Pyrolytic remediation of crude oil-contaminated soil

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HIGHLIGHTS

• Pyrolysis was used to remediate crude oil-contaminated soil.
• Pyrolysis temperature influenced remediation efficiency more than the residence time.
• FTIR and GC × GC confirmed changes in hydrocarbon composition.
• Pyrolysis successfully treated real polluted soils in continuous treatment mode.
• Seed germination was increased >50% in remediated soils.

GRAPHICAL ABSTRACT

ABSTRACT

Deterioration of our terrestrial environment due to decreasing soil quality brought on by crude oil spills and leakages is a major issue. In this study, soil samples were prepared by mixing clay (bentonite) and sand contaminated with 5 and 10 wt% crude oil (in order to study the effect of oil concentration), and weathered in a laboratory to simulate actual contaminated soil. Volatilization of light oil was inhibited in clay rich-soil, resulting in higher contamination after weathering. The efficiency of the pyrolytic treatment was evaluated by comparing the weight change and n-hexane extractable material (HEM) content of the soil samples. The working temperature in fl influenced pyrolysis efficiency more than the reaction time. A residual amount of 0.29–0.61 wt% (below the soil pollution standard) was observed in the samples with high clay content and pollution level (by pyrolysis for 30 min at 400 °C). Infrared analysis of treated soil samples showed a reduction in alkyl functionality (C—H), confirming a decrease in hydrophobicity and an improvement in water holding capacity (WHC). Seed germination and plant growth were relatively better in the pyrolyzed soil. The field applicability of the pyrolytic treatment process was confirmed at laboratory and pilot scale, as well as by treating soil samples collected from actual polluted sites.

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1. Introduction

Rising global energy demand has significantly increased the transportation of crude oil via air, water, and land routes. Oil spillage and leakage occurring during transportation, processing, and through other anthropogenic activities contaminate the terrestrial environment (Anthony and Wang, 2006). The Hebei oil spill of 2007 is the worst accident reported to date in South Korea. A massive amount (~10,900 tons) of crude oil was released, covering 375 km of the western coast line (Yim et al., 2017). More than hundreds of local oil spill accidents were reported in 2018 (SAFETY4SEA, 2019). Soil is the component of the terrestrial environment that is most affected; enormous monetary losses are incurred due to the resultant reduction in agricultural productivity. Various treatment technologies such as incineration (Anthony and Wang, 2006), biological treatment (Zhang et al., 2010), soil washing (Fabbrì et al., 2008; Keskin et al., 2008), a combination of soil vapor extraction and bioremediation (Soares et al., 2010), thermal desorption (Dazy et al., 2009; Lee et al., 1998), and land treatment (Kaimi et al., 2007; Kostecki, 1989; Shailubhai, 1986) have been developed for crude oil-contaminated soil remediation. Among them, the two foremost thermal soil remediation techniques are: incineration and thermal desorption (Li et al., 2009). Incineration is associated with soil contaminants burn off at high temperature (up to 1000 °C) (Rushon et al., 2007) and removes all the organic materials necessary for agricultural application (Enner, 1995; Nyer, 2000), while during thermal desorption, the soil contaminants are desorb, mobilize, and evaporate by rise in temperature (from 100 to 600 °C). In addition, thermal desorption may also promote biodegradation (Kaimi et al., 2007; Li et al., 2009; Rushon et al., 2007). However, large operational costs, energy consumption, large-scale usage, and structural deterioration of soil have restricted the application of the aforementioned thermal treatments processes (Chien, 2012). On the other hand, pyrolysis, an alternate thermal treatment process, can be performed at lower temperature (350–500 °C). The pyrolysis process, relative to incineration, significantly minimizes the heat requirement (40–60%) (Vidonish et al., 2016a). A pyrolytic treatment for 10 min at 250 °C of diesel-contaminated soil reduces total petroleum hydrocarbons from 6272 to 359 mg/kg (Ren et al., 2020). The pyrolysis of contaminated soil at 400 °C for 30 min was facilitated by adding 5% hematite, considerably improving water-holding capacity of the soil (Liu et al., 2020).

Crude oil contaminants along with very low natural organic matter (NOM) have been reported in soil samples of several Middle Eastern countries (Amin Al Manni et al., 2019; Bruckberger et al., 2019; Lee et al., 2019; Tavili et al., 2019). Hence, it requires not only remediation, but also soil amendment. Pyrolysis involves the removal of volatile organic content (VOC) from soil and the development of the soil matrix structure. Available reports on biomass pyrolysis are generally focused on the organic content (VOC) from soil and the development of the soil matrix structure. In the present study, we investigated the remediation of soil contaminated with crude oil using pyrolysis. Factors such as the pyrolysis temperature, heating rate, and residence time may affect physicochemical characteristics of treated soil (Jayaraman et al., 2015; Yan et al., 2014). Therefore, these factors were examined in our treatment of oil-contaminated soil samples with varying particle size and pollution levels. The field applicability of the optimized process was confirmed through experiments using soil samples collected from actual polluted sites, and pilot experiments. The impact of soil treatment on plant growth was also evaluated.

2. Experimental

2.1. Materials and chemicals

River sand samples collected from the Nakdong River (Republic of Korea) were supplied from the HIT FACTORY (Pohang, Republic of Korea). Clay of extra pure quality (Bentonite, CAS NO. 1302–78–9, Duskan Pure Chemicals, Ansan, Republic of Korea) was used to prepare clay-sand mixtures of different compositions. The ACS reagent grade n-hexane (CAS NO. 110–54–3, Sigma Aldrich, Missouri, USA) was used as the extraction solvent in Soxhlet extraction and hydrochloric acid (CAS NO. 7647–01–0, Sigma Aldrich, Missouri, USA) to determine the n-hexane extractable material (HEM). Extra pure granular anhydrous sodium sulfate (CAS NO. 7757–82–6, Junsei chemical, Tokyo, Japan) was used for soil drying.

2.2. Preparation of soil samples

The soil samples for laboratory-scale pyrolysis experiments were prepared by mixing sand and bentonite in different weight ratios (0–20%, 2 mm) followed by contamination with crude oil received from Saudi Arabia. The composition of the crude oil (i.e., the fraction of hydrocarbons) used varies, and the ratio of short-chain hydrocarbons and n-alkane was relatively high (Fig. S1). The mixtures were stored in a fume hood for a week. During storage, most of the light oil was vaporized, leaving behind only heavy oil in the samples. This process can be used to simulate oil weathering (Urum et al., 2004).

Two different oil-contaminated soil samples with 5 and 10 wt% crude oil and an American Petroleum Institute (API) gravity of 27 were used. Moreover, three oil-contaminated and weathered soil samples, collected from different sites in the South East Kuwait oil field (obtained from Kuwait Oil Company) were used in the present investigation.

2.3. Pyrolysis of soil samples

A series of 30 g soil samples were taken in pre-weighed crucibles, pyrolyzed in a muffle furnace under inert (N2) atmosphere at different temperatures (300, 350, and 400 °C) and residence times (30 and 60 min). Pyrolysis is initiated at 300–350 °C, and thereafter proceeds rapidly in temperature range 400–450 °C (Del Bianco et al., 1993; Yoshida et al., 1984) with the better removal efficiency at 60 min residence time (Bulmœu et al., 2014). After pyrolysis, soil samples were weighed again. The weight difference was compared according to the pyrolytic conditions and was used to calculate the remediation efficiency (RE), by the following expression:

$$ RE \% = \frac{(C_i - C_f)}{C_i} \times 100 \quad (1)$$

where $C_i$ and $C_f$ are the initial and residual concentrations of contaminants in crude-oil contaminated soil (mg/kg), respectively.

The soil sample pyrolyzed at 400 °C was analyzed for HEM, commonly oil and grease, using USEPA method 9071B (EPA, 1998), extracted through Soxhlet extraction using an evaporator (RV 10, IKA, Staufen, Germany) at 85 °C. The content volatilized below 85 °C cannot be measured. All the experiments were carried out in triplicate and an average values have been reported.
under the curve for the chromatogram were used for calibration and USA) was used for hydrocarbon analysis. The changes in the area of mass spectrometry (MS, 5977A mass detector, Agilent Technologies, Palo Alto, CA, USA) coupled with mass spectrometry (FID). The surface chemistries of the contaminated and treated soil samples were studied using Fourier-transform infrared spectroscopy (FTIR, Vertex 70v, Bruker, Billerica, MA, USA). The change in soil particle size due to the formation of char was analyzed by dry sieving.

The water holding capacity (WHC; the maximum amount of water that the freely drained soil can hold), which is one of the major benefits of char produced through pyrolysis, was determined using the volumetric method (Priha and Smolander, 1999). The WHC was estimated after the saturated soil sample was allowed to drain (without allowing its moisture stores to be depleted due to evaporation). To estimate the WHC, 20 g of the dried soil sample was transferred into a funnel. After, the funnel was placed over a volumetric cylinder. Twenty mL of water was poured through the soil sample, saturating it. The WHC of the soil sample was then evaluated by measuring the amount of water that had passed through the soil into the funnel.

Pyrolysis of weathered oil-contaminated soil samples, collected from different sites in Kuwait, was performed to investigate the efficiency of the pyrolysis process on actual weathered samples. At the laboratory scale, the soil samples were pyrolyzed at 350, 400, and 500 °C for 20 min. Furthermore, a screw kiln (length: 120 cm and diameter: 30 cm, Fig. S2) was used to investigate the efficiency of the pyrolysis process at the pilot scale. Natural-gas burners (350 ± 3.2 °C) were used for indirectly heating the soil samples. Artificially contaminated soil samples were fed at a rate of 20 kg/h, treated for 30 min, and then analyzed to obtain an estimate of the change in HEM content.

2.5. Germination and plant growth experiments

The effect of pyrolysis on seed germination and early plant growth was tested by both laboratory and field-scale experiments, in triplicate. Twenty grams of contaminated and remediated soil (pyrolyzed for 60 min at 300, 350, and 400 °C) were weighed into petri dishes (80 mm diameter). Ten seeds of the selected plant species, Lactuca sativa L., were planted in appropriate petri dishes. The soil samples were moistened and incubated at 25 °C. The soil samples in the petri dishes were moistened daily, and the percentage of germination was recorded for 15 days. For the field experiment, 40 seeds of Lactuca sativa L., were planted on 1000 g of remediated soil sample (350 °C, 30 min) using the screw kiln. The seed germination and plant growth (as determined by weight) were observed after 7 days.

3. Results and discussion

3.1. Characterization of soil samples

The HEM and moisture content of the contaminated soil samples were determined (Table 1). Volatilization of light oil had occurred during the weathering process. Volatilization caused HEM in the weathered samples to be, 3.27–3.72 wt% (in the 5 wt% oil mixed samples) and 7.76–8.62 wt% (in the 10 wt% oil mixed samples), which is lower than the initial content in the contaminated soil samples (at both 5 and 10 wt%). Further, a higher HEM amount was present in soil samples with more clay content. The higher the clay content, the more the light oil was retained by the soil during the weathering process. The residual oil in the weathered soil showed strong peaks between 3000 and 2800 cm⁻¹ in the FTIR spectrum, due to alkyl functionality on the soil surface (Fig. 1).

The amount of moisture (a sum of water and some light hydrocarbons) was relatively high in the soil samples with higher clay content. The moisture content was calculated by the weight difference before and after heating the samples at 105 °C. Some light hydrocarbons along with water can be released at this temperature. Therefore, the high values of moisture content were observed for highly oil-contaminated soil (Table 1). The difference in moisture content for the soil with clay was larger than the difference in HEM; the moisture content was more affected by water content than light oil. The increased moisture content in the uncontaminated soil sample (raw sand) with high clay content supports this observation (Table 1).

<table>
<thead>
<tr>
<th>Clay (wt%)</th>
<th>HEM (wt%)</th>
<th>Moisture content (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Contaminated</td>
<td>Uncontaminated*</td>
</tr>
<tr>
<td>0</td>
<td>3.27</td>
<td>7.76</td>
</tr>
<tr>
<td>5</td>
<td>3.68</td>
<td>8.36</td>
</tr>
<tr>
<td>10</td>
<td>3.68</td>
<td>8.62</td>
</tr>
<tr>
<td>20</td>
<td>3.72</td>
<td>7.97</td>
</tr>
</tbody>
</table>

* Uncontaminated soil sample is raw sand.

![Fig. 1. FTIR spectra of 5 wt% crude oil-contaminated soil sample before (5 wt% oil soil) and after pyrolysis (300 °C, 60 min, 350 °C, 60 min, 400 °C, 60 min).](image-url)
3.2. Remediation of oil-contaminated soil samples

The synthetic crude oil-contaminated soil samples with varied sand and clay content (wt%) were tested for pyrolytic remediation at different temperatures (300–400 °C) and residence times (30–60 min). The pyrolysis process was divided into three stages. Initially, moisture from the soil samples was evaporated. During the second stage, volatile matter was released. Finally, the decomposition of residual carbonaceous compounds occurred. Generally, the weight loss during pyrolysis of the contaminated soil occurred due to moisture evaporation (at lower temperatures) and decomposition of light hydrocarbons (at higher temperatures). As illustrated in Fig. 2, the overall weight loss for the soil samples were 1.35–3.55 wt% and 2.44–6.24 wt% for the 5 and 10 wt% crude oil-contaminated soil samples, respectively. The pyrolysis temperature, as opposed to the treatment time, had a significant effect on weight loss (Fig. 2A and B). At 400 °C, ~90% RE was achieved within 30 min of treatment time. However, at 300 °C, only 70% RE was reached, even after 60 min of treatment. Therefore, to achieve 90% or higher RE, oil-contaminated soil samples must be treated at 350 °C or higher for 30 min or more. In contaminated soil samples (5 wt% crude oil), the weight loss increased as the clay content increased. Light oil remained in the soil sample with higher clay content during the mimicked weathering process for laboratory sample, resulting in more of the remaining oil being volatilized during the pyrolysis process (Table 1 and Fig. 2A). However, lesser weight loss was observed with 10 wt% crude oil-contaminated soil samples with an increase in clay content (Fig. 2B), compared to 5 wt% crude oil-contaminated soil sample. As the clay content increased, the slope of weight loss was 0–0.040 in 5 wt% contaminated soil and −0.012–0.012 in 10 wt% contaminated soil. These results indicate that the clay content had more influence on soil remediation when the crude oil concentration was lower (5 wt%).

During pyrolysis at 400 °C (30 min), decreases of at least 80% in HEM content for both 5 and 10 wt% contaminated soil samples with varied clay content (wt%) were observed (HEM_V1 in Fig. 3). Furthermore, an additional 30 min of pyrolysis time (total time 60 min) reduced the HEM content further (HEM_V2). The residual HEM content (HEM_R) was decreased ~1 wt% in all the oil-contaminated soil samples, regardless of the amount of clay. For soils with moderate and high contamination levels (5 wt% and 10 wt% of crude oil), the treatment used in this study achieved a contamination level of 1 wt%. The criterion is required from the Kuwait Environmental Remediation Program (KEPA, 2012).

A significant weight loss in the soil samples occurred as the oil contamination increased, without affecting the RE. However, for highly crude-oil polluted soil, additional treatment was required. The highest oil removal rate was observed with fine sand, and the lowest one with coarse sand and clay; this is due to the fact that more oil adsorbs in soil with smaller particle sizes (i.e., soil having larger specific surface area) (Falciglia et al., 2011).

During pyrolysis, the soil grain size increases due to the attachment of hydrocarbons. The char produced due to heavy crude oil pyrolysis is similar to polymer hydrocarbons such as asphaltenes and resins (Ambalae et al., 2006; Vidonish et al., 2016a). After pyrolysis, the initial particles (<600 μm), which were coated with oil components (tar, resin, and asphaltenes), increased their size to above 600 μm due to char formation on the soil surface. The particle size (≤600 μm) in soil was increased by 6.67% and 5.87% to sizes above 600 μm with clay (0 and 10 wt%, respectively) (Fig. 4). The increased particle size due to
Pyrolytic treatment can improve the soil permeability (Childs and Collis-George, 1950; Daneyko et al., 2011; Panda and Lake, 1994; Shepherd, 1989).

The residual crude oil components in the soil before and after pyrolytic treatment (400 °C, 30 and 60 min) (Fig. 5) were quantitatively analyzed using GC equipped with an FID. Peaks of different sizes appeared in chromatograph of the 5 wt% crude oil-contaminated soil sample. A 30 min pyrolysis of a contaminated soil sample resulted in a decrease in peak size. However, many branched-chain hydrocarbons (formed during pyrolytic treatment) were detected in the pyrolyzed soil sample compared to the untreated soil sample. After 60 min of pyrolytic treatment, the hydrocarbons were almost eliminated (Fig. 5).

The FTIR spectrum of a 5 wt% crude oil-contaminated soil sample before and after pyrolysis (please refer to Fig. 1) shows C–H stretching between 3000 and 2800 cm$^{-1}$ due to alkyl functionality on the soil surface. Alkyl (C–H) functionality is an indicator of hydrophobicity (Kinney et al., 2012). The alkyl functionality of contaminated soil sample was removed by pyrolysis. The hydrophobicity was significantly reduced as the pyrolysis temperature increased (300 to 400 °C). This result was confirmed by the disappearance of the alkyl peak, in line with previous studies (Keiluweit et al., 2010; Kinney et al., 2012). The drop in alkyl functionality and reduction in hydrophobicity (due to pyrolysis) eventually increased the WHC of soil samples (Fig. 6). A higher hydrophobicity of oil-contaminated soil samples causes a bypass of water, and pyrolytic treatment allows water to spread throughout the sample, thus increasing the WHC. The WHC increased as the temperature and reaction time increased, which is consistent with the results of HEM removal. Lower HEM and higher WHC can help plant growth.

The effectiveness of the treatment process was confirmed by laboratory scale pyrolysis experiments using three actual contaminated soil samples collected from Kuwait. The observed HEM contents in samples A, B, and C were 4.90, 4.06, and 9.18%, respectively (Table 2). A significant decrease in HEM content was observed after pyrolysis. Only a trace amount of HEM was left untreated in the highly contaminated soil samples after pyrolysis at 400 °C (Table 2). Pilot tests were also conducted to treat large amounts of contaminated soil. The contamination level of 5.21 wt% was reduced to 0.28 and 0.03 wt% after 30 and 60 min of pyrolytic treatment at 400 °C, respectively. Contamination level of 1 wt% (acceptable level in KEPA) could be achieved within 30 min of pyrolysis time. Even if the amount in the soil samples is increased, the process can be performed on a large scale if the heat transfer efficiency is appropriate (Benanti et al., 2011; Fantozzi et al., 2007).

The incineration of coal tar contaminated soil and oil contaminated gravel was carried out in a pilot scale plant at 850 °C with 100% contaminants removal efficiency (Anthony and Wang, 2006). Similar removal efficiencies were observed through thermal desorption. However, previous studies have only reported the remediation of diesel-contaminated soil (Falciglia et al., 2011; Lee et al., 1998, 1999; Piña et al., 2002; Tatano et al., 2013). Recent pyrolytic treatment for crude oil-contaminated soil was carried out at 420 °C for 3 h with residual oil content to be <1 wt% (Vidonish et al., 2016a). Present study reported...
relatively lower pyrolysis temperature (300–400 °C) and shorter residence time (30 and 60 min) to remediate contaminated soil below the regulatory standard. Pilot scale tests also demonstrated the effective remediation of crude oil-contaminated soil. Additionally, germination and plant growth experiments were conducted to test the feasibility of remediated soil for agricultural purposes.

3.3. Germination and plants growth experiment

The seed germination and plant growth of remediated soil samples compared to contaminated soil were examined. Germination experiments were also conducted using soil samples treated in a kiln (laboratory scale) (Fig. 7A). The seed germination rate was lower in the contaminated soil, as well as in the pyrolyzed soil with higher clay content. This might be attributed to the fact that more oil remains unpyrolyzed in soil with high clay content (Espitalie et al., 1980; Ranjbar, 1993). After 15 days, the germination rates in soil with 20 wt% clay before and after the pyrolytic treatment were 10 and ~50%, respectively (Fig. 7A). A soil sample treated at 400 °C showed a 20–40% germination rate (day 3), compared to a 0–20% germination rate in the contaminated soil.

Soil samples treated in the kiln were also tested for seed germination in the field. The seed germination rates were 62% and 65% on day 3 and 15, respectively. The seeds did not germinate at all in the contaminated soil. This result is highly significant, in comparison to the germination rate of 8% (3 days) and 48% (7 days) in uncontaminated soil.

After the germination test in pyrolyzed soil, germinated seeds were collected and their length and weight were measured. The average length (2.95 cm) and total weight (0.976 g) of the germinated seeds in pyrolyzed soil were higher than the average length (2.39 cm) and total weight (0.301 g) of the seeds germinated in uncontaminated soils. Previous research found that the addition of char to agricultural soil substantially reduced nutrient leaching (Gronwald et al., 2015). Higher nutrient retention and availability due to charcoal addition in soil has been reported, and relationships with higher exchange capacity, surface area, and direct nutrient additions have also been found (Glaser et al., 2002).

4. Conclusion

The crude oil-contaminated soil was remediated through pyrolytic treatment and reused. Light oils volatilized during the weathering process. However, clay-rich soil samples retained more oil after weathering. Relatively low temperatures of 300–400 °C, along with 30 and 60 min reaction times, were used to determine the appropriate residence time. More oil was volatilized with higher temperatures and longer reaction times, with the temperature being more effective than the reaction time. The oil pollution level of 1 wt% in soil was achieved by treatment of soil at 400 °C for 30 min. After treatment, the WHC was also increased due to reduced hydrophobicity. The acceptable level of remediation was also achieved in the case of field-contaminated soils.

A screw kiln was used to remediate large amounts of soil samples. If the appropriate level of heat transfer was attained, the process can be efficient at temperatures as low as 350 °C. Seed germination in treated soils increased to >50%; the germination rate and growth rate also increased. Results suggest that for a soil affected by weathered oil,

![Figure 6](image_url)

**Fig. 6.** Effect of pyrolysis at different temperatures on water holding capacity (WHC) of soil samples.

**Table 2**

The n-hexane extractable material (HEM) content in contaminated soil samples collected from Kuwait, before and after pyrolysis.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>HEM content (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample A</td>
</tr>
<tr>
<td>Untreated pyrolysis</td>
<td></td>
</tr>
<tr>
<td>@350 °C/20 min</td>
<td>0.09</td>
</tr>
<tr>
<td>@400 °C/20 min</td>
<td>0.08</td>
</tr>
<tr>
<td>@500 °C/20 min</td>
<td>0.04</td>
</tr>
</tbody>
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![Figure 7](image_url)

**Fig. 7.** Seed germination rates (%) during laboratory scale experiments in contaminated and treated (a: 300 °C, 60 min; b: 350 °C, 60 min; c: 400 °C, 60 min) soil mixtures (clay: sand = 0–20 wt%) (A). Images illustrating plants growth in aforementioned soil mixtures along a control (B).
pyrolysis can be a viable treatment for rapid remediation and improve-
ment in its reusability. Also, compared to incineration lesser energy con-
sumption reduces pollution. In addition, incineration removes all the
organic materials necessary for agricultural application, while pyrolysis
produces biochar, which increases germination and growth rate, and
water holding capacity of the soil. This study is particularly applicable
in countries with a scarcity of water and soil for cultivation.

CRediT authorship contribution statement

Chan-Ung Kang: Investigation, Visualization, Writing - original draft.
Do-Hyeon Kim: Investigation, Project administration. Moonis
Ali Khan: Writing - review & editing. Rahul Kumar: Writing - review
& editing. Seung-Eun Ji: Investigation, Data curation. Kung-Won
Choi: Investigation, Data curation. Ki-Jung Paeng: Resources, Writing
- original draft. Sungmin Park: Data curation, Writing - original draft.
Byong-Hun Jeon: Supervision, Writing - original draft.

Declaration of competing interest

The authors declare that they have no known competing financial
interests or personal relationships that could have appeared to influ-
ence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.

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