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Optimization of the rheological properties of alumina slurries for ceramic processing applications
Part I: Slip-casting

Athena Tsetsekou [1], Christos Agrafiotis *, Aggelos Milias

Ceramics and Refractories Technological Development Company, (CERECO S.A.), 72nd km of Athens – Lamia Ntl. Road, PO Box 146, 34100, Chalkida, Greece

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Abstract

Aqueous powder slurries are widely employed in ceramic manufacturing processes like slip-casting and spray-drying. The aim of the present work was to identify the conditions for the preparation of stable alumina slurries with high solids content for the production of slip-cast objects with improved properties, as well as to correlate the slurry properties to the final object properties. For slurry stabilization, three commercial dispersants were compared. It was found that for each dispersant there exists an optimum concentration range within which low viscosity is achieved for a slurry of high solids content. In addition to the slurry solids content, the choice of a particular dispersant also affects the slurry viscosity and through that the casting rate. The combination of high slurry solids content and slower casting rate results in objects with higher densities both in the green and fired state. © 2001 Published by Elsevier Science Ltd. All rights reserved.

Keywords: Al_2O_3; Dispersants; Rheological; Slip-casting

1. Introduction

In many ceramic shaping processes such as slip-casting, tape-casting or spray-drying, the dispersion of the ceramic powder in an aqueous medium is required. These aqueous suspensions (slurries) have to fulfill several requirements. The particles should not settle fast under the effect of gravity, but they should be able to remain in suspension, because otherwise segregation occurs which causes density inhomogeneities in the cast objects. In addition, the slurries have to be easily reproduced and be insensitive to slight variations in solids content, chemical composition and storage time. They also have to be of high solids content, in one hand to achieve reasonable casting rates and on the other hand to reduce energy consumption in the subsequent drying stage due to the lower moisture content to be removed.[1,2] Furthermore, the prevailing trend in ceramic processing is the development of very fine particles in order to enhance sintering rates as well as to reduce the size scale for mixing uniformity in powder blends. However, the combination of high solids loading and small particles leads to a viscosity increase because of increased particle–particle interactions and, consequently, to difficulties in slurry handling.[3]

Ceramic powders have the tendency to agglomerate due to the attractive intra-particle Van der Waals forces. This tendency can be eliminated with the addition of appropriate dispersants which alter the powder surface properties so that repulsive forces — either due to electrostatic repulsion resulting from the overlapping of electrical double layers or due to steric hindrance resulting from absorption of large molecules — become higher than the attractive ones and the particles can remain separated in suspension.[4] Dispersants that are functioning both via an electrostatic and a steric mechanism are called poly-electrolytes which usually consist of a hydrocarbon chain and a polar ionic part (COO^-, SO_3^-).[5–7] Dispersant addition can dramatically reduce the viscosity of slurries with very high solids content, thus ceramic industry has a constant demand for effective dispersants or deflocculants.[8,9]

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* Corresponding author at current address: Department of Materials Science and Engineering, University of Ioannina, University Campus, 47110, Ioannina, Greece.

E-mail address: chagrafi@cc.uoi.gr (C. Agrafiotis).

[1] Current address: Department of Mineral Resources Engineering Technical University of Crete, University Campus, 73100 Chania, Greece.

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Various dispersants are used for the stabilization of oxide powder slurries (alumina, titania, zirconia, etc.), but poly-methyl-acrylate salts — usually referred with their commercial names, such as Darvan, Dolapix, etc. — are probably the most frequently employed. The kind of a dispersant to be used with a particular ceramic powder as well as its optimum quantity, have to be determined in order to prepare stable slurries of high solids content that can produce defect-free, high quality products. Techniques like adsorption measurements,[10–13] zeta-potential measurements,[12–14] acoustophoresis,[15] etc. are used as preliminary screening among various dispersants ; however, whether or not the choice has been successful should be verified by measurement of the slurry properties and finally reflected on the final properties of the objects produced.

Cesarano et al.[10,11] have tested poly-methacrylic acid and have shown that the degree of dissociation depends on the slurry pH. At pH values greater than 8.5, the polyelectrolyte dissociates almost completely to produce negatively charged anions that exhibit high affinity absorption on the α-alumina powder. On the contrary, the negligible polyelectrolyte dissociation at pH lower than 4.0 makes it a much less effective dispersant at acidic pHs. Zhang et al.[16] used poly-methacrylic acid for the stabilization of nanometer-sized α-alumina and zirconia powders for use in slip-casting. They observed a large increase in viscosity with an increase of the solids content of the slurry, and an optimum in dispersant concentration for slurry stability; for dispersant concentration higher or lower than this value, sedimentation percentage increases. Hampton et al.[17] used sodium polyacrylate for the slip casting of alumina and observed an optimum dispersant concentration for minimum slurry viscosity. They have also found that a mixture of coarse and fine powders produced the cast specimens with the highest green density. The concept of combining coarse and fine particles for the achievement of better particle packing during slip casting has also been employed by several other researchers.[18–23] Other organic dispersants employed include citric acid,[24] benzoic acid and its derivatives[12,26] and polyphosphates.[27,28] The kind of organic dispersant used was found to affect the green density of the cast specimens as well as the grain size of the sintered specimens.[25]

The aim of the present work was to identify the conditions for the preparation of stable alumina slurries with high solids content for the production of slip-cast objects as well as to correlate the slurry properties to the final object properties. Thus, the first part involved the comparison of commercially available dispersants and the identification of the optimum conditions for slurry stability. The second part involved the preparation of cast objects by slip-casting and the study of the effect of the slurry rheological properties on the casting performance. Finally, the last part involved the characterization of the green and fired cast objects so that the effect of slurry properties on the product properties could be identified. A second publication that will follow will involve the study of the compatibility of the same commercial dispersants with commercially available binders, and the preparation of stable slurries appropriate for spray-drying.

2. Experimental

The α-alumina powder used was the type NABALOX No 625-30 (VAW aluminium AG, Schwandorf, Germany), with a characteristic mean diameter of 3 μm (measured with the aid of a Malvern Instruments M5.4 laser particle size analyzer) and a specific surface area of 4.5 m^2/g (measured with the aid of a Micromeritics ASAP 2000 Nitrogen porosimeter). The characteristics of the powder are summarized in Table 1. Three commercially available dispersants were used for slurry stabilization, known with the commercial names Darvan C (R. T. Vanderbilt Co., Norwalk, CT, USA), Duramax D 3005 (Rohm and Haas, Lauterbourg, France) and Dolapix CE 64 (Zschimmer & Schwarz GmbH & Co, Lahnstein, Germany). Darvan C is an ammonium salt of the poly-methacrylic acid with an average molecular weight of 10,000, with low tendency to foam. Duramax D 3005 is also an aqueous solution of an ammonium salt of an acrylic polymer whereas Dolapix CE 64 is a carbonic acid-based, alkali-free polyelectrolyte. All three dispersants are completely water-soluble and were provided from the manufacturers in the form of aqueous solutions. The active matter percentage for each solution is given in Table 2, together with other characteristic properties.

Slurries with solids content 40, 60, 70 and 80 wt.% (corresponding to 14.6, 27.8, 37.4 and 50.6 vol.%) were prepared with the ultimate goal to determine the optimum conditions for stabilization of slurries at high

Table 1
Properties of the alumina powder used

Alumina powder characteristics	
Manufacturer	VAW aluminium AG
Type	NABALOX 625-30
α-Phase content	>95%
Characteristic mean particle diameter d_{50} (μm)	3
Characteristic particle diameter d_{90} (μm)	6
Specific Surface Area (m^2/g)	4.5
Density (g/cm^3)	3.9
Impurities (wt.%)	
Na_2O	0.25 max.
SiO_2	0.05 max.
$Fe2O_3$	0.04 max.

Table 2
Properties of the aqueous solutions of the dispersants used

	Darvan C	Duramax D 3005	Dolapix CE 64
Active matter (wt.%)	25	35	70
Density (g/cm^3)	1.11	1.15	1.10
pH	7.5	6.5	7.0

solids content (80% wt.). After the addition of the dispersant into a slurry of a given solids content, the slurries were treated by ball-milling with alumina grinding media for 8 hours and subsequently aged under mechanical stirring for 16 hours. Immediately after aging, slurry viscosity was determined with a rotating-spindle viscometer (Brookfield RVT DV-II). Sets of measurements with a specific spindle were taken at all shear rates (rotational speeds) where a reading of the viscosity values could be obtained. The slurry stability was evaluated in a sample of each as-prepared slurry, by measuring the particles settling rate inside glass test tubes. For this purpose, the height of the sediment inside these calibrated test tubes was measured as a percentage of the total suspension height, at regular time intervals.

Thereafter, the slurries were used for the production of two kinds of specimens by slip-casting: cylindrical crucibles for the measurements of casting rate and sample density in the green state and cylindrical rods for the measurement of mechanical strength. For the measurement of the casting rate, identical plaster-of-Paris moulds for cylindrical crucibles (dimensions : 49 mm in diameter by 72 mm in length) were filled to the top with a particular slurry. Each mould was kept at this state for a different time period (casting time), after which the slurry was drained and a green final object (crucible) was formed on the mould walls. The casting rate was obtained by varying the casting time between 4 and 55 min and measuring the wall thickness of each crucible. For the preparation of cast samples for bending strength measurements, a particular slurry was poured in a cylindrical plaster-of-Paris mould (14.4 mm diameter by 150 mm length). The amount of water absorbed by the mould walls, was progressively replaced with new slurry, until the whole mould was filled with the green cast specimen and a solid rod was obtained.

After casting, the specimens were dried at 110°C for 2 h and then fired at 1400, 1500 and 1600°C for 2 h with a heating rate of 3°C/min. The fired specimens were evaluated with respect to density (Archimedes' method) and microstructure (scanning electron microscopy, JEOL JSM-6300 microscope). The mechanical strength of the specimens was investigated by performing three-point bending tests in a mechanical testing device (Instron 8562). The value obtained for the bending strength was the average of six measured rods cast from each slurry.

3. Results and discussion

3.1. Optimization of rheological properties of slurries

The particle size distribution of the alumina powder employed as well as an SEM photograph of the powder are shown in Fig. 1. From the particle size distribution measurements occurred that the powder exhibits a wide size distribution with a mean diameter of 3 μm and 90% of the powder being below 6 μm (Fig. 1a). This is further verified by the SEM photograph of the powder (Fig. 1b) where dense particles with dimensions around 1 μm are shown together with particles ranging from 3 to 5 μm.

Stabilization of slurries of high solids content for slip-casting applications is achieved with the addition of an appropriate dispersant, which on one hand will impart low viscosity to the slurry and on the other hand will ensure that the powder particles will not settle within a short period of time (which for slip-casting applications is usually 1–2 h). It is known that slurries of ceramic powders do not exhibit Newtonian flow behavior (i.e. constant viscosity, independent of the shear rate). In the particular study all slurries exhibited shear-thinning behavior (i.e. a reduction in apparent viscosity with increasing shear rate), irrespective of the slurry solids content, and the kind of dispersant added.

Darvan C was employed for the stabilization of slurries with 40, 60 and 80 wt.% solids content, and its concentration was varied from 0.1 to 1.5 wt.% (based on polymer solution, which corresponds to 0.025–0.375 of active matter), per dry alumina powder. The pH of the slurries varied between 9.0 and 9.5 depending on Darvan C concentration. The effect of dispersant concentration on viscosity is shown in Fig. 2. Viscosity measurements for the slurries of 40 and 60 wt.% were obtained with a rotational speed of 100 rpm using the largest spindle (no. 1), whereas those of the slurries of 80 wt.% solids content with the same spindle but with a rotational speed of 50 rpm. Slurries of low and intermediate solids content (40 and 60 wt.%, respectively), exhibit constant, low viscosity values (15 and ~25 mPas, respectively), independent of dispersant concentration. For the slurries of high solids content (80 wt.%), slurry viscosity depended on the dispersant concentration. Very high slurry viscosity is observed for dispersant concentration lower than 0.075 wt.%. In the region of dispersant concentration 0.075–0.125 wt.%, a constant viscosity, equal to 97 mPas was observed. Finally, for dispersant concentration higher than 0.2 wt.%, a significant rise of slurry viscosity again is observed.

Slurry stability was evaluated by measuring the settling of powder (sediment height as a percentage of the total suspension height) as a function of time and dispersant concentration. The stability for the slurries of 40 and 60 wt.% solids content depended on dispersant

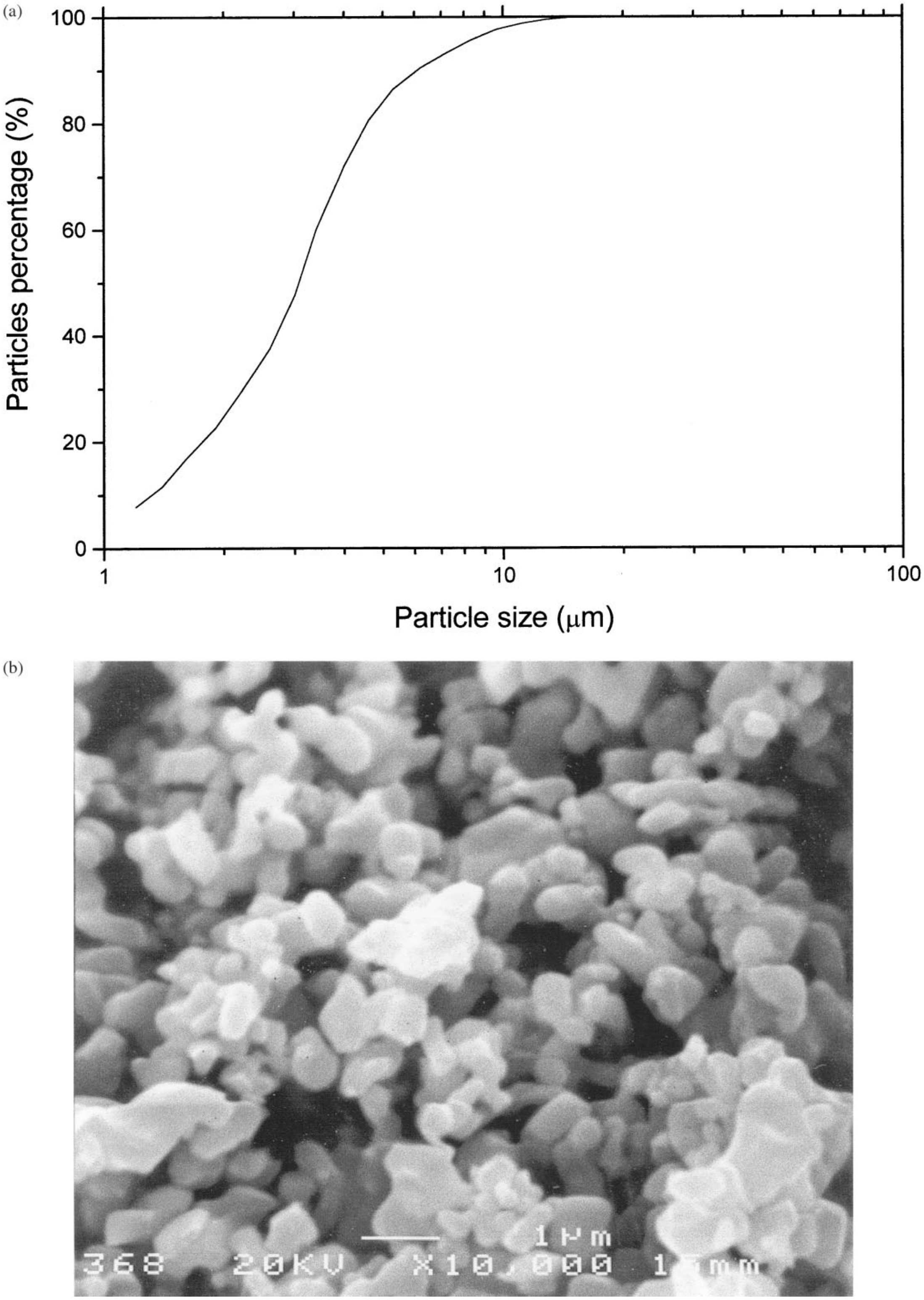

Fig. 1. Size and morphology of the alumina powder employed: (a) particle size distribution, b) SEM photograph.

concentration. This trend can be observed in Fig.3, where the results for a slurry of 60 wt.% solids content are illustrated. The optimum Darvan C concentrations, where minimum settling was observed, were determined to 1 wt.% for the 40 wt.% solids content slurry and to 0.7 wt.% for the 60 wt.% solids content slurry (values equivalent to 0.25 and 0.175 wt.% of active matter concentration, respectively). In contrast, the slurries

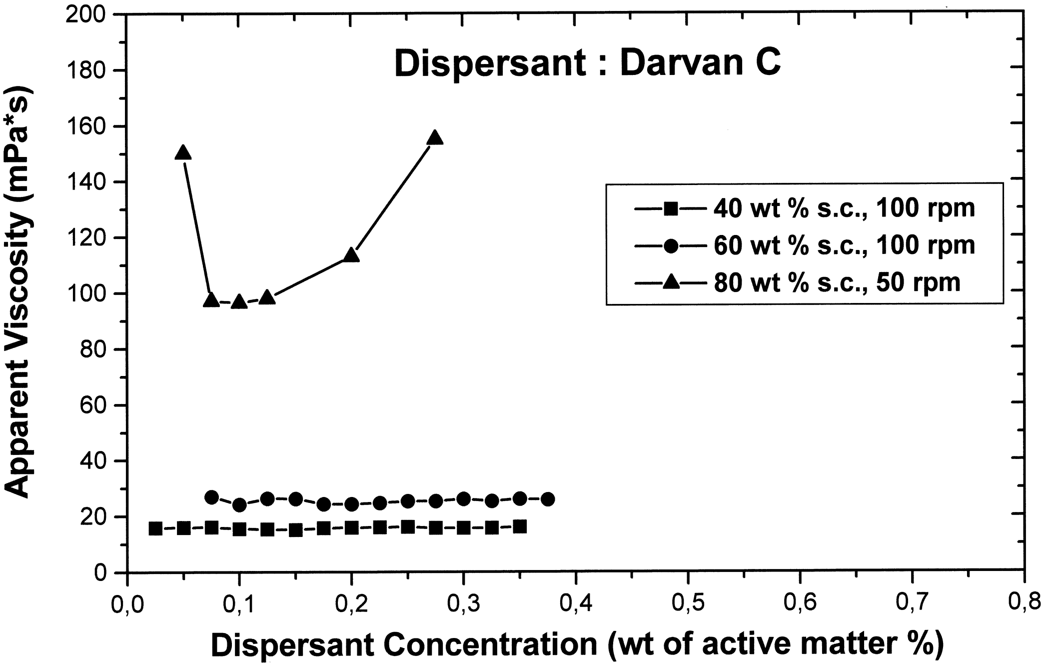

Fig. 2. Effect of Darvan C concentration and slurry solids content on slurry viscosity.

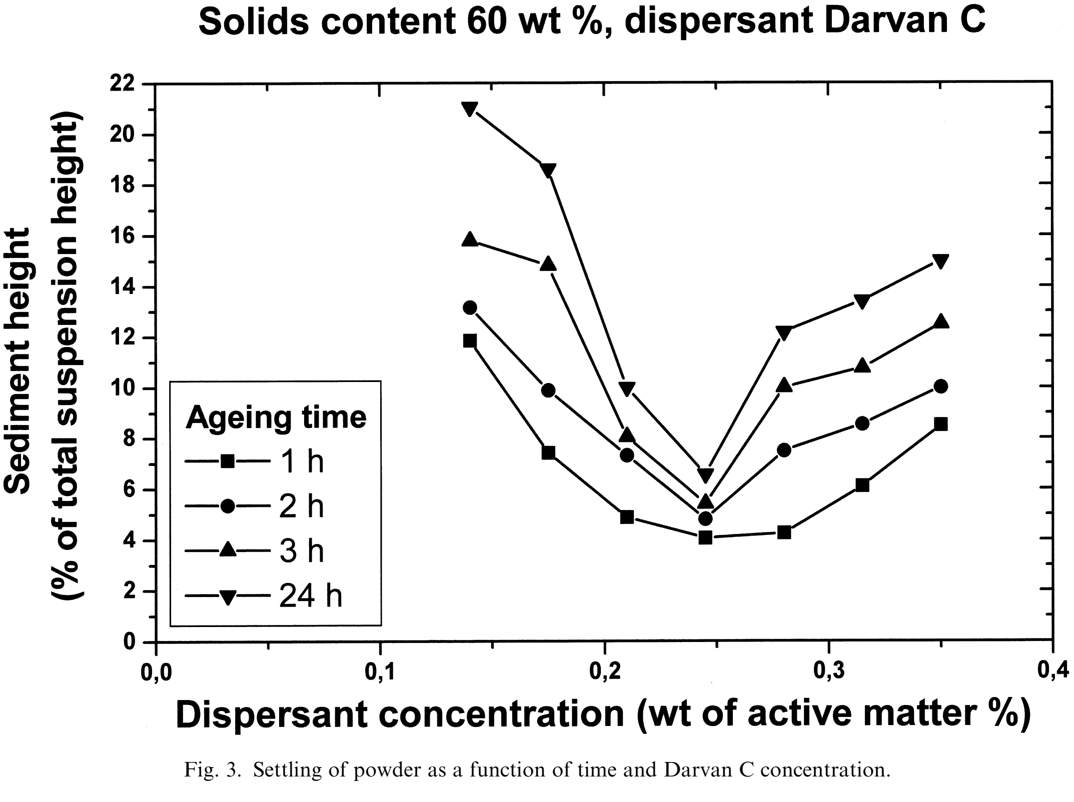

Fig. 3. Settling of powder as a function of time and Darvan C concentration.

with 80 wt.% solids content exhibited minimal settling in the whole range of dispersant concentration values tested. This is because, outside the range of dispersant concentration where minimum viscosity is achieved (0.075–0.125 in this case) due to the high slurry solids content, a flocculated network structure is formed which impedes particle sedimentation. In summary, slurries with 80 wt.% solids content and Darvan C concentrations between 0.075 and 0.125 wt.% (based on active matter) exhibit relatively low viscosity so that they can be handled easily and used successfully for slip-casting.

Duramax D 3005 was used for the stabilization of slurries with 60, 70 and 80 wt.% solids content. Its concentration was varied between 1.0 and 2.0 wt.% (0.35–0.70 wt.% based on active matter) inducing a variation on the slurry pH between 8.5-9.5. Just like the case of Darvan C, a lower shear rate was required for viscosity measurements of the slurries with the higher solids content. The effect of dispersant concentration on slurry viscosity is shown in Fig. 4. For each solids content, a region of dispersant concentration where viscosity is minimum, is observed. This region becomes narrower as the slurry solids content is increased. For 60 and 70 wt.% slurries, minimum settling was observed at a dispersant concentration of 0.07 wt.%, a value that induces low viscosity as well. Just like the case of Darvan C, slurries with 80 wt.% solids content exhibited minimal settling in the whole range of dispersant concentrations studied.

In the case of the dispersant Dolapix CE 64, only slurries with 80 wt.% solids content were investigated so that the three dispersants could be compared with respect to the ultimate goal which was the stabilization of high-solids-content slurries. Dolapix CE 64 concentration was varied between 0.14 and 0.70 wt.% (based on active matter) resulting in a pH variation from 8.9–9.5. The effect of Dolapix CE 64 concentration on slurry viscosity is shown in Fig. 5 where it is compared to the respective slurries (80 wt.% solids content) stabilized with the other two dispersants. A region of Dolapix CE 64 concentration between 0.28 and 0.35 wt.% is observed where minimum slurry viscosity (~105 mPas) is achieved.

From the curves in Fig. 5, it occurs that for each dispersant there exists a concentration range within which minimum slurry viscosity is observed. In general, small dispersant concentrations are not adequate to fully deflocculate the slurry and maintain the colloidal particles in dispersion. On the other hand, high dispersant concentrations result in an increase in viscosity as well. The lowest viscosity value (88.7 mPas) was observed for Duramax 3005 and active matter concentration 0.14 wt.%. However, the concentration range of this particular dispersant where low viscosity is achieved, is very narrow. Slurries stabilized with Dolapix CE 64 exhibited the highest viscosity values for each dispersant concentration. The lowest viscosity with this dispersant (~103 mPas) was observed in the concentration range 0.28–0.35 wt.%, which is much higher than these of the other two dispersants. In the case of Darvan C, low viscosity (~97 mPas) was observed within a wider dispersant concentration range (0.075–15%) a fact that gives an advantage to use this particular dispersant.

3.2. Slip-casting

Cylindrical crucibles were prepared by slip-casting and the casting rate was measured (thickness of crucible wall as a function of casting time). For each solids content and dispersant used, the amount of dispersant in the slurry was that which imparted minimum slurry viscosity, determined from the previous measurements.

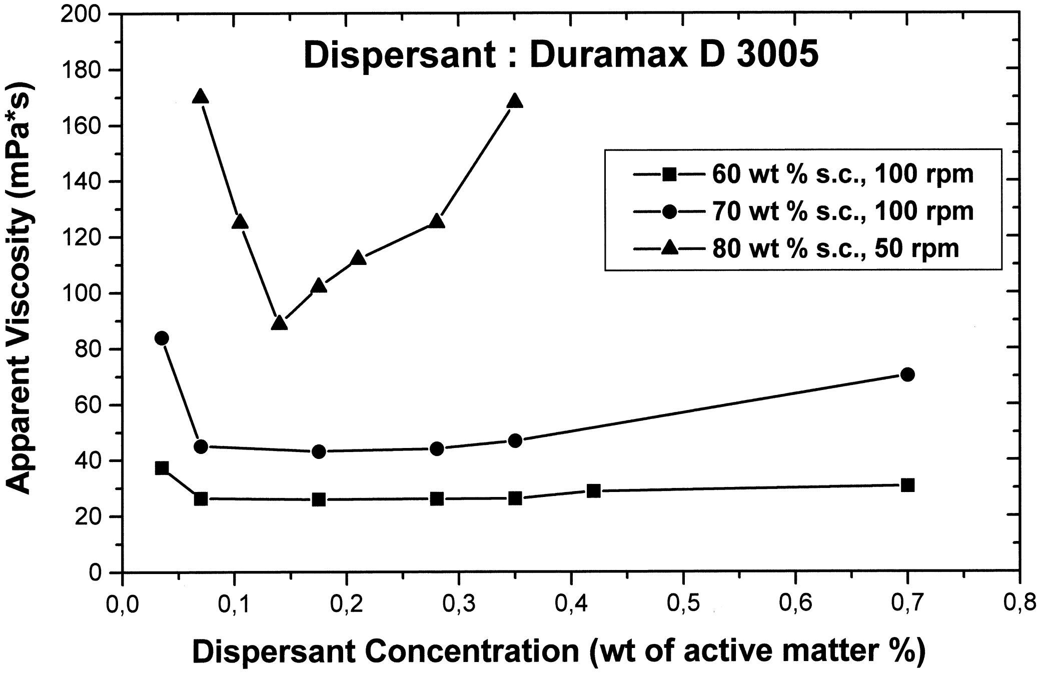

Fig. 4. Effect of Duramax D3005 concentration and slurry solids content on slurry viscosity.

The results are summarized in Fig. 6 (with the proportion of dispersant referring to active matter concentration). The advantage of high solids content in the slurry is evident. For each particular dispersant used, slurries with 80 wt.% solids content exhibit higher casting rate (greater wall thickness in the same casting time) than the slurries with 60 wt.% solids content.

From the comparison among slurries with the same solids content but stabilized with different dispersants, it appeared that the slurry with the highest viscosity (the

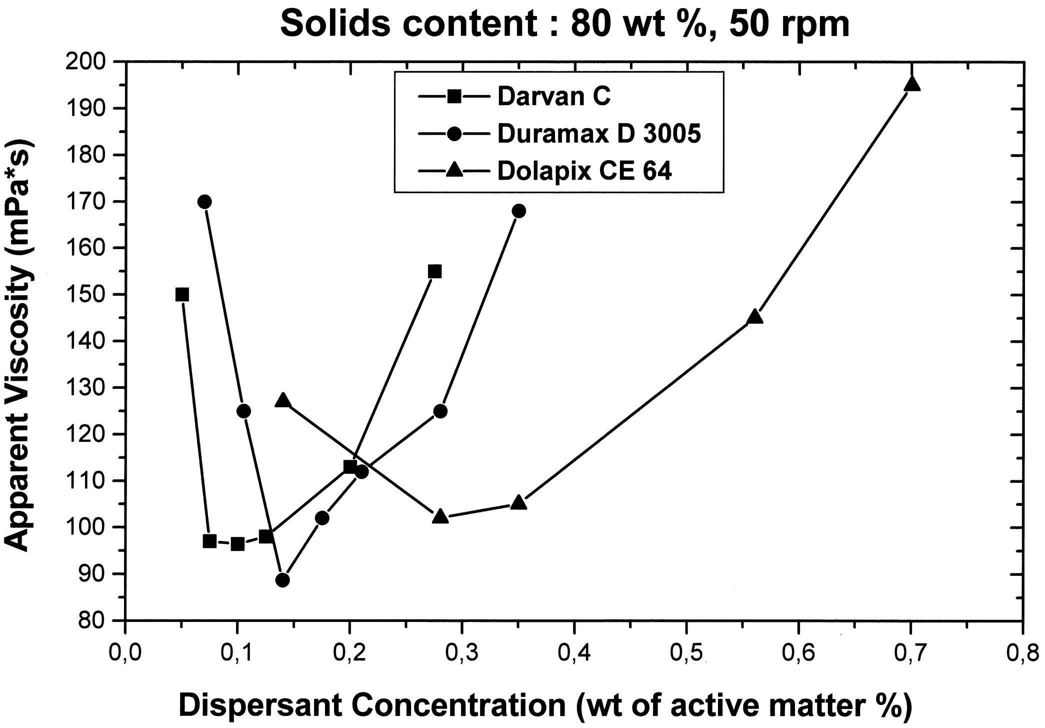

Fig. 5. Comparison among the three dispersants on the effect of dispersant concentration on slurry viscosity for slurries with 80 wt.% solids content.

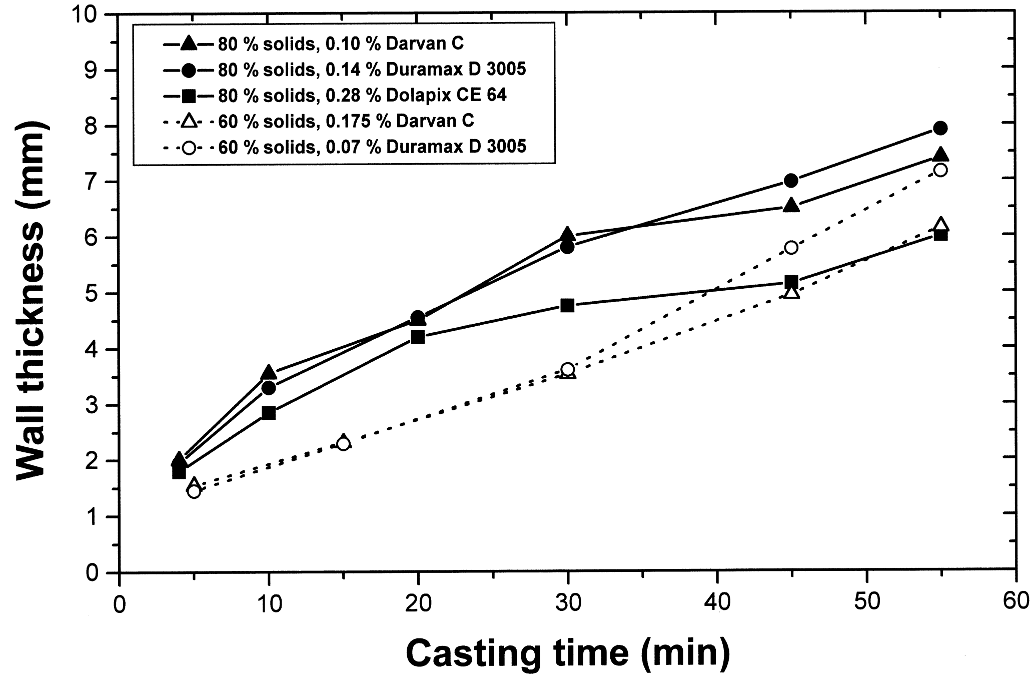

Fig. 6. Effect of slurry solids content and kind of dispersant used on the casting rate of alumina crucibles.

one stabilized with Dolapix CE 64) exhibited the lowest casting rate. This fact is important since casting rate affects the specimen properties, as it will be discussed later. In Fig. 7, the densities of the fired cast specimens are plotted as a function of firing temperature and slurry properties (dispersant, solids content). On one hand, specimens from slurries of higher solids content, exhibit as expected, higher densities. On the other hand, among specimens from slurries with the same solids content, the ones with the slowest casting rate (those from the slurry stabilized with Dolapix CE 64) exhibit the highest density. The slower casting rate results in better "packing" of the ceramic particles and consequently higher specimen densities, both in the green and in the fired state.

The packing of powder particles during slip-casting has been the subject of numerous investigations. For optimum packing, mixtures of coarse and fine powders

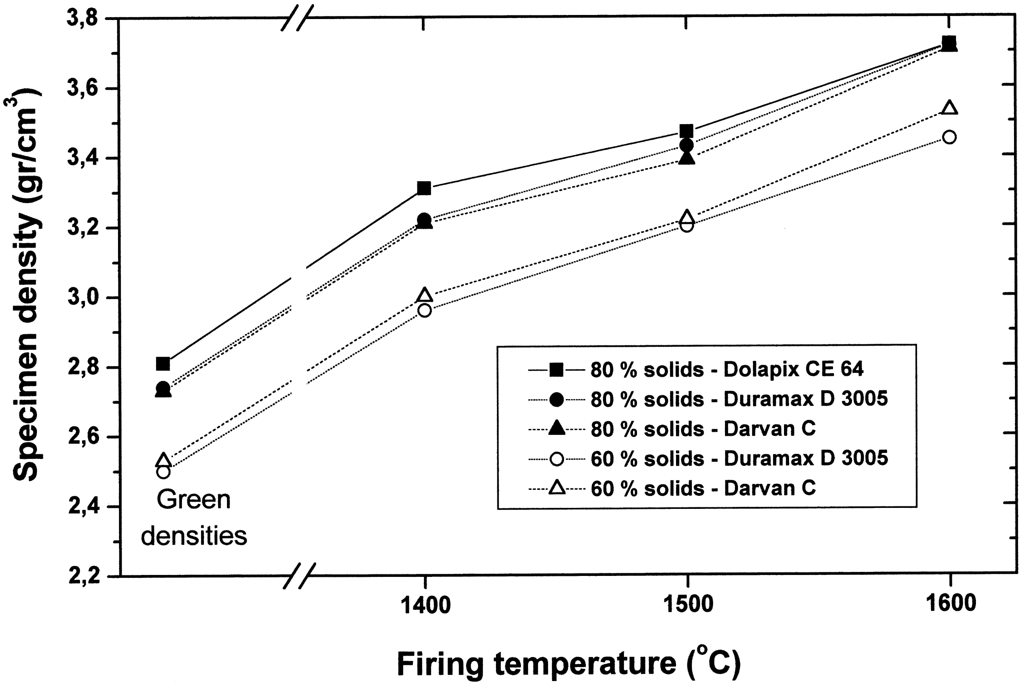

Fig. 7. Effect of slurry solids content, kind of dispersant used and firing temperature on the density of fired alumina crucibles.

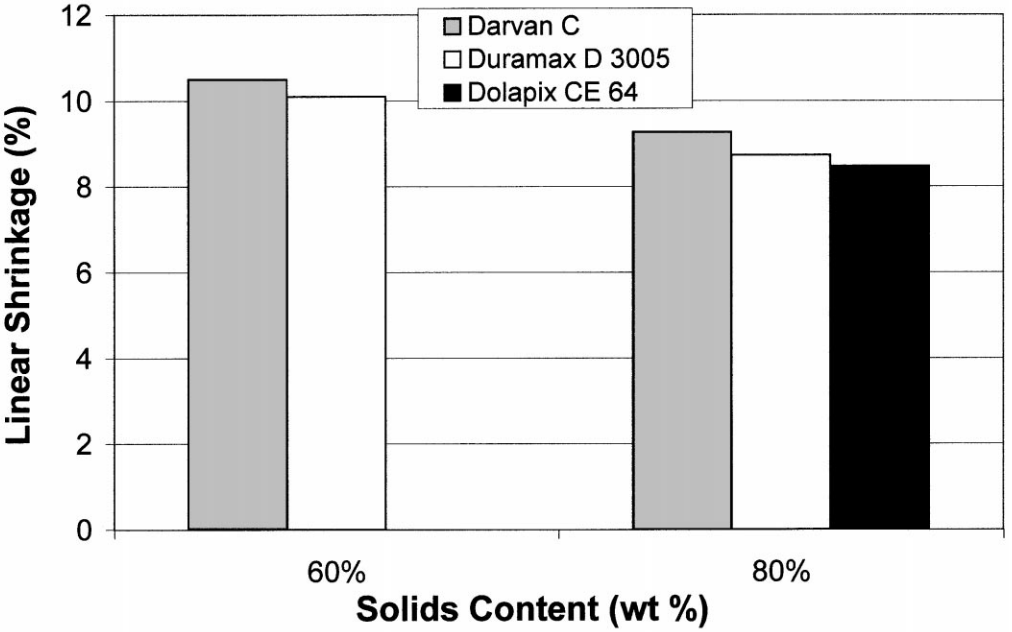

Fig. 8. Effect of slurry solids content, kind of dispersant used and firing temperature on the shrinkage of alumina specimens during firing.

have been frequently used, and various mechanisms have been proposed to explain the experimental observations. Smith and Haber[17,18] used mixtures of coarse (mean particle diameter ~ 6 μm) and fine (mean particle diameter ~ 0.7 μm) alumina particles of various weight ratios and found that a mixture of coarse/fine particles equal to 85/15 exhibited the lowest viscosity, the highest casting rate but in contrast to expectations, the highest green density of cast specimens. They have attributed this fact to the formation of flocs of coarse and fine particles in the slurry, which consolidated during drying and created a bimodal porosity in the green specimens. Ferreira[15] used also a mixture of one fine and one coarse batch of silicon nitride powder in a proportion producing the highest packing density and observed a maximum in this packing density of cast specimens with respect to both dispersant concentration and solids content in the slurry. He attributed the maximum with respect to solids content in the dominance of different driving forces (increasing deposition rate, but decreasing potential for particle rearrangement as the solid loading increases).

In our case, since we did not use mixtures of powders with different mean particle sizes, the consolidation mechanism during slip-casting depends primarily on the slurry viscosity. The reduced potential for particle rearrangement at high solids content, can be counterbalanced by the higher slurry viscosity — obtained with a particular dispersant — which provides for more time for particle rearrangement. Better packing of the powders is achieved not through the use of powder batches of different particle size distributions, but by allowing for more time for particle rearrangement through the use of more viscous — but yet stable — slurries. Among

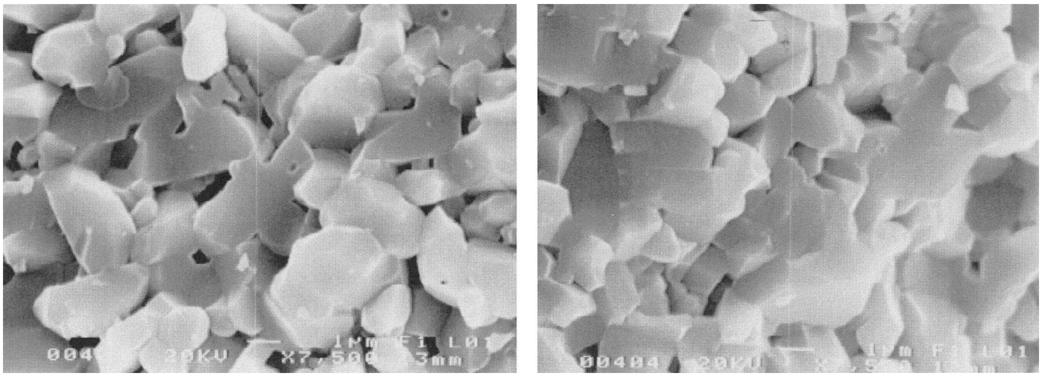

Fig. 9. Morphology of specimens produced from slurries with Duramax D 3005 and fired at 1600°C: (a) slurry solids content 60 wt.% (b) slurry solids content 80 wt.%.

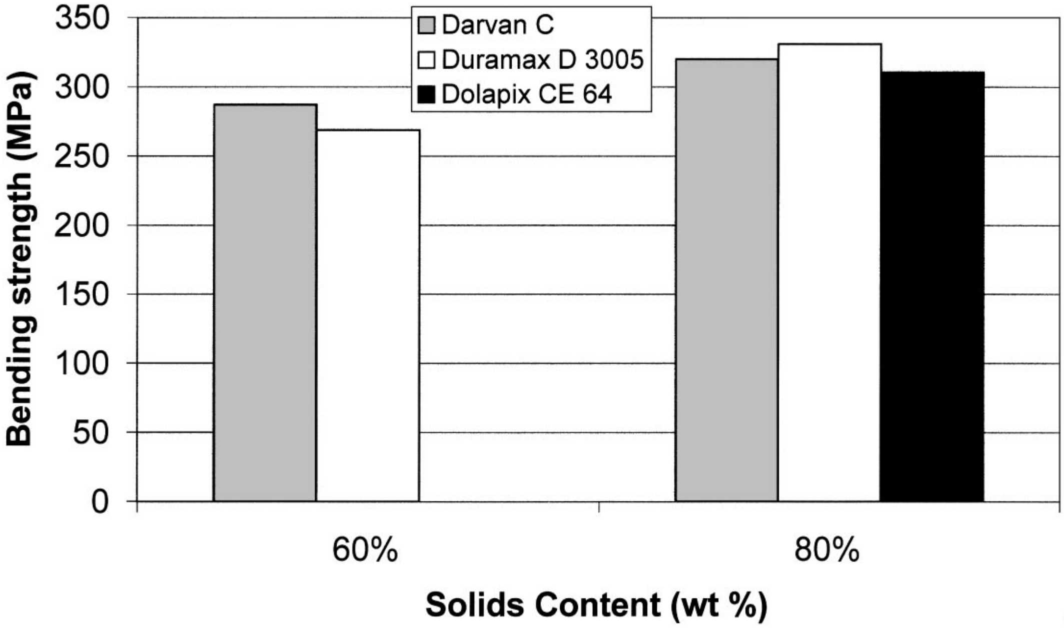

Fig. 10. Effect of slurry solids content and kind of dispersant used on the bending strength of fired alumina specimens.

stable slurries with the same, high solids content, the slower casting rate exhibited from the slurries stabilized with a particular dispersant, gives the time to the particles to re-arrange themselves in a more closely-packed structure and therefore, results in higher green densities.

The linear shrinkage of the specimens after firing at 1600°C (shown in Fig. 8 as a function of slurry solids content and kind of dispersant used), follows as expected, the density variation: it is larger for the slurries of lower solids content which achieve lower green density and least for the slurry with Dolapix CE 64. The effectiveness of Dolapix CE 64 on achieving high green densities on the cast bodies, compared to a purely electrostatic dispersant has been also reported by other research groups.[25] The difference in densities between samples from slurries with high and lower solids content can be further clarified on the SEM images shown in Fig. 9, where specimens from slurries stabilized with Duramax D, 3005 fired at 1600°C are compared. The effect of high solids content in the slurry in reducing the remaining porosity of the cast samples is evident.

Slurry solids content was also shown to give large impact on the mechanical properties of the cast samples. The results of three-point bending tests for specimens (cylindrical rods) from slurries with 60 and 80 wt.% solids content, fired at 1600 °C, are presented in Fig. 10. It is evident that higher slurry solids content leads to higher bending strength, related to the higher density achieved during firing. Among slurries with 80 wt.% solids content, the differences in density at this firing temperature have been eliminated (Fig. 7) and therefore no significant differences in mechanical strength are observed with respect to the kind of dispersant employed.

4. Conclusions

The first part of the work involved the preparation and the stabilization of alumina slurries of high solids content to be used in the production of ceramic objects by slip-casting. For this purpose three commercial dispersants, Darvan C, Dolapix CE 64 and Duramax D 3005 were compared. It occurred that even though all three dispersants are capable of achieving stabilization of high solids content slurries, they exhibit differences with respect to the optimum concentration and the concentration range required for the achievement of minimum slurry viscosity. For instance, for slurries with 80 wt.% alumina, Duramax D 3005 gives the lowest viscosity whereas Darvan C results in low viscosity within a wider dispersant concentration range. The optimum concentration for each dispersant was determined so that stable slurries with low viscosity could be prepared.

The slurries with optimized rheological properties were utilized for the preparation of slip-cast specimens and subsequent properties' evaluation. An increase on the slurry solids content increased the density in the green and in the fired state, decreased the shrinkage during firing and produced specimens with better mechanical properties. The kind of dispersant used, affects slurry viscosity and through that the casting rate and subsequently the green and fired density of the specimens. It was found that for the highest solids content employed (80 wt.%), slurries stabilized with Dolapix CE 64 gave the slowest casting rate, allowing for a denser particle packing during casting and, consequently, produced slip-cast samples with the highest density.

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