



Research paper

Characterization of rectorite from the Beatrix Gold Mine in South Africa

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ARTICLE INFO

Article history:

Received 20 September 2015

Received in revised form 23 February 2016

Accepted 24 February 2016

Available online 2 March 2016

Keywords:

Rectorite

Mixed-layer

Interstratification

XRD

SEM

Solid-state NMR

Thermal analysis

FTIR

ABSTRACT

Three rectorite samples from the Beatrix Gold Mine, South Africa were characterized. Scanning electron microscopy revealed a layered morphology. High resolution transmission microscopy showed well distinguished light and dark layers of about 2.20 nm consistent with the 1:1 interstratified mica-smectite nature. X-ray diffraction measurements confirmed the basal spacing d_{001} of 2.20 nm consistent with a one-water-layer structure. Unit cell parameters, for a monoclinic unit cell with primitive lattice, refined to $a = 5.177 \text{ \AA}$; $b = 8.980 \text{ \AA}$; $c = 22.489 \text{ \AA}$ and $\beta = 97.335^\circ$ with mean crystallite size around 14 nm and calculated cell volume of 1045 \AA^3 . The Greene-Kelly test suggested that the expandable smectite layers have montmorillonite-beidellite composition. Nuclear magnetic resonance spectroscopy indicated a high degree of Al substitution and the presence of two different Al sites corresponding to six- and four-fold octahedral and tetrahedral aluminum respectively. The chemical composition and diffraction data suggest that the mica is Na-Ca-rich, i.e. of paragonite-margarite series. The fixed interlayer regions (mica interlayers) contains proportionally dominant Na^+ and Ca^{2+} and minor amounts of K^+ . The exchangeable smectitic interlayers contain almost equal amounts of Na^+ and Ca^{2+} ions. The distribution of the interlayer Na^+ ions was quantified by ^{23}Na solid-state NMR spectroscopy. It points to a three component mixed-layer structure with considerable variation in the composition of the mica layer of the different samples.

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1. Introduction

Rectorite is a 1:1 regular interstratification of dioctahedral mica and dioctahedral smectite (Bailey, 1981; Bailey et al., 1982; Brown and Weir, 1963). The swelling of the mineral is caused by the hydration of the exchangeable cations of the montmorillonite-like layers.

Rectorite occurrence of economic value exists in China (Hanlie et al., 2008). Rectorite was also discovered in the Beatrix Gold Mine located near Welkom, South Africa (von Rhaden, 1994). There it is found in modified shale bands, up to 0.5 m thick, both in the hanging and foot-wall of the Beatrix reef close to a major underground fault. The bands consist of low-grade metamorphic rock with mineral composition of quartz, muscovite, paragonite, chlorite, pyrophyllite, rectorite, smectite and chloritoid. The rectorite formation is associated with the alteration of other phyllosilicates, pre-existing pyrophyllite in particular, under metamorphic conditions of at least 350°C and 2.5 kbar (von Rhaden, 1994). The shale has been associated with gold losses at the mine in the process of physical and chemical extraction of the gold (Van Vuuren et al., 2000). Beatrix Mine rectorite is a Na-Ca-rich three-

component mixed layer made up of smectite, paragonite and margarite (Jakobsen et al., 1995; Lausen et al., 1999).

The mixed-layer structure of rectorite, made up of alternating non-expandable (mica) and expandable (smectite) layers in a 1:1 ratio, defines a set of specific properties that differ from those of mica and smectite alone. For example rectorite dispersions provide for unique behavior, e.g. facilitating the synthesis of ultralight gels that are useful as adsorbents (Lu et al., 2015; Zheng et al., 2013). The present interest is in pursuing the use of this mixed-layer clay as nano-additive in polymer-based nanocomposites where it has also been found to impart desirable mechanical properties, e.g. improving the ductility of poly(lactic acid) (Li et al., 2009). This communication provides characterization data on Beatrix rectorite and is a first step towards this goal.

2. Materials and methods

2.1. Materials

Three samples containing rectorite from Beatrix Mine were obtained and examined. Sample RT1 was collected in 1995, sample RT2 in 2010 and sample RT3 in 2012. The samples differ in their visual appearance with RT1 containing large light blue-grey platy aggregates with silky luster, while RT2 and RT3 represented disintegrated host rock mine

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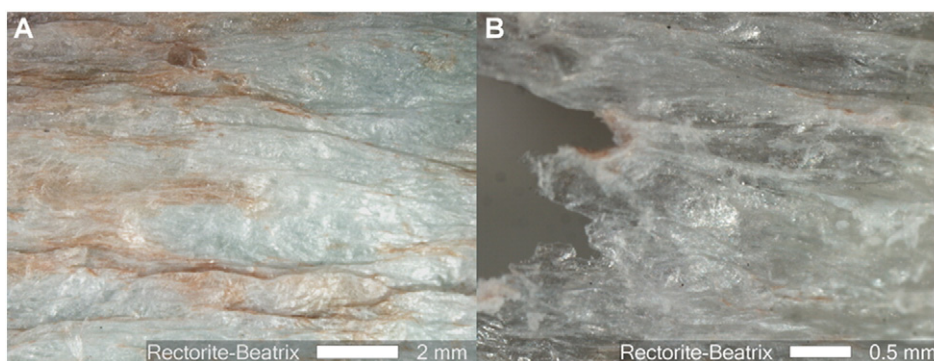


Fig. 1. Binocular microscope photographs of Beatrix rectorite sample RT1. (A) Compact aggregate. (B) Translucent to transparent at sharp edges thin fragment (right).

material. All samples were subjected to various purification procedures aimed to produce high purity material for various tests and experimental applications. Unless otherwise indicated, the characterization presented in this paper is based on purified material with rectorite content above 90 wt% (as determined by XRD analysis). Structurally and chemically the purified materials from the three samples were very similar and were categorized as rectorite, except where applicable specifics are highlighted.

2.2. Sample preparation

Sample preparation was applied as carefully as possible to prevent any physical distortion of the clay structure that may affect the experimental results. The general guidelines on sample preparation for clay minerals (Brindley and Brown, 1980; Moore and Reynolds, 1997) were followed with slight adaptations to accommodate specifics encountered in the preparation process.

2.2.1. Purification

Where applicable, the raw rectorite was first disaggregated with a laboratory jaw crusher (sample RT1) to break down large platy aggregates to around 1–2 mm, followed by further size reduction in a mixer-grinder. Samples RT2 and RT3 were sieved and the <75 μm fraction used for further purification. Material (ca. 50 mg) was dispersed ultrasonically in deionized water (~250 mL) to allow disaggregation and the formation of a suspension. A settling time of 5 min was established for optimal recovery of the clay fraction. The solid was separated from the water by centrifugation at 2000 rpm for 30 min. For oriented preparations, the clay residue, mixed with small amount of water, was deposited onto glass slides by a pipette and air-dried (AD) for XRD analysis. Glycerol (G) and ethylene glycol (EG) saturation was achieved by the vapor method. The AD oriented mounts were placed in a desiccator with glycerol or ethylene glycol for 16–18 h overnight at a temperature of 60 $^{\circ}\text{C}$ and analyzed again on the X-ray diffractometer. The

nature of the expandable component in the mixed-layer structure was determined by the Greene-Kelly test. It entails X-ray diffraction of Li-saturated oriented specimens after heating at 300 $^{\circ}\text{C}$ followed by glycerol saturation (Greene-Kelly, 1952). The test was duplicated with high repeatability of the results. Relative humidity in the laboratory was maintained between 50% RH and 60% RH.

Removal of the calcium carbonate (12–15 wt%) impurity from sample RT1 was accomplished by using 0.3 M acetic acid following a standard method (Ostrom, 1961). The separation was performed at room temperature and the process was closely watched by monitoring the pH after each cycle. The samples were repeatedly washed after effervescence had subsided. X-ray diffractograms were compared before and after exposure to acid to ensure that no damage to the rectorite structure had occurred.

Saturation of rectorite with various cations, i.e. lithium, sodium, potassium, strontium and barium, was achieved by treating about 20 g of sample with 1 M solution of the respective chlorides. The exchange procedure was carried out using a shaker for three days at room temperature. The frequency of the shaker was adjusted to 225 rpm. The saturation was repeated three times per day replacing the liquid each time with fresh solution containing an excess of the exchanging cation. The last round was completed by multiple washing with deionized water through three shaking sessions to remove excess chloride. To ensure that all chloride ions are removed a test was performed by reaction of an aliquot of supernatant solution with 0.1 M AgNO_3 . The washed clay was separated by centrifugation in acetone for 30 min at a speed of 2000 rpm.

Preparations for powder XRD (pXRD) measurements utilized a McCrone micronizing mill with corundum grinding elements. It reduced the mean particle size of the milled sample to ensure homogeneous subsample for X-ray diffraction. About 4 g of milled sample was ground with 10 mL ethanol for 8 min and each sample was prepared in duplicate. Calibration mixtures prepared by adding ZnO (or corundum) as an internal standard were ground and homogenized following

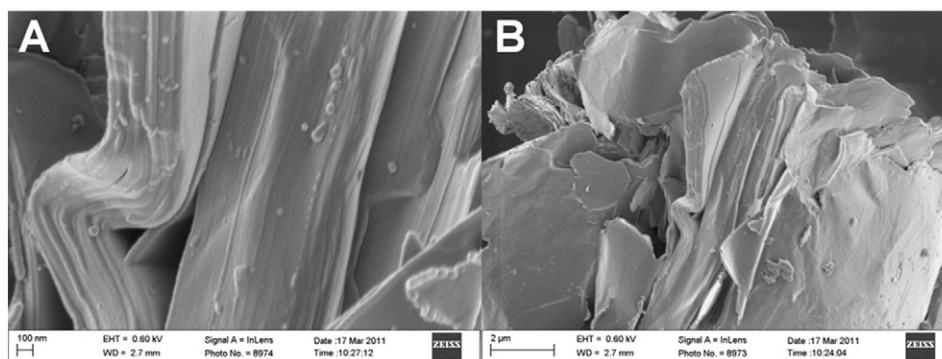


Fig. 2. SEM images of platy folded and curled rectorite aggregates displaying typical layered textures (sample RT1).

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