Applicability of an anionic-nonionic surfactant in p-cresol contaminated soil washing: Finding the optimal mixing ratio

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Abstract

In this study, the parameters influencing p-cresol removal efficiency in soil washing method were investigated. Primarily, extraction efficiencies of three Tween series surfactants (Tween 20, Tween 60, Tween 80) with 10 mM concentration were compared. Tween 80 showed the best results since its value (55%) was 4% and 13% higher than that of Tween 60 and Tween 20. The impact of mixed surfactant on extraction rate was examined by employing a mixture of Tween 80 and one anionic surfactant (sodium dodecyl sulfate) with different molar ratio as the main washing solution. The results denoted that the molar ratio of 3:2 (SDS:Tween80) could enhance the extraction rate up to 38% compared to using SDS and Tween 80 alone. Regarding the initial p-cresol concentration in the collected sample, the cleanup level (390 mg/kg) could only be achieved using the mixed-surfactant. Thus, the minimum required surfactant concentrations to hit the target level was calculated to be 3.54 g/L of Tween 80 and 2.105 g/L of SDS (molar ratio of 0.27 SDS:Tween80). Studying the role of surfactant concentration indicated that its increment from 10 mM to 20 mM, which is way above all the reagents' critical micelle concentration (CMC), does not affect the removal rate considerably. The same results were obtained comparing the effect of washing time in three different levels (30 min, 60 min and 90 min). However, temperature showed to be a more significant parameter as it could enhance the results up to 20% (for SDS).

Keywords Surfactant-Enhanced Remediation · Mixed surfactant · Extraction efficiency · P-cresol · Soil contamination

Introduction

Phenolic compounds are among the most concerning environmental pollutants due to their widespread use, refractory in nature and their short and long-term effects on human health [1]. Cresols are isomeric substituted phenols with methyl groups at one of the ortho, meta or para positions relative to the OH-group [2]. These compounds have been categorized by the EPA as Group C, possible human carcinogens [3–5]. P Cresol or 4-methylphenol, as a derivative of phenol and an isomer of cresol has been believed to exert a substantial influence on biological systems in uremia and can cause nervous system depression [6, 7].

The concentrations of cresols in soil and water are usually low. Because of their relatively rapid rate of biodegradation, only in areas where petroleum products were spilled or produced, could higher levels of cresols be detected [8]. These

areas have to be precisely identified and cleared, otherwise the contaminants will leak and cause great damage to the groundwater. Moreover, due to the high toxicity of cresols, finding a proper method to efficiently remove them from contaminated soil is of great importance.

There are various techniques to extract Hydrophobic organic compounds (HOCs) such as cresols from soils. Ex-situ soil remediation technologies are getting more interest despite the expense and hassle of excavation, while in-situ treatment methods are often time-consuming and expensive [9]. Among the ex-situ methods, Surfactant-enhanced remediation (SER) has been suggested as a promising soil washing technology [10]. Surfactants are chemical compounds used widely for extracting hydrocarbons from soil. Surfactants enhance the solubility of HOCs by partitioning them into the hydrophobic cores of surfactant micelles, thereby enhancing the remediation efficiency [11].

In general, there are four types of surfactants according to the composition of the polarity of the head group: cationic, anionic, non-ionic and amphoteric. Cationic surfactants are often toxic and have reported to be sorbed easily onto the soil particles [12]. Thus, they are usually considered as unsuitable extracting agents and are not selected in remediation works

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[13]. Anionic surfactants account for about 65% of all surfactants manufactured [14]. Too hard subsurface water may decrease the effectiveness of anionic surfactants because they may precipitate and reduce soil hydraulic conductivity [15]. Nonionic surfactants generally have smaller critical micelle concentrations (CMC) than ionic surfactants and are considered to be good solubilizers of hydrophobic substances [13].

According to the literature, non-ionic surfactants have attracted more attention than other types and among them, Tween series, Brij series and Triton X series have been widely studied for the extraction of HOCs [13, 15–19]. Some studies suggest that nonionic surfactants are non-toxic and biodegradable and can show better results compared to other surfactants [20, 21]. Some studies, however, worked on anionic surfactant as the main washing fluid for soil remediation [22–24]. Compared to using single surfactant, it is believed that using a mixture of different surfactants, can cause synergism to happen [15]. The synergistic solubilization of HOCs in anionic/nonionic mixed surfactant has been observed in various research [25–28]. This synergism is because the mixed anionic/nonionic surfactants generally show smaller CMCs, higher cloud point and lower Krafft point [29].

The SER technique is influenced by several factors such as type of surfactant, surfactant concentration, washing time and agitation speed. The objective of this study is to investigate this technology on p-cresol contaminated soil as the studies in this area is very limited in the literature. The effect of type of surfactant (anionic, non-ionic and mixed) on extraction efficiencies was first evaluated and then other parameters including surfactant concentration, washing time and temperature were further examined.

Material and method

Material

The washing agents in this study are Tween 20, Tween 60, Tween 80 and sodium dodecyl sulfate (SDS). They were all purchased from Merck Company with high purity. The properties of selected surfactant are listed in Table 1 [28, 30, 31]. In

this study, all the solutions were prepared with two different concentrations of 10 mM and 20 mM. To achieve these concentrations, the specific amount of each surfactant was scaled and poured into a 50 ml Pyrex flask. Then, distilled deionized water was added and the mixture was agitated for 2 min by a magnetic stirrer. Deionized water had electrical conductivity (EC) of 9–12 $\mu\text{S}/\text{cm}$, total dissolved solids (TDS) of 4.5–5.5 ppm and pH of 6.5–7.5. Distilled deionized water was used to prepare washing solutions containing different concentrations of surfactants.

Soil sampling

P-Cresol-contaminated soil was sampled from a suspected area of contamination (R6 point in Fig. 1) in “Terminals and Tanks Petrochemical Company” located in Bandar-e Emam, south of Iran. According to preliminary analysis, the identified area was considerably contaminated with p-cresol up to 2-m depth. The sample was collected at the depth of 100 cm, stored in a sealed plastic bag and transferred to the laboratory. Before conducting the main experiments, the sample was placed in a refrigerator at 4 °C. The physicochemical properties of the soil are summarized in Table 2 [32–36]. According to chromatography test, the initial concentration of p-cresol in the sample was 2817 mg/kg. For comparison, the cleanup levels of p-cresol in residential soil regulated by different environmental departments are presented in Table 3.

Soil washing

For each experiment, 5 grams of sample was added to 50 mL of surfactant solution in a 100 mL flask. An overhead electric stirrer was used to wash the samples at 150 rpm for 30 min and under the temperature of 20 °C [39]. To examine the impact of temperature and washing time, the tests were performed under different temperature (20 °C, 40 °C and 80 °C) and different time (30 min, 60 min and 90 min). A heater plate was used to provide the demanded temperature.

Following the washing stage, each sample was allowed to precipitate for half an hour and then was centrifuged at 5000 rpm for 30 min to completely separate the solution from

Table 1 Physico-chemical properties of used surfactants in this study

Surfactant	Molecular formula	Chemical name	CMC (mM)	Molecular weight (g/mol)	Hydrophile–lipophile balance (HLB)
SDS	$\text{C}_{12}\text{H}_{25}\text{OSO}_3\text{Na}$	Sodium dodecyl sulfate	8.4 ^b	288 ^b	40 ^c
Tween 20	$\text{C}_{58}\text{H}_{114}\text{O}_{26}$	Polyoxyethylene (20) Sorbitan monolaurate	0.05 ^a	1228 ^a	16.7 ^a
Tween 60	$\text{C}_{64}\text{H}_{126}\text{O}_{26}$	Polyoxyethylene (20) Sorbitan monostearate	0.023 ^a	1311.7 ^a	14.9 ^a
Tween 80	$\text{C}_{64}\text{H}_{124}\text{O}_{26}$	Polyoxyethylene (20) Sorbitan monooleate	0.043 ^a	1309.7 ^a	15 ^a

^a [28], ^b [31], ^c [30]

Fig. 1 Location of sampling in terminals and tanks petrochemical company located in Bandar-e Emam

solid phase [40]. Prior to analysis, the collected supernatant was filtered through a 0.45 μm pore size PTFE filter using a plastic syringe [37, 41].

As a preliminary study, three nonionic surfactants (Tween 80, Tween 60, Tween 20) were selected as the main surfactant and their extraction efficiencies were compared. The concentration of washing solution was opted to be 10 mM and the temperature was maintained at 20 $^{\circ}\text{C}$. All the samples were washed for 30 minutes.

In the next step, the effect of mixing Tween 80 with one anionic surfactant (SDS) on extraction rate was examined. Soil samples were washed with each mixture and their removal rates were measured. The concentration of solution was 10 mM and to validate the results, the same experiments were conducted with 20 mM.

To evaluate the effect of mixing Tween 80 and SDS on removal rate, different washing solution with different molar ratio ranging from 0 to 1 were prepared. Molar ratio in this study is defined by the following equation:

$$\text{MolarRatio} = \frac{\text{mole}(\text{Tween80})}{\text{mole}(\text{Tween80}) + \text{mole}(\text{SDS})}$$

All the experiments were performed in duplicate and the average value was utilized for the following statistical analysis.

Table 2 Physiochemical characteristics of the sample

Property	Value	Source
Moisture content	2.11%	[32]
PH	7.45	[33]
Organic Content	0.96%	[34]
EC ($\mu\text{S}/\text{cm}$) (20 $^{\circ}\text{C}$)	3.11	[34]
Clay (<0.005 mm)	0.44	[35]
Silt (0.005–0.075 mm)	0.03	[35]
Sand (0.075–4.75 mm)	99.53	[35]
Fine gravel (>4.75 mm)	0	[35]
USCS classification	Sand	[36]
P-cresol Concentration	2817 mg/kg	

Sample analysis

Reversed-phase high performance liquid chromatography (HPLC) was used to determine the concentration of p-cresol in each sample. HPLC analysis was carried out by employing a modular Shimadzu LC-10 system equipped with a LC-10AD pump, a CTO-10A column oven, a SPD-10A UV-DAD detector with wavelength of 274 nm, a CBM-10A interface, a FLD detector and a LC-10 Workstation. The mobile phase for HPLC was set as a mixture of acetonitrile: acetate Buffer (30:70; v/v). The buffer solution was prepared by dissolving 13.6 g sodium Acetate trihydrate and 5.8 ml glacial acetic acid in 1000 ml of purified water [42]. Injections were performed with the use of a 20 mL loop into a C18 reverse-phase column at a flow rate of 0.3 mL min^{−1} [37, 38].

Results and discussion

Surfactant type

The comparison of using tween group series as washing agent in p-cresol extraction is illustrated in Fig. 2. It could be seen that the washing solution with Tween 80 achieved the maximum removal rate. The corresponding value (55%) was 4% and 13% higher than the values for Tween 60 and Tween 20, respectively. This could be attributed to the hydrophobic tail length of the tween series. The hydrophilic heads of the three examined tween series surfactants are the same and they can be differentiated by their hydrophobic tail. As it could be seen

Table 3 Permissible concentration of p-cresol in residential soil

Concentrations of p-cresol (mg/kg)	Standards
310	Arizona
310	Wyoming
300	Florida
393	EPA
310	IRIS

Data are taken from [37, 38]

Fig. 2 Comparison of Tween group series in removing p-cresol from soil

from the structure of the surfactants (Fig. 3), tween 80 and tween 60 have similar length of carbon chain tail which is longer than that of tween 20. According to the literature, increasing surfactant tail length will lead to a rise in micellar aggregation number and also micellar diameter that could enhance solubilization [43]. The difference between the efficiency of tween 60 and tween 80 may be attributed to the one double bond in the alkyl chain structure of tween 80 which has a great effect on the solubilization capacity of tween surfactants to water [44].

It can also be observed that all the three Tween series solutions are much more effective in extracting p-cresol from soil than was tap water. This is due to the surfactants molecules that adsorb on the surface of the contaminant and cause a repulsion between head group of the surfactant molecules and the soil particle [31].

In the literature, there were some studies comparing the effectiveness of Tween group surfactants on soil remediation. One study reported higher PCE removal using Tween 80 than that using Tween 40 [16]. Another study indicated that the desorption rate of three PAHs (Fluoranthene, Benzantracene, Pyrene) when Tween 80 is used is similar to that when Tween 20 is applied [45]. The same authors showed that Tween 80 is more efficient than Tween 20 when removing three other PAHs from soil (Anthracene, Benzo[a]pyrene, Phenanthrene) [46].

In the next stage, the effect of using a mixed anionic-nonionic surfactant with different molar ratio was examined. As Fig. 4 shows, the relationship between Tween 80 molar ratio and removal efficiency has a strong compliance with third order polynomial pattern. Regression formulas and correlation coefficients are presented in Fig. 4. Local Maximum point could be calculated using these equations. It could be seen that 0.4 is the most efficient molar ratio that leads to an extraction rate of 91% for 20 mM and 89% for 10 mM. The

corresponding mass ratio of SDS/Tween80 with respect to molar weights of the surfactants will be 0.33 (1:3).

In the literature, different anionic/nonionic ratios have been reported as the optimum mixing ratio for soil remediation. One study presented 1:1 mass ratio of SDBS/Tween80 as the best mixing ratio for pNCB desorption [29]. Another study concluded that for PAHs desorption, 1:9 mass ratio of SDBS/Tween80 is the best option [47]. Lee et al. suggested that 1:1 volume ratio of DSOL/Tween60 is the promising ratio for PCE removal from sandy soil [28].

Considering 393 mg/kg p-cresol as the permissible cleanup level (Table 2), the minimum acceptable extraction efficiency of the sample is 86%. Figure 4 indicates that this rate is achievable under using 0.22–0.62 molar ratio for 20 mM surfactant concentration and 0.27–0.55 molar ratio for 10 mM. The current approach, however, is to minimize the total dosage of surfactant as it increases the operating costs [16]. Thus, the minimum required surfactant concentration to hit the target value for 5-gram sample can be achieved at 0.27 molar ratio (for 10 mM) with the corresponding concentrations of 3.54 g/L of Tween 80 and 2.105 g/L of SDS.

In Fig. 5 a comparison between three different washing solutions was made. It can be observed that using a mixture of SDS:Tween80 (3:2) as the main washing agent is far more efficient than using SDS or Tween 80 alone as the mixed surfactant could enhance their removal rate by 30% and 35% respectively (for 10 mM). This may be attributed to the co-aggregation of hydrophobic chains of the two surfactants that reflects a more rigid cresol microenvironment in the mixed micelles relatively to its microenvironment in the single surfactant micelle [48]. In addition, it is reported that using mixed-surfactant solutions may improve the performance of the soil washing of by increasing the bioavailability and biodegradation of nonaqueous-phase organic pollutants and reducing the level of surfactant pollution [49].

Similar results were obtained for PCE removal from sandy soil, which showed that the mixture of anionic (DOSL) and nonionic (Tween 60) surfactant was 35–38% more efficient than the anionic or nonionic surfactant alone [28].

Concentration

To study the role of surfactant concentration on removal process, samples were washed using 20 mM concentrations of different washing agents and the results were compared with that of 10 mM. It should be noted that the selected concentrations are well above all the reagents' critical micelle concentration (CMC).

Figure 6 compares the extraction efficiencies of five different washing solutions with 10 mM and 20 mM concentration.

It could be observed that the difference in the results of the studied concentrations varies between 4% (for mixed surfactant) and 7% (for SDS). This is in compliance with previous studies that show the positive effect of surfactant concentration on extraction rate. As the surfactant concentration is increased beyond the CMC, surfactant molecules aggregate to form micelles with a hydrophobic core. With an increase in the number of micelles, surface and interfacial tensions both drop, enhancing the desorption of contaminant from the soil particles [50].

However, Fig. 6 shows that increasing the surfactant concentration from 10 mM to 20 mM does not lead to a significant rise in extraction rate. This indicates that although removal ratio is increased with concentration, there is an inflection point for effectiveness. According to the literature, solubilization percentage may drop after a certain point [11].

Fig. 3 Chemical structure of Tween series surfactants

Fig. 4 The relationship between Tween 80 molar ratio and removal efficiency

Similar results were obtained in [45] which reported that the increase in the concentration of Tween20 and Tween80 (ranging from 10 to 30 g/L) does not make a great increase in the results. Another study denoted that the increment in Tween80 concentration up to 5 g/L (= 3.8 mM) would enhance the removal rate sharply, but after that point, further increase of concentration does not lead to appreciable improvement in the removal [11]. Gitipour et al. reported the same results examining the effect of surfactant concentration on cresol removal efficiency. They suggested that a rise in concentration of SDS and Triton X-100 up to 3% would enhance the removal ratio, but after that point this effect disappears [38].

Fig. 5 The comparison between a nonionic surfactant (Tween 80), an anionic surfactant (SDS) and a mixed surfactant (SDS:Tween80 = 3:2)

Time

To evaluate the importance of washing time, experiments were performed in three different time: 30 min, 60 min and 90 min. In these experiments the temperature was fixed at 20 °C and surfactant concentration was 10 mM. Four washing agents including SDS, Tween 80 and SDS: Tween80 (3:2) and tap water were used for comparison.

Results revealed that increasing washing time by an hour only enhanced the results in the range of 3.5- 5% (Fig. 7), which is not considerable. The highest removal rate (91%) was achieved for soil washing with mixed surfactant for 90 minutes. The effect of washing time was more or less the same

Fig. 6 The effect of surfactant concentration on removal rate

for soil washing with tap water. It could be concluded that in remediation of coarse-grained soil, washing process could be efficiently done within 30 minutes. Han et al. came to the same conclusion that the optimal washing condition for crude oil contaminated soil can be reached in 30 minutes [51]. However, for more fine-grained soil, washing time plays more important role and the upward trend for extraction rate could be continued up to 60 minutes [11, 52].

Temperature

The impact of temperature on results was examined by maintaining other factors constant and washing the samples under the temperature of 20 °C, 40 °C and 80 °C. SDS, Tween 80 and SDS:Tween80 (3:2) were utilized as the washing agent with 10 mM concentration and the samples were washed for 30 min. The results indicated that temperature is a significant parameter in p-cresol removal process as its increment raised

the removal efficiency considerably. This could be attributed to the fact that para cresols are semi volatile organic compounds and high temperature could make them vaporize. Moreover, increasing the temperature would lead to a rise in diameter of micelles, that would enhance solubilization.

This is consistent with the findings of other studies that had found this parameter important in soil washing [26, 51]. Among the different washing solutions, the improvement in the results for SDS was more dramatic. As Fig. 8 presents, a 60 °C rise in temperature could increase the extraction rate by 8%, 13%, 20% and 10% for water, Tween 80, SDS and SDS:Tween80 (3:2) respectively.

Conclusion

In this study, the effect of type of surfactant, concentration, washing time and temperature for p-cresol contaminated soil

Fig. 7 The effect of washing time on removal rate

Fig. 8 The effect of temperature on removal rate

washing was investigated. The results revealed that among the three studied Tween series surfactant, Tween 80 was the most efficient one as its removal rate (55%) was 13% and 4% higher than that for Tween 20 and Tween 60 respectively. Soil washing with a mixture of Tween 80/SDS with different molar ratio demonstrated that the proportion of 3:2 (SDS: Tween80) leads to the highest extraction rate, which will be 35–38% greater than that using single surfactant. Regarding the level of contamination, cleanup level for the sample could only be achieved using the mixed surfactant. In this regard, the minimum required Tween80 and SDS was calculated to be 3.54 g/L and 2.105 g/L respectively. The parameter of washing time and temperature were also evaluated in this research. Time of washing in the studied range (30–90 min) demonstrated to be a less affecting parameter as it enhanced the removal rate only up to 5%. However, temperature (ranging from 20 °C to 80 °C) showed to be an influencing factor. Among washing agents, SDS was more sensitive to the changes in temperature and presented 20% rise after increasing the temperature from 20 °C to 80 °C.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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