Contents lists available at ScienceDirect

Applied Clay Science



Research paper Physical properties and Na-activation of Egyptian bentonitic clays for appraisal of industrial applications



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ARTICLE INFO

Article history: Received 26 June 2015 Received in revised form 8 August 2015 Accepted 10 August 2015 Available online 4 September 2015

Keywords: Bentonitic clays Egypt SSA Swelling index Na-activation Industrial applications

ABSTRACT

Selected physical properties of bentonitic clays in the north Western Desert, Egypt were investigated to assess their potential use in industrial applications. Assessment tests included specific surface area (SSA), swelling index (SI), green (GCS) and dry (DCS) compressive strengths, rheological properties, and filtrate volume. The impact of Na-activation on swelling index and rheological properties was also considered. SSA, SI after Naactivation, GCS, and rheological properties were highly correlated with smectite content and a modified AgTU-CEC. Mineral impurities greatly influenced the physical properties. Quartz and feldspars reduced the rheological properties and increased the water loss. Halite and colloidal iron oxides increased swelling index and enhanced rheological properties. DCS was greatly influenced by the percentage of added water and kaolinite abundance. Na-activation improved the swelling index and rheological properties of halite-poor samples. The swelling index of Na-activated samples was a useful tool for determining the quality and grade of bentonitic clays. The suitability of the Egyptian bentonitic clays for drilling mud and bonding of foundry sands was determined by comparing the results with a standard bentonite and published industrial specifications. Egyptian bentonitic clays showed comparable compressive strengths with Wyoming Na-bentonite (SWy-1) by adding 8 and 10 wt.% clay to sand molds and can be used for most metal-type castings. Egyptian bentonitic clays with a high swelling index and low quartz and feldspar content, produced comparable rheological properties with the Wvoming Na-bentonite and met international drilling mud specifications. However, the filtrate volume measurements indicated that the samples require additional modifications to reduce water loss. Many desirable bentonitic clay properties were reduced by large quantities of kaolinite.

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1. Introduction

Assessment is a critical stage in the development or exploitation of bentonitic clay deposits (Harvey and Lagaly, 2013). Once a potential deposit has been identified, its physical and chemical properties have to be determined. Assessment involves laboratory tests of general properties and specific tests related to intended uses. Assessment also involves geologic, mineralogic and petrographic investigations. The geologic origin (Galán and Ferrell, 2013) of the clay has a major impact on its physiochemical characteristics. Clays are used by the industry in bulk form without significant beneficiation or after various physical or chemical processing techniques (Pickering and Murray, 1994; Christidis et al., 1997; Pruett and Pickering, 2006; Murray, 2007). Recently, special attention was directed to the applications of high quality clay minerals in the formulation of clay–polymer nanocomposites (CPN) (Bergaya

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and Lagaly, 2007; de Paiva et al., 2008). The complexities of clay mineral science and some of the relationships among assessment variables have been detailed in the *Handbook of Clay Mineralogy* (Bergaya and Lagaly, 2013a, 2013b).

Today bentonite is an essential raw material in metal and oil production as well as numerous applications of the raw or processed forms for pet litter, waterproofing, sealing, animal feed, fillers, fertilizers, extenders, filtering, catalysts, clarifying, and decolorizing. The "best-use" of bentonite for increasingly complicated and diverse applications depends on an understanding of clay mineral structures, chemical compositions, and physical properties (Eisenhour and Brown, 2009). Several protocols for assessment involve simple and/or complicated test procedures; color, grain size distribution, purity, methylene blue absorption, surface area, Atteberg limits, cation exchange capacity, exchangeable cations, thermal analysis, viscosity and swelling, electron beam microscopy, compactability, compressive strength, chemical composition, and mineral content by XRD or infrared spectroscopy (Inglethorpe et al., 1993). Unique physico-chemical characteristics of bentonites such as sub-micrometer particle size, high cation exchange capacity (CEC),



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large surface area (up to $800 \text{ m}^2/\text{g}$), and variable chemical composition (Eisenhour and Brown, 2009; Güven, 2009) were frequently related to smectite content (i.e. grade).

Assessment tests may be general or specific related to a particular application. For bentonite and other industrial clays, three distinct stages of laboratory assessment are suggested (Christidis, 2011): (i) identification of the clay minerals and associated minerals; (ii) determination of important physical and chemical properties of the clays; and (iii) comparison with standards. The first stage is useful to assess "grade" of bentonite based on smectite content. The second stage can evaluate "quality"; a measure of industrial performance that is related to physico-chemical properties (Inglethorpe et al., 1993). Many industrial associations have established standards for specific categories of use.

Kaolinite- and smectite-rich clay deposits of variable age occur widely within the Nile valley and delta, and the Western and Eastern Deserts of Egypt. Clay deposits with bentonitic characteristics have a long history of use extending, in Egypt, from the time of the Pharaohs to now. Many are used in the production of ceramics, including refractories, white ware, heavy clay products, and Portland cement. Clays in the Fayoum and Alamine areas with swelling characteristics were assessed recently for use in the production of lightweight aggregate (Ismail et al., 2014). Some of the clay deposits contain enough smectite to classify them as bentonitic clays or bentonites with potentially higher value usages.

Mineralogical variability in existing bentonitic clay quarries within the northern Western Desert of Egypt revealed differences that were correlated statistically with Province, age and quarry with a discriminant function analysis (DFA) (Agha et al., 2012). Differences in the mineral content were related to the paleo-weathering environment and sorting by detrital processes (Agha et al., 2013). The relatively low smectite content and high kaolinite content of all but one of the quarries justified their designation as bentonitic clays rather than purer smectitic bentonites derived from volcanic sources. Most of the samples lacked the high smectite content and chemical purity (Agha et al., 2012; Agha, 2015) contributing to the special properties of Na-bentonite from Wyoming.

The chief goal of this investigation was to answer the following questions: Do the physico-chemical characteristics and clay quality measures provided by laboratory assessment suggest ways that the industrial usage of the Egyptian clays might be expanded? What are the relationships between easily determined physical properties (specific surface area and swelling index) and mineralogical and chemical characteristics? Does Na-activation play a role in these relationships? Answers to these questions should be of benefit to the clay industry in Egypt and in other countries where similar clay deposits are located.

2. Methods and materials

Thirty one representative samples were selected for evaluation from 10 quarries in the north Western Desert collection used to support the earlier mineralogical and geological reports prepared by the authors (Agha et al., 2012, 2013). Approximate locations of the sample are summarized in Table 1. Quarry numbers are generally lower in the North. For more information on locations refer to Agha et al. (2013). Sample selection was based on composition and the availability of enough material to conduct assessment tests, including extractable cations, cation exchange capacity, specific surface area, swelling index, viscosity and binding strength. The CMS Na-bentonite Source Clay, SWy-1, was used as a comparison standard for physical properties related to industrial applications.

The whole-rock and clay mineralogy were reported in detail earlier (Agha et al., 2012). Mineral identification was carried out by matching observed d-values with those in MacDiff 4.2.5, and quantitatively

Table 1

Samples used in assessment tests, quarry location and stratigraphic age.

Quarry	Sample	Location	Name	Age
Q2	s007	South of Alamein city	Deir Abul Hegif	Middle Miocene
Q4	s021	South of El Hammam city	El Barkan	Lower Miocene
Q5	s029	Wadi El Natrun	Deir El Baramous	Upper Pliocene
Q6	s033	El Fayoum Depression	Qasr El Sagha	Upper Eocene
	s061			
Q7	s063		Kom Oshim	
Q8	s083		Girza	Middle Eocene
	s091			
Q9	s099		Qalamshah	
	s109			
Q10	s111		Cemetery	?
Q11	s113		Shaklofa	Middle Eocene
Q12	s119		Reigha	?

assessed with the whole-pattern, least-squares fitting method in CLAY + + (Ferrell, 2006; Ferrell and Dypvik, 2009).

A JEOL JSM 840A scanning electron microscope with energy dispersive X-Ray spectroscopy (SEM/EDX) was used to determine the elemental composition of individual grains of all bentonitic clay samples. Freshly broken samples (\leq 5 mm thick) were coated with gold and viewed perpendicular to bedding in the secondary electron imaging mode (SEI).

CEC and extractable Ca, Mg, Na, and K were determined with a variation of the AgTU method of Dohrmann (2006) that avoided the initial rinsing of the solids with distilled water. Extractable cations are those displaced from the clay by AgTU ion exchange as well as the dissolution of soluble minerals or evaporated pore solutions by the reagent solution. Two sample weights (15 and 30 mg) were used to identify the effects of partially soluble minerals on the extracted element quantities.

Specific surface area (SSA, area/mass of dry clay) determinations utilized the Ethylene Glycol Monoethyl Ether (EGME) method (Cerato and Lutenegger, 2002). Thirty one samples were analyzed in 12 batches. For reliability, two replicates of each sample were measured. The quality control material, SWy-1, was employed for each batch of samples. The precision of this method yielded a coefficient of variation (CV) less than 0.1, or CV% = 10%, in accordance with Cerato and Lutenegger (2002). SSA (m^2/g) was calculated based on a K value ($2.86 * 10^{-4} g$) representing the weight of EGME that is required to form a monomolecular layer covering 1 m^2 of clay surface.

Thirteen samples representing a wide range of smectite and kaolinite contents were subsequently selected from the 31 used for SSA to measure swelling index, compressive strength, rheological properties, and filtrate volume. Swelling index (SI) was determined using the method of Inglethorpe et al. (1993) that compares the volume of 10 g of clay after water saturation to the original dry powder volume. Additional aliquots were Na-activated by 1, 2, 3, 4, 5, and 6 wt.% anhydrous sodium carbonate (analytical grade) and tested to determine the maximum swelling capacity (ratio of maximum swelling index of activated sample to raw material * 100).

Compressive strength was determined for wet (green compressive strength, GCS) and dry sand molds (dry compressive strength, DCS) to evaluate the suitability of the Egyptian clays for use as binders for molding sands in the foundry industry. The test specimen size and geometry were prepared in accordance with the American Foundrymen's Society (AFS) standard specification for the preparation of molding sand test samples as described in Ademoh (2008). Washed and screened (400–200 µm) Quikrete Premium Play Sand No. 1113 was used for the molding sand. Percentage dry weight (Ps%) for the -400 µm bentonitic clay additions were calculated according to Inglethorpe et al. (1993). Different percentages of clay (5, 8, and 10 wt%) and moisture contents (2, 3, and 4 wt.%) were added to the sand and thoroughly mixed. Green sand molds (i.e. wet) were tested immediately after core preparation. Each sample was tested twice and the average result was reported. Dry

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