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Recycling of solvent used in a solvent extraction of petroleum hydrocarbons contaminated soil

Guozhong Wu[a,c], Xingang Li[b], Frédéric Coulon[c], Hong Li[b], Jingyan Lian[d], and Hong Sui[b*]

[a]*School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, China;*

[b]*National Engineering Research Centre for Distillation Technology, Tianjin 300072, China;*

[c]*Centre for Resource Management and Efficiency, Sustainable Systems Department, School of Applied Science, Cranfield University, Cranfield MK43 0AL, UK.*

[d]*School of Chemistry and Chemical Engineering, Tianjin University of Technology, Tianjin 300191, China;*

[*]Corresponding author:

Email: suihong@tju.edu.cn

Tel: 86-022-27404701

Fax: 86-022-27404705

Abstract:

The application of water washing technology for recycling an organic composite solvent consisting of hexane and pentane (4:1; TU-A solvent) was investigated for extracting total petroleum hydrocarbons (TPH) from contaminated soil. The effects of water volume, water temperature, washing time and initial concentration of solvent were evaluated using orthogonal experiments followed by single factor experiments. Our results showed that the water volume was a statistically significant factor influencing greatly the water washing efficiency. Although less important, the other three factors have all increased the efficacy of water washing treatment. Based on a treatment of 20 g of contaminated soil with a TPH concentration of 140 mg g^{-1}, optimal conditions were found to be at 40 °C, 100 ml water, 5 min washing time and 660 mg g^{-1} solvent. Semi-continuous water extraction method showed that the concentration of the composite solvent TU-A was reduced below 15 mg g^{-1} d.w. soil with a recovery extraction efficiency > 97%. This finding suggests that water washing is a promising technology for recycling solvent used in TPH extraction from contaminated soils.

Keywords: Solvent extraction, TU-A solvent, Water washing, Orthogonal experiment

1. Introduction

Solvent extraction is a promising technology for the treatment of oil contaminated soil, in which petroleum hydrocarbons are removed from soil using an individual solvent or mixture of solvents [1]. Typically, solvents used within this field include exhaustive organic solvents [2, 3], surfactant aid aqueous solution[4], non-toxic and biodegradable agents [5, 6], and supercritical [7] and subcritical fluids [8]. A great deal of research on the development of composite solvents for removing PAH from soil has been carried out. However, most of the studies were conducted at laboratory scale and the high performances of extraction reported were achieved in controlled conditions such as extraction temperature ranging between 70 and 100 °C. In addition, previous studies on the characterisation of natural soil organic matter (SOM) demonstrated that component molecules of SOM can be altered [9] and transitions between the glassy and rubber phase of soil can occur by the addition of heat[10]. To the best of our knowledge, few extractions with high efficiency have been performed at room temperature and the issue of energy cost needed should be taken into account for implementing this technology at field scale. Additionally, the extraction time reported varied greatly ranging from 30 min to 48 h [3, 4, 11, 12] and comparison under the same conditions should be performed to determine if solvent extraction can remove contaminants faster than other technologies. Hence, there is an increasing demand for extraction with less energy consuming and faster mass transfer rate.

The problem of secondary pollution needs also to be addressed as some of the solvents used may persist in the remediated soil and pose an environmental risk due to their low biodegradability. Most of the studies have focused on the extraction efficiency, but less attention was paid to the solvent regeneration. Some investigations have been conducted on the recovery of solvent from the extracted oil; for instance, the surfactant and vegetable oil were regenerated by activated carbon adsorption[4, 13], acetone and ethyl acetate were regenerated by distillation [2] and cyclodextrin was reused by liquid-liquid extraction [14]. However, very limited information was found for regenerating the residual solvent in the soil as it is difficult to recover its original properties for purpose of reusing. To handle the issue of liquid-solid separation, Soxhlet and solid phase extraction are alternative techniques, but both are time consuming and generate secondary pollution since organic solvents are used. Evaporation and centrifugation are effective approaches and avoid the problem of secondary pollution, but they are energy intensive or inapplicable for the inflammable and explosive solvents. These challenges raise the demand for developing simple cost-effective techniques and alternative approaches promoting use of green chemistry in order to recycle and reuse organic solvents.

In our previous research [15], the TU-A solvent, an organic composite solvent consisting of hexane and pentane (4:1 v/v) was used for extracting total petroleum hydrocarbons (TPH) from a contaminated soil in which the initial TPH concentration ranged between 0.1 and 0.14 g g^{-1} d.w. soil. After solvent extraction at a soil-solvent

ratio of 1:4 (w/w), the TPH removal efficiency after 5 and 15 min at room temperature was 80% and 95% respectively, and the concentration of the composite solvent remaining in the soil was between 330 and 660 mg g^{-1}. Two conclusions were reached in these preliminary results: (i) solvent extraction using TU-A solvent is a promising technique for removing petroleum hydrocarbons from soil which is fast and effective; (ii) the amount of solvent remaining in soil may pose an environmental risk and prevent its use at industrial scale without recycling.

Water is perceived as 'the ultimate green solvent', cheap, non-toxic and recyclable. Its disposal is regarded as benign with little affect on the environment [16]. In this work, water was proposed as a carrier to decontaminate soil from the residual organic solvent. The hypothesis was that the TU-A solvent (nonpolar molecules) distributed in the micro pores in soil would transfer from solid phase to water phase in the presence of concentration gradient, entered spaces between water clusters and associated with each other (rather than with the water molecules) in the void spaces. Both water and organic solvent were reused after separation by density differences. Therefore, the specific objectives of this study were: (i) to assess the feasibility of the water washing technology for recycling organic solvent, (ii) to identify the factors influencing washing efficiency and optimize washing conditions, and (iii) to provide information for implementing a robust remediation methodology associated with solvent extraction.

2. Materials and methods

2.1. Chemicals and soils

Hexane, pentane and tetrachloromethane (CCl_4) were purchased from Tianjin Jiangtian Technology Co. Ltd., China. TU-A solvent was a mixture of hexane and pentane (4:1 v/v). All the solvents used were analytical grade.

The uncontaminated soil used was obtained from a typical oil field in China. Physicochemical properties of the soil were as follows: pH: 8.0, SOM: 0.7%, cation exchange capacity: 164.0 $mmol \cdot kg^{-1}$, moisture content: 1.7%, bulk density: 1.4 $g \cdot cm^{-3}$, and tap density: 2.8 $g \cdot cm^{-3}$. The fraction of clay, sand and silt was 25.3%, 40.4% and 34.3%, respectively. The soil was air-dried and homogenised by screening through a 2-mm sieve to remove extra vegetable roots and stored in glass desiccators before use.

2.2. Optimization of water washing condition

The water washing conditions were optimized using multifactor orthogonal experiment followed by single factor experiment. Previous studies on the soil washing using aqueous surfactant solution demonstrated that the factors largely influencing the washing efficiency were water volume, water temperature, washing time and initial concentration of solvent [12]. In this study, the four parameters were studied at three levels using $L_9(3^4)$ orthogonal array as shown in Table 1. In conventional full factorial experimental design, the number of experiments required to run is $3^4 = 81$, which was reduced to 9 by orthogonal design, offering a great advantage in terms of experimental time and cost. Three statistical coefficients (K, R and F-ratio) were used

to evaluate the orthogonal data. K is the sum of the concentrations of residual solvent extraction mixture in soils for each impact factor at each level, which was used to assess the optimal level of each factor so as to determine the optimum combination of the experimental condition. The lowest the K value, the highest solvent removal efficiency of the factor considered is. The extreme difference R is a parameter representing the fluctuation degree of the washing efficiency in accordance with the variety level of impact factor. The larger the R value, the more obvious influence of the corresponding factor is. F-ratio was applied to evaluate whether the impact factors were statistically significant or not, which was calculated using the method previously described by Gonder *et al* [17] and then compared with the critical F value which could be found in most of the statistics and experimental design books [18].

Briefly soil samples (20 g) were weighed into a 250 ml conical flask and different volume of TU-A solvent were added to achieve the level concentration as shown in Table 1. The flasks were sealed, shaken manually for 5 min and stored at room temperature for 15 min. Water was then added to the flask and stirred on the magnetic stirrer for 1, 3 and 5 min as described in Table 1. The mixture was transferred to a separating funnel, in which the TU-A solvent was extracted from the soil by water. After the solvent float on the top layer, the supernatant was discarded while the bottom slurry was received in a beaker and analysed by ultrasonic extraction.

2.3. Volatilization of solvent from soil

Volatilization of TU-A solvent from soil during solvent recycling process was estimated by mass balance for each experiment in the $L_9(3^4)$ orthogonal array. Volatilization during the ultrasonic extraction (section 2.5) was also considered as it accelerated the loss of solvent by evaporation. Therefore the overall loss of solvent by volatilization equals to loss during the water washing process and the ultrasonic extraction. The difference between the known mass of all the materials added (i.e. 20 g of soil, 6.6 g of solvent, 20g of water and 31.9 g of CCl_4 were added in experiment 1) and the actual overall mass measured at the end of ultrasonic extraction (i.e. 78.2 g in experiment 1) was estimated to be the mass loss by volatilization.

2.4. Semi-continuous concurrent water extraction

The solvent extraction soil system was washed in a water wash column (Figure 1a), which was made of glass and fitted with water distribution tube, alternative water inlet and overflow outlet. Soils (500 g) were weighed into a 2500 ml premix bottle and 500 ml composite solvent was added. The bottle was sealed with corker stopper with parafilm wrapped around. The soil was saturated with solvent after sorption for 15 min. Water (1 L) was added followed by agitation for 5 min on a magnetic stirrer. The water extraction process is shown in Figure 1b. From the top of the water wash column, the mixture of solvent, soil and water (60 $ml \cdot s^{-1}$) was fed through a soil feed tube that was a glass circular tube with an inverted funnel at the end. A water distribution tube that was perforated with regularly spaced holes was fitted at the

middle of the water wash column, through which water (16.7 $ml \cdot s^{-1}$) was fed into the column. Three phase layers and two interfaces were formed in the column. The solvent floated at the uppermost layer and overflew (5.6 $ml \cdot s^{-1}$) into the solvent recycling bottle. The interface between the solvent mixture and the lower emulsion layer was kept below the overflow outlet to minimize the loss of soil. The mixture of soil and water was discharged at the bottom of the column and received in a slurry collection bottle. Once the slurry settled, water was pumped back into the water wash column. Since the whole system was sealed in order to reduce volatilization of the solvent mixture, a connection between the premix bottle, water wash column and the solvent recycling bottle was made with rubber tubes to facilitate the feed and overflow.

2.5. Analytical Method

The concentration of residual solvent mixture in soil after water washing was determined by ultrasonic solvent extraction followed by chemical analysis on gas chromatography (Perkin Elmer AutoSystem GC) equipped with a flame ionization detector (FID) (Figure 2). The slurry was extracted with 20 ml of CCl_4 and sonicated for 25 min at 20 °C. The supernatant was decanted into a Teflon centrifuge tube. After centrifugation for 5 min at 750 rpm, the supernatant was passed through a filter column fitted with receiver tube. Extract (5 ml) was decanted into a GC vial and stored in darkness before GC analysis to prevent photodecomposition of CCl_4. The

residue soil after ultrasonic extraction was dried in oven for 2 h at 120 ºC after volatilization for 24 h in a ventilating cabinet. The beaker was washed, dried and weighed again after discarding the dry soil.

For GC-FID analysis, splitless injection with a sample volume of 1 µl and the injector temperature of 140 ºC were applied. TPH were separated on a PE-1 capillary column (50 m × 0.2 mm internal diameter) with nitrogen as a carrier gas. The oven temperature was increased from 40 ºC to 75 ºC at 6 ºC min^{-1} and kept at this temperature for 20 min. The FID was operated at 140 ºC. External multilevel calibrations were performed using CCl_4/TU-A standards, concentration of TU-A ranging from 0.03 to 0.21 g ml^{-1}.

3. Results and discussion

3.1. Multifactor orthogonal experiments

The standard orthogonal matrix for optimising the water washing conditions including water volume (A), water temperature (B), washing time (C) and initial concentration of solvent (D) is shown in Table 2. The extreme difference analysis showed that the lowest K value for each impact factor was at the second level for water volume and the third level for the other three factors. Therefore the optimum combination could be defined as $A_2B_3C_3D_3$. The coefficient R values of the various parameters showed the following trend: water volume > washing time > water temperature > initial concentration of solvent in soil (Table 2). Water volume was the most significant

factor that influenced efficacy of water washing at 95% confidence level with F-ratio of 40.51 (Table 3). In contrast, the other factors did not significantly influence the washing efficiency as all F-ratio were lower than the critical F-value.

As shown in Figure 3, the efficiency of solvent recycling was ranging from 93% (i.e. experiment 1) to 99% (i.e. experiment 7). In addition, volatilization of the TU-A solvent was relatively low ranging from 0.4% (i.e. experiment 7) to 5.5% (i.e. experiment 2). This finding suggests that water washing can be an efficient process for removing residual solvent in soil. It also suggests that solvent extraction and recycling can be repeatedly carried out if the entire system is well sealed.

3.2. Single factor experiments

The effect of the most significant factor (water volume) on the washing efficiency was investigated by fixing the other three factors at the optimal values. The relation between the concentrations of residual solvent extraction mixture in soil and water volumes showed that the concentration of the residual solvent extraction mixture was decreased to less than 2 mg g^{-1} when the water volume increased to 3.5 times of its initial concentration (Figure 4). As expected, the concentration of residual solvent in soil declined from 7.3 to 1.7 mg g^{-1} when the water volume was increased from 30 ml to 70 ml respectively. The same trends were observed in the orthogonal experiments where the $\overline{k}$ value (average value of K) of the water volume dropped from 6.0 at level 1 to 1.5 at level 2 (Figure 5). However, the surprising increase of $\overline{k}$ up to 2.70

when the water volume was at level 3 suggests that the loss of solvent results from a concomitant effect of the water washing process and volatilization. This finding is further supported by the volatilization data. As shown in Figure 3, the higher the volume of water, the lower the volatilisation. This can be explained by the wicking mechanism identified by Arthurs *et al.* [19, 20]. The wicking behaviour is created by capillary force in the soil and occurs whenever an immiscible phase concentration gradient is present, which then contributes to volatilization rates. This mechanism was also validated by Li *et al.* [21], which demonstrated that appropriate volume of water helps the wicking action but also that too much water stops this mechanism. When large quantity of water exists, the water molecules prefer to take up the micro pores in the soil whereas the solvent molecules in the pores of larger size [22]. The capillarity was therefore weakened and the promoting of volatilization by wicking behaviour was restrained. In addition, this behaviour may be interpreted by the effect of free pores in the soil system [23, 24]. The presence of large amount of water leads to the declining of porosity factor and volatilization area, resulting in restricting of volatilization rate.

In the semi-continuous experiments, water used for extraction consisted of three parts such as the water in the premix step, the fresh water fed into the water wash column and the recycled water, total volume of which depended on the initial concentration of TU-A solvent. Results indicated that the volume of water should be at least 3 times greater than those of TU-A solvent to be regenerated (Figure 4). The concentration of

the residual solvent extraction mixture was 15 mg g^{-1} after concurrent extraction in the column, which was more than 1.5 times higher than the results in the orthogonal experiments. This finding can be explained by the fact that the fluid-solid contact time in the water wash column was too short, resulting that 'new' fluids displaced the incompletely equilibrated 'old' fluids before mass transfer completed.

A biphasic kinetic has been proposed previously to describe the desorption of hydrophobic organic solvent from the soil to the water phase [25]. Initially, a portion of solvent can be extracted by water very quickly (within few minutes) by intraparticle diffusion driven by concentration gradient, whereas the remaining fraction is more slowly or cannot be extracted. This suggests that solvent removal rate and efficiency will increase and then reach a plateau as water volume increases. No such trend was observed in this study suggesting that the slow desorption stage was not reached and the initial fast desorption stage alone could not provide sufficient information for determining the critical water volume. Nevertheless, comparison between the signal factor experiment and the orthogonal experiments showed that the residual solvent in soil was 1.7 mg g^{-1} using 70 ml of water (Figure 4) whilst the lowest concentration was 1.1 mg g^{-1} when the water used was 100 ml (Table 2). It may be concluded that enhancement of water volume above 100 ml will make no significant improvement of solvent removal rate and the critical water volume is estimated to be between 70 ml and 100 ml. Using unnecessarily excessive amounts of water is unfavourable for economic reasons taking into account the cost of subsequent

water recycling. Practically, it is a balance between the water wash efficiency and the operation cost.

3.3. Effects of other factors

Increasing water temperature and prolonging washing time were both beneficial for recycling the organic solvent as shown in Figure 5. This tendency agrees with those reported in previous works [12].

The adhesion between the solvent and the soil is weakened as the temperature increase, resulting in the decrease of the solvent viscosity, increase of its mobility and increase of its surface contact with water. Soil-compound interactions have been shown to be influenced by SOM, both in amount [26] and in nature [27]. According to the polymer model [10], when the temperature is increased, the SOM in glass phase, a highly condensed rigid structure, will be transited into rubber phase which is highly expanded structure. This transition facilitates desorption of the sequestered organic solvent because the SOM in rubber state has flexible chains owing to weak cohesive forces between chains. From this point of view, SOM will drastically influence the removal rate of solvents although not being quantified in this study due to the fact that only one kind of soil was studied. Further investigation needs to be performed to validate the mechanisms of the effects of SOM.

A strong stir that is able to overcome the forces holding the molecules in their association will accelerate desorption of organic solvent. The forces that hold molecules together include inter-particle forces, such as multi-pore interaction,

induction forces, dipole-induced dipole interactions, Vander Waals forces, and hydrogen bonding interactions [28]. Dipole-dipole interactions are strongest when the dipoles are aligned head to tail, and the strongest hydrogen bonds are formed when the two electronegative atoms and the hydrogen are collinear. Thus, the successive stir is an impediment to the formation of these interactions and the removal of organic solvent can be enhanced by reinforcing the stirring strength [12, 29].

3.4. Process treatment conceptualization

Based on these overall findings, a conceptualized process for a soil contaminated with a high concentration of petroleum hydrocarbons was developed (Figure 6). It was estimated that 6000 L solvent and 3500 L water will be required to treat one tonne of soil with an initial TPH concentration of 140 mg g^{-1}. Up to 99% of the solvent and 94.3% of water can be recycled, resulting in 7 mg g^{-1} of TPH, 15 mg g^{-1} of solvent and 200 mg g^{-1} of water remaining in the soil (Figure 6). On this basis, the net volume of solvent and water retained in soil will be 23 L and 200 L, respectively. The volume of solvent lost by volatilization will be 30 L. Bioremediation strategy can be then applied for cleaning of the resulting contaminated soil.

4. Conclusion

This research demonstrates that water washing is a promising technology for recycling and reusing organic solvent used for petroleum hydrocarbon extraction in contaminated soils. Water volume was the most influential parameter and the optimal

conditions for 20 g of soil were as follows: water volume of 100 ml, water temperature of 40 °C, washing time of 5 min and initial solvent concentration of 660 mg g^{-1}. These conditions are mild and easy to achieve, providing encouraging information for implementing solvent recycling with water at field scale. The proposed method is also more environmentally friendly, as water is an 'ultimate green solvent', which will reduce significantly residual concentration of solvent in the environment. Considering the subsequent cost of water treatment, the volume of water used is suggested to be 3 ~ 4 times of that of organic solvent to be regenerated. The water remaining in the washed soil should be recycled by filter pressing or sedimentation.

Acknowledgements

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Tables:

Table 1 Parameters and levels of the orthogonal experimental design

Impact factors	Symbol	Level			Units
		1	2	3	
Water Volume	A	20	100	200	ml
Water Temperature	B	15	30	40	°C
Washing time	C	1	3	5	min
Initial concentration of solvent	D	330	490	660	mg g^{-1}

Table 2 $L_9(3^4)$ orthogonal matrix and statistical data

Experiments	Impact factors				Concentration of residual solvent in soil (mg g^{-1})
	A	B	C	D	
1	1	1	1	1	8.1
2	1	2	2	2	6.2
3	1	3	3	3	3.8
4	2	1	2	3	1.1
5	2	2	3	1	1.3
6	2	3	1	2	2.2
7	3	1	3	2	1.7
8	3	2	1	3	4.3
9	3	3	2	1	2.1
K_1	18.1	10.8	14.6	11.4	
K_2	4.6	11.8	9.4	10.1	
K_3	8.1	8.1	6.8	9.2	
$\overline{k_1}$	6.0	3.6	4.8	3.8	
$\overline{k_2}$	1.5	3.9	3.1	3.3	
$\overline{k_3}$	2.7	2.7	2.2	3.1	
R	4.5	1.2	2.6	0.7	
Optimal Level	2	3	3	3	
Optimal combination		$A_2B_3C_3D_3$			

Table 3 Variance analyses for the impact factors in the orthogonal experiment

Impact Factor	A	B	C	D
Si [a]	32.71	2.52	10.49	0.81
DOF [b]	2.00	2.00	2.00	2.00
MS [c]	16.35	1.26	5.25	0.404
F-ratios	40.51	3.13	13.00	1.00
F_{cr} [d]	19	19	19	19

[a] Square of deviance, [b]degree of freedom, [c] mean square, [d] critical F value.
Confidence level: 95%.

Figures:

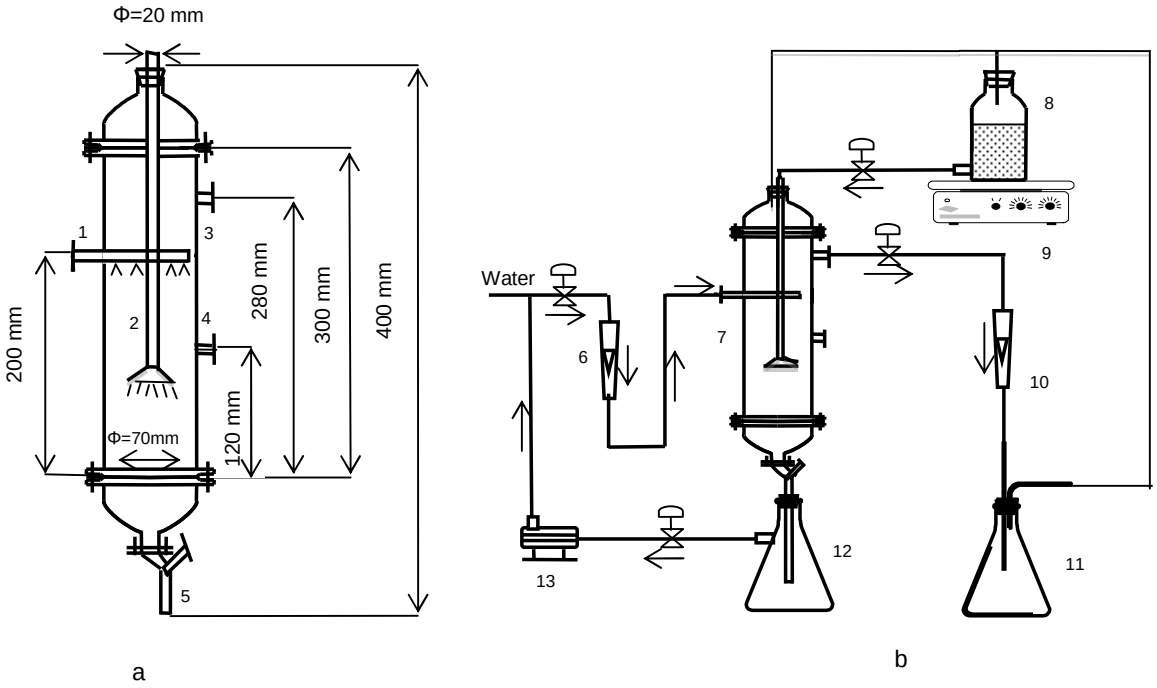


Figure 1 Experimental set-up for the regeneration of composite solvent used to extract petroleum hydrocarbons from contaminated soil. The arrows indicate the direction of the flow. (1) Water distribution tube, (2) soil feed tube, (3) overflow outlet, (4) alternative water inlet, (5) soil outlet, (6) industrial flow meter, (7) water wash column, (8) premix bottle, (9) magnetic stirrer, (10) rotameter, (11) solvent recycling bottle, (12) slurry collection bottle, (13) pump.

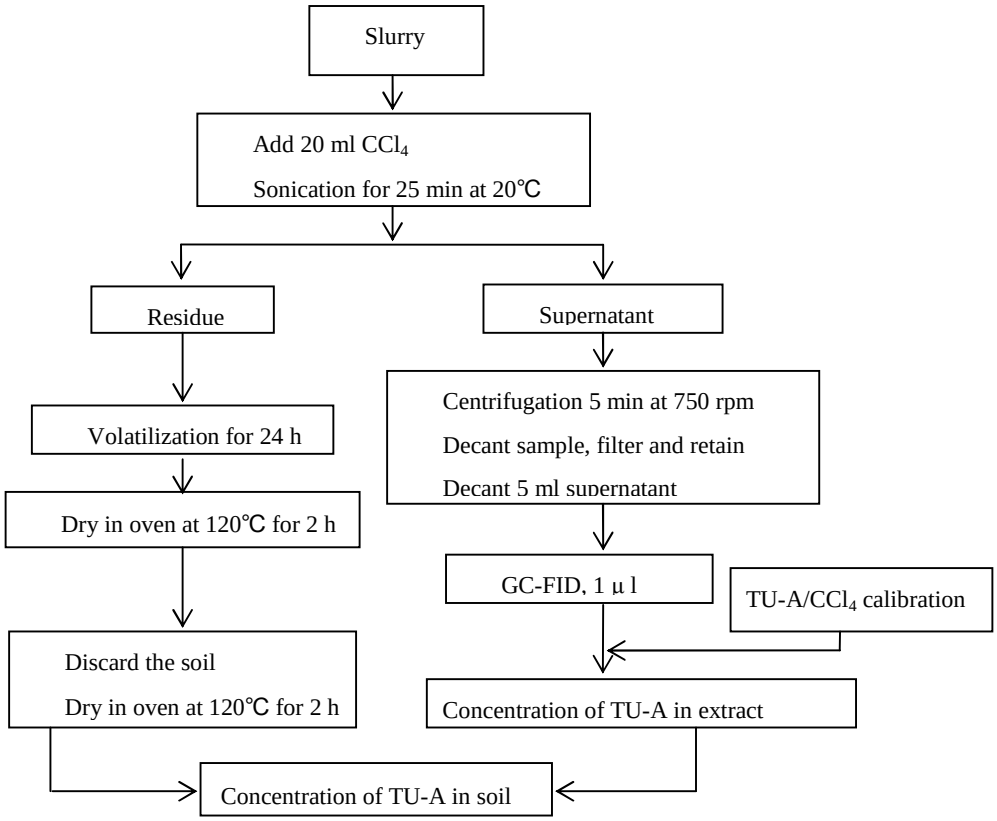


Figure 2 Schematic for analyzing residual solvent in soil after water washing

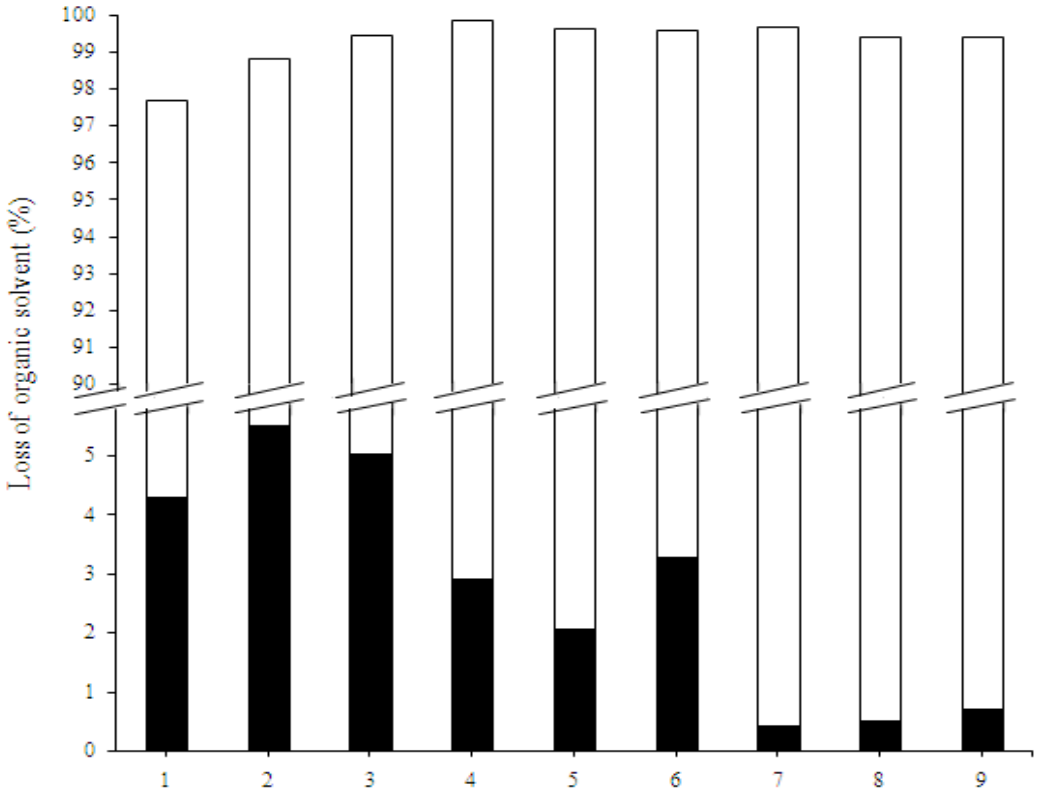


Figure 3 Percentage of organic solvent volatilized (■) and extracted by water (□) in the $L_9(3^4)$ orthogonal experiments

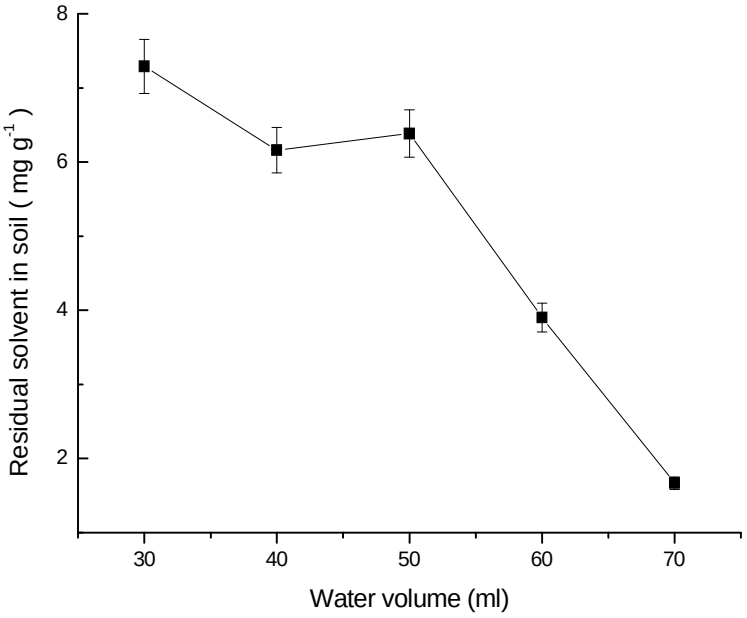


Figure 4 Relationship between concentrations of residual solvent in soil and water volumes at wash conditions of water temperature 40 °C, washing time 5 min and initial concentration of solvent 660 mg g^{-1}.

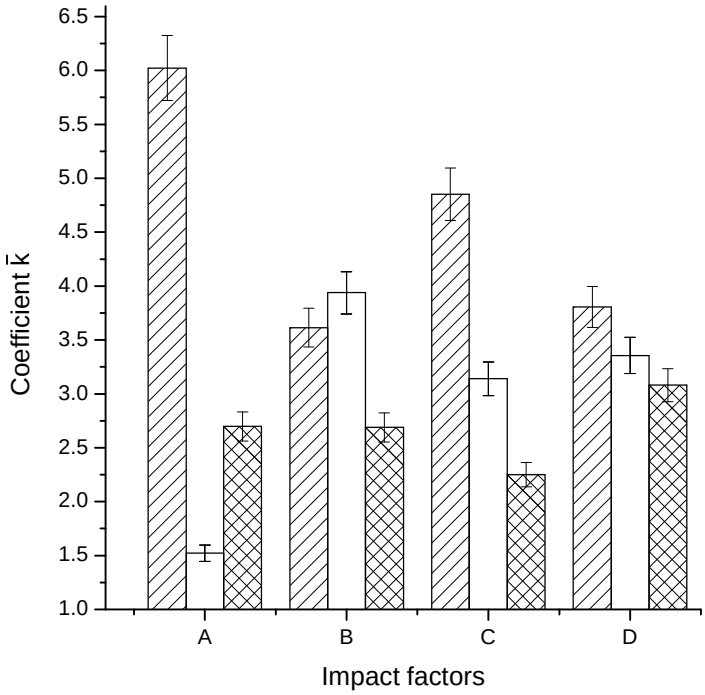


Figure 5 Effects of factors on water washing efficiency at level 1(hatched bars), level 2 (white bars) and level 3(cross bars).

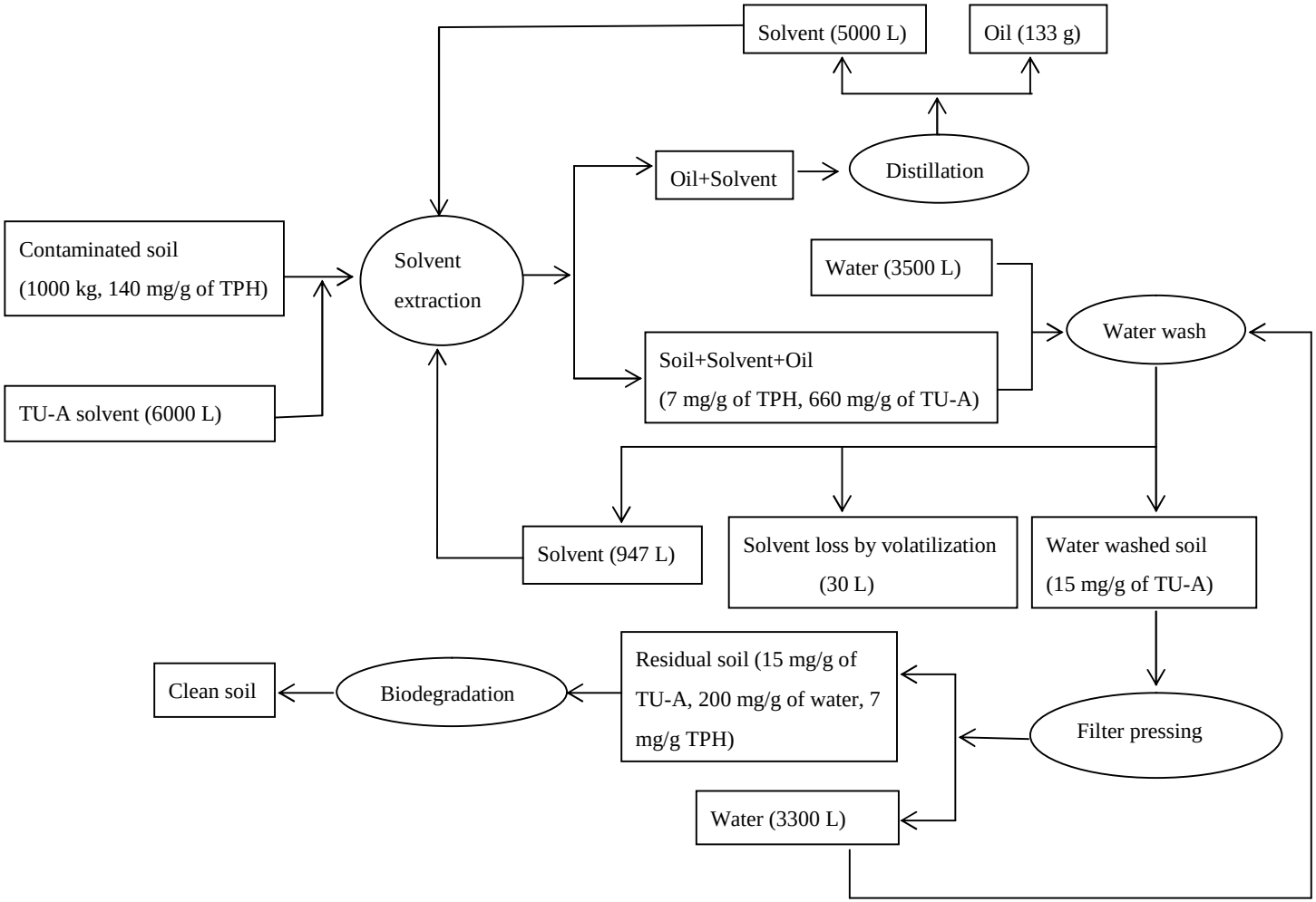


Figure 6 Proposed schematic for remediation of high concentration petroleum contaminated soil using TU-A solvent



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Recycling of solvent used in a solvent extraction of petroleum hydrocarbons contaminated soil.

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Guozhong Wu, Xingang Li, Frédéric Coulon, Hong Li, Jingyan Lian, Hong Sui, Recycling of solvent used in a solvent extraction of petroleum hydrocarbons contaminated soil, Journal of Hazardous Materials, Volume 186, Issue 1, 15 February 2011, Pages 533-539.

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