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Soil flushing using colloidal gas aphron suspensions generated from a plant-based surfactant

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Abstract

Natural surfactant solutions obtained from the fruit pericarps of Sapindus mukorossi, commonly known as Ritha or soapnut, are tested for their ability to remove hydrophobic organic compounds (HOCs) from soil. Colloidal gas aphron (CGA) suspensions generated using the surfactant are used in this study to flush an HOC from a representative soil. Soil is spiked with a chlorinated hydrocarbon, hexachlorobenzene (HCB), serving as a model HOC representative of contamination at a Superfund site north of Baton Rouge, LA. The recovery of HCB from soil columns using CGA suspensions was considerably larger than that for a waterflood. HCB recoveries in the effluent reached a maximum by the fifth pore volume and remained fairly constant for soils contaminated with high levels of HCB. This maximum HCB concentration in the column effluent was proportional to HCB solubility in the corresponding surfactant solutions. Natural surfactant performed marginally better in the form of conventional solutions than CGA suspensions at similar concentration in recovering HCB. HCB removal increased with increasing surfactant concentration due to increased aqueous solubility. The pressure buildup across the soil column remained fairly low when natural surfactant was used at concentrations up to 1%. Alternating the flushing media between CGA and water neither enhanced the recovery of HCB nor changed the pressure buildup across the soil column. © 1998 Elsevier Science B.V.

Keywords: Natural surfactant; Ritha; Sapindus mukorossi; Soil flushing; Colloidal gas aphron (CGA); Hexachlorobenzene

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1. Introduction

Conventional 'pump and treat' technologies for treating hazardous waste sites require long times to significantly reduce the mass of contaminants in the subsurface [1]. Surfactant solutions, when used with existing 'pump and treat' systems, can sometimes enhance the performance [2-5]. Surfactants form aggregates known as micelles with a hydrophobic core at concentrations beyond the critical micelle concentration (CMC). The ability of the surfactant micelles to solubilize adsorbed hydrophobic organic compounds (HOCs) from soil is dependent on the interaction of the compounds with the surfactant and soil, sorption of surfactant on soil and the partitioning of the compound with the surfactant micelle [4]. Although many commercial surfactants have shown good potential in terms of recovery of contaminants from soils, their fate in the subsurface is still unknown. An alternative to commercial or synthetic surfactants are those derived from plants, specifically belonging to the genus Sapindaceae. These plants produce saponaceous substances called saponins, which lather or foam in water. Sapindus mukorossi, Sapindus trifoliatus, Sapindus laurifolius and Sapindus emarginatus are widely grown in India, Pakistan and other tropical and subtropical regions of the world. Fruit pericarps of these plants have been traditionally used as soap substitutes for fabric washing, bathing and in folk medicine [6,7]. The recorded external use of saponins does not site any toxic effects on human skin and eyes [8].

Several researchers have isolated and identified the saponins from the fruit pericarps of the plants of genus *Sapindus* [7,9–11]. Saponins are complex substances and are essentially glycosides with their aglycones related to either sterols or triterpenes. Gedeon [7] quantified the saponin content of fruit pericarps to be 10.1% of the weight of the pericarp and 6.1% based on the weight of the nut. Extraction with water has been the simplest, inexpensive and the most commonly used method for both scientific purposes and domestic use. Preliminary experiments using *S. mukorossi* indicated that this fruit extract can desorb and solubilize significant amounts of hydrocarbons [12–14].

Surfactants can be introduced into the subsurface both as conventional surfactant solutions and as colloidal gas aphron (CGA) suspensions [15–17]. CGAs are micron size gas bubbles generated with a film of surfactant around them and are a class of Kugelschaum foams [18]. CGA suspensions typically consist of 65% gas by volume and therefore form a low-density fluid. The CGA suspensions have viscosities similar to water, which make them suitable for pumping without significant deterioration. Some of the applications of CGA suspensions are flotation of organic contaminants, soil flushing, and in situ bioremediation. CGA suspensions have been shown to sweep the contaminated soil more efficiently than a simple water flood or conventional surfactant solutions in laboratory soil columns [15,16]. Natural surfactant solutions have been used to generate CGA suspensions and their properties such as size distribution and stability have been studied [17]. The results of solubility and desorption studies indicate that natural surfactant solutions are comparable to commercial surfactants in solubilizing HOCs [13,14].

The objective of this study is to investigate the suitability of CGA suspensions generated from natural surfactant solutions for flushing columns containing HOC contaminated soil. The hypothesis is that CGA suspensions, due to their stability, can be

used effectively to remediate contaminated soil and their performance may be superior to conventional surfactant solutions due to their small size and the sweep efficiency. An aromatic chlorinated hydrocarbon, hexachlorobenzene (HCB), which is one of the major contaminants of concern at a local Superfund site is selected as the compound for the study. The performance of CGA suspensions is compared to conventional surfactant solutions and waterfloods. The effect of alternating the introduction of flushing media with intermediate waterfloods on the removal of HCB and the pressure buildup across the soil column were also investigated.

2. Materials and methods

2.1. Materials

Dry fruits of *S. mukorossi* were procured from India. These fruits are golden brown in color and have diameters between 1 and 3 cm. After separating the seed, the pericarps were dried in an oven at 50°C for 2 days. The dried pericarps were ground and sieved through a US standard sieve No. 20 (840 μ m). Hexachlorobenzene (HCB), a chlorinated aromatic hydrocarbon was obtained from Aldrich Chemical (Milwaukee, WI) with 99% purity and was used as supplied. HCB has a molecular weight of 284.8 with a boiling point of 325°C and vapor pressure of 1.9×10^{-5} mm Hg at 25°C.

Soil from an uncontaminated region at a local Superfund site north of Baton Rouge, LA was air dried, homogenized, and kept in an oven overnight at 105°C for drying. Soil was ground and the fraction passing US standard sieve No. 10 (2 mm) was used. This soil is classified as sandy loam and has a low organic matter content (0.2%). The soil was 70% sand, 20% silt and 10% clay.

2.2. Preparation of natural surfactant solutions

Natural surfactant solutions were prepared using the method reported by Roy et al. [13]. An appropriate quantity (10 g per 100 ml water) of dry fruit pericarp powder was added to deionized (DI) water and stirred for 3 h at room temperature. The mixture was centrifuged at 10000 rpm ($\sim 14000 \times g$) for 45 min and the supernatant was filtered sequentially through a 0.44-mm pre-filter and a Metricel 0.45 μ m membrane filter

Table 1 Properties of natural surfactant [13,19]

Empirical formula ^a	$(C_{26}H_{31}O_{10})_n$
Total organic carbon (TOC) ^a	41 g
Chemical oxygen demand (COD) ^a	124 g/l
Nitrogen and phosphorus ^a	not detected
Critical micelle concentration (CMC)	0.1%
pH (1% solution)	4.5

^aFor 10% natural surfactant solution.

(Gelman Scientific, Ann Arbor, MI). The solution thus obtained (10%) was used as stock solution. Composition and some basic properties of the surfactant are listed in Table 1 [13,19].

2.3. Generation of colloidal gas aphrons

Surfactant solutions made from the fruit pericarps were used to generate colloidal gas aphron (CGA) suspensions. A unit was developed in our laboratory for generating CGA suspensions from surfactant solutions [20]. The generator was used in a continuous mode with CGA suspensions being withdrawn from one side and the surfactant fed from the other.

2.4. Soil flushing experiments

Glass columns 10 cm long and 5.75 cm in diameter were used for all soil flushing experiments with a stainless steel top and bottom plates. The bottom plate has a groove of 1.5 cm making the length of soil column equal to 11.5 cm. The schematic diagram of the experimental setup is shown in Fig. 1. The outlet and inlet ends of the column were fitted with a fine wire mesh sandwiched between two coarse wire screens to prevent soil from being washed out of the column and to distribute the flow uniformly across the soil



Fig. 1. Schematic diagram of the one-dimensional soil column flushing experiment.

column [19]. The soil fraction passing US standard No. 10 (2 mm) sieve was spiked with the appropriate amount of HCB dissolved in petroleum ether. The soil was tumbled for about a week after evaporating the solvent. A portion of the contaminated soil was extracted with a acetone:hexane (1:1) mixture and the HCB concentration on the soil was determined. The soil was then dry packed into the column in four equal layers of about 110 g and each layer was compacted by giving 25 strokes with a compacting rod. The bulk density and porosity of the packed soil column measured using the weight of soil and volume of column were about 1.6 g/cm³ and 0.40, respectively.

The packed column was placed vertically and saturated slowly from bottom using water with a hydraulic head of about 50 cm to remove trapped air. Flushing solvents were pumped in to the top of the column at a flow rate of 2.5 ml/min (pore water velocity of 0.24 cm/min) thus providing a downflow mode. The flushing media were natural surfactant in the form of CGA suspensions or conventional surfactant solutions at concentrations of 0.5% and 1.0% or simply water. The pressure at the influent end was monitored using an analog pressure gauge. The effluent from the column was collected in pore volumes (about 120 ml each) using a 125-ml Erlenmeyer flask sealed with parafilm. Duplicate samples from each pore volume were analyzed for HCB and natural surfactant concentration.

For the experiments using alternating water floods, flushing media (CGA suspensions or conventional surfactant solutions) were pumped for the first three pore volumes before switching to water. Water was pumped for the next two pore volumes and switched back to the original fluid for two more pore volumes. Two pore volumes of water and flushing solvent were alternated until a total of 16 pore volumes were collected. The last pore volume was that of water.

2.5. Extraction and analysis of hexachlorobenzene

Commercially available Sep-Pak C₁₈ cartridges were used for extracting HCB from aqueous surfactant solutions using a procedure described elsewhere [19]. The cartridges were activated by passing 5 ml DI water, 5 ml methanol and 5 ml DI water again. Samples were eluted through the cartridge at a rate of 5 ml/min followed by 5 ml of DI water. The cartridge was then eluted with 5 ml of hexane, which was collected and analyzed for HCB on a gas chromatograph, HP 5890 Series II equipped with an HP 7673 auto sampler and a Ni⁶³ electron capture detector (Hewlett–Packard, Wilmington, DE). The column used was a 30-m PTE-5 capillary column (0.32 mm internal diameter and 1.0 μ m film thickness) (Supelco, Bellefonte, PA). The chromatographic conditions were: 1 μ l splitless injection, injection temperature 275°C, temperature program 50°C (initial for 1 min) to 270°C at 10°C/min and hold for 3 min, total run time 26 min, ECD temperature 325°C. Minimum detection limit was 1 pg.

2.6. Natural surfactant analysis

Natural surfactant solution was quantified using a UV/Visible absorbance method developed earlier [13,19]. Effluent samples were centrifuged and the UV absorbance of

the sample was measured. The concentration of the natural surfactant in the effluent was determined using calibrations reported elsewhere [13,19].

3. Results and discussion

3.1. Soil flushing

Solubility enhancements of HOCs in natural surfactant solutions and the desorption of HCB from soil have been previously reported [13,14]. Natural surfactant solutions increased the solubility of naphthalene and HCB by several hundred-times compared to water. This solubility enhancement is comparable to commercial surfactants [13]. In batch desorption experiments, natural surfactant desorbed as much as 90% of the total HCB from soil contaminated with low HCB concentrations (2 mg/kg). HCB concentration in the aqueous supernatant solution approached a maximum (about 90% of the solubility of HCB in the corresponding aqueous surfactant solution) for highly (\sim 100 mg/kg) contaminated soils [14].

Results of soil flushing experiments with different levels of soil contamination are presented in Fig. 2a,b for CGA suspensions generated from 0.5% and 1% natural surfactant, respectively. HCB concentrations on soil were in the range 2–110 mg/kg soil. The removal of HCB during the first pore volume was negligible. During the first pore volume saturation water was replaced with surfactant solution and the effluent contained essentially water in which the solubility of HCB is very low ($< 50 \ \mu g/l$). Surfactant introduced during the initial pore volumes will likely be sorbed onto the soil and not available to solubilize HCB. Natural surfactant concentration in the effluent measured using UV absorbance increased steadily and approached a breakthrough between 3 and 5 pore volumes. The surfactant concentration remained fairly constant beyond once breakthrough was achieved. The delay in breakthrough of the surfactant indicates that the surfactant is undergoing sorption and retardation in the soil. Sorption decreases the amount of surfactant available for micellar solubilization. HCB concentration increased steadily after the first pore volume and approached a maximum by the fifth pore volume. This maximum concentration of HCB in the effluent was proportional to the HCB solubility in the corresponding aqueous surfactant solution. HCB concentration in the effluent remained constant for the remainder of the experiment at 80% of HCB solubility in the aqueous surfactant solution. The removal of HCB appeared to be limited at low contamination levels (2 mg HCB/kg soil). The HCB concentration in the effluent was only a small fraction of its solubility in such a case. However, for soils contaminated with higher amounts of HCB, effluent HCB concentration approached a constant maximum proportional to the solubility of HCB in the corresponding aqueous surfactant solution. CGA generated using 1% natural surfactant solution recovered a total of 120 μ g and 670 μ g HCB at the end of 12 pore volumes from soil contaminated with 2 and 92 mg HCB per kg soil, respectively. The cumulative HCB removed as a percent of total HCB in the column was 15.1% and 1.7%, respectively. As soil contamination level increased, HCB recoveries in the effluent also increased. However,



Fig. 2. Removal of HCB from soil columns using CGA suspensions, generated from (a) 0.5% and (b) 1% natural surfactant solutions.

the recoveries were limited by the solubility of HCB in the flushing media for high soil contamination levels.

Total HCB recovered over 12 pore volumes was about 190 μ g and 670 μ g for CGA suspensions generated with 0.5% and 1.0% natural surfactant solution, respectively. HCB concentration on the soil was 110 mg/kg and 92 mg/kg, respectively. These amounts are about 24 and 84 times more than that recovered with water (8 μ g) from soil

columns contaminated with 92 mg HCB/kg soil. Again, the recovery of HCB was 0.37% for 0.5% and 1.7% for 1.0% suspensions compared to 0.02% for water. HCB remaining in the soil column was quantified by slicing the column into four approximately equal sections and analyzing each section independently, and in duplicate. HCB mass recovered in the effluent and that remaining on the soil were used to perform a mass balance. The total HCB recovered in the effluent and the HCB remaining on the soil accounted for about 90% of the original HCB in the soil column.

3.2. Comparison between CGA suspensions and conventional surfactant solutions

Natural surfactant in the form of conventional solutions and CGA suspensions at 0.5% and 1.0% were used to flush soils contaminated with HCB. A comparison between CGA suspensions and conventional surfactant solutions is shown in Fig. 3a.b for soil contamination levels of 2 mg/kg and 92-110 mg/kg, respectively. The conventional surfactant solutions performed better than CGA suspensions generated with identical surfactant concentration. CGA suspensions generated with 1.0% natural surfactant removed about 670 μ g of HCB in 12 pore volumes and conventional surfactant solutions of similar concentration recovered about 890 μ g under identical conditions. These results are at variance with those reported in earlier studies on the application of CGA suspensions from a commercial surfactant for soil flushing [15,16]. Roy et al. [16] used sodium dodecylsulfate (SDS) in the form of conventional solutions and CGA suspensions to recover residual levels of nonaqueous phase liquids (NAPLs) from soil columns and found CGA suspensions to have better recoveries than conventional surfactant solutions. In another study [15], 2,4-dichlorophenoxyacetic acid (2,4-D) was used as the contaminant in which case CGA suspensions and conventional surfactant solutions had similar recoveries. Roy et al. [21] used several commercial surfactants in the form of conventional solutions and CGA suspensions to flush soils contaminated with naphthalene and reported that conventional surfactant solutions showed greater contaminant removal than CGA suspensions as observed in the present study. The possible reasons for the lower recovery with CGA suspensions could be pore blocking and channelling through the soil matrix due to dispersion of soil colloids. The dislodged soil fines due to the passage of CGA could block the soil pores, restricting flow through certain channels and thus bypass the majority of the soil matrix. Another possible reason could be the loss of HCB in the gaseous phase of the CGA effluent [21]. As mentioned earlier, CGA consists of 65% air and the column effluent will have two distinct phases, air and water. Only the aqueous phase was collected and accounted for in this study. It should be noted that in the reported studies by Roy et al. [16] where the CGA suspensions had better performance than conventional surfactant solutions, the contaminant was a separate phase, a NAPL. In this study and that of Roy et al. [21] the test organic is in adsorbed state and we only the effect of solubility with CGA and/or conventional solution is observed and not free phase displacement.

3.3. Effect of surfactant concentration

Fig. 3a,b also depicts the effect of surfactant concentration on the removal of HCB for both conventional surfactant solutions and CGA suspensions at two levels of soil



Fig. 3. Effect of natural surfactant concentration on the removal of HCB from soil columns (a) 2 mg and (b) 92 mg HCB/kg soil.

contamination (2 and 92–110 mg HCB/kg soil). With an increase in surfactant concentration the number of micelles formed increase and also the number of surfactant molecules per micelle will increase and thus can solubilize more HCB. This suggests that micellar solubilization is the primary mechanism responsible for the mobilization and subsequent washing of HCB from soil. Similar trends were observed for other HCB

contamination levels. When the natural surfactant concentration was increased from 0.5% to 2.5% (by weight) the removal increased from 190 μ g in 12 pore volumes to 3300 μ g in 11 pore volumes. In contrast, a waterflood was able to recover only 8 μ g in 12 pore volumes. However, when 2.5% solutions were employed, a high pressure developed and the run had to be terminated at the end of 11 pore volumes. It should also be noted that surfactant solutions of 1.5% and higher concentration are not suitable for generating CGA suspensions as they tend to produce low quality (gas fraction) suspensions [17]. Liu [22] reported that increased surfactant concentration could change the hydraulic properties and pore geometry of the soil matrix. From our study, it appears that a natural surfactant concentration of 1% is best for soil-flushing operations.

3.4. Pressure buildup in soil columns

Pressure buildup is one of the controlling factors that determine the application of surfactants in field applications [2]. Fig. 4 shows the pressure buildup in the soil columns for CGA suspensions generated with 0.5% and 1% natural surfactant solution. The general trend observed is that pressure increased with an increase in surfactant concentration. However, the increase was not significant when the natural surfactant concentration increased from 0.5 to 1%. When a high concentration surfactant was used in the form of conventional solutions, the pressure increased gradually and approached 60 psi (in 8 pore volumes for 2.5% solution and 3 pore volumes for 5% solution) and the experiment had to be terminated. It is believed that the soil-surfactant interactions are responsible for the increase in pressure [3,22]. There was no significant difference in



Fig. 4. Pressure buildup across the soil columns for the flushing experiment.

pressure buildup between the CGA suspensions and conventional solutions at lower concentrations (0.5% and 1%). This observation is in contrast to those of Roy et al. [16] who used SDS solutions to flush automatic transmission fluid. In some cases, the columns were flushed with conventional solutions and CGA suspensions for 16 pore volumes and the pressure appeared to stabilize after 4 to 5 pore volumes. From our studies it appears that a natural surfactant solution at a concentration of 1% is a good compromise to achieve higher removal and avoid the high pressure buildup in the soil column.

3.5. Effect of alternating flushing solvent

It has been suggested in the literature that by alternating the flushing solvent (conventional surfactant solution or CGA suspension) with a simple waterflood, the high pressure buildup across the soil columns could be avoided and would help in the optimization of the amount of surfactant required to recover the contaminant [15]. As discussed in earlier sections, the surfactant breakthrough occurs after about 4 to 5 pore volumes and the recovery of the contaminant was stable beyond that. In this study, two concentrations of natural surfactant, 1% and 2.5%, and CGA suspensions generated with 1% surfactant solution were alternated with a waterflood.

The results of the study are presented in Fig. 5a,b. HCB concentration for all the experiments was 92 mg HCB /kg soil. As observed earlier, conventional surfactant solutions recovered more HCB from soil columns in 16 pore volumes than the CGA suspensions of similar concentration in the continuous mode of flushing. Higher concentrations of natural surfactant (2.5%) were able to recover significantly more HCB (3300 μ g in 11 pore volumes) than 1% natural surfactant (1325 μ g in 16 pore volumes). The conventional surfactant solutions gave higher recoveries of HCB (510 μ g) than the CGA suspensions (390 μ g) in 16 pore volumes in the alternating mode of flushing as observed for continuous flushing. The alternate runs recovered about 37% of the total mass of HCB removed in continuous mode of operation. However, it should be noted that when the flushing media is alternated, only half of the surfactant used for continuous runs was utilized. For example, for a 16 pore volume experiment, 9 pore volumes of surfactant and 7 pore volumes of water were used. HCB recovered at the end of 16 pore volumes was used to calculate the removal per gram of natural surfactant. For 1% natural surfactant in the form of conventional solution and CGA suspensions the recoveries were 69 and 55 μ g/g of surfactant, respectively in the continuous mode of operation compared to 48 and 35 μ g/g in alternate mode of flushing. In the alternate mode of operation, conventional surfactant solutions or CGA suspensions were pumped for about three pore volumes and then switched to water. The first pore volume of water after switching basically replaced the surfactant that was already pumped and thus the recovery did not decrease from that of the earlier pore volume. However, during the second pore volume, the amount of surfactant available was low and thus could recover only small quantities of HCB due to low HCB aqueous solubility. When the flushing fluid was changed back to conventional surfactant solution or CGA suspension, the first pore volume essentially replaced the pore water and thus the recoveries were lower. By the second pore volume, however the recoveries increased due to an increased amount of



Fig. 5. Effect of alternating flushing media with water on HCB removal from soil columns (a) 1% natural surfactant and CGAs and (b) 1% and 2.5% natural surfactant.

surfactant in the effluent and continued to increase for one pore volume even after switching to water.

As mentioned in Section 3.4, when conventional surfactant solutions of 2.5% concentration were used, a high pressure buildup developed across the soil columns and the experiment was terminated. Roy et al. [15] reported that the high pressure buildup during the surfactant run was lowered considerably when switched to water. However,



Fig. 6. Effect of alternating flushing media with water on the pressure buildup across the soil columns.

the results of this study are contrary to these observations. Fig. 6 shows the variation of pressure buildup across the soil columns for the alternate runs and the corresponding continuous flushing runs. The pressure behavior did not change significantly when the flushing media was alternated with a waterflood. The column runs with 2.5% natural surfactant solution were terminated after 11 pore volumes due to a high pressure. However, conventional surfactant solutions and CGA suspensions at lower concentration (1%) have similar pressure trends and alternating with water did not change the behavior appreciably. The pressure buildup across the soil columns remained stable for all the 16 pore volumes beyond the initial 3 or 4 pore volumes for 1% natural surfactant in the form of both conventional surfactant solutions and CGA suspensions suggesting that the conventional surfactant solution or CGA suspension flush could be extended to still larger pore volumes and recover most of the HCB from the soil. The increased recovery of HCB using surfactant solutions over the waterflood significantly reduces the number of pore volumes required to achieve the cleanup goals as compared to a waterflood.

4. Conclusions

CGA suspensions generated from natural surfactant solutions are used in this study to recover HCB from soil columns and the performance is compared with conventional surfactant solutions. The following conclusions can be drawn from this study.

• CGA suspensions generated from natural surfactant solutions are very effective in recovering HCB from soil than a simple waterflood. CGA suspensions generated with

1% natural surfactant recovered 670 μ g in 12 pore volumes compared to 8 μ g by waterflood.

• The recovery of HCB was lower for low-contamination soils and the recovery increased with increasing contamination on the soil. However, the maximum HCB concentration in the effluent was limited to about 80% of HCB solubility in the corresponding surfactant solution.

• Natural surfactant in the form of conventional solutions outperformed the CGA suspensions in recovering HCB from soil columns under identical conditions.

• Increase in natural surfactant concentration increased the recovery of HCB significantly for both conventional solutions and CGA suspensions. The increased solubility of HCB is responsible for the enhanced recovery.

• The pressure buildup across the soil columns remained fairly low when natural surfactant was used at concentrations up to 1% for both conventional solutions and CGA suspensions.

• Alternating the flushing media with waterflood neither reduced the pressure buildup nor increased the removal of HCB.

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