

RHEOMETRIC TECHNIQUES APPLIED TO REFRACTORY CERAMIC SUSPENSIONS

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ABSTRACT

Ceramic suspensions present great technological and scientific interest due to their many applications. Suspensions with suitable rheological properties are fundamental in application, processing control and optimization of products and properties. Nevertheless, the use of rheometric techniques to evaluate ceramic suspensions has been scarcely studied when compared to other systems, such as molten polymers. In the present work, the steps of a systematic approach, specifically developed for ceramic suspensions are presented. These rheometric techniques could allow a further understanding regarding these materials and their interactions with additives, such as binders and dispersants. In order to point out their applicability, an alumina-silica sol system was carried out through viscosity, storage modulus, loss modulus and normal force measurements. Such results will be the basis for further studies on ceramic suspensions with silica sol as a binder agent for refractory castables.

INTRODUCTION

Ceramic materials present high melting temperatures and hardness [1], which inhibit their conformation through conventional techniques applied to metallic or polymeric materials. Therefore, ceramic products are usually produced by alternative processes using powders dispersed in a liquid medium [2]. These solid/liquid mixes are called suspensions and are characterized by particles homogeneously distributed in a liquid medium, presenting no significant dissolution with time [3]. Ceramic suspensions present great technological and scientific interests due to their many applications, such as in paints, coatings, abrasives and polishing pastes, lubricants, mortars and concretes. The understanding of rheological properties of suspensions is fundamental in applications, processing control and optimization of products and properties.

Several rheological characterization techniques can be found in the literature. [4] One of the most used is viscosimetry. Viscosity describes the physical property of a fluid to resist to shear-induced flow [4] and this test is usually carried out with capillary or rotational viscosimeters using coaxial cylinder, cone-and-plate or parallel-plate sensor systems. The main applications of viscosimetry include determining the optimum pH values or dispersant content, aiming at the maximum solid concentration of suspensions. [2, 5]

Another rheometric technique is the oscillatory or dynamic test. Samples are subjected to oscillating stresses or strains, applied as a sinusoidal time function. [4] In this technique, samples of viscoelastic fluids and even of soft solids will be mechanically disturbed in a recoverable way and their internal structure will not be ruptured during such a test. It enables us to differentiate between elastic and viscous responses, which are expressed by the storage or elastic modu-

lus (G') and by the loss or viscous modulus (G''). G' indicates that the stress energy is temporally stored during the test, but can be recovered afterwards, whereas G'' indicates that the energy which has been applied to initiate the flow is irreversibly lost and transformed into shear heat. [4] It is usually applied when evaluating the curing process of thermosetting resins such as epoxy, polyester and phenolic resins [6-9] and gelation kinetics of organic and inorganic particles. [10-13]

Normal force measurements can be carried out to characterize viscoelastic properties of a material. It can determine the normal force (F_n) generated in a body as a response of an imposed elastic deformation. A normal force test measures the system's elasticity, such as the oscillatory test, but not necessarily affording the same sort of information [14], as samples are subjected to a stationary flow during normal force measurements and to a nonstationary flow in the oscillatory test.

Nevertheless, the use of these techniques to analyze ceramic suspensions has been scarcely studied in comparison to other systems, such as food and molten polymers. [14-16] Moreover, research concerning ceramic suspensions usually applies rheology as a controlling method for process variables and not as the focus of the study. As ceramic suspensions are complex systems, the traditional rheological characterization methods may not be suitable to analyze their behavior. Therefore, rheometric techniques that allow a further understanding regarding these materials and the interactions among their components are necessary.

In the following sections, the steps of a systemic approach, specifically developed for ceramic suspensions, will be presented. This approach introduces the utilization of oscillatory tests and normal force measurements as quantitative tools for the setting behavior and demolding conditions. In order to highlight its potential in refractory castables, the use of silica sol as a liquid medium and binder for alumina suspensions was evaluated. Silica sol, a stable dispersion of discrete particles of amorphous silica [18], has been pointed out as an alternative binder to calcium aluminate cement in refractory ceramics. With the proper selection of controlled gelling agents, silica is gelled around the refractory particles. After the drying process, it generates a skeleton and provides strength to the green refractory castable [19]. The literature reports many inorganic compounds and organic liquids as gelling agents, but magnesium compounds are the most applied. [18, 20]

In this work, viscosity (η), storage modulus (G'), loss modulus (G'') and normal force (F_n) measurements were carried out. The results obtained will be the basis for further studies on ceramic suspensions with silica sol as a binder agent for refractory castables.

MATERIALS AND TECHNIQUES

The tests were carried out in an alumina (A17NE – Almatris, USA) and silica sol (Bindzil – Nalco) suspension with 56.6 vol% of solids (exceptions are described when required). Citric acid PA (Synth, Brazil), polietileneglicol FS10 (SKW Polymers), polycarboxilate FS20 (SKW Polymers) and sodium polimetacrilate Darvan-7S (R. T. Vanderbilt Company, Inc.) were tested as dispersant agents. Magnesium oxide (98%, Synth, Brazil) was added as a gelling agent. [18]

The suspensions were prepared in a laboratory mixer (Ética Equipamentos Científicos S.A., Brazil) under 40 rpm for 2 minutes. The rheological characterizations of the suspension were carried out in a rotary rheometer RS300 (Thermo Haake, Germany), shown in **Figure 1a**.

RESULTS AND DISCUSSION

The results attained will be presented in two parts. In Part I, the steps of the systemic approach specifically developed for ceramic suspensions are presented. Part II shows the systemic approach developed for ceramic suspensions concerning the role of silica sol as a liquid medium and binder for alumina suspensions.

Part I: Standardization of the rheological evaluation

Ultrasound application

A ceramic suspension can be considered homogeneous when its particles are individualized. The ultrasound application is commonly utilized to break up the particles' aggregates. [10, 21] This treatment leads to the particles' individualization and to superior packing efficiency, allowing higher accuracy when determining the optimum dispersant content.

Preliminary tests were carried out in suspensions containing 1.9 mg/m² of FS10 in order to evaluate the effect of the ultrasound technique in dispersion (Unique UltraSonic-Thornthon Unique). First of all, each sample was subjected to an ultrasonic treatment at different times (0, 1, 1.5, 2 and 3 minutes) and then tested under a shear rate variation from 0 up to 50 s⁻¹ with a coaxial cylinder sensor at 25°C. **Figure 2** illustrates the results of the suspension viscosity for different treatment times.

It can be observed that the longer the time that the suspension was subjected to ultrasound application, the greater the viscosity reduction. This could be interpreted as an improvement in dispersion. Nevertheless, a temperature increase from 8 up to 57°C (depending on the treatment time) was also observed comparing to ultrasound-free suspension. Therefore, despite the dispersing effect, ultrasound

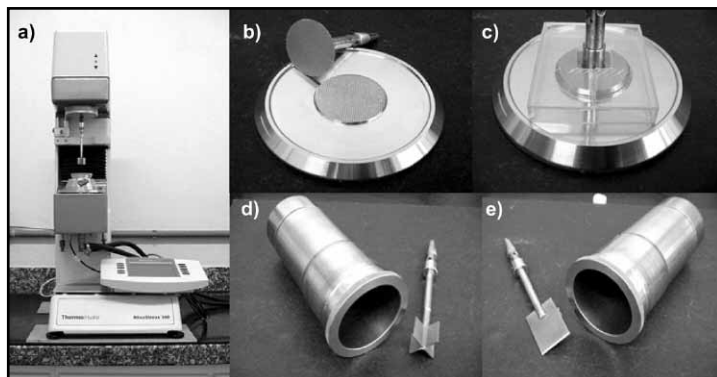


Figure 1. Rheometer and sensors: a) RS300 rheometer, b) serrated parallel-plate, c) serrated parallel-plate with protective device, d) vane sensor and cup test and e) blade-shape sensor and cup test.

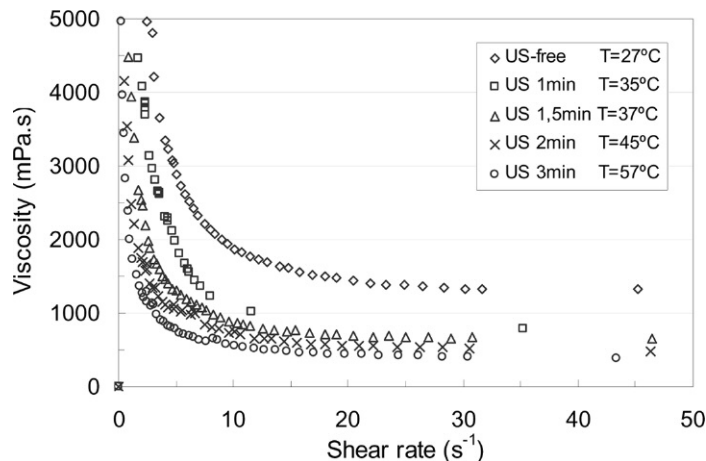


Figure 2. Viscosity as a function of shear rate for alumina-silica sol suspension dispersed with FS10, subjected to different ultrasonification times (T: temperature of the suspension at the end of test).

treatment was not suitable to be applied in silica sol systems because temperature variations can modify their gelling kinetics. As a consequence, the suspensions used in the following tests were not submitted to this dispersion technique.

Measurement system for oscillatory tests

An important concern in ceramic suspension characterization is their susceptibility to the drying that may occur during the test. The liquid removal generally leads to an increase in viscosity, disturbing the movement of the particles. Furthermore, in systems involving gelling reactions, such as those containing silica sol, the drying control acquires greater importance, as gelation denotes the transition from a liquid (sol) to a solid (gel) state without fluid removal [11]. Therefore, the next step was the standardization of the oscillatory tests in order to prevent the suspension drying.

Either parallel-plate or vane sensors (**Figure 1b** and **1d**) can be used in these experiments. [4, 10, 15] The main differences between them are related to their geometry and the consequent contact area with the sample and the surrounding environment. In these tests, an aqueous alumina suspension was used as a reference since no significant reaction is expected to occur throughout the experiments, allowing for an easier detection of drying or moisture absorption.

The oscillatory tests (1 Hz and 1 Pa) were carried out at 50°C in order to select the measurement system which was less susceptible to drying. In the tests performed with the vane, the suspension's surface was covered with a non-polar oil; for those conducted with the parallel-plate, the samples were subjected to different moisture conditions: i) the sample without a protective device (**Figure 1c**) and without water, ii) sample with the device and without water and iii) sample with the device and surrounded by drops of water. **Figure 3** shows the obtained curves.

The significant increase in the storage modulus (G') values indicated that suspensions presented a significant degree of water loss during the tests conducted with the parallel-plate sensor, even with the use of the protective device and water in the surrounding environment. This can be associated to the large area exposed between the plates, which favor the drying. On the other hand, there were no changes in the G' values for the sample tested with the vane sensor with a coating oil layer, showing that drying was suitably avoided.

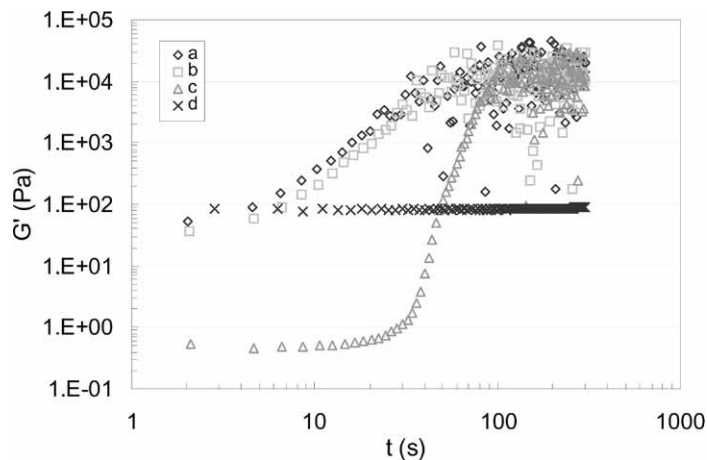


Figure 3. G' as a function of time for aqueous alumina suspension under different experimental settings at 50°C: a) sample without protective device and without water coating, b) sample with the device and without water coating, c) sample with the device and water coating and d) sample with oil coating.

These results showed that the parallel-plate system is greatly susceptible to the water withdrawn, even when the atmosphere surrounding the sample was controlled. The literature reports that certain ceramic suspensions prepared with high vapor pressure compounds (such as alcohols and ketones) can be even more sensitive to the drying effect being measured. [22] This problem was avoided with the use of the vane sensor and a layer of oil coating the suspension's surface. In addition to the drying minimization, as this configuration requires a sample volume greater than the parallel-plate one, the results attained are more representative and less sensitive to minor inhomogeneities of the suspension [15].

Therefore, the vane sensor, with a coating oil layer on the suspension's surface was selected for further use in oscillatory tests.

Standard parameters for oscillatory tests

The equations used in the G' and G'' calculations state that the oscillatory tests must be carried out under a linear viscoelastic regime. It implies that the sample's deformations must occur in a totally recoverable way, with no significant permanent strain, such as cracks or disruption of the sample. [4] After selecting the sensor, the range of frequencies and stresses at which the material, before and after the gelling reaction, fulfills a linear viscoelastic behavior was selected. Frequency (from 0.01 up to 10 Hz) and stress (from 0.01 up to 100 Pa) sweep tests were carried out for the alumina-silica sol suspension (62.5 vol%) with 0.26 mg/m² of citric acid in two conditions: after mixing without MgO addition and after the gelling reaction with 0.6 wt% of MgO. It is necessary to assure that, even after gelling reaction, frequency and stress values are in the linear regime range. **Figure 4** presents the results obtained for the stress sweep test.

Constant G' values in a stress variation (and at constant frequency) or frequency sweep (and at constant stress) indicate that the material is within a linear viscoelastic regime. [4] Based on this affirmation, 1 Hz and 1 Pa were chosen as standard values for the following oscillatory tests in this system.

Normal force test methodology

This test comprises the measurement of the normal force (F_n) generated in a body as a response to an elastic deformation imposed uniaxially. The measurement methodology and the data treatment developed in this work is a novel approach for characterizing ceramic sus-

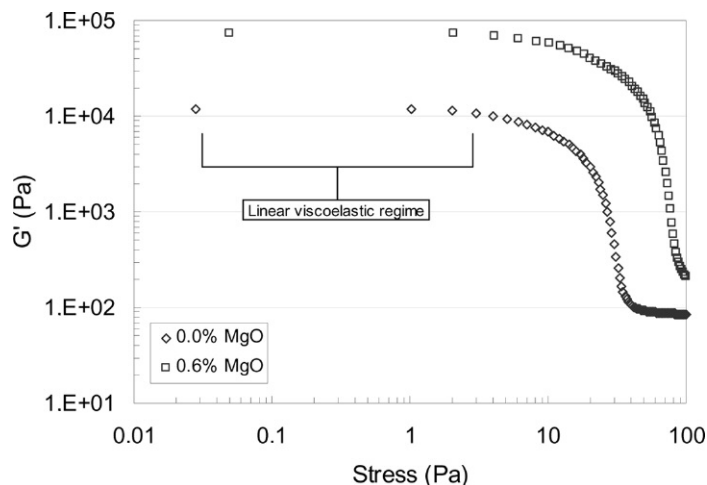


Figure 4. Stress sweep test: storage modulus (G') as a function of applied stress for suspensions with and without MgO.

pensions. It evaluates the sample's response to a blade-shaped sensor penetration at a constant deformation rate (see **Figure 1e**).

In the normal force tests, equal quantities of suspension were poured into several cups. Each sample was covered with oil to avoid drying and kept at a constant temperature in an acclimatized chamber (Vöstch, model 20-20) until the measurement, at a regular time interval (up to 3 h) after the mix. The normal force measurement represents the difficulty that a blade-shaped sensor faces to penetrate the sample's surface at a rate of 1 mm/s for 20 seconds. The sensor velocity was previously set in order to carry out the experiments without damaging the sample. **Figure 5** shows typical normal force results as a function of the collecting time (20 s).

The time derivative of the normal force data, obtained as a response to a constant deformation rate applied in the suspension, supplied the normal force rate. This is a more reliable value than the normal force ones as at the beginning and at the end of the each measurement, the sensor displacement can be affected by the accommodation of the suspension. The results presented are the average rate value as a function of time (3 h) and will be discussed in detail in Part 2.

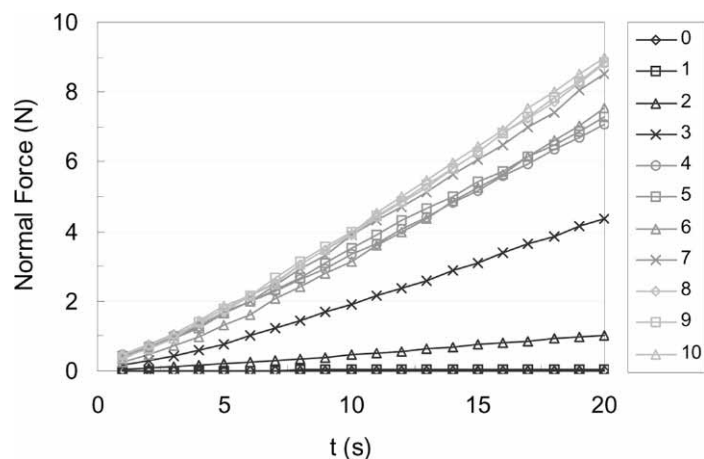


Figure 5. Normal force as a function of time for samples (n°0 to n°10) for alumina-silica sol suspension with 0.3 wt% of MgO.

PART II: EVALUATING SILICA SOL AS A BINDER FOR ALUMINA SUSPENSIONS

Part 2 presents the results obtained using the parameters and testing set conditions defined in Part 1 for the alumina-silica sol system: suspensions without ultrasound treatment, coating oil layer on the sample's surface, the vane sensor and 1 Hz and 1 Pa standard values for oscillatory tests and blade-shaped sensor penetration rate of 1 mm/s for normal force tests.

The literature describes that gel-bonded castable compositions containing silica sol does not need dispersing additives. [19] It is also reported that alumina-silica sol slurries showed a minimum sedimentation volume and reduced viscosity values close to pH 10. In this region, silica sol particles are dispersed and are adsorbed on the surface of large alumina particles, displaying an electrostatic dispersion effect in alumina particles. [5] In order to evaluate these hypotheses, different dispersing agents were tested in the silica sol-alumina suspensions using viscosity measurements (see **Figure 6**).

A reduction in the suspension viscosity was observed for all the dispersants tested. A lower viscosity indicates that a lower stress is necessary to submit the fluid into a constant shear rate. [2] For this particular system, the silica sol dispersing effect can be improved with the use of other dispersing agents. FS20 presented the best result.

The optimized content of each dispersing agent was determined and oscillatory tests were carried out to verify the effect of these additives on the storage and loss moduli.

Comparing the results in **Figure 7**, citric acid and FS20 containing samples behaved similarly to the additive-free one, whereas Darvan-7S and FS10 containing samples showed a different behavior. Darvan-7S increased G' and G'' values of the suspension. This can be attributed to Na^+ releasing in the medium: the sodium ion can be adsorbed at a negative site of a silica particle forming a neutral complex which reduces the overall net repulsion effect, accelerating the coagulating mechanism of silica sol. [18] On the other hand, the FS10 dispersant reduced the initial values of G' and G'' , which were kept constant during the testing time. The effect promoted by FS10 addition in suspension can favor the casting step and it was used in the following investigations.

The influence of MgO content on the alumina-silica sol system was evaluated using oscillatory tests (**Figure 8**).

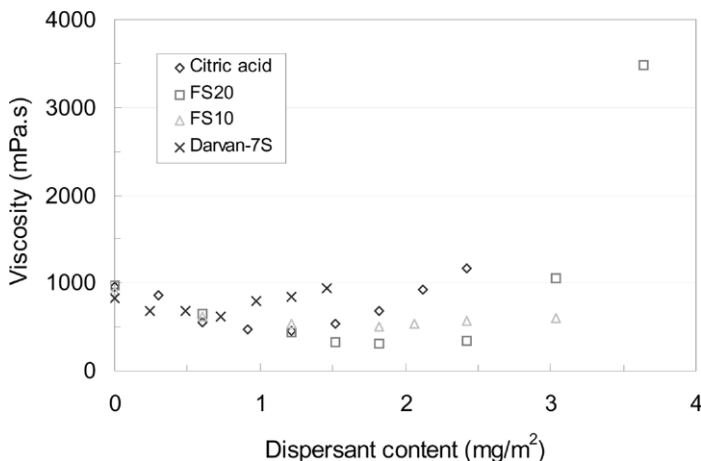


Figure 6. Alumina-silica sol suspension viscosity as a function of dispersant content.

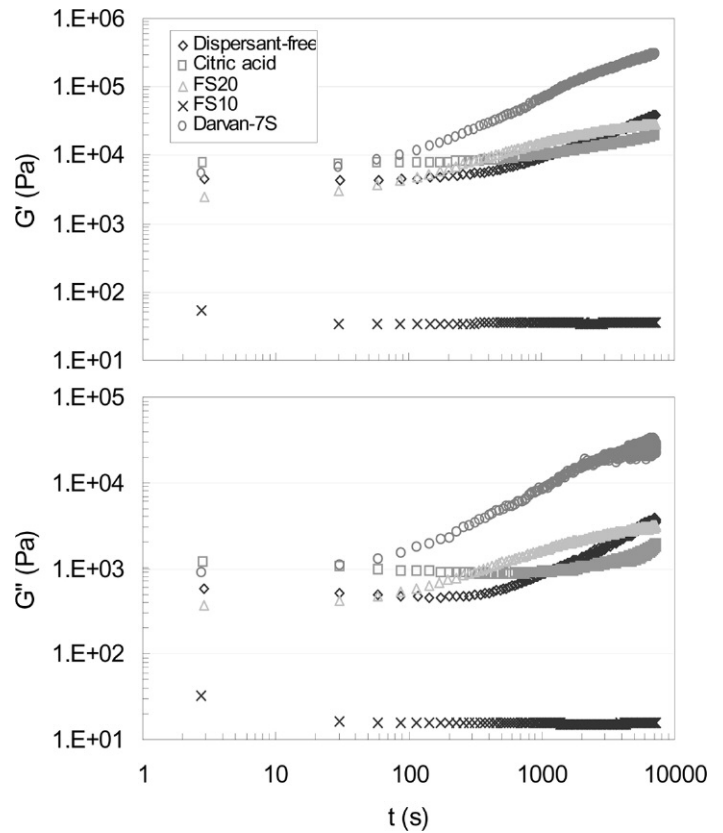


Figure 7. Storage (G') and loss (G'') moduli as a function of time for alumina-silica sol suspensions with different dispersant additives at 25°C.

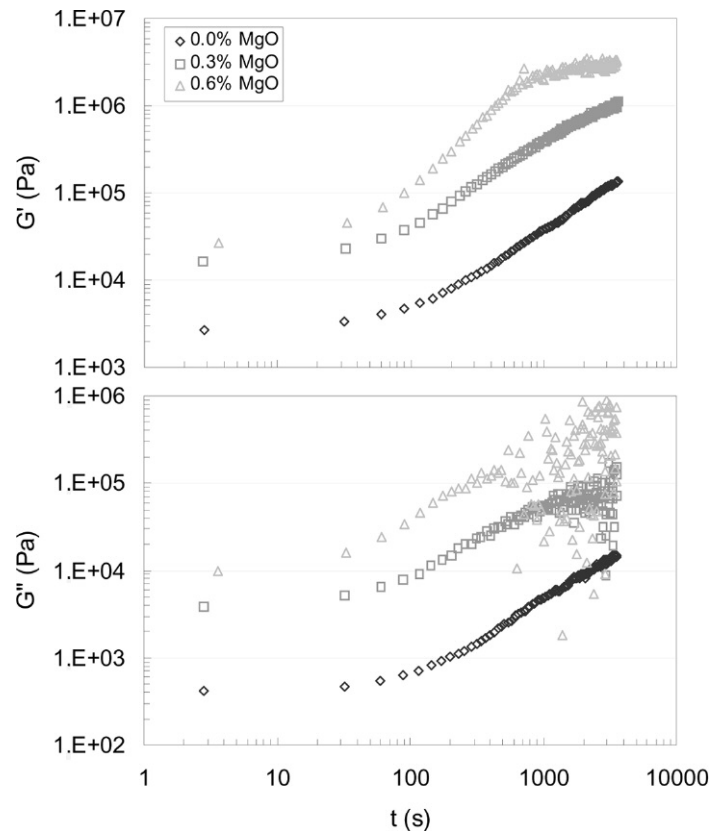


Figure 8. Storage (G') and loss (G'') moduli as a function of time for alumina-silica sol suspensions with different MgO contents at 25°C.

The literature describes different criteria to evaluate the end of the gelling reaction, often named gel time [6], which indicates the transition from a viscous liquid to an elastic solid. After this time, a strong reduction in the material's workability is expected. One of the most used criterion suggests that gel time corresponds to the crossover between G' and G'' curves. However, it was not observed in the studied system. Therefore, another parameter to determine the gel time for these ceramic suspensions was defined, assuming that the end of the gelling reaction occurs at the time that G' and G'' values become constant. In the same way, it was also defined that the time at which the moduli start to increase indicates the beginning of the gelling reaction.

The MgO addition resulted in the increase of G' and G'' initial values and in the decrease of the time required for the moduli to start increasing and reaching constant values ($G' = 10^6$ Pa and $G'' = 10^5$ Pa, approximately). These results showed that the gelling reaction was highly accelerated by adding MgO. As a basic oxide, MgO favors anionic reaction mechanisms on its surface. [23] Consequently, MgO removes hydrogen ions from Si-OH groups on the surface of silica particles, increasing the rate of the formation of siloxane bonds (Si-O-Si) and thus, increasing the gelation rate. The greater the MgO content in the suspension, the more significant these effects are.

The simultaneous G' and G'' increase indicates that the samples become more elastic and that the energy dissipation during deformation was enhanced, compared to the initial condition. In the case of the analyzed suspensions, the loss modulus presented slower increase rates and achieved lower final values than the storage modulus, indicating that this system possesses an elastic component greater than the viscous one.

At the end of the oscillatory tests, it was observed that some samples did not attain enough mechanical strength to be demolded without damage, despite the G' value having been stabilized at a nearly steady value of 10^6 Pa (called G'_{equil}). It means that G'_{equil} is able to indicate the end of the gelling reaction, but it does not provide enough information about the macroscopic consistency of the system. Therefore, in the system studied, oscillatory tests can determine the time that the reaction starts, but are not always suitable to evaluate the time required for demolding and the suspension mechanical strength at early ages (before firing), as pointed out in the literature. [11] These macroscopic aspects were investigated with another parameter, the normal force.

The normal force rate for alumina-silica sol suspensions with different contents of MgO are illustrated in **Figure 9**.

As observed for G' and G'' curves, the higher the amount of MgO added to the suspensions, the shorter the time required to achieve the maximum values. When normal force rate becomes constant, the suspension reached its maximum deformation resistance. After this point, extra gelling time will not bring any benefit. For the studied system, it also means that suspension attained its best demolding condition. Therefore, the time required for normal force stabilization can be used as a more suitable demolding criterion than the maximum G' value (G'_{equil}).

During the testing time, only the suspension with 0.6 wt% of MgO reached a constant normal force rate, after approximately 35 minutes. The normal force rate for the sample with 0.3 wt% of MgO increased, but it did not stabilize under the testing conditions. These results also pointed out clear differences between the gel time and demolding time, indicating that G' and G'' moduli and the normal force rate did

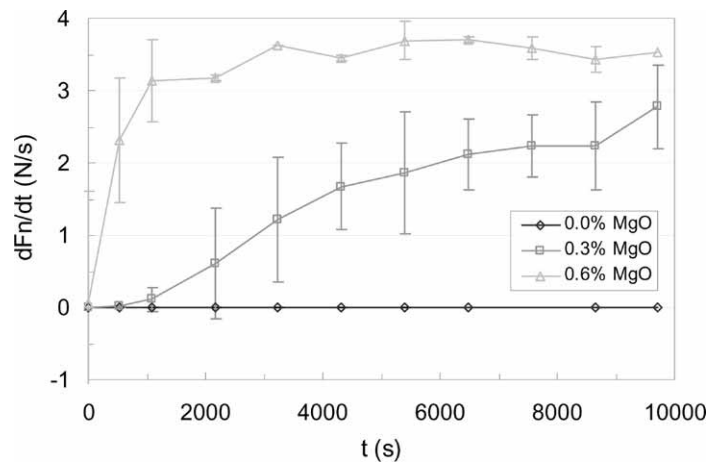


Figure 9. Normal force rate as a function of time for alumina-silica sol suspensions with different MgO contents at 25°C.

not provide the same information. The moduli can be related to the occurrence and kinetics of the gelling reaction, whereas normal force seems to be better related to the mechanical strength development.

CONCLUSIONS


A novel approach for rheological characterization of ceramic suspensions was proposed based on the systemic evaluation of the suspension characteristics, from mixing and molding to the consolidation. Several testing conditions and parameters were carefully set in order to prevent the influence of external variables, such as suspension sedimentation and drying. It was observed that the ultrasound application improves dispersion but can lead to temperature increase. Viscosity measurements indicated that silica sol, without the use of other additives, did not disperse the alumina particles properly. The oscillatory tests showed that suspension drying and the volume of the samples tested are important aspects. The best testing set was attained with the vane sensor and non-polar oil for the suspension surface coating. The G' and G'' curves as a function of time showed that the gelation rate can be modified by the addition of a suitable gelling agent (magnesium oxide). In this study, normal force measurement was adapted to be used in ceramic suspensions. The results obtained complement those attained in the oscillatory tests: whereas G' and G'' moduli behaviors indicate the beginning and kinetics of gelling reaction and are useful to determine the workability time for molding; the normal force better describes the mechanical strength evolution of the sample and can be used to determine the demolding time. Oscillatory and normal force tests have brought new insights regarding gelling mechanism of silica sol in a ceramic system, allowing for a better understanding of its processing. These techniques will also be applied to select shotcrete-setting additives and to further understand the behavior of silica sol containing castable matrices.

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THE 42nd ANNUAL SYMPOSIUM “ADVANCES IN RAW MATERIALS”

The St. Louis Section and the Refractory Ceramics Division of the American Ceramic Society will sponsor the 42nd annual symposium on the theme "Advances in Raw Materials" on March 29-30, 2006, held in St. Louis, Missouri at the Hilton St. Louis Airport Hotel. Dilip Jain of Kyanite Mining and Dana Goski Allied Mineral are the co-program chairs. ASTM C08 will meet at the same hotel on March 29th.

Listed are papers to be presented: “Silica Fume”, J. Scanlon, Globe Metallurgical & G.M. Gapinski, Norchem Concrete Products; “A General Overview of Silicon Carbide Worldwide - With Particular Reference to Refractory Applications; Advantages of Silicon Carbide in Various Refractory Fields; Quality Differences in Silicon Carbide”, G. Wagner, Saint Gobain; “The Advantages of Calcium Aluminate Cement as a Castable Bonding System”, C. Parr, C. Alt & Ch. Wöhrmeyer, Lafarge Aluminates; “Industrial Application Experiences with the Microporous Calcium Hexaluminate Insulating Material SLA-92”, R.P. Racher, Almatis, R. Kockegey-Lorenz & A. Buhr, Almatis GmbH (Germany); “Interchangeability of White Fused Alumina and Tabular Alumina in Low Cement Castable Matrix: Utopia or Reality?”, F. Cantelaube, Alcan Specialty Aluminas (France); “Fused Silica in Refractory”, A.S. Labi, Precision Electro Minerals Co. (PEMCO); “The Rejuvenation of Guyanan Bauxite, a Progress Report”, G. , Cambior; “Economics of Alumina Supply for the Refractory Industry”, P. Ormond, Aluchem; “Chinese Brown Fused Aluminum Oxide and Bauxite-Past and Present”, R.M. Silvestri, Great Lakes Minerals; “Manufactured Aggregate for Concrete and Refractory Applications: The Importance of Controlling the Aggregate-Cement Interface”, W. Carty, Alfred University; “Reactive Spinel Powders for Refractory Applications”, G. Oprea, G. Ye & T. Troczynski, University of British Columbia, and “The Sleeping Giant has Awakened!!”, C.E. Semler, Semler Materials Services (Invited Paper).

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