were consistent with previous findings and showed that the surface charge of the toner was changed by alkali. More negative charge was generated on the surface of the toner and led to the negative effect on agglomeration of this toner (Wang et al. 2011).

Table 3. Toner Surface Charge at Different Reaction Conditions

Experimental Conditions	pH=7; Temp=25 °C	pH=7; Temp=70 °C	pH=12; Temp=70 °C
Toner Charge mEq/g	-0.0022 ± 0.0002	-0.0014 ± 0.0002	-0.0068 ± 0.0015

Note: The HCA was added after slurry was cooled down to room temperature.

Structure Effect of Cationic Surfactants

Three different cationic surfactants, SDBAC, CTAB, and LDBAC, were added as the positive charge donator in addition to 2% 1-octadecanol and 0.5% NaOH. Different amounts of each surfactant were added to evaluate their effect on agglomeration, and the results are shown in Figs. 3 through 6. In Figs. 3 and 4, the weight percent concentration on OD paper for each surfactant was used as the abscissa. In Figs. 5 and 6, the abscissa is the molar concentration.

As shown in Fig. 3, these cationic surfactants showed similar trends in 1-octadecanol agglomeration. As their concentration increased, the agglomeration effectiveness increased and then reached an optimal concentration value. When each surfactant's concentration exceeded its optimal dosing value, the effectiveness decreased or leveled off. Based on these results, even a small addition of these surfactants will dramatically help the 1-octadecanol agglomeration and reduce the NPM value. When 0.02% (on OD paper) LDBAC was used, the NPM was lowered from 200,000 to 68,000, which was a 66% reduction. The optimal dosage of LDBAC occurred in the range between 0.035% and 0.04%, where the NPM value reached its minimum of 10,000, which was a 95% reduction. Above this dosage (0.04%), the NPM appeared to level off at around 20,000. The results of CTAB and SDBAC were almost identical to that of the LDBAC, except that the optimal dosage values were different. The optimal dosage of CTAB and SDBAC appeared at 0.05%. At this point, the NPM was approximately

13,000 for CTAB (a 94% reduction) and approximately 17,000 for SDBAC (a 93% reduction). For all of these surfactants, there existed an optimal value of usage, but a dosage higher than this value will hurt the agglomeration efficiency. This is more clearly illustrated in Fig. 4.

As shown in Fig. 4, the agglomerated ink particle size of LDBAC reached its highest value of 0.22 mm^2 at the 0.035% addition of LDBAC, which was consistent with its optimal NPM value. The optimal addition values of CTAB and SDBAC occurred at 0.05%, which was also consistent with their NPM values. However, all their average ink particle sizes were reduced when more surfactants were used. When 0.06% or more surfactant was added, the agglomeration became worse and the ink particle size became similar to the particles with zero surfactant addition. This means that too much surfactant will disperse the agglomerated ink particle again.

An interesting finding from these two figures was that the optimal dosage of CTAB and SDBAC existed at the same level. These two compounds consisted of very similar long chain alkyl groups, C16 for CTAB and C18 for SDBAC, except that the SDBAC had an extra phenol group on it. It has been reported that the phenol group played an important role in the co-agglomeration system of 1-octodecanol for some types of phenolic non-ionic surfactants, which would help 1-octodecanol agglomeration on some negatively charged toner ink (Welf et al. 2001). From these two figures, it seemed that an extra phenol group showed no obvious benefit in terms of toner ink agglomeration between CTAB and SDBAC. In fact, the CTAB seemed to agglomerate better when the dosage was 0.05%, since the optimal average ink particle size (0.3 mm^2) is bigger than SDBAC (0.2 mm^2). The agglomeration efficiency of LDBAC was different. This compound has a similar chemical structure to SDBAC, except that it consists of a C12 alkyl group and also a phenol group. However, the molecular weight is lower than SDBAC. When this surfactant dosage was 0.035%, the NPM value was only 10,000, which is the lowest one among the three surfactants, and the ink particle size was 0.22 mm^2 , which is also better than SDBAC.

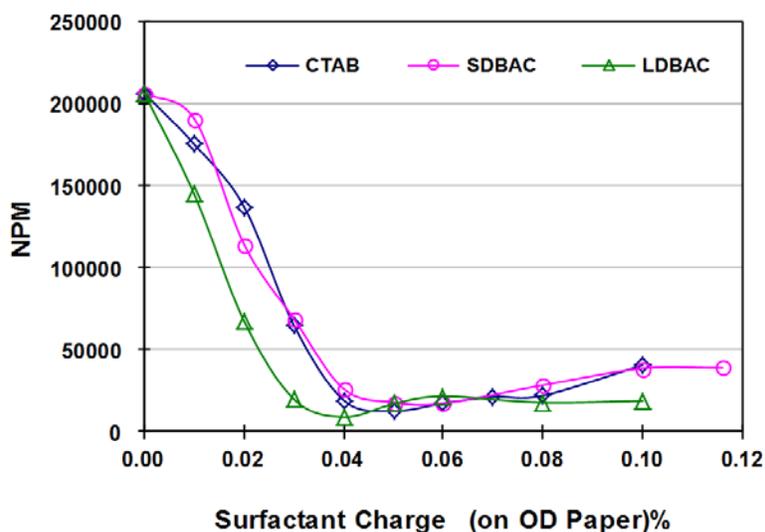


Fig. 3. NPM versus cationic surfactant concentration with 2.0% 1-octadecanol and 0.5% NaOH

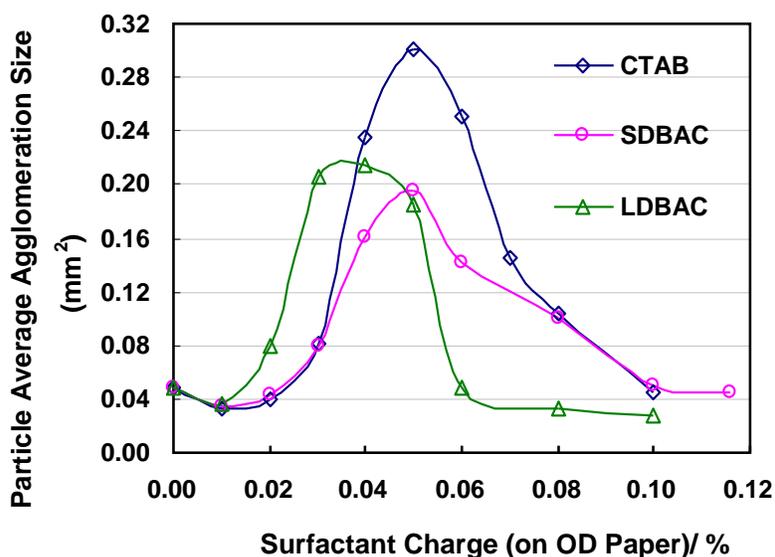


Fig. 4. Average agglomeration size versus cationic surfactant concentration with 2.0% 1-octadecanol and 0.5% NaOH

Since the 0.035% dosage of LDBAC is much less than that of CTAB and SDBAC, the LDBAC seems to be the most efficient chemical based on the chemical usage. Thus, it is difficult to analyze from Figs. 3 and 4, and to conclude which surfactant structure is the best. Since the charge plays an important role in this agglomeration system, it might be helpful to plot the experiment results versus molarities.

Charge Effect of Cationic Surfactants

Figures 3 and 4 show the agglomeration results of each surfactant based on their weight percent concentration. However, the weight percent concentration is not equal to the equivalent molar concentration, and the cationic effect of surfactants may not be reflected thoroughly. In order to analyze the cationic effect of each surfactant, Figs. 3 and 4 were re-plotted based on their equivalent molar concentration, and the results are shown in Figs. 5 and 6.

Comparing Figs. 5 and 6 to Figs. 3 and 4, three major changes are apparent. First, all three lines and optimal values became closer to each other. In Figs. 3 and 4, the optimal values are located at 0.035% for LDBAC and at 0.05% for SDBAC and CTAB. The difference between 0.035% and 0.05% is 43%. In Figs. 5 and 6, the optimal value was 0.035 mmole for LDBAC and SDBAC and 0.04 mmole for CTAB. The difference is only 14%. Since all these cationic surfactants carry one proton, the molar concentration could directly reflect the positive charge density on the agglomeration system. A narrow optimal dosage range for these surfactants also means that the charge effect is more important than the structure effect. Second, the location of optimal value for SDBAC was changed dramatically. It has a similar location of 0.05% to the CTAB in Fig. 4, but it shifted dramatically to 0.035 mmole in Fig. 6, which is almost identical to the LDBAC. Both SDBAC and LDBAC have the same chemical structure, which carries a phenol and a long-chain alkyl group. The only difference is that the SDBAC has a C18 alkyl group

and the LDBAC has a C12 alkyl group. In these two figures (5 and 6), the effect of these two surfactants was plotted based on their charge density. It can thus be concluded that the hydrophobic chain length has no effect on both chemicals' agglomeration, and charge is the most important factor. Third, the optimal dosage of LDBAC and SDBAC is slightly smaller than CTAB. Since both LDBAC and SDBAC carry a phenolic group, plus the SDBAC and CTAB have similar alkyl chain length, it can be concluded that the phenolic group is actually helping the 1-octadecanol agglomeration. However, the contribution is much smaller than that of the charge effect.

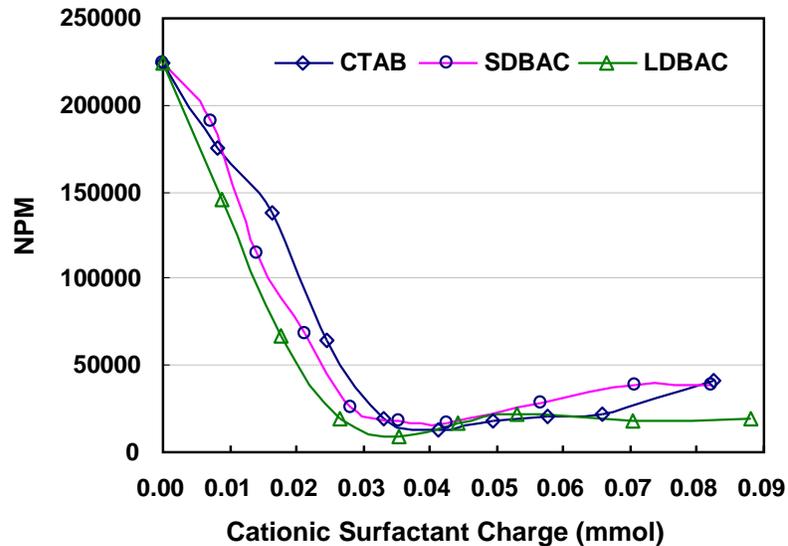


Fig. 5. NPM versus cationic surfactant charge with 2.0% 1-octadecanol and 0.5% NaOH

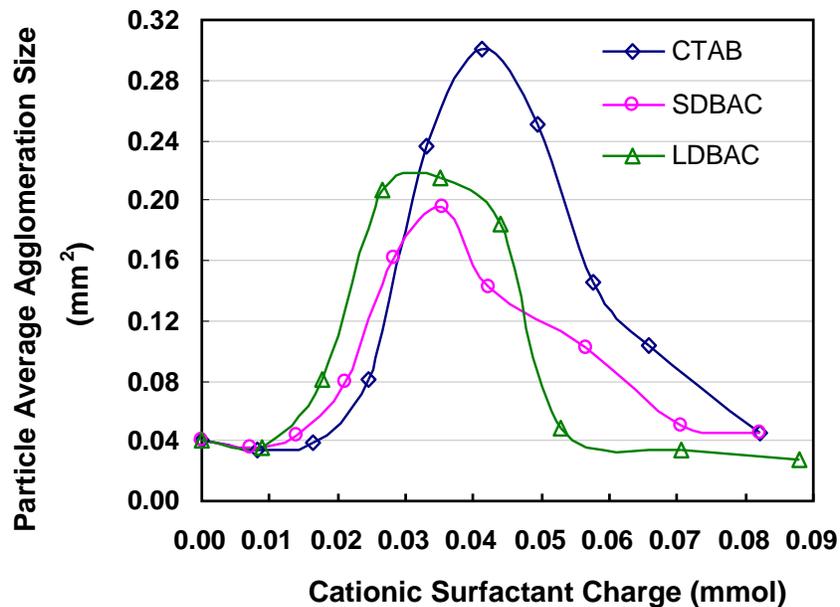


Fig. 6. Average agglomeration size versus cationic surfactant charge with 2.0% 1-octadecanol and 0.5% NaOH

Effect of Cationic Polymer

It has been reported that polyamine and poly-DMDMAC are adsorbed onto the toner surface, where they can inhibit the agglomeration of the negatively charged toner. However, the polyacrylamide did not interfere with negative charge toner agglomeration due to its high molecular weight (Chen et al. 2004). In order to investigate the cationic effect of different polymeric materials on the toner agglomeration under alkaline conditions, DMDAAC, poly-DMDAAC (P-DMDAAC), and cationic polyacrylamide (CPAM) products were added into the pulp with 2.0% 1-octadecanol and 0.5% NaOH, respectively. The results are shown in Figs. 7 and 8.

As shown in Figs. 7 and 8, all three cationic polymers could decrease the NPM from 220,000 to about 150,000 when the dosage was less than 0.03%.

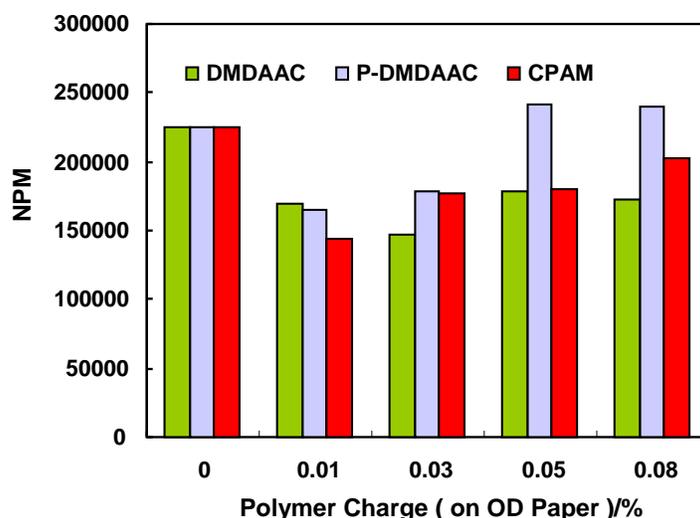


Fig. 7. NPM versus cationic polymer with 2.0% 1-octadecanol and 0.5% NaOH

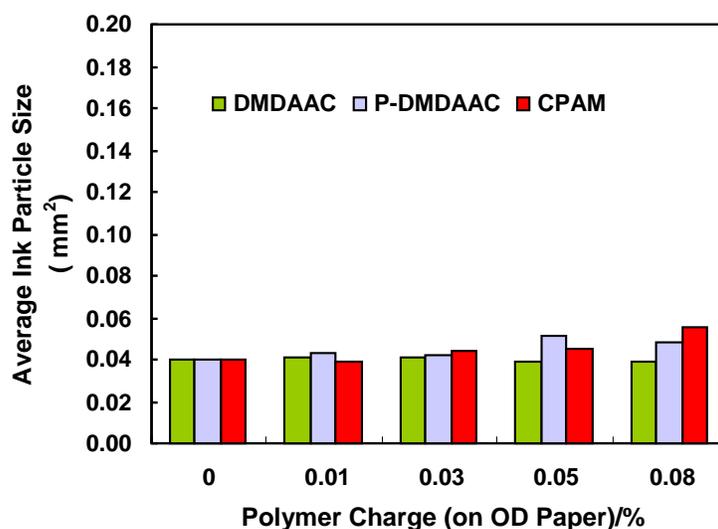


Fig. 8. Average agglomeration size versus cationic polymer concentration with 2.0% 1-octadecanol and 0.5% NaOH

However, the average in particle size was unchanged (0.04 mm^2), compared to that without adding any cationic polymer. This might be due to the polymer, which provided cationic charge and caused some toner to agglomerate. The chemical structure of polymers was not suitable for the particle size increasing as the co-agglomeration reagent. When the dosage exceeded 0.03%, the reduction in NPM value became smaller or got closer to the value that had been obtained without adding any polymeric materials. Thus, the cationic polymers were not useful as a co-agglomeration reagent. This might be caused by adsorption of the cationic polymer onto the negatively charged toner surface, following the mechanism proposed by Chen et al. (2004), thus inhibiting the toner agglomeration.

CONCLUSIONS

1. The agglomeration performance of laser toner carrying a negative charge was negatively affected by alkaline conditions. Addition of a small amount of suitable cationic surfactant greatly improved the agglomeration efficiency of 1-octadecanol under alkaline conditions.
2. Equally charged cationic surfactants with similar chemical structures had the same agglomeration efficiency. It follows that the charge effect and charge density are the most important factors in the cationic co-agglomeration system.
3. The addition of a phenol group in the hydrophobic part of a cationic surfactant makes a positive contribution to the agglomeration efficiency. However, its contribution to effectiveness is much less than that of the charge.
4. A highly charged cationic polymer did not contribute effectively to toner agglomeration.

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