

# A laboratory study on foam-enhanced surfactant solution flooding in removing n-pentadecane from contaminated columns

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## Abstract

The efficiency of a foam flooding process in removing n-pentadecane from a contaminated glass-bead column was evaluated by comparing with the results obtained by a surfactant-solution flooding process. Data on the foaming properties and solubilization efficiency of aqueous surfactant solutions were reported. Moreover, an experimental column containing n-pentadecane-contaminated glass beads was used to explore the effects of surfactant concentration and gas–liquid volumetric flow rate ratio on the recovery efficiency of n-pentadecane. It was found that the efficiency of a surfactant solution in recovering n-pentadecane increased with increasing surfactant concentration. The findings also suggest that in this study the surfactant-enhanced emulsification of n-pentadecane played an important role in removing n-pentadecane from the contaminated column. Using foam flooding, the recovery of n-pentadecane was increased significantly compared with what obtained by surfactant-solution flushing, probably due to the reduced channeling flow effect. These data demonstrated that the dramatic improvement in residual n-pentadecane recovery can be realized by the use of foam. In addition, at a constant liquid flow rate, an increase in gas–liquid volumetric flow rate ratio may result in increased total n-pentadecane recovery. © 2000 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

A major problem in the soil or groundwater remediation is the removal of hydrophobic organic compounds. Non-aqueous-phase organic

compounds usually enter the unsaturated zone as discrete liquid phases, which move due to gravitational and capillary forces. Because of the low solubility of hydrophobic organic compounds in water, the residual organic phase usually represents a long-term contamination source for soil and groundwater.

An effective remediation technique for such situations is the surfactant-solution flushing approach [1]. Surfactant-enhanced soil remediation

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has been proposed as an alternative method for recovering residual organic materials from contaminated soils. This technique is primarily based on two processes. First, as the surfactant is dissolved in water, the surface tension of the aqueous phase is greatly decreased and the ability of viscous force to displace the oil is favored [2]. Second, surfactant molecules in an aqueous solution can form micelles, which could increase the apparent solubility of the oil in water [3–8]. Aqueous surfactant solutions have been successfully used to remove absorbed hydrocarbons from soils [1,9–16]. The performance of the remediation by surfactant-solution flooding is expected to be governed by the properties of the surfactant solution and displaced organic compounds, the flow patterns in a reservoir, and the characteristics of the reservoir system.

Because of its dispersed nature, the presence of foam has a profound effect on the mobility of fluid phases flowing in a porous medium [17–19]. Compared with the permeabilities measured in the absence of foam, the effective permeability of the porous medium to each phase is significantly decreased as foam is present [17,20,21]. This improves the mobility ratio and thus the homogeneity of the flood. In addition, due to its relatively low density, foam can easily overcome gravitational effects and flow through most of the regions in a heterogeneous reservoir. Thus, foam can be expected to improve the process efficiency of surfactant-solution flooding in a contaminated reservoir [22–25]. Moreover, foam was also found to hold promise for improving sweep efficiency in oil recovery processes [26,27]. Apparently, the efficiency of foam-enhanced surfactant solution flooding strongly depends upon the surfactant type, the surfactant concentration, and the ratio of gas to liquid in the foam injected.

The specific objective of this study was to assess the efficiency of a foam flooding process in removing oils from a contaminated glass-bead column in comparison with the results obtained by a surfactant-solution flooding process. The data reported herein are for non-ionic splittable Triton SP-series surfactants, mainly for Triton SP-190. Since the surfactant characteristics of the Triton SP-series surfactants can be easily de-

stroyed by adjusting the pH according to the information provided by Union Carbide Corporation, USA [28], which is convenient for later treatments of the contaminated flushing effluent, they are well-suited for soil remediation. N-pentadecane, a saturated hydrocarbon, was selected as the model oil. The foaming properties and solubilization efficiency of aqueous surfactant solutions were reported first. An experimental column containing n-pentadecane-contaminated glass beads was then used to explore the effects of surfactant concentration and the volumetric flow rate ratio of gas to liquid injected in the foam on the n-pentadecane recovery efficiency.

## 2. Experimental

Commercial Triton SP-series surfactants, Triton SP-135, Triton SP-175, and Triton SP-190, were provided by Union Carbide, USA and were used as received. The major component of Triton SP-series surfactants is polyethylene glycol, and the average degrees of ethoxylation are 3.5, 7.5, and 9.0 mol mol<sup>-1</sup> for Triton SP-135, Triton SP-175, and Triton SP-190, respectively. The active content for all three surfactants is reported to be 100 wt.% by the manufacturer. The densities of Triton SP-135, Triton SP-175, and Triton SP-190 at 20°C are 0.9935, 1.0244, and 1.0336 g ml<sup>-1</sup>, respectively, while the hydrophile-lipophile balance (HLB) numbers are 8, 12, and 13, respectively. Triton SP-135 is insoluble in water, and the cloud points for 1% aqueous solution are 38 and 59°C for Triton SP-175 and Triton SP-190, respectively [28]. N-pentadecane was selected as a model compound for contaminants. Research-grade n-pentadecane was obtained from Tokyo Kasei Kogyo Co., Ltd., Japan. All solutions were prepared with purified water that was passed through a Milli-Q plus purification system (Millipore, USA) with a resistivity of 18.2 MΩ cm.

The information about foaming ability and foam stability for aqueous surfactant solutions was obtained based on the measurements of the Ross-Miles method [29]. In a modified Ross-Miles method, a 200-ml aqueous surfactant solution contained in a titration pipette was allowed to fall

45 cm onto 50 ml of the same solution contained in a cylindrical vessel at room temperature. The height of the foam produced in the cylindrical vessel was measured immediately after all the solution has run out of the titration pipette and then measured again after 5 min.

The surface tensions of aqueous surfactant solutions and interfacial tensions between n-pentadecane and aqueous surfactant solutions were measured at 25°C by using a Wilhelmy plate tensiometer (model CBVP-A3, Kyowa Interface Science Co., Ltd., Japan).

The ability of surfactant solutions to improve the apparent solubility of n-pentadecane in water was investigated as follows. In the batch tests, surfactant solutions having concentrations ranging from 100 to 400 ppm by weight were prepared, and 0.5 ml n-pentadecane was added to 30 ml of the surfactant solutions. The solutions were then continuously shaken with a water bath shaker (model SB-303, TKS, Taiwan) at 25°C for 48 h. The sample vials were then allowed to settle for 24 h for phase separation. Portions of the resulting aqueous phases were then withdrawn for analysis of the n-pentadecane concentration by gas chromatography (model GC-14B, Shimadzu, Japan) equipped with a flame ionization detector.

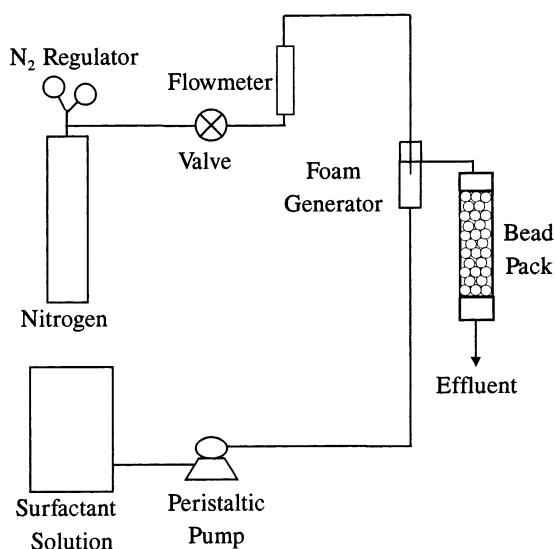


Fig. 1. Schematic diagram of the experimental apparatus for column flushing experiments.

These measurements gave the amounts of n-pentadecane existing in the aqueous surfactant solutions.

A glass column, 10 cm long by 3 cm i.d., was used in the column experiments. Glass beads with an average diameter of 0.1 cm were used as the porous medium. To prepare the glass beads for column experiments, they were mixed with n-pentadecane for 20 min. The mixed sample was then placed in the glass column and was drained with the introduction of nitrogen for 30 min. The amount of residual n-pentadecane in a drained column was determined. The pore volume of the column experiments was estimated about 26.5 cm<sup>3</sup> and the residual n-pentadecane occupied approximately 15% of the pore space. Two separate series of column flushing experiments were conducted, one with a surfactant solution alone and the other with a surfactant solution and foam. For aqueous surfactant-solution flooding experiments, a surfactant solution was introduced through the top of the column and was allowed to flow through the glass beads at a desired volumetric flow rate. For foam-enhanced surfactant-solution flooding experiments, a foam generator, which consisted of a drawn tip that protruded into a surfactant solution-filled chamber, was used. Nitrogen through the tip and the surfactant solution through another port are injected into the chamber at prescribed volumetric flow rates. A schematic diagram of the experimental apparatus for column flushing experiments is shown in Fig. 1. Effluent samples were collected at 30-ml intervals and analyzed for n-pentadecane concentration by gas chromatography. Sonication was required with effluent samples in order to ascertain a homogenous dispersion solution was analyzed.

### 3. Results and discussion

#### 3.1. Foaming ability and foam stability

The results of the modified Ross–Miles test for aqueous surfactant solutions are plotted in Fig. 2. It can be seen that Triton SP-175 or Triton SP-190 surfactant possesses a much better foaming ability than Triton SP-135. The poor foaming

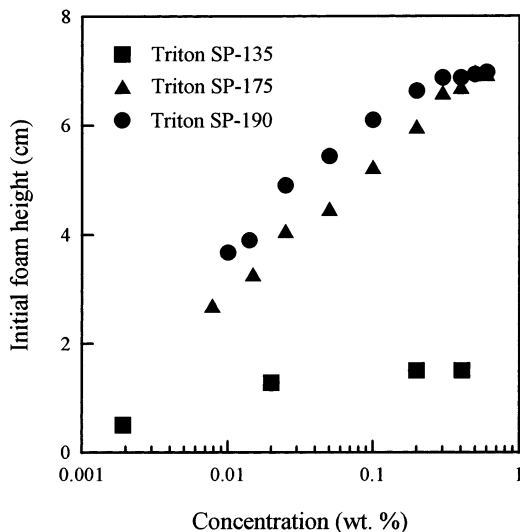


Fig. 2. Initial foam heights of aqueous Triton SP-series surfactant solutions measured by using a modified Ross–Miles method.

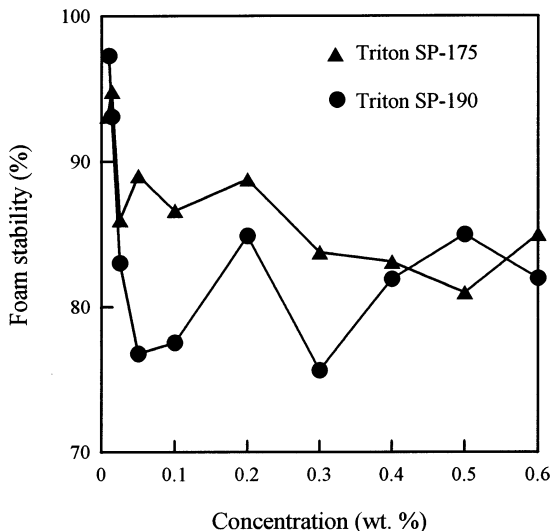


Fig. 3. Foam stability of aqueous Triton SP-175 and Triton SP-190 surfactant solutions measured by using a modified Ross–Miles method.

ability of Triton SP-135 surfactant appears due to its limited solubility, and it is evidently unsuitable for use as a foaming agent. From the plot, one can find that an increase in surfactant concentration would result in increased initial foam heights. However, the results also indicate that the surfac-

tant concentration higher than 0.4 wt.% did not significantly improve the initial foam heights. The stability of foam is defined as the ratio of the 5-min foam height to the initial foam height and is demonstrated in Fig. 3. It seems that Triton SP-175 surfactant has a slightly higher foam stability than Triton SP-190 surfactant.

### 3.2. Foam/oil interaction

Oils are well-known agents for foam inhibition and breaking. It has been suggested that oil can destroy foams by spreading or entering [30]. For a potential defoamer or oil to affect the stability of a foam film, it must be able to penetrate or enter the aqueous solution/gas interface first [31]. From the thermodynamic point of view, the penetrating process is favorable if the entering coefficient,  $E$ , is positive.

$$E = \gamma_{wg} - \gamma_{og} + \gamma_{wo} \quad (1)$$

where  $\gamma_{wg}$  is the surface tension of the foaming solution,  $\gamma_{og}$  is the surface tension of the defoamer or oil, and  $\gamma_{wo}$  is the interfacial tension of the foaming solution/defoamer interface.

Moreover, the spreading coefficient of a defoamer on an aqueous solution/gas interface was proposed as an additional stability criterion [32]. From thermodynamics, the spreading process of a defoamer over a foam surface is favorable if the spreading coefficient,  $S$ , is positive [33].

$$S = \gamma_{wg} - \gamma_{og} - \gamma_{wo} \quad (2)$$

Table 1 lists the tension data and calculated coefficients for the systems of n-pentadecane with 500 ppm Triton SP-175 or Triton SP-190 surfactant solutions. Since the entering coefficients are positive and the spreading coefficients are negative, n-pentadecane would be drawn through, but would not be expected to spread at the gas/aqueous surfactant solution interface. This condition may or may not cause destabilization [30]. For systems with positive entering but negative spreading coefficients, both oil-tolerant and oil-sensitive foam types of behavior were observed [34].

Based on the micro-visualization results, Schramm and Novosad [30] found that upon con-

Table 1  
Entering and spreading coefficients of n-pentadecane on gas/aqueous surfactant solution interfaces

Surfactant	Surfactant concentration (ppm)	$\gamma_{wg}$ (mN m <sup>-1</sup> )	$\gamma_{og}$ (mN m <sup>-1</sup> )	$\gamma_{wo}$ (mN m <sup>-1</sup> )	Entering coefficient	Spreading coefficient
Triton SP-175	500	28.8	27.5	2.1	3.4	-0.8
Triton SP-190	500	29.9	27.5	3.6	6.0	-1.2

tact with this type of foam, the oil spontaneously emulsified into smaller droplets in the plateau borders and the foam possessed a moderate stability to collapse. It was also observed that this type of foam lamella would transport oil droplets some distance before rupturing and releasing the oil. A subsequent lamella would sweep-up this oil and transport the oil some distance farther again [30,35].

### 3.3. Apparent solubility

Experiments were performed to evaluate the ability of surfactants to improve the apparent solubility of n-pentadecane in water. The apparent solubilities of n-pentadecane in aqueous solutions of Triton SP-175 and Triton SP-190 at 25°C are shown in Fig. 4. It was found that the apparent n-pentadecane solubility increased linearly at surfactant concentrations above their critical micelle concentrations, ~100 ppm [28]. These results indicate that Triton SP-175 and Triton SP-190 surfactants can greatly increase the apparent solubility of the hydrophobic n-pentadecane. The increased solubility of n-pentadecane may be due to the incorporation or partition within surfactant micelles [7,13,36]. Dissolution of n-pentadecane into the hydrophobic interior of the surfactant micelles is able to increase the effective solubility of n-pentadecane. The increased solubility may increase potential mobilization of trapped n-pentadecane droplets and reduce the amounts of surfactant solutions required for remediation.

The ability of a surfactant to improve the hydrocarbon solubility may be represented by the ratio of the number of moles of hydrocarbon solubilized to the number of moles of surfactant in the micellar form, which is defined as the molar

solubilization ratio (MSR) [7]. When the surfactant and n-pentadecane concentrations are expressed on a molar basis, the MSR can be calculated from the slope of the solubility curve above the critical micelle concentration of the surfactant. Since the exact molecular weights of Triton SP-175 and Triton SP-190 surfactants are not available, the surfactant concentrations can only be expressed on a mass basis in Fig. 4. However, the slope of the solubility curve in Fig. 4 is still an indication for the solubility capacity of a surfactant. These data suggest that on a mass basis Triton SP-190 surfactant has a similar capacity to solubilize n-pentadecane by comparing with Triton SP-175 surfactant.

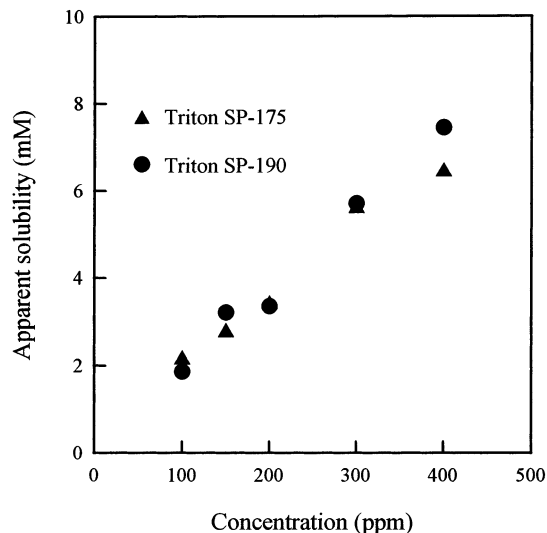


Fig. 4. Apparent solubilities of n-pentadecane in aqueous solutions of Triton SP-175 and Triton SP-190 surfactants at 25°C.

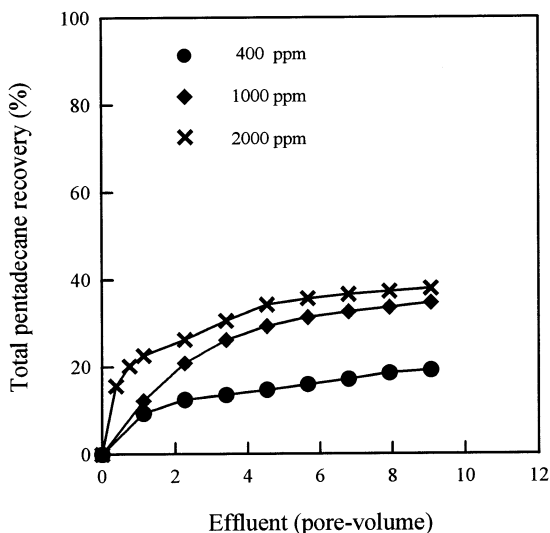


Fig. 5. Recoveries of n-pentadecane after flushing contaminated columns with various concentrations of aqueous Triton SP-190 solutions at a flow rate of  $5 \text{ ml min}^{-1}$ .

### 3.4. Surfactant-solution flooding

The ability of Triton SP-190 surfactant solutions to remove n-pentadecane from a contaminated glass-bead column was next investigated. The mass percents of n-pentadecane recovered from the contaminated glass-bead columns during flushing with different Triton SP-190 concentrations of aqueous solutions are shown in Fig. 5. The results presented in Fig. 5 were from column experiments conducted at surfactant concentrations ranging from 400 to 2000 ppm. In the experiments, the glass beads were initially contaminated with n-pentadecane with the oil volume/pore volume  $\cong 0.150 \pm 0.004$ . The results in Fig. 5 are similar in that the n-pentadecane was rapidly removed from the column in the first few pore-volume of effluent, followed by a slower removal rate. For example, flushing with the 2000 ppm surfactant solution recovered 36% of n-pentadecane in the first 6 pore-volume of effluent, while only another 2% was recovered for additional 3 pore-volume of effluent.

Of the three surfactant concentrations studied, the 2000 ppm solution was the most effective in removing n-pentadecane from the column. In

about 9 pore-volume of effluent, near 38% of n-pentadecane was removed by the 2000 ppm surfactant solution, and only about 19% of n-pentadecane was removed by the 400 ppm surfactant solution. Apparently, an increase in surfactant concentration would result in increased total n-pentadecane recovery. At one of the surfactant concentrations used in the column experiments, 400 ppm, the apparent solubility of n-pentadecane was approximately 7 mM (Fig. 4). However, the n-pentadecane concentrations in the first few pore-volume of effluent were found to exceed the solubility limit. Thus, these findings suggest that the solubilization of residual n-pentadecane in the surfactant solutions may not be the dominant mechanism in removing n-pentadecane from the contaminated glass-bead columns. Except the increased mobilization of residual n-pentadecane caused by reduced interfacial tensions in the presence of surfactants, the improved flushing by using the aqueous surfactant solutions could be due to the surfactant-enhanced emulsification of n-pentadecane from the glass-bead columns. This is supported by visual observations of emulsions forming in the effluents.

### 3.5. Foam-enhanced surfactant-solution flooding

The n-pentadecane recovery in a glass-bead column by a foam flooding process was then measured and compared with what obtained by a surfactant-solution flooding process. In all the experiments, immobile n-pentadecane was present in the glass-bead column prior to the injection of foam. The ratio of the volumetric flow rate of gas to that of liquid in the foam injected varied from 5/1 to 10/1, and the flow rate of the liquid comprising the foam injected was kept at  $2 \text{ ml min}^{-1}$ . Data are presented in Fig. 6 for the total recovery of n-pentadecane as a function of effluent volume at two gas-liquid ratios for a 400 ppm Triton SP-190 aqueous solution. The total recovery of n-pentadecane obtained in 9 pore-volume of effluent increased significantly using foam flooding compared with surfactant-solution flooding. Moreover, the increase of gas/liquid volumetric flow rate ratio would result in an improved total n-pentadecane recovery. Surfactant-solution

flooding recovered about 26% n-pentadecane in 9 pore-volume of effluent. At the gas–liquid ratio of 5/1, about 36% of n-pentadecane was removed by 9 pore-volume of effluent. However, at a higher gas–liquid ratio of 10/1, slightly over 74% of n-pentadecane was removed.

When foam is present, the effective permeability of the porous medium to each phase is greatly reduced compared with permeability measured in the absence of foam [17,20]. Therefore, foam can be expected to reduce the channeling flow effect in a reservoir by decreasing the permeability to the displacing aqueous phase. This improves the mobility ratio and thus the homogeneity of the flood.

Moreover, from visual observations, the foam was susceptible to fast disintegration upon contact with n-pentadecane. Initially, the foam front could only be traced for a short distance from the inlet, beyond that point, the foam began to disperse and deteriorate. Observations indicated that the higher the gas–liquid volumetric flow rate ratio, the faster the foam filled the column. In the case with a gas–liquid ratio of 5:1, the foam front never reached the outlet of the column during the experiment. Because of the early foam breakdown, the fingering flow occurred in the foam

flooding with a gas–liquid ratio of 5:1 was practically more significant than the foam flooding with a gas–liquid ratio of 10:1. In the foam flooding experiment with a gas–liquid ratio of 10:1, the position of foam front could be clearly identified. Propagation of foam front was observed along the glass-bead column as the effluent volume reached about 5 pore-volume. The foam front finally arrived at the outlet after 7 pore-volume of effluent was collected. A sharp increase in n-pentadecane recovery was also observed in about 5–7 pore-volume of effluent at the gas–liquid ratio of 10:1. Beyond this, the n-pentadecane recovery only increased slightly. It was observed in the micromodel that this type of foam lamella with a positive entering coefficient and a negative spreading coefficient could sequentially imbibe and transport oil droplets before rupturing [35]. As a result, the removal of n-pentadecane by foam in this study may be described by the possible mechanism of oil-drop emulsification and imbibition into the foam lamellae structure, which was proposed by Schramm et al. [35].

The effect of surfactant concentration on n-pentadecane recovery efficiency was investigated for 400 and 1000 ppm of Triton SP-190 solutions at two gas/liquid volumetric flow rate ratios. A similar plot as Fig. 6 for the 1000 ppm Triton SP-190 aqueous solution is shown in Fig. 7. The efficiency depended on the surfactant concentration and the ratio of gas to liquid in the foam injected. An increase in surfactant concentration apparently resulted in higher n-pentadecane recovery efficiency. However, it should be noted that an excessively high concentration of surfactants may cause foam barrier resulting in decreased sweep efficiency [37]. Similarly, one can observe in Fig. 7 that foam flooding exhibited significantly higher n-pentadecane recovery than surfactant-solution flooding, and the effectiveness of foam flooding in recovering n-pentadecane increased with increasing gas/liquid ratio. Moreover, visual observations indicated that the higher the surfactant concentration, the faster the foam filled the column. A sharp increase in n-pentadecane recovery was observed in about 4–6 pore-volume of effluent at the gas–liquid ratio of 10:1, compared with 5–7 pore-volume of effluent in the case of 400 ppm.

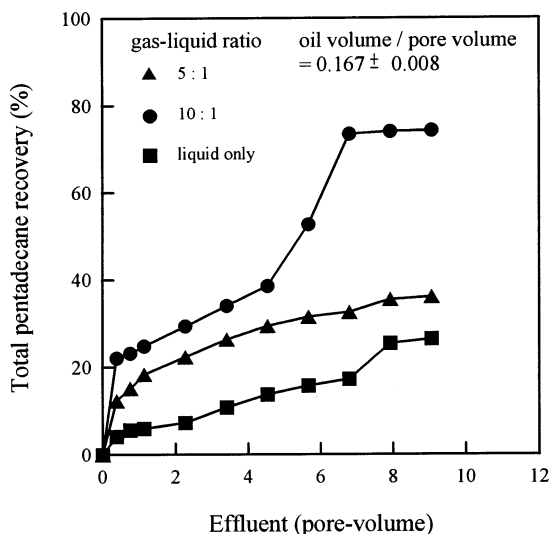


Fig. 6. Recoveries of n-pentadecane after foam-enhanced flushing contaminated columns with 400 ppm aqueous Triton SP-190 solutions at a flow rate of  $2 \text{ ml min}^{-1}$  under different gas–liquid ratio conditions.

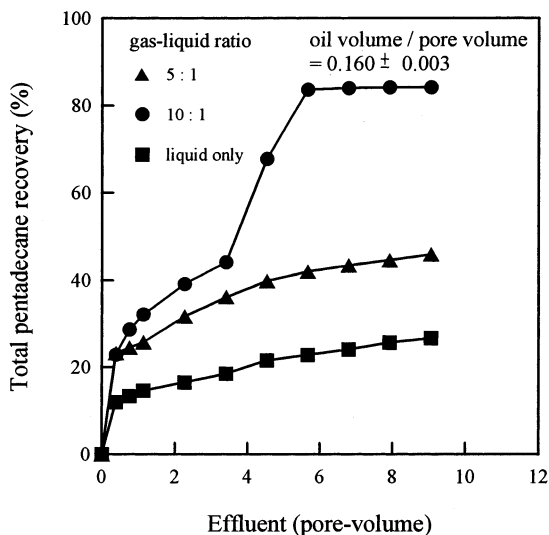


Fig. 7. Recoveries of n-pentadecane after foam-enhanced flushing contaminated columns with 1000 ppm aqueous Triton SP-190 solutions with a flow rate of  $2 \text{ ml min}^{-1}$  under different gas-liquid ratio conditions.

#### 4. Conclusions

This study investigated the efficiency and characteristics of foam-enhanced surfactant-solution flooding in removing n-pentadecane from a contaminated glass-bead column. A surfactant-solution flooding approach was explored first, and it was found that the n-pentadecane recovery was increased with increasing surfactant concentration. Moreover, the surfactant-enhanced emulsification, instead of the solubilization, may play a more important role in recovering n-pentadecane from the contaminated column. The recovery efficiency of n-pentadecane was found to be greatly increased by using a foam-enhanced surfactant-solution flooding approach, probably because of the reduction in the channeling flow effect. These data demonstrated that by the use of foam, the n-pentadecane recovery efficiency can be significantly improved. Moreover, the recovery efficiency may be increased as the gas-liquid volumetric flow rate ratio in the foam injected was increased.

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