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STABILIZATION OF HIGHLY CONCENTRATED ALUMINA SUSPENSIONS WITH DIFFERENT DISPERSANTS

Summary

Slip casting is a widely used colloidal technique for consolidation of ceramic powders from a suspension. The colloidal approach ensures homogeneous microstructure of a green body, thereby reducing post-sintering machining and production costs. The process consists of several steps. The first step is to mix the ceramic powder with a liquid, usually water, where the stabilization of the new obtained suspension with different dispersants follows. When an optimum dispersion of particles is achieved, resulting in low slip viscosity, ceramic slurry can finally be poured into a porous mould, usually a gypsum one.

The influence of different dispersants on rheological properties of highly concentrated alumina (Al_2O_3) suspensions was investigated. The used dispersants were: ammonium polymethacrylate water solution (Darvan C-N), 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt (Tiron) and citric acid. The amount of dispersants was varied from 0.1-1.25 wt. % in order to determine the optimum. Rheological properties of the prepared suspensions were determined by measuring the apparent viscosity at different shear rates. The obtained optimal amount was 0.75 wt. % for Darvan, 0.1 wt. % for Tiron and 0.3 wt. % for citric acid. The obtained results show that the most effective dispersant is 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt, given that only 0.1 wt. % was required to stabilize the 70 wt. % Al_2O_3 suspension.

Key words: *alumina, suspensions, slip casting, stabilization*

1. Introduction

Alumina powder (Al_2O_3) is nowadays one of the most used and investigated ceramic materials due to its various satisfying properties and thus a wide range of industrial applications, such as communication and information technology, electric components, corrosion and wear protection, automobile and spacecraft industry, dental and orthopaedic implants, and environmental industries [1].

To achieve improved mechanical, chemical and wear properties, it is essential to produce green bodies with adequate homogeneity, thus their forming process is an important milestone in manufacturing of ceramics [2]. The first step in the ceramic manufacturing process is the preparation of stable concentrated aqueous suspensions of the chosen ceramic powder. Ceramic powders are usually sub-micron or nano-sized with a large specific surface area, which reduces the sintering temperature. However, a high solid loading with such small particles can cause an increase in viscosity due to high particle-to-particle interactions, which could cause agglomeration and subsequently difficulties in casting and mould filling [3]. Such prepared green bodies may turn out to have low density and inhomogeneous microstructure, hence poor properties [4].

Slip casting is a colloidal technique used for consolidation of ceramic powder from an aqueous suspension. The slip i.e. suspension is poured into a porous mould which withdraws water from the slip producing a green body ready for sintering [5]. Slip casting is a simple and reliable procedure adequate for manufacturing monolithic and composite ceramics. The technique itself is very flexible and cost-effective. However, it requires an adequate understanding of colloidal suspensions and their behaviour [6]. Colloidal techniques may help in the control of the forces between particles and thereby reduce formation of agglomerates, facilitate slip casting behaviour, consequently avoiding coarsening of the sintered microstructure and thus improving performance reliability [7]. To achieve lower viscosity on the one hand, but higher green and sintered density on the other, various additives are added to suspensions, such as dispersants and binders.

Dispersants stabilize ceramic particles in the suspension electrostatically, sterically or electrosterically [4, 8]. Electrostatic stabilization is based on the formation of an electrical double layer. New ionic groups, which adsorb on the surface of the ceramic particles, are added to the suspension, forming a charged layer. An equal number of counterions with the opposite charge surround the particles to maintain their electroneutrality and give rise to overall charge and form neutral double layers. These double layers are mutually repulsed, providing the suspension stability. Steric stabilization is achieved by adding macromolecules which attach themselves to the surface of the particles creating an adsorbed layer, hence stabilizing the suspension. However, these macromolecules can cause deformations in the ceramic material during sintering. The most commonly used stabilization method is electrosteric stabilization, a combination of the two previously mentioned stabilization methods [7]. Electrosteric dispersants are usually polyelectrolytes which attach themselves to the surface of particles, creating enough potential difference to cause repulsion forces between particles. Polyelectrolytes have a greater flexibility for processing multiphase systems and higher consolidated density for slip casting. They ensure also a better control of the flocculation state and thixotropy and are overall more stable in comparison to inorganic dispersants [8].

A wide range of different dispersants have been investigated. For example, ammonium polymethacrylate (“Darvan C”) [10, 11, 12, 15], 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt (“Tiron”) [13, 15] and triammonium salt of aurintricarboxylic acid (“Aluminon”) [15] were employed as aqueous ceramic stabilizing agents for commercial alumina. Studies show that the surface charge of the alumina powder becomes more negative in the presence of Darvan C leading to a well stabilized suspension. All three dispersants: Darvan, Tiron and Aluminon are capable of producing well stabilized alumina suspensions. However, viscosities are slightly higher in the presence of Darvan C than in the presence of Tiron and Aluminon.

Chou and Lee studied rheological properties of concentrated alumina slurries as functions of quantities of two different dispersants: sodium pyrophosphate and diammonium hydrogen citrate [14]. Both dispersants successfully deflocculated concentrated alumina suspensions under optimum conditions (an optimum usage of each dispersant). Other polyelectrolyte dispersants, such as ammonium polyacrylate ("Seruna D-305") [9], carbonic acid salt ("Dolapix CE 64") [12, 16, 17], polycarbonic acid salt ("Dolapix PC 33") and carbonic acid ester ("Dolapix ET 85") [17] were used to stabilize aqueous alumina suspensions. As the coverage with adsorbed dispersant Seruna increases, the zeta potential increases accordingly, leading to a well stabilized suspension. It was found that slurries stabilized with Dolapix CE64 gave the slowest casting rate (i.e. slowest removal of water) and therefore samples with the highest density. To achieve stability with polycarbonic acid salt, high contents were required, whereas in the case of carbonic acid ester, the low content resulted in a too high viscosity.

Organic dispersants with different molecular structure based on a benzene ring substitution with (-OH) and (-COOH) functional groups have been examined on the alumina surface [18] as well as poly(maleic acid), PMA [19]. The interaction energy between dispersant and alumina surface was found to increase in terms of the number and position of the functional groups. PMA was successfully used as an anionic dispersant for the preparation of aqueous alumina slurry. Lately, there has been a growing need for more environmentally friendly dispersants. Studies were carried out with citric acid [20] and ascorbic acid [21]. Citric acid shows a high affinity to the alumina surface and thus results in the formation of a negative charge on the alumina particles. Ascorbic acid successfully decreased the suspension viscosity which is directly related to the adsorption on the alumina surface. Both dispersants are easily available, inexpensive and non-toxic, which makes them adequate for a more environmentally friendly slip casting process of alumina ceramics.

A flow curve or rheogram is used to describe rheological properties of fluids. Rheograms are constructed by plotting the following rheological data: viscosity versus shear rate, and shear stress versus shear rate. A flow model may be considered to be a mathematical equation that can describe the rheological data and that provides a convenient and concise manner of describing the data. A number of models have been developed in order to describe non-Newtonian systems. The most frequently applied mathematical models for the characterization of flow properties of ceramic suspensions are the power law, the Herschel-Bulkley and the Bingham model [3, 6]. These models are effectively used to explain, characterize, and predict the flow and pseudo-plastic behaviour of various systems, such as highly concentrated alumina suspensions, and can be expressed as [3, 6]:

Power law model: $\tau = k\gamma^n$ (1)

Herschel-Bulkley model: $\tau = \tau_0 + k\gamma^n$ (2)

Bingham model: $\tau = \tau_0 + \rho\gamma$ (3)

where:

τ (Pa) is the shear stress,

k is the consistency coefficient,

γ (s^{-1}) is the shear rate,

n is the flow index or the shear thinning constant or the shear rate exponent,

τ_0(Pa) is the yield stress,

ρ (Pa s) is the plastic viscosity.

In this work, rheological measurements were conducted to investigate the influence of three different dispersants (i.e. ammonium polymethacrylate water solution (Darvan C-N), 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt monohydrate - Tiron and citric acid monohydrate) on the stabilization of high concentrated alumina suspensions. The selection of dispersants was based on their different chemical composition. Both Darvan and Tiron are commercially available polyelectrolyte dispersants. However, Darvan is an ammonium salt of polymethacrylic acid, while Tiron is a disodium salt of benzenedisulfonic acid. Citric acid is a naturally occurring tricarboxylic acid and a representative of environmentally friendly dispersants.

2. Materials and methods

2.1 Suspension preparation

Alumina suspensions were prepared with 70 wt. % of alumina powder. High-purity Al_2O_3 powder was used, with the average particle size of 300-400 nm (Alcan Chemicals, USA). Three different dispersants were used: Darvan C-N, which is an ammonium polymethacrylate water solution (Vanderbilt Chemicals, LLC, USA), 4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt monohydrate (Sigma-Aldrich Chemie GmbH, Germany) and citric acid monohydrate, >99.7 % purity (VWR Chemicals, BDH Prolabo, Belgium) to stabilize highly concentrated alumina suspensions.

Different amounts (Table 1) of dispersants were mixed with deionised water and added into the grinding jar of the planetary ball mill, after which 70 wt. % of dry monolithic alumina powder was added into the grinding jar. Ten alumina balls were used for the mixture homogenization, which lasted for 90 minutes at a speed of 300 rpm. Alumina balls were separated from the suspension after the homogenization using a sieve. The suspension underwent an ultrasonic treatment for 15 min in an ultrasonic bath – BRANSONIC 220 (Branson Ultrasonics Corp., USA) to remove air bubbles and achieve better homogeneity. After the homogenization, the pH-value was measured. For each dispersant the pH-values were around 6-8 (Table 2).

Table 1 Composition of prepared suspensions

Dispersant		Dry powder content (wt. %)	Water content (wt. %)	*Dispersant content (wt. %)
1	Darvan C-N (an ammonium polymethacrylate water solution)	70	30	0.4 - 1.2
2	Tiron (4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt monohydrate)	70	30	0.1 - 1.25
3	Citric acid monohydrate	70	30	0.1 - 0.6

*weight percentage based on the amount of alumina dry powder

2.2 Determination of rheological properties

Rheological properties were determined using a rotational viscometer DV-III Ultra (Brookfield Engineering Laboratories, Inc., USA) in a small sample chamber with a spindle SC4-18. Pre-shearing lasted for 2 min at a shear rate of 100 s^{-1}. The shear rate was gradually increased from 0.1 to 180 s^{-1}, and then reduced back to 0.1 s^{-1}. The shear rate increase/decrease interval was divided into 50 equal time frames, each lasting for 3 seconds. Rheological measurements were conducted just before each shear rate change.

Temperature was kept constant at 25±1 °C using a thermostatic bath Lauda EcoRE 415 (LAUDA-Brinkmann, LP, USA). Flow curves were recorded for each dispersant type and each dispersant concentration.

Three mathematical models were used to describe the rheological properties: the power law, the Herschel-Bulkley model and the Bingham plastic model.

3. Results and discussion

3.1 Viscosity of highly concentrated alumina (Al_2O_3) suspensions

The aim of this study was to investigate the influence of different dispersants on the stability of highly concentrated alumina suspensions. The prepared suspensions contained 70 wt. % of pure alumina powder, 30 wt. % of deionised water and the concentrations of dispersants varied from 0.1 – 1.25 wt. % of the dry powder weight (Table 2). The suspension stability must be well achieved in order to establish a complete control over suspension rheological properties. Viscosity measurements and sedimentation tests are often used for the suspension stability estimation. In this paper, suspension stability was estimated by the measurements of viscosity for all prepared suspensions at two shear rates, γ, 50 and 100 s^{-1}. The obtained values are listed in Table 2.

Table 2 Measured viscosity of all prepared suspensions (dispersant 1: Darvan C-N, dispersant 2: Tiron, dispersant 3: citric acid). Optimal amounts are shown in bold.

Dispersant	*wt. %	pH	η (mPa s)	
			$\gamma = 50\ s^{-1}$	$\gamma = 100\ s^{-1}$
1	0.4	8.72	12.47	10.82
	0.5	8.75	27.02	22.23
	0.6	8.57	13.62	13.17
	0.75	**8.74**	**8.85**	**8.42**
	1.0	8.61	10.93	9.50
	1.2	8.58	15.09	12.33
2	**0.1**	**8.76**	**6.85**	**6.63**
	0.25	7.89	8.93	7.54
	0.5	6.50	18.55	13.41
	0.75	6.20	32.94	22.79
	1.0	6.04	37.25	25.11
	1.25	6.16	30.56	21.08
3	0.1	7.93	12.47	10.34
	0.2	7.06	13.16	10.90
	0.3	**5.69**	**11.55**	**9.30**
	0.4	6.13	19.63	14.53
	0.5	5.82	28.32	20.20
	0.6	6.23	36.64	25.35

*weight percentage based on the amount of dry alumina powder

The shear rate of 50 s^{-1} is the exact shear rate usually achieved during gravity slip casting. Viscosity of all suspensions decreased as the shear rate increased (Table 2). The influence of the content of each dispersant type on the viscosity of 70 wt. % alumina suspensions at a constant shear rate of 50 s^{-1} is shown in Figure 1.

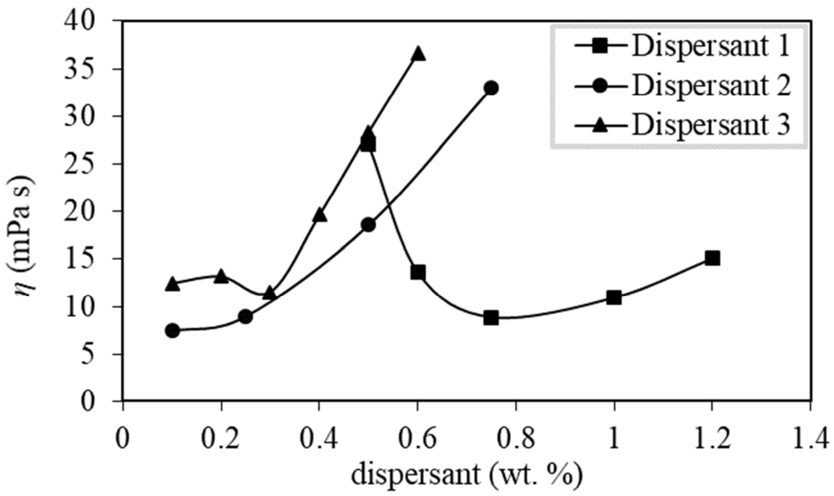

Fig. 1 Dependence of viscosity (η) on dispersant content (wt. %) at shear rate of 50 s^{-1} (dispersant 1: Darvan C-N, dispersant 2: Tiron, dispersant 3: citric acid)

The results presented in Table 2 and Figure 1 indicate that the optimal amount of each dispersant type suitable for slip casting is: 0.75 wt. % for Darvan, 0.1 wt. % for Tiron and 0.3 wt. % for citric acid. The most effective dispersant is Tiron– only 0.1 wt. % was required to stabilize the prepared suspension. However, in the investigated suspensions, citric acid also lowers the alumina suspension viscosity satisfactorily. Given the fact that this dispersant is more economical and eco-friendly, it can be established that citric acid can stabilize the presented system (70 wt. % alumina suspension) equally well as commercial dispersants.

3.2 Rheological flow curves of highly concentrated alumina (Al_2O_3) suspensions

Viscosity measurements are rather limited when evaluating the stability of concentrated suspensions. Therefore, rheological flow curves are used instead. Rheological flow curves show the dependence of shear stress (τ) on viscosity (η) as well as on shear rate (γ), and can be used for predicting the nature of interactions among particles in the suspension. The alumina suspension flow curves, with optimal amounts of different dispersants, are shown in Figure 2. Flow curves, expressed as a dependence of viscosity (Fig. 2A) and shear stress (Fig. 2B) on the applied shear rates, are shown for each optimal amount of particular dispersants.

The obtained results show that the suspension viscosity decreases with an increase in the shear rate (Fig. 2A), that is, all suspensions show typical pseudoplastic behaviour, characteristic of non-Newtonian fluids [22]. Fig. 2B also shows that shear stress increases with an increase in the shear rate of all suspensions, which also confirms the previous statement. The investigated suspensions with the addition of citric acid show a bit higher viscosity values at smaller shear rates, while the viscosities of dispersant 1 and dispersant 2 are very similar. The flow curves show no significant difference among different dispersants at higher shear rates. The shear stress values of all dispersant types are quite similar at very low shear rates. However, the shear stress values of dispersant 2 are notably lower at higher shear rates than those of dispersant 1 and dispersant 3.

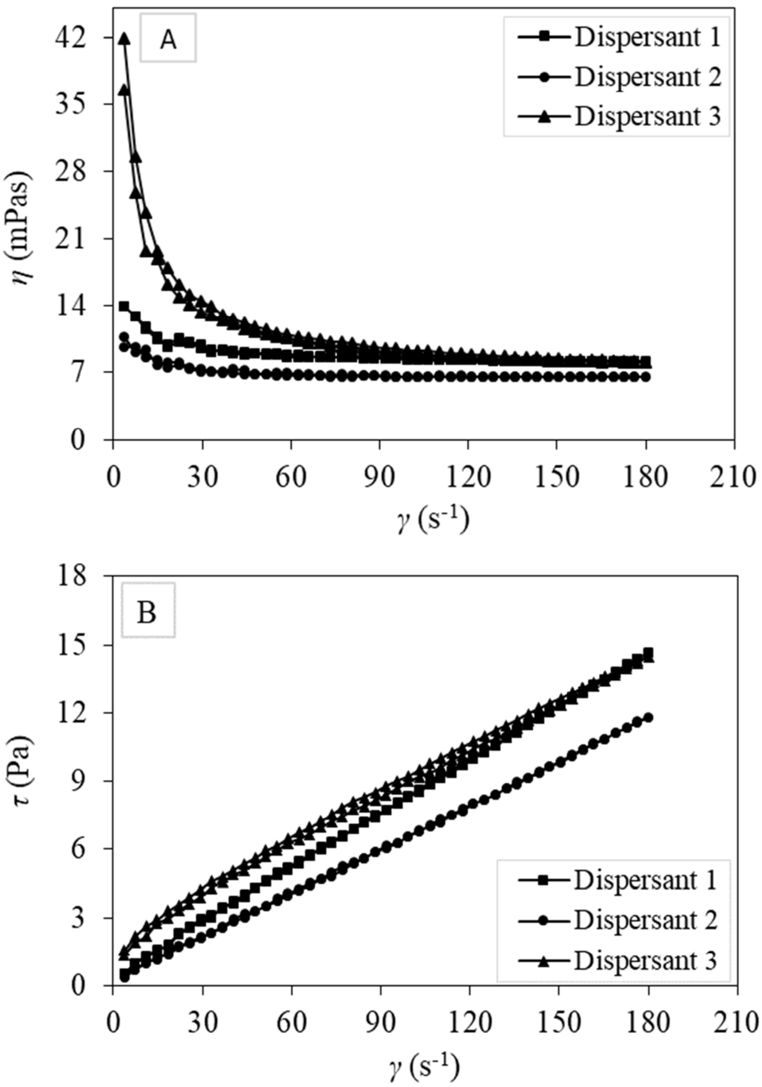

Fig. 2 Flow curves: (A) viscosity (η) vs. shear rate (γ), and (B) shear stress (τ) vs. shear rate (γ) (dispersant 1: 0.75 wt. % Darvan C-N, dispersant 2: Tiron, dispersant 3: 0.3 wt. % citric acid)

3.3 Rheological flow models

The power law, the Herschel-Bulkley and the Bingham model were used to fit the experimental data on optimal concentrations of each dispersant in 70 wt. % alumina suspensions obtained at room temperature. The calculated values are listed in Table 3. The ability of the models to follow the data was analysed. It was shown, that the Herschel-Bulkley model (Equation (2)) fits the measured data with a higher correlation coefficient (R^2) than the power law (Equation (1)) or the Bingham model (Equation (3)). The Herschel-Bulkley model is sometimes referred to as the pseudoplastic model with yield stress. This means that certain stress is required for the flow to start, and when the flowing occurs, the suspension behaves like a pseudoplastic fluid (i.e. the viscosity decreases as the shear rate increases). The shear rate exponent, n, is below 1, indicating that these suspensions behave pseudoplastically according to the power law and to the Herschel-Bulkley model.

Table 3 Linear regression parameters for different rheological models of 70 wt. % alumina suspensions stabilised with different dispersants (dispersant 1: 0.75 wt. % Darvan C-N, dispersant 2: 0.1 wt. % Tiron, and dispersant 3: 0.3 wt. % citric acid)

Dispersant	*wt. %	POWER LAW			HERSCHEL-BULKLEY				BINGHAM		
		k	n	R^2	τ_0 (Pa)	k	n	R^2	τ_0 (Pa)	ρ (Pa s)	R^2
1	0.75	15.3	0.87	0.975	0.03	9.23	0.97	0.999	0.05	7.9	0.977
2	0.1	52.0	0.63	0.971	0.12	5.99	0.85	0.999	0.20	7.05	0.984
3	0.3	10.7	0.90	0.954	0.03	16.3	0.98	0.998	0.02	6.41	0.959

*weight percentage based on the amount of alumina dry powder

4. Conclusion

The focus of this investigation was the influence of three different dispersants on rheological properties of highly concentrated alumina (Al_2O_3) suspensions. The prepared suspension had 70 wt. % of dried Al_2O_3 powder. Three commercially available dispersants were used: Darvan C-N (an ammonium polymethacrylate water solution), Tiron (4,5-dihydroxy-1,3-benzenedisulfonic acid disodium salt) and citric acid. Citric acid is a less expensive and much more eco-friendly alternative compared to the two previously mentioned dispersants Darvan and Tiron. The dispersant amount was varied and the rheological curves were recorded in order to determine the optimal content of each dispersant.

The conducted rheological measurements showed the optimal amount of each dispersant, i.e. the amount that allows the lowest apparent viscosity of the system. The established optimal amount of Darvan was 0.75 wt. %, of Tiron 0.1 wt. % and of citric acid 0.3 wt. %. According to the obtained results, the most effective dispersant is Tiron as only 0.1 wt. % of the dispersant was required to stabilize the prepared suspension. However, in the investigated suspensions, citric acid also lowers the viscosity satisfyingly. Given the fact that this dispersant is more economical and eco-friendly, it can be established that it can stabilize the chosen system (70 wt. % alumina suspensions) equally well as the commercial dispersants.

The applied flow models showed that all investigated suspensions can be categorized as the Herschel-Bulkley fluids: a certain amount of yield stress is present, but when the flow is established, the suspensions show pseudoplastic behaviour.

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