Low-Cost Foam Surfactant from Wood Pulping By-Products

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ABSTRACT
We report the results of laboratory experiments designed to evaluate the performance of the sodium salt of tall oil acid, TOA, as a foam surfactant. The objective of the experiments was to determine if this inexpensive by-product could be used to reduce the chemical costs of the foam mobility control process. Tall oil acid is a commercially available blend of oleic and linoleic carboxylic acids obtained by distillation of the wastes from pine pulping mills. The sodium salt soap, TOA\textsuperscript{Na}, is easily produced by one-step neutralization of TOA with sodium hydroxide.

The performance of TOA\textsuperscript{Na} was measured in terms of the rate and magnitude at which the foam pressure gradient, $\nabla p$, increased when TOA\textsuperscript{Na} and nitrogen were injected into a two-foot sandpack. Performance was also measured as a function of temperature, TOA\textsuperscript{Na} concentration, foam liquid volume fraction, foam velocity, salinity, pH, and oil saturation.

Experimental results show that TOA\textsuperscript{Na} is an effective foaming agent that can produce pressure gradients of hundreds of psi per foot in a sandpack. The dominant parameters found to control TOA\textsuperscript{Na} performance were salinity, temperature, and pH. To generate a large $\nabla p$ at high temperature and in the presence of oil, the salinity of the TOA\textsuperscript{Na} solution had to be increased by adding between 1.0 and 1.5 wt% sodium chloride. The TOA\textsuperscript{Na} was effective up to $204^\circ C$, but a large $\nabla p$ could not be produced at $260^\circ C$. The pH of the TOA\textsuperscript{Na} solution had to be greater than about nine to achieve a large $\nabla p$. Sodium sesquicarbonate was used to buffer the solution at a pH of ten. Other notable results include that TOA\textsuperscript{Na} foam performance was not adversely affected by oil and that stable foams were produced at high and low foam velocities and at low liquid volume fractions.

INTRODUCTION
The recovery efficiency of gas injection EOR methods, such as steam, carbon dioxide, and nitrogen, is often low. This is the result of the poor sweep efficiency caused by the low viscosity and density of these gases. Recent field trials have shown that the sweep efficiency of gases can be improved by injecting a surfactant with the gas to generate a foam in the reservoir. Foam improves the oil recovery efficiency of gas flooding by diverting gas to high oil saturation zones. Foam has been used to improve sweep efficiency near the well bore, in cyclic processes, and more in depth, in gas drive processes. The surfactant described in this paper is applicable in any of these processes as long as the surfactant is soluble in the injection water and the formation brine. The focus in this paper is on application in the steam foam process since this represents the severest test of the surfactant. The steam foam process was recently reviewed by Castanier et al. and Hirasaki.\textsuperscript{1,2}

Recent field experience has helped to identify the key factors that influence the success of steam-foam applications.\textsuperscript{3-4} Pertinent reservoir factors include oil saturation, thermal maturity, and reservoir structure and mineralogy. Key surfactant performance factors include thermal stability, brine compatibility, retention, liquid volume fraction (LVF, the ratio of aqueous foam volume to total foam volume), foam velocity, and foam stability to oil.
Laboratory research has provided important information about the foam process. The effects of gas and liquid velocity on foam rheology have been determined at low and high temperatures, and this information has been used to develop simplified models of foam flow. A better understanding of foam-oil interactions has been achieved by correlating measurements of interfacial phenomena with foam flow in porous media and in microvisual cells.

New surfactants have been developed and marketed that are effective at high temperatures and in the presence of oil. These new surfactants, composed mainly of high molecular weight alkylaryl sodium sulfonates, have been used in technically successful steam-foam field tests.

Despite these technical advances in foam chemistry, physics, and mechanics of field application, the steam foam process is not widely used. One reason for this is that the process is not economical due to the high cost of foam surfactants. If the surfactant cost could be substantially reduced, the economics might be improved to where the steam foam process could be used more often.

A search for such a surfactant led us to one of the oldest types known - an alkali carboxylate soap, in particular the soap derived from TOA. Soaps are more susceptible to precipitation by divalent cations than are sulfonate steam-foam surfactants. However, this concern was surpassed by the low cost of the TOA. The current price for the grade of TOA used in this work is $0.12 per pound, in bulk.

Tall oil acid is a relatively pure blend of unsaturated eighteen-carbon carboxylic acids obtained by distillation of wastes from pine pulping mills. The distillate contains mainly oleic and linoleic acids and small amounts of saturated acids and rosin acids. Various grades of TOA are available, which are priced by their oleic and linoleic acid content. The middle-grade TOA used in this work contained about 90% oleic and linoleic acids, 6% rosin acids, and 4% unsaponifiable material. Tall oil acid is an easily-handled liquid at ambient temperature and can be converted into a water soluble soap, TOA\Na, in a simple, one-step neutralization with sodium hydroxide.

The TOA\Na was assumed to be thermally stable, especially in the anaerobic environment of a steamflood. An anaerobic environment is needed to prevent oxidation of the carbon-carbon double bonds in TOA\Na. The pH of the steam's liquid phase decreases as the sodium hydroxide is consumed by reactions with reservoir sand and clays. An inexpensive buffer, sodium sesquicarbonate, was added to the TOA\Na solutions to keep the pH alkaline a greater distance into the reservoir. This buffer, also known as trona ore, is less reactive with reservoir sands. The buffer will also enhance the transport of TOA\Na by removing divalent cations from reservoir sands via ion exchange and precipitation.

We initially used the TOA\Na as an additive with other surfactants. By replacing part of the expensive surfactant with TOA\Na, the chemical cost was reduced. An unexpected benefit was also discovered - the TOA additive often improved the performance of the other surfactants in the presence of oil. While these results were encouraging, the greatest cost reduction would obviously be achieved if TOA\Na could be used separately as a steam-foam surfactant. In this paper we discuss our initial evaluation of TOA\Na as a single-component steam-foam surfactant.

**EXPERIMENTAL**

**Equipment**

A schematic diagram of the coreflood equipment is shown in Figure 1.

![Figure 1. Foam Coreflood Equipment](image)

A stainless steel cell, 3.8 cm diameter x 61.0 cm, was fitted with internal pressure taps at six, twelve, and eighteen inches from the injection sandface. Pressure taps were also located on the injection and...
production lines, near the sandfaces. Water-filled lines were used to connect the pressure taps to Validyne P300D pressure transducers such that differential pressure could be measured from the injection port across 15.24, 30.48, 45.72, and 61 cm of the sandpack. Sand was retained in the cell by screened distribution plates. The cell was placed inside a mechanical convection oven that regulated the temperature to ± 1°C.

Injection of ultra-high purity nitrogen was controlled with a Brooks 5850E mass flow controller. Nitrogen was humidified before entering the core by percolating through deionized water in a stainless steel cylinder that was partially packed with lead shot. Surfactant injection was controlled with an ISCO A500 continuous-flow syringe pump. Foam was generated just upstream of the sandface by mixing nitrogen and TOA'Na* surfactant in a 60-micron sintered metal frit.

Back pressure was maintained on the core with a Mity-mite dome-loaded back-pressure regulator, BPR. To prevent sand fines from entering the BPR, 60-micron metal filters were placed upstream of the BPR. Additional BPRs were used to prevent accidental over-pressurization of the cell and, when required, to maintain back pressure on the humidifier.

Sandpack vp, temperature, and nitrogen rate data were acquired and recorded at one-minute intervals on a PC using Datascan 7010/7020 A/D modules and RTM 3500 software. A separate PC running in-house software was used to control the nitrogen injection rate.

Fluids and Matrix

The TOA used in this work was a commercial product composed of oleic acid, 44%, linoleic acid, 42%, saturated acids, 4%, rosin acids, 6%, and unsaponifiable material, 4%. The acid number was 188. A stock solution of TOA'Na* was prepared by adding aqueous six molar sodium hydroxide, 35.80 g, into a well-stirred solution of TOA, 60.00 g, and distilled water, 304.20 g. The mixture was stirred at ambient temperature for about 30 minutes. The stock solution was clear, contained 16.1 wt% TOA'Na*, and had a pH of 10.0 and a viscosity of 14 cp (7.34 sec⁻¹, 22°C).

Synthetic Kern River softened water, KRSW, was used in all of the experiments. This low salinity brine, 740 mg/L total dissolved solids, contained NaCl, 0.32 g/L, Na₂SO₄, 0.16 g/L, and NaHCO₃, 0.32 g/L. Injection solutions of TOA'Na* were prepared by diluting the TOA'Na* stock solution with KRSW. In most experiments, sodium sesquicarbonate, Na₂CO₃·NaHCO₃·2H₂O, was added to buffer the injected solution, and sodium chloride was added to raise the salinity. Unless noted otherwise, the pH of all injected solutions was adjusted to ten with sodium hydroxide or hydrochloric acid.

The cell was packed with silica sand, F-95 grade from U.S. Silica Company. The sandpack was evacuated and then saturated with KRSW. Average sandpack pore volume, absolute permeability to water, and porosity were 248 mL, 6.2 μm², and 0.35, respectively.

Foam Coreflooding Procedure

The system back pressure was set at either 2.758 or 5.516 MPa for corefloods run at 38 to 204°C or 260°C, respectively. The back pressure was always well above the saturated steam pressure, but not so high that, when combined with a large foam vp, would exceed the pressure limit of the mass flow controller.

In oil-free corefloods, KRSW was displaced with 2 PV of TOA'Na* solution, and then nitrogen and TOA'Na* solution were injected continuously. In oil-containing corefloods, S₀ was established by sequential injection of Kern River crude oil and KRSW. Nitrogen and KRSW were then injected for one hour prior to the continuous injection of nitrogen and TOA'Na* solution.

The mass flow controller and pump settings needed to achieve a particular foam superficial velocity and LVF, at a given pressure and temperature, were calculated as follows. We assumed, and verified experimentally, that the nitrogen was completely humidified before entering the core. This was important because we did not want water to flash in the core, and we wanted to be sure that the calculated water vapor rate was achieved. The water vapor rate is a large fraction of the total gas rate because the equilibrium partial pressure of water vapor is substantial at the initial temperature and pressure used in these corefloods. The pump and mass flow controller settings were calculated by using pressure and liquid density data from a saturated steam table for the given temperature and by assuming ideal gas behavior.

RESULTS

The performance of TOA'Na* was measured in terms of the rate and magnitude at which vp increased when TOA'Na* and nitrogen were injected into a two-foot sandpack. Nitrogen was used instead of steam so that foam velocity and LVF could be accurately set. Since the initial core pressure, foam rate, and LVF are the same within similar sets of corefloods, the rate and magnitude of the vp increase can be used to directly compare the effects of different...
variables on foam performance. Foam performance was determined as a function of numerous variables, including temperature, TOA\(\text{Na}^+\) concentration, foam LVF, foam injection velocity, salinity, pH, and oil saturation. The experimental conditions for each coreflood are listed in Table 1. The coreflood results are shown in Figures 2 to 8, where \(\varphi_p\) across the entire sandpack is plotted vs. time. To keep the figures readable, \(\varphi_p\)'s across shorter sections of the core were not plotted.

**Corefloods Without Oil - Temperature and Salinity Effects**

To determine the initial feasibility of using TOA\(\text{Na}^+\) as a steam-foam surfactant, the first corefloods, No. 1 to 3, were run under favorable conditions. The sandpack was preflushed with TOA\(\text{Na}^+\) solution and contained no oil. The only additive to the TOA\(\text{Na}^+\) solution in these corefloods was a small amount of buffer. As seen in Figure 2, a large \(\varphi_p\) developed rapidly in coreflood No. 1, which was run at a low temperature just to see if a foam could be generated. In coreflood No. 2, the temperature was raised 10°C, which is typical for steam in a shallow California reservoirs. The rate and magnitude of the \(\varphi_p\) response were lower, but still substantial. In coreflood No. 3, the temperature was raised to 400°F, which is typical for steam in deeper, higher pressure wells. There was no \(\varphi_p\) response beyond that observed for injection of KRSW and nitrogen.

The next corefloods were designed to determine if increasing the salinity of the TOA\(\text{Na}^+\) solution would improve foam performance at high temperature. Buffer concentration was raised to 0.56 wt% in these and subsequent corefloods. Addition of 1.0 wt% sodium chloride dramatically improved the \(\varphi_p\) response at 204°C and caused only a slight decrease in the \(\varphi_p\) response at 149°C, as seen in corefloods No. 5 and 4, respectively.

Experiments were conducted to determine the solubility of the 0.53% TOA\(\text{Na}^+\)/0.56% buffer solution at higher salinity. At 98°C and a pH of 10.0, the sodium chloride concentration could be increased to about 4.0 wt% before the surfactant started to precipitate. The solubility would likely be similar, if not better, at higher temperatures.

With this information, other corefloods were run at 260°C in oil-free cores with sodium chloride concentrations ranging from 1.0 to 3.5 wt% and with TOA\(\text{Na}^+\) concentrations up to 2.0 wt%. This temperature is typical of steam used in deep, high pressure reservoirs. The results were not plotted because no substantial \(\varphi_p\) was observed.

**Corefloods With Oil - Temperature and Salinity Effects**

For corefloods run in the presence of oil, a waterflood residual oil saturation was first established, and then nitrogen and KRSW were injected prior to injecting nitrogen and the TOA\(\text{Na}^+\) solution. There was a delay in the \(\varphi_p\) response relative to corefloods No. 1 to 5. About 30 minutes of this delay was due to the time required for the TOA\(\text{Na}^+\) solution to reach the core.

As seen in Figure 3, a nearly identical \(\varphi_p\) response was observed at 149°C when a TOA\(\text{Na}^+\) solution which contained 1.0 wt% sodium chloride was injected into either an oil-free sandpack, No. 6, or an oil-containing sandpack, No. 7. A nitrogen/KRSW prefush was also used in the oil-free coreflood.

The results for corefloods run in the presence of oil at 204°C are shown in Figure 4. The magnitude of the \(\varphi_p\) obtained with 1.0 wt% sodium chloride, No. 8, was about 2.0 MPa, or about one-half of the \(\varphi_p\) in the oil-free case, No. 5. By increasing the sodium chloride concentration to 1.5 wt%, No. 9, the magnitude of the \(\varphi_p\) response increased to a level comparable to that observed in the oil-free case. A slight decrease in the magnitude and the rate of the \(\varphi_p\) response was observed as the sodium chloride concentration was increased to 3.0 wt%, No. 10, and 3.5 wt%, No. 11.
### Table 1
Foam Corefloods - Experimental Conditions

<table>
<thead>
<tr>
<th>Coreflood Number</th>
<th>TOA Na⁺ (wt%)</th>
<th>Buffer Sat B (wt%)</th>
<th>NaCl (wt%)</th>
<th>pH</th>
<th>Temp. (°C)</th>
<th>Sₗₑw C</th>
<th>TOA Na⁺ Preflush</th>
<th>N₂/KRSW D Preflush</th>
<th>N₂ Inj. (scm)</th>
<th>TOA Na⁺ Inj. (mL/min)</th>
<th>Foam Rate E (m/day)</th>
<th>LVF F</th>
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<td>2 PV</td>
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<td>467</td>
<td>0.62</td>
<td>91</td>
<td>0.01</td>
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<tr>
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<td>10.0</td>
<td>149</td>
<td>—</td>
<td>2 PV</td>
<td>—</td>
<td>1097</td>
<td>0.66</td>
<td>91</td>
<td>0.01</td>
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<td>2 PV</td>
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<td>10.0</td>
<td>149</td>
<td>—</td>
<td>1 hr</td>
<td>—</td>
<td>1097</td>
<td>0.66</td>
<td>91</td>
<td>0.01</td>
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<td>10.0</td>
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<td>0.28</td>
<td>—</td>
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<td>0.66</td>
<td>91</td>
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<td>—</td>
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<td>91</td>
<td>0.01</td>
</tr>
<tr>
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<td>10.0</td>
<td>204</td>
<td>—</td>
<td>2 PV</td>
<td>—</td>
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<td>1.45 / 2.90</td>
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<td>—</td>
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<td>—</td>
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<td>—</td>
<td>469 / 313</td>
<td>0.31 / 0.21</td>
<td>91 / 61</td>
<td>0.005</td>
</tr>
</tbody>
</table>

A 2.76 MPa backpressure was used for all floods.
B Na₂CO₃·NaHCO₃·2H₂O
C oil saturation at start of foam, waterflood residual
D Nitrogen and KRSW were injected at the same rate as used during foam injection.
E superficial velocity, calculated at initial core pressure
F volume fraction of liquid in injected foam, calculated at initial core pressure
Corefloods With Oil - TOA\textsuperscript{Na}\textsuperscript{+} Concentration Effects

As seen in Figure 5, the rate and magnitude of the \(vP\) response decreased when the concentration of \(\text{TOA}\textsuperscript{Na}\textsuperscript{+}\) was lowered from 0.53 wt\%, No. 9, to 0.32 wt\%, No. 12. Reductions in the concentration of \(\text{TOA}\textsuperscript{Na}\textsuperscript{+}\) to 0.2% or less, under the same conditions, produced pressure gradients that would randomly fluctuate at values less than 0.2 MPa. This behavior is indicative of a weak foam. These results were not plotted.

Corefloods Without Oil - pH Effects

Experiments were first conducted to qualitatively determine the phase stability of \(\text{TOA}\textsuperscript{Na}\textsuperscript{+}\) solutions as a function of pH. At 98\(^\circ\)C, the pH of a stirred \(\text{TOA}\textsuperscript{Na}\textsuperscript{+}\) solution (0.53 wt\% \(\text{TOA}\textsuperscript{Na}\textsuperscript{+}\), 0.56 wt\% sodium sesquicarbonate, 1.5 wt\% sodium chloride) was slowly reduced by the drop-wise addition of 1N hydrochloric acid. The phase stability of the solution was monitored by observing the focus of a pin-point source of light directed through the solution. The solution remained clear to a pH of 8.5 and remained translucent, without phase separation, to a pH of about 7.6. As seen in Figure 6, No. 13, the rate and magnitude of the \(vP\) response decreased when the pH of the injected solution was lowered from 10.0 to 8.5.

Corefloods Without Oil - Rate and LVF Effects

To determine foam performance at near-well-bore and in-depth rates, corefloods were run at injection rates that were substantially higher and lower those discussed above. The corefloods did not contain oil because the rates were altered in stages; a separate coreflood was not run for each condition. The effect of lowering the LVF was also determined.

As seen in Figure 7, No. 14, the rate and magnitude of the \(vP\) response were high for foam injection rates of 213 and 427 m/day. In Figure 8, the effects of both lower rates and a lower LVF are shown. The magnitude of the \(vP\) decreased as the foam injection rate was lowered from 91 to 15 m/day at an LVF of 0.01, No. 15, but the foam did not collapse. A similar result was obtained when the LVF was decreased to 0.005.
DISCUSSION

The experimental results indicate that TOA\(\text{Na}^+\) is a viable steam-foam surfactant when used within the envelope of performance variables identified in this work. The focus of this work was not to identify or synthesize the best possible steam-foam surfactant. Instead, the objective was to determine if this inexpensive chemical could be used as-is to replace the expensive steam-foam surfactants now in use, and if so, to determine the general limits of its applicability.

The salinity of the TOA\(\text{Na}^+\) solution had to be increased to about 2 wt% (sodium sesquicarbonate plus sodium chloride) to generate a strong foam in the presence of oil, at temperatures up to 204°C. This requirement is not unique to TOA\(\text{Na}^+\), as researchers have previously reported that sodium chloride was needed to stabilize steam foams generated with \(\alpha\)-olefin sulfonate and alkylbenzene sulfonate surfactants.\(^{23}\) Besides stabilizing the foam, the sodium chloride would improve the propagation rate of TOA\(\text{Na}^+\) through the reservoir by speeding the displacement of divalent cations off reservoir clays.\(^{23}\) Numerous steam-foam field projects have been run with up to 4 wt% sodium chloride added to the surfactant.\(^{1,2}\)

A buffer is needed to help maintain the pH of the TOA\(\text{Na}^+\) solution at least above 8.5, preferably 9.5 or higher. Although a TOA\(\text{Na}^+\) solution (0.53 wt% TOA\(\text{Na}^+\), 0.56 wt% sodium sesquicarbonate, and 1.5 wt% sodium chloride) remained stable to a pH of about 7.5, the foam "coreflood performance decreased when the pH was lowered from 10.0 to 8.5. The greater solubility of the TOA\(\text{Na}^+\) at higher pH is at least partially responsible for this behavior. Sodium sesquicarbonate, also called trona ore, is the buffer of choice for this application. Previous work has shown that alkali consumption by reservoir sand is much lower when trona ore is used instead of sodium carbonate or sodium hydroxide.\(^{20,25}\) Also, trona ore speeds surfactant propagation by exchanging and precipitating divalent cations from reservoir clays and forms petroleum soaps by reacting with crude oil.\(^{19,24}\) These soaps may enhance the rate of oil recovery and may help to stabilize the foam.

In contrast to other steam foams, the TOA\(\text{Na}^+\) foams exhibited little sensitivity to oil. The presence of oil did decrease foam stability, as measured by \(\nu_p\), when the sodium chloride concentration was too low. But a slight increase in salinity restored foam stability to an oil-free level.

Very high temperatures, up to 290°C, are encountered in steamflooding deep, high-pressure reservoirs. Repeated attempts were made to generate a strong TOA\(\text{Na}^+\) foam at 260°C. We increased the sodium chloride concentration and raised the TOA\(\text{Na}^+\) concentration, but no substantial pressure gradient could be produced. We ruled out thermal degradation of the TOA\(\text{Na}^+\) as the cause by running a quick experiment that showed the TOA\(\text{Na}^+\) was stable at 260°C for at least the two days it was monitored. The fact that the stability of surfactant foam decreases at higher temperature is well documented. We also found that \(\text{C}_{16-18} \alpha\)-olefin sulfonate with added salt could generate a strong foam at 149-204°C, but not at 260°C. Based on these and other experiments that we have run, we conclude that the TOA\(\text{Na}^+\) is not hydrophobic enough to produce a stable foam at this temperature. The upper temperature limit for TOA\(\text{Na}^+\) steam foam is in the lower range between 149°C and 260°C.

The coreflood results also indicate that TOA\(\text{Na}^+\) should be used at a concentration of 0.5 wt% or higher. A decrease in \(\nu_p\) was observed when the concentration was reduced from 0.53 wt% to 0.32 wt%. All foams become less stable when the surfactant concentration is reduced to low levels. The surfactant concentration needed to generate a stable foam depends on several factors, including surfactant type, oil type, and temperature.

The TOA\(\text{Na}^+\) surfactant generated a stable foam at 204°C over a wide range of velocities and at low LVFs - from 91 to 427 m/day at 0.01 LVF and from 91 to 15 m/day at 0.01 and 0.005 LVF. This shows that TOA\(\text{Na}^+\) can generate a stable foam at near-well-bore and in-depth velocities and at low LVFs.

The experimental results of this initial evaluation show that TOA\(\text{Na}^+\) is an effective steam foam surfactant when properly applied. The positive results include the following. The TOA is an easily handled liquid that can be converted to TOA\(\text{Na}^+\) surfactant in a simple one-step process. The
TOA'Na+ can be supplied as a low viscosity fluid at 16 wt% activity. The TOA'Na+ can generate strong foams at temperatures up to 204°C, without sensitivity to oil, at high and low rates, and at the LVFs normally used in the steam foam process. These results can be achieved by using low-cost salt additives, which have been, and still are, routinely used in steam foam and other improved oil recovery processes.

The greatest advantage of TOA'Na+ is its low cost. We estimate that the chemical cost of TOA'Na+, including the manufacturing cost of neutralization and the cost of the appropriate amounts of sodium sesquicarbonate and sodium chloride, is about $1.00/active kg ($0.45/lb). The manufacturing cost of neutralization accounts for about 46% of this total. A major portion of this cost could be recovered by performing the neutralization on-site, on-the-fly, which would bring the estimated total cost to about $0.70/kg ($0.32/lb). This cost is about one order of magnitude less than that of traditional foam surfactants.

CONCLUSIONS

Based upon the experimental results described in this paper, we conclude the following.

1. TOA'Na+ is an effective foaming surfactant that can produce large pressure gradients in a sandpack.
2. TOA'Na+ foam stability is not sensitive to Kern River crude oil.
3. TOA'Na+ foams are stable at high and low velocity and at low liquid volume fraction.
4. TOA'Na+ can generate stable foams up to 400°F, but not at 500°F.
5. Sodium chloride must be added to TOA'Na+ to generate a stable foam.
6. The pH of the TOA'Na+ solution must be nine or higher to generate a stable foam.
7. The concentration of TOA'Na+ must be about 0.5 wt% or higher to generate a stable foam.
8. The estimated chemical cost of the TOA'Na+ foam system is about one order of magnitude less than that of traditional foam surfactants.

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