

Precipitated Calcium Carbonate Prepared by Batch and Semicontinuous Processes

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A comparison of the physical-chemical properties of precipitated calcium carbonate (PCC), prepared by batch and semicontinuous processes, is presented. In both processes, PCC, was synthesized by carbonation of slaked lime. In the batch process the experiments were performed by varying the initial mass concentration and temperature, and the semicontinuous process was conducted by varying the initial supersaturation and temperature. Calcite was the only calcium carbonate polymorph that appeared under the experimental conditions investigated. It was found, that in the batch experiments the specific surface area and the average size of calcite particles decreased with the increase of temperature. At the same time, the increase of the initial mass concentration of slaked lime enhanced the degree of precipitate aggregation. PCC prepared by the semicontinuous process, conducted at low temperatures and high supersaturations, was of high specific surface area. By careful tuning of the semicontinuous process parameters, calcite in the form of rhombohedral, scalenohedral or nanosized spherical particles can be produced.

Key words: *Precipitated calcium carbonate, slaked lime, batch and semicontinuous carbonation process*

Introduction

Calcium carbonate is an extremely versatile filler and pigment that is utilized in a wide variety of products including paper, plastics, rubber and pharmaceuticals. Its final application is determined by different physical and chemical properties. Calcium carbonate crystallizes forming a number of polymorphic and hydrate modifications (calcite, aragonite, vaterite, calcium carbonate monohydrate, calcium carbonate hexahydrate and amorphous calcium carbonate). Because of its thermodynamic stability at standard conditions and its possibility to appear in various morphologies (rhombohedral, scalenohedral, spheroidal, etc.), calcite proves to be the most important polymorph in industrial application. Thus for instance, the paper industry uses scalenohedral calcite having the specific surface area of approximately $10 \text{ m}^2 \text{ g}^{-1}$, while plastics and sealants industries prefer rhombohedral calcite with rather high specific surface area, up to $70 \text{ m}^2/\text{g}$.

From the technological point of view, two kinds of calcium carbonate products are distinguished: natural ground calcium carbonate (GCC) and synthetic (precipitated) calcium carbonate (PCC). Development of new technologies, caused by the market demands for the high quality of a product, puts specific needs to the calcium carbonate properties. To meet these needs, calcium carbonate should be prepared under carefully controlled conditions and this can be provided by PCC production only, since precipitation opens plenty of possibilities of yielding the product with predetermined properties.

PCC can be synthesized by two methods:¹ precipitation from aqueous solutions, and carbonation of slaked lime.

Complex solutions of calcium salts and functional organic additives are often added to slaked lime.^{2,3} The first method is often adopted in a laboratory study, because of the easiness in controlling process variables. In this way, for example, *Kitamura*⁴ revealed the effect of supersaturation, feeding rate and temperature on the morphology and crystallization of polymorphs in $\text{CaCl}_2\text{--Na}_2\text{CO}_3$ aqueous solutions. In this way, also, we studied the effect of inorganic additives on the physical and chemical properties of precipitates from $\text{Ca}(\text{OH})_2\text{--H}_2\text{CO}_3$ aqueous solutions^{5,6} and the conversion kinetics of vaterite to calcite in $\text{CaCl}_2\text{--Na}_2\text{CO}_3$ aqueous solutions.⁷ However, in industrial scale the most widely used method to obtain PCC is the carbonation route. This is due to the availability of raw materials and their low costs. Basically, the industrial procedure of preparing PCC consists of three steps:

(a) calcination of limestone to produce quicklime (CaO) and carbon dioxide (CO_2):



(b) the slaking process by controlling the addition of H_2O , in which the quicklime is transformed to slaked lime, i. e. suspension of $\text{Ca}(\text{OH})_2$:



(c) carbonation of slaked lime, i. e. bubbling CO_2 gas through an aqueous $\text{Ca}(\text{OH})_2$ suspension (CO_2 by-product produced in the calcination process is most often used):



Currently, carbonation of slaked lime is performed commercially only in batch reactors.¹ However, by using a semi-continuous carbonation process it is possible to adjust the driving force of this process. The driving force is usually expressed as supersaturation, $S = [(a(\text{Ca}^{2+}) a(\text{CO}_3^{2-}))/K_{\text{sp}}]^{1/2}$, where a denotes the activities of respective ionic species at a certain time, and K_{sp} is the thermodynamic solubility product of a particular calcium carbonate polymorph. By adjusting the supersaturation, it is possible to control the morphology and size of the precipitated CaCO_3 .^{8,9} In this work, the physical-chemical properties of PCC, prepared by carbonation of slaked lime in the batch and semicontinuous processes, are compared.

Experimental

Batch process

PCC was prepared in a 300 cm³ double-walled glass reactor. Stirring was performed by a flat-bladed stirrer with two perpendicular blades ($n = 300 \text{ min}^{-1}$), the diameter being 1/3 of the reactor diameter. The vessel was fitted with two vertical baffle plates to prevent vortex formation during stirring. CO_2 gas was introduced into the vessel through the nozzles placed at the bottom of these two baffles. Initially, the reactor was filled with 200 cm³ of deionized water, saturated by CO_2 gas (100 % CO_2 , Messer). The flow rate of CO_2 was kept constant, $Q = 820 \text{ cm}^3 \text{ min}^{-1}$. The suspension of $\text{Ca}(\text{OH})_2$ (p. a., Merck) was injected into water saturated with CO_2 , so that the initial mass concentration in the system varied in the range $13.0 \text{ g dm}^{-3} > \gamma(\text{Ca}(\text{OH})_2) > 28.6 \text{ g dm}^{-3}$. The temperature was kept constant during each experiment in the range $25 \text{ }^\circ\text{C} < \theta < 45 \text{ }^\circ\text{C}$. The propagation of the reaction was followed by measuring the electrical conductivity of the reaction mixture, κ_{25} (normalized to the value which refers to 25 °C).

Semicontinuous process

PCC was prepared in a thermostated glass reactor of 1.6 dm³ total volume capacity. The electrical conductivity was controlled and kept constant at the predetermined value within the range $0.5 \text{ mS cm}^{-1} < \kappa_{25} < 5.0 \text{ mS cm}^{-1}$. Peristaltic pump was used as actuator, which dosed the thermostated $\text{Ca}(\text{OH})_2$ suspension ($\gamma(\text{Ca}(\text{OH})_2) = 100 \text{ g cm}^{-3}$) into the reactor. A gas mixture with approximately $\psi = 20 \text{ } \%$ $\text{CO}_2/80 \text{ } \%$ N_2 (Messer) was used, simulating the flue gases that are commonly used in the PCC industry during the carbonation process. The gas mixture was introduced at a constant flow rate, $Q = 7.5 \text{ dm}^3 \text{ min}^{-1}$, into the vessel through the nozzles placed at the bottom of the two baffles. Stirring was performed by a flat-bladed stirrer ($n = 400 \text{ min}^{-1}$) with the diameter being 1/3 of the reactor diameter. The flow of the $\text{Ca}(\text{OH})_2$ suspension was stopped 10 min after starting the experiment, while the gas flow was kept constant until the endpoint of the carbonation reaction, which was indicated by a drop of conductivity.

Characterization of precipitate

At the end of each experiment, the final suspension was treated in a centrifuge for 10 min, $n_c = 4000 \text{ min}^{-1}$, the precipitate was separated and dried at $\theta = 105 \text{ }^\circ\text{C}$ for three hours. The composition of the solid phase was determined by Fourier transform infrared, FT-IR, spectroscopy (Mat-

tson, Genesis Series). The size distribution of particles was analyzed by an electronic device, Coulter Multisizer II, and the specific surface area was determined by the multiple BET method (Micromeritics, Gemini) using nitrogen gas as the adsorbate. Samples were also analyzed by means of scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Results and discussion

Batch process

As it is well known, supersaturation is the most important factor that determines physical-chemical properties of a precipitate obtained from aqueous solutions.¹⁰ Fig. 1 shows changes of supersaturation in the precipitation system during the carbonation process, measured as electrical conductivity of the $\text{Ca}(\text{OH})_2$ suspension. The initial, ascending part of the κ_{25} vs. time curves corresponds to the dissolution of $\text{Ca}(\text{OH})_2$, previously injected in the system. After reaching its maximum, κ_{25} value slowly decreases (approximately horizontal part of the curves) and then an abrupt drop is evident from the moment the suspended $\text{Ca}(\text{OH})_2$ disappears from the reaction mixture. The minimum value of κ_{25} indicates the end of the calcium carbonate formation. A subsequent increase of κ_{25} is a consequence of PCC dissolution in the excess carbonic acid that remained in the system. The results of mineralogical analyses of the final products show that calcite was the only CaCO_3 polymorph obtained at the end of each carbonation experiment. The maximum value of conductivity and the carbonation time decrease with decreasing the initial mass concentration of $\text{Ca}(\text{OH})_2$ added to the system (see Fig. 1 and Table 1). Thus for instance, at 25 °C and $\gamma(\text{Ca}(\text{OH})_2) = 13.0 \text{ g dm}^{-3}$ the ma-

Table 1 – Experimental conditions of PCC preparation by the batch carbonation process and physical properties of precipitates obtained

Tablica 1 – Eksperimentalni uvjeti priprave TKK šaržnim postupkom karbonatizacije vapnenoga mlijeka i odgovarajuća fizička svojstva dobivenih taloga

Experiment Pokus	$\theta^{(a)}$ / °C	$\gamma^{(b)}$ / g dm ⁻³	$t_{\text{max}}^{(c)}$ / s	$\kappa_{\text{max}}^{(d)}$ / mS cm ⁻¹	$s^{(e)}$ / m ² g ⁻¹	$\bar{d}^{(f)}$ / μm
1	25	13.0	264	6.60	16.5	5.3
2	25	20.0	460	7.37	21.4	6.3
3	25	28.6	900	7.80	25.7	10.7
4	35	13.0	320	6.72	11.9	4.2
5	35	20.0	496	6.97	12.3	4.2
6	35	28.6	776	7.07	10.7	5.3
7	45	13.0	328	6.30	11.1	3.5
8	45	20.0	536	6.46	11.2	3.3
9	45	28.6	820	6.48	9.6	3.5

(a) – temperature; (b) – mass concentration of $\text{Ca}(\text{OH})_2$ suspension; (c) – time of carbonation; (d) – maximum conductivity of the system; (e) – specific surface area of PCC; (f) – mean particle diameter of PCC

(a) – temperatura; (b) – masena koncentracija suspenzije $\text{Ca}(\text{OH})_2$; (c) – vrijeme karbonatizacije; (d) – maksimalna el. provodnost sustava; (e) – specifična površina TKK; (f) – prosječna veličina čestica TKK

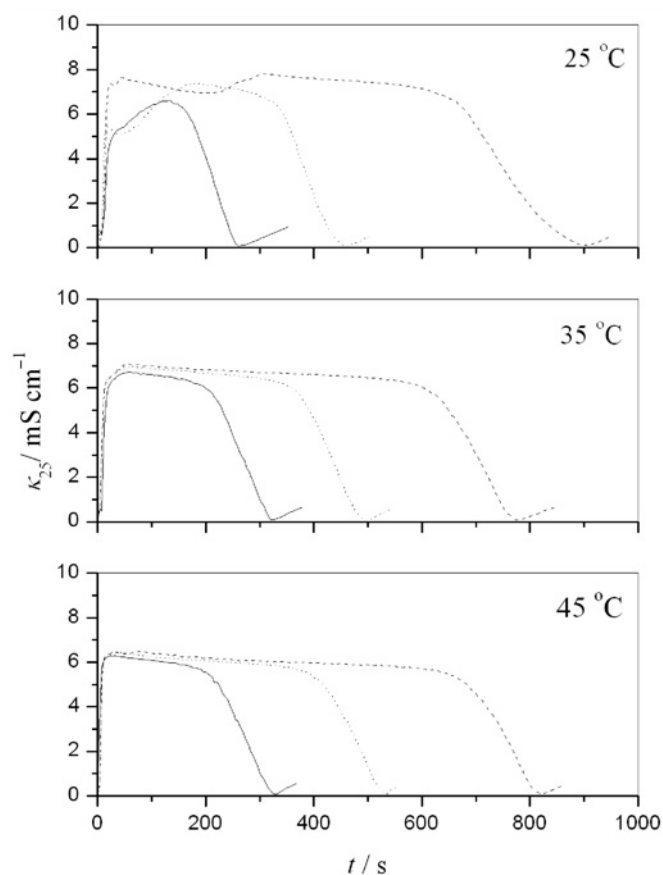


Fig. 1 – Changes of κ_{25} during the batch carbonation process at different temperatures and initial mass concentrations of $\text{Ca}(\text{OH})_2$: (–) 13.0; (---) 20.0, and (.....) 28.6 g dm^{-3}

Slika 1 – Promjena κ_{25} tijekom karbonatizacije suspenzije $\text{Ca}(\text{OH})_2$ u šaržnom reaktoru, pri različitim temperaturama i početnim masenim koncentracijama $\text{Ca}(\text{OH})_2$: (–) 13,0; (---) 20,0 i (.....) 28,6 g dm^{-3}

maximum conductivity of $\text{Ca}(\text{OH})_2$ saturated solution $\kappa_{25\text{max}} = 6.60 \text{ mS cm}^{-1}$ and at the same temperature and $\gamma(\text{Ca}(\text{OH})_2) = 28.6 \text{ g dm}^{-3}$ the maximum value of conductivity $\kappa_{25\text{max}} = 7.80 \text{ mS cm}^{-1}$. As the conductivity of saturated $\text{Ca}(\text{OH})_2$

solution at 25 °C equals 8.640 mS cm^{-1} , the maximum conductivities quoted above roughly correspond to the concentrations of total soluble calcium: $c \approx 0.0165 \text{ mol dm}^{-3}$ and $c \approx 0.0196 \text{ mol dm}^{-3}$, respectively. The maximum conductivities obtained at 35 °C and 45 °C are lower than that obtained at 25 °C, due to the lower solubility of $\text{Ca}(\text{OH})_2$ (and CO_2 !) at higher temperatures. Specific surface area and mean particle diameter (expressed as the maximum value of particle diameter distribution) of the precipitate increase with increasing the maximum conductivity. By comparison of experiments performed at different temperatures, it is evident that the measured specific surface area of PCC decreases with increasing temperature, which is principally due to the lower solubility of $\text{Ca}(\text{OH})_2$ and CO_2 at higher temperatures and with this connected change of the prevailing mechanism of nucleation. For the same reason, the degree of aggregation of the precipitated particles follows the increase of the initial supersaturation, as shown from the scanning electron micrographs of the corresponding samples (Fig. 2). These findings are in accordance with the theoretical models of precipitation by which an increase of supersaturation gives rise to nucleation of a larger number of smaller crystals and to their enhanced aggregation.¹⁰

Semicontinuous process

The main disadvantage of batch carbonation of slaked lime, which is generally used in the PCC industry, is the limited possibility of controlling the supersaturation of the system. Regarding the fact that supersaturation is the main factor affecting the physical-chemical properties of a precipitate, conducting a process under constant supersaturation enables better definition of the final product properties. So, the apparently constant supersaturation was maintained in each experiment of this semicontinuous process. For this purpose, the conductivity was kept at a predetermined value by controlling the feeding flow rate of the $\text{Ca}(\text{OH})_2$ suspension.

Figure 3 shows progress curves (κ_{25} vs. time) obtained during carbonation of slaked lime in the semicontinuous process performed at different temperatures ($\theta = 25 \text{ °C}$, $\theta = 35 \text{ °C}$ and $\theta = 45 \text{ °C}$) and conductivities ($\kappa_{25} = 0.5 \text{ mS cm}^{-1}$; $\kappa_{25} = 1.0 \text{ mS cm}^{-1}$; $\kappa_{25} = 1.5 \text{ mS cm}^{-1}$; $\kappa_{25} = 3.0 \text{ mS cm}^{-1}$

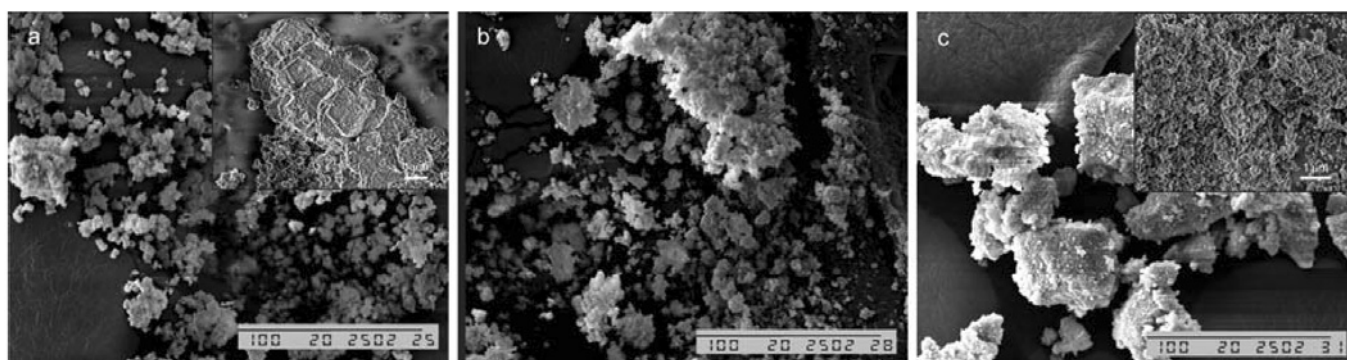


Fig. 2 – Scanning electron micrographs of PCC samples prepared by the batch process at $\theta = 25 \text{ °C}$ and different initial mass concentrations of $\text{Ca}(\text{OH})_2$: (a) 13.0 g dm^{-3} ; (b) 20.0 g dm^{-3} and (c) 28.6 g dm^{-3}

Slika 2 – Pretražne elektronske mikrofotografije uzoraka TKK pripremljenih šaržnim postupkom pri $\theta = 25 \text{ °C}$ i različitim početnim masenim koncentracijama $\text{Ca}(\text{OH})_2$: (a) 13,0 g dm^{-3} ; (b) 20,0 g dm^{-3} i (c) 28,6 g dm^{-3}

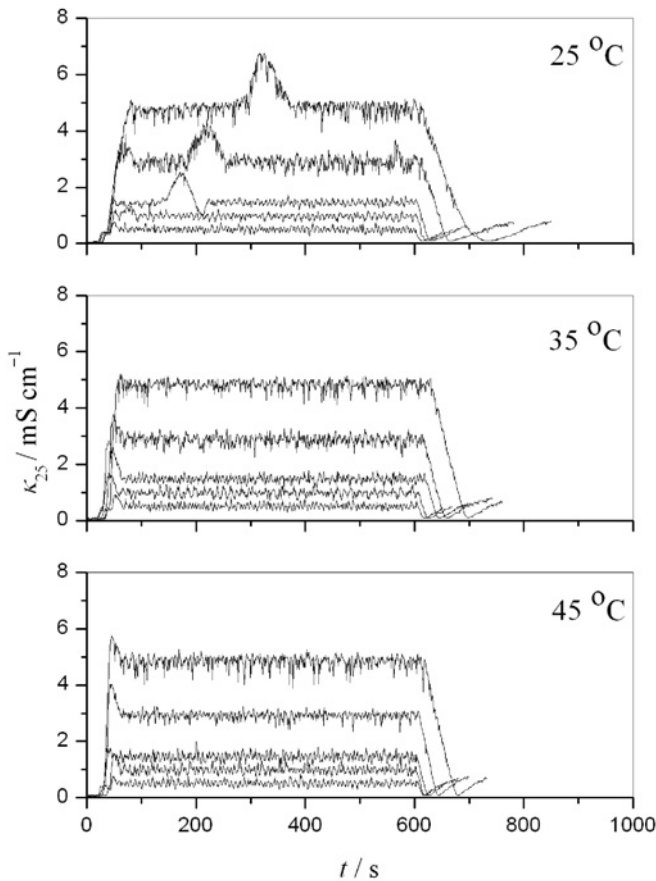


Fig. 3 – Progress curves of κ_{25} during the semicontinuous carbonation process performed at different temperatures and conductivities

Slika 3 – Krivulje promjene provodnosti sustava tijekom semikontinuiranog postupka karbonatizacije pri različitim temperaturama i provodnostima

and $\kappa_{25} = 5.0 \text{ mS cm}^{-1}$). The initial increase of conductivity corresponds to the Ca(OH)_2 dissolution during its introduction to the system. After reaching the predetermined working value of κ_{25} , the conductivity of the system was kept constant (conductivity plateau) by regulating the flow rate of the Ca(OH)_2 suspension. The addition of the Ca(OH)_2 suspension was stopped after 10 min when the carbonation process came to the end, but the flow of the gas mixture went on until precipitation of the excess Ca(OH)_2 was com-

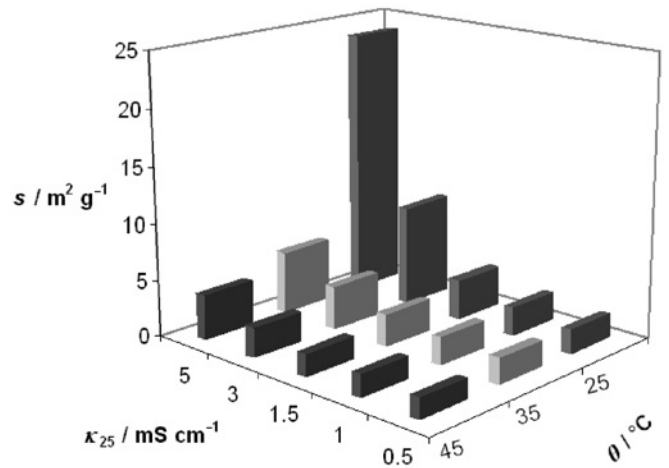


Fig. 4 – Values of the specific surface area of PCC prepared by the semicontinuous process at different temperatures and conductivities

Slika 4 – Vrijednosti specifične površine TKK pripravljenog semikontinuiranim postupkom, pri različitim temperaturama i provodnostima

pleted (Fig. 3, $\theta = 35 \text{ °C}$ and $\theta = 45 \text{ °C}$). This part of the process is denoted by a decrease of κ_{25} , and its minimum value indicates the end of the calcium carbonate formation. A subsequent increase of κ_{25} is a consequence of PCC dissolution in the excess carbonic acid that remained in the system. The shape of the κ_{25} vs. time curve obtained in the experiment carried out at $\theta = 25 \text{ °C}$ and $\kappa_{25} > 1.5 \text{ mS cm}^{-1}$, differs somewhat from the other experiments, since the conductivity increases shortly after a period of constancy. Such a behaviour of the conductivity during the process of PCC formation is explained by being a consequence of nucleation and growth of a certain calcium carbonate precursor phase, probably an amorphous calcium carbonate, on the surface of suspended Ca(OH)_2 particles (so-called "surface mechanism of precipitation").^{9,11} Since the precursor is extremely unstable,¹² it rapidly dissolves in the process of transformation into thermodynamically stable calcite, thus liberating the surface of the Ca(OH)_2 particles and causing the uncontrolled increase in conductivity. Formation of a precursor phase is expected when the supersaturation of the system is higher than the certain critical value, typical for the respective precipitation system. As at higher temperatures the solubility of CO_2 is lower, the respective su-

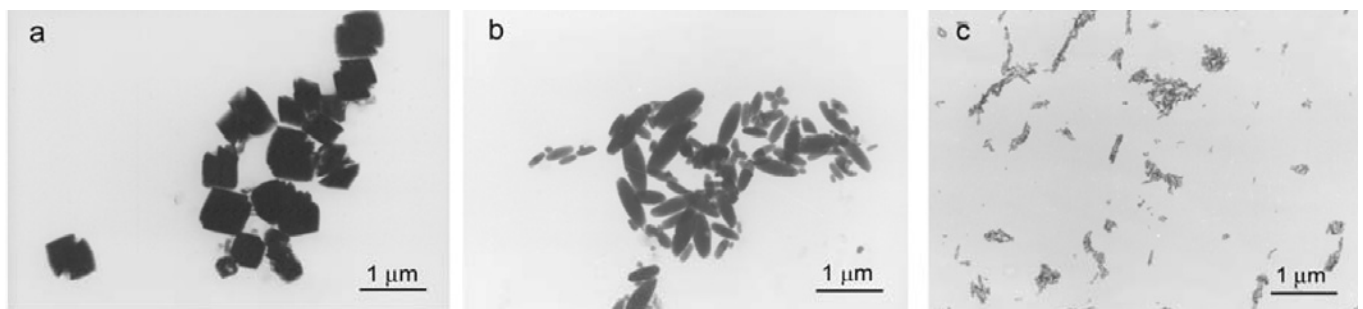


Fig. 5 – Typical TEM images of PCC prepared by the semicontinuous process: (a) rhombohedral, (b) scalenohedral, and (c) spherical
Slika 5 – Karakteristične transmisijske elektronske mikrofotografije uzoraka TKK pripravljenih postupkom: (a) romboedarski, (b) skalenoedarski i (c) kuglasti.

persaturation is lower and, consequently, the formation of precursor phase(s) is less probable.

Calcite was found to be the only CaCO_3 polymorph obtained in the given experimental range. Fig. 4 shows the specific surface areas of PCC prepared by the semicontinuous process. Specific surface area increases with increasing supersaturation and decreasing temperature. PCC with the highest specific surface area of $s = 23.8 \text{ m}^2 \text{ g}^{-1}$ was obtained at $\theta = 25^\circ\text{C}$ and $\kappa_{25} = 5.0 \text{ mS cm}^{-1}$. Typical TEM microphotographs of PCC obtained at different temperatures and conductivities are shown in Fig. 5. At low conductivities ($\kappa_{25} = 0.5 \text{ mS cm}^{-1}$, 1.0 mS cm^{-1} and 1.5 mS cm^{-1}) and at all investigated temperatures ($\theta = 25^\circ\text{C}$, 35°C and 45°C), rhombohedral crystals of calcite, with the mean particle diameter $d_m \approx 0.7 \mu\text{m}$, estimated by TEM, were produced (Fig. 5a). Higher conductivities ($\kappa_{25} = 3.0 \text{ mS cm}^{-1}$ and 5.0 mS cm^{-1}) and temperatures of $\theta = 35^\circ\text{C}$ and 45°C gave rise to the formation of scalenohedral calcite crystals (Fig. 5b). In the experiments conducted at 25°C and at constant conductivities of 3.0 mS cm^{-1} and 5.0 mS cm^{-1} , PCC appeared in a form of nanosized spherical calcite particles, $d_m \approx 30 \text{ nm}$ (Fig. 5c).

Conclusions

– Calcite is the only calcium carbonate polymorph obtained by carbonation of slaked lime in the batch process, at the experimental range of temperatures $25^\circ\text{C} < \theta < 45^\circ\text{C}$, initial mass concentrations of slaked lime $3.0 \text{ g dm}^{-3} > \gamma(\text{Ca}(\text{OH})_2) > 28.6 \text{ g dm}^{-3}$, and at constant CO_2 flow rate of $Q = 820 \text{ cm}^3 \text{ min}^{-1}$.

– Specific surface area and mean particle diameter of the precipitated particles decrease with increasing temperature, while the increase of initial mass concentration of slaked lime enhances the degree of precipitate aggregation.

– Calcite is also the only calcium carbonate polymorph that precipitates in the semicontinuous process of slaked lime carbonation, performed in the bench-scale reactor at experimental range of temperatures $25^\circ\text{C} < \theta < 45^\circ\text{C}$, conductivities $0.5 \text{ mS cm}^{-1} < \kappa_{25} < 5.0 \text{ mS cm}^{-1}$, and at constant N_2/CO_2 flow rate of $7.5 \text{ dm}^3 \text{ min}^{-1}$.

– Specific surface area and mean diameter of particles increase with increasing supersaturation and decreasing temperature.

– Under experimental conditions of low temperatures and high supersaturations ($\theta = 25^\circ\text{C}$, $\kappa_{25} > 1.5 \text{ mS cm}^{-1}$) a precursor phase precipitates initially, probably an amorphous calcium carbonate, which quickly transforms into nanosized calcite.

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List of symbols

Popis simbola

a	– activity – aktivitet
c	– concentration, mol dm^{-3} – koncentracija, mol dm^{-3}
c_s	– equilibrium concentration (solubility), mol dm^{-3} – ravnotežna koncentracija (topljivost), mol dm^{-3}
\bar{d}	– mean particle diameter, μm – prosječni promjer čestica, μm
K_{sp}	– thermodynamic equilibrium constant – termodinamička konstanta ravnoteže
n	– stirring rate, min^{-1} – brzina vrtnje mješala, min^{-1}
Q	– volume flow rate, $\text{dm}^3 \text{ min}^{-1}$ – obujmni protok, $\text{dm}^3 \text{ min}^{-1}$
S	– supersaturation – prezasićenost
s	– specific surface area of precipitate, $\text{m}^2 \text{ g}^{-1}$ – specifična površina taloga, $\text{m}^2 \text{ g}^{-1}$
t	– time of carbonation, s – vrijeme karbonatizacije, s
γ	– mass concentration, g dm^{-3} – masena koncentracija, g dm^{-3}
κ	– electrical conductivity, mS cm^{-1} – električna provodnost, mS cm^{-1}
θ	– temperature, $^\circ\text{C}$ – temperatura, $^\circ\text{C}$
ψ	– volume ratio, 1 – obujamni omjer, 1

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SAŽETAK**Priprava taložnog kalcijeva karbonata šaržnim i semikontinuiranim postupkom**

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U ovom su radu uspoređena fizičko-kemijska svojstva taložnog kalcijeva karbonata (TKK) pripremljenog karbonatizacijom vapnenog mlijeka u šaržnom i semikontinuiranom reaktoru. U šaržnom su postupku varirane temperatura i početna masena koncentracija vapnenog mlijeka, dok su u semikontinuiranom mijenjani temperatura i prezasićenost sustava. Pri svim je istraživanim uvjetima nastao kalcit. Utvrđeno je da se specifična površina i prosječni promjer čestica kalcita, pripremljenog šaržnim postupkom, smanjuju s povećanjem temperature, dok se s povišenjem početne masene koncentracije poboljšava stupanj agregiranosti taloga. TKK pripremljen semikontinuiranim postupkom, vođenim pri niskim temperaturama ili visokim prezasićenostima, velike je specifične površine. Odabirom procesnih parametara semikontinuiranog postupka moguće je pripremiti kalcit u obliku romboedarskih, skalenoedarskih ili kuglastih čestica nanometarskih dimenzija.

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