



Research paper

Characteristics of clay minerals in soil particles from an argillic horizon of Alfisol in central China



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ARTICLE INFO

Keywords:

Alfisol
Argillic horizon
Clay minerals
Nanoparticles
Crystalline structure

ABSTRACT

The soil particles (< 2000, 450–2000, 100–450 and 25–100 nm) in an Alfisol were studied using inductively coupled plasma-optical emission spectroscopy (ICP-OES), conventional and synchrotron X-ray diffraction (XRD), fourier-transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TG) to investigate the mineral composition of clays and changes in crystalline structure in the particles of an argillic horizon. The results showed that the clay minerals in the particle fractions (< 2000, 450–2000 and 100–450 nm) were illite, kaolinite, vermiculite and a trace amount of hydroxy interlayered vermiculite and chlorite. In the nanoparticles (25–100 nm), the main clay minerals were illite and kaolinite. The molar ratios of SiO₂ to Al₂O₃ and SiO₂ to R₂O₃ (Al₂O₃ and Fe₂O₃) were higher in the nanoparticles compared to other particle fractions. With decreasing particles size, kaolinite and vermiculite decreased gradually and illite increased. The Al-Mg-OH and Si-O-Si (Al) stretching modes of vermiculite were broadened in the 100–450 nm particles and disappeared in nanoparticles while the hydroxyl group (–OH) of clay minerals in the samples was reduced with decreasing particle fractions. The broadening of the band characteristics of clay minerals, the dehydration and dehydroxylation were less pronounced in particle fractions from the argillic horizon comparing to the topsoil. Clay minerals from 450 to 2000 and 100–450 nm particle fractions were well ordered whereas illite and kaolinite were poorly ordered in nanoparticles of the argillic horizon. The “crystallinity” of clay minerals was weakly affected in the particle fractions of argillic horizon compared to topsoil.

1. Introduction

Clay minerals are the products of the chemical weathering process and they are frequently used as indicators of pedogenesis (Velde and Meunier, 2010; Hubert et al., 2012; Hong et al., 2014; Jäger et al., 2015). Clay minerals influence the soil properties due to their high exchange capacities, small particles size, and high specific surface areas. This influence depends on the nature and the content of clay minerals species in soils. Several studies (Yoothong et al., 1997; Kanket et al., 2006; Huang et al., 2007) had reported that some clay minerals such as illite, vermiculite, chlorite or smectite were very active in the Alfisol colloids. According to the Soil Survey Staff (2010), Alfisols are characterized by clay-enriched horizon in the subsoils (argillic horizon). This horizon is a subsurface horizon with a higher amount of clay fraction (particles < 2 μm) than the overlying horizon (Soil Survey Staff, 2010, 2014). The argillic horizon can be the result of the translocation, in situ formation and relative loss of clay minerals from topsoil (Birkeland, 1999). The components of nanoparticles from soil colloids considerably influence the soil properties (Wilson et al., 2008; Monreal

et al., 2010). Soil nanoparticles are the natural soil particles having a size < 100 nm. They can contain clay minerals (illite, kaolinite, vermiculite), Fe and Al- (hydr) oxides (hematite, goethite and gibbsite), and organic carbon (Theng and Yuan, 2008; Tang et al., 2009; Monreal et al., 2010; Tsao et al., 2013). The characteristics of clay minerals in the particle fractions of two Alfisol topsoils in China illustrated that, illite and vermiculite were the abundant clay minerals in < 2000 and 450–2000 nm particle fractions while illite and kaolinite were the main clay minerals in 100–450 nm particles and nanoparticles (25–100 nm) (Zhang et al., 2016). Their structures changed with decreasing particles size and evolved from the well ordered structure in particles > 100 nm to the poor ordered structure in nanoparticles. However, it has been recognized that clay minerals from the argillic horizon colloids play a major role in the soil nutrients, water retention, geomorphic stability and transport of metals (Hopkins and Franzen, 2003; Huang et al., 2008). The detailed knowledge about clay minerals along the soil particle fractions from argillic horizon requires further studies to uncover characteristics of clay minerals in Alfisol.

The objective of the current study was to analyze particle fractions

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(< 2000, 450–2000, 100–450 and 25–100 nm) from argillic horizon of Alfisol in order to (1) identify their elements and clay mineral compositions, and compare with those in the topsoil; (2) discuss the changes in the crystalline structure of clay minerals in different particle size fractions; and (3) reveal the characters of clay minerals in nanoscale from the argillic horizon of Alfisol on the quaternary sediment in central China.

2. Materials and methods

2.1. Description of the site and soil sampling

The profile of soil investigated in the present study was located in the Xiangyang city, in Hubei province, central China. The study area was characterized by a subtropical climate, with an average annual temperature of 15.1–16.9 °C and an average annual rainfall of 878 mm/year (Huang et al., 2015). The most abundant vegetation was Peanut (*Arachis hypogaea*). The parent material was Quaternary sediment (Q₃), deposited in the latest Pleistocene. The zonal soil investigated was classified as Fragiudalfs in the Key Soil Taxonomy (Soil Survey Staff, 2014). The soil profile was localized by geographic points N32°18'55.27" and E111°57'21.17" and 117 m of altitude. The soil was sampled from 0 to 27 cm in A horizon (topsoil), and 27–48 cm in B horizon which was an argillic horizon. The soil texture was light clay in topsoil and silty clay in the argillic horizon (Table 1). The Munsell color was 7.5YR 5/6 (yellowish brown) in the topsoil and 7.5YR5/6 (dull orange-yellow) in the argillic horizon. The soil was air-dried, crushed and ground to pass 2, 0.85, 0.25 and 0.15 mm sieves.

2.2. Extraction of soil particle fractions

The soil sample (> 100 mg) was treated with 30% H₂O₂ and heated at 60 °C on the hot plate to remove organic matter. The soil samples were then suspended in the double distilled water (DDW) (600 mL), and dispersed for 6 min using a NEY 300 ultrasonic instrument (NEY, Barkmeyer Division, Yucaipa, USA) with an amplitude of 40%. The suspension was then passed through a 300-mesh sieve (50 μm) to remove the sand fractions (50 μm–2 mm). Then, the clay (< 2000 nm) fractions were collected by sedimentation according to Stokes' Law (Jackson, 1979). The suspension of < 450 nm particles and 450–2000 nm particle fractions were collected after transferring the suspension of < 2000 nm particles in the polycarbonate tubes (250 mL × 6), centrifuging and washing seven times at 3500 rpm for 5 min at 4 °C using Avanti J-26 XP refrigerated centrifuge instrument (Beckman Coulter, California, USA) with a rotor JA-14.

The suspension of < 450 nm particles were collected and transferred to the polycarbonate tubes (250 mL × 6), centrifuged and washed seven times at 9000 rpm for 15 min at 4 °C to collect the suspension of < 100 nm and 100–450 nm particle fractions.

The suspension of < 100 nm particles were collected and filtered to obtain the 25–100 nm particle fractions using an automated ultrafiltration device ((AUDII), Tony nano-techno, Taiwan) and Millipore ultrafiltration disk membrane (Filter type: 25 nm VSWP) (Tsao et al., 2009; Zhang et al., 2016).

Table 1
Principal physical and chemical characterization of the studied soils.

Sample	Depth (cm)	pH	OM (g kg ⁻¹)	CEC ^a (cmol kg ⁻¹)	Exchangeable cation(cmol(+) kg ⁻¹)				Sand (g kg ⁻¹)	Silt (g kg ⁻¹)	Clay (g kg ⁻¹)	Textures
					K ⁺	Na ⁺	Ca ²⁺	Mg ²⁺				
Topsoil	0–27	5.9	7.9	18.3	0.04	0.03	4.25	3.65	390	428	182	Light clay
Argillic horizon	27–48	7.1	5.8	17.2	0.05	0.03	4.15	4.06	271	499	230	Silty clay

^a Cation Exchange Capacity.

2.3. Soil analyses

Soil pH was determined by a 1:2.5 soil: solution ratio using distilled water (National Soil Survey Center, 1996). The cation-exchange capacity (CEC) was determined using the ammonium acetate (1 M and pH 7.0) method (Metson, 1957). Organic matter (OM) was determined by the K₂Cr₂O₇ wet oxidation method. Soil texture was determined using the pipette method (Gee and Bauder, 1986). Concentrations of Si, Al, Fe, Ca, Mg, Mn and Ti in different size particles (< 2000, 450–2000, 100–450 and 25–100 nm) were determined using inductively coupled plasma-optical emission spectroscopy (ICP-OES Visata-MPX, Varian, California, USA). K and Na were tested using a flame photometer (M410, Sherwood, UK). The molar ratio of SiO₂ to Al₂O₃ and SiO₂ to the sum of Al₂O₃ and Fe₂O₃ at the different size of soil particles was calculated.

The tested samples were saturated with Mg-glycerol (Mg-gly) and potassium (K). The Mg-gly samples were examined at 25 °C. The K-saturated samples were tested at 25 °C and then heated for 2 h at 110, 250, 350, 450 and 550 °C (Jackson, 1979). The identification of clay minerals was made according to operational definitions given by Środoń (2006), and Barré and Velde (2010) and the estimations of their semi-quantitative proportions were performed as reported by Dixon (1989) and Pai et al. (1999). The oriented samples were measured with an X-ray diffractometer (D8 Advance, Bruker, Rheinstetten, Germany) with CuKα radiation (λ = 1.5418 Å) generated at 40 kV and 40 mA. The Mg-gly and K saturated oriented samples at 25 °C of soil nanoparticles were measured using synchrotron XRD from the Shanghai Synchrotron Radiation Facility (SSRF) of China. The synchrotron XRD was 18 keV (BL14B1 beamline). The incident X-ray wavelength (λ) was 0.6887 Å. The oriented and powder patterns were recorded by a NaI and Mar345 detector, respectively. Based on Bragg's Law, *d* values of synchrotron radiation were converted into *d* values of Cu target (λ = 1.5418 Å).

Fourier transform infrared spectroscopy (FT-IR) measurements were taken using dry KBr pellets, which were prepared by mixing and pressing 1 mg of the sample with 100 mg of KBr and pressed into a disk. The infrared spectrum was collected with the resolution of 4 cm⁻¹ using 128 scans by a Bruker Vertex-70 (Germany) infrared spectrometric analyzer.

The thermogravimetric and derivative thermal gravimetric (TG-DTG) curves were obtained using the thermal instrument Netzsch TG 209 °C type. The measurement was carried out by heating the sample from 30 to 800 °C in steps of 10 °C min⁻¹ with the flow of dry nitrogen atmosphere (20 mL/min). The percentage of weight loss was calculated as a function of temperature at 30 °C corresponds to 100% of sample mass.

3. Results

3.1. Soil chemical and physical properties

The soil pH was 5.9 in topsoil and increased to 7.1 in the argillic horizon (Table 1). The OM (organic matter) content was 7.9 g kg⁻¹ in the topsoil and decreased in the argillic horizon to 5.8 g kg⁻¹. The CEC (cation exchange capacity) of soil from topsoil and the argillic horizon

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