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Evaluation of dispersibility of aqueous alumina suspension in presence of Darvan C

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Abstract

The effect of ammonium salt of polymethacrylic acid commercially known as Darvan C as a dispersant on stability of aqueous suspension of Al_2O_3 was investigated through sedimentation test, determination of wet and flow points, and measurement of streaming potential. The degree of dispersion estimated by the sedimentation test was correlated with particle surface charge, and the inflection point in streaming potential as a function of concentration. Good agreement was observed between dispersion characteristics and specific charge and inflection in streaming potential. The results also illustrate the applicability of wet point and flow point techniques for examining colloidal dispersions. A convenient and reliable method of rapidly measuring stability maxima with optimum dispersant concentration could be envisaged through measurement of surface charge by PCD and determining wet point and flow point.

Keywords: Dispersibility; Wet point; Flow point; Streaming potential; Surface charge

1. Introduction

The dispersion of powders in liquid is an important process in the ceramic industry. The good dispersion of particles with high solids loading is often a prerequisite to obtain optimum packing state (high green density), which in turn influences the sinterability and the physical as well as chemical properties of the final product. Hence, monitoring of the state of dispersion/aggregation is essential step in colloidal processing of ceramics. However, in practice, colloidal sized particles have a natural tendency to aggregate when dispersed in aqueous medium. The degree of such aggregation, is usually governed by the chemistry of the interface between the solid particles and the suspending fluid. Therefore, in order to control the wet-forming process of these powders, it is important to know the behaviour of the particles in suspension and also the adsorption and distribution of additive phases on the interface, which may alter the processing behaviour and consequently the microstructure of the final product [1].

Interaction of suspended particles with the surrounding environment usually leads to adsorption of ionic species on their surface creating local electric charge, leading to dispersion through different types of forces. The inter-particle interaction energy in presence of additives like surface-active agents in the suspension has been well presented by a number of authors [2–5]. The measurement and control of surface charge on the particles surface can be used to enhance dispersibility of the particles in the suspensions. Hence the surface charge is an important parameter to predict dispersion behaviour.

The success of any wet-forming scheme/process largely depends on a judicious choice of a dispersant to obtain a well-dispersed, highly homogeneous and concentrated ceramic suspension. In practice, the selection of dispersants is usually done on an empirical basis, which involves evaluation of numerous products to achieve acceptable performance. In addition to dispersant selection and characterization, the interaction of the dispersants with the ceramic powder also plays a vital role and must be well characterised to predict dispersion behaviour. Usually, the conventional techniques used to evaluate the dispersion characteristics are related to the macroscopic properties of the suspensions such as sedimentation and viscosity behaviour, whereas relatively

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less is known about the interfacial behaviour of the dispersants with ceramic particles on a molecular scale. This work is an attempt to apply surface chemical principles to select suitable dispersant via different methods/processes.

The paper presents systematic evaluation of dispersion behaviour of colloidal alumina suspensions in presence of dispersant Darvan C. The stability of the suspension was assessed using sedimentation method, turbidity measurement, wet and flow point determination, streaming potential measurement with particle charge detector (PCD-03-pH), etc. Emphasis has been given to elucidate the surface-active behaviour of dispersant and to identify the conditions for most effective use of the dispersant to enhance the dispersibility of alumina in aqueous suspension in terms of its bulk physico-chemical behaviour. These studies have been found to provide vital information for understanding the correlation between various parameters that affect ceramic processing.

2. Experimental

2.1. Materials

High pure α-Alumina (CT-3000 SG) obtained from the Alcoa, Kolkata, India was selected as the model ceramic material for present investigations. It is a very fine powder with an average particle size of 0.7 μm and a specific surface area (BET) of about 7.0 m^2/g. The characteristic properties of the powder are summarised in Table 1.

The dispersant used was an ammonium salt of polymethacrylic acid, supplied by M/S, R.T. Vanderbilt Co. Norwalk, CT, USA and is available commercially with the trade name Darvan C. It has an average molecular weight of about 13000 g/mol. It is supplied in the form of a 25% aqueous solution with an active matter content of about 35%. The detail characteristic properties are presented in Table 2. All other reagents used in the experiments were of analytical reagent (AR) grade. Double distilled water of specific conductivity less than 1.5 μS/cm was used in all studies.

Table 1
Properties of the alumina powder used

Powder characteristics	Typical values
Type	Calcined alumina
α-Phase content	99.9%
Average particle size (μm)	
d_{50} (μm)	0.7
d_{90} ((μm)	2.0
Specific surface area (m^2/g)	7.0
Density (g/cm^3)	3.9[a]
Impurities	
Na_2O	0.02
SiO_2	0.05
Fe_2O_3	0.03

[a] As per literature from the manufacturer.

Table 2
Some characteristic properties of Darvan C

Characteristics properties	Typical values
Molecular weight (g/mol)	13000 (approximately)
Active matter (wt.%)	35
Density (g/cm^3)	1.11
pH	7.5
Ionic nature	Anionic

2.2. Surface charge measurement

Surface chemical properties of alumina powder were determined by specific surface charge measurement using a particle charge detector (PCD-03-pH) of M/S. Mutek, Germany. The principles and detail method of surface charge measurement are reported elsewhere [6,7]. Measurements were done in absence and presence of Darvan C at varying pH adjusted with AR grade HCl and NaOH solution. Incremental concentrations of Darvan C was added drop wise to the 5 wt.% alumina suspension under agitated conditions.

2.3. Sedimentation tests

For sedimentation test, 2.5 g of alumina powder was dispersed in 50 ml aqueous solution containing different amount of dispersant Darvan C. The suspensions were first stirred thoroughly for 10 min in a beaker and then transferred to the measuring cylinder where it was allowed to stand undisturbed for 48 h. The suspension density was measured by pipetting out 5 ml of the cloudy suspension from a predetermined height (corresponding to a volume of 25 ml) from the top of the cylinder. It was then weighed and dried to know the weight of the suspended particles in the 5 ml suspension. The volume of suspension containing dispersed particles above the settled mass at the bottom was calculated after deducting the space covered by the sediment. The total amount of solid remaining in suspension was then estimated from the total volume of suspension over the sediment bed. The remaining solid was assumed to have been reported in the sediment, the volume of which is read directly at the bottom of the graduated cylinder. The relative density of the sediment was then estimated from the ratio of the density of the sediment to the true density of the solid.

2.4. Determination of optimum dispersant dosage

The optimum dosage of the dispersant Darvan C to achieve maximum dispersion of alumina suspension was determined with the help of Particle Charge Detector (PCD-03-pH). For this purpose, the streaming potential of the 5 wt.% alumina suspension was measured in presence of various amounts of Darvan C. The inflection point in the plot between concentrations versus PCD potential gave the optimum dosage of the dispersant for achieving maximum dispersion [7].

2.5. Wet point and flow point determination

The wet point of powder is defined as the least amount of solvent required for a unit amount of powder to form a lump. The flow point represents the least amount of solvent required for the suspension to begin to flow. Measurement of the wet point and flow point is simple and useful technique for a rapid evaluation of the dispersion of any suspension. Exactly 2 g of Al_2O_3 powder was mixed with a fixed volume of an aqueous solution containing different amount of Darvan C on a glass Petri-dish keeping the convex part upward (which is well below the requirement of liquid to form a lumpy mass).

A small volume of solution containing known amount of Darvan C was first added to the Al_2O_3 powder and the balance amount of water was also added drop wise from a burette and mixed intimately with a spatula such that a lumpy mass was just formed. The total amount of liquid thus required was noted and reported as wet point. Further amount of water was added drop wise from the burette and mixed with the lumpy mass till the mixture begins to flow which is clearly visible as the slurry starts flowing from the top of the dish [8]. The total volume of liquid including that required for wet point was reported as flow point. The procedure was repeated for different concentration of Darvan C.

2.6. Turbidity measurement

If the suspension is well-dispersed it will appear to be turbid but the turbidity decreases with de-stabilisation of the suspension. In the present case, the turbidity measurements were carried out using the Nephelometer (Model: Elico CL52, make India) and are presented in terms of Nephelometric turbidity units (NTU). For turbidity measurement, the suspension containing varying concentration of the dispersant were first mixed thoroughly and then allowed to stand for a predetermined time. A fixed volume of the suspension was then drawn from a predetermined height from the top and subjected to turbidity measurement.

3. Results and discussion

3.1. Surface charge effects in ceramic processing

The amount of charge on the surface is an important particle characteristic because it determines many properties of the suspension. The charge in the fluid is in the form of free ions. There is a region around each particle where the particle charge attracts the free ions to form an electrical cloud called the electrical double layer. The interaction between two charged particles in a polar media is related to the osmotic pressure created by an increase in ion concentration between the particle whose electrical double layer overlap [6,7]. In the present investigation, Al_2O_3 slurry has a natural pH of 8.4–8.7 in double distilled water (pH 6.7). The results of surface charge on alumina at varying pH are presented in Table 3. It clearly shows that under prevailing experimental condition of natural pH, the Al_2O_3 particle exhibit a mild positive charge.

Table 3
Specific surface charge density of Al_2O_3 (5%, w/v), distilled water pH 6.8

pH	Specific surface charge (C/g)	Streaming potential (mV)	Observation
2.2	+1.219	+98.0	
2.98	+0.757	+108.5	
5.5	+0.872	+652.7	
8.4 (natural pH)	+0.042	+194.2	Natural pH of water after Al_2O_3 addition
9.14	−0.012	−27.3	pH_{iep}
11.11	−0.221	−482.1	
11.54	−0.325	−172.6	
12.0	−0.203	−150.0	

3.2. Surface chemical properties of alumina suspension

The stability of alumina suspension is closely related to its surface properties in aqueous solution. Well-dispersed suspension can be obtained with sufficiently high surface charge density to generate a strongly repulsive double layer force. Fig. 1 shows the effect of pH on surface charge of Al_2O_3 with and without Darvan C. The isoelectric point (iep) of untreated alumina in aqueous suspension is found to be at pH 9.14, which is in close agreement with the reported value 9.2 [3]. Fig. 1 further depicts that Al_2O_3 contains negative surface charge in the pH range above the iep and positive surface charge at pH values below the iep, which is typical characteristics of oxides. It is further seen that the iep has been shifted to the acidic side (i.e. pH 5.65) with the addition of Darvan C. This is due to interaction of Darvan C with alumina particles leading to specific adsorption at Al_2O_3/water interface. A dose of 2.29 mg/g of Darvan C to the suspension has brought down the iep to pH 5.65. Further, the Darvan C addition results in a more negative surface charge than that without it. Such increase in the magnitude

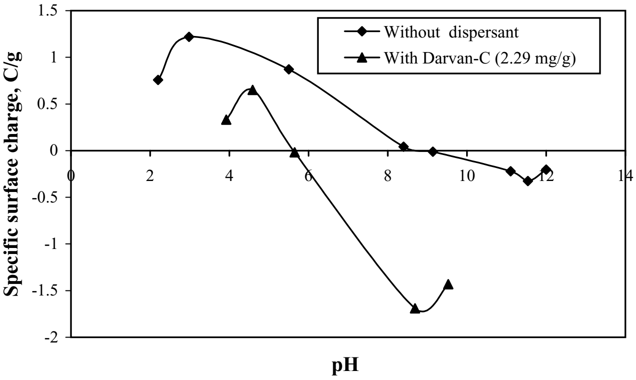

Fig. 1. Effect of pH on the specific surface charge of 5% (w/v) alumina suspension with and without Darvan C.

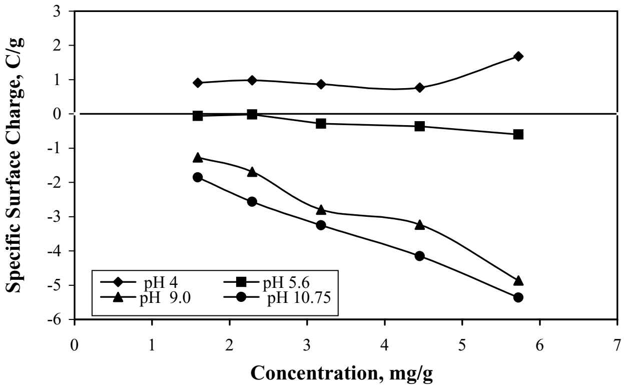

Fig. 2. Effect of Darvan C concentration on the surface charge at different constant pH.

of negative surface charge could be a result of the dissociation of Darvan C in the solution producing more COO^- groups adsorbed on the alumina particles, which could increase the net negative surface charge on powder surface, resulting in more repulsion between the particles. At iep, the surface charge is zero thereby reducing the electrostatic repulsion between the particles to a minimum and hence the particles in the suspension can be easily agglomerated. It can be seen from the Fig. 1 that under strong acidic conditions ($pH < 3$) and strong basic conditions ($pH > 11.5$) absolute magnitude of charge on alumina surface decreases, which can be explained by DLVO theory [3]. Under both the conditions, more and more HCl or NaOH are required to adjust the pH values, resulting in the increased ionic strength in the suspension, which compresses the thickness of the double layer and hence results in the decrease of surface charge at the interface on the particles and hence decrease in stability.

Fig. 2 shows the specific surface charge of alumina suspension as function of Darvan C concentration at four different pH 4, 5.6, 9.0 and 10.7, respectively. The relationship between surface charge and Darvan C concentration shows interesting behaviour. At pH 4, the surface charge is always positive with increasing Darvan C concentration. The magnitude of charge is more or less same throughout the range investigated. At pH 5.6, increasing Darvan C concentration leads to a small increase in the magnitude of negative surface charge from −0.060 to −0.603 C/g. This indicates that the adsorption of Darvan C on alumina at a pH below or around 5.6 is only marginal. Also, there appears no reversal of charge with concentration. It can further be predicted that there could be a limit below which the iep cannot be shifted even though the surface is treated with high concentration of Darvan C solution.

At pH 9, as the concentration of the Darvan C was increased from 1.59 to 5.7 mg/g, the surface charge also became more negative, i.e. from −01.269 to −4.862 C/g, indicating increased adsorption of Darvan C onto alumina. Similar increase in the magnitude of negative surface charge with increasing concentration of Darvan C was also observed at pH 10.7. When the concentration of the polyelectrolyte added is sufficiently high (but not sufficient enough to reach the saturation coverage), the surface charge also becomes highly negative attesting to good dispersion of the alumina particles.

3.3. *Interaction of Al_2O_3 with Darvan C*

Darvan C is a very effective dispersant for oxide system. There is a significant shift in iep from pH 9.14 down to pH 5.65 in presence of Darvan C. As a result of addition of Darvan C the particle surface is negatively charged at a wide range of pH above 5.65. The negatively charged carboxylic group dissociated from Darvan C is easily adsorbed on the positively charged alumina surface via electrostatic attraction and as a result the particles become more negatively charged. The surface being more negatively charged will have demand for more H^+ to neutralise the surface charge and hence shifts iep to acidic pH. The results of sedimentation tests in the presence of optimum dosages of Darvan C at different pH values clearly show that near iep (pH 5.65) dispersion volume is minimum, seemingly due to partial agglomeration of particles while it appears more dispersed at the range of pH above 6. This observation is in good agreement with surface charge versus pH curve (Figs. 1 and 2). The double layer repulsion is small near iep and therefore, van der Waals attraction force makes particles agglomerated while at pH far from iep electrostatic repulsion keeps the particles separated from each other due to high surface charge.

3.4. *Mechanism of interaction/adsorption of Darvan C onto alumina powder*

The specific free energy of adsorption (interaction) for the alumina–Darvan C system based on the explicit relationship between the characteristic shift in the iep and the concentration of the chemisorbing surfactant in solution can be estimated in accordance with the following proposition given by Pradip [9]:

$$\Delta\,\mathrm{pH}_{\mathrm{iep}} = 1.0396\,\mathrm{C_o}\exp\left(\frac{-\Delta G^{\mathrm{o}}_{\mathrm{SP}}}{RT}\right) \tag{1}$$

where $\Delta\mathrm{pH}_{\mathrm{iep}}$ is the shift in the isoelectric point at the dispersant concentration C_{o} and $\Delta G^{\mathrm{o}}_{\mathrm{SP}}$ represents the corresponding specific energy of interaction between the ceramic powder surface and the dispersant. R and T are the standard gas constant and the temperature in Kelvin, respectively.

The specific free energy of interaction obtained using above equation for alumina in presence of Darvan C are summarised in Table 4. The specific energy of interaction $\Delta G^{\mathrm{o}}_{\mathrm{SP}}$ is close to $-3.50\,RT$ and this clearly indicates that Darvan C strongly adsorbs onto alumina, which in turn is indicative of strong interaction of alumina with Darvan C. The surface charge versus pH plot for alumina slurry (Fig. 1) with and without Darvan C indicates that due to adsorption of Darvan C, the isoelectric point of Al_2O_3 has been brought down to pH 5.65 from 9.14, i.e. a difference in $\Delta\mathrm{pH}_{\mathrm{iep}}$ value

Table 4
Specific free energy of interaction for the alumina and Darvan C system

System	pH_{iep}	Concentration (ppm)	ΔpH_{iep}	$-\Delta G^{o}_{SP}$ (*RT* units)
Alumina powder	9.14	–	–	–
Darvan C	5.65	114.5	3.49	3.50

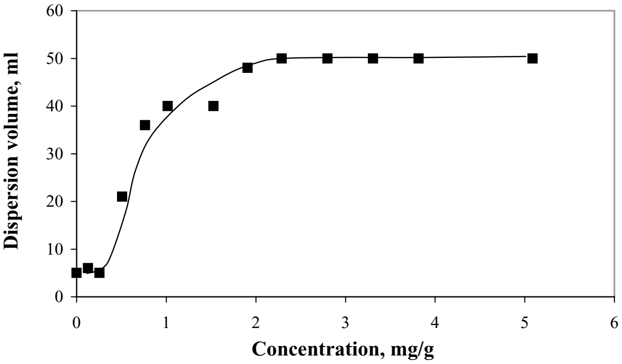

Fig. 3. Sedimentation tests of alumina particles in aqueous suspensions for 48 h in the presence of Darvan C at normal constant pH 8.4.

of around 3.49 (Table 4). It is known higher the value of ΔG^{o}_{SP} for a given system, more the electrical double layer repulsion. The high value (−3.50 *RT* units) of ΔG^{o}_{SP} in the present case suggest that Darvan C is a suitable dispersant.

3.5. Sedimentation

Fig. 3 depicts the results of the sedimentation test for the suspension with various amounts of Darvan C at room temperature 25 °C and constant natural pH 8.4–8.6 with alumina powder. Initially with increasing concentration of Darvan C in the suspension, there is steep rise in the dispersion volume upto concentration of 1.0 mg/g and then plateau is reached at concentration 2.29 mg/g. After plateau value, there is no further increase in dispersion volume. Therefore, 2.29 mg/g of Darvan C concentration at which it gives maximum dispersion volume was chosen as optimum dosages to obtain a stable suspension.

Fig. 4 shows the dispersion quality of alumina suspension at pH ranging from 4.8 to 10.6. The results show that suspension at lower pH range from 4.8 to 7.28 generates good dispersions, which could be due to the high positive surface charge in an acidic environment (Fig. 1). As Al_2O_3 is a basic oxide, it consumes H^+ ions and increases pH resulting in positive charges at the interface in pure water. The suspensions even without Darvan C dispersed well owing to their positive surface charge. On the other hand at pH ranging from 7.2 to 8.5, there is very sharp fall in the dispersion volume as the surface approaches towards point of zero charge and then from pH 8.5 onwards it remains more or less the same. Low dispersibility of alumina in these higher pH ranges can be explained in the light of the surface charge (Fig. 2) where it is clear that the surface charge does not significantly change beyond iep. The iep of alumina particles is found to be at pH 9.14, so the charge on the particle near this pH is minimal/zero. Therefore, all particles have natural tendency of agglomeration and settle very fast. In entire alkaline pH range from 9 to 12, charge on the alumina particles is slightly negative (−0.012 to −0.203 C/g) and hence minimum dispersion volume.

But the situation is different when the surface interacts with Darvan C as depicted in Fig. 5. Dispersion of Darvan C treated Al_2O_3 has been found to be minimum at the iep, i.e. at pH 5.65 but dispersion is high at both acidic as well as alkaline sides of the iep. In this case, Darvan C concentration of 2.29 mg/g was kept invariant at all pH range of investigation. Suspension from pH 6.26 to 9.9 demonstrated excellent dispersion characteristics, as it is evident from dispersion volume depicted in Fig. 5. The better dispersion quality at alkaline pH is due to the fact that alumina particles are highly negatively charged in the presence of Darvan C. As mentioned earlier Darvan C is dissociated fully at high alkaline pH, which induces mutual repulsion among the particles, resulting in better dispersion. In fact it may further be corroborated with the finding that the surface charge of

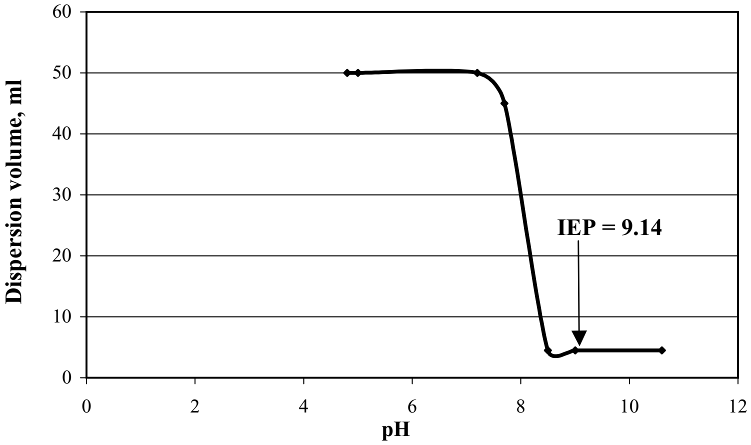

Fig. 4. Sedimentation tests of alumina particles in aqueous suspensions for 48 h at different pH.

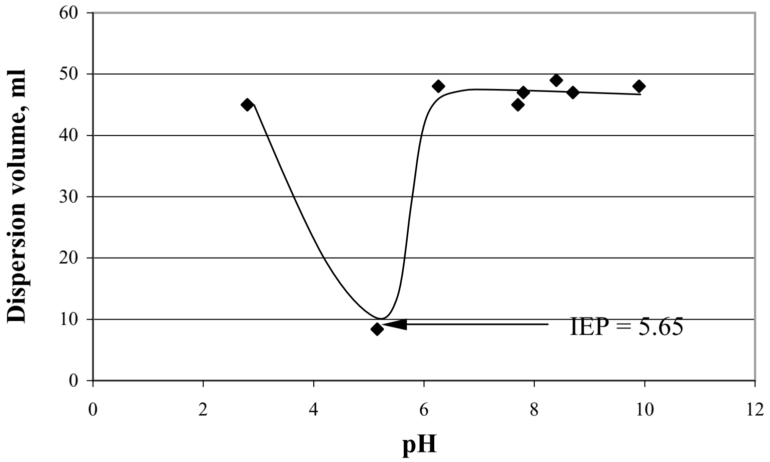

Fig. 5. Sedimentation tests of alumina particles in aqueous suspensions for 48 h in the presence of Darvan C at different pH.

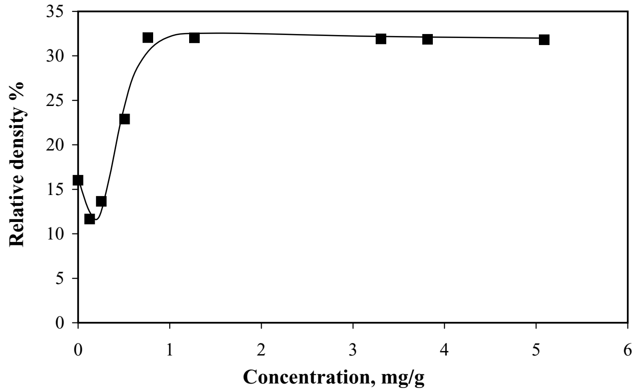

Fig. 6. Sediment density of alumina powder in aqueous suspension with varying concentration of Darvan C after 48 h.

alumina in presence of Darvan C is maximum at pH 8.69, which is about −1.690 C/g. On the contrary, at pH 5.65 dispersion volume is minimal (8.4 ml) and dispersion quality was very poor. This is because of the fact that iep of Al_2O_3 in the presence of Darvan C at the dosage of 2.29 mg/g is 5.6.

3.6. *Packing of particles*

Well-dispersed suspension are characterised by slow sedimentation, a cloudy supernatant, and a higher sediment density [10]. Fig. 6 shows the sediment density of alumina suspension at varying concentration of dispersant Darvan C. As the concentration of Darvan C increases, the relative sediment density increases and reaches a maximum value of 32% at a dispersant dosages 1.2 mg/g. It can be concluded from this study that the packing density is related to the stability of the suspensions.

3.7. *Wet point and flow point of powder*

One of the objectives of adding a dispersant is to obtain a highly loaded suspension while retaining the fluidity. The flow point represents the thickest suspension attainable to retain fluidity, and hence the least water content [8]. Darvan C helps to reduce the water requirement to maintain fluidity. Fig. 7 shows the typical curve of wet point and flow point of colloidal alumina experimentally determined in presence

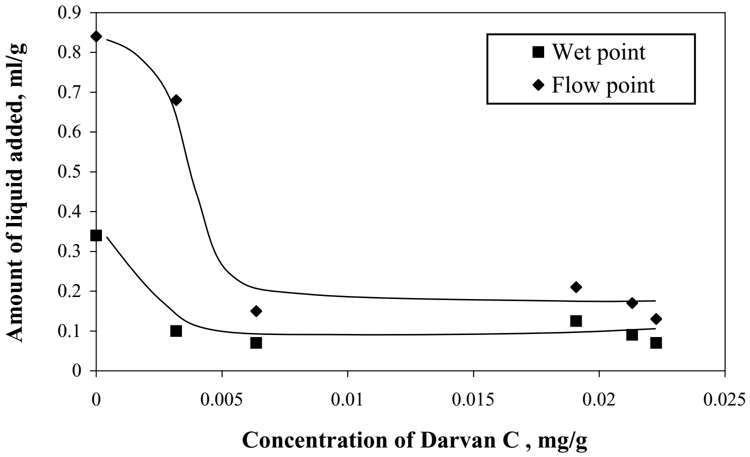

Fig. 7. Wet and flow point of colloidal alumina.

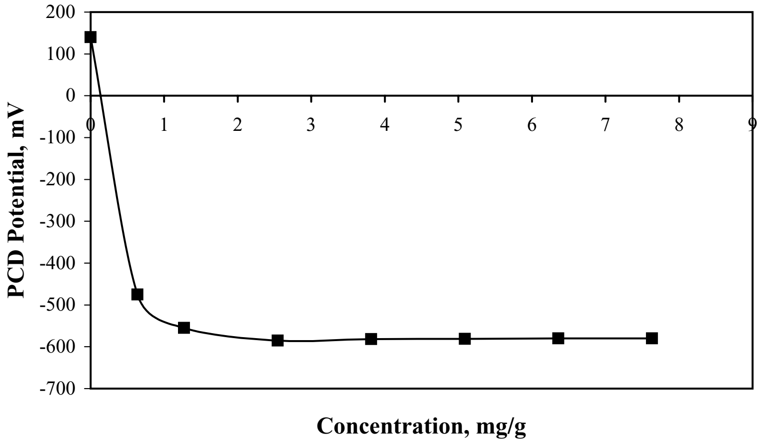

Fig. 8. Effect of Darvan C dispersant concentration on PCD potential of 5% alumina slurry at pH 8.4.

and absence of Darvan C. The value of the wet point first decreases with concentration of Darvan C and is then almost constant for the addition of various amounts of Darvan C. The wet point represents the least amount of water required for a unit amount of powder to form a lump. Similarly, the addition of Darvan C decreases the flow point rapidly upto dosages of 0.0063 mg/g beyond which it remains almost constant. The addition of Darvan C to attain a good dispersion allows decrease in the flow point value seemingly due to increase in negative surface charge and decrease in the viscosity of the suspension. At Darvan C dosages beyond 0.0063 mg/g, the flow point remains the same. Therefore, 0.0063 mg/g of Darvan C can be considered to be the optimum dosages to obtain thickest slurry with reasonably good fluidity. Daniel and Goldman as referred to by Wang et al. [10] have suggested a small gap between the wet points and flow point for a better dispersion of the suspension. The results of wet point and flow point in the present investigation are also in good agreement with the suggestion of Daniel and Goldman. Therefore, it has been concluded that the measurement of the wet point and flow point is a simple but useful technique for a rapid evaluation of a required amount of dispersant for making any flowable suspension with maximum amount of solids loading.

3.8. Determination of stability maxima and optimum dispersant dosage

Fig. 8 shows variation of PCD potential with different dosages of dispersant Darvan C. The untreated alumina suspension exhibits a positive PCD potential. The addition of small amount of the highly negatively charged polyelectrolyte Darvan C neutralises the positive surface charge leading to reversal of the charge. The magnitude of the negative PCD potential continues to increase rapidly with increasing concentration of dispersant, and then remains almost constant after reaching the plateau value (−585 mV). It indicates that at low concentration region, the adsorption of negatively charged species of dispersants on the ceramic particles continues and increases the magnitude of

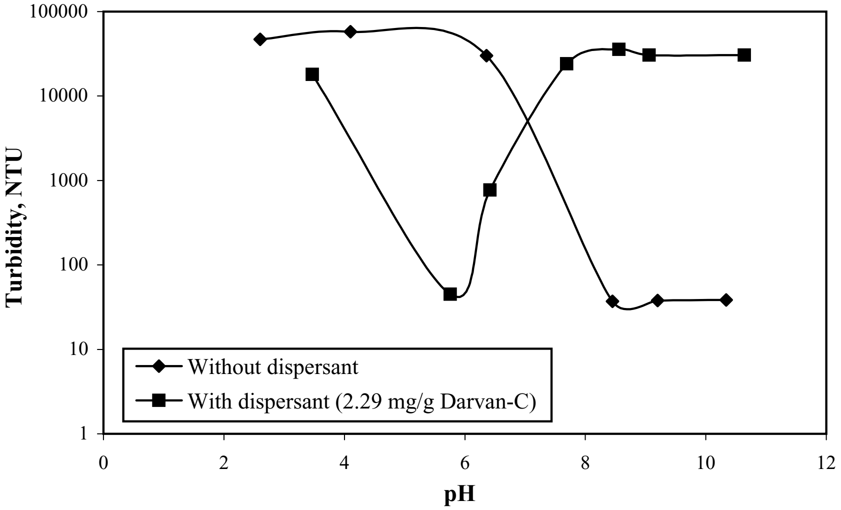

Fig. 9. Variation of turbidity of aqueous suspension of alumina powder at different pH.

negative surface charge on it. The occurence of plateau value in the PCD potential starts at a Darvan C concentration of 2.29 mg/g, which is in agreement with optimum concentration obtained by sedimentation technique. Any amount of the dispersant above optimum concentration remains in the aqueous phase, which may affect the viscosity of the solution. This data is further corroborated with visual observation of the settling behaviour of the suspension in presence of the above dispersants. A distinctly optimum dispersant amount can be determined from the potential characteristics obtained from the PCD and the results can be readily used for practical purposes.

3.9. Turbidity measurement

Fig. 9 shows the variation in turbidity of the suspension at different pH with and without dispersant. The turbidity of the suspension in the absence of dispersant, in the acidic pH range from 2.6 to 6.4 is very high, decreases rapidly between pH 6.4 and 8.45, and then remains more or less same beyond the pH value of 8.45. On the other hand, in the presence of dispersant at optimum dosage (2.29 mg/g), the turbidity values are highest in both very acidic as well as very alkaline pH. The minimum turbidity was obtained at pH value of 5.76, which is very close to the $\mathrm{pH_{iep}}$(5.65).

If we compare turbidity data with surface charge (Fig. 1) it is found to be in accordance with the DLVO theory. When positive charge on the particle in the acidic pH range is sufficiently high the suspension is well-dispersed resulting in high turbidity. At $\mathrm{pH_{iep}}$ as the surface charge is minimum the particles tend to agglomerate and settle for which the turbidity also reaches minimum value. In alkaline pH the particles are negatively charged but the magnitude of charge for alumina in absence of dispersant is very small whereas in the presence of dispersant Darvan C, the surface charge is strongly negative beyond $\mathrm{pH_{iep}}$. As a consequence, the turbidity of the suspension in presence of Darvan C at alkaline pH is comparatively much higher than that in absence of any dispersant.

4. Conclusions

Based on this investigation, the following conclusions can be drawn:

1. This study clearly shows that a good understanding of surface chemistry is required if concentrated suspensions of the desirable rheological properties are to be achieved.
2. Electrokinetic tests show that the surface charge of the alumina powder becomes more negative with increasing concentration as a result of dispersant adsorption.
3. Surface charge density has a tendency to decrease under strong acidic as well as basic pH conditions because of increased ionic strength compressing the thickness of the electrical double layer.
4. Measurement of the wet and flow point is a simple and useful technique for a rapid evaluation of the dispersion characteristics of Al_2O_3.
5. The measurement of surface charge is a strong predictive parameter of aqueous dispersion behaviour.
6. The methods available for observing either the extent of dispersion or the surface charge on the particles are indirect. PCD-03-PH can be precisely used to know directly surface charge density as well as dispersion optima and hence effective dispersant concentration.

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