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COAGULATION MECHANISMS OF IRON SALT AND SALICYLIC ACID

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ABSTRACT

This study focused on the reaction mechanisms exhibited by two iron coagulants and salicylic acid molecules. Experiments were performed to investigate the influence of polymeric and monomeric iron coagulants on treating waters containing natural analog organic matter. As predicted by the formation function theory, polyferric sulfate (PFS) consumed less alkali than $FeCl_3$ during the coagulation process. This means that the PFS forms amorphous $Fe(OH)_3$ colloid more easily than the $FeCl_3$. On the other hand, when the pH value was less than 4, the Fe(III) ion could complex with the salicylic acid through coordination reaction. However, the complex compounds that formed from either PFS or $FeCl_3$ under such conditions were too small to remove with filtration. At higher pH levels, iron-salt coagulates immediately reacted with OH^- ion, forming amorphous $Fe(OH)_3$ colloids and resulting in the adsorption of salicylic acid on $Fe(OH)_3$ colloid surfaces. Because the —OH and —COOH functional groups of salicylic acid have complex-forming O-donors, they coordinated with $Fe(OH)_3$ colloids on the surface

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layer of the oxide. Therefore, salicylic acid adsorbed on the surfaces of hydrous oxides through specific adsorption.

Key Words: Polyferric sulfate; Ferric chloride; Coagulation; Salicylic acid; Adsorption

INTRODUCTION

The presence of organic matter deteriorates the drinking water quality in many aspects, such as the carcinogenic disinfection byproducts (DBP) produced by chlorine reaction. Conventional coagulation can remove natural organic matter (NOM) such as humic acid and fulvic acid from public water supplies to a certain extent (1–3). Previous studies have shown a fraction of NOMs containing aromatic groups (4). Successful elimination of aromatic groups by coagulation depends on the number and the position of the functional —COOH and —OH groups in the aromatic ring. Therefore, a more precise determination of NOM removal requires a knowledge of the interactive mechanism between functional groups and coagulants as well as the role played by the complexation reaction (5). To understand the coagulation mechanisms between aromatic groups and coagulants in a water-treatment process, the behavior of salicylic acid in coagulation processes has been investigated widely (5,6). Because salicylic acid is the simplest compound containing —COOH and —OH functional groups in the ortho positions of the aromatic ring, it facilitates coagulation via complexation reactions. Salicylic acid is also a common aromatic acid and a chelating agent like fulvic acid (7).

Krasner and Amy (8) showed that organic matter could be removed from the solution by: (1) forming metal–organic complexes through electrical neutralization method, or (2) adsorbing onto metal hydroxide colloid surfaces. Generally, mechanism (1) occurs at low-coagulant doses and acidic conditions, while mechanism (2) occurs at high-coagulant doses and alkalinic conditions. Therefore, the NOM removal depends on the type and the dosage of the coagulants, and the pH condition of the reaction (9,10). We can use the basic monomeric Fe(III) salts or pre-polymeric iron species as coagulants when iron salt is used in the water-treatment processes. Direct addition of the basic Fe(III) salt as $FeCl_3$ to the water may cause extremely rapid and uncontrolled hydrolysis to form $Fe(OH)_3$ precipitates (11). In contrast, a controlled hydrolysis process by a polymeric iron species may slow down the rate of hydroxide precipitation upon dilution, and consequently allow the charged polymeric species to remain for a longer duration and enhance charge neutralization (12–14).

Polyferric sulfate (PFS) is a new polymerized inorganic coagulant whose molecular formula is $[Fe_2(OH)_n(SO_4)_{3-n/2}]_m$ $(n < 2,\ m > 10)$. It contains a range of preformed iron(III) hydrolysis species, $[Fe(OH)_x]^{(3-x)+}]$

and $[Fe_m(OH)_x]_n^{(3m-x)n+}]$, including $[Fe_2(OH)_3]^{3+}$, $[Fe_2(OH)_3]_2^{6+}$, and $[Fe_8(OH)_{20}]^{4+}$ (15,16). Therefore, PFS carries high cationic charges. Their enhanced surface activity and improved charge-neutralizing capacity may make them more effective at comparatively low dosages. As reported in our previous study, PFS may also be less sensitive to dosage change, and more likely to produce less residual iron than conventional coagulants (17,18). PFS also has a wider range of applicable pH and leaves less residual iron. The flocs produced from the PFS coagulation separate more easily during filtration. In addition, the less corrosive characteristic of PFS may reduce the maintenance requirements of treatment facilities. Because of these advantages, PFS has been studied extensively and applied to water-treatment systems in recent years (11,14).

The aims of this study included:

(a) to examine the hydrolysis characteristics of PFS and $FeCl_3$ with/without salicylic acid under lower pH conditions,
(b) to identify coagulation mechanisms of salicylic acid and iron salt from hydrolysis and adsorption results,
(c) to investigate the salicylic acid removal efficiency by polymeric and monomeric iron salts at different pH and coagulant dosages, and
(d) to identify the mechanism of salicylic acid reaction on $Fe(OH)_3$ colloid surface.

EXPERIMENTAL PROCEDURES

PFS Preparation

The PFS used in this study was prepared in our laboratory from concentrated sulfuric acid and ferrous sulfate using catalysis and oxidation reactions. The molar ratio of sulfuric acid to ferrous sulfate was kept greater than 0.2 to prevent formation of ferric oxide sediment. Then, $NaClO_3$ ($40\,g\,L^{-1}$) and $NaNO_2$ ($16.7\,g\,L^{-1}\,hr^{-1}$) were added as oxidant and catalyst, respectively, along with the pure oxygen to accelerate the oxidation of ferrous sulfate. After 80–100 min catalysis and polymerization at 303–353K, the final preparation included an aging period of 5–7 hr at 323K to achieve PFS stability. The PFS thus prepared is characterized as follows: $Fe(III) = 160\,mg\,L^{-1}$; $Fe(II) < 1\,mg\,L^{-1}$; $[OH^-]/[Fe] = 0.4$; reddish brown in color with consistent viscosity. Because $[OH^-]/[Fe]$ ratios are generally expressed as γ-ratios, this PFS preparation is written as $PFS_{r=0.4}$.

Hydrolysis of $FeCl_3$ and PFS

In each experiment, $FeCl_3$ and PFS were diluted to desired concentrations by adding the products to double-distilled water containing

$NaClO_4$ (0.01 M) with/without salicylic acid. The pH values of the resulting mixtures were determined with a digital pH meter and calibrated to pH 4 and 7 using phosphatic buffers. B^* calculated from the pH values of the solutions right after dilution represents the initial extent of their hydrolysis at various concentrations.

Salicylic Acid Coagulation Using $FeCl_3$ and PFS

A six-beaker jar-test was set up at room temperature for each trial. Each beaker contained 900 mL of synthetic water prepared using 100 mg L^{-1} $NaHCO_3$, 10^{-2} M $NaClO_4$, and 10 mg L^{-1} salicylic acid. The coagulants were added into the beakers containing the synthetic water, and the pH values were immediately adjusted to the given values using NaOH or HCl. Rapid stirring at 120 rpm for 2 min was followed by a 20-min flocculation period at 30 rpm, and a 30-min settling period. The sample pH values were recorded after settling. Samples were taken from 3 cm below water surfaces and filtered through 0.45 μm membranes. The residual concentrations of salicylic acid in the filtrates were analyzed using a UV analyzer (1 cm cell; U-2000, Hitachi, Tokyo, Japan) at a wavelength of 272 nm. To avoid the interference of the residual iron on salicylic acid analysis, the coagulation pH values were kept higher than 5 (5).

Batch Adsorption of Salicylic Acid

The same procedure as done in the coagulation experiment was followed. $NaClO_4 \cdot H_2O$ was used to adjust Milli-Q water (1 L) to have the ionic strength 0.01 M. After the addition of the required amount of PFS or $FeCl_3$ (10 mg L^{-1} as Fe), the solution was adjusted to pH 5–6 using NaOH. The solution was stirred under the conditions similar to those conducted in the coagulation experiment resulting in amorphous $Fe(OH)_3$. Thereafter, the solution was put into several polyethylene bottles, and each one was adjusted to different designated pH. During the first hour, the pH values were controlled from 5 to 10 using HCl or NaOH and the suspensions were shaken constantly. After 1 hr, given concentrations of salicylic acid were added to the ferric suspensions, and the pH values were readjusted from 5 to 10. The samples were then shaken in a reciprocating shaker for 8 hr at 25°C to allow adsorption. At the end, the solids were separated by membrane filtration with a 0.45 μm filter. The filtered supernatants were then analyzed for residual salicylic acid.

Changing Solution PH After Coagulation to Measure the Removal Rates

To compare the removal rates affected by the pH of the solution, this study was first conducted under pH 5 with coagulant 10 mg L^{-1} added to the bulk solution of salicylic acid. After coagulation, the solution was distributed into six polyethylene bottles. In each bottle, the pH was adjusted to a designated value between 5 and 8, stirred at 100 rpm for 4 hr, and then filtered. After filtration, UV analyzer was used to determine the residual concentrations of salicylic acid in the filtrates.

RESULTS AND DISCUSSION

The Coagulation Mechanism Between Iron Salt and Salicylic Acid

Two coagulation mechanisms may occur between the iron coagulants and the salicylic acid. Under lower pH conditions, a complex reaction may occur resulting in a soluble complex compound that cannot be removed by settling or filtering. The other way is that $Fe(OH)_3$ precipitates form at higher pH conditions and salicylic acid can be removed as a phase change.

Complex Reaction in Low PH and Homogeneous Solution

Under low pH conditions, the addition of Fe(III) ions into salicylic acid solutions will change the solution color from transparent to purple, indicating the formation of a complex of the Fe(III) ions and the salicylic acid. An increase in the pH level will result in the disappearance of the purple color indicating the formation of $Fe(OH)_3$ precipitation, which destroyed the bonding of the Fe–salicylic acid complex. To compare the degree of Fe(III) hydrolysis with that of the formation of the Fe–salicylic acid complex at pH lower than 4, we applied the following function developed by Tang and Stumm (19) :

$$B^* = B_H + B - A \tag{1}$$

In the equation, $B^* = [OH^-]_{bond}/[Fe]_T$; $B_H = [H^+]/[Fe]_T = 10^{-pH}/[Fe]_T$; $B = [OH^-]_{add}/[Fe]_T$; $A = [H^+]_{add}/[Fe]_T$.

B^* is the number of OH^- ions that each iron ion bonds in the hydrolysis process. B_H is the initial ratio, which is the counterpart of the free H^+ in the iron solution and can be calculated from the pH value. $[OH^-]_{add}$ and $[H^+]_{add}$ are the strong base and acid added. Therefore, the pH values of the stock and diluted solutions of $PFS_{r=0.4}$ or $FeCl_3$ were measured under equilibrium conditions to determine the values of A and B_H, and the B^* values were calculated using Eq. (1). The pH values of the diluted $PFS_{r=0.4}$ and

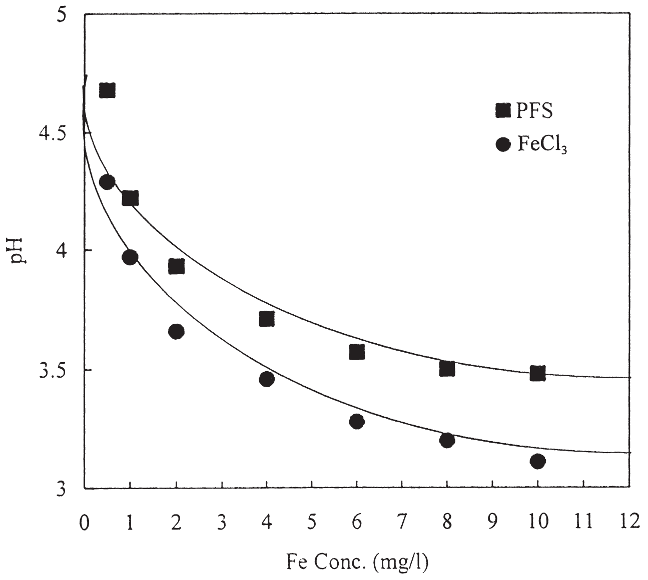
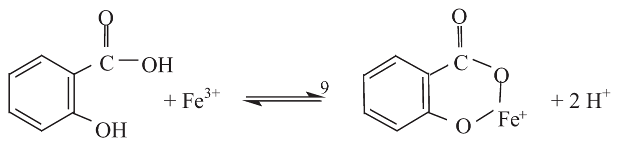


Figure 1. Relationship between solution pH values and PFS and $FeCl_3$ dosages.

$FeCl_3$ solutions shown in Fig. 1 were measured under equilibrium conditions. The results show that the pH level decreased as the Fe(III) ion addition was increased, which suggested that more hydrolysis reaction occurred at higher Fe(III) dosages. A similar experiment was performed by adding larger amounts of Fe(III), and the B^* values were calculated using Eq. (1). The results are shown in Fig. 2. It was found that the differences in B^* values between $PFS_{r=0.4}$ and $FeCl_3$ at the same Fe concentrations were always between 0.3 and 0.4. Because 0.4 OH^- ion had already bonded to each Fe(III) ion during the preparation of PFS, as evidenced by the molecular formula of $PFS_{r=0.4}$ being $Fe_2(OH)_{0.8}(SO_4)_{2.6}$, therefore the PFS consumed less alkalinity than the monomeric ferric chloride. This property of $PFS_{r=0.4}$ benefits the floc production and leaves less residual iron.

On the other hand, if the solution contains organic compounds that has a coordinating ligand, the Fe(III) ions will complex with the organic compounds instead of hydrolysis. As shown in Eq. (2), the functional groups —COOH and —OH of the salicylic acid coordinate with Fe(III) ions to form a soluble complex compound that reduces B^* values and lessens the hydrolysis reaction (5,20).



(2)

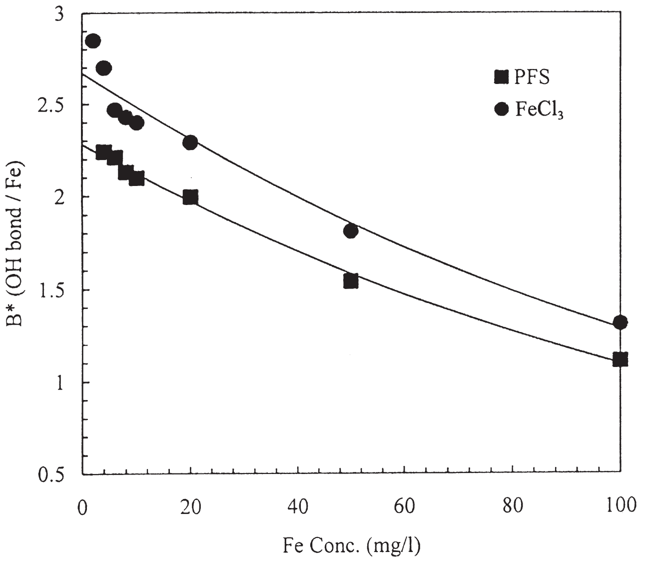


Figure 2. Relationship between B^* and PFS and $FeCl_3$ dosages.

The change in B^* of $10\,mg\,L^{-1}$ salicylic acid solutions when different amounts of PFS and $FeCl_3$ coagulants were added is shown in Fig. 3. At lower Fe concentrations, the differences between the B^* values in Fig. 3 and those shown in Fig. 2 were more significant. This result confirms that iron salt complexes with salicylic acid instead of hydrolysis. Thus, the greater the molar ratio of salicylic acid–iron, the greater influence on the hydrolysis and B^* values of the iron salt. For the above-mentioned reason, the formation of this soluble complex was particularly observable at acidic pH levels and high salicylic acid–iron molar ratios. It is shown that at pH lower than 4, the Fe ions tend to bond with the bidentate functions of salicylic acid using their empty orbital. The concentration of the Fe–salicylic acid complex can be detected at 525 nm with spectrometric method. Figure 4 shows percentage of the Fe–salicylic acid complex at various iron concentrations when the salicylic acid was maintained at $10\,mg\,L^{-1}$ ($0.073\,M$). It can be seen that at $5\,mg\,L^{-1}$ ($0.089\,M$) $FeCl_3$, 90% of the salicylic acid are in complex form indicating that each Fe ion of the $FeCl_3$ combines with one salicylic acid molecule. This result is similar to that from the study by Rahni and Legube (5) that at pH 3.5, salicylic acid reacted with Fe(III) ion to form a soluble complex at one to one stoichiometry. The profile of the complex formation with PFS curve is clearly lower than that of the $FeCl_3$ indicating that every salicylic acid molecule bonds with three or four Fe ions of PFS. This is because the PFS being a preformed hydrolyzing coagulant cannot bond with salicylic acid in a one to one stoichiometry. However,

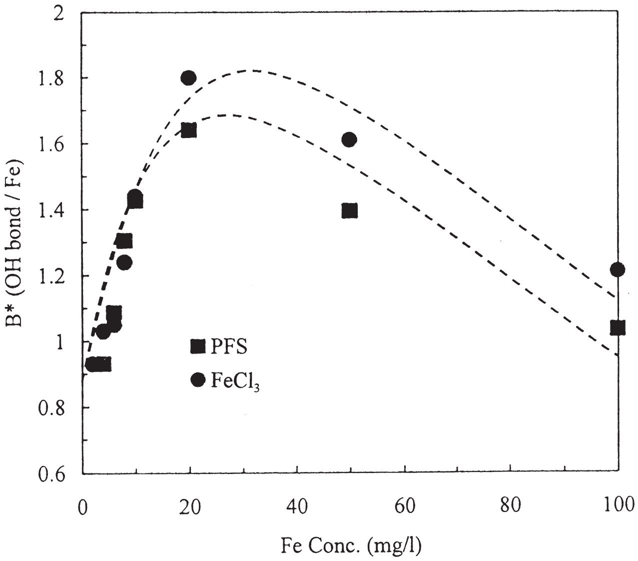


Figure 3. Relationship between B^* and PFS and $FeCl_3$ dosages in $10\,mg\,L^{-1}$ salicylic acid solutions.

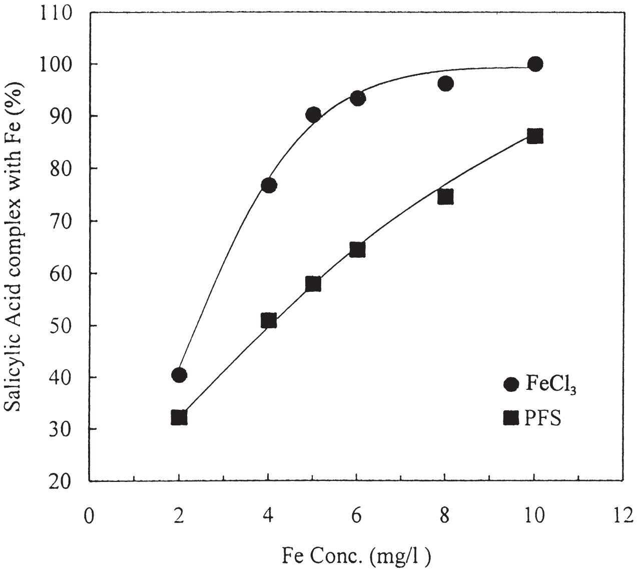


Figure 4. Salicylic acid complex percentage ($10\,mg\,L^{-1}$) vs. PFS and $FeCl_3$ concentrations.

we must notice that no floc was formed during the complex reaction between Fe and salicylic acid at pH values lower than 4, which made removal of the Fe–salicylic acid complex compound impossible by sedimentation or filtration.

Comparison of Salicylic Acid Removed by Adsorption and Coagulation at Higher PH Values of the Solution

Following the above-mentioned experimental steps, as the pH value of the solution increased with addition of NaOH, salicylic acid may be removed by coagulation or $Fe(OH)_3$ precipitation. But there is a point needed to be clarified—under the experimental dosage what is the possibility for iron coagulants to form the flocs of $Fe(OH)_3$ before salicylic acid could be reacted onto it. Figure 5 shows the coagulation capacity of salicylic acid via jar mixing–filtering at same PFS dosage ($10\,mg\,L^{-1}$ as Fe) and different pH values. The salicylic acid residual percentage via amorphous ferric hydroxide flocs adsorption is also included to make a comparison with coagulation capacity at pH values higher than 5. Similar removal curves were obtained for adsorption and coagulation. Therefore, we may conclude that removal of low-molecular-weight organic materials, such as

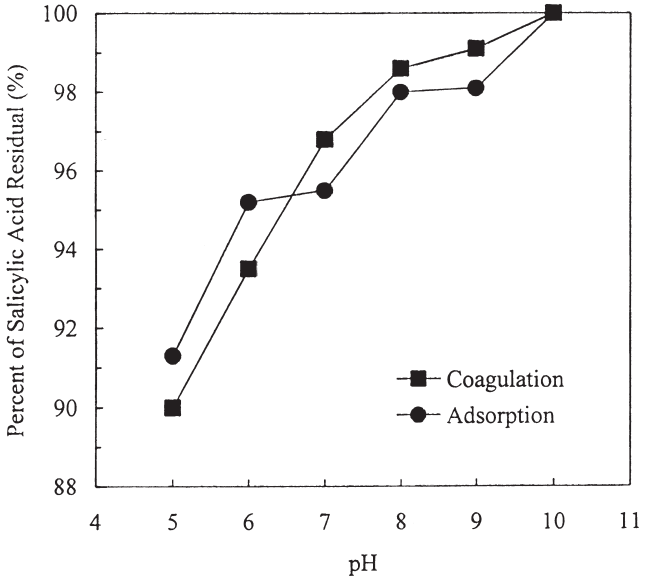
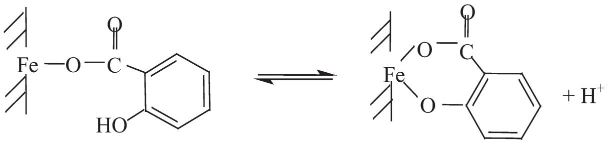


Figure 5. Comparison of differences in salicylic acid residual percentage between adsorption and coagulation reactions (PFS, 10 mg Fe per L).

salicylic acid, by coagulation is attributable primarily to adsorption. As in the study by Huang and Shiu (6), this result also indicates that the coagulation mechanism between salicylic acid and iron coagulant includes the initial formation of the amorphous ferric hydroxide flocs upon the addition of iron salts and followed by the adsorption of the salicylic acid molecular on floc surfaces.

Mechanism of Salicylic Acid Adsorption on $Fe(OH)_3$ Colloid Surface

Evanko and Dzombak (4) used α-FeOOH as an adsorbent to investigate the adsorption mechanism of salicylic acid. In a similar study, Kummret and Stumm (20) investigated salicylic acid adsorption on γ-Al_2O_3 surfaces. Evidence from both studies implies that specific adsorption is the major mechanism of salicylic acid adsorption on metal hydroxide surfaces. As shown in Eq. (3), under specific adsorption, salicylic acid used its –OH and –COOH functional groups to adsorb on the iron hydroxide surface and to produce stable six-angle ring structures.



(3)

The adsorption sites on $Fe(OH)_3$ surface are amphoteric and can be simulated in protolysis reactions of the hydroxyl group. As indicated in Eqs. (4) and (5), the adsorption sites on $Fe(OH)_3$ surface can form protonated and deprotonated hydroxyl groups (21).

$$FeOH + OH^- \leftrightarrow FeO^- + H_2O \tag{4}$$

$$FeOH + H^+ \leftrightarrow FeOH_2^+ \tag{5}$$

Figure 6 shows the effect of pH, ranging from 5 to 10, on the adsorption of salicylic acid onto the PFS and $FeCl_3$ formed $Fe(OH)_3$ colloid surface. Both removal curves shown in Fig. 6 indicate that salicylic acid residual percentage decreases with pH, perhaps because surface protonation increases with a decrease in pH. This increase results in more $FeOH_2^+$ positively charged sites on oxide surfaces, thereby enhancing the attractive force between the ferric oxide and the salicylic acid. Figure 7 shows the overall salicylic acid residual percentage via the process of jar mixing–settling–membrane filtering at various ferric coagulant dosages while a pH of 5 was maintained during rapid mixing. The results show that residual percentage decrease with the increasing coagulation dosage.

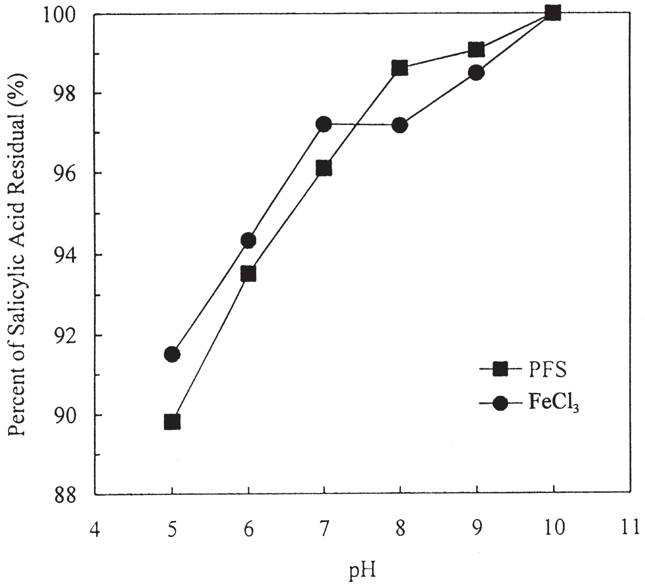
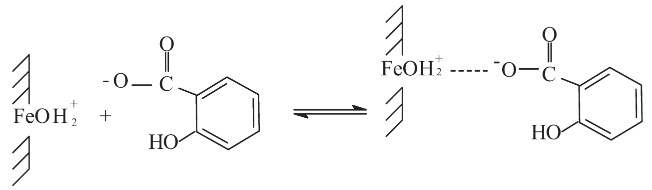


Figure 6. Salicylic acid ($10\,\mathrm{mg\,L^{-1}}$) residual percentage at various coagulation pH conditions using PFS and $FeCl_3$ as coagulants (10 mg Fe per L).

Because more ferric hydroxide precipitated as the dosage increased, more salicylic acid was adsorbed onto the ferric hydroxide surface.

The coagulation behavior of salicylic acid molecules on $Fe(OH)_3$ floc surfaces can be explained using the triple-layer model (22). The salicylic acid molecules undergo surface reaction by either inner-sphere complexation (specific adsorption) as described in Eq. (3), or by outer-sphere complexation (ion-pair adsorption) as described in Eq. (6).



(6)

If outer-sphere complexes are formed, there were one or more H_2O molecules between the adsorbed species and ferric hydroxide surfaces, thereby an increase in the pH values of the solution after coagulation reduces the positive charge of hydroxide surface and affect salicylic acid adsorption. However, if inner-sphere complexes are formed, the adsorbed species directly react with

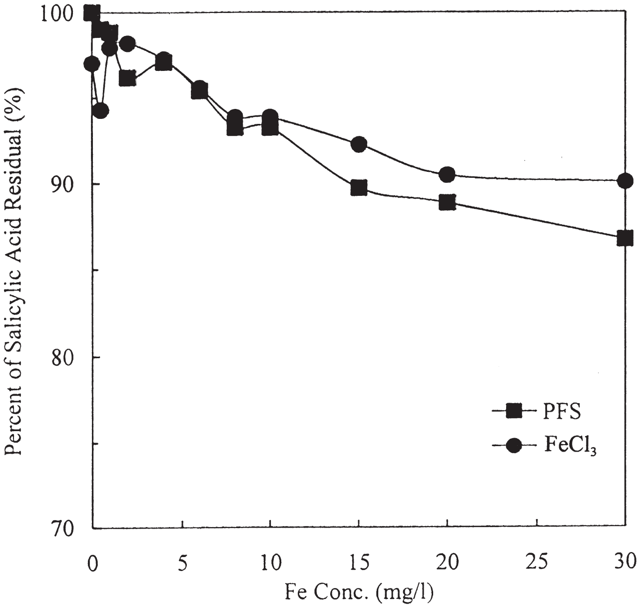


Figure 7. Salicylic acid (10 mg L^{-1}) residual percentage after jar mixing–settling–membrane filtering shown as a function of dosage at pH 5.

$Fe(OH)_3$ surfaces via stable chemical bonding, therefore a change in pH value of the solution after coagulation do not affect the adsorption ability. Following the coagulation experiments performed under pH 5, the solutions were adjusted again to a range between pH 5 and 8. After shaking for 4 hr, the salicylic acid concentrations of solutions were checked. A significant difference was found (Fig. 8) when compared with the data in the coagulation experiments. The circular dot curve in Fig. 8 shows that residual percentage of salicylic acid is not influenced by the gradually increasing pH values of the solution after coagulation. These results indicate that the coagulation mechanism of salicylic acid is adsorption on $Fe(OH)_3$ surfaces by inner-sphere reaction.

CONCLUSION

Insignificant removals of salicylic acid were discovered by coagulation with poly-nucleus (PFS) or mono-nucleus ($FeCl_3$) coagulants even at high dosages. However, at higher solution pH values, the removal efficiency of salicylic acid increased when the coagulant dosage was increased, and the

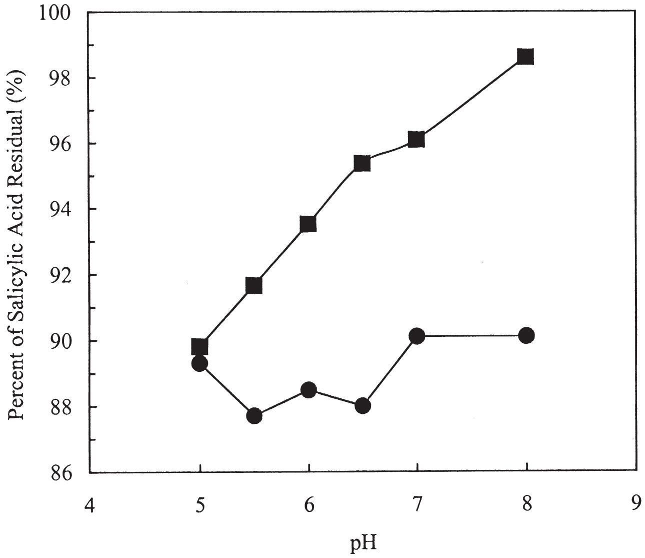


Figure 8. Comparison of differences in salicylic acid ($10\,\mathrm{mg\,L^{-1}}$) residual percentage for changed solution pH values (equilibrium time 4 hr) after PFS (10 mg Fe per L, pH 5) coagulation reaction (●), and for coagulation with PFS (10 mg Fe per L) at various pH values (■).

coagulation-reaction mechanism demonstrated that flocs of amorphous $Fe(OH)_3$ primarily formed during ferric coagulation, followed by the adsorption of salicylic acid onto oxide surfaces via inner-sphere reactions. However, at lower pH values of the solution, the empty orbital of free-ferric-ion tend to bond with salicylic acid with bidentate functional groups to form soluble complexes. Using formation functions, we calculated B^* values, and found that the B^* values were influenced by salicylic acid molecules; this clearly proves that the ferric ion and salicylic acid actually form a soluble complex. The B^* values obtained from our hydrolysis experiments also indicate that the alkalinity consumption of PFS is lower than that of monomeric ferric chloride. This evidence implies that PFS may be a better choice of coagulants in water-treatment processes.

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