

Rheological study of yttrium oxide aqueous suspensions

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Abstract: Yttria (Y_2O_3) has been used in many technological applications areas as luminescence material, high temperature and strength structural material, owing to its excellent optical and refractory characteristics. Applying conformation techniques using Y_2O_3 concentrated suspensions is adequate, if it is well controlled, so that assisting to obtain homogeneous ceramic bodies, reproductive and with complex geometry. Studies involving superficial behavior, stability conditions of suspensions and the behavior related to conformation give important information to control the processes in manufacturing ceramic components. In this work is presented a rheological study of Y_2O_3 aqueous suspensions, which concerns to solids and dispersant concentration and pH of media. Preparation of Y_2O_3 aqueous suspensions with solids concentration of 30vol% was possible, using 1wt% of ammonium polyacrylate (PAA) that was enough to gain the lowest viscosity of the suspensions.

Introduction

Colloidal processing is a powder consolidation method in order to produce ceramics using near net shape conformation process (tape casting^[1], gel casting^[2], impregnation method^[3]). This process results in a more uniformed particle packing, better microestructural control while firing and high mechanical strength of ceramic^[4].

Yttrium oxide is one of the most important rare earth oxides, being applied in a variety of technologies areas as: luminescence materials^[5] and biomaterials^[6]. Some of its main proprieties are: high melting point (2400°C), high refraction index ($\cong 1.9$), low expansion coefficient and high corrosion resistance^[7,8].

In this work is presented a rheological study of Y_2O_3 aqueous suspensions, where is analyzed the effect of some variables on rheological behavior such as: solids concentration, dispersant concentration and pH of the media.

Experimental

Powder characterization

The following commercial powder was used as starting material: Y_2O_3 (Aldrich, Gmb), with a mean particle size of 1.31 μm , a specific surface area of 13.59 $m^2 \cdot g^{-1}$, a density of 5.83 $g \cdot cm^{-3}$ and a purity higher than 90.0%. The details of the physic-chemical characterization of the powders were reported in the our previous work^[9].

The stability of powders in aqueous media was based on measuring the electrophoretic mobility of the particles in a pH range from 2 to 12, using a light phase scatter analyzer (ZetaPALS,

Brookhaven Instruments Corporation, USA). The samples were prepared to a solid content of 0.5 g.L⁻¹ in distilled water. The ionic strength was fixed at 10⁻² M using NaCl (analytical degree, Casa Americana, Brazil) as indifferent electrolyte. Prior to measurements, all suspensions were homogenized with an ultrasounds probe (dr. Hielscher 400US, Germany) for 2 min. The pH adjustments were made by adding appropriate amounts of HCl and KOH. The polyelectrolyte Duramax D3005 (ammonium salt of a polyacrylic acid - PAA, Rohm and Haas Co., PA, USA) was used as deflocculant.

Suspensions preparation

Yttrium oxide suspensions (Y₂O₃) were prepared in distilled water with solids concentration from 15 vol.% on (≅ 47 wt.%). In order to stabilize the suspensions an ammonium salt of a polyacrylic acid (PAA) with concentrations ranging from 0 wt.% to 2 wt.% (referred to dry solids) was used. Basicity of the medium was provided by adding tetramethylammonium hydroxide (TMAH), supplied by Aldrich-Chemie (Germany). The increase in solids concentration in suspensions till 30 vol.% was also studied. All PAA additions were based on dry powders of Y₂O₃.

The rheological behavior of suspensions was performed with a RS600 rheometer, (Thermo Scientific, Germany). The sensor system consisted on a double cone rotor and a stationary plate (DC60/1°). Characterizing the suspensions stability the flow curves were determined in a control rate mode (CR). Measurements were performed by increasing the shear rate from 0 to 1000 s⁻¹ in 5 min., maintaining at 1000 s⁻¹ for 2 min and returning to 0 in 5 min. Temperature was maintained constant at 25°C during these experiments. For each CR cycle 200 points were measured. In order to decrease the agglomerates size, the suspensions were stirred for 3 min at high shear rate (Quimis, Q-252-K18). After that, they were poured at the mechanical mixer (Heidolph, mod. RZR1) for 30 min to promote suspensions homogeneity.

Results and discussion

Powders characterization

Powders morphologies of Y₂O₃ as received and after milling during 3 hours were observed by SEM (Fig. 1), where can be verified that before milling the Y₂O₃ powders consisted of the aggregates platelet particles (Fig.1a). In Fig.1b the milling effectiveness (around from 5 μm to 2 μm) on reduction of the particles aggregates size is observed.

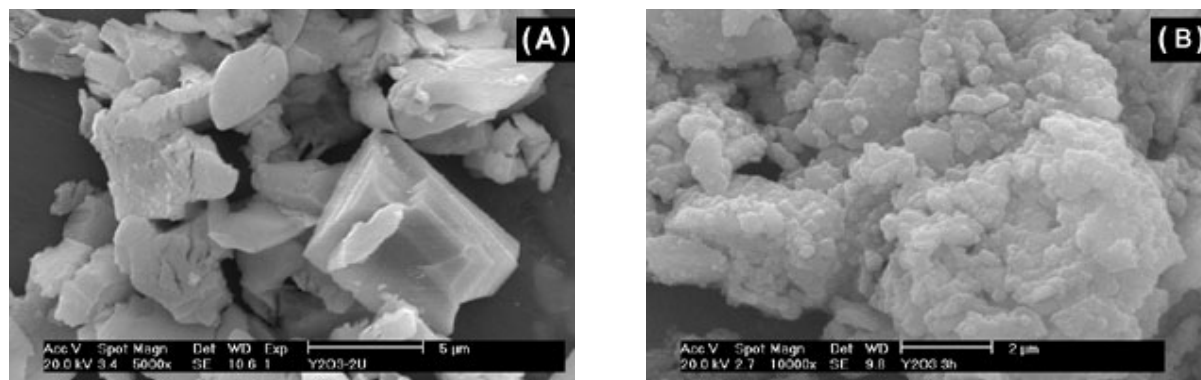


Fig. 1 - Y₂O₃ powders micrographs: (A) as received and (B) after 3h milling

Stability of Y_2O_3 suspensions - Zeta potential determination

The Zeta potential variation and the dispersant effect (PAA) on Y_2O_3 particles surface are indicated in Fig. 2. It's observed that dispersant shifts the IEP of Y_2O_3 from pH 8.5 to pH 6.5. This change is due to the adsorption of dispersant molecules on particles surfaces. The highest Zeta potential value (the highest degree of stability) takes place at pH 10 (\cong 56 mV). Adding 1 wt.% of dispersant was enough to form stable suspensions from pH 8.0 on.

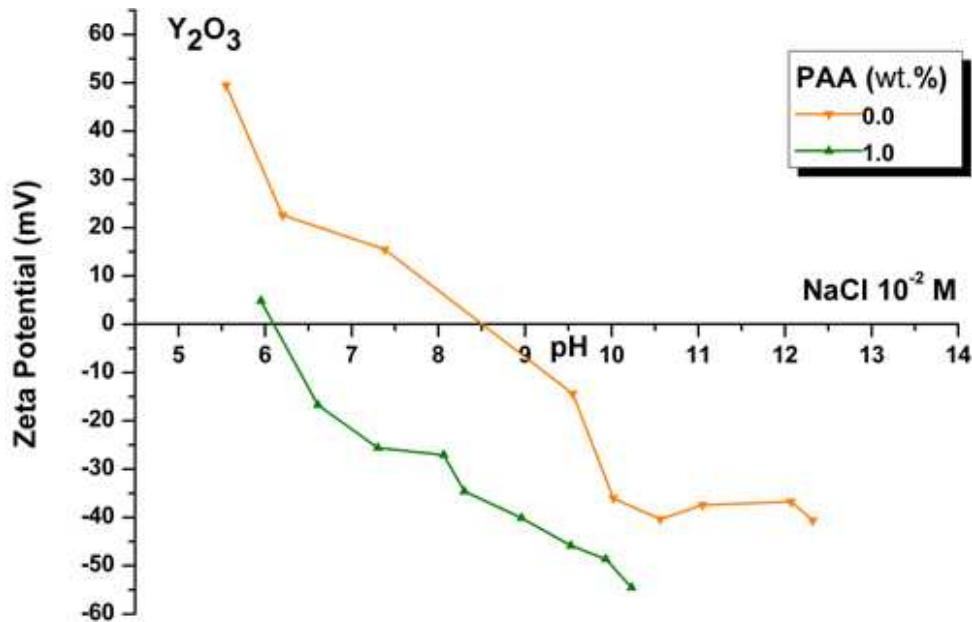


Fig. 2 - Zeta potential variation as function of pH and PAA concentration.

Effectiveness of dispersant concentration and pH on suspensions stability

The effect of dispersant concentration on rheology behavior of Y_2O_3 suspensions was verified varying PAA dosage from 0.5 wt.% to 2 wt.%. Based on the result presented in Fig.3, it's observed that suspension presented the lowest viscosity value with addition of 1 wt.% of PAA. So, from this concentration on is not observed betterment on flow behavior. In agreement with Zeta potential curves (Fig.2), the suspension with pH close to IEP of Y_2O_3 (pH 8.5) showed the highest viscosity value (Fig.4). At this pH, the particles present low surface charge, near to neutrality. Otherwise, at pH 10, the particles get the highest repulsion potential (high negative surface charge), as DLVO^[4] theory predicts, supporting dispersion (via electrostatic repulsion) of the particulate system.

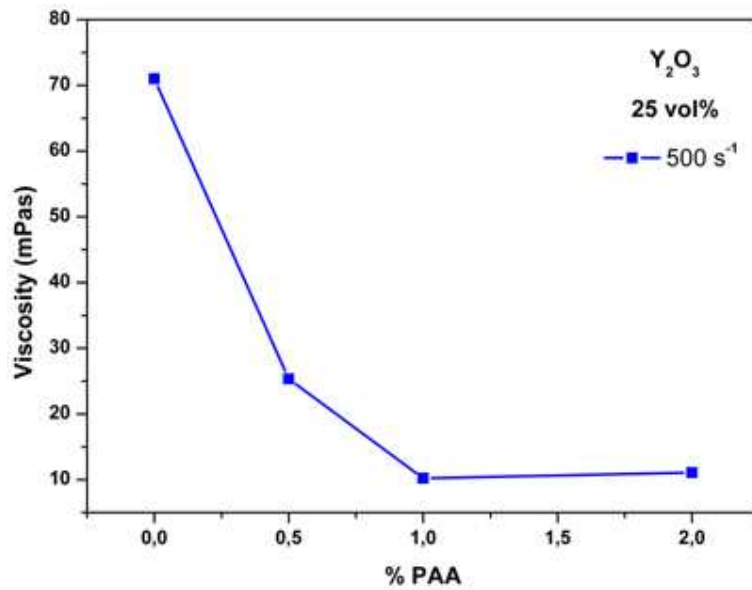


Fig. 3– Viscosity as function of dispersant concentration (PAA).

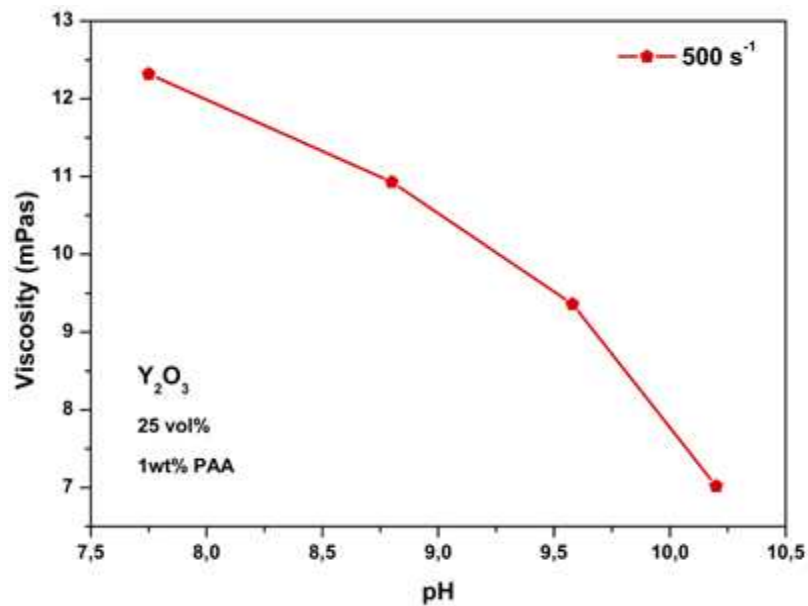


Fig. 4– Y_2O_3 suspension viscosity as function of pH

Effect of solids concentration on rheological behavior

The effect of solids concentration on rheological behavior of yttria aqueous suspensions is presented in Fig. 5 that shows the shear stress (Pa) as function of the shear rate from 0 to 1000s⁻¹. A great increase on flow resistance (viscosity) of suspensions from 15 vol.% to 30 vol.% was observed, due to higher particle concentration and shorter room between particles, which intensifies the interaction degree of particles^[10]. The rheological behavior presented by the flow curve of 30 vol.% exhibits higher flow resistance (viscosity) compared to the others flow curves.

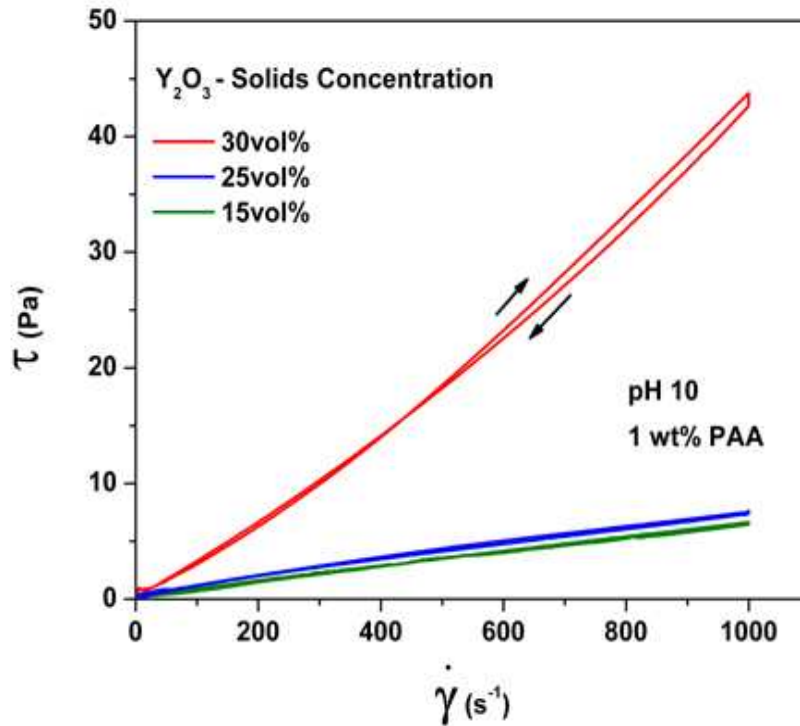


Fig. 5 – Flow curves as function of Y₂O₃ solids concentration

Conclusions

The highest Zeta potential value (the highest degree of stability) of Y₂O₃ suspensions takes place at pH 10 and with 1 wt.% of dispersant the stability is achieved from pH 8 on. The highest solid concentration attained in this study was 30 vol.% with a 1wt.% of dispersant at pH 10 (adjusted with TMAH).

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