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The influence of a permanent magnetic field on the crystallization of calcium carbonate from carbonate aqueous solutions

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The effect of a permanent magnetic field on the crystallization of calcium carbonate from carbonate aqueous solutions was studied. Based on the equations for the first- and second-order dissociation constants of carbonic acid, the stability constants of NaCO_3^- and NaHCO_3^0 complexes, the mass balance and electroneutrality equations, the values of the system components and the ratio between the components of the carbonate subsystem were determined, and it was established that the main species in experimental conditions are CO_3^{2-} , NaCO_3^- and HCO_3^- and the main components of the carbonate subsystem are CO_3^{2-} (86.7%) and HCO_3^- (13.19%). Studies have shown that with a significant excess of CO_3^{2-} over HCO_3^- ions in the solution at temperatures of 18-20°C, calcite and vaterite are most likely formed. During the crystallization of calcium carbonate from an aqueous carbonate solution in a magnetic field of 125 - 250 mT, the size of the crystallites increases significantly compared to the absence of a magnetic field.

Key words: magnetic treatment, crystallization of calcium carbonate, carbonate water system, electrolyte solutions, Debye-Hückel theory, approximation method, XRD, Debye-Scherrer method.

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Introduction

The effect of the magnetic field on the crystallization of calcium carbonate has been known for a long time [1] and it is important both from the point of view of fundamental studies of the crystallization of inorganic sparingly soluble salts [2-4] and from the point of view of applied problems of technological processes, for example, such as heat exchange [5] and chemical condensation with predetermined properties of the solid phase [6]. But currently there are many experiments, the results of which are in contradiction [7], which confirms its complexity and multifactorial nature. Such multifactoriality consists in the polymorphism of the solid phase (calcite, aragonite, vaterite [8]) and in the multicomponent calcium-carbonate water system [2, 9], moreover, such a system (calcium-carbonate) is open to atmospheric CO_2 [10], which significantly affects its condition, which, in the absence of tightness, is fundamentally unstable.

Thus, the search for experiments that would unambiguously and reproducibly show the effect of the magnetic field on the crystallization of calcium carbonate is an important scientific task.

I. Experiment

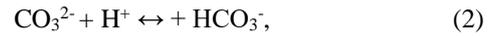
Crystallization was initiated by mixing the solutions. 50 ml of an aqueous solution of CaCl_2 with a concentration of 20.0 mmol/dm³ was added to 50 ml of a solution of Na_2CO_3 with a concentration of 20.0 mmol/dm³. Mixing was done in parallel in two identical chemical beakers with a capacity of 100 ml each. After mixing, one chemical beaker was placed in a magnetic field. During the experiment, the solutions were continuously stirred by hand with glass rods for one minute. All solutions were prepared from chemically pure reagents and distilled water. To test the prepared solutions

before mixing, the concentrations of Na^+ and Cl^- were measured with a PAZ-2 flame liquid analyzer with a relative measurement error of 1%. Also, before mixing, the pH of the Na_2CO_3 solution was measured with an AI-123 ion analyzer with an error of 0.01 units. The magnetic field was created by two flat samarium-cobalt magnets (Fig. 1a). Experiments were performed at temperatures of 18 - 20°C. The distribution of the magnetic field in the chemical beaker was previously measured (without a solution) by a MT-1 magnetometer and is presented in Fig. 1b. Crystallization was initiated by mixing the solutions. 50 ml of an aqueous solution of CaCl_2 with a concentration of 20.0 mmol/dm³ was added to 50 ml of a solution of Na_2CO_3 with a concentration of 20.0 mmol/dm³. Mixing was done in parallel in two identical chemical beakers with a capacity of 100 ml each. After mixing, one chemical beaker was placed in a magnetic field. During the experiment, the solutions were continuously stirred by hand with glass rods for one minute. All solutions were prepared from chemically pure reagents and distilled water. To test the prepared solutions before mixing, the concentrations of Na^+ and Cl^- were measured with a PAZ-2 flame liquid analyzer with a relative measurement error of 1%. Also, before mixing, the pH of the Na_2CO_3 solution was measured with an AI-123 ion analyzer with an error of 0.01 units. The magnetic field was created by two flat samarium-cobalt magnets (Fig. 1a). Experiments were performed at temperatures of 18 - 20°C. The distribution of the magnetic field in the chemical beaker was previously measured (without a solution) by a MT-1 magnetometer and is presented in Fig. 1b.

II. Discussion of results

2.1. Determination of the component composition of the carbonate subsystem

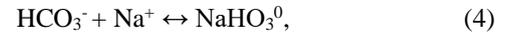
In the work, for the $\text{Na}_2\text{CO}_3 + \text{H}_2\text{O}$ solution, it is important to establish the component composition and quantitative ratios between the components of the carbonate subsystem (CO_3^{2-} , HCO_3^- , CO_2). Such ratios are based on chemical reactions occurring in this system: carbonic acid dissociation reactions of the first and second degrees (1), (2), respectively



water dissociation reactions (3)



and NaCO_3^- and NaHCO_3^0 complex formation reactions (4), (5), respectively



Based on reactions (1) - (5), let's write down the thermodynamic constants of dissociation of carbonic acid in the first (6) and second (7) degrees:

$$K_1 = \frac{a_{\text{H}^+} \cdot a_{\text{HCO}_3^-}}{a_{\text{CO}_2}}, \quad (6)$$

$$K_2 = \frac{a_{\text{H}^+} \cdot a_{\text{CO}_3^{2-}}}{a_{\text{HCO}_3^-}}, \quad (7)$$

and the thermodynamic water dissociation constant K_w (8)

$$K_w = a_{\text{H}^+} \cdot a_{\text{OH}^-} \quad (8)$$

The concentration constants of stability of NaCO_3^- and NaHCO_3^0 complexes (9), (10) will have expressions, respectively:

$$K_3 = \frac{c_{\text{NaCO}_3^-}}{c_{\text{Na}^+} \cdot c_{\text{CO}_3^{2-}}} \quad (9)$$

$$K_4 = \frac{c_{\text{NaHCO}_3^0}}{c_{\text{Na}^+} \cdot c_{\text{HCO}_3^-}} \quad (10)$$

Also, it is necessary to take into account the mass balance equation (11) and the electroneutrality equation (12).

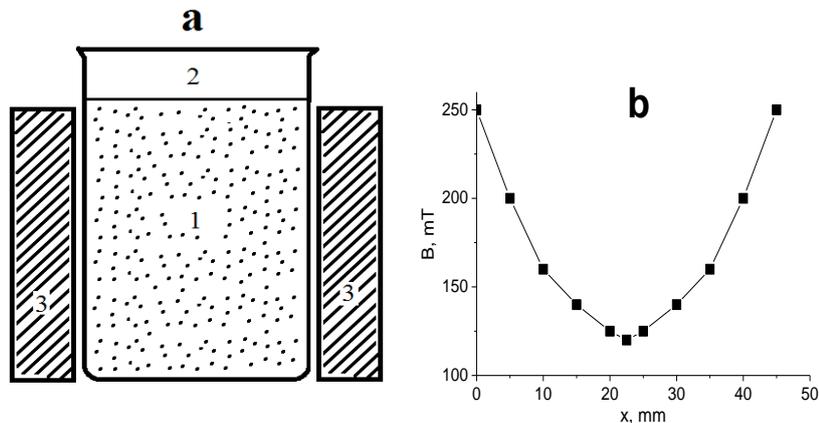


Fig. 1. Scheme of the installation for magnetization of CaCO_3 suspension (a): 1 – CaCO_3 suspension; 2 – chemical beaker; 3 – permanent magnets. Distribution of the magnetic field in the glass (b).

$$(C_{Na^+})_0 + (C_{CO_3^{2-}})_0 = C_{Na^+} + C_{CO_3^{2-}} + C_{HCO_3^-} + C_{CO_2} + C_{NaCO_3^-} + C_{NaHCO_3^0}, \quad (11)$$

$$C_{Na^+} + C_{H^+} = 2C_{CO_3^{2-}} + C_{HCO_3^-} + C_{NaCO_3^-} + C_{OH^-}, \quad (12)$$

The values of the constants are: $K_1 = 1.72 \cdot 10^{-4}$, $K_2 = 4.40 \cdot 10^{-11}$, $K_w = 1.00 \cdot 10^{-14}$. In (7)-(12) we obtain the dependence of CO_3^{2-} on H^+ in the form:

$$(Na^+)_0 \cdot \frac{1 - K_3 \cdot C_{CO_3^{2-}}}{1 + A(H^+) \cdot C_{CO_3^{2-}}} = B(H^+) - C(H^+), \quad (13)$$

$$\text{where } A(H^+) = \frac{K_4 \cdot \gamma_{CO_3^{2-}}}{K_4 \cdot \gamma_{HCO_3^-}} \cdot H^+ + K_3,$$

$$B(H^+) = \frac{\gamma_{CO_3^{2-}}}{K_2 \cdot \gamma_{HCO_3^-}} \cdot H^+ + 2,$$

$$C(H^+) = \frac{K_w}{\gamma_{HCO_3^-} \cdot H^+} - \frac{H^+}{\gamma_{H^+}}.$$

Activity coefficients γ were determined according to the second Debye-Hückel approximation [11]:

$$\log(\gamma) = -\frac{0.512 \cdot Z^2 \sqrt{I}}{1 + 0.328 \cdot a \cdot I}, \quad (14)$$

where Z is the ionic charge; b – the distance of the smallest convergence of ions; I is the ionic strength of the solution, defined as

$$I = 0.5 \cdot \sum_{i=1}^N C_i \cdot Z_i^2, \quad (15)$$

a (effective sizes of ions, Å) are equal [12]: $a_{H^+} = 9$; $a_{Na^+} = 4$; $a_{CO_3^{2-}} = 5$; $a_{HCO_3^-} = 4$; $a_{OH^-} = 3$.

The ionic strength I was determined by the iterative method, the zero approximation of which corresponded to the initial values of concentrations ($(Na^+)_0 = 40 \text{ mmol/dm}^3$ and $(CO_3^{2-})_0 = 20.0 \text{ mmol/dm}^3$). In this case, three iterations were enough (Fig. 2). Since the solution (13) is cumbersome, we introduce additional notations:

$$\begin{aligned} D &= \frac{1}{2AB}, & E &= -(Na)_0 K_3 - AC - B, \\ F &= (Na)_0 K_3 \cdot \left((Na)_0 K_3 + 2B + 2AC + \frac{4AB}{K_3} \right), & G &= (B - AC)^2. \end{aligned} \quad (16)$$

In terms of notation (16), the solution (13) will have the form:

$$C_{CO_3^{2-}} = D \cdot \left[E + (F + G)^{\frac{1}{2}} \right]. \quad (17)$$

For (17), and (6)-(12), as well as (14), (15) were calculated $C_{CO_3^{2-}}$, $C_{HCO_3^-}$, $C_{NaCO_3^-}$, $C_{NaHCO_3^0}$ and C_{CO_2} (Fig. 3) and the relationship between the components of the carbonate subsystem $C_{CO_3^{2-}}$, $C_{HCO_3^-}$, C_{CO_2} (Fig. 4). From fig. 3 shows that at a pH of the Na_2CO_3 dosed solution equal to 10.9, the molar concentrations of the components have:

$$C_{CO_3^{2-}} = 11.6 \frac{\text{mmol}}{\text{dm}^3}, \quad C_{HCO_3^-} = 1.76 \frac{\text{mmol}}{\text{dm}^3},$$

$$C_{NaCO_3^-} = 7.07 \frac{\text{mmol}}{\text{dm}^3}, \quad C_{NaHCO_3^0} = 0.032 \frac{\text{mmol}}{\text{dm}^3},$$

$$C_{CO_2} = 0.0417 \frac{\text{mmol}}{\text{dm}^3}.$$

From fig. 4 it can be seen that at a pH of the Na_2CO_3 dosed solution equal to 10.9 basic components are CO_3^{2-} and HCO_3^- . The calculated ratios between the components of the carbonate subsystem will be as follows components are CO_3^{2-} and HCO_3^- . The calculated ratios between the components of the carbonate subsystem will be as follows (fig. 4):

$$\varepsilon_{CO_3^{2-}} = 86.7\%, \quad \varepsilon_{HCO_3^-} = 13.19\%, \quad \varepsilon_{CO_2} = 3.0 \cdot 10^{-4}\%.$$

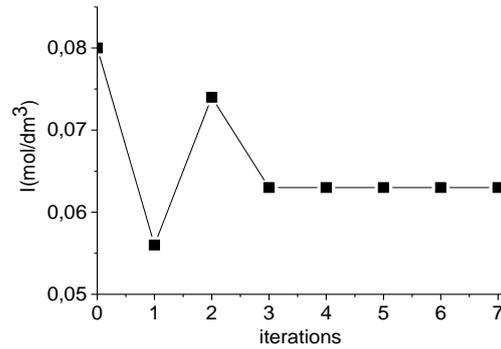


Рис. 2. Залежність іонної сили від кількості ітерацій.

2.2. X-ray studies

X-ray structural analysis of the studied samples was carried out on a DRON-4 diffractometer. Experimental diffractograms were obtained at room temperature with a scan step of 0.050 according to the Bragg-Brentano scheme. The source of X-ray radiation was an X-ray tube with a copper anode at a voltage of 41 kV and a current of 21 mA. Characteristics of X-ray reflexes (angular position 2θ , half width, width at half height) β , integral intensity I were determined using the Gaussian function and were used to interpret the results of X-ray studies.

In Fig. 5. X-ray diffraction patterns of two samples that crystallized without the influence of a magnetic field (Fig. 5a) and with the influence of a magnetic field (Fig. 5b) are shown. It can be seen from the diffraction patterns that they are quite similar. It was suggested that the reflexes of the diffractograms belong to calcium carbonate. It is known that calcium carbonate crystallizes in three modifications: calcite, vaterite and aragonite [13]. Using the known interplane distances [14] and the Wolf-Bragg formula [15]:

$$2 \cdot d \cdot \sin \cdot \theta = k \cdot \lambda, \quad (18)$$

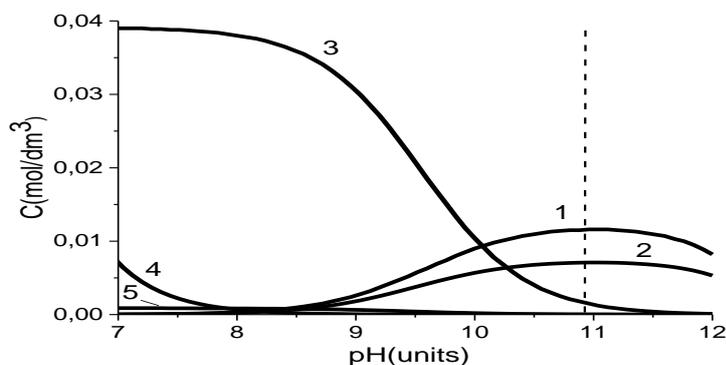


Fig. 3. Concentrations of the components of an aqueous solution of $0.02 \text{ mol/dm}^3 \text{ Na}_2\text{CO}_3$ depending on the pH of the solution. 1 - CO_3^{2-} , 2 - NaCO_3^- , 3 - HCO_3^- , 4 - CO_2 , 5 - NaHCO_3^0 .

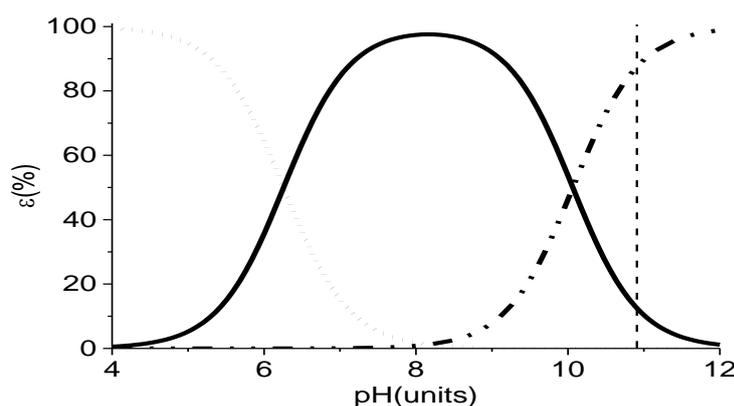


Fig. 4. The ratio (in percent, ε) between the components of the carbonate subsystem of an aqueous solution of $0.02 \text{ mol/dm}^3 \text{ Na}_2\text{CO}_3$ depending on the pH of the solution. - HCO_3^- , · - CO_2 , · - CO_3^{2-} .

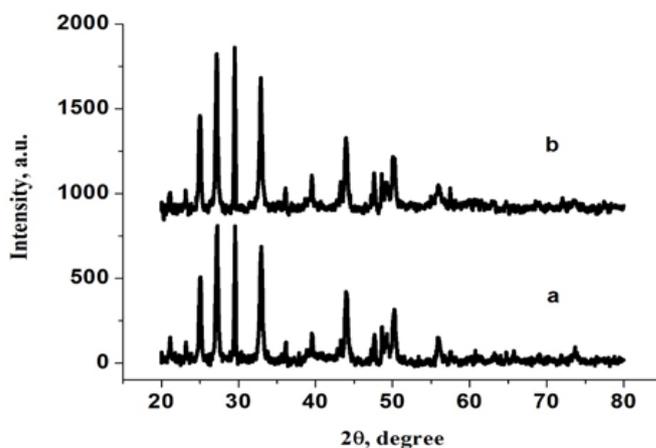


Fig. 5. X-ray diffractogram of the studied samples: a – synthesis without the influence of a magnetic field; b – synthesis under the influence of a magnetic field.

where d is the interplane distance; θ – diffraction angle; k is the order of the diffraction maximum; λ is the wavelength of X-ray radiation, the angular positions of the 2θ reflexes were calculated for various modifications of calcium carbonate. Table 1 shows a comparison of calculation results and literature data with experimental data for two modifications of calcium carbonate (angular positions are given in degrees, intensities in percent).

The table shows that only reflexes characteristic of

calcite and vaterite are present on the diffractograms. Reflexes of another modification, namely aragonite, were not recorded. The calculated and experimental values of the angular positions of the reflexes agree well with each other. The differences in the relative intensities are due to the fact that the literature data are given for peak intensities, while we used integral intensities, in addition, different X-ray radiation was used in both cases.

Table.

Experimental and literature data for calcite and vaterite

calcite				vaterite			
Calculation		Experiment		Calculation		Experiment	
2θ	I	2θ	I	2θ	I	2θ	I
23.0	12%	23.2	21%	20.9	13%	21.1	19%
29.4	100%	29.6	100%	24.9	63%	25.0	60%
36.0	14%	36.1	22%	27.1	75%	27.2	98%
39.4	18%	39.5	50%	32.8	100%	32.9	100%
43.2%	18%	43.3%	15%	44.0	63%	44.0	61%
47.2	5%	47.2	10%	48.8	15%	49.2	28%
47.5	17%	47.7	23%	49.9	63%	50.2	50%
48.6	17%	48.6	25%	55.7	31%	56.0	24%
57.5	8%	57.5	18%				

To index the reflexes of the diffractograms, the Wolff-Bragg formula and the quadratic form for the hexagonal syngony were used [14]:

$$1/d^2 = 4(h^2 + h \cdot k + k^2)/3 \cdot a^2 + l^2/c^2$$

where a and c are unit cell parameters; $(h\ k\ l)$ are the Miller indices of the reflex. As a result, it was established that reflexes with angular positions: 23.20; 29.60; 36.10; 39.50; 43.30; 47.20; 47.70; 48.60 and 57.50 belong to calcite and have Miller indices (102); (104); (110); (113); (202); (204); (108); (116) and (212), respectively. The vaterite reflexes have Miller indices: (002); (100); (101); (102); (110); (112); (104) and (202), to which the angular positions correspond: 21.10; 25.00; 27.20; 32.90; 44.00; 49.20; 50.20 and 56.00 respectively. Our X-ray structural studies correlate well with the results of works [16 - 18] in which the authors studied synthetic modifications of calcium carbonate.

The Debye-Scherrer formulas were used to determine the size of nanoparticles [19 - 21]:

$$D = 0.89\lambda/(\beta \cos \theta), \quad (19)$$

where λ is the wavelength of X-ray radiation; θ – diffraction angle; β – the half-width of the reflex depends on the size of the nanoparticles (the physical value of the half-width). The experimental value of the half-width of the X-ray reflex β_1 contains two components: the physical β and the instrumental β_2 value. Therefore, the physical value of the half-width is calculated according to the formula:

$$\beta = (\beta_1^2 - \beta_2^2)^{1/2}. \quad (20)$$

The instrumental β_2 half-width value was determined from the X-ray diffraction patterns of Si and Al₂O₃ reference powders.

Using the Debye-Scherrer formula, the dimensions for all reflexes of two modifications of calcium carbonate

were determined and the arithmetic mean of the results obtained for each modification was found. As a result, the following sizes of nanoparticles were obtained: sample in the absence of a magnetic field, calcite – 100 nm, vaterite – 24 nm; sample, which is synthesized in a magnetic field vaterite - 31 nm. For the sample synthesized in a magnetic field, the half-widths of calcite reflexes are proportional to the instrumental half-width value. In this case, it is incorrect to use the Debye-Scherrer formula. This means that the size of the crystallites is of the order of 1 μm and their sizes must be determined by other methods.

Conclusions

Studies have shown that with a significant excess of CO₃²⁻ ions over HCO₃⁻ in the solution (6.6 times in this work) at temperatures of 18-20°C, calcite and vaterite are most likely to be formed, and aragonite is unlikely to be formed.

The process of crystallization of calcium carbonate from an aqueous carbonate solution is significantly influenced by an external magnetic field.

The sizes of calcium carbonate crystallites (calcite and vaterite) in the case of synthesis in a constant magnetic field increase significantly.

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Вплив постійного магнітного поля на кристалізацію карбонату кальцію з карбонатних водних розчинів

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Досліджено вплив постійного магнітного поля на кристалізацію карбонату кальцію з карбонатних водних розчинів. На основі рівнянь для констант дисоціації вугільної кислоти по першій і другій ступенях, констант стійкості комплексів NaCO_3^- і NaHCO_3^0 , рівнянь балансу мас і електронейтральності, визначено значення компонент системи а також співвідношення між компонентами карбонатної підсистеми і встановлено, що основними компонентами в умовах експерименту є CO_3^{2-} , NaCO_3^- та HCO_3^- а основними компонентами карбонатної підсистеми є CO_3^{2-} (86,7%) та HCO_3^- (13,19%). Дослідження показали, що при значному перевищенні у розчині іонів CO_3^{2-} над HCO_3^- при температурах 18 - 20°C з найбільшою ймовірністю утворюються кальцит і ватерит. При кристалізації карбонату кальцію з карбонатного водного розчину в магнітному полі 125 - 250 мТл розміри кристалітів суттєво збільшуються в порівнянні з відсутністю магнітного поля.

Ключові слова: магнітна обробка, кристалізація карбонату кальцію, карбонатна водна система, розчини електролітів, теорія Дебая-Хюккеля, метод ітерацій, рентгено-структурний аналіз, метод Дебая-Шеррера.