

in a washing machine. Instead, a small "slug" of surfactant solution must push oil ahead of it as it moves through the reservoir rock. To do that efficiently, the surfactant slug and the "drive" fluid which follows it must be provided with controlled "mobility." Although the mobility of a fluid flowing through rock is a function of both its viscosity and the "permeability" of the rock to that fluid, the concept of mobility control can be grasped by considering only viscosity.

If we attempt to push a liquid through a rock with another liquid of lower viscosity, the driving liquid will tend to channel or "finger" through the driven liquid. On the other hand, if the viscosity of the driving liquid is higher than the viscosity of the driven liquid, fingering will be negligible and displacement of the first liquid by the second will be much more uniform and complete. Therefore, for efficient surfactant flooding, the viscosity of the surfactant solution must be higher than the effective viscosity of the oil-water bank it is displacing, and the viscosity of the drive must be higher than the viscosity of the surfactant solution.

Mobility control in surfactant flooding is achieved almost exclusively with water-soluble polymers, hence the name "micellar-polymer flooding." (Surfactant concentrations in the slug always are above the critical micelle concentration.) Frequently, the surfactant slug and the microemulsions formed in the reservoir, as the surfactant slug mixes with crude oil and formation brine, are viscous enough to require no additional mobility control; but the drive always requires mobility control to be efficient. The most beneficial advance that could be made today in EOR by surfactant flooding

would be a more reliable, less expensive way to control mobility of the drive. Possibly, surfactants will play a role in filling the need for better drives; surfactant-stabilized foams are being studied for that purpose.

Since the viscosity of supercritical carbon dioxide is even lower than the viscosity of water, mobility control can increase the efficiency of enhanced oil recovery by carbon dioxide flooding. Again, surfactant-stabilized carbon dioxide foams and emulsions are being studied.

The furthest advanced application of foams in enhanced oil recovery, now in the field development stage, is in steam flooding. Not only is the viscosity of steam low, but its density also is low. Consequently, steam fingers and also overrides the crude oil and brine in the reservoir. Steam foam produces a more uniform displacement on both counts.

Surfactants that are suitable for EOR by steam foam must be thermally and hydrolytically stable. Temperatures as high as 300-400 F (150-200 C) are common in reservoirs under steam flood. Leading surfactant contenders are alkylbenzene sulfonates and α -olefin sulfonates.

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The Use of Inorganic Sacrificial Agents in Combination with Surfactants in Enhanced Oil Recovery

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ABSTRACT

Current literature on optimization of surfactants in enhanced oil recovery is summarized. Effectiveness of the use of surfactants in chemical EOR processes is dependent on many factors. Uncontrollable factors such as reservoir parameters, mineralogy, and the nature of the crude oil influence the choice of a chemical process. Each reservoir offers a different set of problems to be solved. When the use of a surfactant is warranted, one attempts to optimize further the activity of this surfactant by modifying the chemistry of the reservoir system. Cost aside, maintenance of optimal surfactant activity is essential to minimize the oil/water interfacial tension. Also, loss of surfactant activity due to adsorption on substrate material is particularly disadvantageous because the water wet nature of the rock may be decreased. The use of alkaline, weak acid anions, such as sodium silicate, phosphate and carbonate to enhance surfactant effectiveness has been studied. These sacrificial agents can reduce the hardness (divalent cation) activity of the solution and compete with surfactant for active sites on the reservoir rock surface. Core flood results show that there is an inverse correlation between surfactant retention in the core and residual oil recovery. They also suggest

that surfactants may be recovered for reinjection by the optimal use of sacrificial agents—in particular, the sodium silicates.

INTRODUCTION

Attention turns increasingly toward enhanced oil recovery (EOR) methods as the petroleum yield per effort of drilling decreases (1) (Fig. 1) and as production using conventional methods declines (2) (Fig. 2). The original flow of oil from the reservoir in primary and secondary recovery is less than perfect because of inefficiencies in fluid flow characteristics (physical displacement/sweep) and/or the chemical displacement of oil by the contacting fluids.

The fluid flow characteristics of oil are governed by reservoir permeability and porosity, oil viscosity and pressure gradient factors. For oil in contact with displacing fluids, the displacement efficiency is generally enhanced by reducing the ratio of the viscosities of the oil and displacing fluid, reducing the density of oil, reducing the oil/fluid interfacial

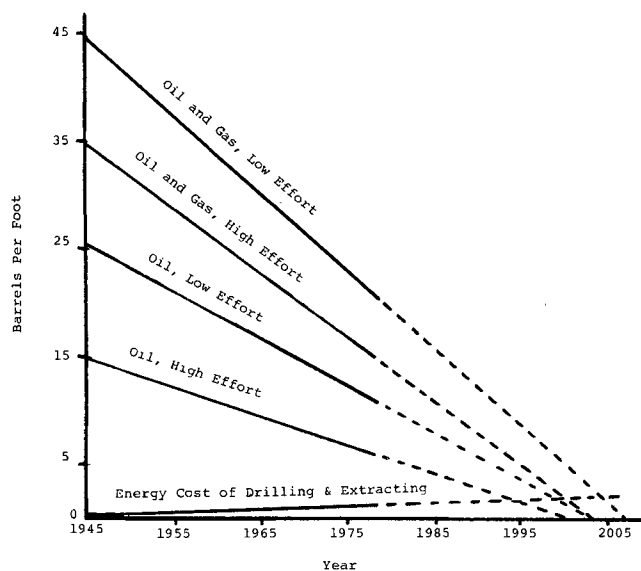


FIG. 1. Energy gains and energy costs of petroleum exploration and development for the U.S. (1).

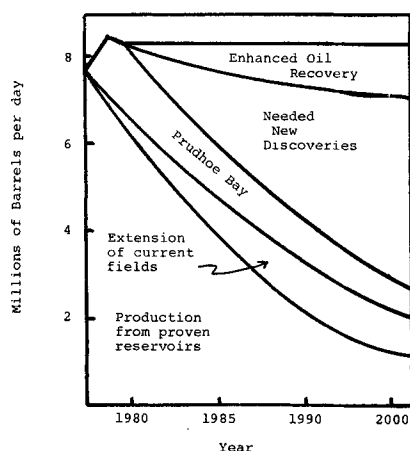


FIG. 2. Distribution of oil sources required to maintain present level of production in the U.S. (2).

tension and maintaining a water wet reservoir substrate. Excluding waterflooding, the various other methods of enhanced recovery attempt to improve sweep and/or displacement efficiency. Table I is a summary of concepts of EOR (3). Of particular interest are the chemical flooding methods, and especially those using surfactants, i.e., alkaline flooding (in situ surfactant formation), surfactant waterflooding and microemulsion flooding (4). Using these methods, one can reduce the oil/water interfacial tension to levels (ca. 10^{-3} mN/m) that release immobilized oil and allow oil bank formation. However, it has generally been observed that the critical factors limiting the successful application of methods using surfactants is their dispersion and retention within the reservoir (5) and reservoir heterogeneity (6).

Surfactants for EOR

It is generally agreed (7,8) that an effective surfactant for EOR, in addition to being capable of reducing oil/water

IFT and being relatively nonadsorbent, should be inexpensive, stable and soluble in reservoir brine at reservoir temperatures and should possess a favorable oil/brine partition coefficient. Anionic surfactants have been of primary interest in EOR due to their relatively low cost; of these, the petroleum sulfonates (PS) have been studied most (4,5). These materials which are made by sulfonating crude oil are quite complex and difficult to characterize (9); however, experience has shown that petroleum sulfonates have been developed with an appropriate distribution of molecular species which can be optimized so that there is an inverse correlation between surfactant oil/water interfacial activity and both solubility and adsorption. The more soluble, lower molecular weight species which are not as active are found to "salt in" the more active, high-weight species. The middle-weight species are thought to serve as sacrificial agents (10).

Akstinat (11) has summarized general criteria for surfactant adsorption: (a) amphiphatic surfactants are readily adsorbed on hydrophobic rock surfaces, depending on their structure; (b) the greater the solubility of a surfactant, the smaller is its adsorption (greatest adsorption of surfactant occurs in high-salinity water because of diminished solubility); (c) with increasing temperature and viscosity of the solvent, adsorption increases; and (d) with increasing surfactant concentration, adsorption increases. He also points out that ionic surfactants are mostly adsorbed in polymolecular layers and that natural surfactants are three to five times more soluble in water than synthetic ones.

An important concept in designing the optimal surfactant system is optimal salinity (12). Optimal salinity must be determined for each surfactant-containing system. At the optimal salinity, one usually finds concurrently minimal o/w IFT, surfactant retention, emulsion coalescence and emulsion viscosity, maximal oil electrophoretic mobility, and most important, maximal oil recovery. The optimal salinity of a given petroleum sulfonate can be increased by adding an ethoxylated sulfonate. This has the effect of changing the equivalent alkane carbon number (EACN) of the surfactant. The EACN/ n_{min} concept is useful in estimating optimal surfactant lowering of oil/water IFT for a given oil (13). The EACN of an oil is the carbon number of a pure alkane which most closely models its IFT behavior. The value of n_{min} for a surfactant is the carbon number of the alkane yielding the lowest o/w IFT at a given electrolyte concentration, temperature and cosurfactant concentration. By equating EACN and n_{min} , this concept allows an estimate of the best surfactant system for a given crude oil.

TABLE 1

Other Methods of Enhanced Oil Recovery (3)

Basic principle	Methods used
Improvement of sweep efficiency	Polymer solutions Water-gas foam injection
Improvement of displacement efficiency	Miscible fluids (alcohol, LPG or rich gas, dry gas) Surfactants Wettability reversing agents
Improvement of both sweep efficiency and displacement efficiency	Microemulsions (e.g., "Maraflood") Alternating water-gas injection Hot water Steam Carbon dioxide Bacterial action Forward combustion

Surfactant Retention

Having designed the appropriate surfactant system, taking into account constraints which arise as a result of the nature of the crude oil, reservoir parameters and available water, one is still faced with a problem of great concern to anyone seeking a cost-effective process—surfactant retention. This problem had been judged in the earlier literature (14) to be of such magnitude as to preclude serious thought of the economic use of ionic surfactants in EOR; however, the promise available from the use of these surfactants has kept efforts alive in spite of these early admonitions. Hurd (15) studied the factors influencing PS adsorption onto sandstone-type minerals and found that the following variables were important: (a) salinity—positive correlation; (b) alkalinity—adsorption is minimal in pH range 9-12; (c) other adsorbing species, e.g., CO_3^{2-} , STPP—adsorption reduction; (d) sulfonate equivalent weight—positive correlation and specific surface area—positive correlation.

It has also been observed that adsorption increases as the PS concentration increases up to the critical micelle concentration (cmc), where adsorption ceases. The addition of alcohol was found to decrease adsorption (16). Bansal and Shah (17) showed that mixtures of PS and ethoxysulfonates increased the salt and hardness tolerance of the surfactant system. Bae and Petrick (18) found that apparent adsorption is dependent on slug flow rate and they found that the use of Na_2CO_3 as a sacrificial chemical preflush reduced adsorption. Even though the adsorption was found to be partially reversible, it was thought to be of value in maintaining high surfactant activity in the front of the slug. The electrical nature of the mineral surface has also been viewed as a significant factor in surfactant adsorption (19), in addition to surfactant activity in solution and factors which increased surfactant solubility.

Novosad (20) suggests that if "adsorption" levels are greater than 1.2 mg/g for PS in Berea cores, one should consider the possibility that part of the loss is due to unfavorable phase behavior, e.g., surfactant entrapment as a result of precipitation or some other mechanism.

Use of Inorganic Additives

Holm and Robertson (21) studied the use of high-pH caustic and silicate solutions as preflushes and NTA and EDTA as adjunct chemicals to micellar slugs. Their results showed that high-pH sodium silicates were effective preflush agents for micellar/polymer floods in sandstone which contain high salinity brine. Sulfonate/solvent use was found to be minimized when 10-25% PV slugs of Na_4SiO_4 at 0.5% were used as preflushes. Celik and Somasundaran (22) have suggested that it is instructive to review the technology of mineral flotation when seeking to better understand surfactant retention.

In mineral technology (23), three classes of additives are generally described (24), i.e., activators, depressants and deactivators. Activators are usually simple electrolytes (hydrolyzable metal ions) having a charge opposite that of the surfactant. Depressants are, again, simple electrolytes (e.g., weak acid anions) that bear like charges to surfactants and are generally thought to compete for surface adsorption sites, whereas deactivators are species that produce their effect by controlling or limiting the solution activity of activators.

Recent studies by Somasundaran and Hanna (25) compare retention of petroleum sulfonates by oil reservoir rock systems to surface adsorption phenomena controlling mineral flotation. They showed that surfactant adsorption could be reduced by the introduction of alkaline weak acid anions, such as sodium silicate, phosphate or carbonate. Sodium silicate was shown to be the most effective additive of those studied.

It has been generally established that anionic surfactant adsorption is primarily due to electrostatic forces (26), whereas secondary driving forces would appear to be related to surfactant chain-chain and substrate-chain hydrophobic interaction (27-29). The latter interaction should be negligible for primarily water wet, i.e., hydrophilic, surfaces.

The surface charge characteristics of the oxide reservoir materials are largely controlled by the ionic make-up of the aqueous phase in the reservoir. For example, Fuerstenau et al. (30,31) found that iron from the steel grinding mills in mineral recovery plants activates silicate surfaces toward sulfonate. It was observed that Ca^{++} ion at high pH values also activated quartz toward sulfonate.

James and Healy (32) suggested that metal ions keep their hydration shells at the oxide interface, and that the surface charge of a typical oxide mineral in the presence of these hydrolyzable metal ions (activators in mineral technology terms) exhibits three charge reversals:

Charge reversal (CR)	Charge change	Influence
CR 1	+ to -	H^+ and OH^-
CR 2	- to +	Hydrolyzable Me^{++}
CR 2	+ to -	$\text{Me}(\text{OH})_n$

It is apparent that the CR 2 region would be favorable to anionic surfactant adsorption; thus, metal ions that affect this type of reversal would act as activators for adsorption. Since many potential activator ions (e.g., Ca^{++} , Mg^{++} , Fe^{+++}) are ubiquitous in nature, especially in oil field brines, additives should be available to minimize these unwanted surface charge characteristics. This can be accomplished as suggested earlier by providing competition for the active sites or sequestering the activator.

It has been shown (33,34) that certain anions, e.g., silicates, orthosilicates, phosphates, pyrophosphates, tripolyphosphates, selenites or fluorides, are adsorbed specifically at the oxide/solution interface. Once adsorbed, these anions render the surface more negative and are relatively difficult to desorb by washing with solutions of nonspecifically adsorbed anions (e.g., Cl^-).

It is also well known that phosphates and carbonates can sequester or precipitate metal ions in solution, thus rendering them inactive. Falcone (35) has shown, through ion electrode studies, that colloidal polysilicate anions are capable of reducing the activities of Ca^{++} and Mg^{++} to a level below those predicted by the metal ion hydrolysis constants. His results suggest that metal ions are sequestered by association with the surface of the colloidal polysilicate anions in a manner analogous to the interaction of metal ions with silica (36). This effect becomes more pronounced as the silicate anions condense to form larger, more complex poly-anions.

Using the above results, Campbell, et al. (37) found that silicates complexed Mg^{++} efficiently in zeolite A-containing detergent systems. Tsai and Falcone (38) demonstrated that activation of oxide-type minerals could be suppressed (i.e., the CR 2 region could be narrowed or eliminated) by the addition of polysilicate anions. The suppression was greater for the more highly polymerized silicates (Fig. 3). It was not determined whether suppression was due to reduction in metal ion activity in solution or to resurfacing of the positively charged oxide by the polysilicate anions.

This summary suggests that common inorganic materials generally classed as surfactant builders should have significant value in EOR in the future.

Laboratory Builder Assessment

The use of Na_2CO_3 , STPP, NaOH and $\text{Na}_2\text{O}:3.22 \text{SiO}_2$ as builders was examined in the low-tension water flood

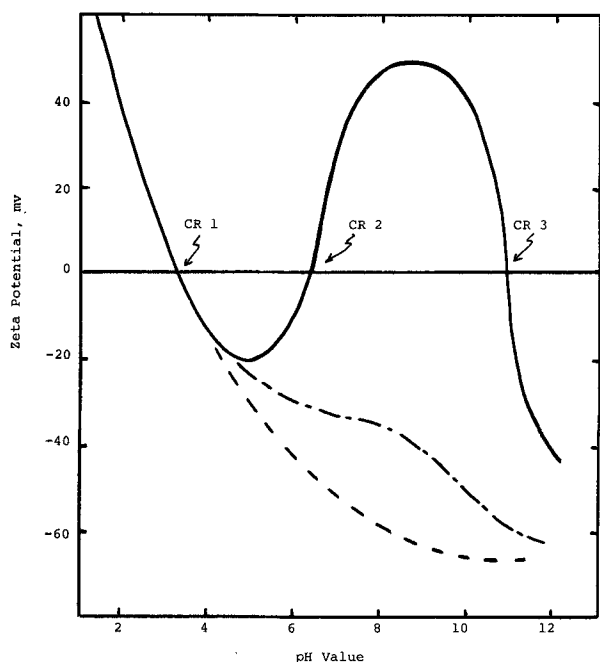


FIG. 3. Zeta potential vs pH value for typical oxide mineral in the presence of: — hydrolyzable metal ion (activator); - - - hydrolyzable metal ion and deactivator, e.g., $\text{Na}_2\text{O}:3.2\text{SiO}_2$; ····· DI water.

(LTWF) of a light, mid-continent crude oil, which had an API gravity of 36.3° and viscosity of 37 centipoises at 26 C, the reservoir temperature (39). A synthetic brine, equivalent to the natural brine, was used for initial saturation of the core and during waterflooding. This brine contained 1190 ppm Ca^{++} , 435 ppm Mg^{++} , and ca. 10% NaCl. Oil recovery after waterflooding and surfactant retention in the Berea core in the absence of oil were studied. The surfactant used was STEPAN's Petrostep® 450 petroleum sulfonate. Experimental details are available in the original paper. Results are shown in Table II.

With no alkali added to the dilute surfactant solution, (Fig. 4) about 0.68 g of surfactant was retained per kg of core material, and hardness levels remained at roughly 200-100 ppm as CaCO_3 . Since so much surfactant is retained in the core, possible wettability changes may occur. It is expected that recovery of oil would be delayed and not dramatic. Sweep in a $\frac{1}{4}$ 5-spot pattern was about normal for this type of low-tension flood.

When sodium hydroxide is added to the surfactant solution, (Fig. 5) there is very little improvement in the amount

of surfactant recovered; however, hardness levels were further depressed.

This indicated that surfactant, as well as appreciable calcium and magnesium hydroxides, were building up in the core and possibly lowering permeability. Recovery was improved only slightly, probably due to the decreased IFT resulting from the high pH value, since very little additional surfactant is available over the case in which no alkali was added.

The addition of sodium carbonate (Fig. 6) results in somewhat reduced surfactant retention (0.26 g of surfactant/kg rock) and lowered elution of hardness ions, but during most of the flood, surfactant levels are less than 50% of the injected concentration. Sodium carbonate will effectively remove Ca^{++} (as CaCO_3); however, Mg^{++} ions are not removed. Therefore many of these ions are still available to deactivate surfactant. Sweep was improved somewhat due to slight permeability reduction and recoveries are higher overall since more surfactant is available and the pH remains high. Most of the carbonate is recovered; therefore, only a minimal amount of permeability reduction can occur.

STPP (Fig. 7) also is very effective at reducing hardness levels of both Ca^{++} and Mg^{++} to less than 10 ppm. STPP effectively sequesters these cations, but may degrade with time and temperature in a reservoir environment. Surfactant retention was correspondingly reduced to about 0.18 g/kg of rock and surfactant effluent levels rose rather quickly to about 0.22% (0.25% injected). Although nearly all of the phosphate anions were detected in the effluent and surfactant retention was reduced, it was noted that permeability was seriously decreased by the STPP. This is difficult to explain, but may be due to either swelling of clays because of very effective ion exchange or possible migration of clays which then block pores. Recovery of the crude oil was excellent because of high surfactant levels which produce optimal IFT.

Sodium silicate (Fig. 8), on the other hand, behaves very differently. Surfactant retention in the core is 0.15 g/kg of sandstone, with effluent concentrations quickly reaching those of the injected solutions. Hardness levels in the effluent are greatly reduced to less than 10 ppm. Appreciable amounts of silicate anions are removed from solution. The silicate anions can react with multivalent metal ions in solution to form either colloidal particles with these ions on the surface or precipitates at the rock and clay surfaces. The latter could trap these ions or slow the ion exchange process, rendering the surfaces less attractive to surfactant molecules. Multivalent metal ions trapped in the clays or at the rock surfaces would not be mobile and free to block pores downstream, but might reduce permeability. Therefore, the action of a silicate builder could be much more effective in controlling these ions so that the surfactant can do its job while beneficial permeability is maintained. Due

TABLE II

Summary of Dynamic Retention Study

Alkali	Hardness, as CaCO_3 (ppm)	Surfactant retention, (g/kg core)	anion consumption	Residual oil recovery (%)
None	100-300	0.68	—	37.6
0.367% NaOH	20	0.65	Very little	37.9
0.367% Na_2CO_3	10-20	0.26 ^a	Very little	54.5
0.367% STPP	<10	0.18	Little	62.3
0.367% $\text{Na}_2\text{O}:3.2\text{SiO}_2$	<10	0.15	80%	69.6

^aMuch of it eluted by the 0.1% NaCl postflush.

to the lower IFT and reduced surfactant adsorption, recovery of oil occurs sooner and at a higher rate than with any of the other alkalis. Therefore, a significant portion of the oil can be recovered and permeability is selectively reduced in the areas where the major portion of the injected solution has passed.

This idea is supported by pattern flood core results which indicate that the addition of silicate enhanced the sweep efficiency as shown in Figures 9, 10 and 11. These results provide further support for the ideas of Somasundaran and

Hanna (25) who indicated that silicates showed some promise as a sacrificial agent for actual applications because they reduced sulfonate abstraction under all conditions of pH and ionic strength, probably by decreasing available sites for abstraction and because they could possibly aid in mobility control.

In general, studies on optimization of the surfactants in EOR show that the analogy to detergency is well documented and suggests strongly the value of the use of inorganic sacrificial agents. The results from several laboratories where

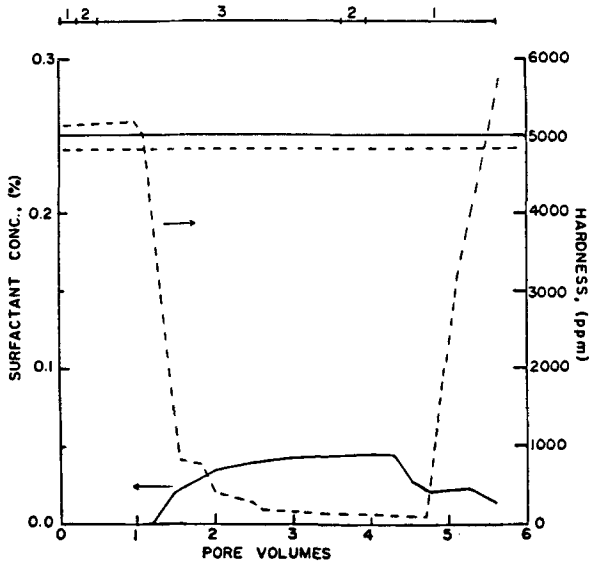


FIG. 4. Effluent surfactant and hardness concentrations in a saline only and dilute surfactant LTWF. 1 = Connate water (4,800 ppm hardness as CaCO_3); 2 = saline preflush/postflush; 3 = dilute surfactant slug.

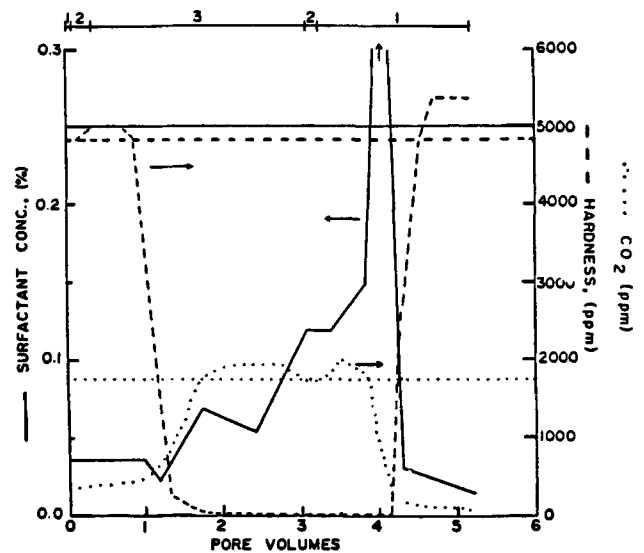


FIG. 6. Effluent surfactant, carbonate and hardness concentrations in a sodium carbonate enhanced dilute surfactant LTWF. 1 = Connate water (4,800 ppm hardness as CaCO_3); 2 = saline preflush/postflush; 3 = dilute surfactant slug.

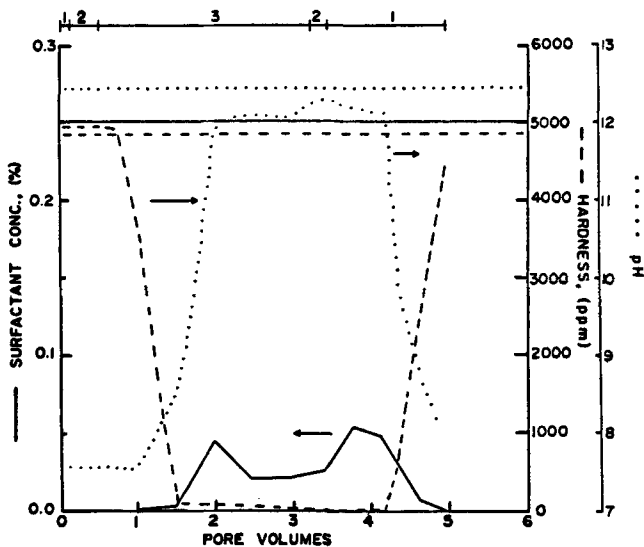


FIG. 5. Effluent surfactant, pH value and hardness concentrations in a sodium hydroxide enhanced dilute surfactant LTWF. 1 = Connate water (4,800 ppm hardness as CaCO_3); 2 = saline preflush/postflush; 3 = dilute surfactant slug.

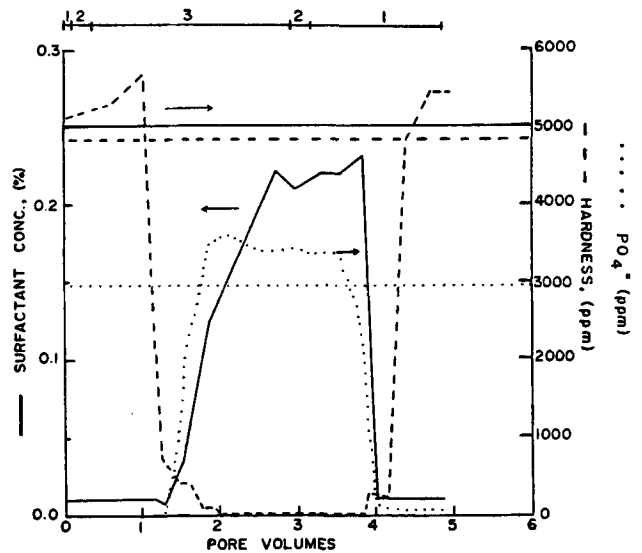


FIG. 7. Effluent surfactant, phosphate and hardness concentrations in a STPP enhanced dilute surfactant LTWF. 1 = Connate water (4,800 ppm hardness as CaCO_3); 2 = saline preflush/postflush; 3 = dilute surfactant slug.

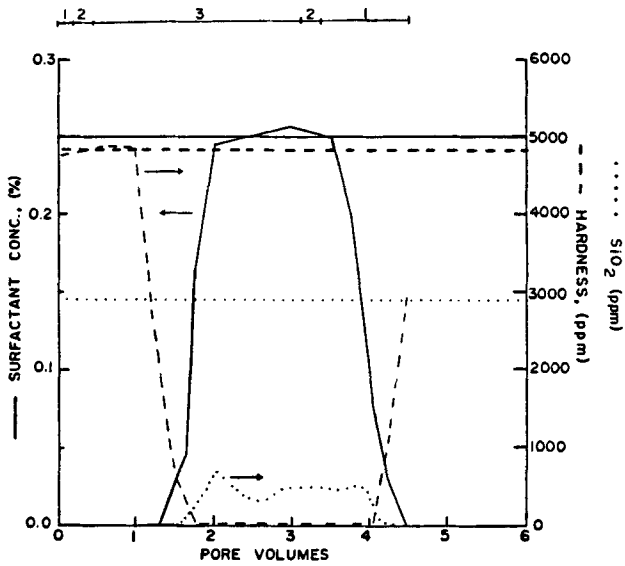


FIG. 8. Effluent surfactant, silicate and hardness concentrations in a sodium silicate enhanced dilute surfactant LTWF. 1 = Connate water (4,800 ppm hardness as CaCO_3); 2 = saline preflush/postflush; 3 = dilute surfactant slug.

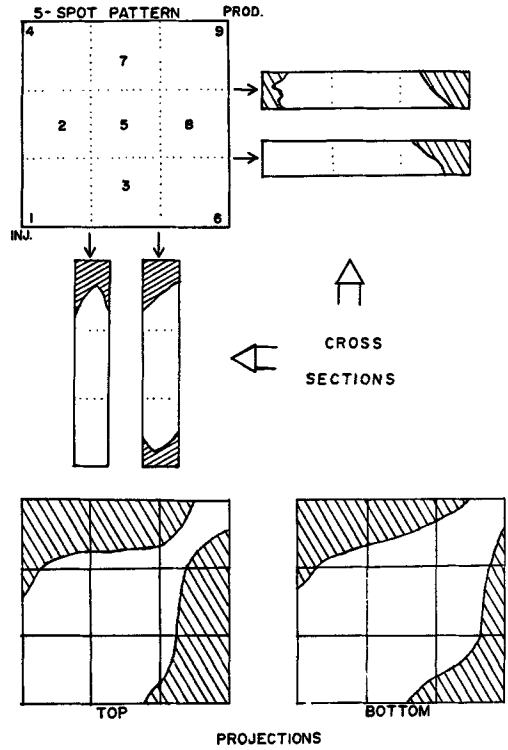


FIG. 10. Projections of the radial sweep patterns using a sodium carbonate enhanced dilute surfactant LTWF.

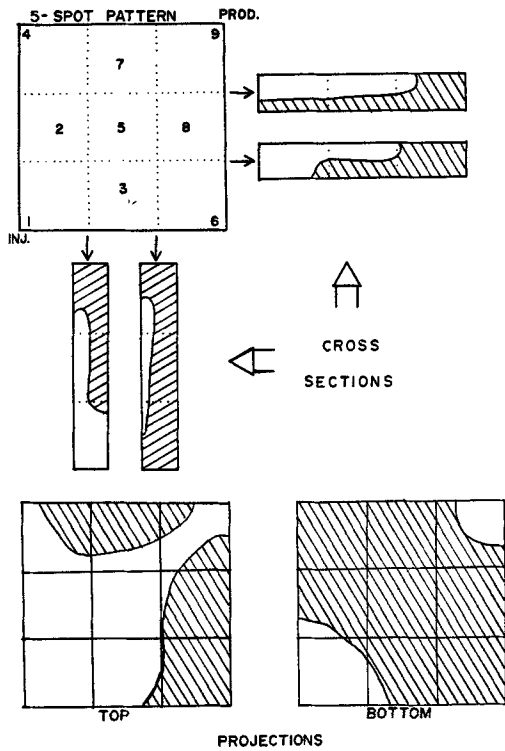


FIG. 9. Projection of the radial sweep patterns using a dilute surfactant LTWF.

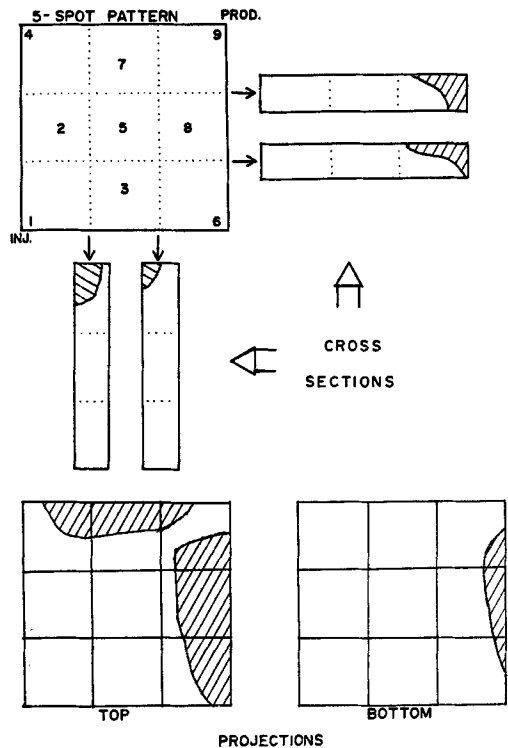


FIG. 11. Projections of the radial sweep patterns using a sodium silicate enhanced dilute surfactant LTWF.

different builders have been compared invariably show that the soluble silicates are of most promise as active additives, either as preflush and/or direct additives in the surfactant slug. Factors which are suggested as favorably influenced by using sodium silicate are: oil/water emulsion viscosity, interfacial tension, surfactant retention, sweep efficiency, early breakthrough and oil recovery, and chemical interaction.

These values for the soluble silicates are not surprising considering their hydrophilicity; their tendency to bind irreversibly to oxide minerals; their association with multivalent metal ions and their general value in detergency. Saubestre (40) states that "... of all inorganic builders, all silicates as a class are the best buffering agents, and the best wetting, emulsifying and deflocculation agents."

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