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Experimental investigation and process intensification of barium sulfate nanoparticles synthesis via a new double coaxial spinning disks reactor

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ABSTRACT

In this article, a new double spinning disks reactor (DSDR) has been proposed and tested successfully for the synthesis of barium sulfate nanoparticles by means of the reactive precipitation. The proposed DSDR consists of two coaxial rotating disks placed horizontally in a cylindrical chamber. Continuous precipitation of barium sulfate nanoparticles as a chemical test system was carried out using this new contacting device and the effects of operating and design parameters such as the disk rotational speed, distance between the disks, feed concentration, feed flow rate, free ion ratio, feed location, and feed distribution pattern on the mean size, size distribution, and morphology of the synthesized barium sulfate nanoparticles were investigated. It was found that the micromixing enhancement obtained by the new DSDR leads to a better performance for the synthesis of nanoparticles with desired mean size and particle size distribution (PSD). Moreover, $BaSO_4$ nanoparticles with the mean size of 23.4 nm were synthesized.

1. Introduction

Over the recent decades, chemical engineering processes have passed an evolutionary period called process intensification (PI). Process intensification as defined by Stankiewicz and Moulijn [1], is “development of innovative apparatuses and techniques that offer drastic improvements in chemical manufacturing and processing, substantially decreasing equipment volume, energy consumption, or waste formation, and ultimately leading to cheaper, safer, and sustainable technologies”. Although PI generally includes all levels of design from chemical system to process design, it mostly refers to the development of new devices with higher performance and less power input requirement. Microchannel reactors [2,3], confined impinging jet reactors (CIJRs) [4,5], rotating packed bed reactors (RPBRs) [6,7], and spinning disk reactors (SDRs) [8–10] are typical examples of contacting devices already proposed for PI and their performances were extensively investigated for various processes from experimental and simulation points of view.

On the other hand, because of the desirable properties of nanoparticles arising from their high surface-to-volume ratio, and hence their high surface energy, the synthesis of nanoparticles has attracted extensive attention. Nanoparticles have found a wide range of applications in ceramic [11], metallurgy [12], electronic [13], optic [14], environmental [15], pharmaceutical [16], photocatalysis [17], and food [18] industries.

Among various methods for the synthesis of nanoparticles, the reactive precipitation process is of great interest. This is due to the simplicity, possibility of mass production, and low cost. Because of the fast kinetics of precipitation process, it is almost impossible to keep the size of synthesized particles in the nanometric range, when the process takes place in the bulk mode. Moreover, because of the non-uniformity of species concentrations within the solution, particles with a wide PSD and various morphologies can be synthesized. Thus, the synthesis of nanoparticles by the precipitation process is usually carried out in narrow channels [19], thin annular ducts [20], small templates [21], etc.

The synthesis of nanoparticles through the reactive precipitation can be carried out in highly agitated contacting devices with high micromixing efficiencies. In the context of PI, numerous configurations have been proposed to improve the performance of precipitation process. Among them, spinning disk reactors (SDRs) with large transfer rates and short mixing times provide a suitable performance in the synthesis of nanoparticles. This is due to the high centrifugal and shear forces exerted on the developed thin

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Nomenclature

a Activity, (mol/m^3)
A Nucleation constant, (−)
B Nucleation rate, $(1/(m^3\,s))$
B^0 Initial nucleation rate, $(1/(m^3\,s))$
C Concentration, (mol/m^3)
C_1 Concentration of $BaCl_2$ in the feed, (mol/m^3)
C_2 Concentration of Na_2SO_4 in the feed, (mol/m^3)
d Disk diameter, (m)
D Mean particle diameter, (nm)
G Growth rate, (m/s)
h Distance between the disks, (m)
k_g Growth rate constant, (m/s)
K_{sp} Solubility product, (mol^2/m^6)
N_f Number of feed points, (−)
Q Feed flow rate, (m^3/s)
Q_1 $BaCl_2$ flow rate, (m^3/s)
Q_2 Na_2SO_4 flow rate, (m^3/s)
r_2 Feed point radius, (m)
R Free ion ratio, (−)
S_a Activity-based supersaturation, (−)
t_{ind} Induction time, (s)

Greek letters
β Half width of the peak, (degree)
$\gamma_\pm$ Mean activity coefficient, (−)
θ Bragg's diffraction angle, (degree)
λ X-ray wavelength, (nm)
ω Disk rotational speed, (rad/s)
ω_{ud} Upper disk rotational speed, (rad/s)
ω_{ld} Lower disk rotational speed, (rad/s)

liquid film with high micromixing efficiency [22–24]. Various nanoparticles such as $BaSO_4$ [25], Ag [26], AgI [27], ZnO [28], CuO [29], TiO_2 [30], hydroxyapatite [31], and magnetite [32] were synthesized through the reactive precipitation in SDRs. The synthesis of barium sulfate has been widely used to evaluate the precipitation process performance, micromixing efficiency, and precipitation models. Table 1 compares the results of this study with those obtained by other researchers regarding the synthesis of barium sulfate particles via SDRs. Cafiero et al. [33] have shown that by using an SDR, micromixing times smaller than the induction time can be achieved for the $BaSO_4$ precipitation. They synthesized barium sulfate particles in the range of 0.5–1 μm and mentioned that this could be achieved with a lower energy consumption compared to other contacting devices with higher power input requirements. Molaei Dehkordi and Vafaeimanesh [25] have investigated the effects of several operating and design parameters such as the supersaturation, rotational speed, free ion ratio, and disk diameter on the size and morphology of $BaSO_4$ particles synthesized in an SDR. They achieved various morphologies for barium sulfate crystals and reported that nanoparticles with mean sizes down to 38 nm could be synthesized. Jacobsen and Hinrichsen [23] have also synthesized $BaSO_4$ nanoparticles with mean sizes down to 27 nm in an SDR. They increased the disk rotational speed to 5000 rpm and used multiple feed zones. They also examined the effects of disk rotational speed, feed location, feed flow rate, disk surface structure, and number of feed points. They reported that SDRs could be appropriate equipment for the controlled synthesis of nanoparticles because of their high micromixing efficiency.

In this work, a new double spinning disks reactor (DSDR) has been proposed and its performance has been tested for the synthesis of $BaSO_4$ nanoparticles with a narrow PSD. In this regard, the effects of different operating and design parameters on the mean size, PSD, and morphology of particles were investigated. The experimental results indicate that by using this new device, not only smaller particles with a narrower PSD can be achieved, but also the nanoparticles can be obtained with a lower rotational speed compared to the conventional SDRs. This is attributed to the enhancement of micromixing efficiency achieved by the DSDR.

2. Theory

Precipitation or reactive crystallization can be regarded as the synthesis of poorly soluble salts using chemical reactions. Generally, the precipitation process includes various phenomena such as nucleation, growth, aggregation, and breakage among them nucleation and growth are primary phenomena. The rates of the primary phenomena mainly depend on the level of local supersaturation within the solution. On the other hand, the rates of the secondary phenomena such as aggregation and breakage are affected by the collision frequency of synthesized particles and the stress forces exerted on them. Therefore, it is possible to introduce primary particles as the uniform identities produced through nucleation and growth and distinguish them from secondary particles, synthesized by aggregation and breakage [33].

In the present work, to evaluate the performance of the proposed DSDR, the precipitation of barium sulfate with the following reaction was studied:

$$BaCl_2 + Na_2SO_4 \rightarrow BaSO_4 \downarrow + 2NaCl \quad (1)$$

The driving force of the precipitation process like the other crystallization processes is supersaturation. When the reactant

Table 1
The operating conditions, design parameters, and characteristics of the synthesized barium sulfate particles in SDRs.

Authors	Rotational speed (rpm)	Total flow rate (mL/min)	Initial supersaturation	Feed entrance radius (mm)	No. of feed points (#)	Disk diameter (cm)	Finest particle size (PSD) (nm)	Min. rotational speed for mean size < 100 nm (rpm)
Cafiero et al. (2002) [33]	100–1000	~160	2000	50	1	50	~700 (500–1000)	–
Molaei Dehkordi and Vafaeimanesh (2009) [25]	500–1500	160	400–2000	10	1	15, 20	~38 (20–130)	1000 (for 100 nm)
Jacobsen and Hinrichsen (2012) [23]	100–5000	180–360	990	10, 20, 30	1, 8	10	27 (not available)	2000 (for 100 nm)
Present work	500–4750	200–400	1500–14000	0, 10, 40, 70	1, 8, 16	20	23 (10–45)	500 (for 60 nm)

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