PARTICLE SIZE DISTRIBUTION ANALYSIS OF AN ALUMINA POWDER: INFLUENCE OF SOME DISPERSANTS, PH AND SUPERSONIC VIBRATION

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Abstract - It is well known that colloidal powder particles (between 1 mm and 0.001 mm) tend to agglomerate due to electrostatic forces. Then assuring an optimal dispersion condition is essential for good particle-size analysis results, since aggregates or weak agglomerates can be measured as single particles. In this paper the particle size distribution of an alumina powder A1000SG (ALCOA) was measured using distinct dispersion procedures. Distilled water was used as dispersant liquid in the pure state and with additives (citric acid and Duramax D-3005). Dispersion by supersonic vibration was also investigated, but only the application time was varied. Particle size analysis was accomplished by laser scattering technique and the dispersion condition was evaluated through zeta potential. The results showed that the Duramax's electrosteric impediment is more efficient than citric acid's electrostatic force, thereby providing better dispersion. Although useful, the supersonic vibration was not good enough to assure an optimal dispersion, at least for the material tested here.

Introduction

Knowing mean particle size and its distribution is a major concern regarding production process involving particulate materials. Mechanical strength, density, thermal and electrical properties of finished products are strongly affected by particle size distribution [1]. Reproducible and accurate results from particle size measurements are a major concern, regarding that economical losses can be resulted from low quality products and high rejection rates.

Various particle size determination techniques have been developed, however a lot of difficulties inherent to each technique arises in order to obtain accurate values [2-6]. For that reason reproducibility becomes more relevant in process control, although accuracy is demanded in research and development activities [7].

The reproducibility of a powder size measurement is mostly influenced by two factors: sampling and powder dispersion. A suitable sample must represent accurately the powder bath in question and above all must be well dispersed; otherwise the size that will be measured is not the one from single particles but from weak aggregates or agglomerates of particles.

Fine particles with colloidal dimensions show a strong tendency to agglomeration, being kept close together by electrostatic forces. In a liquid medium, the high specific surface area of these particles improves the action of superficial forces causing agglomeration. [8]. To assure a good dispersion, repulsion forces should be stronger than attraction ones. Repulsion can be originated from two events: electrical charges at particle surfaces produced by the interaction between the particle surface and liquid medium, forming a double electrical layer (electrostatic

stabilization); adsorption of long-chain polymers, covering particle surfaces and making particle-to-particle contact impossible. (electrosteric impediment).

Dispersion state of a suspension can be controlled by the electrical surface potential and by the thickness of the electrical double layer. This layer is formed by ions of charge opposite to that of the particle close to the surface (named counter ions). Surface potential is related to the so called zeta potential, a measure of the electrical potential at the shear or slipping plane, which is a boundary in this diffuse layer between the counter ions kept surrounding the particles and the ones that go away when an electric field is applied [10].

The aim of the present work is to evaluate the influence of different dispersions states on particle size measurements.

Experimental

For the dispersion experiments proposed here an Alumina powder (A1000SG - ALCOA - 9,2 m^2/g) was chosen for the dispersion conditions have been well studied. Deionized water was used as suspension medium. Tree sets of experiments were conducted according to the dispersant agent utilized: pure water (no dispersant); citric acid in two different concentrations, 0,14 e 0,28 mg/m^2 (mg of citric acid per powder surface area); a commercial polyacrylic acid based polyelectrolyte (Duramax D-3005 from Rohm & Haas) in a concentration of 0,04 mL/g.

Dispersion conditions of the samples were established from the variation of zeta potential with pH. Particle size measurements were done under two different conditions: at a high zeta potential and near isoelectric point (IEP), where zeta potential is zero.

Alumina concentrations in water suspension were determined by the operational conditions of the equipments, e.g. 0,7 mg/mL for zeta potential analysis and 0,2 to 0,3 mg/mL for particle size analysis. Aqueous solutions of KOH and HNO_3 were added for pH control of the dispersions.

Zeta potential measurements were performed on an electrophoretic light scattering (ELS) equipment (ZetaPlus from Brookhaven Instruments Corporation), using a modulated laser beam and an electrolytic system (aqueous solution of KNO_3 10^{-3} M). Particle size distributions were measured by laser scattering (Granulometer 1064 from CILAS -Compagnie Industrielle des Lasers).

The influence of supersonic vibration energy (14W) on dispersion state was also investigated here by changing only the time of application, according to the following: no application (SS - 0), only during the data acquisition time (SS - During), 30 seconds (SS - 30s), 1, 2 and 3 minutes (SS - 1min; SS - 2min. SS - 3min), the four later before the acquisition time. In these tests, the dispersant agents were added just before the analysis, with no time for stabilization, a common practice adopted by analytical laboratories.

Results and Discussion

Fig. 1 shows zeta potential versus pH curves for pure alumina (no dispersant) and alumina plus dispersants. The isoelectric point of pure alumina is found near pH 8, in accordance to the literature [9]. As citric acid is added, the pH at IEP (pHIEP) is changed to 7. A significant increase in the negative charges was seen when Duramax was added, changing the IEP to the acid pH range (pHIEP≅ 3).

Particle size distribution measurements (showed in Figure 2) were based on previous isoelectric point determinations. The pH of each sample was adjusted to this point and also to one corresponding to a high zeta potential (positive or negative).

Particle size distributions of pure alumina and citric acid - alumina suspensions were strongly affected by pH variation. Near IEP all distributions show higher particle size values due to particle agglomeration resulted from low superficial charges. With Duramax addition, distributions at both pHs are very close together, an indication that this dispersant causes a steric impediment avoiding the particles to come near each other even with low superficial charges.
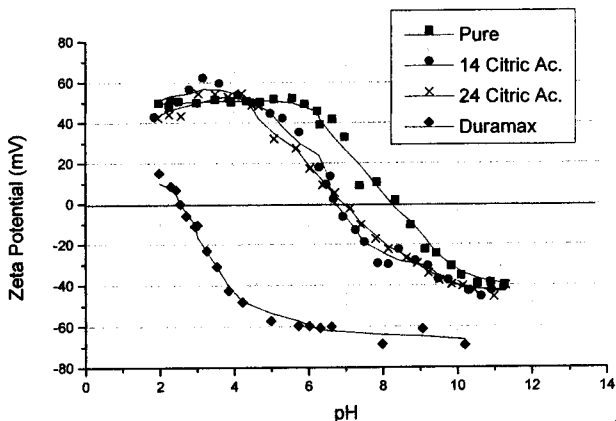
Figure 1 - Zeta Potential versus pH curves for pure alumina and alumina plus dispersants (solid lines are added only for better visualization).
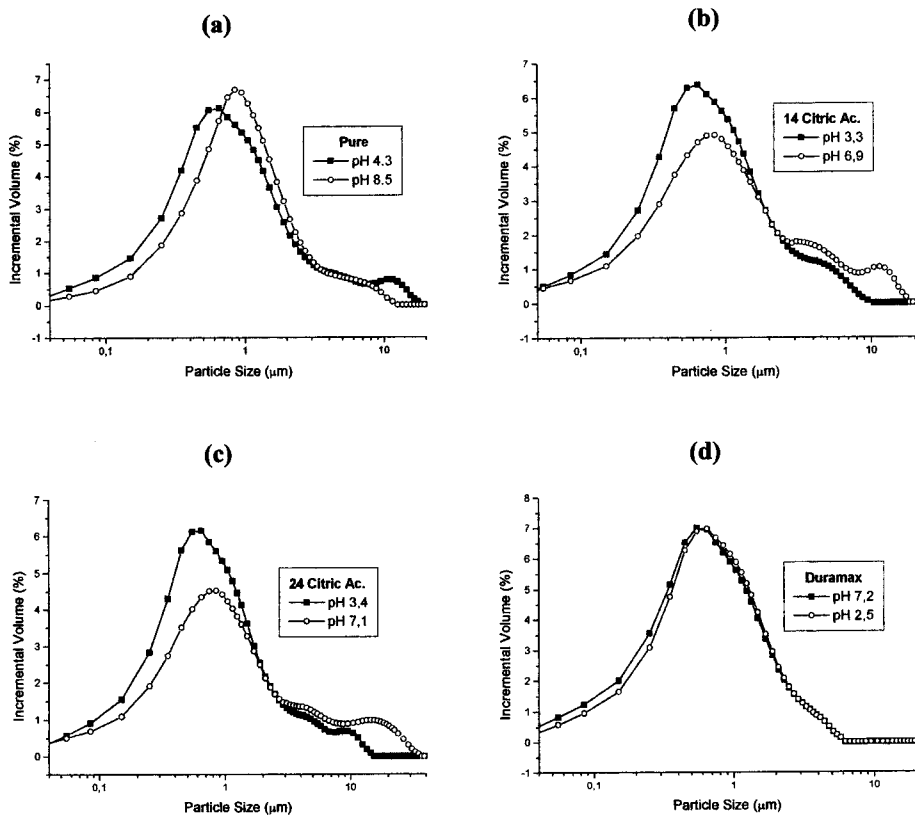

Figure 2 - Particle size distributions of alumina at near-IEP and high-zeta potential pH: (a) pure; (b) 0.14 mg/m^2 of citric acid; (c) 0.24 mg/m^2 of citric acid; (d) Duramax.

In Fig. 3 distribution curves were grouped according to the zeta potential, e.g. low zeta potential at Fig. 3a and high zeta potential at Fig. 3b. Some differences between curves can be noticed as result of different states of agglomeration. Pure alumina and citric acid suspensions show near similar distributions in both cases (low and high zeta potentials) while Duramax suspension distributions were a little dislocated to the left (low particle size). This is an indication that in this system the electrosteric impediment from Duramax caused a better dispersion condition than electrostatic action of the citric acid. Moreover, the addition of the later is irrelevant to the particle size analysis at high zeta potential (> 20mV). The presence of particles higher than 5 µm is also noticed in the pure alumina and citric acid suspension distributions, which confirms the agglomeration. This proves the superior efficiency of Duramax, once no agglomeration was observed at distribution curves with that dispersant, even at low zeta potential condition (pH 2.5).
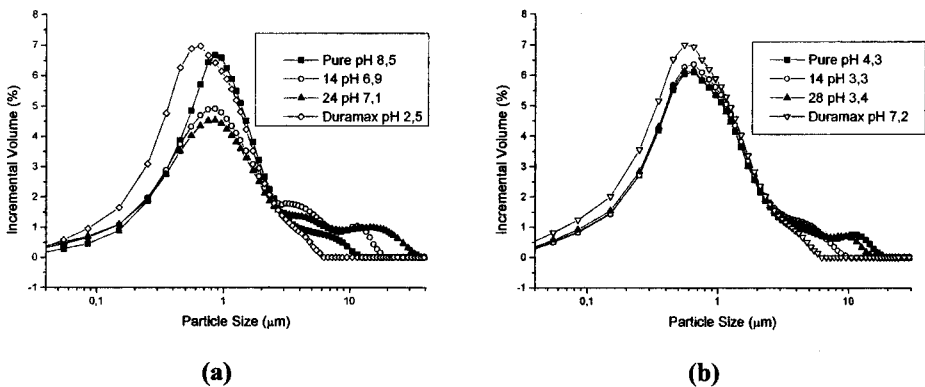

Figure 3 - Particle size distributions of alumina grouped by dispersion condition:
(a) near-IEP pH; (b) high zeta potential pH.

Supersonic vibration tests (Fig. 4) showed that supersonic waves are very efficient and useful to the particle size analysis of alumina. Dispersion state attained at pure alumina suspension with supersonic vibration however was not so high compared to the Duramax suspension (with supersonic vibration), as shown in Figure 4c, where Duramax curves exhibit just lower values.

These observations indicate that supersonic waves although useful are not enough to assure a full dispersion state. Besides, the variation of the distribution curves with the application time can be evidence that the ideal dispersion condition is not attained. Duramax alone was not able to disperse the suspension, which evidences the need of the supersonic-waves energy to the initial breaking of the agglomerated particles, allowing the adherence of the dispersant at particles surface. The reapproximation of particles is then avoided and the dispersion stability is enhanced.

Final Comments

For the case studied here, electrosteric impediment provided a better dispersion state than electrostatic activity, which is changed by the zeta potential or, primarily, the pH of the suspension. The supersonic vibration is not sufficient to assure a full dispersion, although it was necessary to the initial breakup of the agglomerates.
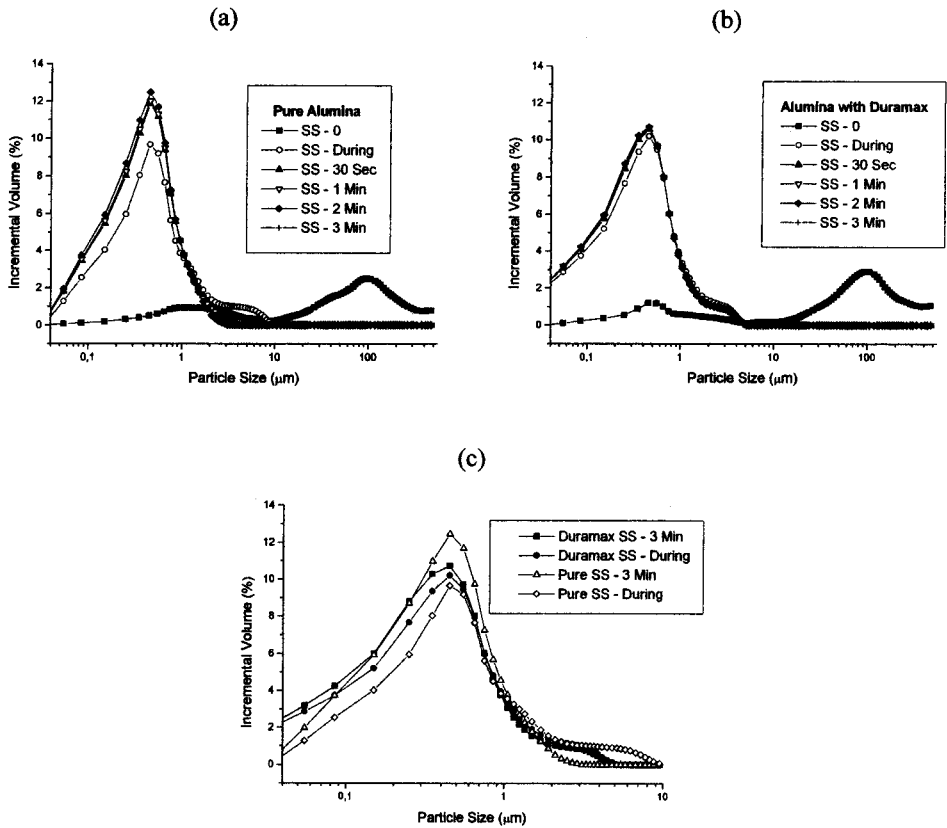
(a)　　　　　　　　　　　　　　　(b)

(c)

Figure 4 - Particle size distribution of alumina at different application times of supersonic vibration ultrasonic: (a) Pure; (b) with Duramax; (c) results grouped by supersonic vibration only during the acquisition time and for 3 minutes.

In the development of new procedures for particle size analysis, it is convenient to investigate dispersion conditions in order to get reproducible results. If no dispersant is utilized, or if the action of a particular dispersant is not known, it is advisable to make measurements changing the pH. This procedure should provide useful data about the agglomeration state and the efficiency of the dispersant.

For the reasons presented here, particle size results must be linked to the dispersion conditions of the analysis.

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