

Understanding the performance of a pilot vermiculite exfoliation system through process mineralogy



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ABSTRACT

This paper reports on the application of process mineralogy to understand the experimental performance of a microwave based system for the exfoliation of vermiculite type minerals. When montmorillonite type clay structures are exposed to high intensity electric fields they expand very rapidly with the degree of expansion being directly related to the applied electric field strength. This basic understanding of the interaction of microwave fields with such materials has been used to develop a pilot scale microwave based process for the exfoliation of vermiculite. During commissioning of the system it was noted that vermiculites from different geographical sources behaved very differently in terms of the achieved expansion ratio. A systematic mineralogical evaluation of these different materials was carried out using Mineral Liberation Analysis (SEM/MLA), Thermo-gravimetric analysis and X-ray Diffraction (XRD) in order to determine the reasons for the variation in performance. Samples from vermiculite mines in Australia, Brazil, China and South Africa were studied. The mineralogy and mineral characteristics such as liberation, size and texture of the samples were quantified for each ore sample. The South African and Australian samples were found to be predominantly hydrated forms of mica. The Brazilian sample was predominantly composed of vermiculite while the Chinese sample was composed almost totally of hydrobiotite. The relationships between the form of hydration and degree of expansion were derived and it is shown that pure vermiculite exfoliates at lower field intensities than the other forms of hydrated mineral. The paper concludes with deductions obtained from the mineralogical assessments of the vermiculites, thereby elucidating the reasons for the performance observed for each feed material in the microwave exfoliation system.

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1. Introduction

Vermiculite typically refers to a group of 2:1 phyllosilicate minerals including the mineral vermiculite composed of hydrated magnesium–aluminium–iron sheet silicates (mica), which contain water molecules within their layered structure (Grim, 1968; Mitchell and Soga, 2005; Walker, 1961). The vermiculite structure comprises 2 tetrahedral silica sheets sandwiching a central octahedral sheet. The interlayer between each 2:1 sheet unit contains water and exchangeable cations. The distance between the silicate sheet layers and the exchangeable cations is referred to as the basal spacing (Mitchell and Soga, 2005). Fig. 1 shows an illustration of the silicate structure.

When heated rapidly to between 870 and 1100 °C, the interlayer water is turned to steam and the pressure generated within the structure disrupts the vermiculite silicate layer by a process

known as exfoliation (Marcos and Rodríguez, 2010; Obut and Girgin, 2002). Resulting from this process, its volume expands to several times that of the unprocessed material (Hillier et al., 2013; Justo et al., 1989). Exfoliated vermiculite is lightweight with thermo-insulating properties. It is also highly porous, insoluble in water, non-toxic and has good absorption properties (Mysore et al., 2005; Suquet et al., 1991). These physical properties enable its use across a wide range of applications (Bergaya et al., 2006; Mouzadahir et al., 2009; Muiambo et al., 2010).

Water exists in clay minerals such as hydrated halloysite, montmorillonite and vermiculite as adsorbed, interlayer and lattice hydroxyl water, and there is a considerable variation in the total water content of different clay minerals (Bergaya et al., 2006; Grim, 1968; Velde, 1992). The adsorbed water in vermiculite is not chemically bound to the vermiculite structure or interlayer cations, and this is released from either the surface or the interlayer space as free water below 115 °C. The high temperature hydroxyl water, also known as crystalline water is strongly bound to the vermiculite structure. The release of this water type from the

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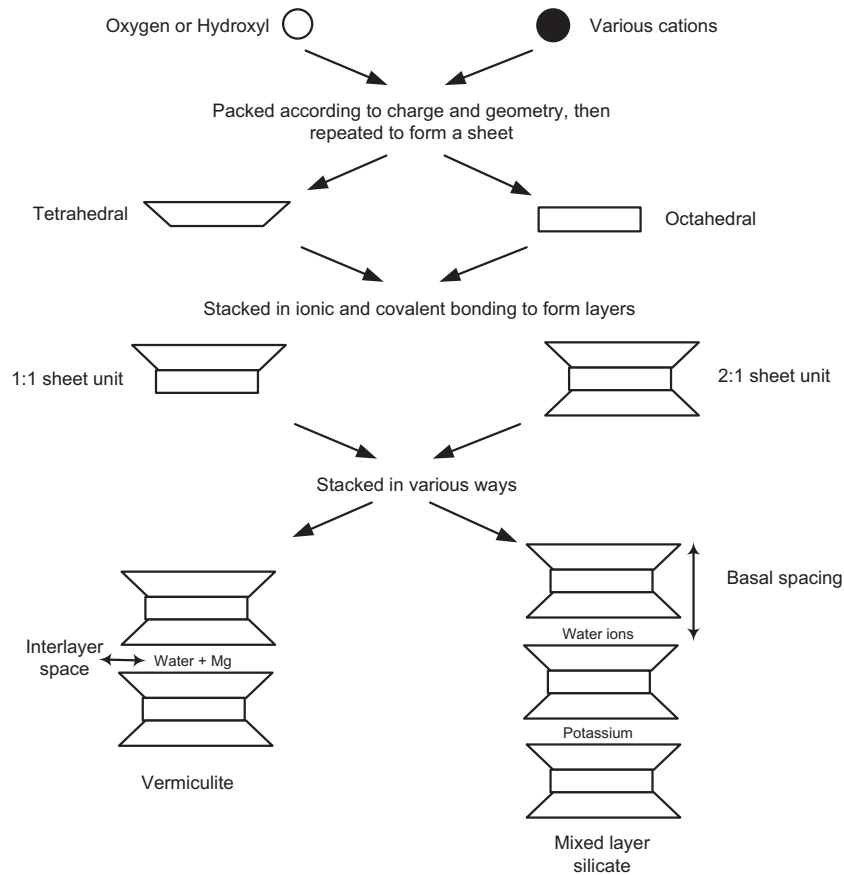


Fig. 1. Illustration of the silicate structure for vermiculites (Adapted from Mitchell and Soga (2005)).

structure starts at 500 °C and is completely removed at temperature higher than 800 °C. The removal of the hydroxyl water causes permanent decomposition of vermiculite structure (Gillott, 1987; Grim, 1968; Marcos et al., 2009; Mouzdahir et al., 2009; Velde, 1992).

Conventional heating methods using fossil fuelled furnaces have been used industrially for vermiculite exfoliation. It has been reported by many researchers that mixed layer (mica-vermiculite) minerals, such as hydrobiotite produce better exfoliation than the pure vermiculite when subjected to conventional heating (Justo et al., 1989; Marcos et al., 2009; Marcos and Rodríguez, 2014; Midgley and Midgley, 1960). Similar observations were recorded for exfoliation using microwave heating (Marcos and Rodríguez, 2010, 2014; Obut et al., 2003). However, microwave exfoliation has been found to be a more robust means of exfoliation in terms of energy savings, reduced processing time and low exfoliation temperature (Folorunso et al., 2012; Marcos and Rodríguez, 2010, 2011; Obut et al., 2003). Apart from the work of Folorunso et al. (2012) that uses a continuous high throughput and high power (>5 kW) microwave system operating at 2.45 GHz, most of the research on microwave exfoliation of vermiculites are limited to laboratory investigations, using domestic microwave ovens which are typically between 0.5 and 1.3 kW (Marcos and Rodríguez, 2011; Obut et al., 2003). Inherent limitations such as, low power density, spatial non-uniform distribution of electric field and creation of multiple hotspots within the cavity, impedes the use of domestic microwave ovens for scientific investigations of microwave exfoliation of vermiculites.

Important parameters in microwave heating of materials are the dielectric properties which provide information about the

energy coupling efficiency of electromagnetic waves to the materials. Dielectric properties also control the applicability of microwave energy for processing a wide variety of non-conductive materials (Meredith, 1998; Schubert and Regier, 2005). The dielectric constant (ϵ') is a quantitative factor which describes the ability of a material to be polarised and store electromagnetic energy while the loss factor (ϵ'') represents the efficiency with which the stored energy is converted to heat (Meredith, 1998; Metaxas and Meredith, 1983). Kaviratna et al. (1996) reported that total water content has a considerable effect on dielectric properties of clay minerals such as montmorillonite and laponite. This is due to the high dielectric constant and loss factor of about 77 and 13 respectively possessed by water at 2.45 GHz (Metaxas and Meredith, 1983). Another reason is that water molecules within the mineral structure easily polarise when placed in alternating electric field, especially free water held to the mineral by van der Waals forces, (Meredith, 1998; Metaxas and Meredith, 1983). However, some silicate minerals such as feldspars, muscovite and quartz are transparent to microwave energy because of their low dielectric properties as a result of the absence of either adsorbed or bound water or any conductive species (Chen et al., 1984). Therefore, the effect of the change in the mineral water network on dielectric properties when heated to a high temperature is significant in the context of microwave heating.

Initial test work on the continuous microwave exfoliation system, suggest different exfoliation outcomes due to mineralogy for the various samples at the same operating conditions. The aim of this work was to understand the mechanisms behind the different exfoliation results obtained. To achieve this aim the following objectives were set:

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