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Microwave thermal remediation of crude oil contaminated soil enhanced by carbon fiber

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Abstract

Thermal remediation of the soil contaminated with crude oil using microwave heating enhanced by carbon fiber (CF) was explored. The contaminated soil was treated with 2.45 GHz microwave, and CF was added to improve the conversion of microwave energy into thermal energy to heat the soil. During microwave heating, the oil contaminant was removed from the soil matrix and recovered by a condensation system of ice-salt bath. The experimental results indicated that CF could efficiently enhance the microwave heating of soil even with relatively low-dose. With 0.1 wt.% CF, the soil could be heated to approximately 700°C within 4 min using 800 W of microwave irradiation. Correspondingly, the contaminated soil could be highly cleaned up in a short time. Investigation of oil recovery showed that, during the remediation process, oil contaminant in the soil could be efficiently recovered without causing significant secondary pollution.

Key words: microwave heating; crude oil remediation; carbon fiber

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Introduction

During normal operation of oil fields, leakage and spillage of crude oil result in soil contamination at many sites (Lee *et al*., 1999), and thus affect environment by the alteration of essential elements of the habitat and direct toxic effects (Mansurov *et al*., 2001). To date, the technologies of soil remediation have been developed including biological treatment (Cassidy and Irvine, 1997), soil washing with surfactants (West and Harwell, 1992), air stripping (William *et al*., 1993; Buettner and Daily, 1995), thermal treatment (Bucalá *et al*., 1994; Lee *et al*., 1999), and so on. Among these technologies, thermal treatment is an effective method to remediate heavily contaminated soil (Bucalá *et al*., 1994; Kawala and Atamanczuk, 1998; Risoul *et al*., 2005). However, for conventional thermal treatment, the outside part of the soil substrate must be heated to an elevated temperature to transfer heat to the center of the substrate, which leads to an overmuch energy consumption.

Microwave technology, for its advantages of rapid, selective and simultaneous heating, is a feasible thermal method to remediate contaminated soil, avoiding overheating the surface of soil substrate. The materials are often classified into three groups based on the response to microwave: microwave conductors, microwave insulators and microwave absorbers (Sutton, 1989). The interactions between microwave absorbers and microwave result in the conversion of microwave energy into thermal energy (Jones *et al*., 2002). Materials such as many organic compounds and soil are, in general, almost transparent to microwave, so they cannot be heated directly up to high temperatures. It is usually required to remove the contaminant from the matrix. Nevertheless, microwave thermal treatment will be possible if the raw material is mixed with strong microwave absorbers (Menéndez *et al*., 2002).

Studies of thermal remediation using microwave heating enhanced by strong microwave absorbers have been performed on the soil contaminated with organic pollutants or toxic metal ions (George *et al*., 1992; Abramovitch and Huang, 1994; Abramovitch *et al*., 1998, 1999, 2003; Yuan *et al*., 2006; Liu and Yu, 2006). George *et al*. (1992) investigated the soil decontamination via microwave heating enhanced by carbon particles. The decontamination process was performed under reduced pressure and inert gas presence to prevent the combustion of contaminant. Their results showed that removal efficiencies of near 100% could be achieved for phenanthrene in simulated API (American Petroleum Institute) separator sludge and 60% for pentachlorophenol in contaminated soil using microwave heating enhanced by 40 wt.% carbon particles. The decomposition of polycyclic aromatics and polychlorobiphenyl in soil using microwave energy, with the addition of NaOH in company with Cu_2O (powdered Al,

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metal wire, etc.) to serve as both reaction catalysts and microwave absorbers was also investigated (Abramovitch and Huang, 1994; Abramovitch *et al*., 1998, 1999). The results show that the decomposition products were probably either mineralized or very tightly bounded to the matrix except that minor mixture of decomposition could be extracted. Furthermore, Abramovitch *et al*., (2003) reported that, with the addition of pencil lead or iron wire, the contaminated soil could be remediated safely to preset depths without the toxic metal ions leaching out for a long time. Yuan *et al*. (2006) investigated the microwave remediation of the soil contaminated with hexachlorobenzene (HCB) using powdered MnO_2 as a microwave absorber. Their results revealed that a complete removal of HCB was obtained with 10 min microwave treatment by the addition of 10 wt.% powdered MnO_2 and about 30 wt.% H_2SO_4 (50%).

Carbon fiber (CF) is a kind of strong and environment friendly microwave absorber (Carrott *et al*., 2001). However, the application of its intense interaction with microwave on soil remediation is rarely reported. If CF can enhance the microwave heating of soil with high efficiency, it means that the contaminated soil can be remediated rapidly through microwave process with less microwave absorbers. In this work, practical crude oil-contaminated soil was thermally remediated using microwave heating enhanced by CF. The objectives were to investigate the enhancement of CF on microwave heating of soil, and to assess the removal of soil contaminant with oil and its recovery in the microwave thermal remediation.

1 Materials and methods

1.1 Materials

Soil contaminated with high concentration of crude oil was collected from a leakage area in Liaohe Oilfield, Liaoning Province in China. The soil was dried under ambient conditions, then ground and screened through a 60-mesh sieve to remove the debris. Basic properties of the soil are listed in Table 1. The commercial polyacrylonitrile-based activated carbon fiber (ACF; diameter 3–5 μm, length 3–8 mm) and graphite fiber (GF; diameter 5 μm, length 6 mm) were obtained from Anshan Activated Carbon Fiber Company, China. They were heated in boiling deionized water for 1 h and washed to remove impurities.

1.2 Methods

The experimental setup is shown in Fig. 1. A modified domestic microwave oven (max. power 850 W) operating at 2.45 GHz with continuous adjustable power settings was used to supply microwave energy. A quartz reactor was designed to protect the contaminant from incineration and to provide a desirable reduced pressure condition, using nitrogen and vacuum pump to remove air in the reactor. The reactor installed into the microwave oven comprised two parts: a quartz pipe (outer diameter (o.d.) 50 mm, length 300 mm), with two entrances at the top, one for the thermocouple and the other for the connection to the condensation system. The bottom of the pipe allowed the entrance of nitrogen. A sample holder (o.d. 40 mm, height 60 mm) placed in the pipe. A sample of 20 g soil, mixed with CF for rapid heating, was placed in the quartz reactor. The reaction pressure and nitrogen flow rate were set to the desired values and then microwave irradiation was applied to the soil sample. After a desirable residence time, the microwave oven was turned off and the temperature was immediately measured with a sheltered type-k thermocouple as described by Kawala and Atamanczuk (1998). Then the system was cooled down to room temperature for analyzing the soil sample. The oil and water left the soil matrix during the process were condensed and collected in the condensation system of ice-salt bath. The above procedures were repeated thrice with a new sample each time, and the average readings are reported.

Table 1 Physicochemical properties of the test soil

pH	Particle density (g/cm^3)	Moisture (wt.%)	Organic carbon (wt.%)	Oil content (wt.%)	Granularity distribution (%)		
					Sand	Slit	Clay
6.5	2.12	3.21	1.38	7.81	9.3	80.7	10.0

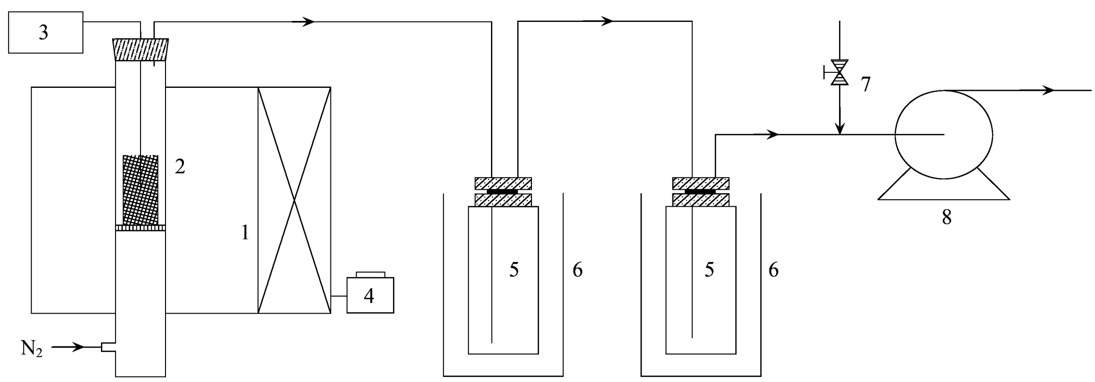

Fig. 1 Experimental apparatus for the microwave thermal remediation. (1) microwave oven; (2) quartz reactor; (3) thermocouple; (4) transformer; (5) collector; (6) ice-salt bath; (7) pressure relief valve; (8) vacuum pump.

1.3 Analysis

Soil sample was collected from the quartz reactor after microwave treatment, and the removal efficiency (R_E) of oil contaminant was determined. The oil concentration in the soil sample was measured before and after remediation, then R_E was calculated as follows:

$$R_E = \frac{C_o - C_r}{C_o} \times 100\% \tag{1}$$

where, C_o is the original concentration of the oil contaminant in soil sample, C_r is the residual concentration of the oil contaminant in soil sample after remediation.

Concentration of the oil contaminant in soil sample was determined according to an ASTM Method (D 7066-04) as TPH (Total Petroleum Hydrocarbons)-IR (Infrared spectrometer). Briefly, the soil sample was extracted with S-316 (a polymer of chlorotrifluoroethylene) and an aliquot of the extract was pretreated with silica gel to remove polar compounds, then placed into a quartz cuvette for the measurement by an infrared analyzer (Horiba Instrument Co., France). The calibration standard used in this TPH-IR method was 25% (*V/V*) *n*-hexadecane, 37.5% (*V/V*) isooctane and 37.5% (*V/V*) chlorobenzene.

The analysis of oil composition was performed by a gas chromatograph (GC-FID, Shimadzu GC-2010, Japan) through comparing with the chromatogram of the standard petroleum hydrocarbon. The column used was a DB-5 (30 m in length, 0.25 mm inner diameter and 0.25 μm in thickness) with nitrogen carrier gas at a flow rate of 3 mL/min, hydrogen gas 47 mL/min and air 400 mL/min. The temperature program was as follows: 3 min hold at 40°C, 5°C/min to 300°C and 20 min hold at 300°C.

2 Results and discussion

2.1 Enhancement of carbon fiber on microwave heating of soil

2.1.1 Temperature profile

Carbon fiber was mixed with the contaminated soil to enhance the microwave heating of the soil. Figure 2 illustrates the temperature profile of the soil mixed with different CF dosages at 800 W. The steady substrate temperature without any absorber addition is lower than 230°C, whereas the addition of 0.1 wt.% ACF brings the test sample to a temperature higher than 800°C after 10 min microwave heating. Similar temperature profile is drawn when 0.1 wt.% GF is added into soil. Microwave heating of soil enhanced by some other accepted strong microwave absorbers such as activated carbon powder (ACP), granular activated carbon (GAC), MnO_2 and Cu_2O was also investigated to compare with CF (Fig. 3). After 10 min microwave irradiation, the temperatures of the test soil, mixed with 10 wt.% whichever of ACP, GAC, MnO_2 or Cu_2O, are all lower than 700°C, while this value can be achieved in approximate 4 min when only 0.1 wt.% CF is added into soil. This reveals that the presence of CF can more efficiently enhance the ability of the soil mixture to absorb microwave with relatively low additional dosage.

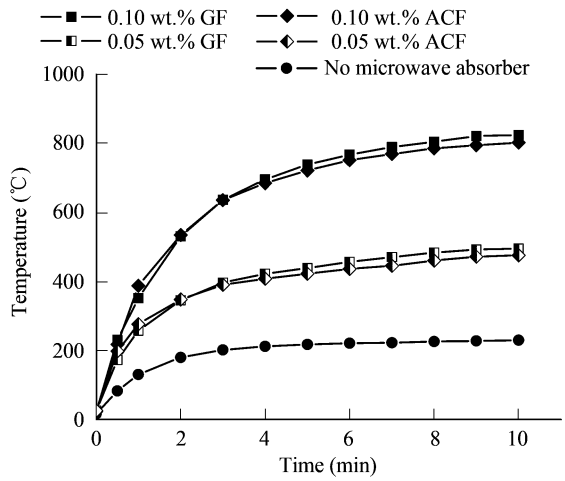

Fig. 2 Temperature profiles of the soil during 800 W microwave heating enhanced by carbon fiber. GF: graphite fiber; ACF: activated carbon fiber.

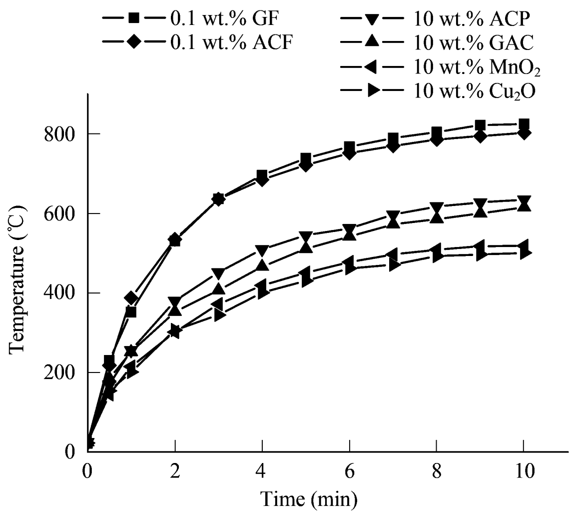

Fig. 3 Comparison of temperature profiles of the soil during 800 W microwave heating enhanced by different microwave absorbers. ACP: activated carbon powder; GAC: granular activated carbon.

2.1.2 Dielectric properties

In order to better understand the role of CF in the enhancement of microwave heating, the authors, using micro-disturbance method in microwave resonant-cavity, measured the imaginary part of the complex permittivity of the soil and the soil mixed with selected microwave absorbers such as ACF and ACP, which were just different in material geometry (Fig. 4). This imaginary part is the effective dielectric loss factor (ε''_{eff}), which determines the temperature increase of the material in the microwave field as follows (Clark *et al.*, 2000):

$$\frac{\Delta T}{\Delta t} = \frac{2\pi f \varepsilon_0 \varepsilon''_{eff} |E|^2}{\rho C_p} \tag{2}$$

where, T is temperature, t is rradiating time, f is microwave frequency, ε_0 is permittivity of free space, E is local electric field intensity, ρ is density, and C_p is specific heat capacity. ε''_{eff} generally comprises some terms, such as a term of dielectric loss (ε''_{di}), a term of Maxwell-Wagner loss (ε''_{mw}) and a term due to electrical conduction loss

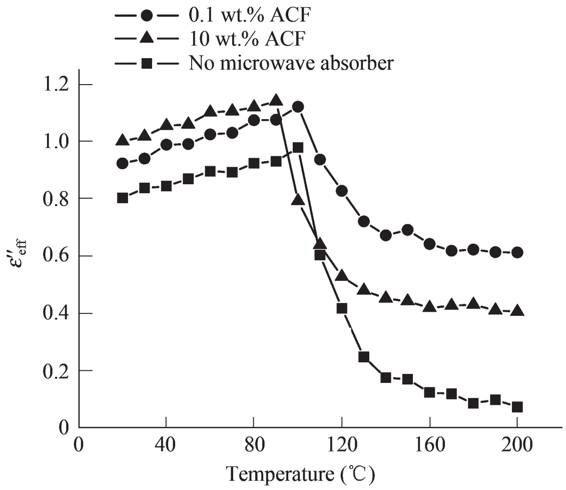

Fig. 4 Evolution of effective loss factors of soil sample with temperature for different microwave absorbers.

($\varepsilon''_{\text{cond}}$) (Wise and Froment, 2001).

As shown in Fig. 4, the $\varepsilon''_{\text{eff}}$ values of all the test soil samples decrease with temperature increasing when the temperature is beyond 100°C. This is because of loosing moisture, which strongly contributes to the $\varepsilon''_{\text{eff}}$ of the material with the increase of temperature (Bilali *et al*., 2005). When the temperature increases to 200°C, moisture is removed completely and $\varepsilon''_{\text{eff}}$ decreases towards a constant minimum value, which is the values resulted from microwave interaction with soil or the mixture of soil and microwave absorbers. It can be seen that the soil sample without microwave absorbers possesses a $\varepsilon''_{\text{eff}}$ value of about 0.07 at 200°C, which comes from $\varepsilon''_{\text{di}}$ due to dielectric loss caused by some weak microwave absorbers in soil. This $\varepsilon''_{\text{eff}}$ value increases to 0.41 when 10 wt.% ACP is mixed into soil. It is believed that the Maxwell-Wagner loss, as a term of $\varepsilon''_{\text{mw}}$, contributes to the $\varepsilon''_{\text{eff}}$ enhancement (Wise and Froment, 2001). It can also be seen from Fig. 4 that, when only 0.1 wt.% ACF is mixed into soil, the $\varepsilon''_{\text{eff}}$ value of soil sample at 200°C increases to 0.61. This infers that, ACF can more strongly enhance $\varepsilon''_{\text{eff}}$ of the soil sample even though it has the same base composition as ACP. It may be due to that, for the fiber shape of CF, the electrical conduction losses are prone to occur and play an important role in microwave energy dissipation during the microwave heating of soil sample besides the mechanism of Maxwell-Wagner losses. Hence, $\varepsilon''_{\text{eff}}$ of the soil sample is strongly enhanced by CF as terms of $\varepsilon''_{\text{cond}}$ and $\varepsilon''_{\text{mw}}$, and CF thereby strongly enhance microwave heating of soil according to Eq. (2).

2.2 Orthogonal analysis

ACF was selected as a model of CF to enhance microwave thermal remediation of the contaminated soil based on the investigation of CF enhancement on microwave heating of soil. An orthogonal experiment (Table 2) was designed to investigate the influences of ACF dosage, microwave power, microwave irradiating time, reaction pressure and nitrogen flow rate, which were

Table 2 Parameters in $L_{16}(4)^5$ orthogonal experiment

Level	ACF dosage (wt.%)	Microwave power (W)	Time (min)	Reaction pressure (MPa)	Nitrogen flow rate (mL/min)
I	0.025 (A_1)	200 (B_1)	0.5 (C_1)	0.10 (D_1)	150 (E_1)
II	0.05 (A_2)	400 (B_2)	1 (C_2)	0.08 (D_2)	650 (E_2)
III	0.075 (A_3)	600 (B_3)	1.5 (C_3)	0.06 (D_3)	1150 (E_3)
IV	0.1 (A_4)	800 (B_4)	2 (C_4)	0.04 (D_4)	1650 (E_4)

considered to be related to the contaminant removal (Shang *et al*., 2005, 2007). Sixteen tests were carried out, and R_E was calculated for each case after measuring concentration of the residual oil. The results of the tests are shown in Table 3, along with the experimental sequence of each case. Investigation of the matrix can reveal several key parameters influencing oil removal, and a comprehensive understanding is possible whereby the quantification of the contribution of each factor can be conducted according to the analysis results.

The predominance of a particular factor can be determined by investigating the range of the maximal difference of the summed R_E values (R). It can be known from Table 3 that microwave power, ACF dosage and microwave irradiating time have strong impacts, which may be due to their close relationship with temperature increase during microwave heating. In Table 3, it can also be seen that nitrogen flow rate and reaction pressure have relatively weak influences on the removal of oil contaminant. The increase of nitrogen flow rate can improve the mass transfer of the volatiles leaving the soil sample and induce more uniform heating of sample. A lower pressure can reduce the boiling point of the oil in soil matrix, allowing equivalent amounts of oil to be removed at slightly lower treating temperature (Shang *et al*., 2005). However, the changes of nitrogen flow rate and reaction pressure do not directly influence the ability of samples to absorb microwave. Therefore, their influence on oil removal is not significant.

2.3 Maximum removal of oil contaminant

According to the orthogonal analysis in Section 2.2, the condition of 0.1 wt.% ACF, 800 W microwave power, 0.08 MPa reaction pressure and 150 mL/min nitrogen flow rate was chosen to investigate the maximum removal of oil contaminant. Comparison between CF and other microwave absorbers on the enhancement of the maximum oil removal was also performed. As shown in Fig. 5, with 0.1 wt.% CF (or GF) more than 99% of oil contaminant can be removed within 4 min, while the similar value is just able to be achieved after 15 min for 10 wt.% ACP or GAC and only less than 90% of oil removal can be reached after 15 min when 10 wt.% MnO_2 or Cu_2O is used. In addition, the experimental result reveals that, after the microwave processing, about 80% ACF can be recovered and reused in the next process without changing its ability to enhance microwave thermal remediation obviously. These results indicate that, using CF to enhance microwave thermal remediation, high oil removal can be obtained in a short time with relatively low dosage.

Table 3 Results and analysis of the $L_{16}(4)^5$ orthogonal experiment

Experiment no.	A	B	C	D	E	R_E (%)
	ACF dosage (wt.%)	Microwave power (W)	Time (min)	Reaction pressure (MPa)	Nitrogen flow rate (mL/min)	
1	0.025	200	0.5	0.1	150	2.2
2	0.025	400	1	0.08	650	21.2
3	0.025	600	1.5	0.06	1150	30.3
4	0.025	800	2	0.04	1650	47.4
5	0.05	200	1	0.06	1650	16.3
6	0.05	400	0.5	0.04	1150	9.7
7	0.05	600	2	0.1	650	45.0
8	0.05	800	1.5	0.08	150	57.2
9	0.075	200	1.5	0.04	650	25.9
10	0.075	400	2	0.06	150	56.3
11	0.075	600	0.5	0.08	1650	32.5
12	0.075	800	1	0.1	1150	59.4
13	0.1	200	2	0.08	1150	54.8
14	0.1	400	1.5	0.1	1650	53.8
15	0.1	600	1	0.04	150	52.9
16	0.1	800	0.5	0.06	650	50.0
K_1	101.1[a]	99.2	94.4	160.4	168.6	
K_2	128.2	141	149.8	165.7	142.1	
K_3	174.1	160.7	167.2	152.9	154.2	
K_4	211.5	214.0	203.5	135.9	150.0	
R	110.4[b]	114.8	109.1	29.8	26.5	

[a] $K_i^x = \sum R_E$ at x_i, [b] R^x = max. (K_i^x) – min. (K_i^x), where, x represents A, B, C, D, or E, i = 1, 2, 3, or 4.

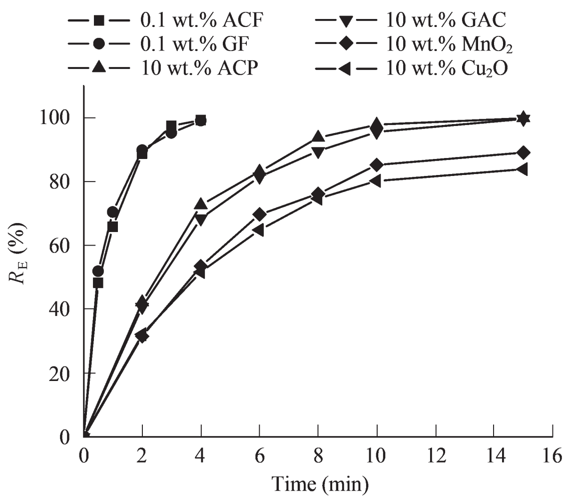

Fig. 5 Removal efficiency of oil contaminant against irradiating time during 800 W microwave heating enhanced by different microwave absorbers.

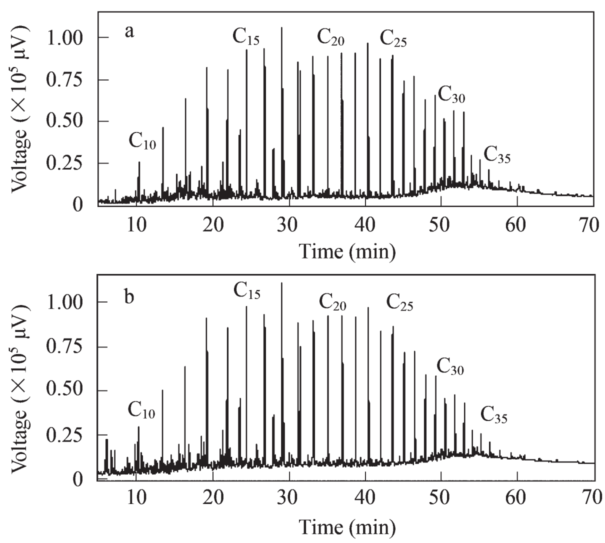

Fig. 6 Gas chromatogram for original oil (a) and recovered oil (b).

2.4 Analysis of recovered oil

During the microwave thermal remediation, the oil and water in the soil sample were removed from matrix and collected after subsequent condensation in the ice-salt bath. A small amount of anhydrous Na_2SO_4 was added into the oil/water mixture for dehydration after microwave processing, and the dehydrated oil was quantitatively and qualitatively analyzed. Quantitative measurement of the recovered oil reveals that more than 94% of the oil contaminant in soil can be recovered when the removal efficiency of the oil contaminant is more than 99%. It infers that the oil contaminant removed from soil can be recovered with a little loss.

Figure 6 shows the chromatograms for the original oil and the recovered oil. Comparison of Fig. 6a and Fig. 6b reveals that the oil contaminant in soil matrix is removed to the oil collector without obvious changes in composition during the microwave thermal remediation. This may mostly benefit from the selective and simultaneous advantages of microwave heating. Microwave can heat the soil sample to vaporize the oil contaminant without causing hyperthermia in the whole reactor. Besides, microwave heating can deliver the heat energy to the inside of the heated substrate without developing excessive temperatures at the outside surface of the substrate. Thus, when the temperature of the soil sample is high enough to vaporize the oil contaminant during microwave heating, the vaporized oil contaminant escapes from soil matrix without passing through a higher temperature region. In general, the temperature for oil vaporization is lower than that for composition destruction. Consequently, the most of oil contaminant has been removed from soil matrix without changes in composition before the temperature level for composition destruction is achieved. Whereas, from this comparison, it can also be known that slight cracking of some heavier hydrocarbons to lighter hydrocarbons occurs in the process. The cracking may be a result of pyrolysis of minor oil that has not escape from the soil matrix in time. Accordingly, besides removing and recovering the oil contaminant in soil, this remediation process may avoid evident production of air pollutant such as some lighter hydrocarbons.

3 Conclusions

Microwave heating enhanced by CF can be applied for thermal remediation of crude oil-contaminated soil. Compared to other microwave absorbers, CF can enhance microwave heating of soil more effectively. Microwave power, CF dosage and microwave irradiating time are important parameters governing the removal of oil contaminant, while the influences of reaction pressure and nitrogen flow rate on the oil removal are not significant. Under the optimum condition, a successfully remediation (99% oil removal) can be achieved in 4 min with 0.1 wt.% CF. This may reveal a lower operating cost for the remediation process.

In addition, more than 94% of the oil contaminant can be recovered in the remediation process and recovered oil does not change obviously in composition, compared with the original oil. Therefore, this microwave thermal process can not only clear up the contaminated soil rapidly but also recover the usable oil contaminant efficiently without causing apparent secondary pollution.

Acknowledgments

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