Changing $BaCl_2$ particles size by varying ionic ratios of the reactants

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Abstract: The initial concentration ratios of reactants were varied in order to investigate their influence on decreasing mean particle size and morphology of $BaCl_2$ particles in a stirred tank reactor. The experiments were carried out via a simple precipitation reaction of calcium chloride and barium sulfate. Samples were characterized with scanning electron microscopy (SEM). Based on the results obtained, varying ionic ratios of the reactants (Ba to Cl (R)) significantly changes the resulted barium chloride particle size.
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1. Introduction

The chemical synthesis of inorganic materials of specific size and morphology has attracted considerable attention due to the potential to design new materials and devices. Small Particles has generated important scientific interest because of the advantages resulting from the size reduction. Barium chloride widely used in the plastics, rubber, paint and oil industries (Joselevich and Willner, 1994; Qi et al., 2000; Rautaray et al., 2002; Yu et al., 2004).

Precipitation of barium chloride has been widely studied in the past decades while numerous theoretical and experimental research studies are reported about the role of initial conditions and about the influence of reactor geometries and mixing conditions on resulting particle size or to test precipitation models (Cheng et al,. 2004; Pohorecki and Baldyga,1983; Jianfeng et al,. 1996; Schwarzer and Peukert, 2004; Aoun et al,. 1999).

However, most of these studies were carried out under stoichiometric conditions, with mixing affecting particle size distribution. This is the first report on investigating non-stoichiometric concentrations of reactants on the average size and morphology of the barium chloride particles in stirred reactor tank.

2. Material and Methods

Analytical grade barium sulfate and calcium chloride were purchased from the Merck, (Germany). Synthesis of $BaCl_2$ particles took place in a semi-batch reactor mode. A schematic diagram of the reactor used for this study is shown in Fig.1 which consists of a stirred 1 Litter Pyrex tank with water jacket for temperature control equipped with 4 baffles and Rushton turbine impeller. The feed input was located below the initial liquid level. Effect of excess ion concentration by adding $BaSO_4$ or $CaCl_2$ as feed to the tank containing the opposite component was investigated.

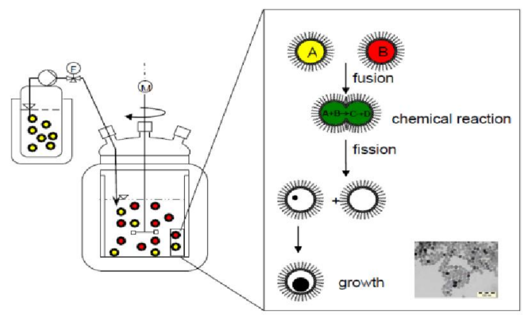


Figure 1. Schematic diagram of single-feed semi-batch precipitation system

3. Results

In each run, spontaneous precipitation was initiated by adding always 300 ml of feed solution to 300 ml of second reagent present in the reactor. While the feed concentration was adjusted from 0.1 to 1 M, the solution concentration in the tank was always kept constant at 0.1 M to provide the total molar ratio (see Table 1). Therefore, both globally stoichiometric and non-stoichiometric precipitation conditions were investigated and the total barium to chloride ions molar ratio was varied from 1 to 1:10 when $BaSO_4$ used as a feed or 1 to 10 while using $CaCl_2$ as feed. In all the experiments the feeding rate was 10 ml/ min, the reaction temperature 40 °C and the stirring speed 600 rpm were kept constant. In all cases barium chloride precipitates were formed instantly. After feeding was completed, samples were taken from the reaction mixture. All precipitation experiments were conducted at least three times to assess the reducibility of the results. The precipitates were separated from the original solution by centrifugation and washed with distilled water several times. The precipitates were then dried at 100 °C. Size analysis was performed scanning electron microscopy (LEO 1455 VP, UK) with an accelerating voltage of 10 kV.

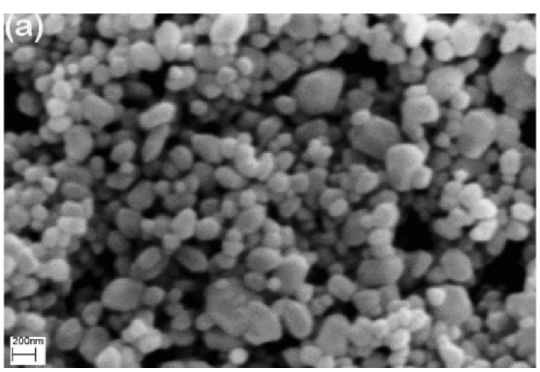
Table 1. Effect of molar ratio on mean particle size with two feeding modes.

Feed (M)	$BaSO_4$	$CaCl_2$	Molar ratio (R)	Average particle size (nm)
$BaSO_4$	0.1	0.1	1:1	1658±1.2
$BaSO_4$	0.2	0.1	1:2	534±1.4
$BaSO_4$	0.4	0.1	1:4	542±2.2
$BaSO_4$	0.6	0.1	1:6	223±1.6
$BaSO_4$	0.8	0.1	1:8	142±1.8
$BaSO_4$	1	0.1	1:10	146±1
$CaCl_2$	0.1	0.1	1:1	1695±1.3
$CaCl_2$	0.1	0.2	2:1	610±0.9
$CaCl_2$	0.1	0.4	4:1	480±1.1
$CaCl_2$	0.1	0.6	6:1	220±1.5
$CaCl_2$	0.1	0.8	8:1	159±1.2
$CaCl_2$	0.1	1	10:1	162±1

Values are reported as mean ± standard deviation.

Table 1 clearly shows the mean size, d (0.5), obtained at various molar ratios of barium to chloride (R). The mean crystal size generally decreased as the mean concentration of excess ions increased in the reactor. Bigger mean sizes and wider size distributions were obtained for the total molar ratios close to stoichiometric (R= 1 to 1:6) and with further increase from the stoichiometric conditions (R= 1:6 to 1:10) smaller mean sizes and size distributions were found, while using $BaSO_4$ as feed, also, feeding with $CaCl_2$ far from the stoichiometric condition (R= 6 to 10) has the similar result on mean particle size and size distribution when compared with the case of feeding with $BaSO_4$.

Fig. 2 shows a comparison between the smallest particles obtained by adding (2 M) $BaSO_4$ (Fig. 2a) and 2 M $CaCl_2$ (Fig. 2b) as feed. Uniform rod-like barium chloride particles were obtained in addition to spherical particles in both pictures. No significant changes in the shape or width of the distributions are observed.



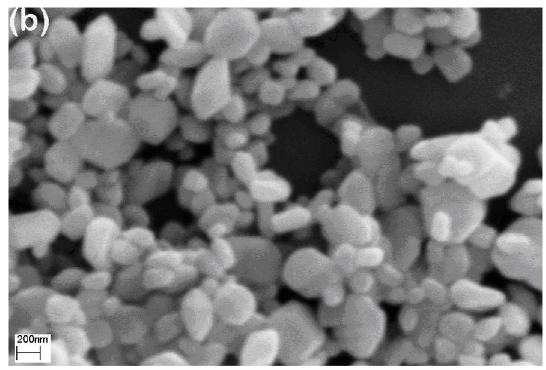


Figure 1. Schematic diagram of single-feed semi-batch precipitation system

4. Discussions

In this paper, effect of the initial concentration ratio of the two reactants on product properties consisting of particle size and morphology were elucidated. It can be concluded that the molar ratio of barium to chloride (R) proved to be the significant parameter in controlling the particle size and may also influence nucleation and growth rates. The smallest precipitate particles are obtained by feeding with barium sulfate or calcium chloride far from the stoichiometric condition. The effect of adding $BaSO_4$ or $CaCl_2$ as feed on the morphology of barium chloride particles far from the stoichiometric condition (R= 1:10 and 10:1) was also investigated. However, it was found that there were no obvious morphological differences.

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