



Research paper

Evaluation of polymeric Al-modified bentonite for its potential application as ceramic coating



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ABSTRACT

A polymeric Al-modified bentonite (B-OHAL) obtained by intercalation of OH-Al species was used as precursor for mullite-containing coatings. Raw clay and B-OHAL were characterized by X-ray fluorescence (XRF), Infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric and differential thermal analysis (TG-DTA). Rheological and electrokinetic behavior of B-OHAL dispersions were optimized by the addition of sodium polyacrylate (NaPA) for its use in dip coating on ceramic substrates. Moderate densification and mullite development of B-OHAL by thermal treatment up to 1200 °C was observed by mercury intrusion porosimetry (MIP), XRD and scanning electron microscopy SEM. NaPA addition on dispersions and an increase in porosity values in substrate led to higher amounts of coating retained in dip coated composites sintered at 1200 °C.

1. Introduction

Ceramic coatings are commonly used in industry, as they can develop interesting properties, such as good corrosion and wear resistance, hardness, high temperature-strength, and thermal stability. These materials have a wide array of applications, as hard coatings for cutting tools (Bobzin, 2016), nuclear fuel cladding (Alat et al., 2016), antibacterial coating (Lee et al., 2016) and as thermal barriers (Bernard et al., 2016), among others.

Mechanical properties of ceramics are linked to the presence of certain crystalline phases. Being the only stable intermediate phase in the Al₂O₃-SiO₂ system at atmospheric pressure, mullite (3Al₂O₃·2SiO₂ theoretical composition, with 72 mass% alumina and 28 mass% silica) is one of the most important ceramic materials, due to its refractory nature, good chemical stability, high-temperature strength, low thermal conductivity and expansion coefficient (Duval et al., 2008). Mullite is usually synthesized, as it is rarely found in nature. Anggono (2005) reported that there are mainly three synthesis methods for mullite. The more classical method involves the mixture of clays (kaolin) and other aluminum containing materials, which, upon sintering, form mullite. Another method involves the melting of the raw materials (for example, alumina and sand) in furnace at temperatures above 2000 °C. The third method is the so-called “chemical-mullite” synthesis, which involves the mixing of Al³⁺ and Si⁴⁺ on scales ranging from the atomic to the micron level. This method has the advantage of being able to reduce the sintering temperature to lower values than with mixed solids.

The interlayering of smectites with inorganic polycationic species has been widely studied, as these materials form Pillared Interlayer Clays (PILC) when thermally treated at 350 °C–750 °C (Brindley and Sempels, 1977; Vaughan and Lussier, 1980; Vicente et al., 2001; Aouad et al., 2005). PILC have seen widespread application in various fields such as the production of biofuels (Kloprogge et al., 2005), catalysis (Kanda et al., 2009), adsorption of heavy metals (Bhattacharyya and Gupta, 2008) among others; due to their high surface area and thermal stability (Kloprogge, 1998). Also, the intercalating polycation [Al₁₃O₄(OH)₂₄(H₂O)₁₂]⁷⁺, usually known as Al₁₃ cation, is simple to synthesize reproducibly (Wang et al., 2016). One interesting feature of these Al-interlayered clays, which has been scarcely studied, is that they can develop mullite when thermally treated. Martínez et al. (2017) used two montmorillonitic clays, namely a Wyoming and a Cheto one (both samples from USA) to obtain Al-interlayered clays, and found mullite presence after thermal treatment above 1000 °C. Jagota et al. (1995) studied an Al-interlayered smectite and found evidence of mullite formation at 800 °C, which is a relatively low temperature.

At temperatures above 800 °C, the Al-interlayered clays lose their characteristic layered structure as their interlayer space collapses (Kloprogge et al., 1994; Vicente et al., 2001; Volzone and Garrido, 2012). At higher temperatures, they develop stable crystalline phases (Jagota et al., 1995; Martínez et al., 2017). Due to their natural origin, the formation of crystalline and liquid phases at high temperature in clays is conditioned by several factors, such as the chemical and mineralogical composition of the clay mineral, the presence of structural

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defects and impurities, etc. (McConville and Lee, 2005; Martinez et al., 2017). Consequently, the microstructure and properties of the fired Al-intercalated clay mineral greatly differs from those of the untreated (fired) one.

There are many ways of producing mullite-containing coating on substrates. Plasma spraying (Schrijnemakers et al., 2009), sol-gel (Zhongliu et al., 2015), hot dipping (Zhang et al., 2016), electrophoretic deposition (Wang et al., 2013), and dip coating (Ramasamy et al., 2011) among others. Dip coating is a simple, inexpensive method of depositing uniform thin layers of particles in dispersion onto a substrate which, upon evaporation of the liquid, forms a film of coating. The film thickness is set by the competition among viscous force, capillary force and gravity (Scriven, 1988). Thus, a basic requirement for the application of this method is the preparation of stable and fluid dispersions. To achieve these characteristics, the understanding of the dispersion of the particles in a liquid medium is of paramount importance. Viscosity of aqueous dispersions can be modified by the addition of different chemical agents.

Although rheological properties of bentonite dispersions and the influence of the addition of dispersants were extensively investigated (Lagaly, 2006; Liang et al., 2010; Santagata et al., 2014), there are limited studies available on colloidal, electrokinetic and rheological properties of Al-intercalated smectite dispersions (Harsh et al., 1988; Avena et al., 1990). Jagota et al. (1995) used Al-intercalated smectite dispersions to coat SiC fibers which, upon firing, were included in a borosilicate glass matrix, improving its mechanical properties; though no rheological properties of the dispersions were measured. The measurement and control of these properties are essential for the preparation of stable dispersions in order to produce dip-coated composites.

The objective of this work was to study the properties of a polymeric Al-modified bentonite (B-OHAL) in order to evaluate its possible application as a precursor for mullite-containing coatings on ceramic substrates. Crystalline phases and textural properties developed in B-OHAL through thermal treatment at high temperatures (1000–1200 °C) were determined by powder X-ray diffraction (XRD), infrared spectroscopy (FTIR) and mercury intrusion porosimetry (MIP), respectively. Stability of B-OHAL aqueous dispersions for dip coating and its modification by sodium polyacrylate addition was also examined by electrophoretic mobility and viscosity measurements. Coated composites were obtained by dip coating a porous substrate in stable and unstable B-OHAL dispersions. Mullite formation and microstructure of coated surfaces was observed by XRD and scanning electron microscopy (SEM) respectively.

2. Experimental

2.1. Materials and methods

2.1.1. B-OHAL preparation

A bentonite from Argentina (B), with 15.6 mass% Al₂O₃ content was used to prepare the coating precursor. This bentonite has been characterized in a previous work (Vidal and Volzone, 2012) and is composed mainly by montmorillonite with traces of quartz and feldspar. The calculated structural formulae of the montmorillonite is: [(Si_{3.94}Al_{0.06})₄(Al_{1.36}Fe_{0.06}Mg_{0.60})O₁₀(OH)₂].

Swelling index was obtained by adding 2.0 g of bentonite in a 100 ml measuring cylinder with water. After 24 h the height of sediment in ml was read. The swelling index of B was 4 ml. This value is low compared with an expansive bentonite, which reaches value > 12 ml.

The polymeric Al-modified bentonite (B-OHAL) preparation method has been previously reported (Volzone and Garrido, 2008), therefore, only a brief summary is described in this work. A polymeric hydroxyl-Al solution (0.10 M in Al) was prepared by diluting the commercial chlorohydroxy-Al solution (6.0 M) that was aged during 7 days at room temperature. The raw clay was dispersed in water (2 mass%) and subsequently the polymeric solution OH-Al was added. The aluminum/

raw clay ratio was 3.25 mmol/g. After 24 h, the solid was separated by centrifugation and washed several times with distilled water in order to remove excess electrolyte.

In order to study of the influence of the heating temperature on the textural properties of B-OHAL, cylindrical casted samples were made by consolidation of the B-OHAL dispersion without additives. Slip casting of this dispersion was carried out in a plaster mould, obtaining samples of about 10 mm in diameter and 8–10 mm of thickness. The resultant cast cylinders were dried at room temperature for 24 h.

Both powdered samples and B-OHAL compacts were thermally treated in an electric furnace at a heating rate of 5 °C min⁻¹ up to 1000, 1100, 1200 °C for 1 h.

2.1.2. Preparation of dip-coated composites

In order to obtain substrates with different porosity for dip coating, disk-shaped ceramics (diameter = 20 mm, thickness = 5 mm) were prepared using a commercial clay by uniaxial compression of the powder at 30 MPa and subsequent thermal treatment at 800 °C (S800) and 1100 °C (S1100) in an electric furnace. The commercial clay is mainly composed by quartz, kaolinite, illite and feldspar. Substrate showed an open porosity of 32.1% and a mean pore diameter of 65.3 nm when thermally treated at 800 °C for 1 h and an open porosity of 18.1% and a mean pore diameter of 330 nm after firing at 1100 °C for 1 h.

Aqueous dispersions of B-OHAL with and without the addition of sodium polyacrylate (NaPA, Dolapix PC 67, Zschimmer & Schwarz) were used as coating precursor. They were obtained by dispersing 6 g of B-OHAL in 100 ml of distilled water containing the dispersant up to desired concentration and stirred for 1 h at room temperature.

Preliminary dip-coating tests were made in order to evaluate the influence of dip coating time (5, 10, 20 and 40 s) on the amount of coating retained. It was observed that the latter increased with coating time up to 10 s, while for longer times it remained relatively constant but favored cracking of the coating surface. Thus, dip-coated composites were obtained by dipping the calcined substrate disks in these dispersions for 10 s, and left to dry at room temperature until constant mass. This process was repeated 3 times. The dip-coated substrates were dried overnight at room temperature and then 1 h at 60 °C in an electric oven prior to thermal treatment, which was carried out in an electric furnace at a heating rate of 5 °C min⁻¹ up to 1200 °C for 1 h. The adherence of the fired coating was evaluated by pushing a scotch tape onto the coated surface and then pulling fast. This test made it possible to establish that the coating adhered firmly to the substrate after doing so.

2.2. Characterization techniques

Chemical analysis of samples was done by X-ray fluorescence (XRF, Shimadzu EDX800-HD). The identification of crystalline phases and structural characterization of raw clay, B-OHAL and the thermally treated samples was carried out by X-ray diffraction (XRD), using a diffractometer Philips with goniometer 3020 and PW3710 controller with radiation of Cu-K α and Ni filter in the range of 3–70°(2 θ). FTIR analysis of B and B-OHAL samples were performed by using a Spectrum One Perkin Elmer equipment, in the wavelength range of 400–4000 cm⁻¹. The samples were dispersed in KBr (1 mass%) and compacted in a thin pellet form.

Thermogravimetric and differential thermal analysis (TG-DTA) of samples up to 1200 °C was performed with a Netzsch 409 equipment, using α -Al₂O₃ as reference at a heating rate of 10 °C min⁻¹.

Electrophoretic mobility measurements at different pH were made. Powdered sample (< 144 μ m) was dispersed in a 500 ml KCl solution (10⁻³ M) to 0.1 mass% solid concentration. The pH of the dispersions was adjusted by addition of 0.01 M NaOH or 0.01 M HCl. Electrophoretic mobility measurements were carried out using a ZETA PLUS zeta potential analyser (Brookhaven Instruments, USA).

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