



Research Paper

An experimental study on oven-drying methods for laboratory determination of water content of a calcium-rich bentonite



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ABSTRACT

Studies on the dehydration of montmorillonites imply that the standard oven-drying method (drying at $110 \pm 5^\circ\text{C}$) may underestimate the water content of bentonites. This study quantifies, identifies, explains, and removes the residual water in a calcium-rich bentonite dried using the standard oven-drying method. A thermogravimetric-differential scanning calorimetry (TG-DSC) test was performed to determine the true water content. According to this test, the effect of the drying time and temperature on the amount of residual water in the bentonite was examined, including the oven-drying and vacuum-drying methods. To identify and explain the residual water, specific surface area (SSA) derived from the nitrogen adsorption isotherm and dehydration reaction characterised by TG-DSC curves of samples with a residual water content of 0–13.38% were obtained. The experimental results demonstrate that the standard oven-drying method underestimates the water content of calcium-rich bentonites by 2.1–3.1%. The 2.1–3.1% of residual water includes both interlayer water and adsorbed water on the external surface. The 2.1–3.1% of residual water is the water forming the inner shell around Ca^{2+} cations in the interlayer and on the external surface of montmorillonite particles. It is impractical to distinguish between the adsorbed water and interlayer water only based on the dehydration reaction characterised by TG-DSC curves. Drying profiles of 12 h of drying at 200°C in an oven and 1 h of drying at $150\text{--}200^\circ\text{C}$ under vacuum are sufficient for removing the adsorbed and interlayer waters from the bentonite.

1. Introduction

Compacted bentonites are considered the main component of engineered barrier systems for high-level radioactive waste disposal due to their low ion diffusion, adequate swelling potential, and low permeability (Bergaya et al., 2006; Pusch and Yong, 2006). To interpret these properties, the specific surface area (SSA), cation exchange capacity (CEC), and soil-water characteristic curve (SWCC) are necessary parameters. The SWCC is defined as the relationship between the amount of water in the soil and the total potential (suction); this parameter is key to analysing the permeability and volume change of unsaturated compacted bentonites (Fredlund et al., 2012). Measurements of the SSA, CEC, and SWCC of bentonites involve determining the water content or drying bentonites.

Because bentonites mainly comprise montmorillonites, drying bentonites, to a large extent, means removing water from montmorillonites. The montmorillonite comprises repeating 2:1 unit layers where one octahedral sheet (O) is sandwiched between two tetrahedral sheets (T). Many such unit layers (TOT) are stacked one above the

other, in a parallel arrangement, to form a quasi-crystal or particle. Because of isomorphous substitution in the octahedral or tetrahedral sheets, the unit layer has a negative charge that is balanced by exchangeable interlayer cations. Water can enter the interlayer space and interact with interlayer cations and surfaces. As shown in Fig. 1, montmorillonites could contain water in several forms: free water, capillary water, adsorbed water, interlayer water, and hydroxyl water (Bergaya et al., 2006; Velde, 1992). The first two types of water, held by macropores (e.g., inter-aggregate, inter-particle pores), can be easily removed by drying under ambient conditions. The adsorbed water is attracted to the surfaces of montmorillonite particles and could be removed by heating to $80\text{--}90^\circ\text{C}$. The interlayer water is associated with the hydration of interlayer cations, the interaction of clay surfaces with water molecules, and interlayer cations and water activity in the clay-water system. It normally leaves the interlayer at $100\text{--}250^\circ\text{C}$, depending on the nature of the interlayer cations. Hydroxyl water is also called crystalline water and is present in the form of OH units within the sheet. The OH oxidises upon heating and forms H_2O (dehydroxylation reaction). The dehydroxylation reaction in montmorillonite

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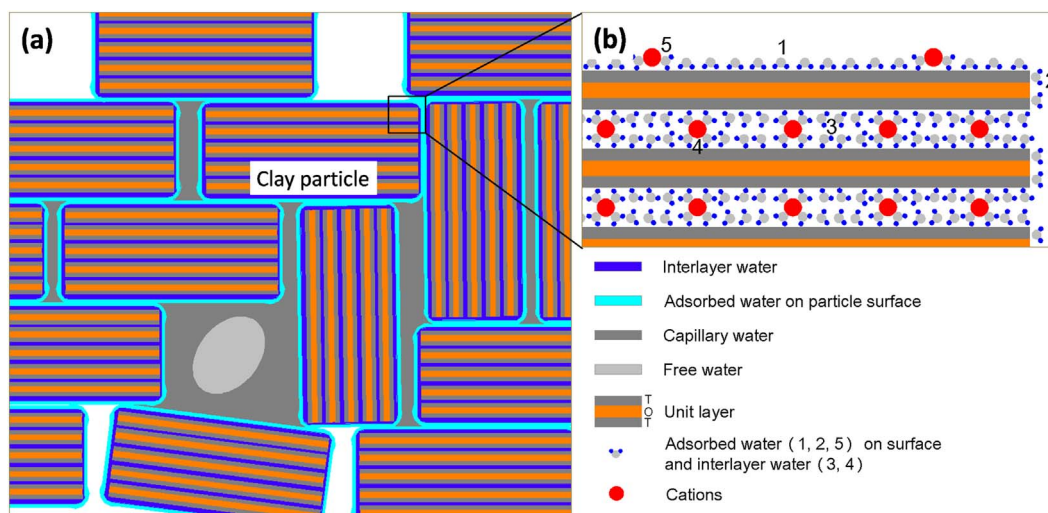


Fig. 1. Schematic diagram of waters in montmorillonite in the case of (a) inter-particles and (b) particle.

occurs above 400°C. Therefore, drying bentonites means removing the free water, the capillary water, the adsorbed water, and the interlayer water from montmorillonites.

In environmental and geotechnical engineering, the method for drying clays is the standard oven-drying method (e.g., GB/T-50123 (1999), ASTM-D2216 (2010), ISO-17892-1 (2014)). In this method, clay samples are heated in a drying oven at a temperature of $110 \pm 5^\circ\text{C}$ for 12 h or until there is no further mass loss. There is no doubt that this method is sufficient for removing the free water and capillary water. However, many studies on the dehydration of montmorillonites have inferred that this method may not successfully extract the adsorbed water and interlayer water. Mooney et al. (1952) and Cerato and Lutenegegerl (2002) suggested that a temperature higher than 120°C is necessary for adequately drying bentonites with oven-drying. X-ray diffraction (XRD) analysis showed that dehydration of the interlayer of montmorillonite was not complete after heating at 110°C (Bray et al., 1998; Ferrage et al., 2007a,b; Morodome and Kawamura, 2009). Thermal analysis of montmorillonites demonstrated that heating at a temperature higher than 200°C was necessary for extracting the interlayer water (Bray and Redfern, 1999; Caglar et al., 2009; Derkowski et al., 2012; Onal and Sarıkaya, 2007; Środoń and MaCarty, 2008; Waclawska, 1984). Thus, if the standard oven-drying method is used to dry bentonites, the measured water content is probably underestimated.

An underestimate of water content will result in an underestimate of SSA and CEC; further, SWCC to move upwards in the graph of chemical potential versus water content. Thermal analysis indicated that if 110°C was used to dry montmorillonites, CEC and SSA would be underestimated by up to 6% and 7%, respectively (Środoń and MaCarty, 2008). A slight shift of the SWCC of bentonites due to correcting water content can induce significant underestimation of total potential. For instance, if the water content of a bentonite was underestimated by 3%, the total potential after correcting the water content could be several times higher than the uncorrected one. Thus, it is crucial to perform a complete and systematic investigation to quantify, identify, interpret, and remove the residual water in the bentonites dried by standard oven-drying.

Layer charge density, the nature of the exchangeable cations, and drying time and temperature influence the dehydration of montmorillonites. After montmorillonites were dried at 110°C, the amount of their residual water depended on the layer charge density (e.g., Emmerich et al. (2015), Środoń and MaCarty (2008)). Compared to the monovalent cation montmorillonite, the divalent cation montmorillonites required a higher temperature for complete dehydration (e.g., Cuadros (1997), Morodome and Kawamura (2009, 2011), Waclawska

(1984)). The first aim of this study is to evaluate the standard oven-drying method for determining the water content of a calcium-rich bentonite. The second aim is to study the effect of the drying time and temperature of standard oven-drying on the amount of the residual water of the bentonite. The last aim is to identify and interpret the residual water and to propose an effective and practical drying profile. To fulfil these objectives, thermogravimetric-differential scanning calorimetry (TG-DSC) tests were first performed to obtain the true water content. Based on the true water content, the effect of the drying time and temperature on the amount of residual water in the bentonite was then examined, including the oven-drying and vacuum-drying methods. To identify and interpret the residual water, changes in the SSA and the dehydrating reaction of the samples with decreasing residual water content were obtained.

2. Materials and methods

2.1. Materials

The bentonite used in this study was a white powder that was provided by Shouguang Montmorillonite Group Co., Ltd, China. This bentonite was produced by physically crushing bentonite rock and purified by hydraulic elutriation. The particle size of this bentonite was less than 2 μm , and montmorillonite composed more than 95% of the mineral constituents of this bentonite. To confirm production information provided by the company, the mineral constituent, chemical composition, cation exchange capacity, and total specific surface area of this material were measured.

Identification and quantification of minerals in the bentonite powder were performed by XRD (D8-FOCUS with Cu-K α radiation, Ni filter, at 40 kV–40 mA) with $0.02^\circ 2\theta$ and 2 s steps in the 2–66° range. The XRD patterns shown in Fig. 2 were analysed with the software program, including Rietveld refinement to carry out phase quantification. Based on Rietveld based quantification results, this bentonite comprised 95% montmorillonite and 5% quartz, which agreed well with the production report from the company.

The chemical composition of the bentonite after drying at 105°C for 4 h was (in mass percentage): SiO₂, 61.52; Al₂O₃, 16.62; Fe₂O₃, 4.73; MgO, 5.45; CaO, 2.73; Na₂O, 0.13; K₂O, 0.11; MnO, 0.03; TiO₂ 0.30; P₂O₅, 0.03, and loss on ignition (LOI) 8.88. The chemical analysis was performed by X-ray fluorescence (XRF). According to the chemical analysis, the half unit structural formula of montmorillonite was determined based on Stevens (1946) (the calculation details are given in the appendix). Therefore, the montmorillonite in this bentonite was a calcium-dominant montmorillonite, which was further confirmed by

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