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Changes in Ecological Properties of Petroleum Oil-Contaminated Soil After Low-Temperature Thermal Desorption Treatment

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Abstract Effects of low-temperature thermal desorption (LTTD) treatment on the ecological properties of soil contaminated by petroleum hydrocarbons were assessed. For this purpose, various ecological properties related to soil health and physicochemical properties of the oil-contaminated soil before and after LTTD treatment were investigated. Total petroleum hydrocarbon concentration, electrical conductivity, organic matter, and total nitrogen decreased while water-holding capacity and available P_2O_5 increased. The soil color was also changed but textural class was not changed after LTTD. The microbial number and dehydrogenase activity increased following LTTD, but there was no significant difference in the β-glucosidase and acid phosphatase activities. Seed germination succeeded after LTTD, but the germination rate was still lower than that in non-contaminated soil as the growth of plants and earthworms was. The results showed that overall soil health related to biological productivity and environmental functions was improved after LTTD and suggested that LTTD could be a better alternative to other harsh remediation methods. However, ecological indicators still show differences to the adjacent non-contaminated level. Therefore, to ensure safe soil reuse, the change in eco-physiochemical properties as well as contaminant removal efficiency during the remediation process should be considered.

Keywords Soil health · Remediation · Physicochemical property · Microbial activity

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1 Introduction

Over the past few decades, there has been increasing public attention on soil contamination by petroleum hydrocarbons from leaking storage tanks, transit mishaps, abandoned petrochemical manufacturing facilities, and industrial facilities (Peng et al. 2009; Vasudevan and Rajaram 2001). Soil contaminated by hydrocarbons pose a potential risk to humans and the ecosystem (Sarkar et al. 2005; Saterbak et al. 1999). Hence, there is an increased need for remediation of contaminated soil, and various approaches to clean contaminated soils have been developed thus far (Alkorta and Garbisu 2001; Falciglia et al. 2013).

Thermal desorption treatment is a very popular remediation technique because it is highly effective in removing high concentration of organic chemicals (Tatàno et al. 2013). It can be classified as low-temperature desorption (100–350 °C) for volatile organic compounds and high-temperature desorption (350–600 °C) for semi-volatile organic compounds. They have been used to remove organic contaminants from soil, sludge, or sediment (US EPA 2012; Falciglia et al. 2011). However, the increased-temperature conditions required in thermal remediation processes can affect soil properties. Changes in surface area and porosity due to the significant structural modifications of soils after thermal treatment were reported (Merino et al. 2003). Cébron et al. (2011) used fauna and microbial bioindicators to assess the biological functioning of PAH-polluted soil after 500 °C of temperature thermal treatment. They showed that thermally treated soil had a high abundance and diversity of microorganisms and fauna, but enzymatic activities were weak because of significant changes in the physical and chemical properties of soil. High-temperature thermal desorption can increase genotoxicity to soil fauna such as earthworms, especially when the soil is also contaminated by heavy metals (Bonnard et al. 2010).

Soil remediation is generally considered complete when health risk-based criteria are met (Dawson et al. 2007; Dudka and Miller 1999). Although contaminant concentration in remediated soil may be below a target concentration level, soil functions cannot be restored if the soil characteristics are also modified during the remediation (Ouvrard et al. 2011). Then, soil health which is the soil capacity to sustain biological productivity and maintain environmental functions cannot be fully recovered (Doran and Parkin 1994). When considering further soil reuse, it is very important to evaluate the soil health following changes to soil properties during remediation.

Physicochemical and ecological soil properties can be used to monitor changes in soil quality in a short time period and help to assess the recovery of soil health during remediation processes (Dose et al. 2015). Soil microbes play an important role in the decomposition of soil organic matter, the degradation of toxic compounds, mineral changes, and nitrogen fixation (Brady and Weil 2010). Soil dehydrogenase activity is known to be proportional to microbial basal respiration (Garcia et al. 1997) and sensitive to toxic contaminants, which can therefore be used as an indicator in soil quality assessments (Scholz and Xu 2002). β-Glucosidase, acid phosphatase, and arylsulfatase activities are involved in the decomposition of organic matter into inorganic nutrients and play important roles in nutrient cycles of carbon, phosphorus, and sulfur. Plant germination and growth can represent the potential of natural regeneration and biological productivity in the remediated soil (Fowler 1986). Earthworms are bioindicators of soil quality because of their ecological function in the soil matrix (Bonnard et al. 2010). Therefore, microbial cell number, enzyme activities, plant germination and growth, and earthworm growth can be used as ecological indicators of soil health that can be affected by changes in the soil physicochemical properties (Ana Cláudia et al. 2008; Scharenbroch et al. 2013).

Although low-temperature thermal desorption treatment (LTTD) uses the lower-temperature than high-temperature thermal desorption treatment (HTTD), their potential effects on soil health cannot be ignored. Most studies have been conducted focused on the change of soil properties under HTTD, but little work has been done to evaluate the impact of LTTD on the change of soil properties related to soil health. If the soil properties are changed and soil functions are still impacted during LTTD treatment, sustainable productivity or material cycling, which is fundamental soil function, can be inhibited even after remediation is completed. Therefore, the changes to soil properties and functions during LTTD treatment must be investigated to ensure the safe reuse of remediated soil.

The aims of the study were to analyze the change in the physicochemical properties of soil after LTTD treatment and to investigate their effects on various ecological properties related to soil health. Physicochemical properties such as soil texture, soil color, organic matter content, water-holding capacity, cation exchange capacity (CEC), nutrient concentrations, and ecological properties such as microbial number, enzyme activities, seed germination and growth rate, and earthworm growth were investigated.

2 Materials and Methods

2.1 Soil Samples

Soils were sampled before and after LTTD treatment to investigate changes in ecological and physicochemical properties. Non-contaminated soil (NCS) near the contaminated site was also analyzed as a control. The total petroleum hydrocarbon (TPH) concentration in the soil before LTTD treatment was 5133 ± 508 mg/kg. To clean the TPH-contaminated soil by LTTD, contaminated soil was placed in a rotary kiln after removal of rubble larger than 50 mm. LTTD consisted of heating the soil to 200 °C for 15 min using heated air generated from a regenerative thermal oxidizer. The samples were air-dried, and particles larger than 2 mm were removed using a 10-mesh sieve. Each sample had three replicates.

2.2 Physicochemical Properties of Soil

The fractions of sand, silt, and clay in the soil were determined using the pipette method, and soils were classified according to the international soil society textural classification (Moeys 2012). Soil color was determined using a Munsell soil color chart (Wang et al. 2010). The water-holding capacity (WHC) was measured by soaking 20 g soil samples in water for 2 h and then allowing the water to drain for 10 h. Soil pH, organic matter, CEC, total nitrogen (TN), available phosphorus (P_2O_5), and TPH concentration were analyzed. The pH was measured using an Orion 4 Star pH electrometer (Thermo Electron Co., USA) in 1:5 soil/distilled water suspensions. The organic matter content was measured using the loss-on-ignition method (Nelson and Sommers 1996). CEC was measured using the 1-N acetic acid replacement method, and TN was measured using the micro-Kjeldahl method (NAAS 1988). Available P_2O_5 was measured using the Bray P2 soil test procedure (Jones 2001). The TPH concentration was determined using a gas chromatography with flame ionization detector based on the Korean Standard Methods of Environmental Pollution for Soil Contamination (KMOE 2009).

2.3 Ecological Properties of Soil

The total number of soil microbial organisms was analyzed using Petrifilm™ (3M, USA). After inoculation, 1 mL of diluted sample was spread on a plate and incubated at 37 °C for 24 h. This gave the number of colony-forming units per gram of dry soil (CFU/g). The activities of the enzymes dehydrogenase (DHA), β-glucosidase, acid phosphatase, and arylsulfatase were also determined (Lee and Sung 2014; Yi and Sung 2015). DHA was determined using 2,3,5-triphenyltetrazolium chloride (TTC) as a substrate and measuring the intensity of the reddish color of triphenyl formazan (TPF) release at 485 nm using a spectrophotometer (Shimadzu, Japan) after incubation for 24 h at 37 °C). β-Glucosidase, acid phosphatase, and arylsulfatase activities were determined using *p*-nitrophenyl-β-D-glucoside, *p*-nitrophenyl phosphate, and *p*-nitrophenyl sulfate as substrates, respectively, and measuring the intensity of the yellow color of *p*-nitrophenol release at 400 nm using a spectrophotometer (Shimadzu, Japan) after incubation for 1 h at 37 °C.

A plant germination and shoot growth experiment was performed according to a previously reported method (Dawson et al. 2007; Wang et al. 2009). *Brassica juncea* was selected because it showed lower tolerance to diesel oil (Graj et al. 2013). Ten *B. juncea* seeds were added in triplicate to a petri dish containing 45 g of dried soil. The experiments were conducted for 8 days using a plant growth chamber (Hanbaek Scientific Technology, Korea) with a daylight period of 16 h, 3500 ± 800 lx light intensity, and 80 % humidity at 22 °C. When more than 70 % of seeds germinated in the non-contaminated soil, the number of seeds germinated in other soil samples was counted. The height of aboveground plant parts at day 8 was measured to assay the effects on plant growth.

Eisenia andrei, which is a test species recommended by the Organization for Economic Cooperation and Development (OECD), was used in the experiment (OECD/OCDE 2004). For each test, 450 g of dried soil was used, and water was added to adjust the soil water content to 25 % on a soil dry weight basis. Five adult worms, each of which possessed clitella and had a wet weight of 350–400 mg, were placed in 1-L glass containers filled with soil. The containers were placed in an incubator (Dasol Scientific, Korea) at 22 °C in darkness. After 7 and 14 days incubation, the weight of the *E. andrei* specimens was measured. Each sample had three replicates.

2.4 Statistical Analysis

We applied a *t* test to determine the effects of thermal desorption treatment on soil properties and functions. The presence of significant differences between the NCS and the soil before LTTD (BLTD) and after LTTD (ALTD) was established using analyses of variance (ANOVA). A 95 % significance level was used for the ANOVA and *t* tests. All statistical analyses were performed using SPSS 18. Principal component analysis (PCA) was conducted using CANOCO for Windows (Ter Braak and Šmilauer 2012), and an ordination diagram was drawn to assess the overall soil property changes after LTTD.

3 Results and Discussion

3.1 Changes in Physicochemical Properties of Soil

The textural class of soil was sandy loam and was not changed during LTTD treatment. After LTTD, average contents of sand (2–0.02 mm) and silt (0.02–0.002 mm) decreased from 77.2 to 74.5 % and from 13.8 to 12.0 % respectively, whereas clay (<0.002 mm) content increased from 9.0 to 13.5 %. Owing to heating, silt- and clay-sized particles form sand-sized aggregates through a cementing effect between 300 and 500 °C (Terefe et al. 2008). However, this was not the case in our study, which showed a decrease in the sand and silt content of soil samples following LTTD. This is because an indirect-heating system with a low temperature (200 °C) and short duration (15 min) was used in the study. Huang et al. (2011) reported that an indirect-heating system did not significantly change the composition of sand, silt, and clay in soils. The increased clay content of soil samples observed in this study was attributed to the wearing of sand and silt particles during mixing in the rotary kiln. The altered particle size distribution affected other soil functions such as its water-holding capacity.

After LTTD, the water-holding capacity (WHC) increased by 0.272 ± 0.002 to 0.292 ± 0.004 g H_2O/g dry soil ($p < 0.05$). The increased WHC is owing to the increased clay content, which can absorb water more readily, and decreased sand content, which can allow water to percolate easily.

The color of soils changed from light olive brown to yellow brown and the hue was reddened (2.5Y → 10YR). Chroma (3 → 4) increased slightly but there was no difference in brightness (5 → 5). In general, Fe-laden minerals in soils tend to transform into hematite when the soil is completely dehydrated, which reddens the soil in a high-temperature environment (Schwertmann et al. 1999). Another factor that can affect soil color is its organic carbon content, which can darken soil (Huang et al. 2011). Removal of the organic compound during the LTTD process may affect the soil color. The increased water-holding capacity and darkened color of soil following LTTD can reduce the albedo and facilitate more energy storage. If soil color changes considerably, the aesthetic function of soil can be affected, limiting its future reuse.

The chemical properties of the soil used in this study are listed in Table 1. LTTD resulted in a decrease of 94.7 % of TPH concentration. The pH of soil was changed ($p = 0.007$) and closer to neutral. Heating increases soil pH (Biache et al. 2008; Bonnard et al. 2010; Cébron et al. 2011; Huang et al. 2011; Yusiharni and Gilkes 2012). The pH change following LTTD could have been caused by the denaturation of organic acids (Certini 2005) or the release of alkali cations during the oxidation of organic matter (Terefe et al. 2008). The cation exchange capacity decreased slightly, but the difference was not significant ($p = 0.67$). Organic matter and total nitrogen contents decreased after LTTD by 11.2 and 3.5 %, respectively. LTTD increased 56.4 % of the available P_2O_5 concentration in soil ($p = 0.001$). Biache et al. (2008) and Cébron et al. (2011) reported that the organic matter content and nitrogen concentration of soil changed after thermal treatment. Organic carbon and nitrogen levels generally decrease with increasing temperature due to the combustion of organic matter and the loss of gaseous oxides of C and N. Organic phosphorous can also be converted to inorganic forms, increasing the available P_2O_5 (Yusiharni and Gilkes 2012). Changes in the nitrogen and phosphorus concentration in soil affect the soil fertility and the C/N ratio. Increased available phosphorous concentrations

Table 1 Chemical characteristics of soil before and after LTTD treatment

Parameters	Unit	Low-temperature thermal desorption	
		Before	After
TPHs**	mg/kg	5133 ± 508	272 ± 107
pH**		6.53 ± 0.07	6.80 ± 0.03
EC**	dS/m	1.55 ± 0.09	0.87 ± 0.01
Organic matter*	%	5.26 ± 0.34	4.67 ± 0.11
CEC	cmol/kg	5.35 ± 0.58	3.96 ± 0.54
TN[a]	mg/kg	230.32	222.48
Available P_2O_5**	mg/kg	38.8 ± 2.2	60.7 ± 2.2

TPHs: total petroleum hydrocarbons, *EC*: electrical conductivity, *CEC*: cation exchange capacity, *TN*: Total nitrogen

*Indicates a significant difference at $p < 0.05$ (two-tailed) between soil samples before and after LTTD; **indicates a significant difference at $p < 0.01$ (two-tailed)

[a] Not applicable

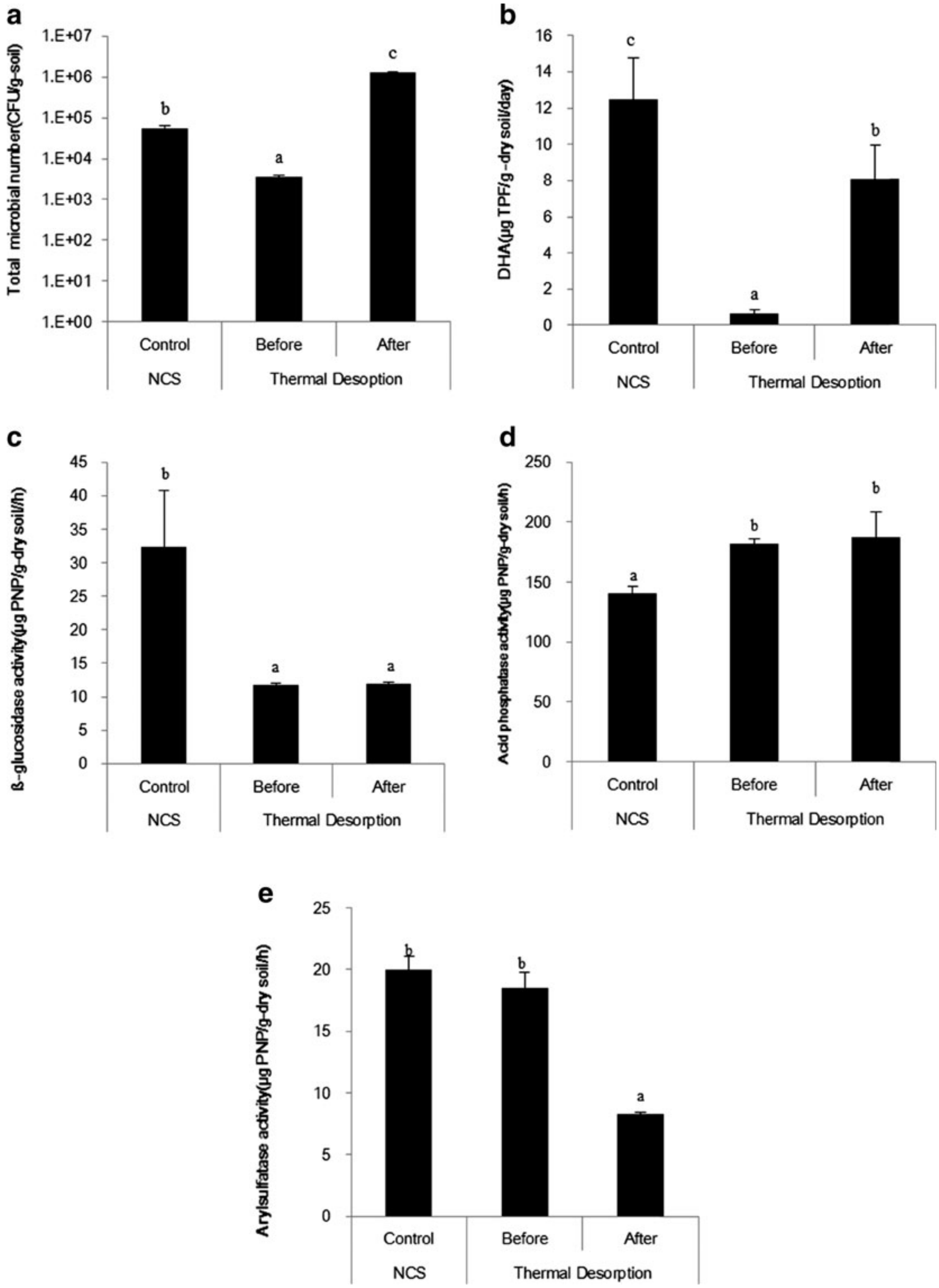

Fig. 1 Changes in total microbial number and soil enzyme activity; **a** total microbial number, **b** dehydrogenase (DHA), **c** β-glucosidase, **d** acid phosphatase, **e** arylsulfatase in the non-contaminated soil, before and after the thermal desorption treatment. *Different letters* indicate significant differences at $\alpha = 0.05$. *PNP para*-nitrophenol

can have beneficial effects on plant growth if other soil properties are unchanged.

3.2 Changes in Ecological Properties of Soil

3.2.1 Microbial Number and Soil Enzyme Activity

Figure 1 shows the microbial number and enzyme activities in the soil samples. Both the microbial number and DHA increased following LTTD, and the total microbial numbers were even higher than those in NCS. Because DHA in soil decreases when the hydrocarbon concentration is higher than the threshold values (Wang et al. 2010), the increase in DHA following LTTD was likely due to the decreased level of contaminants. Further, the total microbial number was higher but DHA was lower than in that in NCS. This shows that DHA per microbial number is still lower than that in NCS even after some of the TPHs were removed. It is likely due to the inhibition effect of TPHs remaining in the soil or changes of microbial community after LTTD (Margesin et al. 2007).

The activity of other enzymes displayed different patterns than that shown by DHA following LTTD. There was no significant difference in the β-glucosidase and acid phosphatase activities following the treatment ($p > 0.05$), but these activities were different to their corresponding activity levels in NCS. β-Glucosidase activity in soil with a history of TPH contamination has been shown to be lower than that in NCS, but phosphatase activity in the same soil was higher than that in NCS. β-Glucosidase helps transform polysaccharides in organic matter to glucose and also helps soil microorganisms to utilize them as nutrient or energy sources (Eivazi and Tabatabai 1988; Tian et al. 2010). The lower β-glucosidase activity in the BLTD and ALTD soils than in NCS suggests that β-glucosidase activity is sensitive to the presence of TPHs in soil and microorganisms have trouble in obtaining energy from the soil organic matter. Phosphatase in soil helps microorganisms to mineralize and utilize organic phosphorous (Garcıa-Gil et al. 2000). The high levels of acid phosphatase activity in TPH-contaminated soil are due to the hydrocarbons in soil. When the soil contains more than 1000 mg/kg of residual hydrocarbons, phosphatase activity gradually increases (Wang et al. 2010). Increased acid phosphatase activity may also help plant and microbial growth by increasing the inorganic phosphorous concentration in soil.

Arylsulfatase activity decreased from 18.48 ± 1.28 to 8.26 ± 0.15 μg PNP/g dry soil/h ($p < 0.05$). The similar values in NCS and soil BLTD suggested that it could be less affected by the TPHs in soil but was more sensitive to the changes in the soil properties. Organic sulfur levels can decrease owing to volatilization (Knudsen et al. 2004), and decreased sulfur concentration results in reduced arylsulfatase activity following LTTD. Because arylsulfatases are important for the mobilization of sulfate for plant nutrition, reduced arylsulfatase activity can affect plant growth (Elsgaard et al. 2002).

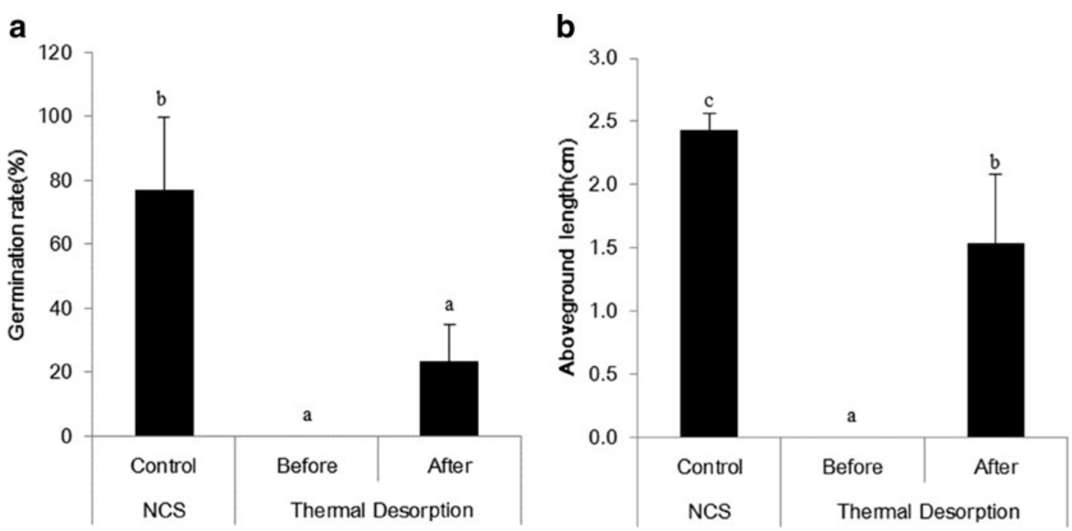

Fig. 2 **a** Seed germination percentage and **b** average length of aboveground parts of *Brassica juncea*. *Different letters* indicate significant differences at $\alpha = 0.05$

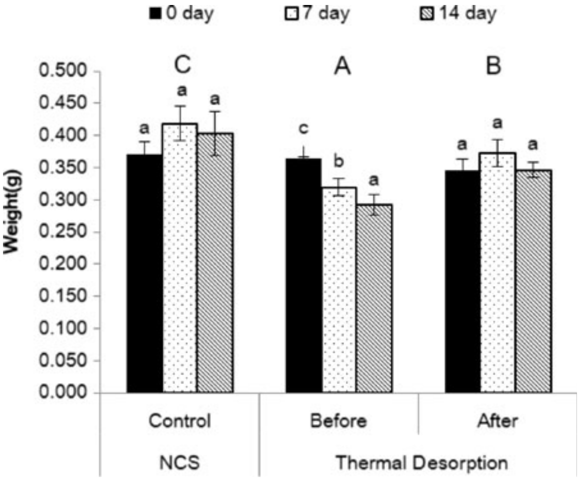

Fig. 3 Changes in earthworm weight in soil after 7 and 14 days of incubation. *Capital letters* (*A*, *B*, *C*) indicate significant differences among treatments at day 14, and *small letters* (*a*, *b*, *c*) indicate significant differences with time for each soil sample at $\alpha = 0.05$

3.2.2 Plant Seed Germination and Growth

The germination rate and aboveground growth of *B. juncea* are presented in Fig. 2. They were determined 8 days after sowing when the germination rate exceeded 70 % in the NCS. In the NCS, the germination rate was 76.6 ± 23.1 % on the eighth day, but the germination of *B. juncea* failed in soil BLTD, which was contaminated with high levels of TPH. It is known that oil pollution has negative effects on plant germination (Achuba 2006). The volatile fraction of diesel fuel has an inhibitory effect on seed germination (Adam and Duncan 2002). Because the TPH concentration decreased and most of the volatile fraction of the TPHs was removed by LTTD, the germination rate increased by 23.3 ± 11.5 % but was still lower than that in NCS. This result is supported by Dazy et al. (2009) who reported an increase in germination in soil treated by thermal desorption, but the germination rate was still lower than that in NCS.

The length of the aboveground parts of *B. juncea* was 2.43 ± 0.13 and 1.53 ± 0.55 cm in NCS and soil ALTD, respectively. The measurements of the height of the aboveground parts of plants produced similar results as the germination test. The results suggest that LTTD has a positive effect on the germination and growth of *B. juncea* by decreasing the TPH concentration in soil, but the complete recovery of soil health in terms of plant germination and growth cannot be guaranteed. Plant germination is crucial for the success of natural succession in ecosystem restoration. If plants are not able to germinate and grow well naturally, ecosystem restoration will be retarded and further efforts will be needed. *B. juncea* used in the experiment was not tolerant to diesel oil (Graj et al. 2013), and thus, the TPHs remaining in the soil result in a lower germination rate and the lower shoot growth in soil ALTD.

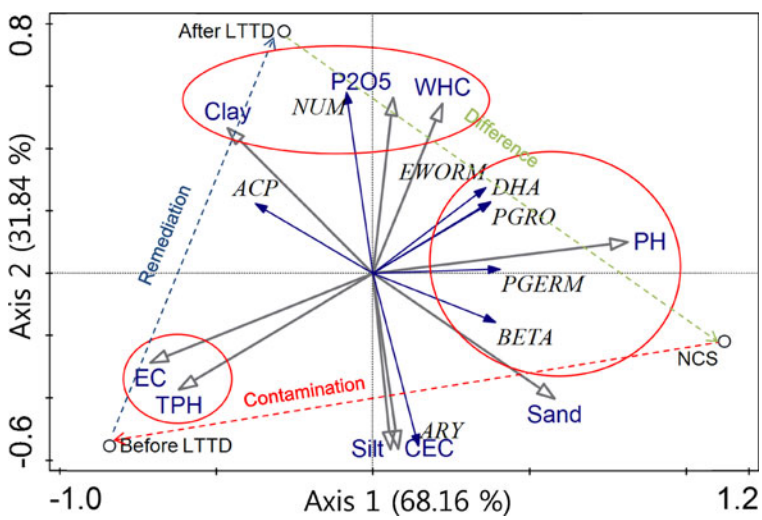

Fig. 4 Principal component analysis biplots showing the effect of low-temperature thermal desorption treatment (LTTD) on soil properties. *WHC* maximum water-holding capacity, *EC* electrical conductivity, *CEC* cation exchange capacity, *P2O5* available P_2O_5, *TPH* total petroleum hydrocarbons, *ACP* acid phosphatase activity, *ARY* arylsulfatase activity, *DHA* dehydrogenase activity, *BETA* β-glucosidase activity, *NUM* total microbial number, *PGERM* plant germination, *PGRO* plant growth, *EWORM* earthworm growth, *NCS* non-contaminated soil

3.2.3 Earthworm Assay

The changes in the earthworms' weights in soil after 7 and 14 days of incubation are presented in Fig. 3. In the earthworm assays, no dead earthworms were found during the experiment but a consistent decrease in the weight of earthworms was observed in soil BLTD. There was no difference in the initial weight and the weight after 14 days in soil ALTD and NCS. Tang et al. (2011) reported that the mortality rate of earthworms was 0, 90, and 100 % in 0.5, 2, and 3 % of TPH, respectively. The concentrations experienced in this study were not high enough to be lethal for earthworms, but they had a negative effect on their growth. In the soil ALTD, the similar initial weight of earthworms to that after 14 days indicated an improved environment for earthworm growth. However, growth was still low compared to the weight of earthworms in NCS. This suggests that the toxicity to earthworms was reduced in soil ALTD, though these values are still considered high for the healthy earthworm growth considering the weight of earthworms in NCS.

3.3 Overall Soil Health Change

PCA ordinations of the soil properties show the clear interpretation of the changes in overall properties of soil after LTTD (Fig. 4). Proximity implies similarity in the ordination diagram. The results showed that overall soil health was improved after LTTD. TPH concentration and EC decreased after LTTD while total microbial numbers, water-holding capacity, and available P_2O_5 increased. LTTD also had a positive effect on plant germination and growth as well as on earthworm growth in TPH-contaminated soil. Therefore, LTTD could be a better alternative to other harsh remediation methods that showed deterioration of soil fertility and ecological soil functions (Laurent et al. 2012; Yi and Sung 2015). However, ecological indicators still showed some differences to the non-contaminated soil level and ecological functions of soil need to be improved to achieve better soil quality.

4 Conclusions

Changes in various ecological properties related to soil health and physicochemical properties of the oil-contaminated soil before and after LTTD treatment were evaluated. Some physicochemical properties of soil were changed during LTTD processes. Total petroleum hydrocarbon, electrical conductivity, and organic matter decreased while water-holding capacity and available P_2O_5 increased. Seed germination succeeded after LTTD, but the germination rate was still lower than that in non-contaminated soil as the growth of plants and earthworms was. The results showed that LTTD improved overall soil health related to biological productivity and environmental functions and suggested that LTTD could be a better alternative to other harsh remediation methods. However, ecological properties still show differences to the adjacent non-contaminated level. Therefore, the change in eco-physiochemical properties should be considered to ensure safe soil reuse.

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