



The influence of ammonia and selected amines on the characteristics of calcium carbonate precipitated from calcium chloride solutions via carbonation



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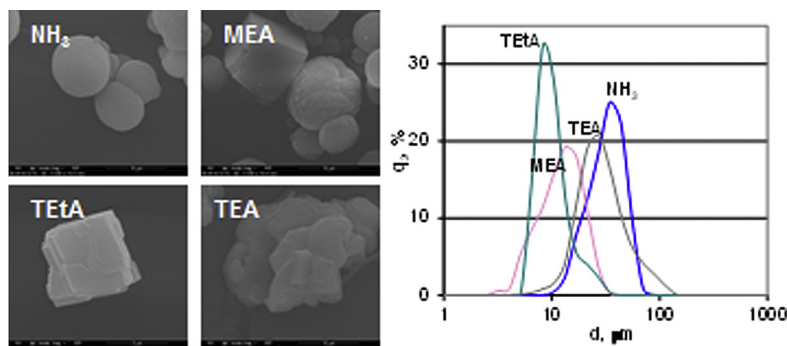
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HIGHLIGHTS

- Calcium carbonate was precipitated from calcium chloride solution via carbonation.
- The characteristics of CaCO₃ depended on used CO₂ absorption promoters.
- Fast CO₂ absorption in NH₃ or MEA solutions favored vaterite formation.
- Slower carbonation in the presence of TEtA or TEA resulted in calcite precipitation.

GRAPHICAL ABSTRACT



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ABSTRACT

The precipitation of calcium carbonate was carried out by passing a gaseous mixture of carbon dioxide and air into a calcium chloride solution. The selected compounds enhancing carbon dioxide absorption were used as additives which promote the formation of carbonate ions in the solution. The additives were ammonia, monoethanolamine, triethylamine and triethanolamine. The resulting calcium carbonate particles varied in polymorphic composition and the particle size depending on the used absorption promoter. When absorption occurred quickly in ammonia or monoethanolamine solutions, the obtained calcium carbonate particles were mainly vaterite. Calcium carbonate particles were precipitated as calcite in triethylamine or triethanolamine solutions, while the rate of carbon dioxide mass transfer from gas to liquid phase was much slower. All precipitated calcium carbonate particles had monomodal particle size distributions. Smaller particles of vaterite were produced in the monoethanolamine solution and of calcite in the triethylamine solution.

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1. Introduction

Precipitated calcium carbonate is widely used in paper, paint, plastics and rubber industries. Particles with well-defined

characteristics, i.e., crystalline form, size and shape, are required for specific applications. The product of defined characteristics can be formed by the careful control of process parameters such as supersaturation, temperature, pH, a manner of conducting the reaction, the intensity of mixing, presence of additives. Therefore, research on the production of CaCO_3 particles of defined characteristics is carried out intensively over the years.

There are many methods to produce precipitated calcium carbonate. A typical industrial method of CaCO_3 production is a carbonation process based on the reaction of gaseous carbon dioxide with the milk of lime [1,2]. A large amount of precipitated calcium carbonate is also produced as a by-product in the ammonia-soda process (Solvay method) [3–5]. However, the quality calcium carbonate obtained in the conventional process is low, and the product contains lots of impurities [3,4]. Precipitated calcium carbonate is also formed in the lime-soda process as a byproduct of the reaction of calcium hydroxide with sodium carbonate, when the sodium hydroxide recovery is the main objective [2]. In the laboratory, the most commonly used method of calcium carbonate precipitation is mixing of solutions containing soluble calcium salt and carbonate salt, because of its simplicity in the operation or its easiness in the control of the process variables [6].

However, the new methods have been proposed recently for preparation of calcium carbonate particles. Among them are high pressure processes, when CO_2 reacts with calcium acetate at increased pressure to shift the reaction equilibrium towards the formation of calcium carbonate [7], or hydrothermal precipitation of calcium carbonate with CO_2 [8] or CO_2 precursors [9–11]. Because the dissociation constant pK_a of carbonic acid decreases with the increasing of CO_2 partial pressure and consequently the concentration of carbonate ions in the reaction solution increases, supersaturation is higher during the precipitation carried out under such condition. The reaction of calcium salt with carbon dioxide formed in situ from CO_2 precursors at atmospheric pressure and elevated temperature are also proposed [12,13]. Another group of developed calcium carbonate precipitation ways are those involving the use of CO_2 absorption promoters in carbonation processes. High quality calcium carbonate can be produced by this method [14,15]. Reagents that improve CO_2 absorption can be an ammonia [16–20] and amines [14,15,20–25]. Calcium salt solutions [14–22,25] or calcium hydroxide slurry [23,24] have been used as a source of calcium ions in these reactions.

The aim of this work is to compare the effect of selected compounds that enhance CO_2 absorption on the precipitated calcium carbonate characteristics. The influence of the type of absorption promoters on the production of calcium carbonate particles by the carbonation process was investigated by Popescu et al. [20] and Chuajiw et al. [24]. The first studied reactions were carried out in CaCl_2 solutions when ammonia, monomethylamine, dimethylamine and trimethylamine were added as absorption promoters [20]. The pH of initial solutions was about 10.4–10.6 in all experiments. The obtained particles were a mixture of calcite and vaterite, wherein vaterite was the main form in ammonia and monomethylamine solution, content of vaterite and calcite was comparable for dimethylamine, while calcite predominant in a trimethylamine solution. The differences in the characteristics of obtained products were attributed to the vaterite stabilization by the carbamate ions forming during the CO_2 absorption in solutions of ammonia, primary and secondary amines. In other research, the substrate was calcium hydroxide and CO_2 absorption promoters were amines, diamines and amino acids having different carbon chain lengths [24]. Calcite was obtained when calcium carbonate was precipitated in the solution without additives. A mixture of aragonite and calcite were produced with butylamine, while amorphous calcium carbonate was obtained with addition of

hexylamine and octylamine. Aragonite with a small amount of calcite was precipitated in the presence of diamines of the formula $\text{H}_2\text{N}(\text{CH}_2)_n\text{NH}_2$ with $n = 4$ and $n = 6$, vaterite, aragonite and calcite was obtained with 1,2-diaminoethane, while the only vaterite was detected in the product formed in the solution with 1,8-diaminooctane. When amino acids of formula $\text{H}_2\text{N}(\text{CH}_2)_n\text{COOH}$ were added to the reactive mixture, vaterite with small amount of calcite was precipitated with glycine ($n = 1$) and vaterite with a small amount of aragonite and calcite was obtained in the presence of 4-aminobutyric acid and 6-aminohexanoic acid. The differences in the characteristics of precipitated calcium carbonate particles were ascribed to the interaction of amines, diamines or amino acids with precipitated particles and retardation of the dissolution-recrystallization process in solutions as a result of the adsorption or covering of calcium carbonate surfaces by added organic substances.

In summary, there is a little work on the effects of the type of amine on the characteristics of the of precipitated calcium carbonate particles, especially on the effects on particle size. In our work, we focused on four absorption promoters: ammonia, monoethanolamine (MEA), triethylamine (TEtA), and triethanolamine (TEA), which have been selected because the detailed mechanisms of carbon dioxide absorption in aqueous solutions of these compounds have been described. During CO_2 absorption in ammonia or MEA aqueous solutions, carbamate ions are formed [26,27] and as noted previously, carbamates are considered as ions stabilizing vaterite form in aqueous solutions. However, TEtA and TEA are both tertiary amines and CO_2 absorption carries out according to the base-catalyzed hydration mechanism [28].

2. Experimental

2.1. Reagents

Anhydrous calcium chloride (CaCl_2 , pure p.a. Lachema), 25% ammonia solution (NH_4OH , pure p.a. CHEMPUR), monoethanolamine (99% Aldrich), triethylamine (99% Sigma-Aldrich), triethanolamine (pure, POCh) were used in this study. All chemicals were used without further purification. Deionised water was used to prepared solutions. Carbon dioxide was purchase from Oxygen s.c.

2.2. Preparation

The reaction was carried out in the airlift reactor constructed of two coaxial columns causing the internal circulation of the reactive mixtures. The detailed description of experimental set-up is given in Ref. [29].

Calcium carbonate precipitation was conducted at 20 °C. The concentrations of calcium ions and organic additive in the solution were equal and amounted to 0.2 M. The liquid volume in the reactor was 1.8 dm³. The mixture of air and CO_2 was used as a gaseous reagent, and the gas flow rate was 1 dm³/min. The volume fraction CO_2 in the gas stream was 20%. The reaction was carried out during 60 min. The 10 ml samples of liquid phase were taken at regular time intervals. Calcium ions concentration was determined in the filtrate sample by the EDTA titration method. To minimize the errors of Ca^{2+} determination the adequate volume of sample and the concentration of EDTA solution were used.

2.3. Characterization of CaCO_3 particles

The samples of CaCO_3 were filtered, washed with ethanol and dried at 100 °C for 6 h. The size of calcium carbonate particles was determined by a laser diffraction method (SALD 2300 Shimadzu). Precipitated particles were observed by a microscope (DELTA

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