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Research Paper

Mineral abundances quantification to reveal the swelling property of the black cotton soil in Kenya

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ABSTRACT

This research provides a combination method in quantifying mineral abundances of the black cotton soil (BCS) to reveal its swelling property. The soil was sampled from a roadbed to be built in Nairobi, Kenya, and was found to experience large volumetric expansion (~166%) due to the moisture variation. The wet-chemical analyses and X-ray characterizations (XRD) were undertaken to study the chemical compositions and mineral phases that are presented in the BCS. The quantification of minerals was determined by combining chemical compositions, physical properties (cation-exchange capacity = 75.5 meq 100 g⁻¹, hydroxyl water loss = 10.5%) and the phase identification. A local regression relationship was proposed based on the mass balance of major elements. The BCS was found to consist with clay minerals as nontronite (17.3 wt.%), smectite (10.9 wt.%), chlorite (21.7 wt.%) and interstratified chlorite/smectite (31.9 wt.%). The nonclay minerals of quartz and feldspar were determined to have proportions of 10.2 wt.% and 8.4 wt.%, respectively. This evaluation was also confirmed by the electron microprobe analysis (EMPA) linked with a graphic software and experimentally by the multiple-step precipitation plus additional alkaline/acid and other chemicals treatments. The high content of Na, Ca-based clay minerals found in this work accounts for the large volumetric expansion when the BCS is exposed to water.

1. Introduction

Black cotton soils (BCS) are commonly found in the mid-south Africa, south-Asia, Australia, and other part of places including China (Shelke and Murthy, 2010; Patel and Shahu, 2015). A geological survey suggested that Robi valley in Central Africa is a lacustrine sedimentary (Ackroyd and Husain, 1986; Behrensmeyer et al., 1995) where the capital of Kenya, Nairobi is located, and > 60% of the area is covered by BCS. The clays are highly expansive with presence of water, but shrink when water is lost (Rao and Shivananda, 2005; Gilbert et al., 2015). The BCS in Kenya was reported to have a volume change of 120–300%, and the swelling pressure is as high as 8–10 kg/cm² (Maneli et al., 2013; Mehta et al., 2014; Patel and Shahu, 2015). The properties give rise to serious problems in civil engineering, e.g., the highly volumetric change, weakness in strength, and the low bearing capacity would always result in damages of highways (Uppal et al., 1970). Particularly

these disasters occur periodically in Kenya due to the alternating rainy and dry seasons. To cure this highly expansive BCS as well as to find a strategy in stabilizing such swelling soils for road construction (Patel and Shahu, 2015; Miao et al., 2017), it is necessary to conduct a mineralogical study on quantitation of phase abundance in the Nairobi BCS.

Various methods have been demonstrated to quantify the mineral phases in clayey soils. For example, means of internal standards in the X-ray diffraction (XRD) characterizations (Chung, 1975; Brown and Brindley, 1980; Moore and Reynolds, 1989; Batchelder and Cressey, 1998; Kahle et al., 2002), infrared spectral absorption plus diffraction measurements (Norrish and Taylor, 1962; Hillier, 2000; Wattel-Koekkoek et al., 2001; Omotoso et al., 2006), full-pattern simulation of diffractograms or the so-called Rietveld methodology (Bish and Post, 1989; McManus, 1991; Hughes et al., 1994; Kolka et al., 1994; Taylor and Matulis, 1994), thermogravimetric analysis (Barshad, 1965), and

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others (Komadel and Stucki, 1988; Merodio et al., 1992; Blum et al., 2011) can be found. Review papers can also be found as mineralogical quantification on clay minerals by analyzing diffraction data (Kahle et al., 2002; Zhou et al., 2016). Unfortunately few documents have been reported to deal with mineralogy of the Nairobi BCS soils due to some unknown reasons (Nagelschmidt et al., 1940; Ola, 1978). Although the XRD-based techniques have been developed to analyze the mineral phases (Zevin, 1977), numerous procedural flaws exist due to the inevitable need of suppositions and variability in phase chemistry, or at least knowledge of chemical compositions/structural characteristics of minerals are required. Other difficulty lies in the unknown peak interferences/absorption coefficients of X-ray (Ouhadi and Yong, 2003; Omotoso et al., 2006), defects/compositional complexity/inter-stratification in clay minerals (Moore and Reynolds, 1989; Środoń et al., 2001; Alves et al., 2007), amorphous/disaggregation states/size effects (geometric factor) of clay particles (Brown and Brindley, 1980; Jackson, 2005), and arbitrary treatments (random/preferred crystallites orientation) (McManus, 1991) during the XRD measurements. The only use of X-ray techniques is thus unable to obtain accurate quantitative mineralogical data. Consequently some combined means have been proposed by using the XRD-based techniques and supplementary data such as elemental analyses, grain-size separation, and other spectroscopic measurements (Kolka et al., 1994; Hillier, 2000). The most commonly used is the “normative” procedures for elemental mass balance which was developed by Hussey (1973). In these methods a set of simultaneous linear-equations were founded by using rigorous evaluation of the chemical compositions and physical properties (cation-exchange capacity, CEC) which were distributed in different phases and summarized to the total soil mixture. Pearson (1978) used total silicate-analysis data in simultaneous equations for the quantitative clay mineral analysis of fine-grained sedimentary rocks. Johnson et al. (1985) developed a program of simultaneous linear equations been used to calculate component proportions for mineral mixtures in soil clays and sediments. Calvert et al. (1989) designed another computerized program for the mineral quantification of geological samples based on the identified phases and specific surface analysis. Galindo et al. (1996) for the first time applied this method in the quantification of sepiolite-palygorskite deposits. This ‘normative’ strategies are now becoming more and more widely in the mineral phase quantification. More reports can be found as Hodgson and Dudeney (1984), Braun (1986), Johnson et al. (1985), Ammar et al. (2014) and Jozanikohan et al. (2016).

In this research the combination method was revisited, and was used to quantify the mineral abundances in a type of swelling soils, the BCS in Kenya, in a view of understanding the extra expansive behavior. Excremental procedures as well as theoretical calculation were developed to quantify the mineral phases. Parameters of chemical compositions, the CEC and water-loss within 300–900 °C were taken into consideration for the simultaneous linear equations. The amount of each element shown as oxide (SiO₂, Al₂O₃, Fe₃O₂, MgO, etc.) in the BCS was determined with bulk chemical analyses and instrumental measurements in micro-areas. The chemical composition plus XRD results performed on different soil specimens (random, oriented, ethylene glycol (EG) saturated, high-temperature and HCl treated slides) were used to formulate simultaneous equations, upon which solving yielded the percentages of various clay minerals. Uncertainties were minimized by refining the input of elemental data of minerals. To confirm the accuracy of this result, the electron microprobe analyses (EMPA) were performed on an arbitrary cross-section of the BCS, and the mineral ingredients were determined. The phase abundances were evaluated based on integrated areas using a software of graphic analyzer. The formulae of clay minerals was calculated based on the O₁₀(OH)₂ principles using the EMPA data. The micro-structures were also studied to reveal the swelling behaviors. These characteristics and the obtained mineralogical data would help us to understand the BCS, and to give guidance to treatment of highly swelling soils in view of civil

engineering/geotechnical applications.

2. Materials and methods

2.1. BCS sampling sites and pretreatments

The BCS was sampled from a subgrade site (36°31'E, 1°35'S), south of Nairobi, Kenya, where a city loop road is to be constructed by China Road and Bridge Co. The pristine soil with a fine-grained texture was collected at a depth of 1.5 m below the organic matter, and photographs of BCS and sampling were shown in Fig. S1 in the supporting information. The samples were air-dried for several weeks, and were crushed into a preliminary coarse size using a heavy mortar. The grinding was performed in methanol instead of water to accelerate drying of the ground sample and to avoid swelling of shale which could liberate individual clay crystals (Jackson, 2005). Fractions of sand (2.0 mm - 75 μm), silt (75–2 μm) and clay (< 2 μm) with different sizes were obtained gravimetrically via the standard dispersion sedimentation and pipette method (Ghosh et al., 2006). The BCS was confirmed to have a percentage of ~77% of clayey and silts (< 75 μm). The free swell index of BCS was determined to be 166% (Miao et al., 2017), which suggests that the BCS belongs to a type of highly expansive clayey soil or the A-7-6 category according to American Association of State Highway and Transportation official (AASHTO) classifications, or to the CHE type soils based on JTG E40-2007 (Ghosh et al., 2006; China, 2007; Highway and Officials, 2011).

2.2. Wet-chemical analysis

Methods for the bulk chemical analysis were described as followed. The soil was digested by melting soil samples with KOH, and the test solution was prepared by the HCl dissolving procedures (LCACUGB, 1990). The weight percentage of SiO₂% was measured via a gravimetric method on the precipitation from the digested solution. Gravimetric measurements of testing SiO₂% were conducted after flocculation from solutions with the aid of poly(ethylene oxide). Other composition items such as Fe₂O₃%, Al₂O₃%, and TiO₂% were measured by volumetric methods as described in literatures (LCACUGB, 1990). The alkalis (Na₂O% and K₂O%) were determined by the flame photometry (FP6450). Percentages of CaO%, MgO%, MnO%, P₂O₅%, and MnO% were evaluated by the inductively coupled plasma - atomic emission spectrometry (ICP-AES, HK-2000). Prior to the ICP-AES measurements, the soil samples were dissolved in a mixture of acid solution consisting with HF, HClO₄, HNO₃ and HCl (HF (40%): HClO₄ (70%): HNO₃ (65%): HCl (36%) = 0.5:1:0.5:1, v/v). This digestion was completed till the disappearance of white fume of HClO₄. To determine the FeO%, the soil sample was digested with HF-H₂SO₄. The residual F⁻ ions were eliminated by adding a saturated solution of boric acid. The amount of FeO% was determined by using diphenylamine sulfonate as an indicator. The percentage of H₂O⁺% (adsorbed H₂O) was measured by gravimetric methods after treating at 105 °C for 24 h. The content of H₂O⁻% (structural H₂O, e.g., -OH) was tested by gravimetric method of using H₂-flame combustion. The loss of ignition (LOI) was measured by treating the BCS specimen at 1000 °C in a constant weight of crucible, and the differential weight is to evaluate the LOI. For the CO₂% which represents carbonates were evaluated by the HCl treatment, and the produced CO₂ are to be measured. The cationic exchange capacity (CEC) of the BCS was evaluated to be 75.5 meq·100 g⁻¹ which was determined by the copper ethylenediamine complex method (Bergaya and Vayer, 1997).

2.3. Other characterizations

The mineral phases are qualitatively analyzed by XRD characterizations performed on the BCS prepared in forms of random and oriented slides. For the preparation of randomly oriented aggregates, the

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