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Research Article

First Report to Characterize the Rheological Properties at Consecutive Transient Low Shear Rate of the Alumina Suspension Stabilized by Phosphate Ester

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Abstract

Rheological measurements are widely used to characterize dispersions, both as a means of studying their fundamental properties and as assessment of their suitability for particular technological purposes. Knowledge of their rheological behavior is essential for almost all dispersion in order to handle them satisfactorily. A numerous methods such as control tests for degree of dispersion, particle size analysis, sedimentation behavior, rheological determination, and optical analysis are established for characterization of dispersion. Among them rheological determination method play a very significant role in the assessment of the dispersion. In this paper, we report a cumulative impact of transient shear with time on viscosity profile for well dispersed alumina suspension having different degree of aggregation stabilized by phosphate ester at low shear domain.

Keywords: *Rheology, Alumina Suspension, Transient Viscosity, First Report.*

1. Introduction

The characterization of colloidal suspensions depends upon the purposes for which the information is sought, because the total description would be an enormous task. Among the properties to be considered are the nature, distribution of purity, size, shape defects, pores, adsorbed surface films, internal surface stress, stability, and state of agglomeration (Barnes H.A. 1997, Berzov W.H. et al. 1990, Mukherjee A. et al. 2008, 2001, Olphen H.V. 1977, Wiese R. and Healy T.W. 1970). The rheological behavior of the dispersion is of considerable importance in a large number of industries and indeed many of the popular terms used to describe consistency are derived from the properties of particular type of materials. Rheological properties have significant values in the production of dispersion where certain type of mill may operate satisfactorily only within a limited range of flow properties, but more particularly in the use of the finished products. The most important rheological measurement techniques to assess the state of aggregation (Nicolai T. and Cocard S. 2001, Mukherjee A. et al. 2008), in a suspension include steady state measurements, transient state measurements as well as dynamic (oscillatory techniques) measurements. Viscosity measurements are widely used to characterize dispersions both as a means of studying their fundamental properties as well as an assessment of their suitability for a particular technological purpose (Cesarano J. et al 1988, Ushifusa N. and Cima M. 1991, Parfitt G.D. 1969). In this paper our aim is to investigate the transient shear and time dependent viscosity behavior of well dispersed alumina suspension having different degree of aggregation stabilized by Phosphate ester. For its successful completion and reproducibility, we performed the experiment number of times with a keen accuracy and systematically at low shear rate domain.

2. Experimental

2.1 Materials used

In the present work, Alumina powder A17NE (ACC-Almatis) was used.

Table 1: Characteristics of A17NE alumina powders taken for study

Alumina powder		***A 17 NE***
Particle size		d_{50} = 2.3 μm d_{90} = 7 μm
Specific Surface area/BET (m^2/g)		2.9
Chemical Composition	Al_2O_3	99.81%,
	Na_2O	0.10%,
	Fe_2O_3	0.03%,
	MgO	0.01%,
	SiO_2	0.03%
	CaO	0.02%

The composition of the powder according to the manufacturer's information is given in the (Table 1). The

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binder used was Poly vinyl Butyral (PVB) of grade Maripol B-30 (Parekh Chemical).Solvents used were azeotropic mixture of 50 % (by weight) Methyl Ethyl Ketone; MEK (Merck) and 50% by (weight) of 99.9 % Ethanol; EtOH, (ChangShuYangyuan Chemical, China). The dispersant used was Phosphate Ester dispersant; Surfonic PE -1168 lot 7716-33-38 (Witco Chemicals, USA). The composition for the preparation of the slurries and the formulation particulars are given in table 2.

Table 2: Composition of the Alumina Slurry and Formulation particulars used

Components	**Function**	**Wt. % (vol. %)**
Polyethylene Glycol 400 (PEG) Merck, $[(C_2H_4O)_nH$ where n = 380-400]	Plasticizer	6.08(8.29)
Butyl Benzyl Phthalate (BBP) Aldrich, $[2\text{-}(CH_3(CH_2)_3O_2C)C_6H_4CO_2CH_2C_6H_5]$	Plasticizer	1.38(1.90)
Mixture of ethanol & Methyl ketone	Solvent	30.12(58.09)
Alumina	Powder	55.29(21.77)
Poly vinyl Butyral (PVB)	Binder	7.13 (9.95)
Phosphate Ester Surfonic (PE)	Dispersant	0.25-1%

2.2 Preparation of suspended slurries

To ensure a negligible moisture content of the powders, we prepared the slurries (Table 1) by vacuum heating the powders at 100^0 C for 7days prior to their use. In the first stage, to a portion (13.96%) of the total azeotropic solvent mixture utilized in the formulation, 6.07% by weight of PEG, 1.38% by wt BBP and 1.38% of the binder PVB was added. The resultant mixture was stirred and kept overnight. After the time period, 55.2% by wt of the heat treated powder was then weighed and poured into the above mixture containing solvent & plasticizers. The resultant slurry was subsequently ball milled at 73rpm for 10 h with 10mm alumina balls (138% by wt of the total formulation) as the grinding medium. In the second stage, the solution of 5.74 % by weight of (PVB) and 16.28% of solvent mixture (viscosity: 665 $\mp$ 5 mPa.s) was added to the slurry. The slurry was further ball milled for 14h. The weight of the container containing the slurry was measured before and after the ball milling process to rule out any solvent or material loss. The alumina suspension thus formed was poured into a container and was de-aired under slow stirring and in vacuum conditions (300 mm Hg) till no more bubbles were seen to form. The weight loss during the de-airing process was found to be nominal (0.2%). Graduated cylinders (Borosil) fitted with stoppers and sealed with Parafilm M (Pechiney Plastic Pakaging) were used to investigate the sediment nature. The graduated cylinders containing the slurry were placed in an oven fixed at 60^0 C for 72 h before the sediment nature to be investigated.

2.3 Experimental set up

A programmable Brookfield concentric cylinder Rheometer DV-III ULTRA was used to measure viscosity. This Rheometer was coupled with a small sample adaptor (SSA 13RP, Brookfield) with EZ-lock assembly and embedded RTD temperature probes. The data was gathered with the help of Rheocal program supplied by Brookfield at a time interval of 1 min. During the experiment the temperature of measurements was kept constant at 20^0C $\mp$ 0.2 with the help of Brookfield water bath TC 502. Prior to any measurement, the slurries were kept at rest in the SSA till the required temperature was obtained. As the required temperature was reached, the suspensions were pre sheared ($\dot{\gamma}t$ = 1800) and after the pre shear stage the suspensions were left at rest for a period of 30 minutes in order to let the system develop an homogeneous level. The optimum dosage of the dispersant PE was chosen by measuring the viscosity of the suspensions at 4 sec^{-1} after an arbitrary 24 rotations of the spindle.

3. Results and Discussion

Powder loading of the alumina slurry prepared was taken 55% (w/w) (Table 1). It is observed that suspended slurries taken for experiments were remained high turbid (Parfitt G.D. 1969) even on standing at accelerated weather condition (60^0C, 72h). Moreover, at the end of sedimentation tests it is found that the suspensions of alumina powder remained very turbid. The high turbidity of the suspension emphasizes that sufficient number of particles is well-dispersed. Furthermore, it was also observed that there was no any visible phase separation after four months of standing at ambient conditions in the suspension taken for experiment which ensure retained high turbidity. These observations pointed out that the suspensions to be taken for experiment are well dispersed. To ensure the systematical accuracy of the experiment, we utilized fresh suspensions for each of these measurements. As per the standard procedure (Mukherjee A. et al. 2008, Olphen H.V. 1977, Reed J.S. 1995) of choosing the optimum dosage of the dispersant for preparing a well dispersed suspension, viscosity measurements were carried on these suspensions at a shear rate of 4 sec^{-1} under similar conditions of temperature, sample volume and time of shear respectively. It is well documented in the literature (Hibder P. et al. 1998, Parfitt G.D. 1969, Ushifusa N. and Cima M. 1991) that the suspension having the minimum viscosity is the best dispersion.

As it can be observed (Figure 1), the suspensions of 0 % PE, 0.25% PE and 0.55% PE respectively have much lower viscosity compared to 0.75% PE and 1.0% PE respectively. The suspension viscosities of the suspensions varied as: 0.55% PE < 0.25% PE < 0 % PE << 0.75% PE< 1.0% PE. Sedimentation tests also showed that the 0 % PE, 0.25% PE and 0.55% PE suspensions are well dispersed (Parfitt G.D. 1969, Ushifusa N. and Cima M. 1991) as evidenced by the high turbidity of these suspension even on standing at accelerated weather conditions (60^0C, 72h) as well as the presence of hard

sediments at the end of the test. The high turbidity of these suspensions did not allow the measurement of the sediment height. It was further observed that standing these suspensions undisturbed even for a period of four months under ambient conditions did not show phase separation and the turbidity still remained high. On the other hand, sedimentation tests also showed 0.75% PE and 1.00% PE suspensions were prone to sedimentation and a clear supernatant (Figure 2) started to appear within half an hour. The sediments of 0.75% PE and 1.0% PE suspensions were also found to be soft. Thus, the suspensions of 0.75% PE and 1.00 % PE were agglomerated.

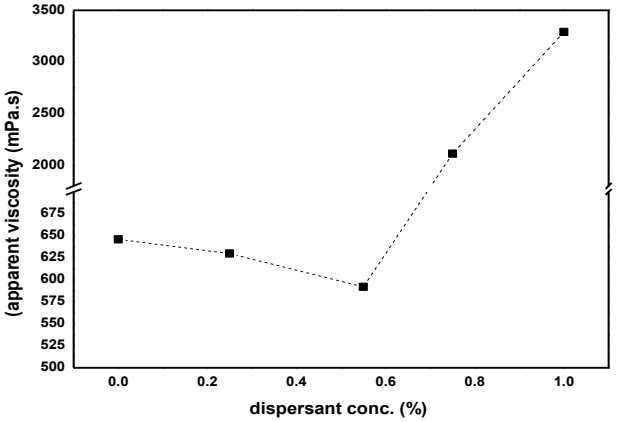

Figure 1: Change in apparent viscosity of the suspensions with the concentration of dispersant

Figure 2: Photograph of slurries taken for sedimentation study (left to right 0%PE, 0.25%PE, 0.50%PE, 0.75%PE, and 1.0% PE respectively)

For our experimental studies, we have taken only consideration of well dispersed suspensions which are 0% PE, 0.25% PE and 0.50% PE. After 30 minutes rest period before shearing (depicted in figure 3) shows interesting rheological behaviors which may be summarized as follows: (i) well dispersed suspensions of 0% PE, 0.25% PE and 0.50% PE respectively showed a continual increase in viscosity with time at each transient shear, however the 0% PE suspension showing the maximum positive slope followed by 0.25% PE and 0.50% PE suspension respectively (ii) in beginning, the raise in viscosity is seemed to be much more for all the suspensions which are aggregated at different level (iii) these suspensions shows a decrease in viscosity with the increase in applied transient shear rates (iv) the trend in slope of viscosity curve of the suspension sheared almost similar, although the magnitude in viscosity drift is significant to be considered (v) Overall summation in viscosity curve follows the order 0.50% PE<0.25%PE<<0%PE with the application of successive transient shear.

In this figure 3 the inset boxes represent the state of transient applied shear rate with progressive application of time, whereas before shearing, rest time period is 30 minutes to equalize the structural level.

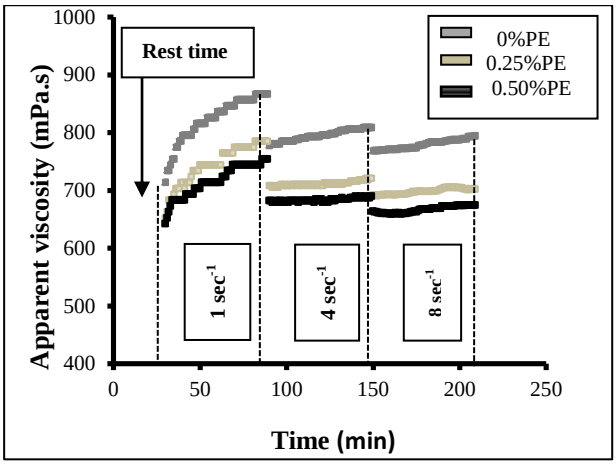

Figure 3: Comparative analysis for transient behavior (shear and time) of well dispersed slurries with different degree of aggregation

Thus, we report here transient shear and time dependent behavior of well dispersed alumina suspension having different degree of aggregation stabilized by Phosphate Ester at low shear transient domain. This identification represents the possibility of numerous effects such as state of agglomeration, aging, internal surface stress, shear applied, and degree of shear induced diffusion (Bye G. C. and Sing K.S.W. 1973, Viasnoff V. and Lequeux F. 2002, Leighton D. and Acrivos A. 1987,Struik L.C.E. 1978, Graham A.L. and Bird R.B. 1984, Nicolai T. and Cocard S. 2001, Ruzicka B. et al 2007) which may alter the stability of dispersion and there is a need for a more comprehensive study to correlate the above observed cumulative transient viscosity profile (figure 3). This will form the basis of our future investigations. Results of this investigation are very essential for the successful exploitation of materials to understand the equilibrium and transport properties of the suspension.

Conclusions

The present investigation conducted with 55% (wt/wt) alumina suspension having different degree of aggregation measures the transient viscosity which shows diverse time dependent and shear dependent viscosity characteristics in the low shear domain. The corresponding drift in viscosity for these suspensions will be different under

similar shearing conditions. However, to the best of our knowledge a time and shear dependent behaviors of suspensions at low shear rates are hitherto unreported in literature. It will be subsequently shown in this report that such investigation reflects some fundamental physics occurring in the suspension under flow which may also open up a new vista for characterizing well dispersed suspensions.

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