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Influence of Polymer Architecture on the Stabilization of Iron and Manganese Ions in Aqueous Systems

The influence of polymers as stabilizing additives for iron and manganese ions was investigated in an aqueous system. The polymers tested include: homo-polymers of acrylic acid, maleic acid, methacrylic acid, 2-acrylamido-2-methylpropane sulfonic acid; co-polymer of acrylic acid : vinyl acetate, acrylic acid : hydroxypropyl acrylate, acrylic acid : carbitol acrylate, acrylic acid : 2-acrylamido-2-methylpropane sulfonic acid; and ter-polymer of acrylic acid : methacrylic acid : acrylic acid : acrylic acid : 2-acrylamido-2-methylpropane sulfonic acid : sulfonated styrene, acrylic acid : 2-acrylamido : t-butylacrylamide. It was found that polymer architecture i. e., monomer type, monomer charge, molecular weight, and polymerization solvent, strongly influences the performance of polymer as iron and manganese ions stabilizing additives. The stabilization data reveal the following order in terms of decreasing polymer performance: ter-polymer > co-polymer > homo-polymer.

Key words: Iron, manganese, stabilization, polymers

Einfluss der Polymerarchitektur auf die Stabilisierung von Eisen- und Manganionen in wässrigen Systemen. Der Einfluss von Polymeren als Stabilisierungsadditiv für Eisen- und Manganionen wurde in einem wässrigen System untersucht. Die getesteten Polymere waren: Homopolymere der Acrylsäure, Maleinsäure, Methacrylsäure, 2-Acrylamido-2-methylpropansulfonsäure; Copolymere von Acrylsäure : Vinylacetat, Acrylsäure : Hydroxypropylacrylat, Acrylsäure : Carbitolacrylat, Acrylsäure : 2-Acrylamido-2-methylpropansulfonsäure; und Terpolymere von Acrylsäure : Methacrylsäure : Acrylsäure : Acrylsäure : 2-Acrylamido-2-methylpropansulfonsäure : sulfoniertes Styrol, Acrylsäure : 2-Acrylamido : t-butylacrylamid. Es wurde gefunden, daß die Polymerarchitektur, d. h. Monomertyp, Monomerladung, Molekulargewicht und Polymerisationslösungsmittel die Leistung des Polymers als Stabilisierungsadditiv für Eisen- und Manganionen stark beeinflusst. Die Stabilisierungsdaten zeigen die folgende Reihenfolge bezüglich abnehmender Polymerleistung: Terpolymer > Copolymer > Homopolymer.

Stichwörter: Eisen, Mangan, Stabilisierung, Polymere

1 Introduction

In virtually all processes in which untreated water is heated, fouling of equipment surfaces is the single most serious problem encountered. Affected application areas include laundry, dairy, dishwashing, cooling, boiler, geothermal, power generation, semi-conductor manufacturing and many other production processes. The deposits that are formed greatly reduce heat transfer causing energy losses or material damage especially when coupled with corrosion, clogging of pipelines, emergency shutdown, and premature equipment replacement. Among the various dissolved impurities present in natural waters, iron-based compounds present the most serious problems in many domestic and industrial applications. In the reduced state, iron (II) or ferrous (Fe^{2+}) ions are very stable and pose no serious problems, especially at low pH values. However, upon contact with air, ferrous ions are oxidized to higher valence state and readily undergo hydrolysis to form insoluble hydroxides.

In addition to the feed water, sources that may also contribute to iron fouling include boiler condensate, corrosion products from pumps and pipes, and biological activity (transformation of iron during bacterial processes). The solubility of iron compounds in industrial water systems is determined by their form as well as their solubility product constant. The amount of the dissolved iron containing compounds in water is dependent on several factors including pH, temperature, total dissolved solids, type and concentrations of anions. When the corrosion rate is very low, the iron hydroxide formed may deposit on the heat exchanger surface as a permeable or impermeable film at the corrosion site. Under severe corrosion conditions, the circulating water transports iron ions and oxides. Iron oxides that form under these conditions are colloidal and form stable suspensions [1]. Other types of iron-based compounds that can potentially deposit on the heat exchanger surfaces include iron carbonate, iron silicate, iron sulfide, etc. Additionally, it has been reported that soluble metal ions (i. e., Cu, Fe, Zn, Mn) interferes with the performance of calcium phosphate scale inhibitors [2–4].

Manganese (Mn) ions, like $Fe^{3+}$and Al^{3+} ions, also cause serious problems including staining of toilet bowls and sinks, graying of laundry, fouling of heat exchanger and reverse osmosis membrane surfaces, and pitting corrosion. In well waters, Mn ions fouling and deposition is commonly associated with specific microorganisms. Manganese is usually present below 0.5 mg/l in public water supplies. Private office supplies often contain higher Mn ions levels, mostly in a dissolved form that precipitates as hydroxide on exposure to the oxygen. In cooling water applications, Mn based foulants have been identified in both once through and re-circulating systems. In these cases various factors such as the presence of oxidizing agents (e. g., oxygen, chlorine), pH, temperature, and natural organics appear to influence the precipitation of manganese oxides. Manganese solution chemistry is very complex. Manganese exists in several oxidation states among which Mn^{2+} and Mn^{4+} are the most important with respect to water problems. Mn^{2+} is soluble and less susceptible to oxidation at pH = 7. In alkaline pH conditions, Mn^{2+} readily oxidizes to less soluble Mn^{4+} (e. g., MnO_2). Manganese-based deposits typically encountered in cooling water systems contain MnO_2. Ferrous ions oxidation occurs at a somewhat lower pH than a Mn^{2+}oxidation and this is perhaps the reason that Fe and Mn salts are typically encountered together in domestic and industrial water applications.

The use of acrylic acid/maleic acid based homo-, co-, and ter-polymers for domestic and industrial applications are well known [5–7]. For example, in home, institutional, and industrial (HI&I) applications polymers are incorporated in the formulations to achieve various performance objectives i.e., to prevent precipitation of Ca-surfactant, Ca-polyphosphate, calcium carbonate salts; dissolve deposits from hard surfaces; remove clay/soil from fabric; suspend and prevent re-deposition of suspended matter on washed fabric/glass. In industrial water treatment, polymers are used to prevent the precipitation of scale forming salts and depositions of un-wanted materials on heat exchangers and reverse osmosis membrane surfaces.

During the last two decades the performance of polymers as precipitation inhibitors and dispersants has attracted the attention of academic researchers and industrial technologists. It has been reported that water chemistry and polymer architecture play important roles in inhibiting the precipitation of scale forming salts and also in dispersing suspended matter under stressed system conditions i.e., high hardness, high alkaline pH, high temperature [8–10]. However, the role of polymer architecture in stabilizing various metal ions has been mostly overlooked.

The term stabilization used in this paper refers to an ability of an additive (polymeric or non-polymeric) to form soluble complexes and/or inhibit metal ion hydrolysis or metal hydroxide precipitation [11]. In practice, it may be difficult to differentiate between truly soluble and very finely dispersed particles. Under the conditions employed in the present work, % stabilization is defined as that concentration of the ionic species which is not removed by filtration. On the other hand, dispersants function by de-agglomerating the preformed suspended matter (i.e., clay, iron oxide, manganese dioxide). Closely related to dispersion is crystal modification of scale forming salts (i.e., calcium carbonate, calcium sulfate, calcium phosphate). In this process, the morphology of growing crystals is modified and extent of crystal growth is diminished thereby delaying the settling of crystals. The terms chelation, sequestration, and complexation are generally applied to phenomena where ions or complex species are maintained in a soluble form.

This paper is a continuation of Noveon's efforts to understand the impact of polymer architecture (i.e., polymer composition, molecular weight, ionic charge) in stabilizing iron and manganese ions in aqueous systems. In addition, experiments were also conducted to study the influence of polymer composition on metal oxides/hydroxides particles. It is hoped that the data presented in this paper would enable the industrial technologists to recognize the importance of polymer architecture in selecting the appropriate polymers for their formulations.

2 Experimental

2.1 Apparatus

The pH of solutions containing stabilizing additive and Fe^{3+} and Mn^{2+} ions in synthetic tap water was measured with pH meter (Model 605, Brinkmann Instruments, Westbury, New York) equipped with a combination pH electrode (Model 910500), Orion Research, Inc.

2.2 Chemicals

Stock solutions of calcium chloride, magnesium chloride, sodium chloride, and sodium sulfate were prepared by dissolving known amounts of reagent grade chemicals in distilled water. Sodium bicarbonate solution was made fresh for iron and manganese ions stabilization experiments. Stock solutions (0.1 %, active solids) of polymers were prepared by dissolving known amount of polymers in distilled water (100 ml). The pH of polymers solutions was adjusted to 8.00 using sodium hydroxide.

2.3 Ferric ions stabilization test method

A known amount of Fe^{3+} as $Fe(NO_3)_3$ was added to a known volume of synthetic tap water containing varying amounts of polymer stock solution in a 125 ml glass bottle. The synthetic tap water was prepared by mixing standard solutions of calcium chloride, magnesium chloride, sodium sulfate, sodium chloride, and sodium bicarbonate. The synthetic water composition was 100 mg/l Ca^{2+}, 30 mg/l Mg^{2+}, 314 mg/l Na^+, 200 mg/l SO_4^{2-}, 571 mg/l Cl^-, and 60 mg/l HCO_3^-. After the addition of Fe^{3+} solution to the synthetic tap water, the pH of the solution was adjusted to 7.00 with dilute sodium hydroxide. The experimental solutions were left at room temperature (23 °C) without stirring. After 2 hr the solutions were filtered through 0.22 micron filter and the filtrate analyzed for iron by Inductively Coupled Plasma – Optical Emission Spectroscopy.

2.4 Manganese ions stabilization test method

A known amount of $MnCl_2$ stock solution was added to a known volume of distilled water containing 100 mg/l of sodium bicarbonate and known amounts of polymer stock solution in a 125 ml glass bottle. After the addition of $MnCl_2$ solution, a known amount of bleach or sodium hypochlorite solution was added, followed by pH adjustment of final solution to 7.50 with dilute HCl. The final concentration of hypochlorite in the experimental solutions was ~10 ppm. The experimental solutions were stored without agitation in a water bath maintained at 40 °C. After 3 hr the solutions were filtered through 0.22 micron filter and the filtrate analyzed for Mn ions by Inductively Coupled Plasma – Optical Emission Spectroscopy.

The performance of the stabilizing agent for M (Fe or Mn) was calculated according to the following equation:

$$S(\%) = \frac{[M]_{sample} - [M]_{blank}}{[M]_{initial} - [M]_{blank}} \times 100\,\%$$

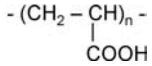
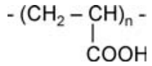
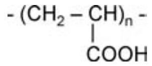
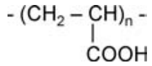
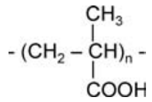
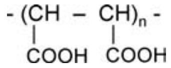
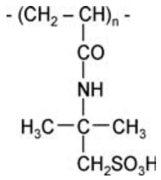
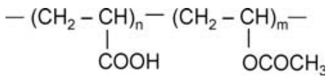
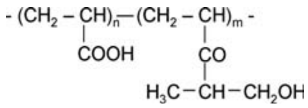
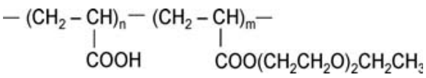
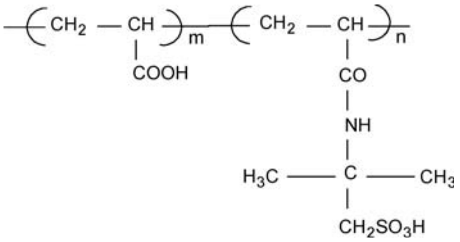
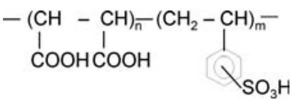
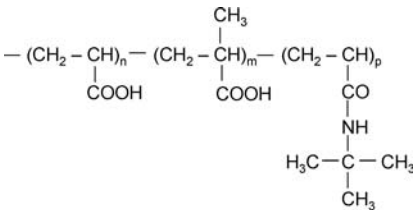
Where:

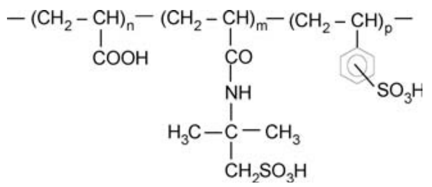
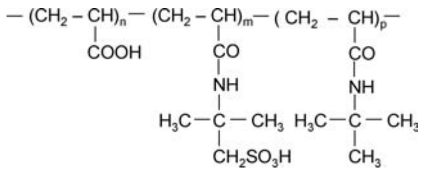
S = Stabilization, %
$[M]_{sample}$ = M concentration in the presence of polymer at 2 or 3 hr.
$[M]_{blank}$ = M concentration in the absence of polymer 2 or 3 hr.
$[M]_{initial}$ = M concentration in the beginning of experiment.

3 Results and Discussion

3.1 Iron ion stabilization

Using the experimental procedure outlined above a series of experiments was conducted to evaluate the performance of polymers as iron stabilizing additives. The experiments were designed to test the efficacy of polymers as a function of polymer dosage, polymerization solvent, and polymer architecture. Table 1 lists the polymers tested in the present study.

Inhibitor	Structure	Acronym	Mol. Wt.
poly(acrylic acid)^{o}	 $-(CH_2-\underset{COOH}{CH})_n-$	H1	~2000
poly(acrylic acid)^{o}	 $-(CH_2-\underset{COOH}{CH})_n-$	H2	~5000
poly(acrylic acid)^{w}	 $-(CH_2-\underset{COOH}{CH})_n-$	H3	~2000
poly(acrylic acid)^{w}	 $-(CH_2-\underset{COOH}{CH})_n-$	H4	~6000
poly(methacrylic acid)	 $-(CH_2-\overset{CH_3}{\underset{COOH}{CH}})_n-$	H5	~5000
poly(maleic acid)	 $-(\underset{COOH}{CH}-\underset{COOH}{CH})_n-$	H6	~1000
poly(2-acrylamido-2-methylpropane sulfonic acid)	 $-(CH_2-\underset{CO-NH-C(CH_3)_2-CH_2SO_3H}{CH})_n-$	H7	~7000
poly(acrylic acid:vinyl acetate)	 $-(CH_2-\underset{COOH}{CH})_n-(CH_2-\underset{OCOCH_3}{CH})_m-$	C1	~7000
poly(acrylic acid: hydroxypropyl acrylate)	 $-(CH_2-\underset{COOH}{CH})_n-(CH_2-\underset{CO-H_3C-CH-CH_2OH}{CH})_m-$	C2	~7000
poly(acrylic acid:carbitol acrylate)	 $-(CH_2-\underset{COOH}{CH})_n-(CH_2-\underset{COO(CH_2CH_2O)_2CH_2CH_3}{CH})_m-$	C3	~8000
poly(acrylic acid: 2-acrylamido 2-methylpropane sulfonic acid)	 $-(CH_2-\underset{COOH}{CH})_m-(CH_2-\underset{CO-NH-C(CH_3)_2-CH_2SO_3H}{CH})_n-$	C4	<12000
poly(maleic acid:sulfonated styrene)		C5	<10000
poly(acrylic acid:methyacrylic acid: t-butyl acrylamide)	 $-(CH_2-\underset{COOH}{CH})_n-(CH_2-\overset{CH_3}{\underset{COOH}{CH}})_m-(CH_2-\underset{CO-NH-C(CH_3)_3}{CH})_p$	T1	<12000

Inhibitor	Structure	Acronym	Mol. Wt.
poly(acrylic acid:2-acrylamido-2-methylpropane sulfonic acid:sulfonated styrene)		T2	< 15 000
poly(acrylic acid:2-acrylamido-2- methylpropane sulfonic acid:t-butyl acrylamide)		T3	< 15 000

^{o}organic solvent polymerized, ^{w}water polymerized

Table 1 Polymers tested

3.1.1 Polymer dosage

The performance of a homo-polymer of acrylic acid, H1, as Fe^{3+} ions stabilizing additive was investigated by carrying out a series of experiments at varying concentrations of H1. Results presented in Figure 1 show that H3 exhibits poor stabilizing ability at low concentrations. For example, %S values obtained in the presence of 5 ppm and 10 ppm of H1 are 4 % and 20 %, respectively. Figure 1 also shows that increasing the polymer concentration from 10 ppm to 20 ppm results in ~3.5 folds increase in %S value and at 25 ppm concentration almost complete stabilization (> 95 %) is observed.

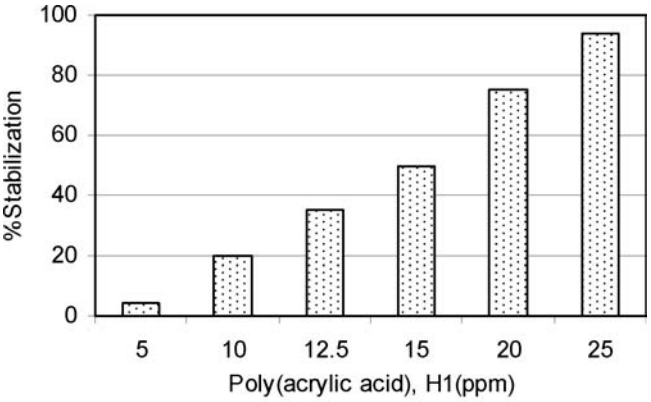
3.1.2 Polymerization solvent

We have previously reported [12–14] that polymerization solvent plays an important role in the performance of poly(acrylic acids), P-AAs, as precipitation agents for sparingly soluble salts and also as dispersants for suspended matter. In addition, we have also reported [15] that P-AAs polymerized in organic solvent are more tolerant to calcium ions than the water polymerized P-AAs of similar molecular weight, MW.

To understand the impact of polymerization solvent, experiments were carried out in the presence of 15 ppm of P-AAs made in different polymerization solvents (organic solvent and water). Results illustrated in Figure 2 clearly show that P-AAs (i.e., H1, H2, MW ~ 2000 and ~ 5000 Da) made in organic solvent exhibit ~ 1.5 times better performance than water polymerized P-AAs (i.e., H3, H4, MW ~ 2000 and ~ 6000 Da). The observed difference in performance for P-AAs made in different polymerization solvents may be attributed to the end group and/or branching. It is worth noting that the influence of polymerization solvent as noted in the present investigation has been previously reported for scale inhibition, calcium ion compatibility, and dispersion properties of P-AAs [12–15].

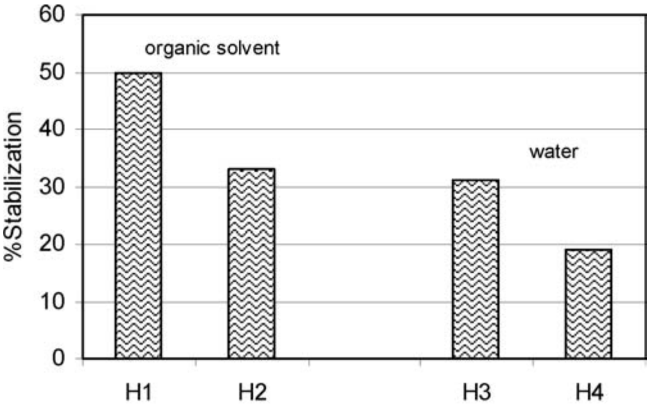
3.2 Polymer architecture

In industrial water treatment formulations various types of additives are used to achieve a variety of benefits (e.g., scale inhibition, chelation, corrosion prevention, dispersancy, etc.). Some commonly used additives include polyphosphates, organophosphonates, homo-polymers of acrylic acid, maleic acids, and acrylic acid/maleic acid-based co- and ter-polymers. Numerous studies on the influence of polymer composition have shown that both the monomer unit ratio and the type of the functional group in the polymer play key roles in on the inhibitory activity of the polymer in terms of preventing the precipitation of scale forming salts. In addition, it has also been demonstrated that nature of the co-monomer exhibits marked inhibitory effect on the kinetics of crystal growth and also on the crystal morphology [16–18].



4 ppm Fe, synthetic tap water, pH 7, 23 °C, 2 hr

Figure 1 Fe^{3+} ions stabilization at various concentrations of H1



4 ppm Fe, synthetic tap water, 15 ppm polymer, pH 7, 23 °C, 2 hr

Figure 2 Fe^{3+} ions stabilization by P-AAs polymerized in different solvents

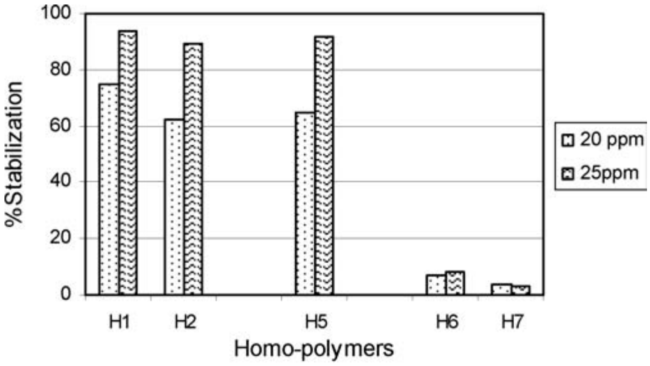
3.2.1 Homo-polymers

To understand the influence of functional groups, a series of experiments were carried out in the presence 20 ppm of homo-polymers. The stabilization data presented in Figure 3 show that performance of homo-polymers strongly depends on polymer functional group. For example, %S values obtained in the presence 20 ppm polymers containing –COOH group i. e., poly(acrylic acid, H2), poly(methacrylic acid, H5) and poly(maleic acid, H6) are 62 %, 65 % and 4 %, respectively compared to 7 % obtained for sulfonic acid (SO_3H) containing polymer (H7). As indicated in Figure 3 further increases in polymer concentration (i. e., 20 ppm to 25 ppm) exhibits negligible influence on the performance of H6 and H7. The poor performance shown by H6 (containing two –COOH groups attached to adjacent carbon atoms) and H7 (containing SO_3H group) may be attributed to polymer MW (~ 1000 Da for H7), and lack of iron-sulfonic acid interaction in the case of H7.

3.2.2 Co and ter-polymers

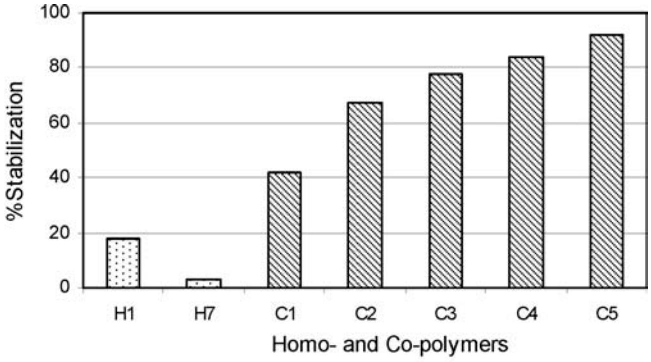
The influence of substituting the carboxyl group with other groups of varying chain length and ionic charge (i. e., vinyl acetate, hydroxypropyl acrylate, carbitol acrylate, and 2-acrylamido-2-methylpropane sulfonic acid) was investigated for their ability to stabilize Fe^{3+} in the presence of 10 ppm of polymer concentration. Results presented in Figure 4 clearly show that, compared to poly(acrylic acid), H1, the co-polymers exhibits superior performance as Fe^{3+} stabilizing agents. For example, %S values obtained for poly(acrylic acid), H1; poly(acrylic acid : vinyl acetate, C1; poly(acrylic acid : hydroxypropyl acrylate, C-2); poly(acrylic acid : carbitol acrylate, C3; acrylic acid : 2-acrylamido2-methylpropane sulfonic acid, C4; and maleic acid : sulfonated styrene, C5 are 42 %, 67 %, 78 %, 84 % and 92 %, respectively. It is worth noting that where as homo-polymers containing COOH group (H1) and SO_3H group (H7) show mediocre to poor performance, co-polymers (i. e., C4 and C5) containing both these functional groups exhibit excellent iron stabilization property. It should also be pointed out that among the two co-polymers containing sulfonic acid groups i. e., C4 and C5, the polymer with higher ionic charge (i. e., C5) performs better as iron stabilizing additive. Thus, for these co-polymers both factors i. e., anionic character and the monomer size, play important roles in imparting Fe^{3+} stabilizing power to the polymer.

Figure 5 presents Fe^{3+} stabilization data for three ter-polymers containing different functional groups (i. e., COOH, SO_3H, $–CH_3$, OC–NH–). It is evident from Figure 5 that incorporation of bulkier group i. e., tertiary butyl acrylamide (i. e., ter-polymers T1, T3) and tertiary butyl acrylamide and sulfonic acid groups (i. e., T2 and T3) significantly improves the performance of ter-polymers. For comparison, performance data on homo- and co-polymers collected in the presence of 7.5 ppm polymers are also shown in Figure 5. Based on the stabilization data, the overall ranking in terms of decreasing effectiveness of the polymers is: ter-polymer > co-polymer > homo-polymer.



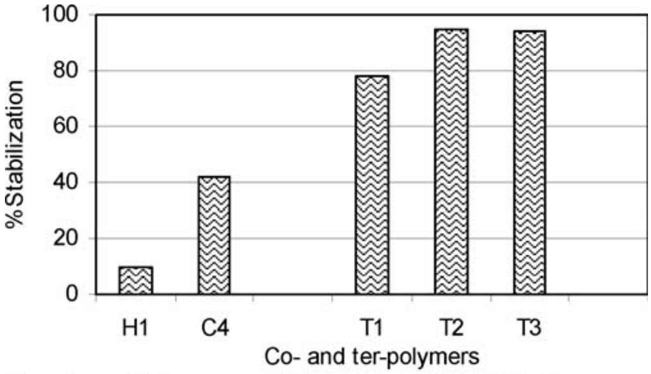
4 ppm Fe, synthetic tapwater, 20 ppm polymer, pH 7, 23 °C, 2 hr

Figure 3 Fe^{3+} ions stabilization by homo-polymers



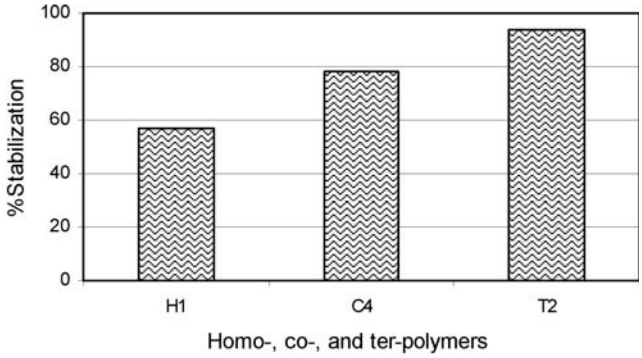
4 ppm Fe, synthetic tap water, 10 ppm polymer, pH 7, 23 °C, 2 hr

Figure 4 Fe^{3+} ions stabilization by homo- and co-polymers



4 ppm Fe, synthetic tap water, 7.5 ppm polymer pH 7, 23 °C, 2 hr

Figure 5 Fe^{3+} ions stabilization by homo-, co-and ter-polymers



3 ppm Mn, 10 ppm Cl_2, pH 7.50, 40 °C, 2 ppm polymer, 3 hr

Figure 6 Mn^{4+} ions stabilization by homo-, co-, and ter-polymers

3.3 Manganese ions stabilization

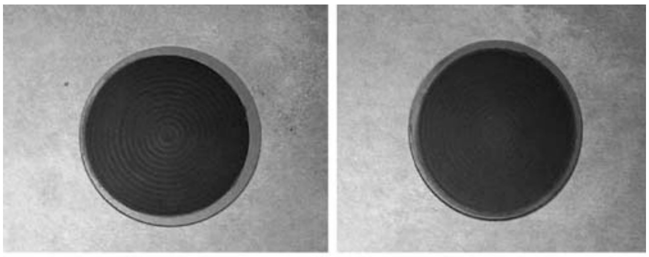
The efficacy of polymers to stabilize Mn^{4+} ions was evaluated by carrying out series of experiments in the absence and presence of variety of polymers as shown in Table 1. As noted in Table 1, the polymers vary significantly both in terms of composition, molecular weight, and functional groups. The polymers were evaluated at various dosages and also as a function of polymer composition.

3.3.1 Homo, co-, and ter-polymers

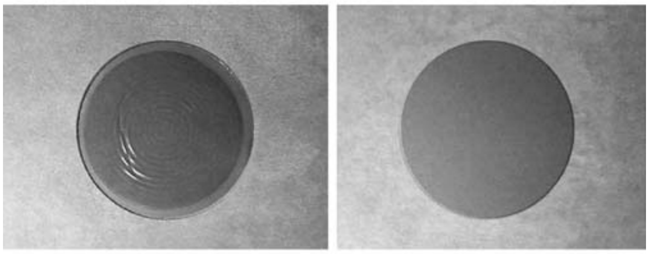
The performance of polymers containing different functional groups was investigated by carrying out stabilization experiments under similar experimental conditions. The data presented in Figure 6 clearly show that the incorporation of SO_3H containing monomer significantly improves the performance of C4 over H1. It is worth noting that incorporation of another SO_3H containing monomer (i. e., sulfonated styrene, SS) in C4 results in further improvement in the performance of C4. For example, %S value obtained for T2 (acrylic acid : 2-acrylamido-2-methylpropane sulfonic acid) is 94 % compared to 78 % obtained for C4. It is evident from Figure 6 that all polymers stabilize Mn^{4+} ions but to a varying degree. Interestingly, among all the polymers evaluated, ter-polymer exhibits the best performance in stabilizing Mn^{4+} ions in aqueous solutions.

3.4 Effect of polymers on iron hydroxide particles stabilization

The influence of polymeric and non-polymeric agents on iron hydroxide particles was investigated. Figure 7 shows optical micrographs of iron hydroxide particles collected on the filter papers at the end of experiments for homo-polymer (H1), co-polymer (C4), and ter-polymer (T2). As shown, T2 is an effective Fe^{3+} ions stabilizer (the darker the yellow color, the more iron hydroxide precipitated from solution).



No Polymer Poly(acrylic acid), H1



Co-polymer (C4) Ter-polymer (T2)

Figure 7 Fe $^{3+}$ ions stabilization in the presence and absence of polymers

4 Summary

Minimizing the precipitation of iron and manganese oxide/hydroxides in aqueous systems is strongly dependent on the polymer dosage and polymer architecture. Preventing the precipitation of iron and manganese oxides/hydroxides plays an important role in determining whether a polymer will be effective in aqueous systems. Mono functional polymers i. e., poly(acrylic acid) are ineffective in stabilizing iron ions but show poor to mediocre performance for manganese ions. Incorporation of acidic and/or bulkier non-ionic group into acrylic acid-based homo-polymer significantly improves the performance of co- and ter-polymers as iron stabilizing additives. Thus, to control metal ions fouling, the incorporation of an effective polymer (i. e., T2) into the HI&I and water treatment formulations can ensure better overall system performance.

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