Research Article

Hydrodynamic Cavitation-Assisted Synthesis of Nanocalcite

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A systematic study was made on the synthesis of nanocalcite using a hydrodynamic cavitation reactor. The effects of various parameters such as diameter and geometry of orifice, CO₂ flow rate, and Ca(OH)₂ concentration were investigated. It was observed that the orifice diameter and its geometry had significant effect on the carbonation process. The reaction rate was significantly faster than that observed in a conventional carbonation process. The particle size was significantly affected by the reactor geometry. The results showed that an orifice with 5 holes of 1 mm size resulted in the particle size reduction to 37 nm. The experimental investigation reveals that hydrodynamic cavitation may be more energy efficient.

1. Introduction

The effect of acoustic cavitation on different chemical reactions is well established. Gedanken [1] has reviewed the use of sonochemistry for fabrication of inorganic nanomaterials of various shapes, size, structure, and phases. Acoustic cavitation in liquids leads to two of major effects: physical (streaming, turbulence, microjet, shear, etc.) and chemical (radical production). While acoustic cavitation-induced chemical reactions have been successfully achieved, hydrodynamic cavitation is found to be efficient for applications involving continuous processing such as industrial carbonation operation. It is expected that hydrodynamic cavitation would increase the rate of carbonization reaction by lowering the mass transfer resistance. Hydrodynamic cavitation, in which a liquid is passed through constrictions, such as orifice plate or Venturi, has been found useful in specific chemical reactions. Hydrodynamic cavitation occurs due to the changes in the pressure of liquid flow in a pipe fitted with orifice or Venturi. A liquid experiences a sudden drop in pressure at downstream resulting in the collapse of formed cavities. The collapse of the cavities generates

highly reactive radicals, which are responsible for specific chemical reactions. In gas-solid reactions, the dissolution of solids is enhanced due to the turbulent mixing generated by hydrodynamic cavitation. The vigorous mixing enhances the transport of gas solutes to the solid surface that results in an increase in the mass transfer and hence the overall reaction rate [2–4].

Hydrodynamic cavitation has been found useful in the hydrolysis of fatty oils [5] and polymer solutions [6] and in the formation of styrene butadiene rubber nanosuspensions [7]. Morison and Hutchinson [8] have shown the limitations of the Weissler reaction as a model reaction for measuring efficiency of hydrodynamic cavitation. Senthil kumar et al. [9] and Moholkar et al. [10] have reported that the generation of cavities in a hydrodynamic reactor is very much dependent upon the design and the geometry of the reactors. Gogate and Pandit [11] have reviewed the effect of hydrodynamic cavitation on different industrially important reactions, such as the oxidation of toluene, xylene, and transesterification. Suslick et al. [12] studied dependence of tri iodide formation rate on the hydrodynamic pressure used to induce cavitation. Find and Moser [13] have reported on

FIGURE 1: (a) Hydrodynamic cavitation setup for nano- CaCO_3 production. (b) Orifice Configurations used for Cavitation.

the hydrodynamic cavitation-assisted synthesis of Cu-Zn-Al metal oxides using a mixture of aqueous Na_2CO_3 and NaOH solutions by precipitation.

Calcium carbonate synthesis is one typical example of the carbonation process, in which CO_2 gas is bubbled through $\text{Ca}(\text{OH})_2$ slurry, which is also known as “reactive crystallization process.” Lin et al. [14] studied the mass transfer effect for the reactive crystallization of calcite crystals in the presence of sodium tripolyphosphate in a multiphase system. The effect of ultrasound on the crystallization process has been extensively studied [15]. Sonawane et al. [16] synthesized nanosized CaCO_3 particles using a sonochemical carbonation process in which CO_2 gas was passed through a hole of an ultrasonic horn to achieve effective micromixing during the synthesis.

To the authors’ knowledge, no systematic study has been carried out on the hydrodynamic cavitation-assisted synthesis of CaCO_3 . Hence, the aims of the current investigation were to synthesize CaCO_3 nanoparticles using hydrodynamic cavitation under various experimental conditions and to study the effect of parameters such as reactor design on the crystal size and size distribution.

2. Experimental

2.1. Experimental Apparatus and Procedure. The experimental assembly (Figure 1(a)) consisted of a closed loop reactor with a holding tank of 5 L volume, a centrifugal pump (2880 RPM, 0.5 HP/ 0.37 KW) to recycle the $\text{Ca}(\text{OH})_2$ slurry, and two flanges for holding the orifice plate. Downstream pipe (20 mm dia) was made up of a transparent acrylic material in order to observe the generation of cavities. Upstream pipe was made of (SS 316) stainless steel. Pressure gauges were provided to measure the inlet pressure (P_1) and fully recovered pressure (P_2). Three different orifice plates and one having different geometry were used in the present study (Figure 1(b)). CO_2 (99.9% pure) gas was introduced

at different flow rates from a hole drilled at the downstream part of pipe. Samples were withdrawn after every 3 minutes interval and were titrated against HCl and the pH and conductivity were measured. $\text{Ca}(\text{OH})_2$ of analytical grade was procured and used. HPLC grade water (Millipore) was used for preparing all the slurry suspension and filtered to get uniform particle size of $\text{Ca}(\text{OH})_2$. The recycling of slurry enabled the particles to be in suspension without settling at the bottom. The diameter of the orifice was calculated using C_v values which was calculated from P_2 (downstream pressure), ρ (density of water), V_0 (average velocity near orifice), and P_v (vapor pressure of water) [17].

2.2. Structural Properties and Particle Size Characterization. Structural properties of the CaCO_3 synthesized by the hydrodynamic cavitation process were evaluated using powder XRD (Philips PW 1800). The Cu- $K\alpha$ radiation (LFF tube 35 kV, 50 mA) was selected for the analysis. Transmission electron microscopy (TEM) was performed on Technai G20-stwin working at 200 kV. The measurement of particle size distribution of the nanocrystals was done by dynamic light scattering technique (via Laser input energy of 632 nm).

2.3. Cavitation Number (C_v). In order to quantify various cavitation conditions and to represent appropriately the intensity of cavitation, the concept of cavitation number was utilized [6, 17].

The cavitation number is a dimensionless quantity defined as

$$C_v = \frac{(P_2 - P_v)}{(0.5 \times \rho \times V_0^2)}, \quad (1)$$

where C_v is the cavitation number; P_2 is the downstream pressure; P_v is the vapor pressure of water; ρ is the density of water at 25°C , V_0 is the average velocity near orifice.

For different orifice sizes C_v values were calculated using (1) and presented in Table 1. Using appropriate orifice

diameter and flow rate to reduce the pressure at vena contracta to fall below the threshold pressure for cavitation (which in many cases is the vapor pressure of water at that temperature) millions of cavities could be produced. The collapsing noise of cavities was heard using stethoscope in all the experiments when an orifice was used.

3. Results and Discussions

Prior to incorporating orifice into the experimental assembly, it was necessary to carry out a set of experiments to find out the consumption of Ca(OH)_2 and the corresponding changes in the solution pH and conductivity (Figure 2). The above three variables were measured for 4% Ca(OH)_2 and 5 l/min flow rate of CO_2 . All three plots (Figures 2(a), 2(b), and 2(c)) show three distinct regions corresponding to an induction period, nucleation, and precipitation. In the first region, nuclei are formed in the reaction mixture. In the second region nucleation around the crystals takes place, in the third region precipitation process occurs. In case of Ca(OH)_2 consumption (Figure 2(a)), first constant period is observed as the solution is saturated with Ca(OH)_2 . The initial induction period (Figure 2(a)) where no change in $[\text{Ca}^{2+}]$ was observed indicates that there was no spontaneous nucleation when the reaction was carried out without the orifice. The nucleation process appears to be slow and it takes relatively longer time (about 15 minutes) for the reaction to complete when the reaction was carried out in the pipe without orifice. It can also be noticed that the carbonation process was not 100% complete and there was some residual Ca(OH)_2 present at the end of the reaction. It was also observed that the particles synthesized without the orifice are coarse in nature and they tend to agglomerate during drying.

It is observed that without the orifice there is a delay in the pH drop (Figure 2(b)) compared to the $[\text{Ca}^{2+}]$ drop. This behavior is expected since the carbon dioxide was fed with a constant flow rate with a high initial concentration of Ca(OH)_2 which led to no change in pH. This data is also supported by the conductivity measurement (Figure 2(c)), which is an indirect measure of the total concentration of the charged species in the liquid phase. The first region of the conductivity plot gives information about the “induction time”, in which the nuclei are formed by absorption of CO_2 onto the surface of Ca(OH)_2 . The formation of CaCO_3 leads to a decrease in the concentration of dissolved ionic species and hence the drop in the conductivity. The relatively slow precipitation process in the absence of the orifice increases the size (101 nm) of the crystals generated, as shown in Table 2 and discussed in the next section.

3.1. Effect of Orifice Diameter and Geometry of the Orifice on the Rate of Consumption of Ca(OH)_2 . Three different orifices with one hole of 2, 3, and 4 mm diameter and one orifice plate having 5 holes of 1 mm diameter were used for cavitation purposes as shown in Figure 1(b). Cavitation number (C_v) and β (ratio of orifice diameter to pipe diameter) for the above orifices are presented in Table 1. The consumption profiles of Ca(OH)_2 slurry with different

FIGURE 2: (a) Consumption of Ca(OH)_2 during the carbonation process without orifice (4% Ca(OH)_2 and 5 l/min CO_2 flow rate). (b) Change in pH during the carbonation process without orifice (4% Ca(OH)_2 and 5 l/min CO_2 flow rate). (c) Change in conductivity during the carbonation process without orifice (4% Ca(OH)_2 and 5 l/min CO_2 flow rate).

orifice diameters are shown in Figure 3. It can be seen that the rate of reaction is much faster with all orifices compared to the rate observed in the absence of an orifice (Figure 2).

TABLE 1: Hydrodynamic conditions of different orifice plate used for experiments.

Sr. No	Diameter of orifice	β value (ratio of orifice diameter to pipe diameter)	C_v (cavitation number)
1	2 mm	0.10	0.15
2	3 mm	0.15	0.75
3	4 mm	0.20	2.15
4	5 holes of 1 mm diameter	0.05	0.21

TABLE 2: Effect of slurry concentration and CO_2 flow rates, orifice diameter on crystallite size (calculated from XRD), and particle size distribution (from TEM).

Ca(OH)_2 Slurry%	CO_2 l/min	Orifice Dia. mm	Crystallite size nm	Particle size distribution nm
Effect of change in CO_2 flow rate				
4	3	4	74	65–92
4	5	4	54	62–53
4	7	4	47	35–55
Effect of change in Ca(OH)_2 concentration				
2	5	4	50	69–52
4	5	4	54	62–53
6	5	4	61	50–72
Effect of change in orifice diameter				
4	5	1 $\times$ 5 holes	37	29–38
4	5	2	39	30–41
4	5	3	49	43–56
Without orifice				
4	5	4	54	62–53
4	5	—	101	90–168

FIGURE 3: Effect of different orifice on consumption of Ca(OH)_2 Slurry with respect of time (showing constant rate period and falling Rate periods) (4% Ca(OH)_2 and 5 l/min CO_2 flow rate).

During the precipitation process, the rate of nucleation step is considered as the rate limiting step. It is observed that

the nucleation is enhanced by the hydrodynamic cavitation and hence enhances the rate of precipitation as reported in earlier section. Nishida [18] has observed that acoustic cavitation generated physical effects of microstreaming that influenced the precipitation of calcium carbonate. Our investigation predicts the same effect on the calcium carbonate precipitation by hydrodynamic cavitation. Lyczko et al. [19] reported the effect of cavitation on the primary nucleation of potassium sulphate. They found that the induction period is drastically reduced in the presence of acoustic cavitation. Similar reduction in induction period and enhancement of primary nucleation (no external addition of seed particles) is observed in our case for calcium carbonate precipitation due to hydrodynamic cavitation. As in the case of without orifice, there is an induction period prior to the consumption of Ca(OH)_2 . However, the length of the induction period is shorter for the 4 mm dia orifice compared to the rest. This indicates that the 4 mm orifice initiates the reaction due to a relatively stronger shear forces generated by the hydrodynamic cavitation process. The larger diameter orifice might have generated the stronger hydrodynamic forces. On changing the geometry of orifice to 1 mm size, 5 holes (Figure 1(b)) orifice geometry, the reaction is initiated without any induction period. This arrangement also yielded smaller particle size during synthesis of calcium carbonate

FIGURE 4: Effect of different experimental conditions on X-ray diffraction spectra. A: Without orifice, 4% Ca(OH)_2 slurry, 5 l/min CO_2 flow rate; B: 1 mm orifice, 4% Ca(OH)_2 slurry 5 l/min CO_2 flow rate; C: 2 mm orifice 4% Ca(OH)_2 slurry 5 l/min CO_2 flow rate; D: 3 mm orifice 4% Ca(OH)_2 slurry 5 l/min CO_2 flow rate; E: 4 mm orifice 4% Ca(OH)_2 slurry 7 l/min CO_2 flow rate; F: 4 mm orifice 2% Ca(OH)_2 slurry 5 l/min CO_2 flow rate; G: 4 mm orifice 6% Ca(OH)_2 slurry, 5 l/min CO_2 flow rate; H: 4 mm orifice 4% Ca(OH)_2 slurry, 3 CO_2 l/min flow rate; I: 4 mm orifice, 4% Ca(OH)_2 slurry, 5 CO_2 l/min flow rate.

in the carbonation process. X-ray diffraction analysis was carried out for all the powder samples as shown in Figure 4. The grain size (crystallite size) was calculated for all the samples by using Debye Scherrer formula:

$$X_d = \frac{k\lambda}{\beta \cos \theta}, \quad (2)$$

where $k = 0.9$, $\beta = \text{FWHM}$, and θ is glancing angle of X-rays with the sample holder, $\lambda = 1.5405 \text{ \AA}$.

As shown in Table 2, the 1 mm $\times$ 5 holes orifice generated the smallest crystal size of 37 nm. The powder XRD is found to be preferably oriented along (1 0 1 0) plane for samples including the CaCO_3 without the orifice. No effect is observed on phases due to change in orifice diameter and geometry of orifice. Single calcite phase is observed in all the samples. Wide particle size distribution was observed for the sample without orifice ranging from 90 to 168 nm as shown in Figure 5(a), while narrow particle size distribution is observed ranging from 30 to 41 nm for the orifice with 2 mm orifice as shown in Figure 5(b). The confirmation of calcite phase observed in the XRD data is confirmed by the TEM images. As shown in Figure 6, the TEM image shows the cubic shape particles of calcite particles synthesized using 4 mm orifice diameter 4% slurry concentration and 5 l/min CO_2 flow rate.

The data shown in Figures 4–6 and Table 2 indicates that the rate of consumption of calcium hydroxide slurry during CaCO_3 synthesis using hydrodynamic cavitation is significantly dependent on the geometry of the orifice and its cavitation number. The use of orifice geometry significantly reduces the grain size and hence, increasing the surface area of the synthesized calcite and making it more suitable for the surface coatings applications. Patil and

Pandit [7] also have reported the reduction in particle size of styrene butadiene rubber nanoparticles by using the different geometries of orifice used in hydrodynamic cavitation. Luque de Castro and Priego-Capote [20] reported the influence of ultrasonic power and horn tip onto the carbonation process. They found that acoustic cavitation had an effect on the consumption of calcium hydroxide, which shows similar reduction in the induction period and particle size reported in Figure 3 and Table 2, respectively.

Figure 7 shows the effect of cavitation number (C_v) and the ratio of orifice diameter to pipe diameter (β factor) on the rate of consumption of Ca(OH)_2 slurry. It is found that the rate of consumption of Ca(OH)_2 increases with an increase in the ratio of orifice diameter to plate diameter (β). Ca(OH)_2 consumption rate has gone up exponentially with an increase of β factor which indicates that there is a significant effect of geometry of orifice plate. We speculate that the β factor has significant influence on the generation of hydrodynamic cavitation. Generally for effective cavitation, it is recommended that the cavitation number should be less than 2.5 and it is important to know that severity of cavitation increases with decreasing the cavitation number [10], which is the case for all orifices. The higher cavitation number for 4 mm orifice also supports the higher rate observed in Figure 3.

3.2. Effect of CO_2 Flow Rates onto the Conductivity for Orifice with 4 mm Diameter. A change in the electrical conductivity of the solution with respect to time in the presence of 4 mm diameter orifice is presented in Figure 8. Three different flow rates (3, 5, and 7 l/min) were used at constant 4% Ca(OH)_2 concentration. It is found at higher flow rate (7 l/min) that the drop in the solution conductivity is faster in comparison to the other two flow rates. It can also be observed that there is small induction time for higher flow rate 7 l/min while longer induction time for 3 and 5 l/min flow rates. Induction time for 7 l/min flow rate is less than 5 minute, while for 3 and 5 l/min induction time is nearly 10 minute. In comparison to other two flow rates, numbers of nuclei are formed at 7 l/min and hence the drop in conductivity is faster. With increasing the CO_2 flow rate, the reduction in crystallite size and no change in calcite phase are observed as shown in Figure 4 and Table 2. At 3, 5, 7 l/min flow rates, the crystallite size was found 74, 54, and 47 nm, respectively. The particle size distribution is found to be narrow at higher flow rate (7 l/min) ranging from 35 to 55 nm.

3.3. Effect of Ca(OH)_2 Slurry Concentration on the Rate of Reaction. Effect of three different Ca(OH)_2 slurry concentrations on the solution conductivity at 4 mm orifice diameter is shown in Figure 9. At higher Ca(OH)_2 concentration (6%), the drop in the conductivity takes longer time in comparison to 2% Ca(OH)_2 slurry concentration which indicates that there is longer induction time for higher Ca(OH)_2 and shorter induction time for low Ca(OH)_2 slurry concentration. At 2% of slurry concentration the drop in conductivity occurs very fast (within 5 minutes). It is also found that the available CO_2 gas concentration is more

FIGURE 5: (a) Particle size distribution of CaCO_3 particles synthesized without orifice in the pipe (4% $\text{Ca}(\text{OH})_2$ slurry, 5 l/min CO_2 flow rate). (b) Particle size distribution of CaCO_3 particles synthesized using Hydrodynamic cavitation (2 mm orifice, 4% $\text{Ca}(\text{OH})_2$ slurry, 5 l/min CO_2 flow rate).

FIGURE 6: TEM Image of nano- CaCO_3 synthesized using hydrodynamic cavitation (4 mm orifice, 4% $\text{Ca}(\text{OH})_2$ slurry, 5 l/min, CO_2 flow rate) (Scale 500 nm).

for the 2% slurry and hence induces the massive nuclei formation.

The effect of calcium hydroxide slurry concentration on the pH values, that is, the completion of reaction was also studied. The reaction was carried out at constant flow rate of CO_2 (5 l/min CO_2 flow rate) and by using 4 mm diameter of orifice. It is seen that the completion of reaction takes longer time (20 minutes) with 6% slurry concentration and on the other hand 2% slurry takes less than 10 minutes for completion. The constant period zone and falling rate zone are observed in the case of 4 and 6% slurry, while 2% slurry shows only falling rate period. As shown in Table 2, with increasing the concentration of calcium hydroxide there is an increase in the crystallite size. This observation suggests that increasing the $\text{Ca}(\text{OH})_2$ has no favorable effect on the crystallite size. The XRD patterns for calcite synthesized at different $\text{Ca}(\text{OH})_2$ concentration indicate that the calcite powder is crystalline in nature and exhibits pure calcite phase [21].

FIGURE 7: Effect of β factor onto rate of calcium hydroxide consumption and cavitation number (C_v).

4. Conclusion

This study has demonstrated that nanocalcite can be synthesized by using a hydrodynamic cavitation reactor, without formation of vaterite phase. The effects of three different orifice diameters and the geometry of orifice were evaluated. The average grain size of the calcite synthesized without cavitation was found to be 101 nm. It is seen that there is a wide distribution of particle size in the range 90–168 nm for the setup without an orifice. The change in the geometry of orifice has resulted significant effect on the crystallite size. In

FIGURE 8: Effect of CO₂ flow rates on conductivity of reaction mixture (orifice diameter 4 mm, 4% Ca(OH)₂ slurry concentration).

FIGURE 9: Effect of Ca(OH)₂ slurry concentration onto the conductivity at 5 l/min CO₂ gas flow rate.

case of 5 holes of 1 mm size orifice found to give reduced size of size of 37 nm.

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