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Enhanced removal efficiency and influencing factors of nitrobenzene in soil by foam flushing

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Abstract. Laboratory scale experiments were conducted to investigate the removal efficiency of nitrobenzene in soil by different kinds of flushing liquid. In addition, the influencing factors, such as foam injection rate, foam quality and permeability of medium for the removal efficiency of nitrobenzene in soil were discussed in the paper. The results showed that the residual rates of nitrobenzene in soil were 62.15%, 63.18%, and 2.65%, while the flushing liquid were water, SDS solution, and SDS foam, respectively. The removal efficiency of nitrobenzene 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 nitrobenzene recovery efficiency can be significantly improved. Within the scope of the study, the foam quality and foam injection rate has little effect on the total removal efficiency of nitrobenzene in soil. Moreover, the total removal efficiency of nitrobenzene for low permeability (0.1~0.25mm, 0.25~0.5mm) is greater than that in high permeability medium. Because of the time necessary for distribution of foam through the soil and desorption process is much longer for soil with a low hydraulic conductivity, this technique is more effective in soil with lower hydraulic conductivity and higher porosity.

1. Introduction

Surfactant is widely used in in-situ leaching technology of organic contaminated soil due to the enhanced solubility of non-aqueous liquid. Surfactant solution leaching technology is a recommended method by US EPA to use for soil and groundwater contaminated by hydrophobic organic compounds (HOCs)[1]. Surfactants enhance organic contaminant recovery through two mechanisms. First, surfactants reduce the interfacial tension between water and contaminants that slows the mobility of the organic components. Therefore surfactants are able to transfer the HOCs to the mobile phase[2]. Secondly, surfactants are capable of forming aggregates known as micelles, thus solubilizing HOCs[3]. However, some intrinsic challenges are associated with the solution-based leaching technology. These challenges include the predominant vertical transport of surfactant solution and the preferential percolation of the solution through highly permeable pathways[4].

Based on these disadvantages, some laboratory studies have used surfactant foam instead of surfactant solution as leaching agent[5-8]. Foam is a multiphase dispersed system and could uniform distributed in medium. Surfactant foam flushing is able to avoid channel effect, resulting in larger influence region and enhanced contaminant displacement[9]. In addition, due to its relatively low density, foam can easily overcome gravitational effects and flow through most of the region in a heterogeneous reservoir. Another advantage of using foam in soil treatment processes is that high



volumes of air per unit volume of foam are injected into the soil, some contaminant is able to be removed by volatilization[10]. Numerous studies have indicated that foam can enhance recoveries of non-aqueous phase liquids (NAPLs)[3,8,11] and pentachlorophenol (PCP)[12] from contaminated soils. The efficiency of foam-enhanced surfactant solution flooding strongly depends upon the surfactant type, the surfactant concentration, foam injection rate, foam quality, and initial pollutant concentration[11]. Although, very few papers dealing with the contaminants removal mechanisms and the mass balance of foam flushing process are available.

The specific objective of this study was to assess the efficiency of a foam flooding process in removing nitrobenzene from a contaminated sand column in comparison with the results obtained by a surfactant-solution flooding process. At the same time, an experimental column containing nitrobenzene-contaminated sand was used to explore the effect of foam injection rate, foam quality, and the permeability of medium on the nitrobenzene recovery efficiency. Additionally, the contaminant removal mechanisms were studied in the foam flushing process through mass balance calculation.

2. Materials and methods

2.1. Surfactant

Sodium Dodecyl Sulfate (SDS) from the Beijing Chemical Plant of China, of analytical grade purity, was used in all experiments. This anionic surfactant has a molecular mass of 288.4 kg/kmol and a critical micelle concentration (CMC) of 8.2mM (2.3 g/L) at 25°C[13]. Distilled water was used to prepare solution containing SDS concentration of 0.5%.

2.2. Contaminated soil

River bank sand was washed with water and a diluted nitric acid solution to remove all organic material possibly adsorbed on sand particles. Dried sand was sieved in order to obtain particles of a selected size range (0.1~0.25 mm, 0.25~0.5 mm, 0.5~1 mm, 1~2 mm). The permeability (k) was obtained using the Darcy Law. The permeability was 10, 25, 73, and 113 darcy for particle sizes correspondent to 0.1~0.25 mm, 0.25~0.5 mm, 0.5~1 mm, 1~2 mm, respectively.

Pure nitrobenzene (30mL) was dissolved in pure methanol (170mL) to give concentration of 180 g/L of nitrobenzene. The solution was then added together into the soil sample, forming contaminated soil containing theoretical nitrobenzene content of 4500 mg/kg. After drying for 2 hours, the contaminated soil was filled into the simulated column, which was chosen to simulate the in-situ condition. Some characteristics of the compacted soils used in this investigation are summarized in Table 1.

2.3. Experimental setup

Remediation experiments were conducted in the system described in Figure 1. A plastic column (L=20 cm, D=2 cm) with circular porous stone plates was used for foam generation in the presence of the surfactant solution and air. A peristaltic pump fed the surfactant solution and a flow meter controlled the flow of solution and air before entering the foam generation column. Varying the flow rates of the surfactant solution and air enabled control of the foam quality and generation rate.

The soil column (L=12.5 cm or 25 cm, D=5 cm) was connected to the foam generation column (Figure 1). The contaminated soil was packed uniformly in 4 layers and each layer was compacted by hitting the column with a wood bar on four different sides to avoid flow channeling in the column. A pressure gauge was installed upstream of the soil column to measure the inlet pressure. The pore volume of the column was measured by saturating the soil column at the pressure gradient close to zero. A three-way valve was placed before the soil column to prevent injection of foam into the soil before its uniformity and steady-state quality were reached. It was also used for taking foam samples before injection into the soil column.

To compare with results for the foam experiments, surfactant solutions without foam generation

were also pumped through the column. Distilled water was used as the controls. To compare with the nitrobenzene removal efficiencies for different foam injection rates, foam qualities and medium permeability, foam with different qualities and flow rates under different medium permeability were used to investigate the nitrobenzene removal efficiencies. The operating parameters of all simulation columns were listed in Table 2. For aqueous surfactant-solution and foam flooding experiments, the solution and foam were introduced through the top of column and was allowed to flow through the contaminated soil at desired volumetric flow rate.

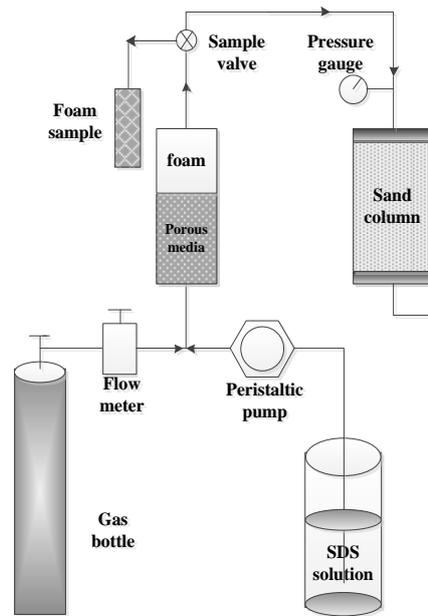


Figure 1. Schematic setup of the column experiments

Table 1. Properties of the soils used in column experiments

Medium size	1~2mm	0.5~1mm	0.25~0.5mm	0.1~0.25mm
Porosity	0.37	0.38	0.40	0.42
Density (kg/m ³)	1559.6	1517.7	1483.4	1479.4
Permeability (darcy)	113	73	25	10

Table 2. Parameters summary of column experiments

Column number	Soil grain size (mm)	Flushing liquid	Foam quality* (%)	Foam injection rate (mL/min)	Initial concentration of nitrobenzene (mg/kg)	Column size (L*ID)
1	0.5~1	Pure water	—	4	3669	25*5
2	0.5~1	SDS solution	—	4	3582	25*5
3	0.5~1	Foam	91	4	3465	25*5
4	0.5~1	Foam	91	6	3248	25*5
5	0.5~1	Foam	91	8	2968	25*5
6	0.5~1	Foam	94	6	3092	25*5
7	0.5~1	Foam	88	6	2887	25*5
8	0.5~1	Foam	91	6	2865	12.5*5
9	0.25~0.5	Foam	91	6	3636	12.5*5

10	0.1~0.25	Foam	91	6	3329	12.5*5
11	1~2	Foam	91	6	1315	12.5*5

*Foam quality means the ratio of the gas volume to the foam volume

2.4. Analytical methods

The foam exiting the soil column was collected for analyzing the nitrobenzene concentration. Samples were collected according to the number of pore volumes passed through the contaminated soil and were left to collapse completely. Liquid chromatography was used to analyze the nitrobenzene concentration in the liquid effluent samples. At the end of each test, samples of soil were taken from the column. Soil samples were weighed and methanol was used as the solvent to extract nitrobenzene from soil samples before analysis of nitrobenzene concentration. Liquid chromatography was used to analyze the nitrobenzene concentration in the methanol solution.

3. Results and discussion

3.1. Foam-enhanced surfactant-solution flooding

The nitrobenzene recovery in sand column by a foam flooding process was then measured and compared with what obtained by water and SDS-solution flooding process. The quartz sands with sizes of 0.5~1.0 mm, the foam quality was controlled at 91%, and the flow rate of the foam injection was kept at 4 mL/min. Data are presented in Figure 2(a) for the total recovery of nitrobenzene as a function of effluent volume. The spatial distribution of nitrobenzene along with the column for the series of experiment was also presented in Figure 2(b). The removal efficiency of nitrobenzene in effluent liquid obtained in 5.5 pore-volume increased significantly using foam flooding compared with water and surfactant-solution flooding. The residual rates of nitrobenzene in soil were 62.15% and 63.18% for water and SDS solution flooding, and far more than what obtained by foam flooding (2.65%). During the process of foam migration, the volatilization of nitrobenzene is also a key factor in its removal. From Figure 2, the removal rate of nitrobenzene in the effluent is 41.72%, and the residual rate of nitrobenzene was 2.65%. Therefore, the volatilization rate was 55.63% when the recovery rate was 100%.

Two reasons can account for the enhanced removal efficiency of nitrobenzene for foam flooding. On the one hand, when the liquid stream is converted into a foam stream, the density of the liquid is significantly reduced. As shown in Figure 3, air is the main component of foam, the density of foam is 0.1 times of the liquid when the foam quality is 90% so that the foam flow can effectively overcome the gravity flow through most of the areas in the simulated column, thereby enhancing the fluid uniformity and reducing the medium channel effect[7]. In addition, the main reason is that the migration of foam in the medium is the migration of piston flow. The contact area and contact efficiency of the foam solution with the contaminants were enhanced to allow the nitrobenzene to be fully dissolved in the foam solution. It has been shown that if the bubble shape in foam is a regular circular shape when the foam quality is 95%, the volume increased 20-fold, leading directly to the contact area of the liquid film increased by 7 times[13].

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[14]. 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. The effect of foam leaching on the removal of nitrobenzene in the soil can also be characterized by the hydraulic conductivity of the foam in the medium. The average hydraulic conductivity of the foam through sediment was calculated based on Darcy's equation using the following equation (1):

$$k = \frac{\Delta L \cdot V_{\text{foam}} \cdot \rho_{\text{water}} \cdot g}{A \cdot \Delta P} \quad (1)$$

where ΔL is the column height (cm), V_{foam} is the foam injection rate (mL/min), ρ_{water} is the water density (kg/m³), g is the gravity (m/s²), A is the cross-sectional area of column (cm²), and ΔP is the

pressure (kPa). So the average hydraulic conductivity of the foam through sand size of 0.5~1 mm was 2.84×10^{-3} cm/s for foam quality of 91.4% at foam flow rates of 4 mL/min. As shown in Table 2, the hydraulic conductivity of water through the same sediment was 73 darcy, corresponding to 0.07 cm/s. It was obvious from the data that the value of hydraulic conductivity for foam was almost one order of magnitude lower than the values for water, thus reducing the velocity of the foam flow. In this case, the foam liquid has more contact time with pollutants, thereby enhancing the nitrobenzene removal efficiency in the soil for foam flooding. This is expected due to the higher viscosity and lower density of foam as well as the Jamin effect[15,16].

The effective viscosity of the injection foam is an important parameter to account for the enhanced removal of nitrobenzene for foam flooding[17]. The effective viscosity is calculated using the following equation (2):

$$\mu_{(foam)} = \frac{kA}{q_{(foam)}} \frac{\Delta P}{\Delta L} \quad (2)$$

Where μ is the effective viscosity of foam (Mpa·s); k is the intrinsic permeability of porous medium (darcy); q is the foam flow rate (mL/s); A is the column cross-section (cm²); ΔP is the pressure drop (atm); ΔL is the column length (cm). It can be observed that when the injection flow is same, the effective viscosity of the foam in the medium is large, not only the contact area between the foam liquid and the pollutant is enhanced, but also the effect of the foam on the nitrobenzene. Accordingly, compared with water and SDS solution, the foam enhances the removal efficiency of nitrobenzene in the soil.

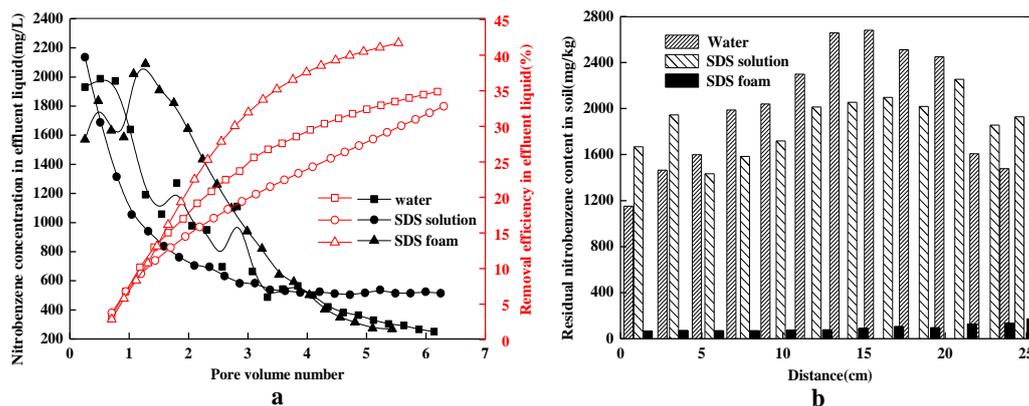


Figure 2. Curve of the concentration and removal rate of nitrobenzene in different eluents (a), and the spatial distribution of nitrobenzene in the medium (b)

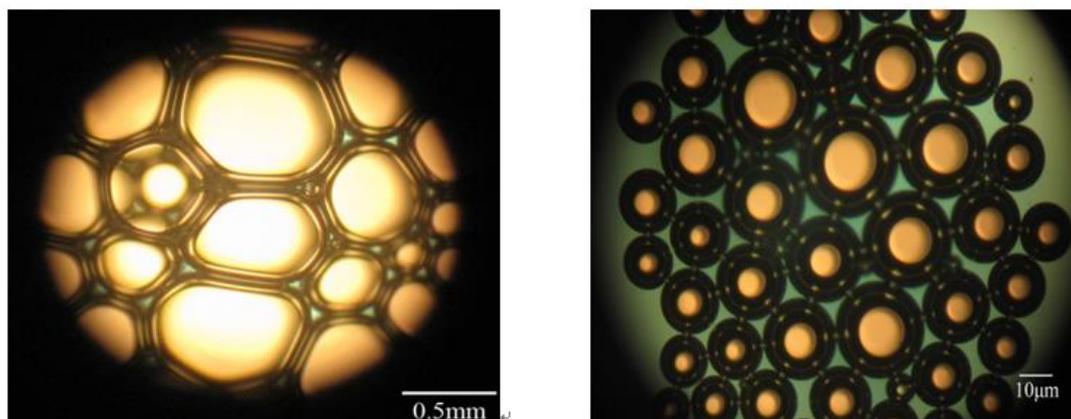


Figure 3. Micrograph of foam bubbles

3.2. Effect of Foam Injection Rate

Figure 4a shows the concentration of nitrobenzene in the effluent and the change of the removal rate

with the volume of effluent (PVs) at the rate of 4 mL/min, 6 mL/min and 8 mL/min, respectively. Figure 4b shows the effects of different foaming rates on the spatial distribution of nitrobenzene mass in the medium.

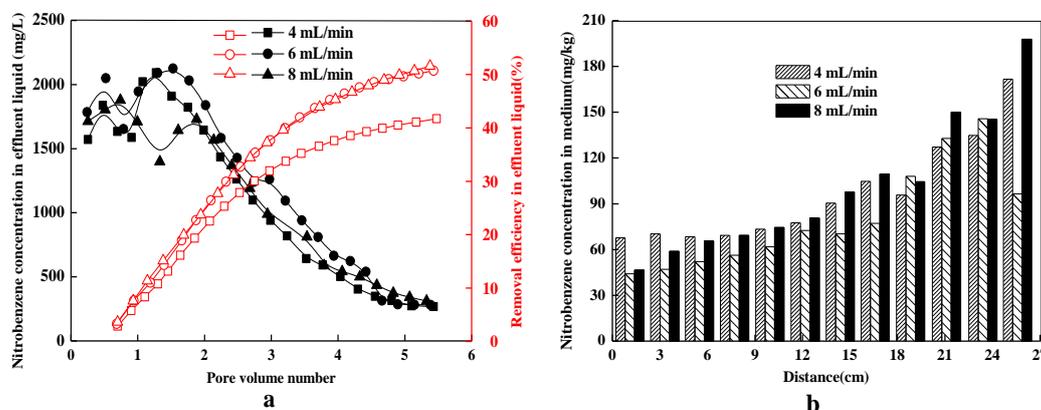


Figure 4. Influence of foam injection rate on the nitrobenzene concentration in effluent liquid and accumulated removal efficiency of nitrobenzene (a) and the spatial distribution of nitrobenzene in the medium (b)

It can be observed from Figure 4(a) that the concentration of nitrobenzene in the effluent decreases gradually with the increase of the volume of the effluent, and the concentration of nitrobenzene in the effluent remains constant when the effluent volume is more than 5PVs when the rates of foam were 6 mL/min and 8 mL/min, the removal rates of nitrobenzene were almost the same, which were 50.70% and 51.76%, respectively, which were higher than that of nitrobenzene at the rate of 4 mL/min (41.72%). However, the concentrations of nitrobenzene in the foam were not significantly different at the three foam injection rates. The main reason for the small removal rate of nitrobenzene in the foam was that the initial nitrobenzene concentration is low in medium.

Figure 4(b) showed that the spatial distribution of residual nitrobenzene in the medium is basically the same, and the residual rates were 2.65%, 2.5% and 3.33%, respectively. Accordingly, the effect of foam injection rate on the removal efficiency of nitrobenzene in soil was not significant. Three foam injection rates could make the contact time between the foam liquid and the pollutant have sufficient contact time, so as to achieve the purpose of pollutant removal.

The spread efficiency of foam in the medium is an important factor to determine the removal efficiency of nitrobenzene, and the capillary number (C_a) of foam in the medium is the key factor to determine the foam spread efficiency. It has been shown that when the capillary is more than 10^{-3} , the removal efficiency of medium pollutants can reach 100%. C_a is the ratio of viscous force and capillary force, the formula ^[16] is as follows:

$$C_a = \mu u / \sigma \quad (3)$$

In the abovementioned formula, μ is the viscosity of the foam, u is the Darcy rate of foam, σ is the interface tension between the foam liquid and the contaminant. It can be seen from the formula (3) that when the viscosity and the interfacial tension remain unchanged, C_a increases with the increase of foam injection rate, leading to the increasing removal efficiency of pollutants. However, the removal efficiency of nitrobenzene in this study does not follow the above rule, the reason was mainly due to the fact that when the foam injection rates were 4, 6, and 8 mL/min, the C_a of foams were 0.0063, 0.0094, and 0.0125, respectively, all more than 10^{-3} , so the removal efficiency of nitrobenzene were more than 96%.

From Figure 4, it can be observed that the removal efficiencies of nitrobenzene by foam air were 55.63%, 46.80%, 44.91%, when the foam injection rates were 4, 6, and 8 mL/min, respectively. This is because that when the foam injection rate is small, the foam and the medium contact more excellently. Accordingly, larger amount of foam burst and more foam air formed, leading to intensively volatilization of nitrobenzene. The drag force of nitrobenzene remaining in the medium by foam flow

is more intensively by increasing foam injection rate, so as to the larger removal efficiency of nitrobenzene by foam liquid at the larger foam injection rate.

3.3. Effect of Foam Quality

Figure 5(a) shows the nitrobenzene concentration and removal efficiency of nitrobenzene in effluent liquid in different foam qualities of 88%, 91% and 94%, respectively. Figure 5(b) shows the spatial distribution of nitrobenzene in the medium in different foam qualities.

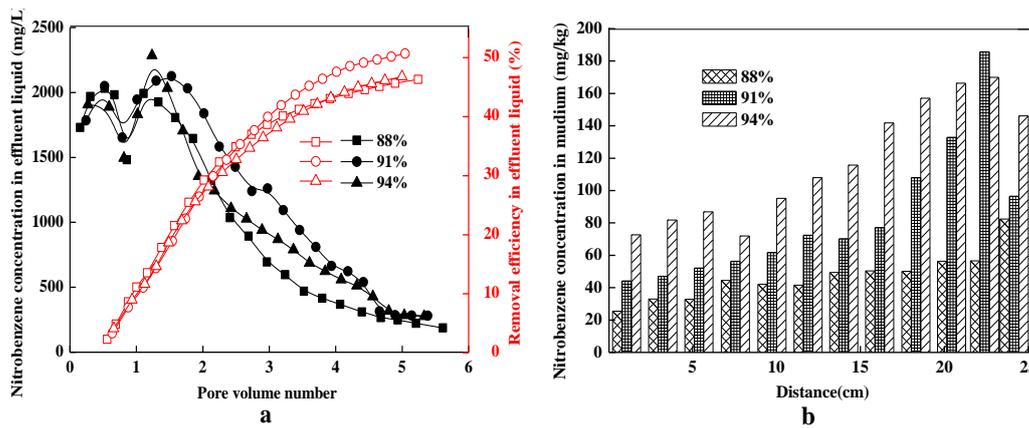


Figure 5. Influence of foam quality on the nitrobenzene concentration in effluent liquid and accumulated removal efficiency of nitrobenzene (a) and the spatial distribution of nitrobenzene in the medium (b)

It can be seen from Figure 5(a) that the concentration of nitrobenzene in foam liquid decreases with increasing of the pore volume. Figure 5(b) showed that the concentration of nitrobenzene in medium is less than 200 mg/kg. The nitrobenzene concentration in medium gradually increased with the farther away from the injection point, mainly due to enhanced mobility of nitrobenzene by foam flushing.

Nitrobenzene solubilized in the effluent liquid is indicated as mobilized. Transferring of contaminants adsorbed to the soil to the gas phase is called volatilization. In all cases, a major portion of nitrobenzene was removed by foam air. This was determined only through mass balances due to the difficulties in capturing the vapour-phase nitrobenzene. Two removal processes are involved in treating the contaminated soil. Mobilization occurs after contaminant compounds dissolved in the flushing agent are washed out along with foam flows in the contaminated soil. Because more than 80% of foam volume is air, it increases the rate of volatilization when it flows through the soil contaminated with semi-volatile compounds.

It is also interesting to note that the mobilization removal efficiency of nitrobenzene was almost equal to that for foam air volatilization. The volatilization rates were 52.08% and 49.46% when the foam quality were 88% and 94%, which were slightly larger than nitrobenzene removal efficiency by mobilization (46.3% and 46.81%). While the volatilization mechanism plays a less important for nitrobenzene removal by foam quality of 91% compare to foam qualities of 88% and 94%. The main reason is that the foam stability for 88% or 94% was weaker than that for foam quality of 91%, and the foam was easy to rupture in the process of migration. The foam liquid migrated downward under gravity and produce channel effect, resulting in reduced contact between surfactant and nitrobenzene. Thereby, the nitrobenzene removal efficiency by mobilization was larger for foam quality of 91%.

3.4. Effect of Particle Size

Figure 6(a) shows the nitrobenzene concentration and removal efficiency of nitrobenzene in effluent liquid in different sand sizes of 1~2mm, 0.5~1mm, 0.25~0.5mm, and 0.1~0.25mm, respectively. Figure 6(b) shows the spatial distribution of nitrobenzene in different medium.

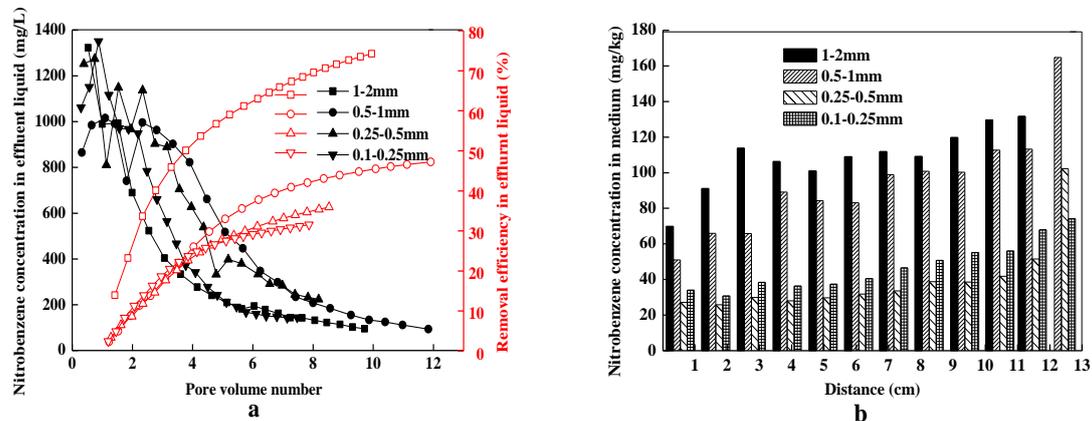


Figure 6. Influence of medium permeability on the nitrobenzene concentration in effluent liquid and accumulated removal efficiency of nitrobenzene (a) and the spatial distribution of nitrobenzene in the medium (b)

In Figure 6(b), we can see that the residual contaminant content in coarse sand increases faster than that in fine sand. It seems this technique is more effective in soil with higher porosity. The reason may be the time necessary for distribution of foam through the soil and desorption process that is much longer for soil with a low hydraulic conductivity. As shown in Figure 6(a), the removal efficiency of nitrobenzene by mobilization (effluent liquid) were 70.66%, 43.06%, 35.68% and 31.56%, respectively when the flushing volume was 8PVs.

According to the Figure 6b, the total removal efficiency of nitrobenzene decreases with the increase of particle size. When the foam injection rate is constant, the pore size of the smaller size medium is smaller, which leads to the increase of the linear migration rate of the foam in the pore. The residual stripping force of nitrobenzene is enhanced, so the foam flushing is more effective in nitrobenzene removal in the smaller particle size[18]. However, according to the literature, the larger particle size medium has higher permeability, which enhances the migration of the foam in the medium, increases its spread efficiency and increases the efficiency of oil removal[19]. The results of this study are different from those of previous studies, mainly due to ignore the volatilization of oil by foam air in previous studies. When the medium particle size is less than the foam size, a large number of foam rupture in the process of migration, the pollutants removal efficiency by volatilization is greater.

For the sand size of 1~2mm, the pore size is larger than the foam bubble size, so the rupture of foam bubble may not be the dominant mechanism in the process of foam migration, while the migration mechanism of the foam in the medium may be mainly deformation[20]. Therefore, mobilization of nitrobenzene may be the dominant mechanism in removing contaminant from contaminated columns instead of volatilization. For the sand size of 0.5~1mm, the pore size of the medium is similar to that of the foam bubbles. At this time, the mechanism of the foam migration is mainly rupture,

regeneration and deformation[20]. The amount of foam gas and the volatilization efficiency of nitrobenzene by foam flushing in the medium is more than that for sand size of 1~2mm. For the sand size of 0.25~0.5mm and 0.1~0.25mm, the foam bubble size is larger than the pore size, while the capillary force is much larger than the critical capillary force of the foam rupture, so the mechanism of the foam migration is mainly rupture, the dominant mechanism in removing nitrobenzene is volatilization. Therefore, the volatilization efficiency of nitrobenzene is greater than the mobilization (effluent liquid) efficiency. The migration rate of the foam in the two smaller particle medium is much smaller than that of the other two kinds of medium. So the time necessary for distribution of foam through the soil and desorption process that is much longer for soil with a low hydraulic conductivity. It seems this technique is more effective in soil with higher porosity (0.1~0.25mm and 0.25~0.5mm).

4. Conclusions

For water, SDS solution, and SDS foam, the total removal efficiencies of nitrobenzene in soil were 34.86%, 32.80% and 97.35% respectively. Compared with water and SDS solution, SDS foam enhanced the nitrobenzene removal efficiency apparently. The foam injection rate and the foam quality have little effect on the total removal efficiency of nitrobenzene in the soil. However, the nitrobenzene removal through mobilization and volatilization is affected by foam injection rate by affecting the contact time of the foam liquid with the pollutants and the dragging effect. The foam quality determined the volatilization and mobilization efficiency of nitrobenzene by affecting the stability of the foam. For coarse sand with size of 1~2mm, the volatilization efficiency of nitrobenzene is less than that by mobilization mechanism. For fine sand with size of 0.1~0.25 mm, the volatilization efficiency is greater than the mobilization efficiency. This technique is more effective in soil with lower hydraulic conductivity and higher porosity.

Acknowledgments

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