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SHORT COMMUNICATION

Effect of ball size on milling efficiency of zinc oxide dispersions

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ABSTRACT
Zinc oxide (ZnO) was wet milled using inert Al_2O_3-ceramic balls having different diameter at different milling intervals and the milling efficiency of the resultant dispersion was followed through particle size analysis and zeta potential measurements. The results indicated that small-sized balls improved the milling efficiency. The highest share (%) of lower-size particles was obtained after 24 h of ball milling.

KEYWORDS
Ball milling; ball size; ultra-fine zinc oxide; zeta potential

Introduction

Nano zinc oxide (ZnO) is one of the multifunctional inorganic materials with broad range of applications. Preparation of stable dispersions of ultra-fine ZnO particles is a subject of interest among researchers. Preparation of ZnO dispersion using ball milling has been studied extensively (Glushenkov and Chen 2006; Giri et al. 2007; Salah et al. 2011; Amirkhanlou, Ketabchi, and Parvin 2012). The optimum milling conditions for the preparation of ultra-fine ZnO dispersions have been reported (Anand, Varghese, and Kurian 2015). The aim of the present research work is to study the effect of balls having different diameters on wet milling of ZnO dispersion. The average particle size and stability of wet-milled ZnO using different ball sizes as a function of milling time is also explored.

Experimental

ZnO used in this study has an average particle size (D50) of 2–4 μm. D50 represents maximum particle diameter below which 50% of the sample volume exists. It is also known as mass median diameter (MMD) or the median of the volume distribution. Disodium methylene *bis*-naphthalene sulphonate (Dispersol-F) was used as the dispersing agent. A 50% aqueous dispersion of ZnO having 2% Dispersol-F was prepared in a steel cylindrical vessel and ball milled. The balls used are ¼, ½, and ¾ inches in diameter. In all the cases, the ball to powder weight ratio has been kept at 5:1. The speed of the jar rotation was 35 rpm. Ball milling was carried out for different periods say 0.5, 6, 12, 18, 24, and 30 h. The average particle size of wet-milled ZnO dispersion was measured at frequent intervals using a Mastersizer 3000 (Malvern Instruments, UK) particle size analyzer. Zetasizer (Nano Z, Malvern Instruments, UK) was used to measure the zeta potential of the dispersions.

The ball-milled ZnO dispersions (wet milled using balls having different diameters) were withdrawn at different milling intervals and are used for zeta potential measurements. The dispersions were diluted to very low concentration using distilled water and stirred well. The diluted samples were taken in a syringe and carefully filled in the cuvette for zeta potential measurements. Care has been taken to avoid air bubbles and foaming inside the cuvette.

Results and discussion

Figure 1a–c represents the cumulative distribution curves of particles of ZnO dispersions milled with balls having diameter ¼, ½, and ¾ inches, respectively. From Figure 1a it is obvious that, small-sized balls shift the curve toward the left side (i.e., particle size decreases). As the ball size increases, with increase in milling time, curve shifts toward right side (particle size increases). Larger balls provide greater free volume in between the balls. This results in coarsening of particles. Further, larger balls deliver low collision rate, as a result the bonding mechanism dominates over fracture mechanism (Eom et al. 2011). However, irrespective of the ball size, 30 h of wet ball milling generated larger particles. The results are in accordance with our earlier findings (Anand, Varghese, and Kurian 2015).

The specific surface area (SSA) of wet ball-milled ZnO dispersion with different ball sizes as a function of milling time is shown in Figure 2. In the beginning of ball milling (up to 6 h), irrespective of the ball size, sharp increase in SSA of ZnO was observed followed by a sharp decline. An increase in SSA of ZnO is observed after 18 and 24 h of wet milling with balls having ¼ inch diameter. However, irrespective of the ball size, reduction in SSA was observed after 30 h of milling. The decrease in SSA after prolonged ball-milling indicates that the ultra-fine particles undergo aggregation (Anand, Varghese, and Kurian 2015).

The average particle size (D50) of the wet-milled ZnO dispersion is shown in Figure 3 as a function of milling time for different ball sizes. For the first 12 h of ball milling, no

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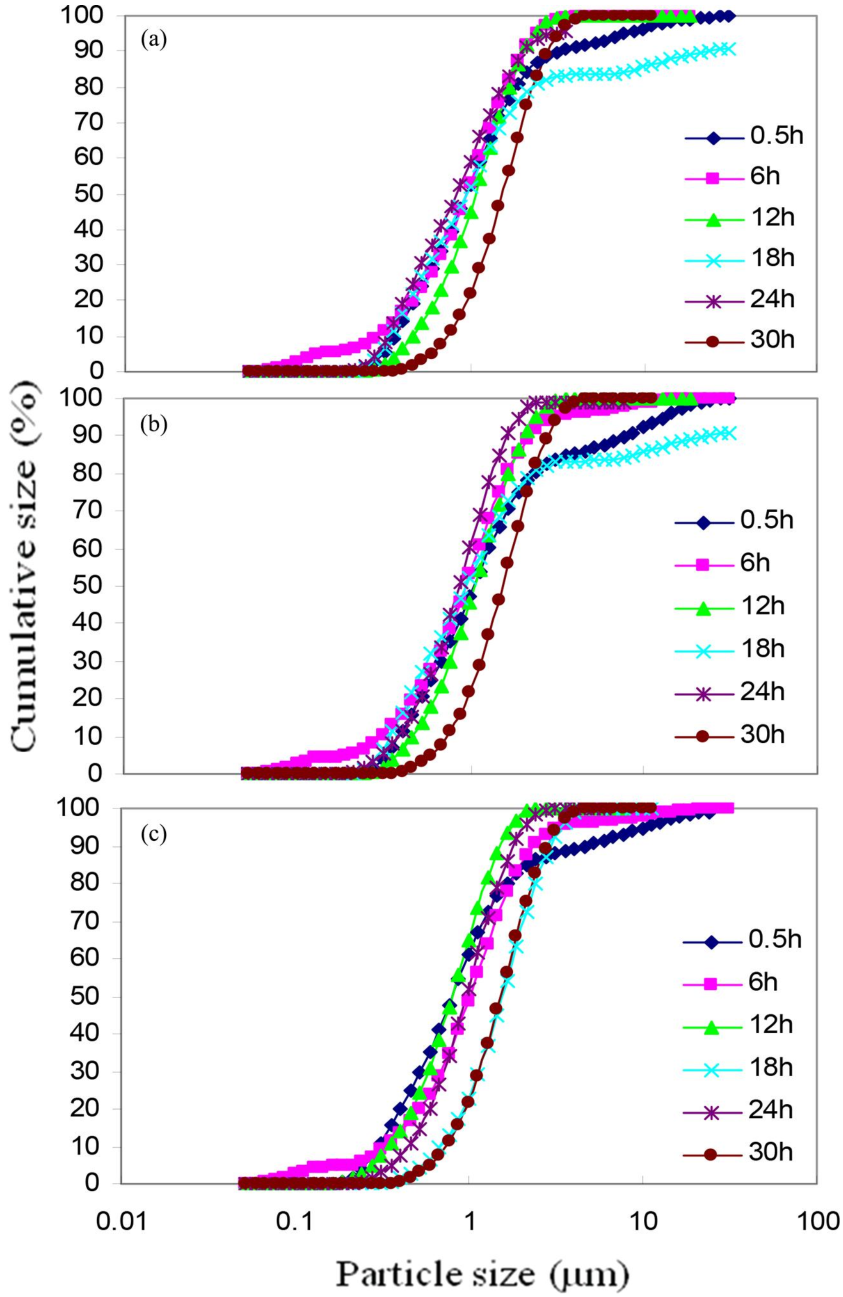

Figure 1. Cumulative distribution curves of ZnO wet ball milled with balls having different diameters: (a) with ¼″ balls, (b) with ½″ balls, and (c) with ¾″ balls.

significant variation in mean particle size is seen with ball size. The optimum size of 0.98 μm has been achieved after 24 h of ball milling using ¼ inch balls. Reduction of particle size would result in an increase in the formation of fresh surfaces (Du et al. 2013). Reduction in particle size after 18 h of grinding with small-sized balls indicates that the rate of formation of fresh surfaces is high compared with other cases. Further increase in milling time resulted in an increase in particle size. This might be due to cold welding of particles caused by the repetitive shear forces (Eom et al. 2011). In the case of ½ inch and ¾ inch balls also, increase in D50 is observed after 18 h of milling.

When the particle becomes smaller, the milling efficiency decreases with increase in milling time (Johansson 2012). As the milling process advances, the temperature goes up very fast and the impact of energy will not be enough to break down the particle. Results on mechanical alloying of Al powder shows that it started to agglomerate when the milling time was increased up to 40 h (Ramezani and Neitzert 2012). From this result, it can be said that the optimum milling time (24 h) for ZnO results in efficient milling, when the ball size is ¼ inch in diameter.

The size (%) of ZnO (particles having size < 0.5 μm) after milling with balls having different diameters is shown in Figure 4. It can be seen that size (%) of ZnO ball milled with ¼-inch balls was high in comparison with balls having larger diameter (½ and ¾ inches). The highest share of 23.4% was achieved after 24 h of milling. Further increase in milling time resulted in a drastic reduction in the share of lower-sized particles. As the ball size decreases, more surfaces are generated and results in effective impacts. The particle breakage

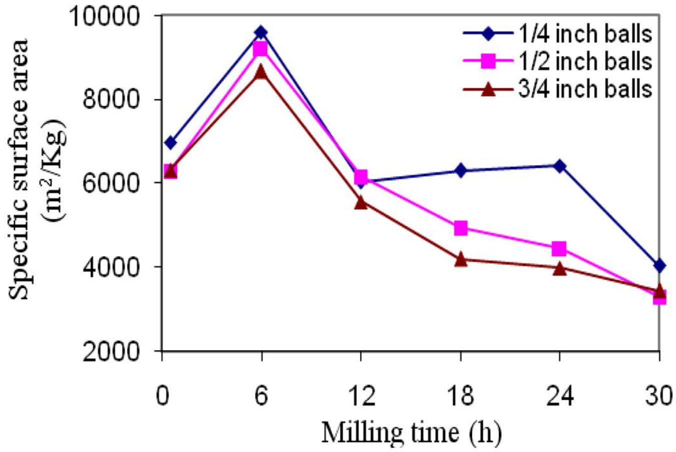

Figure 2. The specific surface area of ZnO for varying ball size as a function of milling time.

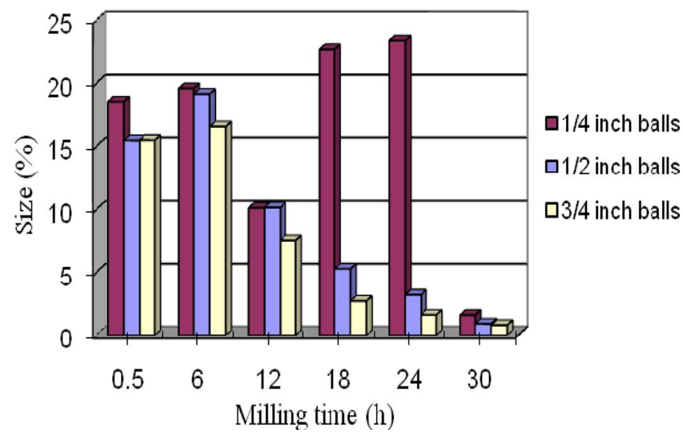

Figure 4. Size (%) of ZnO (size < 0.5 μm) with milling time for different ball sizes.

takes place mostly at the contact sites of the balls. Hence, smaller balls possess a high milling efficiency because of the increased number of contact points between the balls (Shin et al. 2013). The share of particles ball milled using ¾-inch balls is low. Interestingly, after 6 h of wet milling of ZnO with balls having different diameters, the share of particles ball milled with ¼ and ½ inches balls is almost same. This shows that at lower milling time (up to 12 h), the effect of ball size on particle size reduction of ZnO is not much significant. Figure 3 supports the result. However, increased milling time (18 and 24 h) using balls with smaller diameters gave high share to lower-sized particles showing effective impacts predominate over collision.

The colloidal stability of wet ball-milled ZnO with different ball sizes as a function of milling time is shown in Table 1. Zeta potential measurement brings detailed insight into the factors affecting the stability of dispersion, aggregation/flocculation, etc. The zeta potential of wet ball milled ZnO using different ball sizes did not change significantly. However, with increase in milling time, reduction in zeta value was observed in all cases. This indicates that the agglomeration tendency of the particles upon prolonged milling is high because zeta potential values are low.

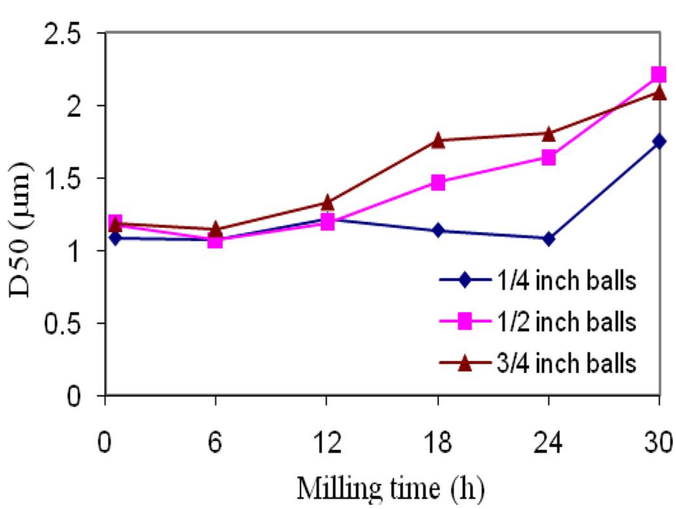

Figure 3. Variation in the mean particle size (D50) of ZnO with the milling time for different ball sizes.

Table 1. Magnitude of the zeta potential with ball sizes at various milling time.

	Zeta potential (mV)		
Milling time (h)	¼″ balls	½″ balls	¾″ balls
0.5	−40.4	−44	−42
6	−40.8	−39.8	−39.8
12	−39.1	−39.6	−39.3
18	−38.7	−37.2	−36.1
24	−36.8	−38	−35.6
30	−33.3	−35	−33.2

Conclusions

ZnO was ball milled in aqueous medium using inert Al_2O_3 ceramic balls having different diameters (¼, ½, and ¾ inches) for different intervals (0.5, 6, 12, 18, 24, and 30 h) and the milling efficiency was analyzed. It was found that small-sized balls increased the milling efficiency and it increases with increase in milling time. At lower milling time (up to 12 h), the average particle size (D50) of ZnO did not vary significantly with ball size. The highest share of lower-sized particles (23.4%) was achieved with ¼-inch balls after 24 h of milling. The zeta potential of ZnO particles wet milled using different ball sizes was nearly the same.

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