Rheological Behavior of Alumina Ceramic Pastes

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Abstract:
The rheological behavior of ceramic oxide hydroxide alumina pastes with high solid loading is investigated. In order to enable an adequate and experimentally rheological characterization, the measurements are carried out with a Rheostress viscometer under isothermal conditions. Various compositions of a commercial AlOOH powder and binder mixture are investigated. We discuss the variation of loss modulus *G'*, storage modulus *G''*, apparent and complex viscosities η, η^* as function of frequency and shear rate. The solid phase used here is the boehmite; the most important precursor for the γ-Al_2O_3 phase for several applications such as catalysts or functional layers of ceramics. Solid phase compositions used are justified by the applications of boehmite in the manufacturing of catalytic materials. A transition zone that appears at a concentration of 55 %wt of the solid phase (Pural) and at which the rheological behavior changes from viscoelastic to elastic is observed. This transition is of a importance as far as ceramic manufacturing is concerned.

Zusammenfassung:
Das rheologische Verhalten von Aluminiumhydroxid Keramikpasten (mit hohem Feststoffgehalt) wurde untersucht. Um eine angemessene und experimentell rheologische Charakterisierung zu realisieren, wurden die Messungen mit einem Rheostress Viskosimeter unter isothermen Bedingungen durchgeführt. Verschiedene Zusammensetzungen von einem kommerziellen AlOOH Pulver-Binder Gemisch wurden untersucht. Die beide Module *G'* und *G''* sowie die Viskositäten η und η^* wurden sowohl oszillierenden als unter Scherung vermessen. Die Feststoffphase, Boehmite, ist das wichtigste Vorprodukt für γ-Al_2O_3 Produkte für verschiedene Anwendungen wie zum Beispiel die Herstellung von Katalysatoren oder funktionelle Keramikbeschichtungen. Die Zusammensetzungen der Feststoffphasen sind durch die Anwendungen von Boehmite bei der Herstellung von katalytischen Materialien definiert. Ab einer Zusammensetzung von 55 %wt der festen Phase (Pural) wurde beobachtet, dass das rheologische Verhalten von viskoelastischen zu elastischen wechselt. Dieser Übergang ist von großer Bedeutung bezüglich der Keramikherstellung.

Résumé:
Le comportement rhéologique de pâtes de céramiques (suspensions hautement concentrées) á base d'hydroxyde d'aluminium a été étudié. Dans le but de permettre une caractérisation expérimentale adéquate, les mesures ont été effectuées en utilisant un rhéomètre (Rheostress viscometer) sous des conditions isothermes. Différentes compositions de mélanges de poudre d'hydroxyde d'aluminium commercial et de liants ont été étudiées. Les modules *G'* et *G''* ainsi que les viscosités apparente et complexe (η et η^*) ont été mis en évidence aussi bien en mode statique que dynamique. Les variations de ces paramètres ont été discutées. La phase solide, utilisée dans ce travail, est la Boehmite. Elle joue un rôle très important en tant que précurseur de la phase γ-Al_2O_3 dans plusieurs applications telles que les catalyseurs et les couches fonctionnelles des céramiques. Les taux de phase solide utilisés dans cette étude sont justifiés par ceux utilisés dans les fabrications des matériaux catalytiques à base de Boehmite. Une transition de comportement rhéologique a été observée à partir de la composition 55% en poids, celle-ci met en évidence un changement du comportement visqueux vers celui élastique. Cette transition est d'une grande importance s'agissant de la fabrication des céramiques.

Key words: alumina, paste, Herschel-Bulkley model

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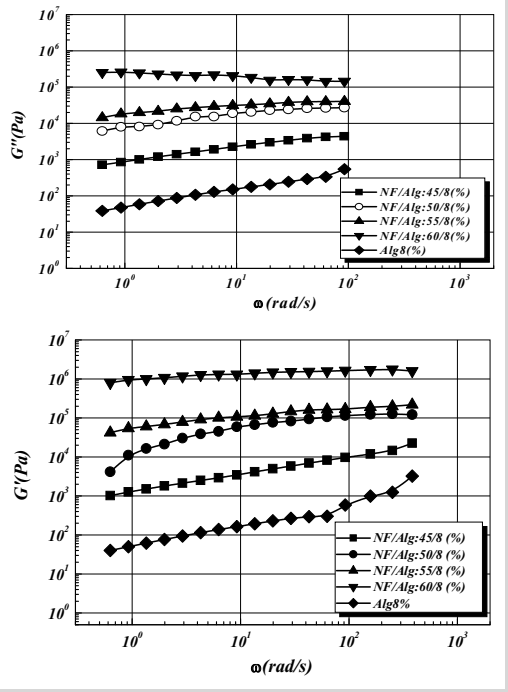

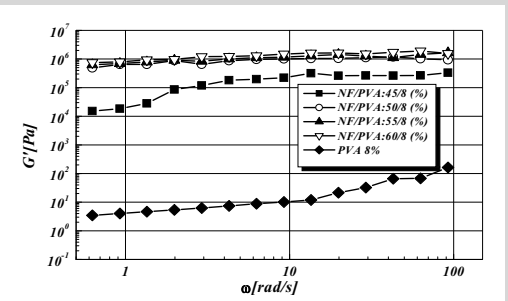

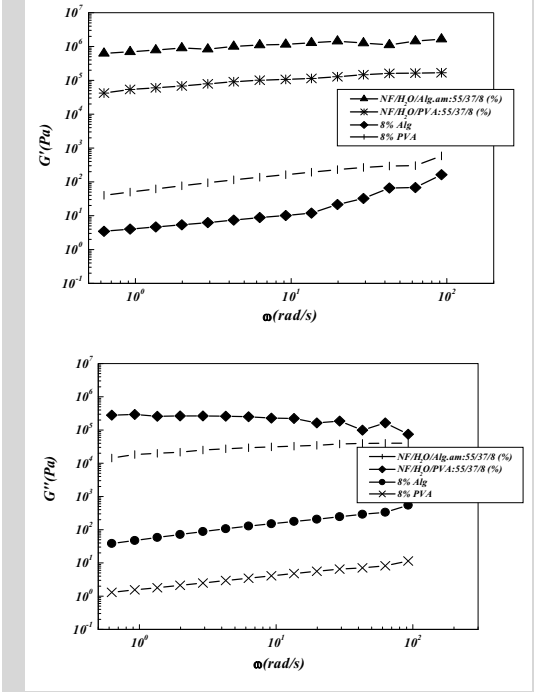

Figure 5 (left above): Effect of various concentrations of Ammonium alginate on variations of G'' (a) and G' (b).

Figure 6 (left below): Effect of various concentrations of Polyvinyl alcohol on variations of G''.

Figure 7 (right): Effect of fluid phases on the variations of G' (a) and G'' (b) for a Pural NF (55 %) suspension.

makes possible to delimit the viscoelastic domain from that of the purely elastic one.

The effect of the second binder, polyvinyl alcohol is illustrated in Figure 6. It highlights the difference from the viscosity point of view between the two binders. It is to be noted that the transition in the behavior observed for suspensions based on ammonium alginate also appears for those containing PVA with a certain convergence of curves from 50 %wt in the solid phase. This can emphasize the effect of the phenomenon of particle compacting (densification) in the presence of PVA as a binder, which reaches a limiting value beyond which the viscosity of the suspension remains constant.

Figure 7 presents the comparison of the evolution of *G'* and *G''* components as a function of frequency for the two kinds of organic binders. The alumina suspensions are dominated by a viscous behavior in the presence of ammonium alginate whereas the elastic behavior is predominant with the PVA. The effect of the nature of the solid phase is shown on Figure 7. The variations at the same time of *G'* and *G''* are much more comparable in the case of the ammonium alginate than in the case of the PVA. If we take in consideration the case where this difference is pronounced, initially, we see that the nature of the solid phase influences much more the flow behavior. This effect is much more significant in the field of shearing (long times) domain.

4 STEADY SHEAR

The effect of shear rate on the shear stress of concentrated suspensions will depend primarily on the particle-particle interactions but to a less extent on the deformability of the particles. The experimental data obtained from the flow curves for different concentrations of the solid phase were correlated using the Herschel-Bulkley equation, which fitted rheological curves having a non-linear plastic trend, to calculate the rheological parameters [8 – 11].

$$\tau = \tau_o + K\dot{\gamma}^n \qquad (1)$$

Where τ is the shear stress (in Pascal), τ_o the yield stress point (in Pascal), K the consistency (in Pa·s), $\dot{\gamma}$ the shear rate (in s^{-1}) and n the flow index. The evolution of the shear stress τ for different suspensions is illustrated on Figure 8. The analysis of these rheogramms and the rheological parameters calculated using the Herschel-Bulkley equation showed that all the suspensions have a behavior similar to that of a Casson fluid (Bingham) [9, 12]. The shear stress of these systems increased with increasing shear rates, but this dependency was not linear. The calculated flow indices ($n < 1$) were in agreement.

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Figure 8 (left):
Effect of the solid phase on G' with a Pural NF/H_2O/Ammonium Alginate (55/37/8) suspension.

Figure 9:
Effect of various concentrations of Pural NF with 8 % Ammonium alginate on flow curves.

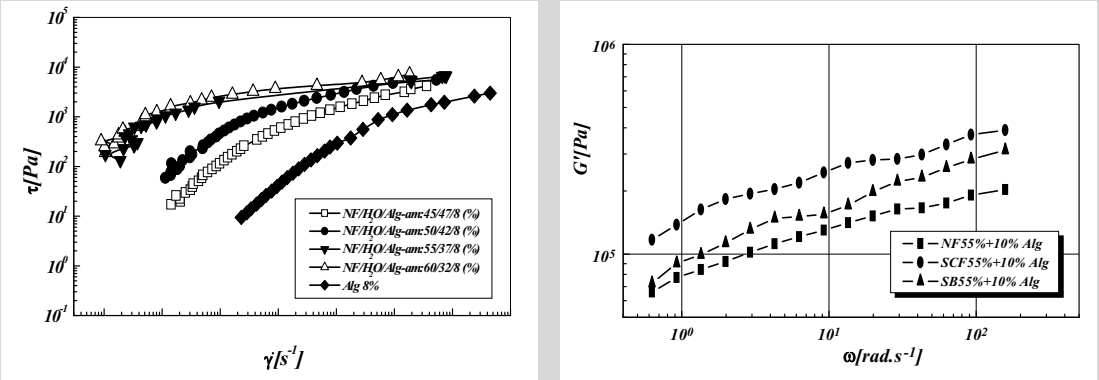

Figure 9 shows the evolution of the storage modulus as a function of frequency for the different kinds of Purals. If the trend seems to be in agreement with respect to particle size for both NF and SB Purals, it is certainly not the case for SCF Pural which exhibits a dominant effect due not only to the size of particles but also to their form which is somewhat heterogeneous in the case of Pural SCF in addition to the effect of agglomerates which can also reverse the trends.

4 CONCLUSION

The rheological behavior of the ceramics pastes in both oscillatory and steady states enabled to value the degree of dependency of the pastes compared to the concentrations of the solid phase and that of the organic binder. A transition zone in the rheological behavior which changes from viscoelastic to elastic was observed. This transition which appears starting from a concentration of 55 %wt of the solid phase (Pural) is of a great interest as far as ceramic manufacturing is concerned. In general the phenomenon which is related to the type of the organic binder has been put in evidence and was predominant with suspensions containing PVA where one notes that starting from a certain composition (50%) the storage modulus curves get superposed. The two kinds of organic binders exhibit viscoelastic behaviors. Our study provides some understanding of the transition phenomenon observed in the rheological behavior of highly concentrated suspensions which is very useful for some industrial applications such as the use of ceramic as honeycomb-shaped as a catalyst support.

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REFERENCES

[1] Sarraf H: High performance ceramics by advanced colloidal processing, Appl. Rheol. 19 (2009) 119-120.
[2] Kaldasch J, Senge B, Laven J: Shear thickening in electrically stabilized non-aqueous colloidal suspensions, Appl. Rheol. 19 (2009) 23493.
[3] Carty WM: Suspension preparation and rheology, Introduction to Ceramic Powder Processing, NYS College of Ceramics, Alfred (1998) 1-35.
[4] Chia-Yi Y, Wan YS, Wei-Heng SJ: Gelation, consolidation, and rheological properties, Am. Ceram. Soc. 83 (2000) 1879-1884.
[5] Götz J, Kreibich W, Peciar M: Mortar physical properties evolution in extrusion flow, Rheol. Acta. 41 (2002) 134-143.
[6] Carreau J.P, De Kee D.CR, Chhabra R.P: Rheology of Polymeric Systems, Hanser (1997) 163.
[7] Fourati N, Léger D, Fakhfakh Z: Influence of physio-chemical parameters on rheological properties of concentarted aqueous kaolin suspensions, Les Cahiers de Rhéologie. XVI (2000) 17-26.
[8] Andreola F, Castellini E, Romagnoli M: Release of ions from kaolinite dispersed in deflocculant solutions, Am. Ceram. Soc. Bull. 81 (2002) 53-56.
[9] Quemada D: Aging, rejuvenation, and thixotropy in complex fluids: Time-dependence of the viscosity at rest and under constant shear rate or shear stress, Appl. Rheol. 18 (2008) 53298.
[10] Masalova I, Malkin AY, Foudazi R: Yield stress as measured in steady shearing and in oscillations, Appl. Rheol. 18 (2008) 44790.
[11] Kelessidis V, Maglione R: Shear rate corrections for Herschel-Bulkley fluids in Couette geometry, Appl. Rheol. 18 (2008) 34482.
[12] Feys D, Verhoeven R, De Schutter G: Extension of the Poiseuille formula for shear-thickening materials and application to Self-Compacting Concrete, Appl. Rheol. 18 (2008) 62705.
[13] Estelle, Lanos C, Perrot A, Amziane S: Processing the vane shear flow data from Couette analogy, Appl. Rheol. 18 (2008) 34037.

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