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Application of Hydrogen Peroxide and Fenton as Pre- and Post-treatment Steps for Composting of Bottom Sludge from Crude Oil Storage Tanks

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In this research the feasibility of chemical oxidation with H₂O₂ and Fenton's reagent for degradation of petroleum hydrocarbons before and after composting of bottom sludge from crude oil storage tanks was investigated. Results showed that total petroleum hydrocarbons removal in composting reactor was 80.2%. In chemical oxidation steps, petroleum hydrocarbons removal enhanced with increasing oxidant concentrations. Increasing oxidation time from 24 to 48 h had a little effect on petroleum hydrocarbons removal. The study showed that chemical oxidation as a pre-treatment step was more effective than post-treatment. It was also deduced that petroleum hydrocarbons in the sludge and composted mixture can be oxidized by hydrogen peroxide without adding supplementary iron.

Keywords: bottom sludge, crude oil storage tanks, Fenton, H₂O₂, petroleum hydrocarbons

1. INTRODUCTION

Due to the significant amounts and toxicity of oily sludge, environmental pollution by petroleum hydrocarbon is one of the most important issues (Kriipsalu and Nammari, 2010; Xu et al., 2011). Remaining of crude oil in storage tanks usually lead to accumulation of oily sludge at the bottom of the tank which should be disposed of as solid waste in an environmentally safe manner. This sludge waste is a mixture of crude oil, salt water, silt, sand, clay, soil, heavy metal salts, paraffin, asphaltene, and the scale from the tank (Baek et al., 2007; Gallego et al., 2007). Because of the cost and risk of disposing this type of sludge in landfills, alternative solutions should be used. In the past, these wastes were mixed with soil and then disposed but nowadays, they are generally treated by expensive physicochemical methods (Gallego et al., 2007).

Application of bioremediation process to treat hazardous wastes has been shown to be effective in biodegrading petroleum hydrocarbons (Godoy-Faundez et al., 2008; Yu et al., 2010) at laboratory or field-scales. In spite of advantages of biodegradation in petroleum hydrocarbon remediation (Bento et al., 2005; Marin et al., 2005), It however has some limitation on degradation of complex aromatic compounds. (Hansen et al., 2004). In chemical oxidation with hydrogen peroxide (H₂O₂)

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TABLE 1
Physicochemical Properties of the Sludge and Compost

<i>Parameter</i>	<i>Unit</i>	<i>Oily Sludge</i>	<i>Unmaturated Compost</i>
OC	mg kg ⁻¹	374167	169777
N	mg kg ⁻¹	1504	1487.6
P	mg kg ⁻¹	1128	2462
Moisture content	%	6.1	40.9
pH	—	6.89	7.46
TPH	mg kg ⁻¹	104300	1450

and Fenton ($\text{H}_2\text{O}_2 + \text{Fe}^{+2}$), hydroxyl radical (OH°) are generated. Hydroxyl radical is a strong and nonspecific oxidant that reacts with most organic compounds at near diffusion controlled rates of 10^7 to $10^{10} \text{ M}^{-1} \text{ sec}^{-1}$. These two oxidants are able to oxidize organic compounds with an aromatic moiety (Neyens and Baeyens, 2003). Thus, hydrogen peroxide and Fenton's reagent are the most commonly used chemical oxidants to overcome the limitations of bioremediation in degrading toxic and biorefractory petroleum hydrocarbons (Tsai and Kao, 2009; Lu et al., 2010).

Due to the need for decontamination large amounts of these hazardous wastes in Iran, this research was conducted. The aim of the present study was to determine the feasibility of chemical oxidation as a pre and post treatment for the sludge composting.

2. MATERIALS AND METHODS

2.1 Oily Sludge and Unmaturated Compost

The oily sludge used for this study was obtained from an oil refinery plant located in Tehran, Iran. The sludge was brought to the laboratory, milled, and passed through a 2 mm sieve. The sludge was not collected directly from the tanks, but from an open temporary storage area. As a result, the moisture content of the sludge was low (6.1%). Unmaturated compost was obtained from a composting facility in Tehran. In that facility the compost was produced from commingled waste and the compost samples contained high amounts of impurities such as glass. As a result, the organic carbon (OC) of unmaturation compost was low (269.8 g kg⁻¹). The compost was taken from the third week of process period, brought to the laboratory, passed through a 4 mm sieve and stored in the laboratory at room temperature before mixing with the sludge. Table 1 shows the characteristics of the sludge and compost used for this research

2.2 Composting Conditions

The cylindrical 1 L capacity composting reactors were set-up at laboratory-scale and operated continuously during 10 weeks. The reactor held 500 g of total composting mixture. The experimental design of sludge:compost (dry weight) ratio was 1:8. In order to increase bioavailability, the sludge was gradually added to unmaturation compost. The reactors stood vertically with air flowing continuously, to avoid oxygen content limitation, and vented outdoors to avoid volatilized compounds accumulation in the reactors. Air flow through the media was provided by means of oil free diaphragm pumps (HAILEA Model ACO 5505), air-delivery tubes and diffusers inserted into the bottom of the reactors. Water content of the mixture was adjusted at 55% before the input of sample in the reactor. Every day moisture content was measured to ensure that it was maintained at the required level, and

amended with tap water when necessary. The primary C:N:P of mixtures was adopted at 100:5:1. Nitrogen was supplemented using NH_4Cl while KH_2PO_4 was added as supplement for phosphorus. To obtain a homogeneous mixture and increase in contact between existing microorganisms and contaminant, the material was mixed three times in day.

2.3 Chemical Oxidation

Both sludge (as pre-treatment) and composted mixture (as post-treatment) was subjected to hydrogen peroxide and Fenton's reagent. The oxidation was carried out at room temperature in a bench-scale glass reactor of 0.5 L capacity. As ferrous salt, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was used and the hydrogen peroxide was of 30% concentration. The molar ratio of $\text{H}_2\text{O}_2:\text{Fe}^{+2}$ was 10:1, which is the optimum ratio between hydrogen peroxide and ferrous ions (Goi and Trapido, 2004) for removal of petroleum hydrocarbons. Hydrogen peroxide and Fenton's reagent were added in six concentrations including 2%, 5%, 10%, 15%, 20%, and 30% (w w⁻¹) for a period of 24 and 48 h of reaction time. The oxidants were added both in a single and stepwise addition (after 0, 2, 4, and 8 h of treatment) way. In order to increase reaction rate of oxidation process in pre-treatment experiments, the sludge was also saturated with distilled water. Thus, the oxidants were added to both pristine and saturated sludge.

2.4 Sample Analysis

The samples were taken randomly every week for TPH, OC, phosphorous (P), and nitrogen (N) analysis. Prior to sampling, the contents of each bioreactor were thoroughly mixed and then sub-samples were collected from the three equidistant points and mixed for analyses. TPH was extracted with n-pentane and analyzed with GC-FID (VARIAN Model CP-3800) according to Texas Natural Resource Conservation Commission (2001). The extract was injected into a gas chromatograph equipped with a flame ionization detector and CP-Sil 8CB capillary column (30 m length, 0.32 mm internal diameter, and 0.25 μm film thickness). The OC content of samples was determined by using a loss-on-ignition procedure (TMECC, 2002). 10 g samples were dried at 110°C for 24 h (moisture content) and then transferred to a furnace held at 600°C for 2 h. Volatile solid was calculated from the difference of pre and post ignition sample weights. The moisture of the samples for each analysis was determined to produce the results on a dry matter basis. Phosphorous and nitrogen contents in the samples were determined spectrophotometrically and by the Kjeldahl method according to Standard Method (American Public Health Association, 2005). Heavy metals were analyzed with ICP after digesting the samples with HCl and H_2SO_4 mixture. All measurements were done in duplicate.

3. RESULTS AND DISCUSSION

Metal concentrations in oily sludge are shown in Table 2. As indicated, the sludge contain high concentrations of Fe, Al, Cu, and Mn and low concentrations of Be, La, Li, Mo, Sn, As, and Co. Al-Futaisi et al. (2007) reported that concentrations of zinc, copper, nickel, and chromium for this type of sludge were 74–759, 2.4–33.4, 0.4–18.8, and 3.9–17.7 mg kg^{-1} , respectively. When comparing these ranges with the sludge sample under consideration, it can be concluded that the sludge in this study have higher concentrations of copper, chromium, nickel, and lower concentrations of zinc.

Chemical analysis by GC-FID revealed low TPH concentrations in the pristine compost (1450 mg kg^{-1} dry weight). According to TNRCC, nonpetroleum organic compounds which are soluble in n-pentane and which have boiling points in the range of TPH can be measured under the conditions of this method. Thus, it is not clear that the measured TPH in the compost is a petroleum hydrocarbon or

TABLE 2
Physicochemical Properties of the Sludge

<i>Element</i>	<i>Unit</i>	<i>Concentration</i>
Fe	mg kg ⁻¹	7167.43
Al	mg kg ⁻¹	1451.98
Cu	mg kg ⁻¹	707.36
Mn	mg kg ⁻¹	134.43
Pb	mg kg ⁻¹	77.60
K	mg kg ⁻¹	51.63
Cr	mg kg ⁻¹	49.95
Ti	mg kg ⁻¹	49.91
Ba	mg kg ⁻¹	45.03
Ni	mg kg ⁻¹	38.75
V	mg kg ⁻¹	28.66
Cd	mg kg ⁻¹	11.78
Zn	mg kg ⁻¹	11.08
Co	mg kg ⁻¹	5.84
As	mg kg ⁻¹	4.99
Sn	mg kg ⁻¹	3.85
Mo	mg kg ⁻¹	3.42
Li	mg kg ⁻¹	1.58
La	mg kg ⁻¹	0.99
Be	mg kg ⁻¹	0.42

nonpetroleum hydrocarbon. However, it is obvious that TPH concentrations in the pristine compost in comparison with the initial TPH concentrations of the sludge (104300 mg kg⁻¹ dry weight) was very low and can be neglected.

Table 3 shows the amount of TPH, OC, N, P, OC:TPH, C:N, C:P, and N:P during the composting period. As indicated, total TPH removal in composting reactor was 80.2%. In all reactors, rapid degradation of TPH was observed in the early stages of reaction and removal rates were decreased significantly during the next weeks. The slowing of the degradation rate during the later stages could be attributed to the properties of the remaining hydrocarbons. Although the exact nature of the remaining hydrocarbons is not known, they likely consist of recalcitrant components (Leonardi et al., 2007). In this regard, further investigation in the toxic effect of each TPH component should also be

TABLE 3
Residual Amount of Different Parameters During the Composting Period

<i>Time (Week)</i>	<i>TPH, mg kg⁻¹</i>	<i>OC, mg kg⁻¹</i>	<i>N, mg kg⁻¹</i>	<i>P, mg kg⁻¹</i>	<i>OC:TPH</i>	<i>C:N</i>	<i>C:P</i>	<i>N:P</i>
0	11469	231944	11597	2319	20.22	20.00	100.00	5.00
1	9690	216125	10877	2151	22.30	19.87	100.48	5.06
2	8272	205015	9853	2009	24.78	20.81	102.05	4.90
3	6883	198438	9212	1735	28.83	21.54	114.37	5.31
4	5834	192932	8923	1494	33.07	21.62	129.14	5.97
5	4665	188614	8470	1338	40.43	22.27	140.97	6.33
6	3876	184941	7808	1299	47.71	23.69	142.37	6.01
7	3417	179612	7031	1249	52.57	25.55	143.80	5.63
8	2907	174800	6731	1198	60.13	25.97	145.91	5.62
9	2597	170400	6702	1141	65.60	25.43	149.34	5.87
10	2550	167498	6618	1130	65.69	25.31	148.23	5.86

TABLE 4
TPH Removal in Pre-treatment Step

Oxidant	Concentration, %	Time, h	TPH Removal, %			
			Single Addition to Pristine Sludge	Stepwise Addition to Pristine Sludge	Single Addition to Saturated Sludge	Stepwise Addition to Saturated Sludge
H ₂ O ₂	2	24	0.78	0.89	2.53	3.36
		48	0.79	0.92	2.58	3.61
	5	24	3.62	6.34	10.21	14.28
		48	4.61	6.60	11.47	13.91
	10	24	16.86	19.79	27.44	33.56
		48	18.57	21.56	28.03	34.44
	15	24	24.99	30.60	35.15	41.57
		48	26.51	32.06	37.26	44.18
	20	24	35.11	38.84	37.42	46.63
		48	35.22	43.62	39.86	48.87
	30	24	45.24	50.79	53.92	60.50
		48	47.49	52.69	55.88	63.80
Fenton	2	24	0.24	0.73	2.57	3.69
		48	0.55	0.78	2.26	3.50
	5	24	4.79	6.75	11.63	14.85
		48	5.67	7.06	12.05	16.80
	10	24	16.19	18.59	26.53	30.28
		48	15.80	19.59	27.68	32.97
	15	24	23.75	31.94	34.40	43.69
		48	24.76	34.64	36.56	47.05
	20	24	34.15	40.74	40.70	48.44
		48	36.04	40.90	45.07	51.06
	30	24	44.26	48.19	53.64	60.07
		48	45.76	52.88	55.26	61.99

conducted. The carbon source in the amendment materials must not represent a preferential carbon source that retard and limit degradation of the target contaminants. In this research, unmaturation compost added as a carbon source did not act as competing energy sources, but caused an increase of degradation rate of the TPH. This fact was deduced indirectly from the increase of OC:TPH ratio. Thus, TPH removal was faster than carbon consumption. As indicated, nitrogen and phosphorus consumption was significant during the length of the treatment. The amounts of OC was decreased, thus an increase of C:N and C:P ratios was indicative of a faster decrease of nitrogen and phosphorus as compared to OC decrease. As well, phosphorus consumption was higher than that of nitrogen. Plateau trends of N and P in three later weeks in the composting reactors would attribute to decrease of microorganism's activities, which might have resulted in a limited removal of petroleum hydrocarbons.

TPH removal in pre-treatment experiment is shown in Table 4. In the experiment with low concentrations of H₂O₂ and Fenton's reagent (2% and 5%, respectively), oxidation reaction is not efficient enough to degrade TPH from the sludge. Watts (1992) reported that high concentrations of H₂O₂ is required for contaminants removal in soils and a strong oxidizing condition is required to degrade contaminant efficiently. The results indicated that TPH removal can be enhanced with increasing oxidants concentrations. Increasing oxidation time from 24 to 48 h has a little effect on TPH removal. On the other hands, stepwise addition of oxidants (both H₂O₂ and Fenton's reagent) to saturated sludge has the highest TPH removal. Kroger and Fels (2007) reported that stepwise addition of H₂O₂ was more effective in destroying contaminants. In Fenton treatment, various weight

TABLE 5
TPH Removal in Post-treatment Step

<i>Oxidant</i>	<i>Concentration, %</i>	<i>Time, h</i>	<i>TPH Removal, %</i>	
			<i>Single Addition to Composted Mixture</i>	<i>Stepwise Addition to Composted Mixture</i>
H ₂ O ₂	2	24	0.73	1.09
		48	0.73	1.09
	5	24	5.09	7.27
		48	5.82	9.82
	10	24	12.00	15.64
		48	13.09	16.36
	15	24	20.00	25.45
		48	22.18	27.09
	20	24	28.73	33.09
		48	31.27	36.00
	30	24	34.91	37.45
		48	36.73	38.18
Fenton	2	24	0.73	1.45
		48	0.73	1.45
	5	24	9.45	12.36
		48	12.00	14.91
	10	24	22.91	24.73
		48	23.64	26.55
	15	24	29.45	33.09
		48	32.73	35.27
	20	24	34.55	37.45
		48	35.64	38.18
	30	24	38.55	39.27
		48	38.55	40.73

ratios between hydrogen peroxide and sludge were used while keeping the molar ratio of hydrogen peroxide to ferrous ions constant at 10:1. It was also established that degradation of TPH depended on applied concentrations of Fenton's reagent. Similar to H₂O₂, further increasing of Fenton oxidation time from 24 to 48 h resulted in very low additional removal of TPH.

In this research oxidation with Fenton's reagent were conducted at natural pH (without adjustment of pH). Although a low pH range of 2–4 is preferred to facilitate the generation of OH radicals in Fenton system, the process is feasible in neutral pH (Yin and Allen, 1999). Results showed that H₂O₂ and Fenton's reagent had relatively similar efficiency on TPH. This may be due to the fact that the sludge contains significant amounts of iron (more than 7 g kg⁻¹) that may promote Fenton reactions. Thus, it was deduced from this study that TPH could be oxidized by hydrogen peroxide without adding supplementary iron. Kong et al. (1998) reported that the naturally occurring iron minerals can catalyze H₂O₂ and initiate Fenton reaction and may have a promising application to petroleum-hydrocarbon contaminated site remediation.

Table 5 presents the efficiency of H₂O₂ and Fenton's reagent in TPH removal from composted mixture (initial TPH of the 1:8 mix ratio = 2550 mg kg⁻¹).

In this section, the H₂O₂ and Fenton's reagent was applied as a post-treatment after composting for destruction of biorefractory organic compounds remaining after microbial degradation. The 10 weeks of composting and the subsequent oxidation by hydrogen peroxide with and without supplementary ferrous ions (the same 30% concentration in both cases) resulted in TPH decrease to

1511 and 1576 mg kg⁻¹, respectively. It was deduced from the study that application of H₂O₂ and Fenton's reagent as a pre-treatment step was more effective than post-treatment.

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