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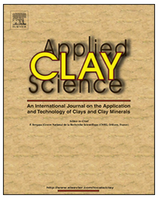

Research paper

Roasting-leaching experiments on glauconitic rocks of Bakchar ironstone deposit (Western Siberia) for evaluation their fertilizer potential

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ABSTRACT

This study examines factors favouring optimal release of potassium from a glauconitic rock by moderate roasting-leaching. Sieving and electromagnetic separation of oolitic iron ore deposit increases the concentration of glauconite grains substantially. Roasting and chemical leaching of this concentrate enhances the release of potassium as interlayer structures of glauconite collapses. This study records the percentage of potassium recovered from glauconite under different experimental conditions, viz., temperature, reaction time, concentration as well as volume of HCl and stages involved in the leaching treatment. Leaching of glauconite without roasting results a low recovery (< 15%) of K at room temperature. The recovery of K content from the sample increases markedly with increase in concentration of HCl, leaching time, and the ratio by mass of sample to HCl, and marginally with increased stages of leaching. Roasting at 900 °C recovers maximum K up to 62–63% for one-stage leaching treatment at 100 °C for 120 min with a 4 $mol \cdot l^{-1}$ HCl solution for a sample: HCl ratio of 1:5. The roasting-leaching method, therefore, holds promise for converting glauconite to potash salts for agronomic applications.

1. Introduction

Potassium is considered to be one of the vital macro-nutrients in the agricultural sector (Manning, 2010; Prakash and Verma, 2016). Its main source is marine potassium salt deposits, found largely in Canada, Russia, Belarus, Chile, Germany, and USA (Manning, 2010; FAO, 2015; Zheng et al., 2015; Al Rawashdeh et al., 2016; Dmitrieva et al., 2017). Increasing demand, limited availability and rising prices of potash fertilizers have motivated researchers to find alternative sources (Coles et al., 2002; Zharikova and Golodnaya, 2009; Rahimzadeh et al., 2015; Santos et al., 2016; Torqueti et al., 2016; Bakhshandeh et al., 2017; Basak et al., 2018). One of the most promising alternative sources for potash could be glauconite (Manning, 2010; Karimi et al., 2012; Franzosi et al., 2014; Shekhar et al., 2017a; Rudmin et al., 2017a, 2017b). Glauconite is a di-octahedral, iron-rich phyllosilicate mineral consisting of 2:1 layers linked by interlayer K cations (McRae, 1972; Odin and Matter, 1981; Drits, 1997; Drits et al., 2010). It contains variable amount of K_2O depending on factors like depositional environment, substrate composition and stratigraphic condensation (Banerjee et al., 2012a, 2012b, 2015, 2016; Bansal et al., 2017).

Glauconite concentrate act as an effective fertilizer when added to soil (Castro and Tourn, 2003; Heckman and Tedrow, 2004; Karimi et al., 2012; Merchant, 2012; Franzosi et al., 2014; Rudmin et al., 2017b). Chemical or biochemical leaching of glauconite produce potassium salts (Yadav and Sharma, 1992; Mazumder et al., 1993; Rao and Rao, 1999; Yadav et al., 2000; Shekhar et al., 2017a, 2017b). Previous studies investigated the recovery of potassium from glauconite using high concentration of acids, for example, 6 $mol \cdot l^{-1}$ hydrochloric acid (Yadav and Sharma, 1992) and 9 N sulphuric acid (Rao et al., 1993). Roasting and addition of salts effectively removes potassium from glauconite (Rao et al., 1993). Recently Shekhar et al. (2017a, 2017b) recovered 98% potassium from a glauconitic sandstone under rather moderate conditions of leaching and reduction roasting.

This study investigates potential of glauconitic rocks of Western Siberia as non-conventional source of potassium fertilizer. These rocks occur within the Upper Cretaceous oolitic ironstone deposits which are removed as tailings after extraction of iron (Rudmin et al., 2017b). This study examines the factors favouring the optimal release of potassium salts from glauconite rocks by roasting and chemical leaching. We have attempted to explore the recovery of potassium salts from glauconite rocks by using moderate chemical conditions in lieu of hazardous means of most other investigations (Yadav and Sharma, 1992;

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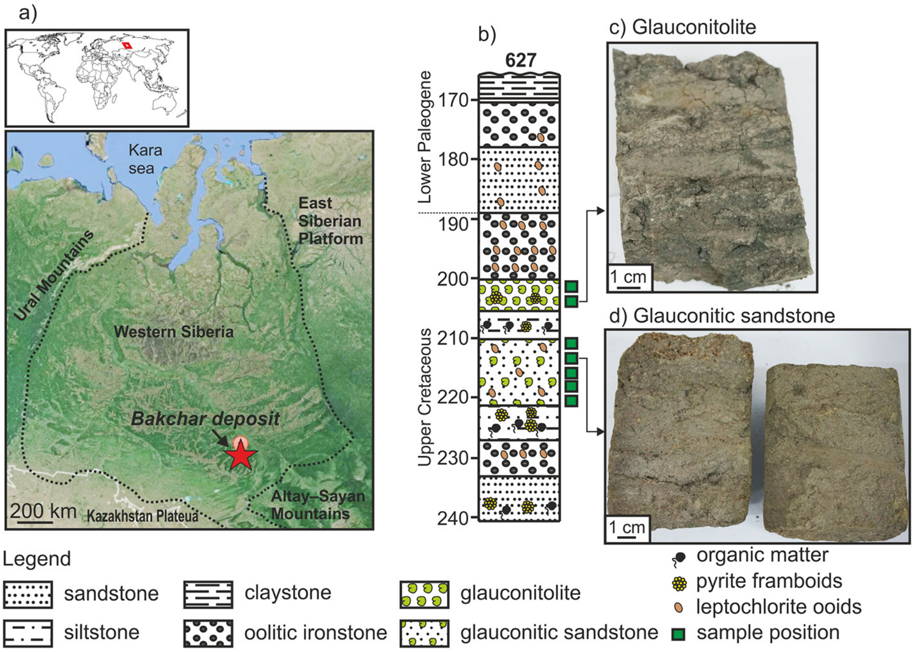

Fig. 1. Location map of studied deposits in Western Siberia (a), stratigraphic log of Bakchar deposit showing occurrence of glauconitic rocks (b), and photos of glauconitolite (c) and glauconitic sandstone (d).

Mazumder et al., 1993; Rao et al., 1993; Rao and Rao, 1999; Yadav et al., 2000). The present investigation also involves the conventional roasting method rather than reduction roasting adopted by Shekhar et al. (2017a), Shekhar et al., 2017b). On the basis of laboratory experiments we evaluate the roles of various parameters influencing the recovery of K that include roasting temperature, concentration of hydrochloric acid (HCl), leaching temperature, leaching time, stages in leaching process, and the ratio by mass of sample to acid.

2. Samples and methods

2.1. Rock sampling and preparation

The Bakchar oolitic ironstone deposit occurs approximately 200 km west of the Tomsk city, Russia (Fig. 1a). The site is a part of the West Siberian iron ore basin at the eastern and southeastern edges of the West Siberian plain. Glauconitic rocks occur at a depth of 180–230 m (Fig. 1b). The deposit comprises a thick sequence of Turonian to Eocene shallow marine sedimentary rocks formed in the ancient epicontinental sea of Western Siberia (Belous et al., 1964; Rudmin and Mazurov, 2016; Rudmin et al., 2018). The ore-bearing sedimentary sequence consists of five major transgressive-regressive cycles (Rudmin et al., 2017a). Transgressive system tracts during the Turonian, Coniacian-Santonian, Campanian, Lower Maastrichtian and Eocene are characterized by siltstones and clays with organic matter, while highstand system tracts are dominantly made of sandstones. Authigenic glauconite content increases from the bottom to the top in the Campanian and the Upper Maastrichtian (Rudmin et al., 2017a). The early diagenesis of marine deposits forms a mineralogical assemblage of glauconite pellets, goethite-chamosite ooids, siderite, illite, and pyrite framboids (Belous et al., 1964; Rudmin and Mazurov, 2016; Rudmin et al., 2017a).

The drill hole 627 provided representative samples of glauconite rocks (RSGR) (Fig. 1b). The sample preparation followed the procedures of Rudmin et al. (2017b). Approximately 20 kg of core samples were crushed and homogenized in order to prepare the representative samples. The samples were then pulverized to < 2 mm with a jaw crusher and a roll crusher for analytical purposes. Standard stainless steel sieves (with mesh openings of 500 and 80 μm) were used to separate lightly crushed samples into three granulometric fractions: coarser than 500 μm, between 500 and 80 μm, and finer than 80 μm. The intermediate fraction was passed through electromagnetic separator ECM 10/5 (JSC “Mining Machines”, Russia). The magnetic roll retained glauconitic grains largely from the feed stream leaving a “nonmagnetic concentrate” at current 3.5A. The magnetic concentrate was reprocessed at the same current 3.5A for further concentration of the glauconite fraction.

2.2. Physical, chemical, and mineralogical characterization

The mineralogical characterization of the studied samples was carried out using a combination of optical microscopy, X-ray diffraction (XRD), and scanning electron microcopy (SEM) coupled with an energy-dispersive spectrometer (SEM-EDS). Bulk mineralogical composition of samples was determined by using a Rigaku Ultima IV X-ray diffractometer at the Tomsk Oil and Gas Research and Design Institute, with Cu-Kα radiation at a current of 30 mA and a voltage of 40 kV. < 10 μm size-fractions of powdered samples were scanned from 3°–70° 2θ, with a step of 0.02° at a scanning rate of 1 s, divergence slit (DS) being 1.2 mm. Each sample was scanned initially after air drying (i.e., untreated), and subsequently by ethylene-glycol solvation by vaporization in a desiccator for 24 h. Comprehensive identification of clay minerals was carried out following procedures provided in Moore and Reynolds Jr. (1997) and Hillier (2003). Quantitative mineralogical analyses of the whole rock data were performed by a Rietveld analysis (Bish and Post, 1993) using PDXL and Siroquant software (Taylor, 1991). Polished glauconitic grains were produced using diamond paste after mounting them in an epoxy base (film balsam Russian GOST 2290-76). The glauconitic grains were studied under scanning electron microscope (SEM) TESCAN VEGA 3 SBU and energy-dispersive adapter OXFORD X-Max 50 with 20 kV accelerating voltage, specimen current of 12 nA, and spot diameter of approximately 2 μm.

Major element concentrations of the powdered samples were estimated by X-ray fluorescence microscope (XRF) HORIBA XGT 7200 operated at a tube current of 1 mA, beam diameter of 1.2 mm, and a voltage of 50 kV. Both pressed and fused pellets were prepared for the purpose of analysis by XRF. Detection limits for major elemental concentration were 0.01 mass%. SEM and XRF investigations of glauconite samples were carried out at the Department of Geology in Tomsk