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Zircon based ceramics by colloidal processing

Liliana B. Garrido *, Esteban F. Aglietti

CETMIC Centro de Tecnología de Recursos Minerales y Cerámica (CIC-CONICET-UNLP), Cno. Centenario y 506,
C.C.49 (1897) M.B. Gonnet, Prov. de Buenos Aires, Argentina

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Abstract

Zircon is an excellent raw material to elaborate ceramics with good thermo-chemical properties. Also, mullite–zirconia materials can be prepared by reaction sintering of zircon–alumina mixtures. Concentrated aqueous alumina–zircon suspensions were prepared to study the effect of the slip properties on the density and microstructure of the compacts obtained by slip casting and pressure filtration at 8 MPa. The effect of the degree of dispersion and solid volume concentration on the viscosity of the suspension was studied. Optimization of the amount of polyacrylate dispersant added and pH of the suspension improved the solid loading and consequently the green and sintered properties of the cast samples.

Keywords: A. Slip casting; Zircon; Pressure filtration; Rheological properties

1. Introduction

Zircon based ceramics are used for many applications due to their optimal mechanical, high resistance to thermal shock and good chemical properties. Mullite–zirconia ceramics can be obtained from zircon raw material and alumina. According to a chemical reaction in a solid state between zircon and alumina, mixing the suitable (stoichiometric) amounts of reactives will produce a mullite and zirconia ceramic. The preparation of mullite–zirconia composites using the reaction sintering method has attracted the attention of the scientists since good-quality zircon is ready available from beach sand and at a cheaper price thus making the development cost effective [1]. The mechanism of mullite–zirconia formation was described earlier in detail [2,3].

This material finds applications as refractories, kiln furniture, grinding media. Kiln linings are made for temperatures above of 1800°C due to zirconia only being slightly wetted by siliceous and metallic melts. Since the solubility potential of zirconia in a silicon oxide is very low the chemical attack on the refractory material is comparatively low [4].

To achieve improvements in the density and microstructure of the green consolidate, the colloidal stability and rheological behaviour of the suspension must be controlled. Control or manipulation of the interparticle colloidal forces by adjusting the pH or by adding dispersants is generally used to improved the properties of the suspensions that, in turn, improve the microstructure and the resulting properties of the green and sintered material. Well dispersed suspensions can be packed at high relative density without large pores and agglomerates.

The poor dispersion of the powders produce difficulties in the densification and the presence of agglomerates is responsible of a poor mechanical properties. In addition some negative effects such as low filtration rates and some mass segregation of the constituents can appear during the consolidation step of a ceramic multicomponent. To minimize these problems, solid concentration is usually increased or a flocculated or coagulated suspension is used [5]. Thus, optimization of the slip properties is required for suitable control of the casting process.

Previous studies on the casting behaviour of zircon and zircon–alumina commercial powders proposed the use of organic surfactant/polyelectrolytes and found that extremely high dispersant concentration was deleterious for the final properties of the cast [6,7].

In this work, the effect of various slip casting and pressure filtration parameters such as dispersant content, pH and solid volume concentration on the viscosity of alumina–zircon suspensions was studied. Adsorption isotherm of polyacrylate on the mixed powder was

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* Corresponding author.
E-mail address: garridol@netverk.com.ar (L.B. Garrido).

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determined. Slip properties were evaluated by rheological measurements and by zeta potential and correlated with the characteristics of the dried and sintered (1600°C, 2 h) compacts.

2. Materials and methods

Alumina powder used is a fine and pure α-Al_2O_3 (A-16 SG, Alcoa Inc., USA) and contains Na_2O and MgO as main impurities [8,9]. The mean particle size determined was 0.4 μm and the specific surface area (BET) 9.5 m^2/g [8]. The isoelectric point has been determined to be 9 [10].

According to the chemical analysis, the zircon powder used (Mahlwerke Kreutz, Mikron, Germany) contains minor amounts of (wt.%) Al_2O_3, 0.90; TiO_2, 0.11; Fe_2O_3, 0.05; P_2O_5, 0.10 and HfO_2, 1.30. The mean particle size determined was 2 μm and the specific surface area (BET) 4.1 m^2/g. The isoelectric point ($pH_{iep} = 5$) was determined through the electroacoustic technique (ESA 8000, Matec Instruments) on a 1 vol.% suspension.

2.1. Suspension preparation and characterization

Alumina–zircon suspensions of stoichiometric (theorical) composition were prepared. The alumina/zircon (wt.%) ratios of the mixtures was: 45.5/54.5. This ratio is which after a theoretical complete reaction at high temperature a mullite–zirconia composite is obtained. Aqueous 41–60 vol.% suspensions were prepared by adding the powder to aqueous solutions with different dispersant content (Dolapix CE64, Zschimmers & Schwartz). The suspensions were treated in an ultrasonic equipment during 30 min for dispersion.

Flow curves of the suspensions were obtained using a rotational viscometer Haake vt550 of coaxial cylinders with NV measure system at 25°C.

2.2. Adsorption isotherm of polyacrylate

The adsorbed amount of polyacrylate on the mixed powder was determined by thermogravimetry. The 48–57 vol.% suspensions were centrifuged, the solid was washed in water and centrifuged. Then, the solid was dried at 60°C. The weight loss between 200–600°C due to polyacrylate decomposition was measured using a Netzsch STA 409 thermobalance and used to calculate the amount of polymer adsorbed.

2.3. Slip casting, pressure filtration and characterization of the compact

The consolidation of suspensions by slip casting was carried out in a plaster mould to produce prismatic bars of 12×12×60 mm^3.

Pressure filtration was done on a metallic sintered porous filter with a wet filter paper placed on it, uniaxial pressure was employed. The pressure of 8 MPa was applied from the top and the liquid moved through the porous filter. Pressure was maintained constant for 30 min. Cylindrical cast samples of 25 mm diameter and 12–15 mm thick were obtained.

Bars and cylindrical compacts were dried 24 h at room temperature and then at 110°C up to a constant weight. Relative density (% of the theoretical) was measured by Hg immersion.

Sintering was carried out in an electric furnace under air atmosphere, density and open porosity were determined by the water absorption method.

Crystalline phases of the fired bodies were characterized by XRD using a Phillips 3020 goniometer with PW3710 controller, Cu–K_α radiation and Ni filter at 40 kV–20 mA. Distribution of Zr and Al were obtained with a Phillips 505-Scanning electron microscope with EDAX accessory.

3. Results and discussion

3.1. Powder characterization

The isoelectric point (iep) of alumina used has been determined to be near 9 [10]. The zeta potential vs pH curve (Fig. 1, curve a) showed that the magnitude of the zeta potential at low pH values was higher than that of the basic pH range. The negative zeta potential slightly increased to −5 mV with increasing the pH from 9 to 10.

Fig. 1 (curve b) shows the zeta potential as a function of pH for zircon. The iep was found to be at pH 5 in agreement with previous results [6,7]. A nonsymmetric curve was also obtained, at the acidic pH range the magnitude of the zeta potential was lower than that of the basic pHs. The negative zeta potential increased strongly to −80 mV by changing the pH to 9.

As the zeta potential is a measure of the electrostatic repulsion between particles in a suspension, a well dispersed suspension of alumina is expected to occur at $pH = 4$ where the alumina particle had a high surface charge at low electrolyte concentration (high zeta potential). Good dispersion of zircon powder was expected at $pH = 3$ or at higher pHs than 7 due to a high negative surface charge on the particles. At pH close to 9 electrostatic attraction between the neutral alumina particle and the highly negative charged zircon particle can occur.

3.2. Rheology

3.2.1. Effect of polyacrylate concentration

Fig. 2 (curve a) shows the apparent viscosity at 500 s^{-1} as a function of the amount of polyacrylate added for

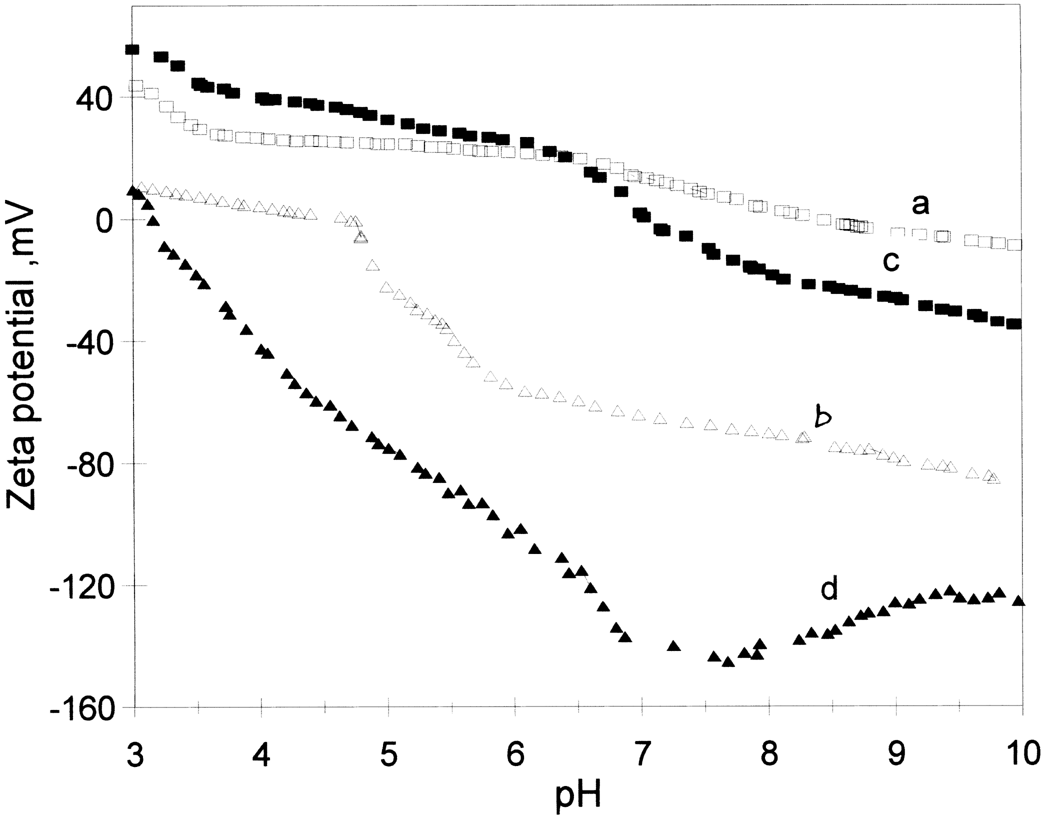

Fig. 1. Zeta potential versus pH curves: (a) alumina, (b) zircon, (c) alumina with polyacrylate adsorbed, (d) zircon with polyacrylate adsorbed.

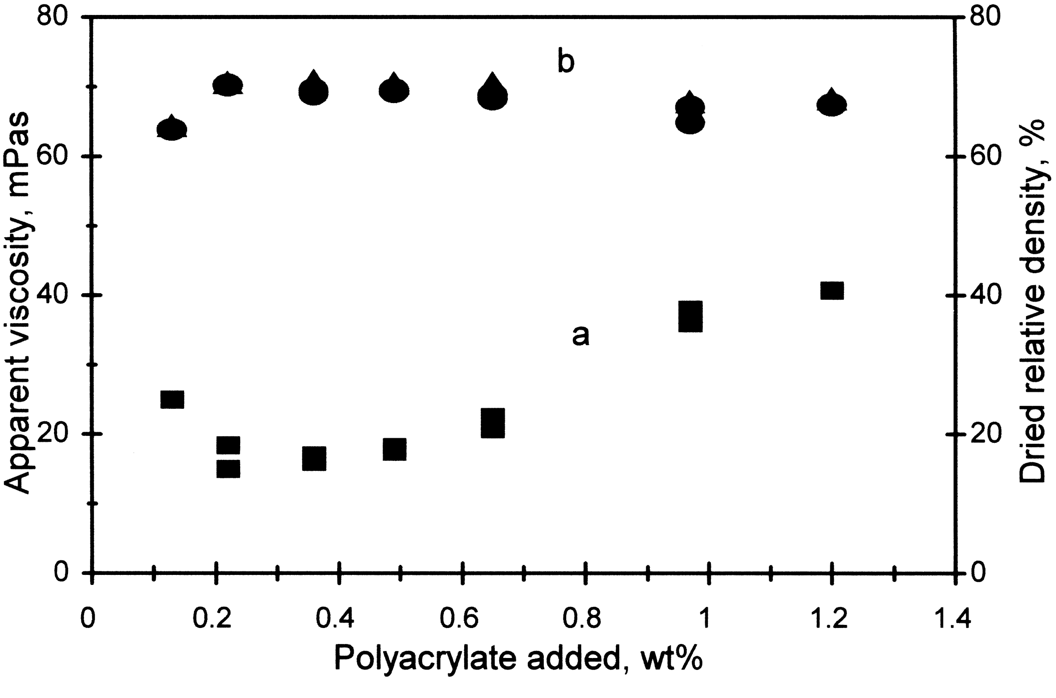

Fig. 2. (a) Apparent viscosity at 500 s^{-1} and (b) relative density of green compacts obtained from 48 vol.% suspensions by slip casting and pressure filtration as a function of the amount of polyacrylate added (▲ slip casting; ● pressure filtration).

48 vol.% suspension (pH = 9.1–9.2). At low dispersant concentrations high viscosity suspensions were obtained. The addition of 0.24 wt.% of polyacrylate slightly improved the degree of dispersion of the particles as lower apparent viscosity values (15 mPas) than the one of the suspension with 0.13 wt.% (25 mPas) was obtained. Further additions of dispersant up to 0.49 wt.% produced a minor variation on viscosity. For polyacrylate concentrations higher than 0.65 wt.% the apparent viscosity increased to 37–40 mPas probably due to the presence of an excess of polyelectrolyte.

The adsorption isotherm of polyacrylate on the mixed powder is shown in Fig. 3. The adsorbed amount reached a constant value up to a polyacrylate concentration of 0.65 wt.%. This may be explained by electrostatic repulsion between the polyacrylate anion on the surface groups and the polyelectrolyte present in the solution. The amount of polyacrylate at the plateau was 0.06 mg m^{-2}. As the adsorbed amount remained unchanged, for added polyacrylate concentrations up to 0.65 wt.%, a further increase in the amount of polyacrylate added gradually increased the amount of polyacrylate in solution.

However, as the added polyacrylate concentrations increased to 0.9 and 1.2 wt.% the amount of polyacrylate adsorbed increased to 0.12 mg m^{-2}. This may indicate that electrostatic repulsion decreased increasing ionic strength of the solution. Nevertheless, the amount of dispersant remaining in solution was high.

Polyelectrolytes like polyacrylate may be electrostatically adsorbed or chemically bound on the surfaces of the particles. Thus, the adsorption of polyacrylate depends mainly on the suspension pH, nature and the charge density on the powder surface [11].

The polyelectrolyte adsorption behaviour on alumina has been studied by Cesarano et al. [12]. They reported that the dissociation of COOH groups of the polyelectrolyte increases with increasing pH and the polyelectrolyte is fully dissociated at pHs above 8.5 [12]. According to these results, at suspension pH 9.1–9.2, the polyelectrolyte was in a dissociated form and negatively charged.

At pH 9.2, zircon particles had a very high negative surface charge generating a significative electrostatic repulsion between the zircon surface and the negatively charged polyelectrolyte in solution that resulted probably in a very low amount of polyacrylate adsorbed.

Whereas, at pH values 9.1–9.2, the alumina powder surface is neutral or slightly negatively charged as suspension pH was near pH_{iep}. Furthermore, adsorption of polyelectrolytes on alumina surface was attributed to both the electrostatic interaction between the COO^- groups of the polyacrylate and the positive sites on the powder surface and to the specific adsorption on the Al–OH groups. Thus, polyacrylate adsorption on the mixed powder may be controlled by the adsorption on the alumina surface as a weak adsorption on the zircon surface was expected.

Fig. 1 (curves c and d) shows zeta potential vs pH curves for alumina and zircon after adsorption of polyacrylate. The amounts of polyacrylate added for alumina and zircon were 0.44 and 0.05 wt.% respectively and corresponded to the concentrations required for optimum deflocculation at pH near 9.

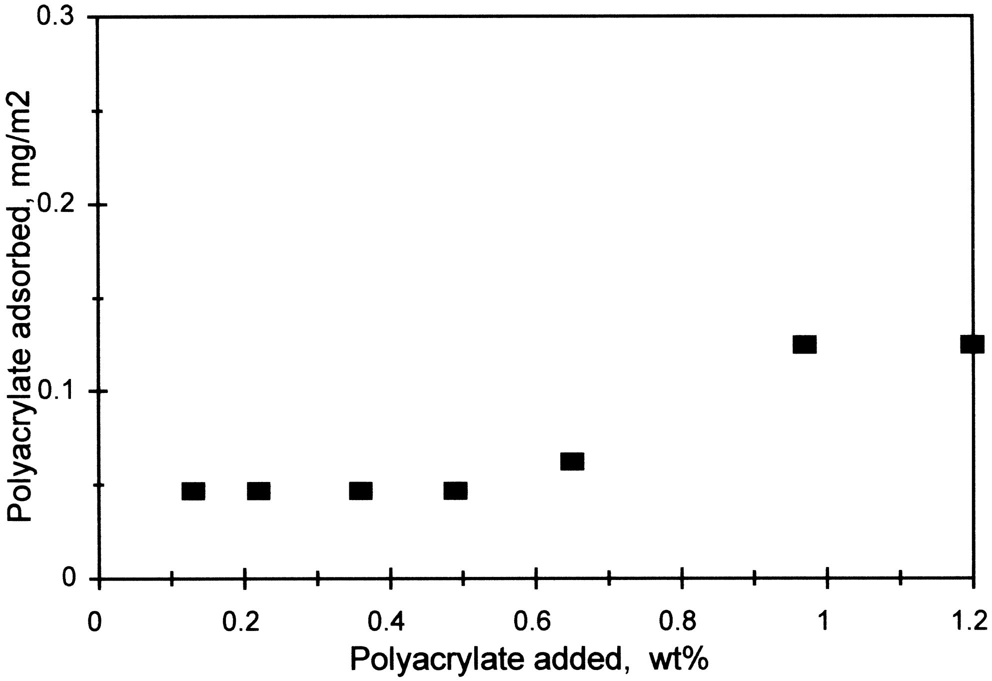

Fig. 3. Adsorption isotherm of polyacrylate on the mixed powder at pH 9.1–9.2.

Zeta potential vs pH curve for zircon with dispersant shows that the pH_{iep} decreased from 5 to 3.5 (indicative of anion adsorption) and the magnitude of the negative zeta potential increased approximately from −80 to −120 mV at pH 9.

Adsorption of COO^- groups on the alumina surface shifted the pH_{iep} to 7 and the surface charge became negative of in a wide range of pH values (Fig. 1, curve c) in agreement with previous results [14]. A negative zeta potential of −30 mV for the polyacrylate-coated alumina was found at pH near 9. Cesarano et al. [12] proposed that polyacrylate and methacrylate based dispersants stabilized alumina suspensions by a combined electrostatic and steric mechanism: adsorption of anionic polyelectrolytes increases the negative surface charge and produce some steric stabilization. Therefore, adsorption of polyacrylate gave to the particles a large charge (Fig. 1, curves c and d) and thus an effective electrostatic stabilization that decreased the viscosity of the suspensions.

At high polyelectrolyte concentrations, higher amounts of free polyelectrolyte remaining in solution caused a decrease in the degree of dispersion. Poor dispersion of the suspensions was attributed to double layer compression as the ionic strength of the solution increased or to depletion flocculation [11–14]. As attraction was significant the particles tend to form flocs with some volume of liquid immobilized. Hence, the suspension showed higher viscosities values and a typical shear thinning behaviour of flocculated suspensions.

3.2.2. *Effect of the solid volume concentration*

Fig. 4 shows the minimum viscosity of alumina–zircon suspensions deflocculated for solid volume concentrations up to 60 vol.% at pH 9.1–9.2. The amount of polyelectrolyte required to obtain the minimum viscosity was previously determined by the deflocculation curve and varied slightly with the solid content (from 0.22 to 0.24 wt.% for solid volume concentrations of 41 and 60 vol.%, respectively). The apparent viscosity at 500 s^{-1} slightly increased with the solid content up to 57 vol.%. Viscosities values of 15, 22, 30 and 56 mPas were obtained with increasing solid concentration from 41, 45, 48 to 57 vol.%, respectively. Increasing the solid content the viscosity of well-deflocculated suspensions mainly increase due to hydrodynamic interactions between particles.

However, a pronounced increase in viscosity from 56 to 260 mPas was observed with increasing solids content from 57 to 60 vol.%. Suspensions containing more than 57 vol.% of the mixed powder exhibited a strong shear thinning behaviour. Then, a solid concentration of 57 vol.% was chosen to study the effect of pH of the suspension on the viscosity and on the adsorbed amount of polyacrylate.

3.2.3. *Effect of pH*

Fig. 5 curve a shows the apparent viscosity at 500 s^{-1} as a function of suspension pH for a 57 vol.% suspension containing 0.24 wt.% of polyacrylate. At pH 8.6 a high viscosity value (192 mPas) was obtained. Increasing the

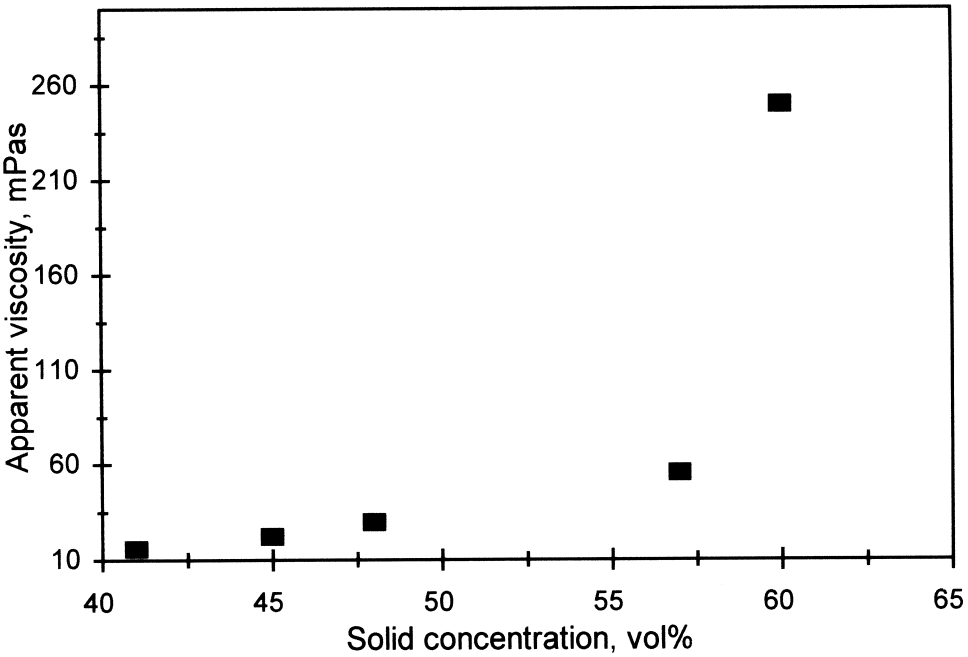

Fig. 4. Minimum apparent viscosity at 500 s^{-1} of alumina-zircon suspension containing 0.22–0.24 wt% of polyacrylate at pH near 9 for different solid volume concentrations.

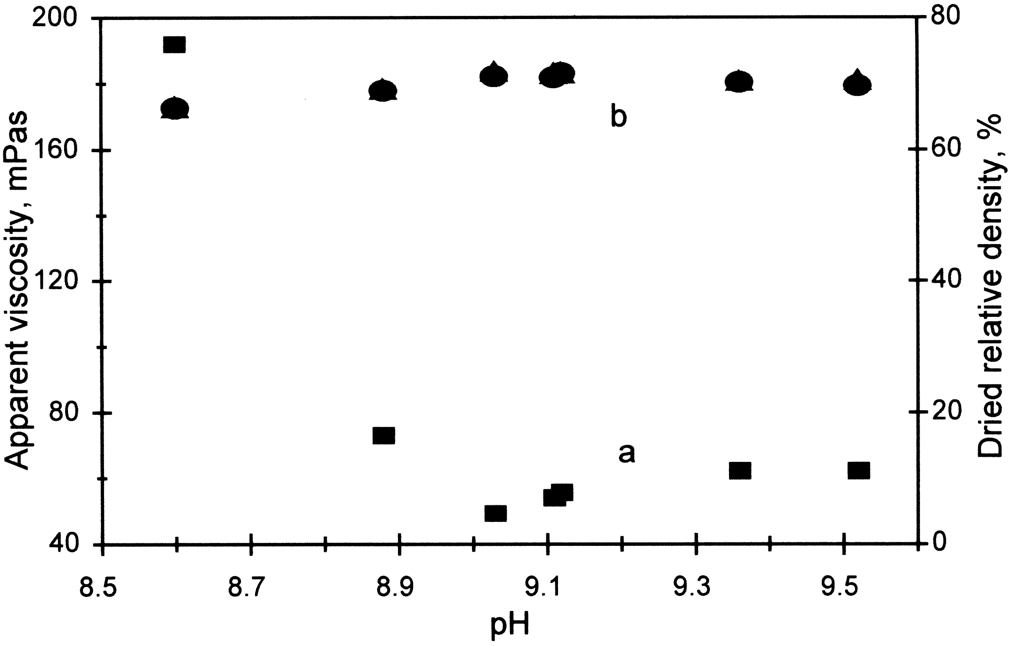

Fig. 5. (a) Apparent viscosity at 500 s^{-1} and (b) relative density of green compacts obtained from 57 vol.% suspensions by slip casting and pressure filtration as a function of the amount of polyacrylate added (▲ slip casting; ● pressure filtration).

suspension pH close to 9 improved the degree of dispersion of the particles and the minimum viscosity was obtained (49 mPas) then, further increase in pH produced a slight increase in the viscosity values. For pH 9.5, the apparent viscosity increased to 63 mPas.

The adsorbed amount of polyacrylate on the mixed powder as a function of pH is shown in Fig. 6. The adsorbed amount of polyacrylate increased from 0.03–0.04 to 0.06 mg m^{-2} with reducing pH from 9.5 to 9. At pH lower than 9 the amount adsorbed remained nearly constant.

At pH 8.6, more positive surface groups developed on the alumina surface that reduced the negative charge of the particles and increased the amount of polyacrylate anion adsorbed. At fixed dispersant concentration, the amount of polyacrylate added became insufficient and the net negative charge on the particles decreased with a consequent increase in viscosity. In addition, some electrostatic attraction between the fine and neutral alumina particles and the coarse and negatively charged zircon particles probably occurred [10]. Therefore, the increase in viscosity may be explained by some heterocoagulation.

At pH 9.5, zircon and alumina particles had a negative surface charge (negative zeta potentials) that increased the electrostatic repulsion between the powder surface and the polyacrylate anion in solution and therefore a low adsorption was observed. High viscosity values may be explained by the high free polyelectrolyte concentration in solution which decreased zeta potential by double layer compression or by depletion flocculation [11–14].

However, the magnitude of negative zeta potential of the powders gradually increased with increasing pH and the negative effect of the free polyelectrolyte on viscosity was less significative.

3.3. Green density vs viscosity

Suspensions were consolidated by slip casting in a plaster mould and by pressure filtration at 8 MPa.

Fig. 2 (curve b) and Fig. 5 (curve b) show the relative density of green of the compacts obtained from the mixed alumina–zircon suspensions with different dispersant concentrations for solid loadings of 48 and 57 vol.%, respectively. For a fixed solid content, the highest density was obtained from the suspension with the low viscosity. Compacts prepared by slip casting from a minimum viscosity suspension of 48 vol.% (15 mPas) reached a density of 3.03 g cm^{-3}, 70.5% theoretical density. Although an increase in the solid loading to 57 vol.% increased the minimum viscosity to 49 mPas, the density of green bodies increased to 3.09 g cm^{-3}, 71.9%. In a presence of an optimum dispersant concentration increasing the solid content (increasing the viscosity) the settling of the particles is reduced and the suspension remained homogeneous. Homogeneous suspensions can pack more efficiently. For 48 vol.% suspension containing a concentration of dispersant of 0.36–0.65 wt.% the green density values varied between 70.6–70.4%. Shear thinning was an important rheological property of these suspensions because at low shear rates the suspension had a high viscosity so that settling of the particles can not occur.

At higher additions of polyacrylate, green densities slightly decreased as the viscosity increased because of the presence of flocs in suspensions that decreased the

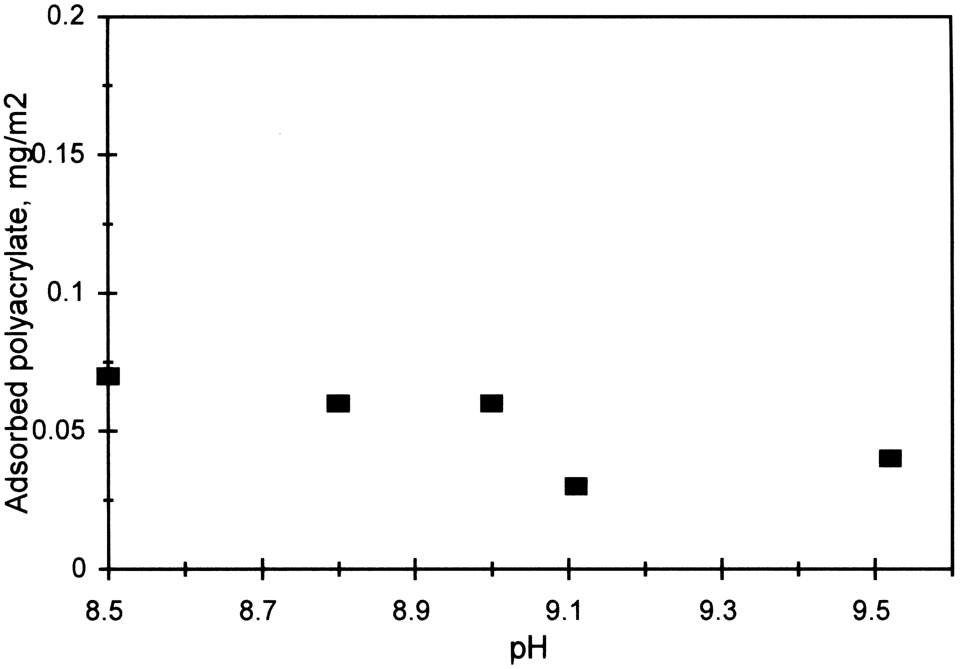

Fig. 6. Adsorbed amount of polyacrylate as a function of pH.

packing density (increased the porosity) of the green compact.

Pressure filtration of 48 vol.% suspension of the minimum viscosity (15 mPas) gave compacts with a green density of 70.3%. Green density values of 70.9% were obtained by pressure filtration 57 vol.% suspension having a viscosity of 49 mPas.

Furthermore, the effect of the degree of dispersion on the green density was similar to that previously reported [5]. Compacts resulting from weakly flocculated suspensions were less dense and had a high open porosity.

3.4. Sintering of green compacts

Final density of zircon-alumina bodies depends on the thermal treatment employed. Up to 1450°C densification takes placed and a composite of zircon and alumina with a density near 4.05 g cm^{-3} can be obtained for a stoichiometric mixture. Reaction sintering leads to a mullite–zirconia composite at temperatures up to 1600°C controlling the sintering time. If reaction is complete the composite reaches a density near 3.84 g cm^{-3} depending on the relative content of tetragonal and monoclinic zirconia (t or m-ZrO_2).

In Fig. 7 XRD diagrams of the sintered pressure filtered compacts at 1450 and 1600°C (2 h) are shown. At 1450°C no reaction occurred and zircon and alumina were the phases present. At this temperature the density reached was 3.88 g cm^{-3}. At 1600°C mullite and zirconia (t and m) appeared as the principal phases. The presence of minor quantities of zircon indicated that reaction was not complete.

Fig. 8 shows the sintered density of compacts prepared from 48 to 57 vol.% suspensions by slip casting and pressure filtration. Fig. 8 showed a linear relation between green and sintered density values. Samples with high green density produced compacts with the highest sintered densities.

Composites prepared from stable and low viscosity suspensions containing 0.22–0.24 wt.% of dispersant at pH 9 reached a final density of 3.70–3.74 g cm^{-3} for 48 and 57 vol.% suspensions, respectively whereas for the unstable suspensions a reduction in density to 3.68–3.7 g cm^{-3} was observed. A pronounced reduction of open porosity to 0.3% was found for the compact with the highest sintered density. Moreno et al. [6] reported sintered densities $>97\%$ TD resulting from slip casting of zircon–alumina suspensions.

Although in a few percentage, sintered at 1600°C, 2 h samples prepared by slip casting produced bodies with a final density higher than that obtained by pressure filtration.

The distribution of Al_2O_3 and ZrO_2 contents in a sintered sample (1600°C, 2 h) prepared from a well disperse 57 vol.% suspension by pressure filtration was examined using EDAX. Al_2O_3 and ZrO_2 contents were measured in a cross section area along seven points situated from the top to the bottom of the compact. The results showed that Al_2O_3 and ZrO_2 were uniformly distributed as the atomic Al/Zr ratio varied between 3.6 to 3.2. The differences between the Al/Zr ratio obtained and the expected ratio (3.0) is related to a high error in the determination of Zr. Due to an overlap of the principal Kα peak of Zr, a lower intensity peak was used for the measurement.

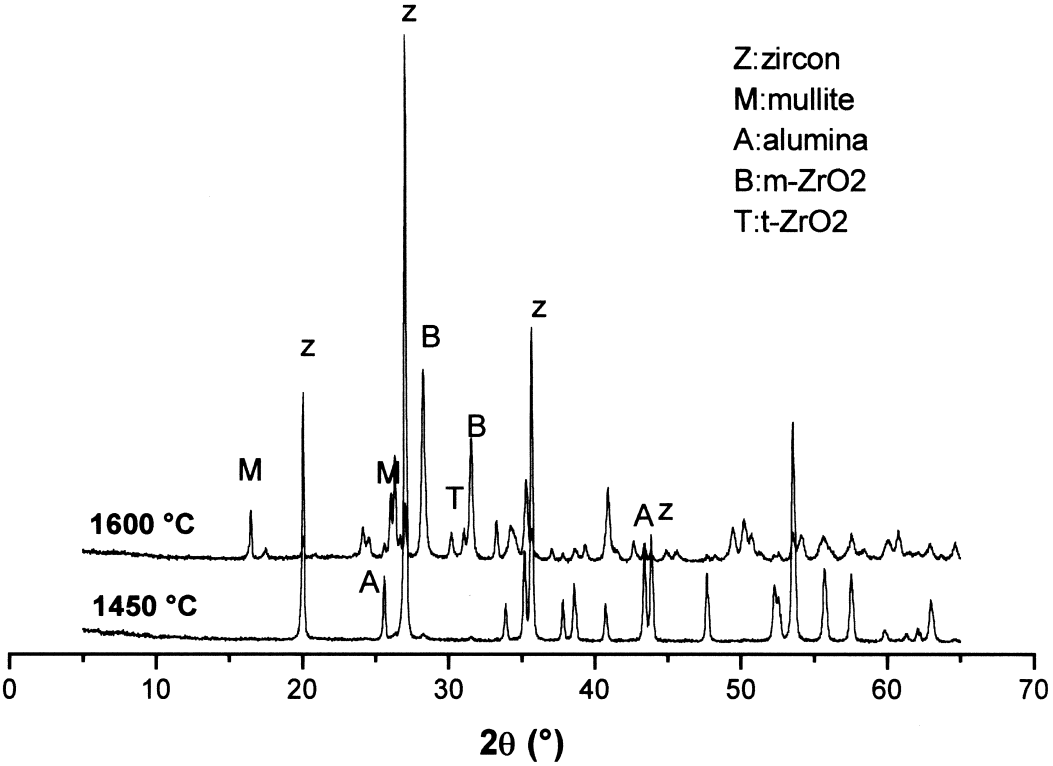

Fig. 7. XRD diagrams of the sintered pressure filtered compacts at 1450 and 1600°C (2 h).

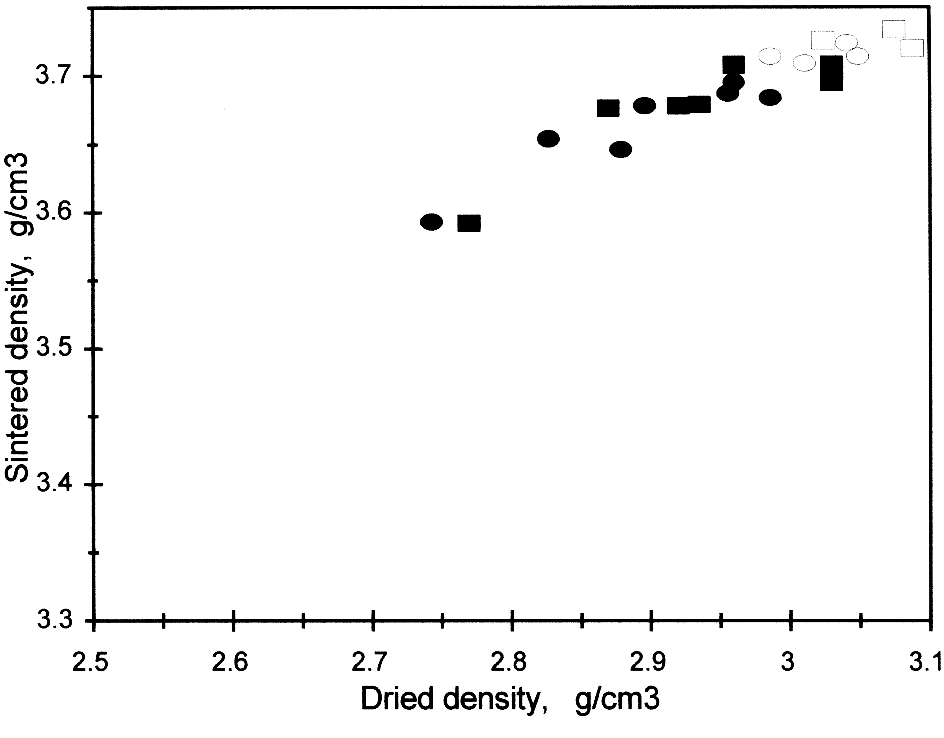

Fig. 8. Sintered density versus density of green compacts obtained from 48 and 57 vol.% suspensions by slip casting and pressure filtration (48 vol.%: ■ slip casting ● pressure filtration 8 MPa; 57 vol.%: □ slip casting, ○ pressure filtration 8 MPa).

If segregation of the dense and coarse zircon particles took place there would be a continuous variation in the Al/Zr ratio from the top to the bottom. The small variation in the ratio is an indication of a good dispersion of the particles in the suspension that was maintained during the filtration process. According to Salomoni et al. [15] a barrier for possible segregation of components increased with increasing the rate of the forming process.

4. Conclusions

Concentrated aqueous suspensions of alumina–zircon mixtures containing up to 57 vol.% solid concentration were prepared using a polyacrylate based dispersant. Addition of 0.24 wt.% of dispersant at pH 9 was appropriate to obtain a colloidally stable (low viscosity) suspension. An adsorbed polyacrylate amount of 0.06 mg m^{-2} was sufficient to produce the minimum viscosity. As the added concentration increased, the adsorbed amount remained low as compared to the amount remaining in the solution and the excess of polyacrylate produced a weakly flocculated suspension.

It was possible to prepare dense composites of zircon and alumina by slip casting and pressure filtration at 8 mPa controlling the degree of dispersion of the particles that produced after sintering a dense mullite–zirconia ceramics. Optimization of the amount of polyacrylate added and pH of the suspension improved the solid loading and consequently the green density of the cast samples.

Colloidally stable (low viscosity) suspensions produced green compacts with the highest relative density. As indicated by a correlation between green and sintered densities values, high green density is desirable in achieving a high fired density. Examination of the atomic Al/Zr ratio has shown an uniform distribution of the two components in the sintered sample.

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