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Use of surfactants to remove water-based inks from plastic film: effect of calcium ion concentration and length of surfactant hydrophobe

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Abstract Effective plastic film deinking could permit the reuse of recycled polymer to produce clear film, reduce solid waste for landfills, reduce raw material demand for polymer production, and aid process economics. In this study, the deinking of a commercial polyethylene film printed with water-based ink was studied using surfactants in the presence of hardness ions (calcium ions) at various pH levels. The electrostatic properties of ink particles in a washing bath were also investigated. Synthetic anionic surfactant or fatty acid soap in the presence of calcium ions at alkaline pH levels was found to be nearly as effective at deinking as cationic, nonionic, or amphoteric surfactants alone. However, adding calcium ions decreases the deinking effectiveness of cationic, nonionic, and amphoteric surfactants. Increasing the length of the ionic surfactant hydrophobe enhances deinking. Zeta potential measurements showed that water-based ink particles in water reach the point of zero charge (PZC) at a pH

of about 3.6, above which ink particles are negatively charged, so cationic surfactant tends to adsorb better on the ink than anionic surfactant above the PZC in the absence of calcium. As the cationic surfactant concentration is varied between 0.005 and 25 mM, the zeta potential of the ink particles reverses from negative to positive owing to adsorption of cationic surfactant. For anionic surfactants, added calcium probably forms a bridge between the negatively charged ink and the negatively charged surfactant head groups, which synergizes adsorption of the surfactant and aids deinking. In contrast, calcium competes for adsorption sites with cationic and nonionic surfactants, which inhibits deinking. All the surfactants studied here disperse ink particles effectively in the washing bath above pH 3 except for the ethoxylated amine surfactant.

Keywords Deinking · Plastic film · Polyethylene · Surfactants · Water-based ink

Introduction

Polymer usage has rapidly increased for many applications because of the light weight, strength, durability, moldability, colorability, and relatively low energy requirements in converting resin to manufactured products. The fraction of plastic in solid waste has also

increased. Plastic with about 9% by weight and 20% by volume is the second biggest contributor to municipal solid waste in the USA [1, 2]. The fraction of plastic film in total plastic waste in the USA is 21–57% [3].

Even though the potential market for recycled polymer is theoretically large, only about 3.5% of all plastic and 5% of plastic films are now recycled because of

limitations in the technology of sorting, cleaning, and reformulating the recycle [1, 4]. In-plant recycling, which is estimated to be 4 billion pounds per year, is more successful than postconsumer recycling since the source is better defined. Technological developments in processing the former may help in developing techniques for the latter [4]. Most recycled plastic films are heavily printed in order to describe the product contained within. Polymers produced from the recycle are not as valuable as virgin polymers because residual ink can color repelletized resins and change the physical properties, and gases associated with decomposition can release and crack pellets [5, 6].

Effective deinking technology could permit reuse of the polymer as a suitable raw material for clear plastic film production. Solvent-based deinking techniques such as the NOREC process have been developed and used in Europe [7]; however, this process is expensive, complicated, and produces hazardous waste. For this study, we investigated the use of aqueous surfactant solutions for plastic film deinking since surfactants are more environmentally compatible [5, 6, 8].

Low-density polyethylene (LDPE) and high-density polyethylene (HDPE), polyethylene terephthalate, ethylene vinyl acetate, polyvinyl chloride, and polypropylene are used for film-making. Most plastic films are made of either LDPE, HDPE or a mixture of LDPE and HDPE [3].

Plastic films are printed with either water-based or solvent-based ink. Water-based ink occupies a large and increasing fraction of plastic film inks since environmental regulations restrict the amount of volatile compounds from printing press emissions [9, 10]. The surface energy of the film should be at least 46 mN/m for water-based ink printing or 38 mN/m for solvent-based ink printing [11]. In order to achieve this surface-energy level, the plastic film surface just before printing is modified with the chemical, flame, plasma, corona discharge, or UV surface treatment methods. Corona discharge is commonly practiced in industry and involves ionizing air with a high voltage to oxidize and roughen the film surface [11]. The corona discharge treatment of the polymer surface produces carbon radicals from the hydrocarbon backbone which form unstable hydroperoxides through rapid binding with oxygen in air. Hydroperoxides decompose with additional oxygen to produce various oxygen-based groups such as hydroxyl groups, ethers, ketones, aldehydes, carboxylic acid groups, and carboxylic ester groups [12]. The carbon radicals may also bind among themselves to produce several types of unsaturated carbon bonds [13, 14, 15]. In this study, the removal of water-based ink printed on corona-treated polyethylene film is investigated.

Deinking involves the removal of various colors and layers of inks from the plastic film [5, 6, 8]. It can be considered as a laundering process: the detachment of soil

(inks) from the substrate (plastic film) by a chemical mechanism and separation of the detached soil from the substrate and dispersion of the detached soil in a washing bath by mechanical action. Mechanical action, process chemistry, pH, time, and temperature are important in both plastic deinking and laundering textiles [5, 6, 8, 16]. Furthermore, ink could be considered as a particulate soil. The best way to remove particulate soil from a substrate is to use a surfactant that adsorbs at the water–solid particle interface. With the effective adsorption of surfactant, the interfacial tension between particulate soil and the substrate and the adhesion forces binding the particles together will be reduced. Then, the particulate soils can be dispersed in a washing bath by mechanical action [16].

In our previous study, surfactants were determined to be effective in the removal of both water-based and solvent-based inks from polyethylene film [5, 6, 8]. It is found that cationic surfactants are the most effective for deinking of solvent-based ink but a pH of at least 11 is required [8]. Nitrogen-based surfactants (quaternary ammoniums, amine oxides, and amine ethoxylates) were identified as the most effective surfactants for deinking of water-based inks at various pH levels. The nonionic surfactants (nonylphenol polyethoxylates and alcohol ethoxylates) were also found to be effective for deinking of water-based inks at basic pH levels. However, anionic surfactant is not very effective at deinking [5, 6, 8].

In this paper, the effect of addition of a hardness ion (calcium ions) with anionic, cationic, amphoteric, and nonionic surfactants on the deinking of water-based ink and on the zeta potential of the ink particles is discussed. The effect of varying the length of anionic and cationic surfactant hydrophobes on deinking is also discussed.

Experimental

Materials

Sodium dodecyl sulfate (SDS), sodium dodecanoate (SD), hexadecyltrimethylammonium bromide (CTAB), tetradecyltrimethylammonium bromide (TTAB) and dodecyltrimethylammonium bromide (DTAB) were obtained from Sigma Chemical Company with a purity of 99%. Sodium decyl sulfate (SDES) was from Eastman Kodak Company and sodium tetradecyl sulfate (STS) as a 27 wt % aqueous solution was from Pfaltz & Bauer. Primary tallow amine ethoxylate with an ethoxylation number of 5 (AMEO₅, trade name Varonic T-205) and alcohol ethoxylate with an ethoxylation number of 5 (AEO₅, trade name Witconol SN-70) were obtained from Witco Corporation. Some properties of these surfactants are given in Table 1. All the surfactants were used as received. The reagent-grade calcium chloride from Fisher Scientific was used as received. The water was double-deionized.

Table 1 Properties of surfactants used for the deinking of plastic film packaging

| Surfactant | Surfactant Type | Structure | Molecular weight (g/g mol) | CMC (mM) [22] |
|------------------------------------------------|-----------------|------------------------------------------------------|----------------------------|------------------|
| Sodium decyl sulfate (SDS) | anionic | $C_{10}H_{21}SO_4^-Na^+$ | 260.32 | 33 |
| Sodium dodecyl sulfate (SDS) | anionic | $C_{12}H_{25}SO_4^-Na^+$ | 288.38 | 8.2 |
| Sodium tetradecyl sulfate (STS) | anionic | $C_{14}H_{29}SO_4^-Na^+$ | 316 | 2.1 |
| Sodium dodecanoate (SD) | anionic | $C_{10}H_{23}COO^-Na^+$ | 200.3 | 23 |
| Dodecyltrimethylammonium bromide (DTAB) | cationic | $C_{12}H_{25}^+N(CH_3)_3Br^-$ | 308.3 | 16 |
| Tetradecyltrimethylammonium bromide (TTAB) | cationic | $C_{14}H_{29}^+N(CH_3)_3Br^-$ | 336.4 | 3.6 |
| Hexadecyltrimethylammonium bromide (CTAB) | cationic | $C_{16}H_{33}^+N(CH_3)_3Br^-$ | 364.5 | 0.92 |
| Tallow amine 5 ethoxylate (AMEO ₅) | amphoteric | $C_{18(18:1)}H_{36}N-(C_2H_4O)_3H$ $(C_2H_4O)_2H$ | 470–500 | – |
| Alcohol 5 ethoxylate (AEO ₅) | nonionic | $C_{10(10-12)}H_{21(21-25)}O(C_2H_4O)_5H$ | 387 | 0.39–1.3 (Witco) |

The plastic film was composed of 70% LDPE and 30% HDPE, and was reverse-printed with various colors of water-based ink (black, white, purple, pink, and orange) by the flexographic printing process after surface treatment with corona discharge. The printed surface was then coated with white ink to give an opaque appearance and to protect the ink layers [5]. This film was commercial plastic packaging supplied by Kimberly-Clark Corporation. Printed and clear parts of the plastic film were segregated, and the clear part was used as a standard for comparison with deinked plastic film.

Methods

All of the glassware was cleaned with a regular laboratory detergent and then acid-washed with nochromix solution (Godax Laboratories) for at least 1 h. Surfactant solutions were prepared fresh with double-deionized water for each of the experiments, and the pH level of the aqueous surfactant solution was adjusted immediately with Fisher-brand certified sodium hydroxide and hydrochloric acid before the deinking experiment was performed. The surfactant solutions containing calcium were first cooled to near freezing temperatures to force precipitation. The solutions were then heated to 25 °C and shaken periodically for 4 days to ensure equilibrium in order to determine that the solution composition was isotropic (i.e., no precipitate present at 25 °C) [17].

A representative sample (2.5 cm×2.5 cm) printed with each of the colors white, black, orange, purple, and pink was cut from the printed part of the plastic film and soaked in a 20-mL surfactant solution with 20 irregular-shaped porcelain beads. The sample tube was then placed on a reciprocating Eberbach shaker for 4 h. After 4-h agitation, plastic samples were taken out of the washing bath, rinsed with deionized water, and air-dried. All the experiments were performed at room temperature and each experiment was repeated at least twice.

Each deinked plastic film sample was first visually evaluated and then analyzed using a Hunterlab Ultra-

Scan spectrophotometer SN7834 d/8° system (Hunter Associates Laboratory, Reston, VA) in regular transmission mode. The intensity of the light passing through the plastic film was measured as a function of wavelength. The level of deinking was quantified by determination of the total color difference (DE^*) between the standard (clear plastic) and the deinked sample at the chosen standard illuminant (D65, representing daylight with a correlated color temperature of 6,500 K) and observer (10° standard observer). The working principle of the Hunterlab UltraScan spectrophotometer is explained in detail in Ref. [5].

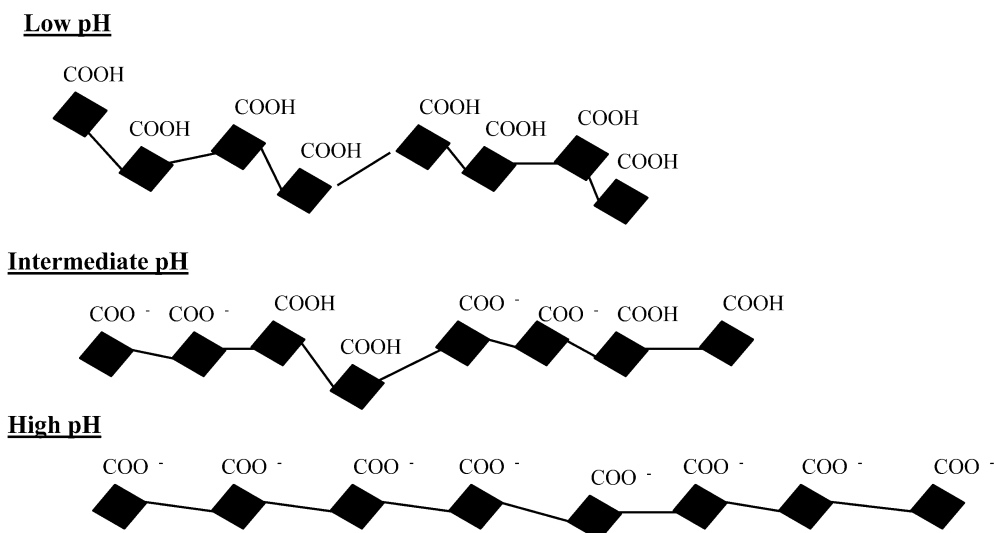
For zeta-potential measurements, water-based ink was extracted from the plastic film surface by soaking 0.5 g of the printed plastic film in 100 mL water at pH 12 and agitating it at room temperature with a reciprocating Eberbach shaker for 48 h. Ink extract (2 mL) was mixed with 23 mL water containing various amounts of surfactant and calcium. The solution pH was adjusted to the desired level with Fisher-brand certified sodium hydroxide and hydrochloric acid, stirred in closed vials for 24 h, and the suspension pH was readjusted if necessary before the zeta-potential measurements. The average zeta potential of 15 or 20 ink particles in each solution was determined at room temperature with a Zeta-Meter system 3.0 (Zeta-Meter) microelectrophoresis instrument, in which the velocity of individual ink particles in the electrical field was monitored optically. A molybdenum anode and a platinum cathode were used as electrodes.

Results and discussion

Effect of calcium ions on deinking

Ink is a complex suspension which includes pigments, binder, carrier, and additives (wax, surfactants, drying agent, and antioxidizing agents). Binder, which is mostly acidic acrylate for water-based ink, aids in the suspension of the pigments in ink before printing, and traps the pigments on the plastic film surface after printing. Acidic

Fig. 1 Water-based ink binder structure at different pH levels



acrylate includes carboxylic acid groups in its molecular structure (Fig. 1). At low pH, carboxylic acid groups are protonated and binder agglomerates. At intermediate pH, some carboxylic acid groups deprotonate to form anionic carboxylates, and the binder is somewhat

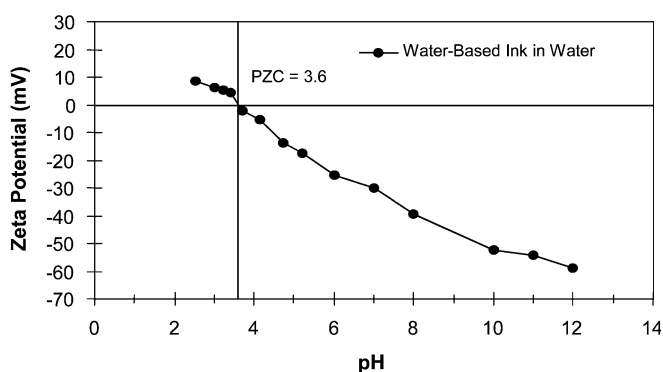


Fig. 2 Zeta potential of water-based ink particles as a function of pH

extended. At high pH, most of the carboxylic acid groups deprotonate, and the binder is fully extended. By changing the pH, pigments disperse in the carrier before printing and bind on the plastic surface after printing [9]. Zeta-potential measurements of water-based ink particles in water as a function of pH are consistent with these protonation properties of the binder at various pH (Fig. 2). As seen in Fig. 2, the point of zero charge (PZC) of water-based ink particles is about 3.6. Below this pH, ink particles are positively charged, while they are negatively charged above it. As illustrated in Fig. 3, at moderate pH levels, cationic surfactants are expected to adsorb better on the ink surface than anionic surfactants, which probably explains the superior deinking of the former [5].

Anionic surfactants (often fatty acids) are commonly used to deink paper in a flotation process [18]; however, calcium is required for effective deinking because it enhances the adsorption of the surfactant through coadsorption [19]. In this study, the effect of calcium on plastic deinking is tested.

Fig. 3 Adsorption of hexadecyltrimethylammonium bromide (CTAB) or sodium dodecyl sulfate (SDS) with calcium ions on water-based ink particles

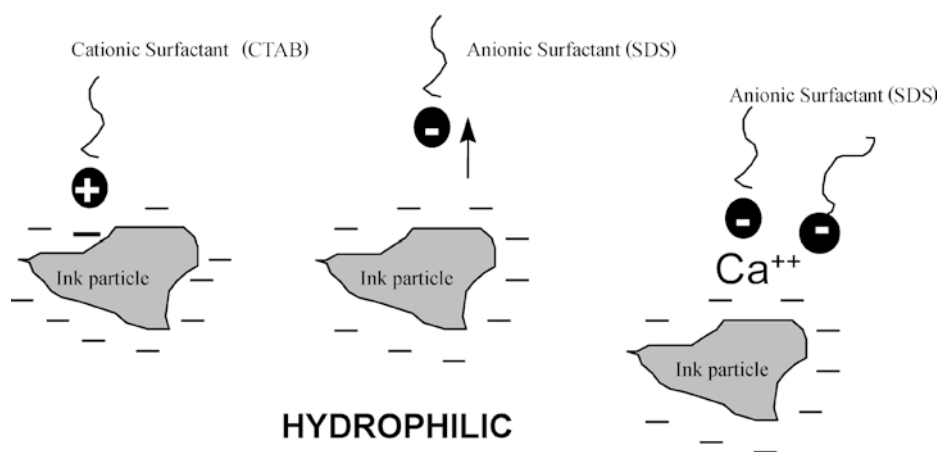


Fig. 5 DE^* between the plastic film (treated with water and 25 mM SD with various concentrations of calcium) and the clear plastic film as a function of pH

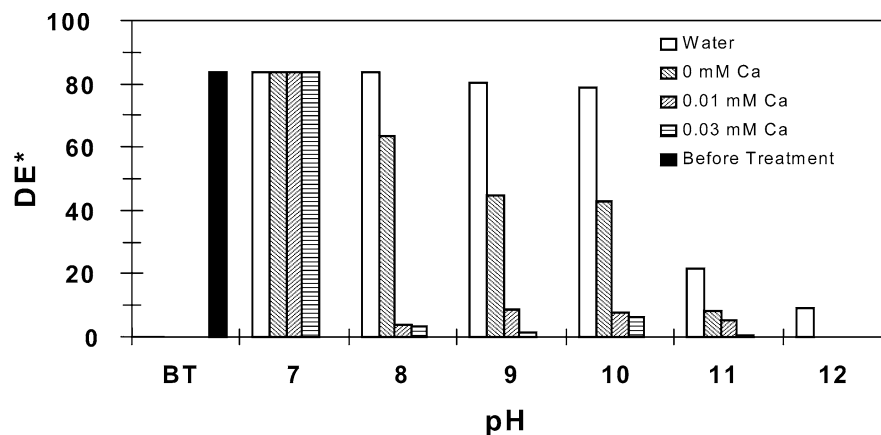
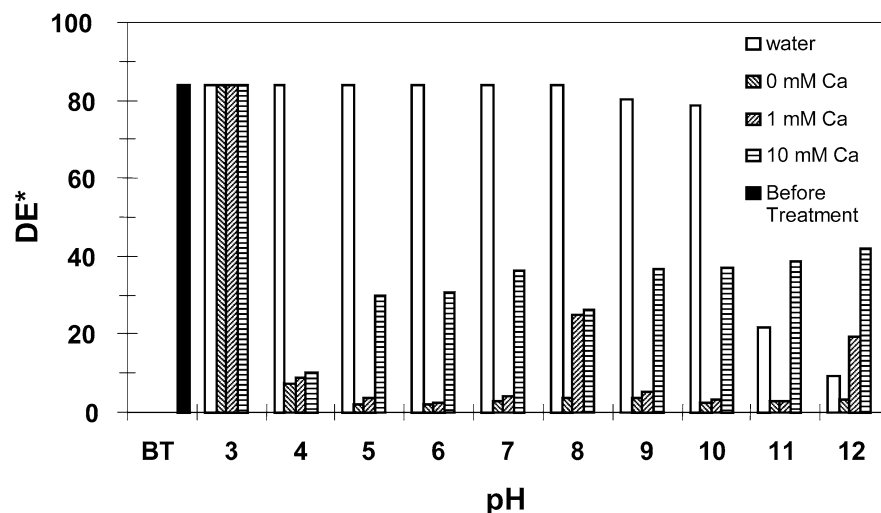


Fig. 6 DE^* between the plastic film (treated with water and 25 mM CTAB with various concentrations of calcium) and the clear plastic film as a function of pH



As seen in Fig. 7, adding calcium to the $AE O_5$ (nonionic surfactant) solution decreases the level of deinking at pH 7–11, and increasing the calcium concentration worsens the deinking level at all pH levels except at pH 12. As shown in Fig. 8, addition of calcium ions to $AMEO_5$ solution decreases the deinking capacity of $AMEO_5$ at both acidic and basic pH levels. Increasing the calcium concentration from 1 to 10 mM worsens the deinking level at all pH values. $AMEO_5$ behaves as an amphoteric surfactant [6]. It is nonionic at pH 8 and above; however, below pH 8, the surfactant exists as a mixture of cationic and nonionic surfactants. The partial cationic character of $AMEO_5$ at acidic pH explains the decrease in deinking level in the presence of calcium ions; however, the cause of the decrease in the deinking level at basic pH is not obvious.

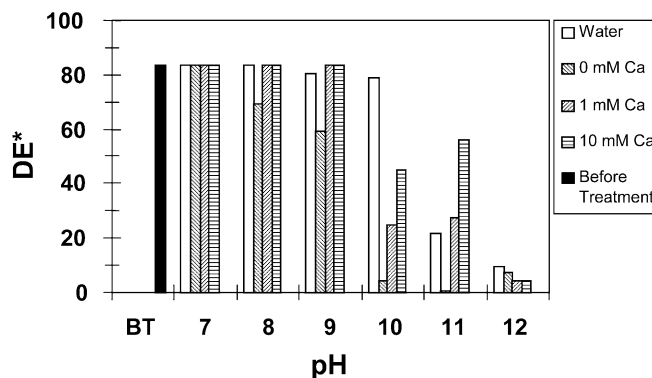


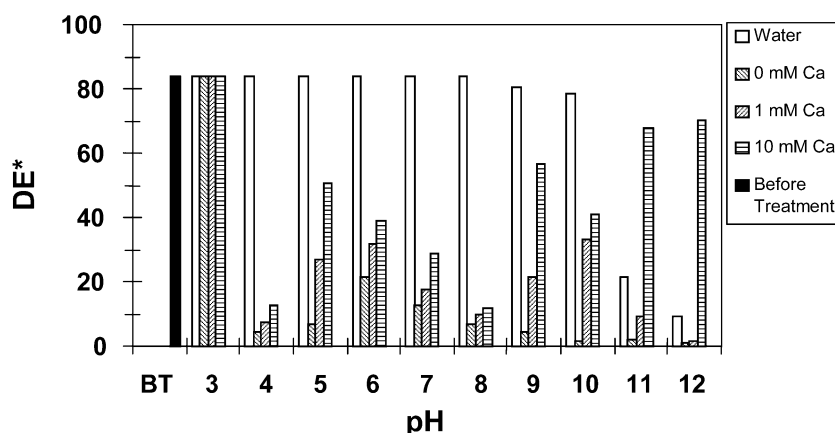
Fig. 7 DE^* between the plastic film (treated with water and 5 mM $AE O_5$ with various concentrations of calcium) and the clear plastic film as a function of pH

Effect of hydrophobe length of ionic surfactants on deinking

Experiments were conducted with three cationic and three anionic surfactants differing only the length of the

hydrophobe (number of carbon atoms) in the linear alkyl chain. These surfactants are cationic DTAB having 12 carbons, TTAB having 14 carbons, and CTAB having 16 carbons, and anionic SDES having 10 carbons, SDS having 12 carbons, and STS having 14 carbons in their hydrophobes. Increasing the chain length decreases

Fig. 8 DE^* between the plastic film (treated with water and 5 mM $AMEO_5$ with various concentrations of calcium) and the clear plastic film as a function of pH



the surfactant solubility in water and the CMC of the surfactant (Table 1).

The surfactant concentration was 25 mM for all solutions, and the pH was varied from 3 to 12 for cationic surfactants and from 7 to 12 for anionic surfactants. Experimental results are shown in Figs. 9 and 10 for anionic surfactants and cationic surfactants, respec-

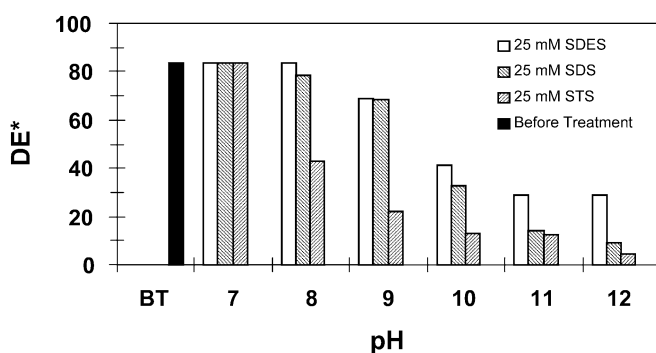
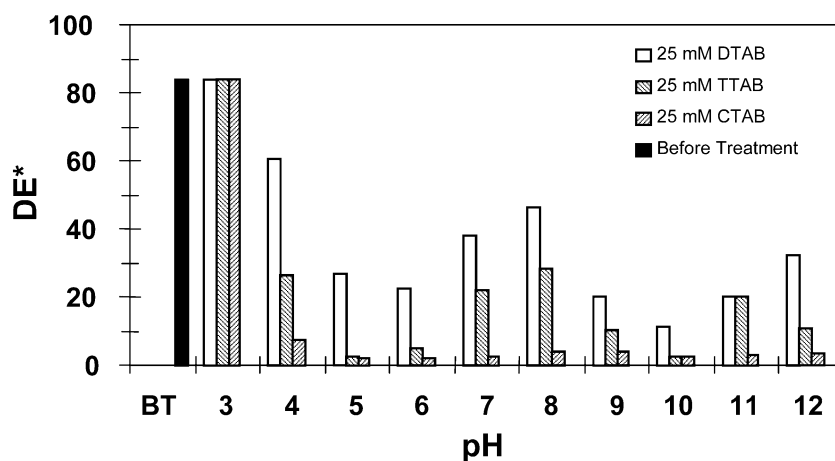


Fig. 9 DE^* between the plastic film treated with various lengths of anionic surfactant hydrophobe and the clear plastic film as a function of pH

Fig. 10 DE^* between the plastic film treated with various lengths of cationic surfactant hydrophobe and the clear plastic film as a function of pH



tively. Increasing the carbon chain length of ionic surfactants increases the deinking level at any pH. Use of the surfactant with the largest hydrophobe while the surfactant is still soluble at the conditions present appears to yield optimum deinking for single-component anionic or cationic surfactants. This is probably due to the increased adsorption of surfactant on the plastic film surface and on the ink with increasing hydrophobe size. In textile detergency, optimum cleaning with ionic surfactants generally occurs when the hydrophobe is as large as possible without becoming insoluble [23].

Zeta potential of ink particles

The electrical potential of the ink particles is anticipated to be important for the removal of the ink from the plastic film surface and also for the dispersion stability of the detached ink particles in the washing solution. This surface electrical potential is approximated by the zeta potential (electrical potential at the shear layer surrounding the particle) [24]. Adsorption of surfactant on ink particles could be indicated by the changes in

zeta-potential values. Some general guidelines for dispersion of negatively charged particles in water indicate that stability is observed for zeta potentials at a value lower than -30 mV, with values between -45 and -70 mV being desirable for electrostatic dispersion stabilization [24].

The zeta potential of water-based ink in water is shown in Fig. 2 as a function of pH. The PZC (pH where the zeta potential is zero) of water-based ink particles occurs at about pH 3.6. The fact that the zeta potential changes its sign with variation in pH indicates that H^+ and OH^- are potential-determining ions for water-based ink. Ink particle settling was qualitatively observed at pH 3–6, whereas ink particles were dispersed at pH 8–12. At pH 7, some ink particles agglomerated and settled, and some dispersed. Water-based ink particles disperse at basic pH levels probably because the binders (acidic acrylate) have carboxylic acid groups which deprotonate to form anionic carboxylates under basic conditions (Fig. 1). For water-based ink formulation, the amine is used as an organic base to form a water-soluble salt by neutralizing the acid resin binder. During printing, the amine is released as drying occurs, causing the precipitation of the resin and the formation of a film that binds the pigment on the plastic film; however, as determined here, this film can easily be redissolved when a basic solution is used.

The zeta potentials of water-based ink particles in the presence of 0.5 and 25 mM CTAB at pH 3–12 are presented in Fig. 11. The distinct difference between the zeta potential of water-based ink particles in water and that in surfactant solutions is consistent with substantial adsorption of surfactants on the ink surface. Over the range of pH and CTAB concentrations studied, adsorption of the surfactant on the ink surface causes the ink particles to become positively charged. Stable dispersions of ink particles were observed at all the pH values studied. The zeta potential of water-based ink particles was also determined at pH 5, 7, and 10 and CTAB concentration from 0.005 to 25 mM as shown in

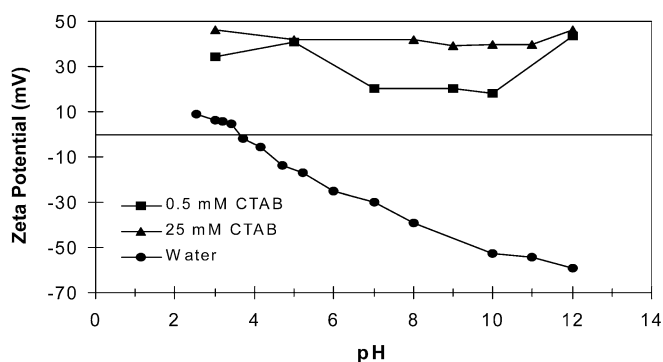


Fig. 11 Zeta potential of water-based ink particles as a function of pH in the presence of 0.5 and 25 mM CTAB

Fig. 12. Changes in the zeta potential indicate that the surfactant adsorption appears to occur in the Stern layer [25]. At pH 5, the zeta potential of water-based ink particles in water was measured to be about -15 mV. At this value, the agglomeration and settling of ink particles were observed. However, as seen in Fig. 12, the zeta potential of ink particles at pH 5 in all the CTAB solutions studied is positive. Stable dispersions of ink particles in CTAB solution over 0.01 mM were observed at pH 5. The zeta potential of ink particles in water at pH 7 and 10 is about -30 and -52 mV, respectively. At low concentrations of CTAB solutions (0.005, 0.01, and 0.05 mM) with pH levels of 7 and 10, the zeta potential of ink particles increases and as the CTAB concentration increases beyond 0.05 mM, the value of the zeta potential increases more substantially. Interpolation of the zeta-potential data in Fig. 12 results in an estimated zeta potential of zero around 0.07 mM at pH 7 and 0.09 mM at pH 10. Further increase in the CTAB concentration causes the zeta potential to become positive, probably because of the higher surface coverage of surfactants [26].

As seen in Fig. 13, the zeta potential of the ink particles increases in 5 mM AEO₅ above pH 5 and in 5 mM

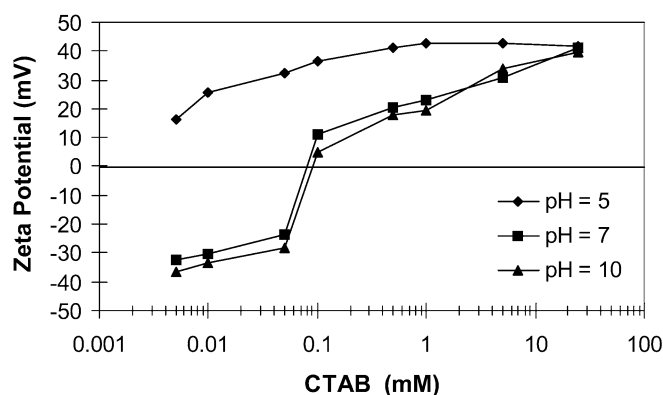


Fig. 12 Zeta potential of water-based ink particles as a function of CTAB concentration at various pH

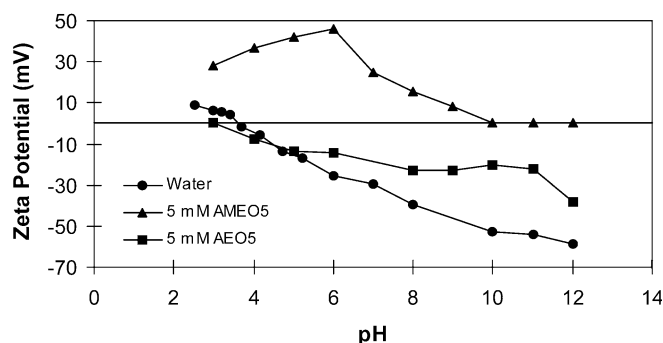
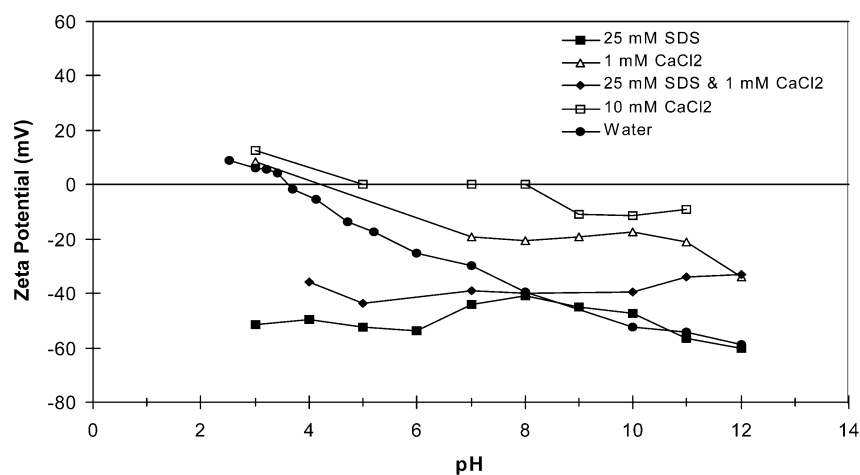


Fig. 13 Zeta potential of water-based ink particles as a function of pH in the presence of 5 mM AEO₅ and 5 mM AEO₅

Fig. 14 Zeta potential of water-based ink particles as a function of pH in the presence of 25 mM SDS, 1 and 10 mM calcium, and 25 mM SDS with 1 mM calcium



AMEO₅ at all the pH levels studied. The adsorption of nonionic surfactant onto a surface generally does not affect its charge. The increase in zeta potential with the adsorption of nonionic surfactants on the ink surface may be due to the shift in the position of the shear plane and the change of adsorption characteristics of the ions present [27, 28]. Ink particles qualitatively formed stable dispersions in AEO₅ solutions at all pH levels except 3. Below pH 10, the zeta potential of ink particles in AMEO₅ solution is positive, probably because the cationic form of AMEO₅ adsorbs on the ink surface. Ink particles settled in AMEO₅ solution for all the pH values studied.

The zeta potentials of ink particles in SDS (25 mM), calcium (1 and 10 mM), and the mixture of SDS/calcium (25 mM/1 mM) are shown in Fig. 14 as a function of pH. The decrease in electronegativity at pH 4–7 could be explained by the adsorption of SDS on ink particles even though the surface charge has the same sign as the surfactant. This could be due to surface heterogeneity; even though the net overall surface charge may be negative, there could be patches with wide variation in net charge including positively charged patches. Another potential explanation is chemisorption of the anionic surfactant on the negatively charged surface [29]. However, SDS does not change the zeta potential of ink particles at pH 8–12, where the ink is highly negatively charged. Calcium (1 mM) increases the zeta potential above pH 7 because positively charged calcium ions adsorb on the negatively charged ink particles. Addition of calcium ions to SDS solutions could synergize the adsorption of SDS molecules on the ink surface particularly above pH 9, probably because calcium ions contribute a bridge between the surfactant molecules and ink particles. Increasing the calcium concentration to 10 mM in water increases the zeta potential further above pH 5 and balances the ink surface charge

between pH 5 and 8. Ink particle settling was observed at pH 3–5 with 1 mM calcium and at pH 3–12 with 10 mM calcium in water.

Conclusion

Water-based ink particles in water have the PZC at a pH of about 3.6 and are negatively charged above this pH; therefore, cationic surfactant, which is the most effective surfactant for deinking, would tend to adsorb on the ink better than anionic surfactant. Both synthetic and natural anionic surfactants at alkaline pH were found to be effective for the removal of water-based ink from plastic film only in the presence of calcium, probably because calcium forms a bridge between negatively charged ink and the anionic surfactant head group. Cationic or anionic surfactants deink better as the number of carbons in the hydrophobe increase, with the largest hydrophobe yielding optimum deinking. The presence of calcium decreases the effectiveness of cationic, nonionic, and amphoteric surfactants because calcium competes for adsorption sites with these surfactants. The zeta-potential measurements of ink particles in water and surfactant solutions with and without calcium ions showed the possibility of surfactant and calcium ion adsorption on ink particles. The presence of surfactants, except for the ethoxylated amine (AMEO₅), stabilizes the ink particle dispersion in the washing bath above pH 3.

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Corporation, Henkel Corporation, Huntsman Company, ICI Americas Inc., Kerr-McGee Corporation, Lever Brothers Company, Lubrizol Corporation, Nikko Chemicals Ltd., Phillips Petroleum Corporation, Pilot Chemical Company, Procter & Gamble, Reckitt Benckiser North America, Schlumberger

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