Available online at www.sciencedirect.com

ScienceDirect

Particuology 6 (2008) 120–124

PARTICUOLOGY

www.elsevier.com/locate/partic

Influence of suspension stability on wet grinding for production of mineral nanoparticles

S. Sakthivel, Venkatesan V. Krishnan, B. Pitchumani *

Particle Science and Technology Laboratory, Department of Chemical Engineering, Indian Institute of Technology, New Delhi, India

Received 4 August 2007; accepted 24 December 2007

Abstract

Grinding behavior of nanoparticles in an attritor mill and the minimum achievable particle size are strongly influenced by the suspension stability. In the present work, suspension stability (i.e. ζ-potential) of nanoparticles was studied by measuring pH as a function of grinding time in the wet milling process. It was found that after a certain time in an attritor mill, there is no further size reduction and the average product particle size increases monotonically. One of the reasons is that the production of submicron particles leads to more particle–particle interactions and consequently pH of the suspension decreases with grinding time. Usually pH value is related to suspension stability and it can be enhanced by addition of NaOH solution. The maximum negative ζ-potential of −51.2 mV was obtained at pH of 12 for silica. The higher the ζ-potential with the same polarity, higher will be the electrostatic repulsion between the particles. Hence, the maximum electrostatic repulsion force was maintained by the adjustment of pH value in wet milling. The experiments were conducted at different pH conditions which were maintained constant throughout the experiments and nanosized particles were obtained consequently.

Keywords: Suspension stability; ζ-Potential; Nanosilica particle; Attritor mill

1. Introduction

The production of micron sized particle (3% residue on 63 μm sieve) of silica was studied in an industrial wet batch ball mill. Sakthivel, Pitchumani, and Pitchumani (2008) observed by conventional grinding in an industrial ball mill that only micron sized particles are obtained, even after a long time of grinding. They mentioned that it is difficult to grind for a longer time due to the formation of paste and even ultimately of cake. Much of the published work reported on the effect of operating conditions such as ball loading, solid mass fraction, ball size and pin tip velocity on the production of micron particle in an attritor mill (Mankosa, Adel, & Yoon, 1986; Ramian & Pitchumani, 1992; Stehr & Schwedes, 1983; Thirunavukkarasu, Raghuraman, & Pitchumani, 2003). Recently production of nanoparticles reported by Mende, Stenger, Peukert, and Schwedes (2003) and Stenger, Mende, Schwedes, and Peukert (2005a), Stenger, Mende, Schwedes, and Peukert (2005b) in stirred media mill, showed that electrostatic stabilization appears to be the key factor to producing stable nanoparticle suspensions in a stirred ball mill. Sakthivel, Thirunavukarasu, and Pitchumani (2006) detailed the production of talc mineral nanoparticles with a narrow particle size distribution and spherical shape particle by controlling pH of the suspension. The average particle size is obtained 100–250 nm in batch mode.

The colloidal behavior of suspension changes with grinding time because of the production of fine particles and the change of their surface charge. The dispersion of silica powder in an aqueous solution is due to the charge being developed on the particle surface, as was confirmed by Milonjić (1987). The surface charge gives rise to surface forces that in many cases impart colloidal stability and strongly affect the rheological properties of the suspension. Suspension stability is directly dependent on the zeta potential (ζ) of the powder that represents the potential difference between the surface of the grains and the external plane of Helmholtz (see the model for the electric double layer in Fig. 1). The ζ-potential is thus defined as the electrical poten-

---

* Corresponding author.
E-mail addresses: bpmani@chemical.iitd.ernet.in, b_pitchumani@hotmail.com (B. Pitchumani).

1674-2001/$ – see inside back cover © 2008 Chinese Society of Particuology and Institute of Process Engineering, Chinese Academy of Sciences. Published by Elsevier B.V. All rights reserved.
doi:10.1016/j.partic.2007.12.001

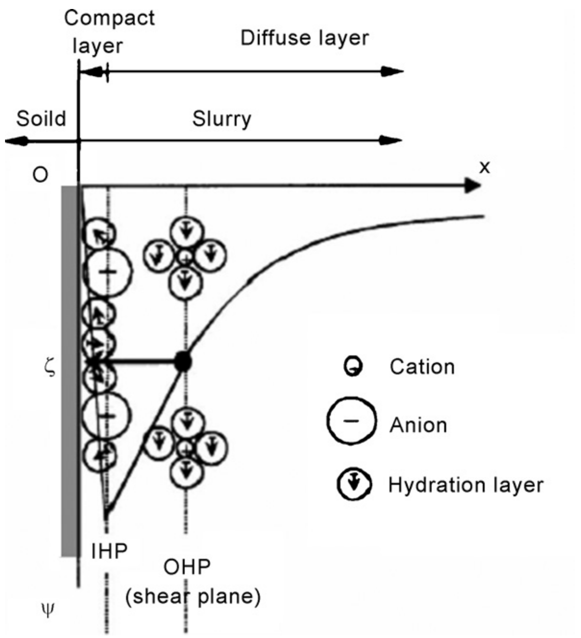

Fig. 1. Model for the electric double layer at the solid–solution interface (Vallar et al., 1999).

tial developed at the solid–liquid interface in response to the relative movement of solid particles and liquid or as the strength of the particle electrical barrier. The higher this potential with the same polarity is, the more higher the electrostatic repulsion between particles becomes. On the other hand, when the suspension is close to the iso-electric point ($\zeta = 0$), the particles tend to flocculate. Most of the mineral particles have negative surface charges, such as quartz, tin oxide and hence those mineral particles have maximum suspension stability (ζ-potential) obtained at pH values ranging from 8 to 11.

The electrostatic stabilization mechanism is based on the adsorption–desorption of ions onto/from the particle's surface, forming a charged particle surface surrounded by an electrical double layer. This mechanism has fast kinetics because of the adsorption/desorption of small ions. The attractive and repulsive particle–particle interactions are based on DLVO [Derjaguin–Landau–Verywey–Overbeek] theory and thus suspension stability is analogous in concept to the electrostatic stabilization discussed by Stenger and Peukert (2001).

The present work investigates the behavior of suspension stability (i.e. zeta potential) in wet milling with the variation of pH. Suspension stability was enhanced by addition of NaOH.

2. Experimental set-up

Grinding experiments were carried out in a laboratory vertical attritor mill in batch mode, as shown schematically in Fig. 2. The mill consists of a double walled stainless steel vessel of 900 cm^3 capacity and a stirrer with four cylindrical rods, and driven by a 1 HP motor. The grinding chamber is kept on a movable adjustable platform for centering the shaft of the grinding chamber. A 5-mm gap was maintained between the tip of the shaft and the bottom of the grinding chamber, which was also designed to facilitate charging of the grinding media and slurry and emptying the mill. The speed of rotation was checked with a non-contact tachometer. The mill is sealed from the top. Stainless steel balls, 2 mm in diameter and 7.24 g/cm^3 in density, were loaded as grinding media up to 50% loading. The pin tip tangential velocity was maintained at 7.1 m/s.

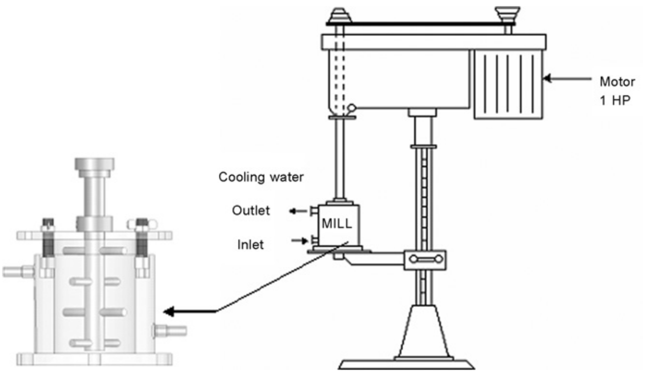

Fig. 2. Schematic diagram of attritor mill.

2.1. Material and suspension preparation

The feed material of silica slurry sample to the attritor mill was obtained from a ceramic tile plant. The density of this material is 2250 kg/m^3. The silica slurry was prepared with distilled water with a solid mass fraction of $C_m = 0.40$, 0.35 and 0.31, respectively. The initial pH of the sample with $C_m = 0.40$ was noted to be 9.8.

2.2. Characterization

The feed sample was well mixed and then the material was measured in a Malvern^{TM} laser particle size analyzer. The average size of the feed material was measured 116 μm.

For zeta-potential measurement, using ZETASIZER Malvern^{TM}, silica slurry samples were prepared at different pH values by drop wise addition of sodium hydroxide (NaOH) and/or nitric acid (HNO_3). The pH of the suspension was measured by a digital pH meter after vigorous stirring with a glass rod until the pH of the suspension became constant, which may take about 3 min.

3. Results and discussion

3.1. Effect of solid mass fraction

Particle size distributions of the product were measured at grinding time of 10, 20, 30, 40, 60, 90, 120 min with a laser particle size analyzer and then the average particle size, X_{50} was calculated. Other operating conditions such as ball loading, pin tip velocity and ball size were maintained constant throughout the experiment. Experiments were conducted at different solid mass fractions ($C_m = 0.40$, 0.35, 0.31). Fig. 3 shows the variation of average particle size with grinding time, indicating that average particle diameter decreases with time up to a grinding