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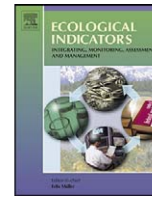

Assessing the quality of oil-contaminated saline soil using two composite indices

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ABSTRACT

It is essential to ascertain the quality status of contaminated soils prior to implementing bioremediation. In this study we assessed the quality of oil-contaminated saline soils in the Yellow River Delta, China using two composite indices. Five sites were selected according to the distance to the Yellow River estuary shoreline, and soils in each site were sampled from different distances to oil well port along the four cardinal directions. Four enzymes including dehydrogenase (DHO), catalase (CAT), polyphenol oxidase (PPO) and lipase (LIP), and physicochemical properties of soils at depth of 0–20 cm were analyzed. Soil electrical conductivity (EC) and total organic carbon (TOC) decreased significantly with the increasing of distance to the shoreline or to the well port. Enzyme activities decreased with the increasing EC except for the PPO, while they oppositely responded to TOC. Considering inconsistencies in assessing soil quality using individual indicators, we introduced two composite indices (geometric mean of the assayed enzyme activities (GMea) and integrated quality index derived from principal components analysis). The GMea decreased with EC, while it increased with TOC and the integrated quality index. The threshold values of GMea and the integrated quality index for healthy soil were confirmed according to the EC and TOC of the uncontaminated soils. If the values of the two indices moved far away from the thresholds, the soil quality became worse. The soil quality was mainly affected by crude oil contamination when the values of the two indices were higher than the thresholds; oppositely, the soil quality was influenced by soil salinity. Our results suggest that the two composite indices could be used for evaluating the quality of crude-oil-contaminated saline soil.

1. Introduction

Soil is one of the earth's most important natural resources to sustain human life. One of the main threats to soils, compromising their ability to perform functions we expect, is pollution (Mikkonen et al., 2011). Oil hydrocarbons are the most common soil contaminants. It has been estimated that the natural crude-oil seepage amounts to 6×10^5 metric tons per year, with a range of uncertainty of 2×10^5 to 2×10^6 metric tons per year (Kvenvolden and Cooper, 2003). In China, the Yellow River Delta is an important region of petroleum production. The crude oil output of Shengli Oilfield, which is located in this delta, was 2.78×10^7 metric tons in 2009. Many of the delta areas have been inadvertently contaminated by oil during crude oil extraction and processing (such as oil well blowouts and oil pipe leaks). The contaminated area accounts for 24% of the total delta; falling-oil is the largest contaminant, and the content of oil in the soil is 9.2–180.9 mg kg^{-1} (He, 2006). Another feature of the soil in the Yellow River Delta is the high salt content; the dominant chemicals in saline soil are NaCl and $MgCl_2$ (Weng et al., 2008), with the content of salt ranging from 6 to 30 g kg^{-1} (Wang et al., 2009). High salt content in the soil makes it more difficult to conduct soil bioremediation. However, few studies have comprehensively assessed the quality of the oil-contaminated soil coupled with saline stress. Therefore, it is important to ascertain the main factors that influence the soil quality, then to establish suitable soil quality indicators to monitor the change of soil quality during the implementation of soil bioremediation.

A large number of physical, chemical and biochemical properties that determine soil processes and their spatial and temporal variations contribute to defining soil quality (Doran and Parkin, 1994). In terms of the chemical properties of oil-contaminated soil, total organic carbon (TOC) is a useful indicator for monitoring petroleum hydrocarbon contamination because TOC includes all weight fractions of total petroleum hydrocarbon (TPH) (Schreier et al., 1999). Wang et al. (2010) found that there were strong

Abbreviations: CAT, catalase; DHO, dehydrogenase; EC, electrical conductivity; GMea, geometric mean of the assayed enzyme activities; LIP, lipase; LSD, Fisher's least significant difference; PC, principal components; PCA, principal components analysis; PPO, polyphenol oxidase; TOC, total organic carbon; TN, total nitrogen; TP, total phosphorous; TPH, total petroleum hydrocarbon.

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positive correlations between TOC and TPH in the soils of the oil field. In terms of soil biochemical properties, many researchers have focused on the response of soil enzymes to oil contamination (Margesin, 2005; Wyszkowska et al., 2006; Chu et al., 2007) or to saline soil (Gianfreda and Ruggiero, 2006). However, Trasar-Cepeda et al. (2000) found that the individual enzymatic activities had serious limitations as soil quality indicators, as they could not discriminate the effect of different contaminants and the extent of degradation they caused. Moreover, the activities of different enzymes generally had different responses to the variation of environmental conditions (Duarte et al., 2012). It is thus difficult to draw appropriate conclusions about soil quality using individual soil properties. A good soil quality indicator has to be integrative due to the complexity of soil structure and functionality (Garcia-Ruiz et al., 2009). Typically, composite indices, like geometric mean of the assayed enzyme activities (GMea), are calculated by means of algebraic combinations of different soil enzyme activities (Trasar-Cepeda et al., 1998; Garcia-Ruiz et al., 2008, 2009). Also, the physical and chemical properties are proposed to condense through principal components analysis (PCA) (Sena et al., 2002; Duarte et al., 2012) or factorial analysis (Shukla et al., 2006). The combination of different physicochemical and biochemical properties into one or two composite indices makes it possible to better reflect the complexity of the soil system.

In this study, we collected 76 crude oil contaminated saline soil samples from the Yellow River Delta. The primary objective was to assess the quality of oil-contaminated saline soil by using two composite indices (GMea derived from the four tested soil enzyme acitivities: dehydrogenase (DHO), catalase (CAT), polyphenol oxidase (PPO) and lipase (LIP) and integrated quality index obtained by PCA). Specifically, we analyzed: (1) soil physical and chemical properties (including electrical conductivity (EC), TOC, total N (TN), total P (TP), C/N ratio, and pH), and acitivities of four enzymes; (2) relationships of EC and TOC with enzyme activities and the GMea; (3) PCA using data for soil physical and chemical properties and soil enzyme acitivities; the relationships of the integrated quality index derived from the first three principal components with these soil properties; and (4) the validation of the GMea and integrated quality index in assessing the quality of oil-contaminated saline soil.

2. Materials and methods

2.1. Site description

The sampling sites are located in Shengli Oilfield in Dongying of Shangdong Province, China (Fig. 1). The region was formed by the fluvial sedimentation of the Yellow River, the second largest river in China. The average annual temperature is 11.7–12.6 °C. The annual precipitation is 530–630 mm, of which 70% occurs during summer (May–July) (Fang et al., 2005). Due to the low and flat terrain, high groundwater table, high mineralization rates, poor drainage conditions, and infiltration of seawater, soil salinization in this area has been very severe (Zhang et al., 2011). The mean value of soil salt content was 10.45 g kg^{-1} (Yang and Yao, 2007). The dominant soils along the seashore are commonly Salic Fluvisols and Gleyic Solonchaks (Fang et al., 2005).

2.2. Soil sampling

In May 2010, five sites were selected according to the distance to the Yellow River estuary shoreline; the nearest site (site A) to the shoreline was 3 km, and the distance between the two adjacent sites is about 20 km with a range of 16–23 km (Fig. 1). One oil well was selected at each site. Taking the oil well as the center, we sampled soils in four different directions. In each direction, four quadrats with a size of 1 m × 1 m were installed at 5-m intervals. Five soil cores (Φ = 2.5 cm) were collected from 0 to 20 cm layer in each quadrat in the shape of a "W" and combined together to be a composite sample. Each sample was sealed in a separate plastic bag, put in an ice box, and sent to the laboratory within 6 h. A total of 80 samples were obtained (four of the samples were abandoned because of their soaking in ice water during transport). Field-moist soil samples were sieved (2 mm), and each sample was divided into two subsamples and stored separately at 4 °C and −20 °C until analysis.

2.3. Soil chemical properties and enzyme activities analysis

The soil samples used for the test of EC and pH were air-dried and passed through a 2-mm sieve. Soil EC and pH were measured in a 1:5 sample/water mixture with a DDS-307W microprocessor conductivity meter (Shanghai Lida Instrument Factory, China) at 25 °C after shaking for 30 min. Soil TOC concentration was determined using the $K_2Cr_2O_7$–H_2SO_4 wet oxidation method of Walkley and Black (Nelson and Sommers, 1996). Soil TN concentration was analyzed by the Kjeldahl method, and soil TP was determined by the molybdenum-stibium colorimetry method with a continuous-flow analyzer (AutoAnalyzer III, Bran + Luebbe GmbH, Germany) after the samples were digested with H_2SO_4.

The DHO activity was measured according to Dick et al. (1996). Briefly, 0.06 g $CaCO_3$ and 6 g air-dried soil (<2 mm) were thoroughly mixed, 1 ml of 3% 2,3,5-triphenyltetrazolium chloride (TTC) and 25 ml distilled water were added and mixed, then the mixture was incubated at 37 °C for 24 h. The concentration of the reddish colored triphenyl formazan (TPF) product was measured colorimetrically (485 nm) and the DHO activity was expressed as μg TPF g^{-1} dry soil 24 h^{-1}. The CAT activity was measured by the method of Trasar-Cepeda et al. (1999). Briefly, 0.5 g air-dried soil was suspended in 40 ml distilled water and shaken for 30 min on a rotary mixer at 30 r min^{-1}, and then 5 ml of 0.3% H_2O_2 was added. The mixture was reacted with shaking for further 10 min at 20 ± 2 °C. After that, 5 ml 3 N H_2SO_4 was added to stabilize the undecomposed H_2O_2. Finally, the mixture was filtered and measured colorimetrically at 505 nm, and the CAT activity was expressed as mmol H_2O_2 g^{-1} dry soil h^{-1}. The PPO activity was measured as described by Zhou (1987). Briefly, 1 g air-dried soil was incubated for 2 h in a water bath at 30 °C with 10 ml 1% pyrogallic acid after shaking. A portion of 2.5 ml 0.5 M HCl was added after 1 h incubation, then purpurigallin was extracted by ether. The extract was measured spectrophotometrically at 430 nm and the PPO activity was expressed as mg purpurogallin g^{-1} dry soil h^{-1}. The LIP activity was measured in accordance with the method of Margesin et al. (2002). Briefly, 0.1 g field-moist soil was weighed and mixed with 5 ml 100 mM NaH_2PO_4/NaOH buffer, pH 7.25, and pre-warmed at 30 °C in a water bath for 10 min. Then 50 μl of substrate solution (100 mM *p*-nitrophenyl butyrate (*p*NPB) diluted in 2-propanol and stored at −20 °C) was added and mixed thoroughly. The mixture was incubated at 30 °C for 10 min. The *p*-nitrophenol (*p*NP) concentration was measured spectrophotometrically at 400 nm and the LIP activity was expressed as μg *p*-NP g^{-1} dry soil 10 min^{-1}.

2.4. Statistical analyses

One-way ANOVA was used to analyze the effects of different sampling sites on EC and the effects of different distances to the oil wells on TOC, respectively. Difference of the EC among sampling sites and difference of TOC among different distances to the oil wells were tested using Fisher's least significant difference (LSD) test. Two-way ANOVA was used to analyze the effects of EC, TOC and their interactions on the four soil enzymes. Pearson's correlation