



Research paper

Influence of preparation methods on textural properties of purified bentonite



Liaqat Ali Shah^{a,b,*}, Maria das Graça da Silva Valenzuela^c, Muhammad Farooq^d,
Shaukat Ali Khattak^e, Francisco Rolando Valenzuela Díaz^c

^a Department of Physics, University of Peshawar, Peshawar, Pakistan

^b State Key Laboratory of Multiphase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, Beijing, China

^c Polytechnic School, University of Sao Paulo, 05508-900 São Paulo, SP, Brazil

^d National Center of Excellence in Physical Chemistry, University of Peshawar, Peshawar, Pakistan

^e Department of Physics, Abdul Wali Khan University Mardan, Mardan 23200, Pakistan

ARTICLE INFO

Keywords:

Bentonite
Soda activation
Purification
Microporosity
Mesoporosity
Montmorillonite

ABSTRACT

The aim of this work was to study the effect of three different purification methods, using chemical solution of $(\text{Na}(\text{PO}_3)_6)$ (method-I), NaCl (method-II) and Na_2CO_3 (method-III), followed by Stokes law of sedimentation, on the textural properties of a Pakistani bentonite, containing $> 70\%$ Ca^{2+} -montmorillonite. The raw and the purified samples were examined through X-ray Diffraction (XRD), N_2 adsorption-desorption, and laser diffraction method (LDM). The quartz contents were found absent in all the purified samples. It was confirmed that the sample obtained by the method-I and other two methods resulted in to be, typically, Ca^{2+} -bentonite and Na^+ -bentonite, respectively. In comparison with the raw sample, higher percentage volume of fine particles was detected in purified samples. The fine particles, however, exhibited significant enhancement in the samples purified by method-II and method-III. Higher pore volume and specific surface area were noticed for samples purified by the trio methods in comparison with the raw sample. Specific surface area of the sample obtained by the method-III was much higher than that of the other purified samples. Mesopores volume was increased significantly by purification, especially by the method-I and III. Considerably higher value of micropores volume/area and mesopores volume/area were detected for sample purified by method-III than for the samples purified by the other methods. A highly porous sample with the excellent surface area and small particle size was obtained by purification method-III which can be used as a good adsorbent, catalyst and catalyst support.

1. Introduction

Bentonite is the most important industrially applied clay because of its main montmorillonite (Mt) component which gives it a very high sorbent property and swelling capacity (Caglar et al., 2009; Elzea and Murray, 1990; Murray, 2007). As compared to pure smectite, bentonite is relatively impure, containing Mt and other clay/nonclay minerals. The amount of pure Mt in bentonite rock is the key to its purity and performance. Higher the contents of the Mt in bentonite rock, the better it will execute as an industrial raw material (Murray, 2007; Thuc et al., 2010). The particle size of Mt usually ranges from 0.1 to $2\ \mu\text{m}$ with an average particle size of about $0.5\ \mu\text{m}$ (Grim and Güven, 1978; Thuc et al., 2010). The expected presence of other clay, non-clay mineral, heavy and trace elements, and pathogenic microorganism in bentonite dilutes its potential use in industries (Bergaya and Lagaly, 2013; Choo and Bai, 2015; Lopez-Galindo et al., 2007; Massat et al., 2016; Murray,

2007). It is well known that the macroscopic properties and application of bentonites are closely connected to the quantity and quality of Mt, pH, particle size and their porous microstructure, which are usually characterized by quantities such as their surface area, porosity, pore size, pore size distribution, and pore geometry (Altin et al., 1999; Ayari et al., 2005; Barrer, 1989; Bergaya and Lagaly, 2013; Bihannic et al., 2009; Carretero and Pozo, 2009, 2010; Dardir et al., 2018; Gamiz et al., 1992; Grim and Güven, 1978; Kaufhold, 2006; Keller et al., 2013; Likos and Wayllace, 2010; Lopez-Galindo et al., 2007; Massat et al., 2016; Murray, 2007; Neaman et al., 2003; Onal and Sankaya, 2007; Onal et al., 2001; Pinnavaia, 1983; Srasra et al., 1989; Takashima et al., 1993). The high contents of Mt contained in bentonites are the key source of the porosity. The International Union of Pure and Applied Chemistry (IUPAC) classified the pores by their internal pore width (Sing et al., 1985). The pores with internal widths $> 50\ \text{nm}$, between $2\ \text{nm}$ and $50\ \text{nm}$, and smaller than $2\ \text{nm}$ are classified as macropores,

* Corresponding author at: Department of Physics, University of Peshawar, Peshawar, Khyber Pakhtunkhwa, Pakistan.
E-mail address: liaqat@upesh.edu.pk (L.A. Shah).

mesopores, and micropores, respectively. These pores often exist in clay including bentonites in their dry state and have been described by the following structural units: layer (in case of smectite, its thickness is about 1 nm and lateral extension is 1–2 μm), particle (consists of elementary clay minerals layers), aggregate (consists of particles); and by arrangement of these units in space (Jullien et al., 2005; Kuila and Prasad, 2013; Massat et al., 2016; Neaman et al., 2003; Sedighi and Thomas, 2014; Touret et al., 1990). First, the micropores, which correspond to the clay structural unit layers, reported in the literature are interpreted in a number of ways: Diffusion between the clay layers is one of the interpretations (Thomas and Bohor, 1968), however, such an explanation can only be considered for bulky interlayer cations such as Cs^+ ion (Berend et al., 1995; Cases et al., 1997; Michot and Villieras, 2013; Rutherford et al., 1997). The other currently accepted interpretation in case of smectite is the turbostratic piling of elementary clay minerals layers in particles, which creates slit-shaped micropores on the broken edges of the layers (Alymore et al., 1970; Cases et al., 1992; Quirk and Aylmore, 1971). The presence of preferential adsorption sites on the basal (planer) surfaces of smectite is also one of the interpretation when using a classical procedure such as α_s -plots or t-plot (Michot and Villieras, 2013). Second, the particles arrangement within the aggregates creates the inter-particle pores, which are typically in the size range of mesopores (2–40 nm) (Jullien et al., 2005; Neaman et al., 2003). Lastly, aggregates, on their turn, are arranged in clusters. The aggregates arrangement inside clusters may contain inter-aggregate pores in the mesopores size range (> 40 nm) and macropores (Kuila and Prasad, 2013; Neaman et al., 2003).

Indeed, the porosity especially microporosity originated from the turbostratic piling of elementary layers in particles is expected to be strongly influenced by the nature of the clay such as size of the particles, size of elementary layer, the layer charge, nature of cations, and preparation condition (Kuila and Prasad, 2013; Michot and Villieras, 2013; Neaman et al., 2003).

Bentonite can be used both as found naturally and after some physicochemical treatment such as ion exchange, acid leaching and heat treatment according to the application area (Dardir et al., 2018; Komadel, 2003; Mukosa-Tebandeké et al., 2015; Onal and Sarikaya, 2007; Stojiljkovic et al., 2015; Stojiljković et al., 2013; Yener et al., 2012). The purification process is also used to improve the quality and quantity of bentonite. Different purification methods have been introduced in the literature to separate Mt (< 2 μm) from bentonite materials such as sieving (Chipera et al., 1993; Ottner et al., 2000), magnetic separation (Chipera et al., 1993), sedimentation based on Stokes' law (Dontsova et al., 2004; Kaufhold et al., 2002; Lee and Kim, 2002; Patel et al., 2007; Sato, 2005; Shah et al., 2013b; Thuc et al., 2010), and centrifugation (Chipera and Bish, 2001; Gong et al., 2016; Ottner et al., 2000; Thuc et al., 2010). The porous structure of bentonite can be altered during hydration and dehydration processes (Saiyouri et al., 2000; Salles et al., 2009). It has been reported that, during the hydration of bentonite, the number of layers in a particle reduces significantly, forming smaller particles of about 10 layers (Saiyouri et al., 2000; Saiyouri et al., 2004). It is well known that the Na⁺-smectite can swell during hydration to a higher degree and its interlayer space can, theoretically, increase to infinity, leading to the formation of individual 2:1 layers as separate particles. In contrast, Ca²⁺-smectite has limited swelling due to both the van der Waals and electrostatic attractions between negatively charged 2:1 layers and interlayer cations (Segad et al., 2010). This swelling characteristic may result in either an increase in size of the particle via water intercalation or decrease by exfoliation. The purification of bentonite by sedimentation is also one of the hydration and dehydration processes. The different chemical treatment such as NaCl, Na₂CO₃, (Na PO₃)₆ and NaOH etc. before purification by sedimentation process can alter the swelling of bentonite differently and thus the level of purity, size of the particle and the porous structure of bentonite. However, a comparative study of the chemically modified bentonite followed by sedimentation process on

the particle size distribution, level of purity, pore size distribution, average pore size, specific surface area and their mutual correlation is scarce.

The objectives of this study are: First, to investigate the effect of three different preparation methods on particle size distribution, pore size distribution, specific surface area and average pore size of a purified bentonite. Second, to probe the effect of preparation methods on dispersion process, during particle size analysis by laser diffraction-based particle size analyzer, level of purity, and micro-porosity of two different purified Na⁺-bentonites. The findings of the research are expectedly of interest to the researchers working on the clay and clay minerals processing for the use of different industrial applications.

2. Material and methods

2.1. Collection of sample

The bentonite in raw form was collected from Peshawar mine of Khyber Pakhtunkhwa province, Pakistan. The sample was dispersed in distilled water and passed through a sieve of mesh No-325 (ASTM). The sieved clay suspension was then dried, ground and designated as "PWR-O". The selected bentonite PWR-O mainly consists of Ca²⁺-Mt (77%), illite (< 1%), albite (16%) and quartz (6%) as investigated (Shah et al., 2017).

2.2. Purification of raw bentonite

2.2.1. Method-I: simple sedimentation using (Na PO₃)₆ as a dispersing agent

5 g of bentonite was added to 1 L deionized water containing 0.25 g (Na PO₃)₆ as a dispersing agent and stirred for about 1 h. The dispersion was placed undisturbed at 25 °C and after 15 h, the supernatant above 10 cm depth was decanted to clean beaker. The process of sedimentation was applied successively till the clay mineral particles collected in the supernatant were found negligible. The separated part of supernatant was then washed, dried and ground to fine powder. The sample purified by this method was labeled as "PWR-I".

2.2.2. Method-II: NaCl treatment followed by Stokes law of sedimentation

In this method, the sample was first treated with NaCl and then washed to get the chloride free slurry. The Stokes law of sedimentation was applied to obtain purified Na⁺-bentonite. The detail of the procedure as described elsewhere (Shah et al., 2013a,b). The sample purified by this method was designated as "PWR-II".

2.2.3. Method-III: Na₂CO₃ activation followed by sedimentation

To obtain pure sodium Mt, the soda activation procedure, that has recently been described, was applied (Shah et al., 2013a). 5 g of Na₂CO₃ per 100 g of clay sample was added to 1000 mL boiling water and stirred continuously for 1 h. After stirring; the activated dispersion was diluted and placed undisturbed for 24 h to settle down the impurities. The supernatant was then decanted and the process of sedimentation was repeated until the supernatant became clear. The collected supernatants were centrifuged at 10000 RPM and the separated clay sample was dried at 60 °C. The sample was further ground and labeled as "PWR-III".

2.3. Methods of investigation

2.3.1. X-ray diffraction

X-ray diffraction (XRD) technique was used to study the effect of modification and purification on raw sample. The oriented samples for raw (PWR-O) and purified bentonites were prepared. X-ray diffraction patterns of entire samples were obtained by D500 diffractometer equipped with monochromator of graphite using CuK α radiation. All the samples were scanned from 2° to 40° 2 θ range using 40 mA and

Download English Version:

<https://daneshyari.com/en/article/8045665>

Download Persian Version:

<https://daneshyari.com/article/8045665>

[Daneshyari.com](https://daneshyari.com)