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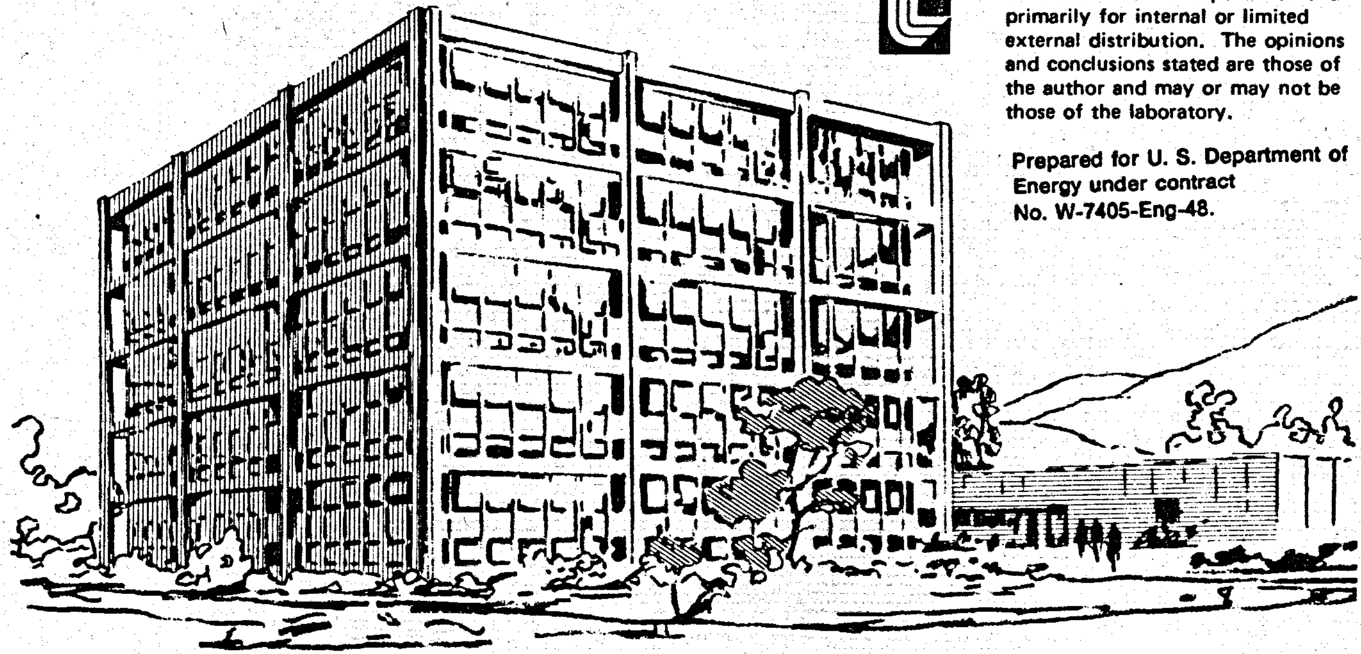
ON-LINE TESTS OF ORGANIC ADDITIVES FOR THE INHIBITION OF THE PRECIPITATION OF SILICA FROM HYPERSALINE GEOTHERMAL BRINE II. TESTS OF NITROGEN-CONTAINING COMPOUNDS, SILANES, AND ADDITIONAL ETHOXYLATED COMPOUNDS.

J. E. Harrar, F. E. Locke, C. H. Otto, Jr., L. E. Lorensen and W. P. Frey

JUNE 1, 1979



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Contents

Abstract

Several new classes of organic compounds have been screened as potential geothermal scale control agents by examining their effect on the precipitation of silica from Magmamax No.1 brine. The substances were tested using the Lawrence Livermore Laboratory Brine Treatment Test System at the Niland, California, Test Site. Solutions of the test substances were injected into flowing brine at 210°C, the brine was flashed to 125°C, and then the kinetics of solids and silica precipitation from effluent brine held at 90°C were measured. Three new types of compounds were shown to have activity as precipitation inhibitors: polyethylene imines, polyethyloxazalines, and quaternary ammonium compounds containing polyoxyethylene. Among the latter, Ethoquad 18/25, which is methyl-polyoxyethylene(15) octadecylammonium chloride, is the leading candidate antiscalant. It is a more powerful inhibitor of silica precipitation than the pure polyoxyethylene polymers, and it apparently has no high temperature solubility limitations. Measurements were made of the concentrations of monomeric silica and the effect of addition of inhibitor at various points in the Brine Treatment Test System. Five different silane compounds showed no activity toward silica.

Introduction

This report is a description of further results of our field test program to evaluate organic chemical compounds for the abatement of scale formed from the geothermal brines of the Salton Sea Geothermal Field. These tests are a continuation of our investigation previously described,[1] in which a series of hydroxyethyl celluloses, other ethoxylated compounds, and surfactants were examined as inhibitors for the precipitation of silica. Proprietary additive mixtures have also been subjected to scaling rate measurements in an earlier series of tests.[2]

The precipitation tests of the ethoxylated or ethylene oxide compounds[1] clearly showed that the polyoxyethylene ($-CH_2CH_2O-$) moiety strongly interacts with the colloidal silica in the hypersaline ($pH \approx 6$) brine at temperatures below 100°C. One specific compound, Carbowax 14,000 (Union Carbide Corporation), which is essentially pure polyoxyethylene polymer of molecular weight 14,000, emerged as the most powerful inhibitor of silica precipitation in the 90°C incubation tests. This compound, at a concentration of 20 ppm in the brine, also retarded the formation of the soft, snowflake-like scale that forms at effluent brine temperatures below 100°C. However, it was apparent in a short plant test that scale reduction at 125°C, and probably higher, would be minimal. In fact, it appeared that the polyoxyethylene may itself deposit or tend to increase the volume of scale at the 125°C point of measurement. This was attributed to the known lowered solubility of the polyoxyethylene polymer at higher temperatures.[3] The polyoxyethylene may still be bound to the colloidal silica, but its diminished solubility may cause it to be incorporated as an inactive agglomerate into the matrix of scale as it is formed.

Another way of viewing the failure of the polyoxyethylene to retard the formation of the higher temperature silica scale is in terms of the current understanding of the steric stabilization of colloidal systesm.[4,5] The mechanism of the inhibition of the precipitation of the silica by the polyoxyethylene probably involves the adsorption of the polymer molecules on the surfaces of the colloidal particles to prevent or retard their aggregation. Our incubation tests at 90°C entail holding the supersaturated brine in an unstirred, stagnant condition.

Ideally, the stabilizing molecules anchor themselves to the particles in such a way that their surfaces are covered with a layer that is compatible with, or soluble in, the dispersion medium. The particles thus remain dispersed and do not flocculate. In the case of the colloidal silica in geothermal brine, the particles themselves are hydrophilic, thus there is a delicate balance between the interaction of the polymer with the particle surfaces and with the aqueous medium. In the case of the polyoxyethylenes, the anchoring and the stabilizing (solubilizing) moiety are one and the same. This makes it vulnerable to "displacement flocculation," which is the dislodgement of the adsorbed polymers by Brownian-motion collisions of the particles, leading to aggregation on flocculation.[4] Stirring, or the existence of solution flow, increases the collision rate, the rate of flocculation, and the rate of scaling of an appropriate surface.

To obtain maximum colloidal stability, especially in the presence of solution velocity, a molecule is required that has a strongly anchoring functional group that is highly specific for silica, together with a solubilizing moiety that is soluble in the highly saline brine at high temperatures. It should be noted that we have not completely discounted the possibility that an additive may be found that controls scale without stabilizing colloidal silica. But we have chosen the experimental route via the precipitation tests as being the one most likely to lead to the most effective antiscalant.

Additives Tested

For the May, 1979, test series discussed here, several types of compounds were selected in an attempt to meet the criteria of higher solubility and stronger bonding between the additive and silica. These are listed in Table 1. The first group, the quaternary ammonium compounds, have ionic (cationic) character, thus are inherently more soluble than nonionic substances in polar, aqueous media. Several of those selected also incorporate polyoxyethylene which, it was thought, would convey activity toward silica. Stronger interaction between the cationic additives and the negatively charged colloidal silica was also envisioned as a possibility.

Table 1. Organic Compounds Tested as Inhibitors of the Precipitation of Silica from Hypersaline Geothermal Brine.

Trade Name	Manufacturer	Chemical Name and Other Characteristics[a]
Quaternary Ammonium Compounds:		
Ethoquad 18/25	Armak Chemicals	Methyl-polyoxyethylene(15) octadecylammonium chloride
Hyamine 1622	Rohm and Haas	Di-isobutyl-phenoxyethoxyethyl-dimethyl benzyl-ammonium chloride
Q-C-50[b]	Tomah Products	Methyl-polyoxyethylene(50) cocoammonium chloride
Variquat K-1215	Sherex Chemical	Methyl-polyoxyethylene(15) cocoammonium chloride
Varstat 66	Sherex Chemical	Methyl-polyoxyethylene(15) tallowammonium ethyl sulfate
Hipochem ADN	High Point Chemical	Methyl-polyoxyethylene(15) tallowammonium chloride
Quat 188	Dow chemical	N-(3-chloro-2-hydroxypropyl) trimethyl ammonium chloride
Emcol CC-36	Witco Chemical	Methyl-diethyl-polyoxypropylene ammonium chloride, M.W. = 1600
Other Ethoxylated Compounds:		
Carbowax 14,000	Union Carbide	Polyoxyethylene, M.W. = 14,000
Lipal 200C	PVO International	Polyoxyethylene(200) ricinoleic acid triglyceride
Ethomeen 18/25	Armak Chemicals	Polyoxyethylene(15) octadecylamine
CMHEC-420L	Hercules	Carboxylated hydroxyethylcellulose, M.S. = 4.2., M.W. ≈ 85,000
Tetronic 1504	BASF - Wyandotte	Polyoxyethylene-polyoxypropylene block copolymer
Jeffox FF-200	Jefferson Chemical	Polyoxyethylene-polyoxypropylene random copolymer

Table 1. (Continued)

Trade Name	Manufacturer	Chemical Name and Other Characteristics
Silanes:		
B-2014-25[b]	Dow Corning	Carboxy-functional amino-siliconate
B-2080-182[b]	Dow Corning	Phosphonate-functional siliconate
A-1160	Union Carbide	Urea-functional triethoxysilane
Z-6020	Dow Corning	3-(2-aminoethylamino)-propyl trimethoxysilane
Z-6071	Dow Corning	Phenyltrimethoxysilane
Other Compounds:		
Corcat P-200	Cordova Chemical	Polyethylene imine, M.W. = 20,000
Corcat P-145	Cordova Chemical	Polyethylene imine, M.W. = 40,000
XD-8779.00[b]	Dow Chemical	Polyethyloxazline, M.W. = 60,000
XD-8779.00[b]	Dow Chemical	Polyethyloxazaline, M.W. = 100,000
XD-30469.00[b]	Dow Chemical	1:1 copolymer of acrylic acid and hydroxyethyl acrylate
Darex 41s	Dearborn	Proprietary carboxylated polyelectrolyte
Alkaphos Q.S.	Alkaril Chemicals	Proprietary amphoteric coco-imidazoline derivative
Alkateric 2CIB	Alkaril Chemicals	Proprietary free acid of complex organic phosphate ester

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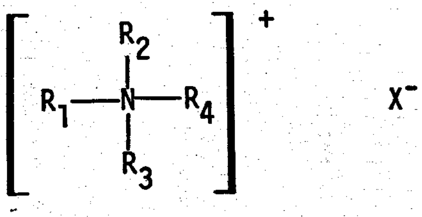
[a] Number in parentheses denotes the number of moles of ethylene oxide per molecule of compound.
[b] Experimental chemical; not commercially available in large quantities.

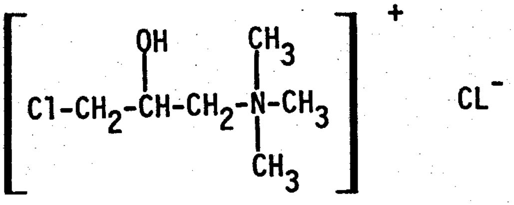
The quaternary ammonium compounds have the generalized structure



$$\left[R_1 - \overset{\overset{R_2}{|}}{\underset{\underset{R_3}{|}}{N}} - R_4 \right]^+ \qquad X^-$$

where the R's are various organic groups, and X^- is the companion anion (usually chloride). In five of the quaternary ammonium compounds tested (see Table 1), one of the R groups is methyl, and two of the R groups are approximately equal-length chains of polyoxyethylene. The fourth R group is a hydrocarbon chain derived from a fatty acid; since it is hydrophobic, it lends a surfactant character to the molecule. The hydrocarbon chain on Ethoquad 18/25 contains 18 carbon atoms (derived from stearic acid, a saturated fatty acid). The "coco-ammonium" compounds are derived from coconut oil, which is predominantly lauric acid (12-carbon), also a saturated fatty acid. The "tallow-ammonium" compounds involve mostly oleic (18-carbon) unsaturated fatty acid.

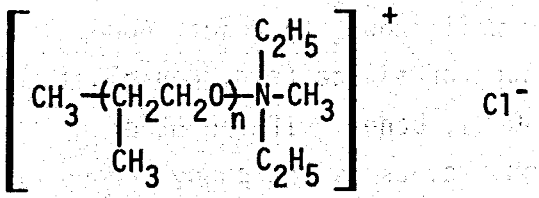
The structures of the other quaternary ammonium compounds are considerably different. Quat 188 is



$$\left[\mathrm{Cl}-CH_2-\overset{\overset{OH}{|}}{CH}-CH_2-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{N}}-CH_3 \right]^+ \qquad \mathrm{CL}^-$$

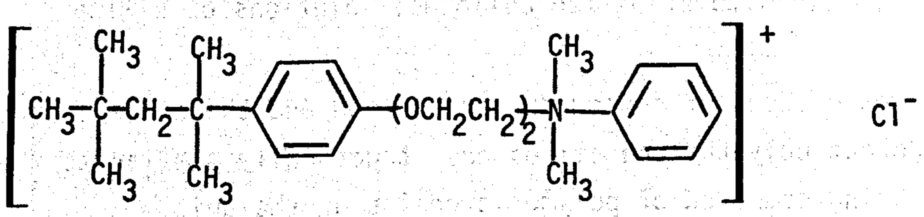
a low-molecular weight compound compared to the others tested.

Emcol CC-36 is



$$\left[CH_3-(\underset{CH_3}{CH_2}CH_2O)_n-\overset{C_2H_5}{\underset{C_2H_5}{N}}-CH_3 \right]^+ \quad Cl^-$$

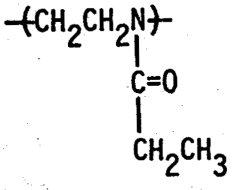
where n ≈ 25 polymeric units of polyoxypropylene. Unlike polyoxyethylene, polyoxypropylene chains are not water soluble, thus this molecule has considerable hydrophobic character even though ionic. Hyamine 1622 is



The second group of substances tested are all high in polyoxyethylene content and are related in various ways to those previously tested.[1] Carbowax 14,000 was again tested. Lipal 200C is a fatty acid derivative containing 200 moles of ethylene oxide. Ethomeen 18/25 is the tertiary amine analog of Ethoquad 18/25 and is very similar to the Ethomeen C/25 tested previously[6] with acidified brine and found to be a good precipitation inhibitor. The compound CMHEC-420L is similar to the previously tested[1] hydroxyethyl cellulose, CMHEC-420H, the former being the low molecular weight version. Tetronic 1504 and Jeffox FF-200 are both medium molecular weight (12,500 and 10,000, respectively) copolymers of polyoxyethylene and polyoxypropylene. As in the Emcol CC-36, the polyoxypropylene moiety, like the hydrocarbon chains of the Lipal 200C and Ethomeen 18/25, is nonpolar and hydrophobic, thus the ethylene oxide-propylene oxide block combination results in a surface-active compound. Tetronic 1504 contains ~40% ethylene oxide, Jeffox FF-200 ~75%; they differ principally because Tetronic 1504 contains large, continuous blocks of ethylene oxide, whereas in Jeffox FF-200, the ethylene oxide is randomly distributed.

The third group of compounds tested (Table 1) comprise five different silanes. These compounds as a class are well known[7] to react, under certain conditions, with the silanol groups of silica (e.g., on glass and silica gel) to form strong siloxane, Si-O-Si, bonds. If the other functional group of the silane is hydrophobic, as is the phenyl group of the Z-6071 compound, the surface "silanized" by such a reaction with the silane is rendered hydrophobic. The first four of the silanes listed in Table 1 were chosen because they have hydrophilic, water-soluble functionality. In the hoped-for reaction with colloidal silica, they could thus provide both the strong anchor bond and the solubilizing moiety. Two such compounds, which are experimental silanes obtained from Dow Corning, have been shown under laboratory conditions to stabilize colloidal solutions of silica.[8]

Several other new classes of compounds were tested and these are listed in Table 1. The Corcat polyethylene imines are structurally similar to the polyoxyethylenes, being composed of polymeric chains of the unit $-CH_2CH_2NH-$; however, they differ in containing considerable branching, and they advantageouly lack the inverse solubility/temperature characteristic of the polyoxyethylenes. Another new polymer tested is the polyethyloxazaline, composed of units of



$$\begin{array}{l} -\!(\mathrm{CH_2CH_2N})\!- \\ \qquad\qquad\quad | \\ \qquad\qquad\quad \mathrm{C{=}O} \\ \qquad\qquad\quad | \\ \qquad\qquad\quad \mathrm{CH_2CH_3} \end{array}$$

Experimental Technique

The additives were tested with Magmamax No. 1 brine using the apparatus and technique described previously.[1] The additives were injected into the separated, 210°C brine, and after collection at the atmospheric-flash temperature of 105°C, the kinetics of the precipitation of silica were measured by anaerobic incubation of the brine at 90°C. As before, the suspended solids were measured by filtration and the silica remaining in solution was measured by atomic absorption spectrophotometry at 0.5-, 1-, and occasionally 2-hour intervals after collection.

Measurement of the monomeric silica in the brine at various points of the Brine Treatment Test System was performed by means of a Beckman Model DB spectrophotometer.

Results and Discussion

The ranges of the characteristics of the brine during the experiments were as follows (effluent brine, flashed to atmospheric pressure):

Chloride concentration:	3.5 - 3.9 Mol/liter
pH:	5.8 - 6.1
Density at 25°C:	1.146 - 1.157 g/cm^3
SiO_2 Concentration:	443 - 486 mg/kg

LLL was again the sole user of the well, but the brine was somewhat more saline than before.[1]

Table 2 summarizes the results of the measurements of the suspended solids and silica in the incubated brines after treatment with the various additives. Only the 1-hour incubation data are presented for brevity; the 30-minute data were always consistent with those at 1 hour. Also shown for comparison in Table 2 are data obtained in the present test series during brine acidification experiments.

Table 2. Concentration of Suspended Solids in Brine and Percentage of Silica Retained in Filtrate after Incubation for 1 hour at 90°C. Magmamax No. 1 Brine (3.5-3.9 Mol/l Chloride). 20 ppm Additive Concentration.

Additive	Concentration of Suspended Solids, mg/kg	Silica Retained, %
Control, no additive	285	48
Quaternary Ammonium Compounds:		
Ethoquad 18/25	55, 53	96, 92
Hyamine 1622	81	85
Q-C-50	97	85
Variquat K-1215	95	81
Varstat 66	121	75
Hipochem ADN	238	53
Quat 188	294	46
Emcol CC-36	275	52
Other Ethoxylated Compounds:		
Carbowax 14,000	102	89
Lipal 200C	111	73
Ethomeen 18/25	67	95
CMHEC-420L	150	70
Tetronic 1504	276	51
Jeffox FF-200	193	62

Table 2. (Continued)

Additive	Concentration of Suspended Solids, mg/kg	Silica Retained, %
Silanes:		
B-2014-25	273	54
B-2080-182	293	51
A-1160	288, 286[a]	53, 47[a]
Z-6020 (40 ppm)	265[a]	58[a]
Z-6071 (40 ppm)	277[a]	55[a]
Other Compounds:		
Corcat P-200	105	88
Corcat P-145	176	80
XD-8779.00	244	60
XD-8779.00	267	46
XD-30469.00	302	46
Darex 41s	284	45
Alkaphos Q.S.	279	49
Alkateric 2CIB	280	50
Acid:		
pH 4.3	4	96
pH 5.0	11	97

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[a] Additive solution in methanol

Among the organic additives tested, those most effective as inhibitors of silica precipitation were Ethoquad 18/25, Ethomeen 18/25, Carbowax 14,000, Corcat P-200, Tomah Q-C-50, and Hyamine 1622. Carbowax 14,000 did not perform quite as well as previously,[1] perhaps because of the greater brine salinity. In any event, it was not as effective as the Ethoquad 18/25 and Ethomeen 18/25. Corcat P-200, the polyethylene imine, appeared equal in activity to Carbowax 14,000, and the data suggest that a still lower molecular weight polyethylene imine might show still greater activity. The high activity of Ethomeen C/25 tested previously,[1,6] together with the present data, show clearly that nitrogen-based, cationic substances and/or those contining polyoxyethylene interact most strongly with the colloidal silica.

The superiority of the Ethoquad 18/25 compared to some of the other substances tested is also illustrated in Figures 1 and 2. The additional polyoxyethylene in Tomah Q-C-50 renders it more effective than the otherwise similar Variquat K-1215; perhaps a 50-mole Ethoquad analog would be still more effective than Ethoquad 18/25. The high activity of the Hyamine 1622 is surprising in view of its minimal amount of the polyoxyethylene moiety. Hyamine 1622-electrolyte solutions are among those known to exhibit the phenomenon of coacervation.[9]

As described above, the quaternary ammonium compounds Quat 188 and Emcol CC-36 are structurally quite different from the other compounds in this group. They do not contain polyoxyethylene, thus this may be the reason for their lack of activity as dispersants. However, like two other additives, Alkateric 2CIB and Darex 41s, they appeared on the basis of the 30-minute incubation measurements, to accelerate slightly -- rather than retard -- the precipitation rates. The possibility to enhance precipitation always exists with such substances that interact strongly with a colloidal system, and this fact is a source of concern in testing of this sort. Polymeric substances, especially those of higher molecular weight and size, may act as bridges between particles thus causing flocculation; and this may occur in a polymer concentration range below that at which the polymer acts as a stabilizing agent.[10]

FIGURE 1

INHIBITION OF FORMATION OF SUSPENDED SOLIDS FROM GEOTHERMAL BRINE BY ORGANIC ADDITIVES

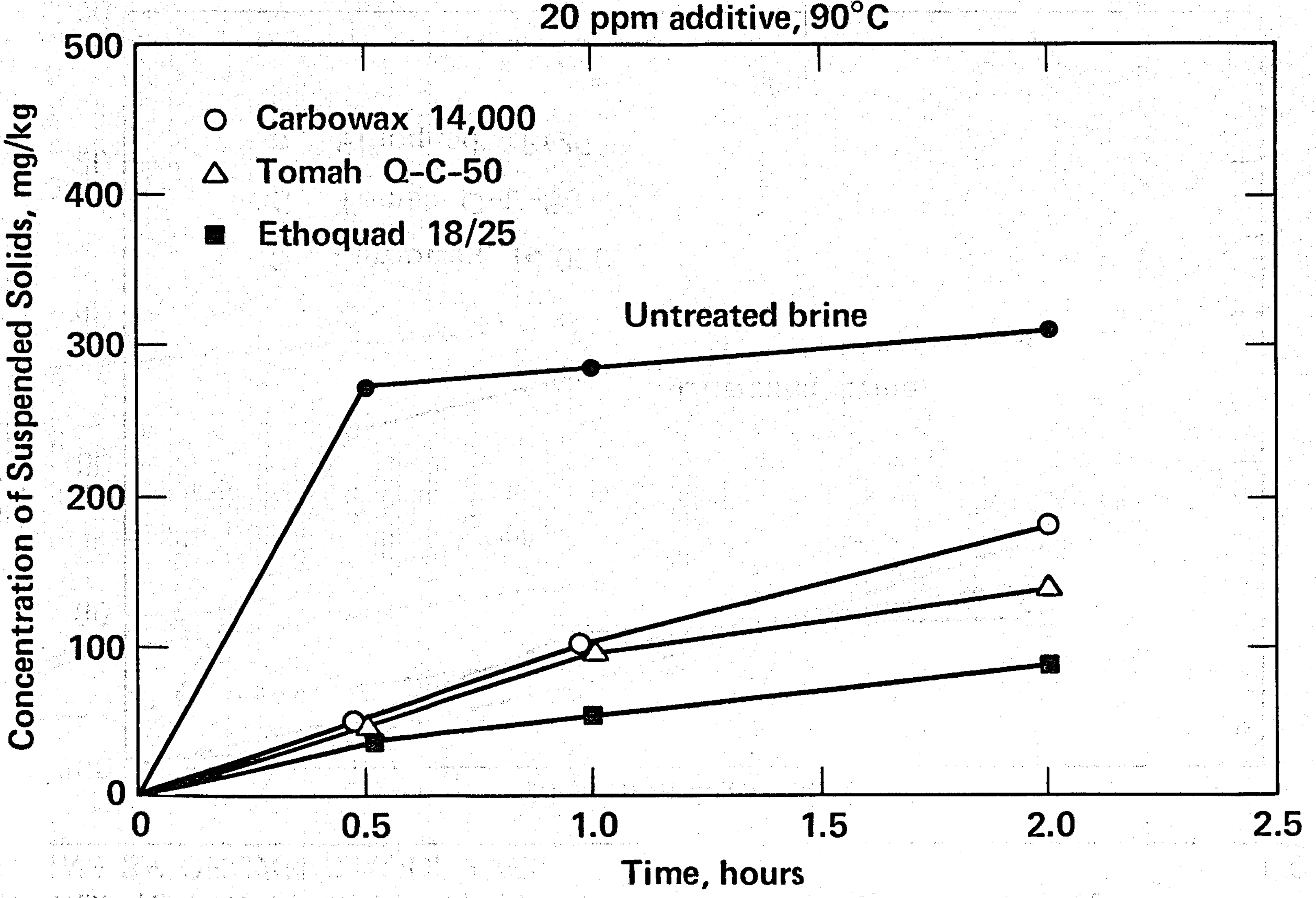
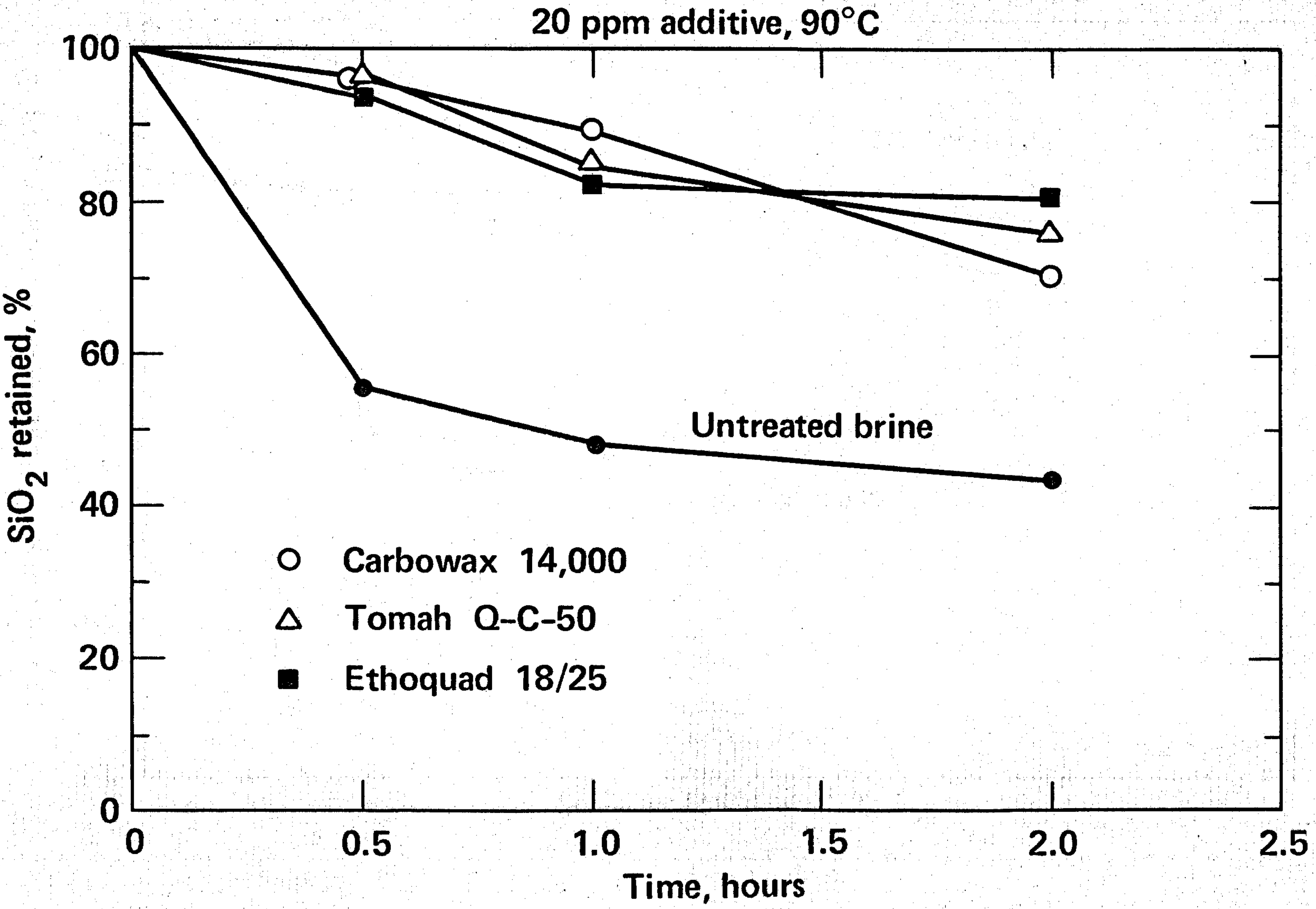


FIGURE 2

INHIBITION OF PRECIPITATION OF SILICA FROM GEOTHERMAL BRINE BY ORGANIC ADDITIVES



The lower molecular weight (60,000) polyethyloxazaline (XD-8779.00) also showed slight activity as an inhibitor. Still lower moleuclar weight versions (in the 10,000 to 20,000 range found to be best for the polyoxyethylenes and polyethylene imines) would probably be better. For these types of polymeric chains, this molecular size seems to be an optimum match for the sizes of the colloidal silica particles with which we are dealing.

The results for the other ethoxylated compounds also showed some interesting effects of additive structure on activity. The CMHEC-420L was no better than the corresponding higher molecular weight compounds tested previously.[1] However, both have anionic character, and the results obtained here for the acrylate-type, carboxylate, and phosphate ester additives, as well as most of the proprietary additives,[2] have consistently shown that anionic surfactants and polymers exhibit very little activity as precipitation inhibitors. These findings agree with a published report[11] that carboxylated polymers do not adsorb on quartz in the pH range of 5 to 8.5.

It is clear from some of the previous measurements[1] and the present results for Lipal 200C, Tetronic 1504, and Jeffox FF-200 that increasing the hydrophobic fraction of a polyoxyethylene-containing molecule decreases its activity. The large fraction of polyoxypropylene (~60%) in Tetronic 1504 completely neutralizes the effect of the polyoxyethylene. Jeffox FF-200 is higher in polyoxyethylene (~75%) but the diluting polyoxypropylene is also randomly distributed, which may diminish its influence. Surfactant character, derived from having both hydrophobic and hydrophilic portions in the same block molecule, generally does not appear to be a useful feature for a compound for stabilizing colloidal silica in geothermal brine.

As can be seen in the data of Table 2, none of the silanes had a measurable effect on the precipitation of silica, even though several were injected as methanolic solutions to prevent hydrolysis before injection. Assuming that our failure to detect a reaction means that none occurred, the lack of a reaction may have been due to the unfavorable pH of the brine,[8] and/or insufficient time in the system for reaction to take place. To allow more time, one experiment was performed in which silane Z-6020 was injected as the methanolic solution into the input of the first-stage separator. This is not the normal point of addition because the brine/steam two-phase flow rates, and hence the concentration of additive, cannot be as accurately measured here. However, we estimated from the accumulation of effluent fluid, and the estimated percentage of steam flashed, that the flow rate was 50 gpm, and the silane was metered accordingly to achieve a concentration of 20 ppm in the brine. Still there was no evidence of a reaction. In future work, a silane will be tested that has a higher probability of reaction at the brine pH of ~6.

The effect of the point of addition of the most effective inhibitor Ethoquad 18/25 was also briefly investigated and the results are listed in Table 3. In the Brine Treatment Test Sytesm, the residence times are mainly those in the two separators. At the usual flowrate of 7-gpm/channel, and 50 gpm overall, the residence time in the first-stage separator (at ~210^{o}C) is ~42 seconds, and that in the second-stage separator (125^{o}C) is ~2.3 minutes. The data of Table 3 show that, compared to the normal point of addition between the first and second stages, there is a decrease in effectiveness of the inhibitor when injected into the second-stage separator, and possibly a slight increase when injected into the input of the first-stage separator, although the latter differences are within the usual measurement uncertainties. In any event, the results show that the Ethoquad 18/25 is quite resistant to the temperature/time conditions at the front end of our system, and could be injected there in a two-stage flash system.

To increase our understanding of the nature of the process of silica scaling in our test facility, attempts were made to measure the concentration of monomeric silica at three points in the system. The procedure used was based on that of Marsh et al,[12] which employs the yellow color of the silicomolybdate complex. Potassium permanganate was used to oxidize the ferrous ion in the brine and prevent reduction of the silicomolybdate. The results of these analyses are given in Table 4, along with the total concentration of silica determined by atomic absorption spectroscopy in the usual manner.

Table 3. Effect of Point of Addition on the Performance of Ethoquad 18/25 as a Precipitation Inhibitor. Incubation of Effluent Brine at 90°C. Magmamax No. 1 Brine (3.8 Mol/l Chloride)

	Point of Addition					
	Input to 1st Stage Separator	Between 1st & 2nd Stages	In 2nd Stage Separator	Input to 1st Stage Separator	Between 1st & 2nd Stages	In 2nd Stage Separator
Incubation Time	Concentration of Suspended Solids in Effluent Brine, mg/kg			Concentration of SiO_2 in Filtered Effluent Brine, mg/kg		
0	0	0	0	481	481	481
30 min.	45	45	43	463	454	444
1 hr.	54	53	71	422	444	407
2 hr.	77	90		422	389	

Table 4. Concentration of Monomeric Silica in Untreated Brine in Scale Control Test System

Sample Location	Total SiO_2 mg/kg	Monomeric SiO_2 mg/kg	Percentage of Monomeric SiO_2 %
Output of 1st Stage Separator 210°C	455	388	85
Output of 2nd Stage Separator 125°C	468	424	90
Output of Delay Stage 85°C	468	333	71

Rather surprisingly, most of the silica appears to be still in the monomeric form even at the 125°C point of scaling rate measurement. This result is important because it suggests that the 125°C scale may be formed chiefly from monomeric silica. This in turn is a potential difficulty for additives that can inhibit silica precipitation only by adsorption on relatively large polymeric silica particles. Inhibition by adsorption on particles should be, and has, thus far, been more effective on the scales formed at the output of the delay stage where the brine has a higher concentration of particles. Experiments designed to investigate these ideas are being planned for the future.

Based on the investigations carried out thus far, the leading candidate for the reduction of scaling from the hypersaline brine is Ethoquad 18/25, and it will soon be subjected to a complete scaling rate test by the techniques previously described.[2] This compound has other potential advantages as an antiscalant. At ambient temperatures it is a liquid which could be injected neat*; and it also may have some activity as a corrosion inhibitor, which should further reduce the scaling of corroding surfaces.

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* Although potentially useful as antiscalants because of their activity as precipitation inhibitors, the hydroxyethyl celluloses now appear impractical because they cannot be prepared as low-viscosity high-concentration aqueous solutions.

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