# SAMPLE PREPARATION AND RHEOLOGY OF CERAMIC SUSPENSIONS

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### ABSTRACT

The present work compares experimental results obtained from common procedures in the laboratory with results from procedures that simulate the preparation process of ceramic suspensions on an industrial scale. The objective was to evaluate the influence of measurement process parameters on the rheological properties of suspensions of ceramic raw materials. The samples analysed presented viscosity variations depending on the measurement process. The results showed that differences in sample preparation procedures cause significant changes in the rheological properties of suspensions. The traditional method of rheological characterization used in the laboratory does not represent the industrial preparation process. The changes in the rheological properties due to suspension preparation can be minimized when sample preparation history is considered.

**KEYWORDS:** sample preparation, rheology, rheometry, viscosity, ceramic suspensions, ceramic slurries, ceramic raw materials.

## INTRODUCTION

The experimental methodology for rheological measurements has a fundamental importance for the characterization of ceramic suspensions, with regard to scientific and technological aspects. On one hand the measurements should accomplish a certain level of reproducibility and precision, on the other hand the measured values should properly represent industrial processing.

The rheological characteristics of slurries influence the quality of ceramic products made by wet grinding processes, which are employed in many processing routes of traditional and advanced ceramics. Usually a deflocculant is added to the slurry to ensure a high solids content, making the drying process economically viable. In order to define the optimum amount of additive, a deflocculation curve is determined by adding increasing amounts of deflocculant to the slurry and measuring the respective viscosity. The suitable amount of additive to be used is usually associated with a certain range of low viscosity in the deflocculation curve.

In this paper, the influence of the measurement process on the rheological behaviour of ceramic suspensions was analysed. Two experimental procedures were compared: a traditional method and an alternative one. The traditional method represents the measurement process usually used in laboratory and the alternative method represents the measurement process proposed for the rheological characterization of ceramic raw materials simulating the industrial processing.

#### EXPERIMENTAL PROCEDURE

Raw materials of different characteristics were selected to compose a typical mixture of a clay-based slurry for industrial applications, e.g., for the production of ceramic tiles.

The characterization of the powder raw materials was accomplished by X-ray fluorescence (XRF, Philips, model PW2400) and the mineralogical analysis by X-ray diffractometry X (XRD, Philips, model Xpert), using CuK $\alpha$  radiation and Ni filter. Quantification of phases was performed by rational analysis, according to the method of Coelho et al.<sup>[1]</sup>. Particle size analysis was carried out by laser diffraction (Cilas, model 1064L).

The raw materials were initially passed through a 8 mm sieve in order to guarantee good homogeneity. Moisture of the samples was determined after heating at 110°C to constant mass. The samples were wet milled in a ball mill till a residue from 4 to 5% on a 200-mesh sieve was reached. The grinding media consisted of 1200 g of 20 mm diameter alumina spheres.

The amounts of dehydrated sodium silicate (Manchester, powder, 27% Na<sub>2</sub>O) used as a deflocculant were calculated on a solid dry mass basis. The viscosity measurements were accomplished with a rotational rheometer (Bohlin, model V88BV) with concentric cylinder geometry. Shear rates of 40 s<sup>-1</sup> were used, which correspond to the magnitude of shear rates in industrial mixing and milling processes.

#### **RESULTS AND DISCUSSION**

Based on the chemical and mineralogical analyses, the raw materials A, B and C can be characterized as clays. Clays A and B present relatively high amounts of sintering aid elements (Na, K, Ca). Clay B has a significant amount of illite, what makes it a good plastic clay. Raw material C is characterized as a kaolin with a high silica amount. Raw material D presents approximately 24% MgO due to its major talc phase. Raw materials E and F are fluxing clay minerals consisting of potassium feldspar with a high amount of iron. Raw material F also presents good plasticity, due to the presence of 34% illite.

The rheological behaviour of the raw materials exhibited variations when sample preparation procedures were compared. In the traditional and alternative sample preparations, the deflocculation curves of raw materials A, B, D and F are quite distant from each other. Raw materials C and E presented narrower distances between the curves. These two samples contained the highest amount of the non-plastic phases quartz and feldspar, 56% and 64%, respectively. Thus, the sample preparation procedure affected the plastic clayey materials more markedly. Clay minerals typically have adsorbed ions, which migrate to the liquid phase when the clay particles are suspended in water<sup>[2,3]</sup>. This leads the surfaces of clay particles to be charged electrically, contributing to the deflocculation mechanism by electrostatic repulsion<sup>[4]</sup>.

At the same deflocculant concentration, e.g. 0.05%, raw material A presented much smaller viscosity values after traditional sample preparation compared with the alternative method. This material presented the highest value for loss on ignition and was the only one with calcite and plagioclase phases. Apparently some flocculating constituent of this sample was washed out by the traditional procedure, making the action of the sodium silicate easier.

For the amount of deflocculant corresponding to the point of lowest viscosity, raw materials C and E presented quite close viscosity values. The minimum viscosity obtained for the deflocculation curves of the raw material D (0.1 Pa·s) corresponded to 0.02% deflocculant for the traditional and 0.15% for the alternative method. Raw material F, also at a viscosity of 0.1 Pa·s, presented an amount of 0.1% deflocculant for the traditional method and 0.25% for the alternative method. No raw material presented overdeflocculation across the range of additive additions.

Particle size distribution was measured in the deflocculated condition. The distribution curves presented the same form. Only raw materials E and F presented a slightly different mean particle size. Even the samples prepared traditionally, which have undergone two grinding steps, had no significant modification in particle size distribution.

All the raw materials analysed presented remarkably different deflocculation behaviour according to the sample preparation method. The raw materials prepared by the traditional method presented significantly lower viscosity values compared with the samples prepared by the alternative method. This fact is also observed in industrial processes.

The alternative method of sample preparation for viscosity measurements proposed here seems to better accomplish the process that occurs in ceramic industries. Complementary experiments using the combination of constituents in a ceramic mass composition should be performed to confirm this tendency.

## REFERENCES

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