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RESEARCH INTO THE PROCESSES OF WOLLASTONITE SYNTHESIS USING A TWO-STAGE TECHNOLOGY

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Abstract. The effect of the mineralogical form and chemical composition of siliceous and calcareous components on the physicochemical processes of calcium silicate and hydrosilicate formation has been investigated under two-stage wollastonite synthesis conditions. It was established that the degree of SiO_2 crystallinity and the presence of foreign impurities in the raw materials have a decisive influence on the formation of the tobermorite phase during hydrothermal treatment. The patterns of microstructure formation of the synthesized products have been studied. It has been shown that amorphous varieties of silica form more crystallized calcium hydrosilicates than quartz. Using XRD and SEM methods, it has been established that the transformation of tobermorite into β-wollastonite occurs within the temperature range of 900–1100 °C. As the burning temperature increases, the degree of wollastonite crystallinity and crystal sizes increase. The optimum temperature for obtaining β-wollastonite with a distinct columnar morphology and crystal sizes of 2–5 μm is 1100 °C. The proposed technology enables the synthesis of wollastonite with controlled structural characteristics at lower temperatures compared to classical methods of solid-phase synthesis. It also allows for reduced energy costs and the use of available secondary raw materials.

Keywords: calcium hydrosilicate, tobermorite, wollastonite, hydrothermal treatment, chemical technologies.

1. Introduction

One of the promising raw materials widely used in various fields of technology and industry is natural and synthetic calcium silicate – wollastonite. Thanks to its unique combination of physical, mechanical, and chemical properties, this mineral is used in the production of traditional and technical ceramics, porcelain, metallurgy, oil-well cements, composites and polymer materials, paints and varnishes, as well as in biomedical practice.[1–8] Wollastonite is characterized by a complex of valuable functional properties: high mechanical strength and thermal stability, stability under high temperatures, low thermal conductivity, and chemical inertness. In addition, it is characterized by high specific electrical resistance, bioactivity, and biocompatibility with human bone tissue, which determines its potential for use in biomedicine.[9–14]

Wollastonite helps improve the performance characteristics of materials, in particular their flexural strength, impact resistance, thermal stability, and durability, while reducing the weight of products and heat loss. In refractory production technology, it is used as a raw material component for the creation of heat-insulating refractory materials, in particular slabs, blocks, and coatings for the thermal protection of furnaces, boilers, and other industrial units. In addition, wollastonite is included in the composition of charges and repair compounds, where it acts as an additive that improves the structure and stability of materials under high-temperature operating conditions. This mineral is particularly important in technologies for producing ceramic products using high-speed firing modes, when the limited duration of the product's stay in the maximum temperature zone does not provide the necessary conditions for the formation of a sufficient number of crystalline phases that determine the required operational properties of the body.[15]

The environmental safety of wollastonite contributes to its gradual replacement of a number of traditional fillers, the use of which harms human health and the environment. Its complex physical and chemical properties make it a multifunctional material for creating environmentally safe and energy-efficient technological solutions and open up wide opportunities for the development of new highly efficient composite and composite materials.

The constant growth in demand for wollastonite, combined with the limited availability and gradual depletion of industrial deposits of this mineral worldwide, makes it particularly important to find new effective

methods for its extraction and to develop innovative synthesis technologies. One promising direction is a two-stage technology that involves hydrothermal synthesis of calcium hydrosilicate of tobermorite composition, followed by its calcination to form calcium metasilicate (wollastonite).

The two-stage method of synthesizing artificial wollastonite is implemented in two stages. At the first stage, the initial raw material mixture (silica and lime) undergoes hydrothermal treatment, resulting in the formation of an intermediate phase – calcium hydrosilicate of tobermorite composition. In the second stage, the resulting product is fired, which ensures its conversion into crystalline calcium silicate, wollastonite. The feasibility of using a two-stage technology for the synthesis of wollastonite compared to the classical method of solid-phase sintering of quartz sand with calcium carbonate is due to a significant reduction in energy costs at the firing stage.[16] This is explained by the fact that low-base calcium hydrosilicates during further heat treatment transition to the wollastonite phase at significantly lower temperatures (approximately 950±50 °C) than in the case of direct interaction of silicon and calcium oxides (approximately 1200±50 °C). The proposed method has prospects for practical application both at the stage of hydrothermal synthesis of calcium hydrosilicate and at the stage of obtaining the final product – wollastonite. At the same time, questions remain regarding the rational selection of raw materials, the establishment of optimal technological parameters for hydrothermal treatment and burning, as well as comprehensive research into the structure and properties of synthesized products. At the same time, taking into account economic feasibility, a promising direction for research is the use of available raw materials and waste from various industries for the synthesis of wollastonite.[17,18] Therefore, the development and improvement of a two-stage technology for the synthesis of artificial wollastonite is of considerable scientific and practical interest.

The aim of the work is to study the influence of the nature of raw materials and impurities present in their composition on the course of tobermorite and wollastonite formation processes and the regularities of microstructure formation in synthesized products.

2. Experimental

2.1. Materials

Quartz sand, amorphous SiO_2 (chemically pure grade), silica, microsilica, calcium oxide (chemically pure grade), and technical lime (grade 1) were used as raw materials for the research. The oxide composition of the starting materials is shown in Table 1.

Table 1. Oxide composition of starting materials

Name	Content of oxides, wt. %						
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O+Na_2O	Lab*
Quartz sand	99.14	0.20	0.05	0.31	0.10	–	0.20
Silica	94.50	0.58	0.44	1.76	0.12	0.25	2.35
Microsilica	94.70	0.38	2.25	0.85	0.74	1.08	–
Technical lime	1.60	1.10	0.40	93.15	1.40	–	2.35

* Losses after burning.

Quartz sand has the same chemical composition as quartz used in glass production and contains a minimal amount of impurities. It was pre-ground using a dry method in a ball mill until it passed through a No. 0063 sieve, after which it was dried to a constant weight.

The waste from the mechanical processing of silica is a by-product of grinding ball production at JSC "Gipsovik." According to X-ray phase analysis (Fig. 1, *a*), it is characterized by a disordered cryptocrystalline structure. This is evidenced by the presence of a halo in the 20–30° range, which corresponds to the most intense lines of β-quartz (d/n = 0.425; 0.335 nm). For further research, the waste was ground in a laboratory ball mill to a residue of no more than 1 % on sieve No. 0063 and dried to a constant weight.

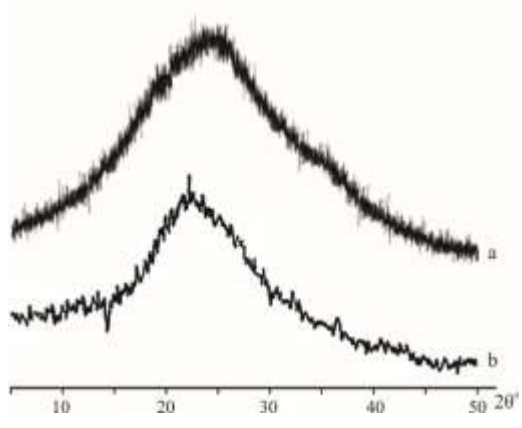


Fig. 1. Diffractograms of silica (a) and microsilica (b)

Microsilica (trademark Elkem Microsilica Grade 940-U) is a by-product of ferrosilicon production. Its diffractogram (Fig. 1, *b*) also has a halo, indicating the presence of cryptocrystalline quartz.

Lump quicklime of the 1st grade (produced by private entrepreneur "Arka-Service") was ground in a laboratory ball mill for 10 h before use.

2.2. Methods

The experimental samples were prepared in the form of a viscous suspension with a water : solid ratio of 1:1 and a molar ratio of SiO_2:CaO of 1:1, which corresponds to the stoichiometric composition of wollastonite. Hydrothermal treatment was carried out at an excess pressure of 1 MPa for 10 h. After autoclaving, the samples were burned in a muffle electric furnace at temperatures of 900, 1000, and 1100 °C with isothermal holding at the maximum temperature for 2 h.

The degree of interaction between silica and lime (completeness of the reaction) was evaluated by the content of residual unreacted $Ca(OH)_2$ after autoclaving, which was determined by complexometric titration.

The true density of the synthesized products was determined by the pycnometric method.

The phase composition of the samples was studied using diffractograms obtained on a modernized DRON-3M diffractometer using copper Kα radiation (λ = 0.154185 nm). The interpretation of the diffractograms was carried out in accordance with the Crystallography Open Database (COD), an open database of crystal structures.

The processes during the burning of the masses were studied using a Q-1500D derivative thermos-gravimetric analyzer of the Paulik-Paulik-Erdei system connected to a personal computer. The samples were analyzed in the temperature range of 20–1000 °C in dynamic mode at a heating rate of 10 °C/min in an air atmosphere. Aluminum oxide served as the reference substance. The weight of the samples was 100 mg.

Electron microscopic studies were performed on a REM-16I (Selmi) scanning electron microscope. To increase the conductivity of the samples, a copper conductive film was applied to their surface by thermal vacuum deposition. The film thickness did not exceed 50 nm. Micrographs were processed using computer morphometry tools.

3. Results and Discussion

The physical prerequisite for the interaction between SiO_2 and $Ca(OH)_2$ is an increase in the solubility of silica in water under hydrothermal treatment conditions. Under normal conditions (0.1 MPa, 20±2 °C), due to the extremely low solubility of silica in water, there is practically no chemical interaction between crystalline SiO_2 and $Ca(OH)_2$. The solubility of silica significantly depends on its structural state (crystalline or amorphous), the degree of order of the atomic structure, dispersibility, and the conditions of determination. Thus, for crystalline forms of SiO_2, it is 0.003–0.012 g/L, while for amorphous forms it is 0.022–0.225 g/L. During hydrothermal treatment, the solubility of SiO_2 increases significantly with increasing temperature and water vapor pressure. For example, at a temperature of 190 °C and an excess pressure of 1.255 MPa, the solubility of SiO_2 amorphous forms reaches 0.872 g/L. This creates the necessary conditions for intensive interaction of dissolved silicon oxide with calcium hydroxide and the formation of calcium hydrosilicates, the basicity of which will be determined by the ratio between SiO_2 and CaO.[19]

As is well known, the nature and degree of purity of the raw materials significantly influence the rate and completeness of their interaction under hydrothermal treatment conditions. Therefore, to study the effect of the degree of order of the SiO_2 structure on the intensity of interaction between the siliceous and lime components, experimental samples were prepared, the charge compositions of which are given in Table 2. The same table shows the results of complexometric titration after hydrothermal treatment of the samples.

Table 2. Charge compositions and results of complexometric titration of experimental samples

Sample number	Siliceous component	Lime component	Content of residual $Ca(OH)_2$, %
1	SiO_2 cryst.	CaO*	31.3
2	SiO_2 cryst.	CaO (technical lime)	27.4
3	SiO_2 amorph.*	CaO*	14.0
4	SiO_2 amorph.*	CaO (technical lime)	6.1
5	silica	CaO (technical lime)	3.0
6	microsilica	CaO (technical lime)	2.6

* Chemically pure

The obtained results confirmed that the completeness of the interaction between SiO_2 and $Ca(OH)_2$ primarily depends on the degree of structural order of the silica and fully correlates with its solubility in water for different degrees of structural order. Thus, even after 10 h of autoclaving quartz sand-based mixtures (samples No. 1 and 2), the residual content of unreacted calcium hydroxide ranged from 27.4 to 31.3 %. This reaction is much more intense in mixtures based on amorphous forms of SiO_2: for chemically pure amorphous SiO_2 (samples No. 3 and 4), the residual $Ca(OH)_2$ content was 14.0 and 6.1 %, respectively, for quartz (sample No. 5) and microsilica (sample No. 6) – 3.0 and 2.6 %, respectively. Obviously, the lower the order of the SiO_2 structure, the more intense its dissolution in water and the formation of calcium hydrosilicate.

Along with the structural features of silica, the presence of foreign impurities (Al_2O_3, Fe_2O_3, MgO, K_2O+Na_2O) in technical raw materials, which act as catalysts for physicochemical transformations, has a significant impact on the completeness of its interaction with calcium hydroxide. During hydrothermal treatment for both components, silica and lime, the use of technical products, due to the presence of impurities in their composition, ensures a greater degree of interaction between the starting components, which is confirmed by the lower content of unreacted $Ca(OH)_2$ in the samples after autoclaving. Thus, for compositions based on crystalline quartz, replacing chemically pure CaO (sample No. 1) with technical lime (sample No. 2) reduces the amount of unreacted $Ca(OH)_2$ after autoclaving from 31.3 to 27.4 %. A similar pattern is observed for amorphous chemically pure SiO_2: after interaction with chemically pure CaO (sample No. 3), the content of unreacted $Ca(OH)_2$ is 14.0 %, and when using technical lime (sample No. 4), it decreases to 6.1 %. The minimum values of residual $Ca(OH)_2$ are recorded for the samples in which technical lime was combined with quartz (sample No. 5) – 3.0 % and with microsilica (sample No. 6) – 2.6 %.

The dependence of the rate and completeness of the interaction of silica with slaked (hydrated) lime on the nature and purity of raw materials is fully consistent with the results of X-ray phase and electron microscopic analysis. The diffractogram of sample No. 1 (Fig. 2, *a*), which is based on pure quartz sand and slaked lime shows intense diffraction maxima of unreacted quartz (d/n = 0. 424, 0.334, 0.245, 0.228, 0.223, 0.212, 0.154, 0.148 nm) and portlandite (d/n = 0.494, 0.310, 0.263, 0.192, 0.179, 0.168, 0.144 nm), as well as very weak in intensity maxima of calcium hydrosilicate (d/n = 1.130, 0.308, 0.281 nm), indicating the initial stage of tobermorite crystallization. This is consistent with the complexometric titration data and indicates a low reaction rate between silica and the lime component. Replacing quartz sand with chemically pure amorphous silica in combination with chemically pure lime (sample No. 3, Fig. 2, *b*) significantly accelerates the chemical interaction between the components. Diffractogram shows a significant decrease in the intensity of the residual $Ca(OH)_2$ maxima and an increase in the intensity of the calcium hydrosilicate maxima (d/n = 1.130, 0.540, 0.308, 0.297, 0.281, 0.183 nm). Weak quartz reflections on the diffractogram indicate that the amorphous component of quartz is primarily involved in the formation of calcium hydrosilicate, while the system is enriched with crystalline SiO_2. The use of technical raw materials (sample No. 5, Fig. 2, *c*) ensures maximum interaction between components. This is confirmed by the intense reflexes of tobermorite (d/n = 1.130, 0.540, 0.308, 0.297, 0.281, 0.228, 0.219, 0.183, 0.167 nm) and the absence of portlandite reflections on the diffractogram, as well as the minimum content of unreacted quartz.

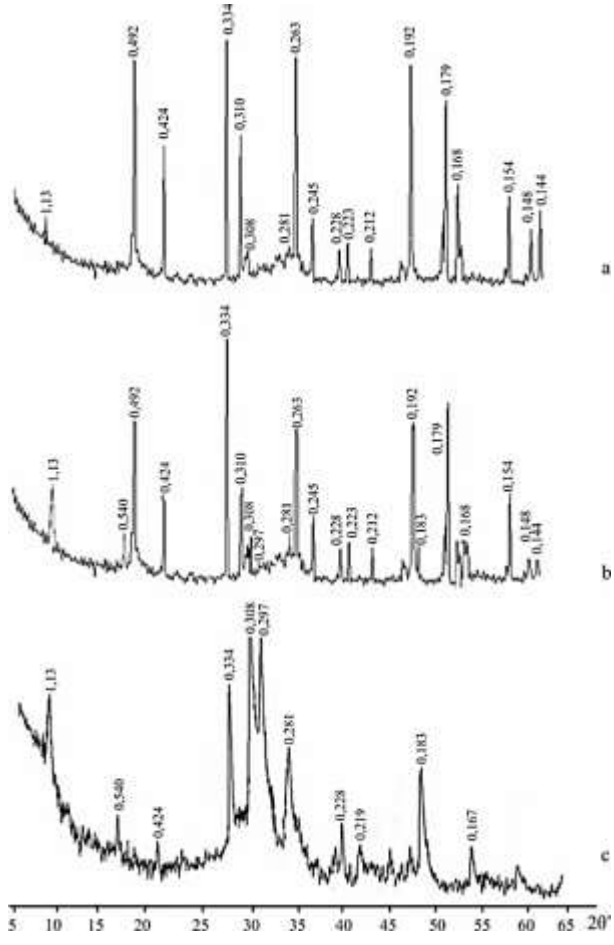
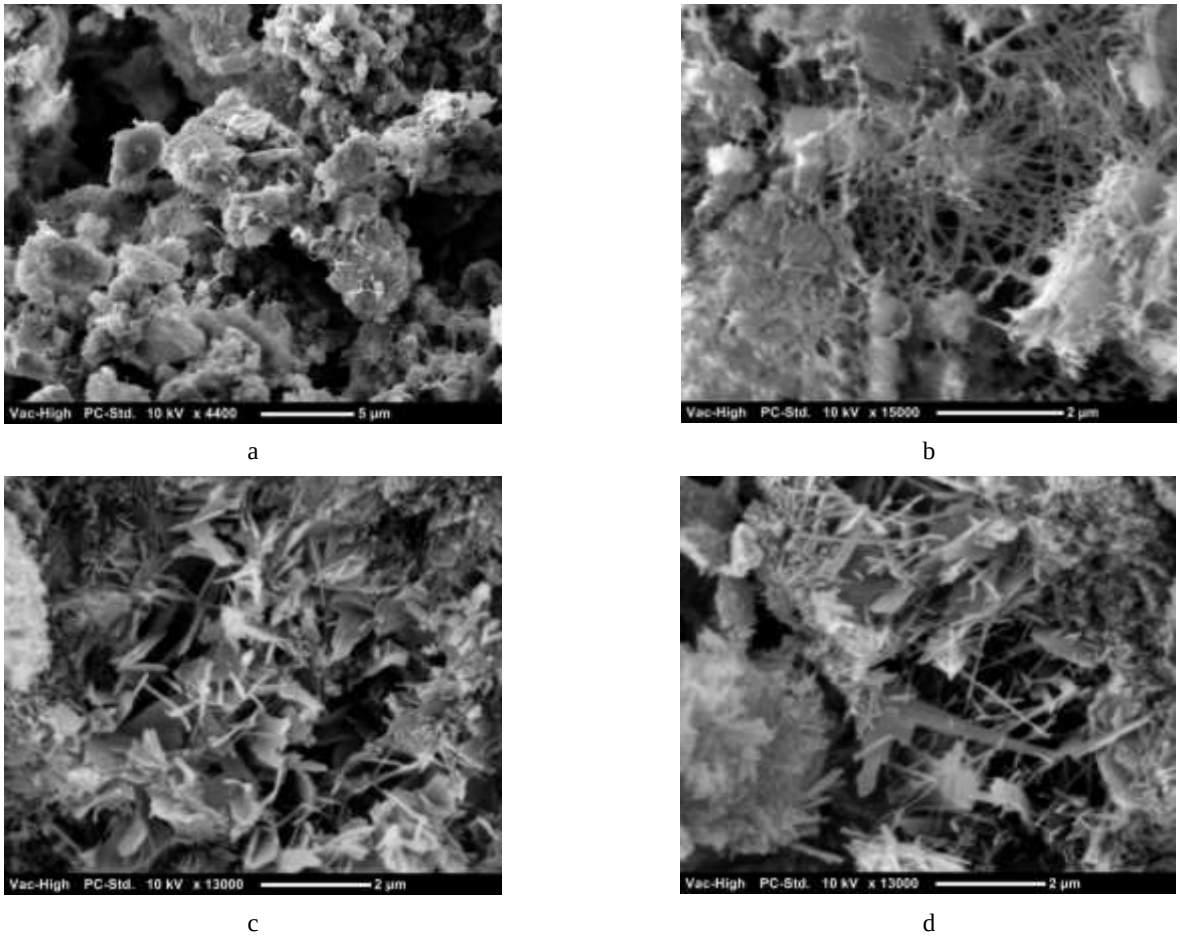


Fig. 2. Diffractograms of the samples after autoclaving: No. 1 (a), No. 3 (b), No. 5 (c)

The results of electron microscopic study also confirm the low intensity of interaction between crystalline SiO_2 and chemically pure $Ca(OH)_2$ (sample No. 1, Fig. 3, *a*). In general, the microstructure of the mentioned samples is characterized by significant morphological heterogeneity and is represented mainly by a porous matrix, consisting of strongly corroded silica grains covered with an amorphous gel-like substance. In the pore space of this matrix, there are areas where tobermorite is formed in the form of thin, chaotically intertwined fibers (Fig. 3, *b*). However, in general, there are not many such areas, which is consistent with the results of X-ray phase analysis.

In contrast, for the sample based on quartz and technical lime (sample No. 5, Figs. 3, *c*, *d*), a high degree of crystallization of the matrix with needle-like and lamellar formations of tobermorite was observed. At the same time, while for crystalline SiO_2 (Fig. 3, *b*) the crystallization of calcium hydrosilicate occurs only in separate areas of the sample's pore space in the form of ultrafine thread-like formations, for the sample based on quartz, crystallization covers the entire sample array. The structure of the latter is represented by a clearly defined crystalline phase of tobermorite with a practical absence of silica grains and gel-like substances.



a b c d

Fig. 3. Microstructure of the autoclave samples: sample No. 1 (a, b), sample No. 5 (c, d)

The second stage of wollastonite synthesis involves tobermorite burning to recrystallize it into calcium silicate. According to thermal studies for a quartz sand-based sample (sample No. 1), two endothermic processes were recorded on the DTA curves (Fig. 4, *a*). The first endothermic effect occurs at relatively low temperatures, with a maximum of approximately 100 °C. In the temperature range of up to 460 °C, a gradual mass loss of up to 2.5 % is observed, which is due to the release of hygroscopic moisture and interlayer water from gel-like weakly crystallized calcium hydrosilicates. The obtained results are in agreement with the data represented in the literature.[20] The absence of a clearly defined crystalline form of calcium hydrosilicates and the presence of a significant amount of calcium-silicate gel are further confirmed by the results of X-ray phase and electron microscopic analyses.

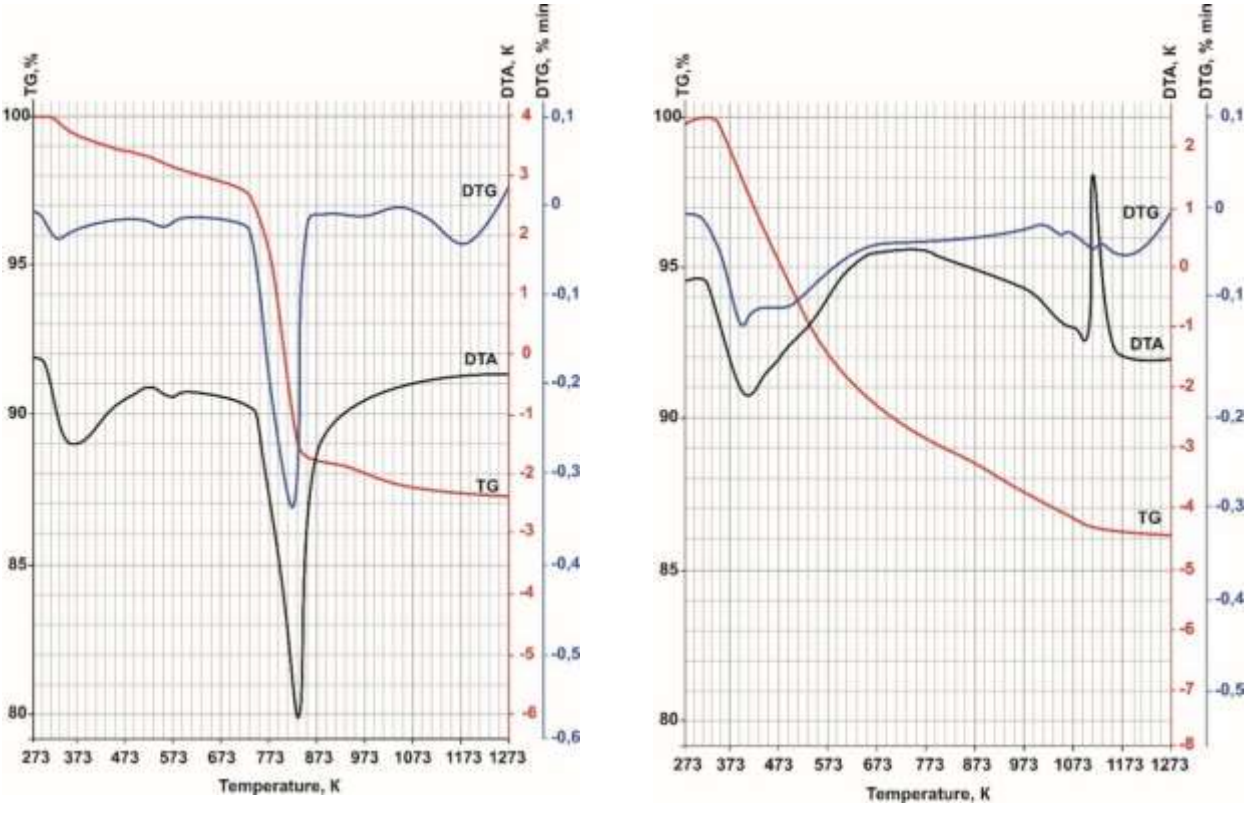


Fig. 4. Thermograms of the samples after autoclaving: No. 1 (a) and No. 5 (b)

During further heating, a second intense endothermic effect is recorded on the DTA curve in the temperature range of 460–620 °C with a maximum at 550 °C, accompanied by a mass loss of 8.8 %. This effect is caused by the dehydration of $Ca(OH)_2$, a significant amount of which in the sample is confirmed by complexometric titration. Further heating to 1000 °C is accompanied by a slight mass loss (1.2 %), which corresponds to the removal of chemically bound water residues from the tobermorite gel.

A fundamentally different nature of thermal processes is observed in the thermogram of the sample based on quartz and technical lime (sample No. 5, Fig. 4, *b*). The DTA curve shows a deeper endothermic effect in a much wider temperature range from 80 to 390 °C, caused by the release of hygroscopic water and bound water from crystalline hydrosilicates. At the same time, there is no endothermic effect with a maximum at 550 °C, characteristic of $Ca(OH)_2$ decomposition. Instead, an intense exothermic effect is observed in the temperature range of 820–900 °C with a maximum at 840 °C, which corresponds to the recrystallization of dehydrated tobermorite into wollastonite.

Taking into account the results of complexometric titration, X-ray phase, electron microscopic, and differential thermal analyses, a mixture based on quartz and technical lime (sample No. 5) was selected for further burning. The sample is characterized by a high degree of interaction between SiO_2 and $Ca(OH)_2$ and the formation of calcium hydrosilicate with a distinctly crystallized structure, which provides optimal conditions for the synthesis of low-temperature wollastonite.

The samples were burnt for 10 h at temperatures of 900, 1000, and 1100 °C with isothermal holding at the maximum temperature for 2 h. X-ray phase analysis shows that after burning under all specified conditions, all samples show a single crystalline phase – β-wollastonite (d/n = 0.760, 0.383, 0.352, 0.331, 0.297, 0.218, 0.183, 0.172 nm). With an increase in the maximum burning temperature from 900 to 1100 °C, there is an increase in the intensity of the diffraction maxima of wollastonite (Fig. 5), which is due to the ordering of the mineral crystal structure. The increase in the degree of order of the crystal structure is also evidenced by an increase in the true density of the test samples, g/cm^3:

- initial tobermorite – 2.39;
- wollastonite (T_{max} = 900 ºC) – 2.64;
- wollastonite (T_{max} = 1000 ºC) – 2.90;
- wollastonite (T_{max} = 1100 ºC) – 2.92.

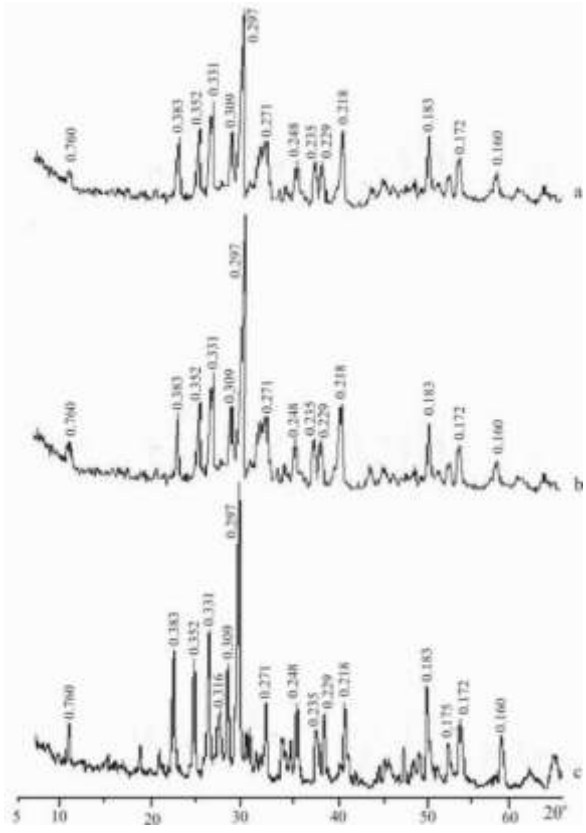
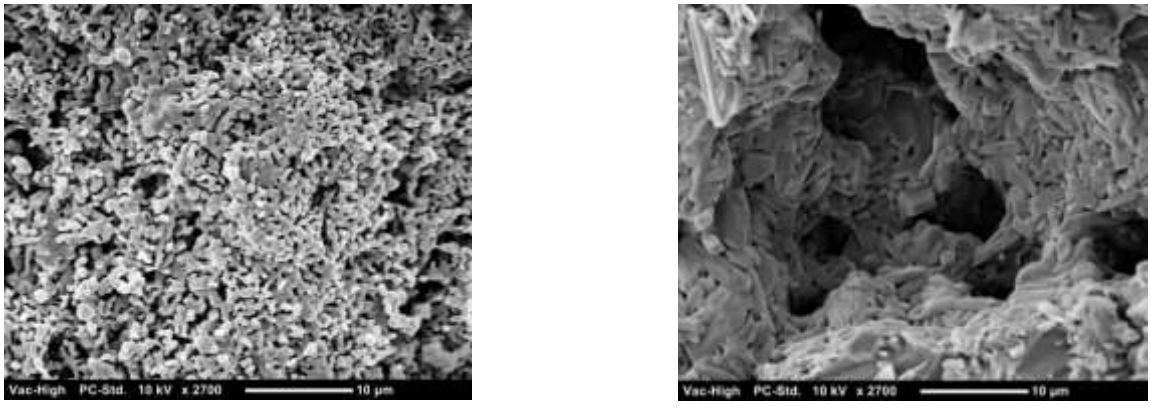


Fig. 5. X-ray images of sample No. 5 after burning at 900 ºC (a), 1000 ºC (b), and 1100 ºC (c)

Electron microscopic studies of the structure of burnt samples also confirm the dependence of the intensity of crystallization processes on the maximumburning temperature. Thus, the microstructure of samples burnt at a maximum temperature of 1000 °C (Fig. 6, *a*) is represented by a sintered conglomerate of rounded wollastonite crystals approximately 0.5–1.5 μm in size. When the maximum burning temperature is increased to 1100 °C (Fig. 6, *b*), the calcium silicate crystals acquire a distinctly faceted columnar shape, and their size increases to 2–5 μm.

4. Conclusions

The studies conducted have established the influence of the nature and chemical composition of raw materials on the physicochemical processes of tobermorite and wollastonite formation in the two-stage synthesis technology. It has been shown that in the first stage of hydrothermal synthesis in the CaO-SiO_2-H_2O system, the structure of silica and the presence of foreign impurities in raw materials have a decisive influence on the completeness of the interaction between the siliceous and lime components and, accordingly, on the intensity of tobermorite synthesis. It has been established that amorphous and cryptocrystalline forms of SiO_2, in particular quartz and microsilica, as well as impurities in technical materials, ensure the maximum completeness of calcium hydrosilicate formation and a high degree of its crystallinity. In contrast, crystalline quartz reacts much more slowly with calcium hydroxide during hydrothermal treatment. The result is that under the same synthesis conditions, the autoclaved product contains significantly less tobermorite. For economic reasons, it is advisable to use silica, which is a widely available and inexpensive raw material, as the starting material for the synthesis of wollastonite.



a b

Fig. 6. Microstructure of wollastonite synthesized at 1000 ºC (a) and 1100 ºC (b)

X-ray phase and electron microscopic analyses confirmed that increasing the burning temperature from 1000 to 1100 °C contributes to the ordering of the crystalline structure of wollastonite and an increase in crystal size from 0.5–1.5 to 2–5 μm, respectively. Studies have shown that the optimal conditions for obtaining β-wollastonite with high

crystallinity and minimum content of unreacted $Ca(OH)_2$ are the use of quartz and technical lime under excess pressure of 1 MPa with a holding time of 10 h during hydrothermal treatment, and a maximum burning temperature of 1100 °C with a holding time of 1 hour.

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ДОСЛІДЖЕННЯ ПРОЦЕСІВ СИНТЕЗУ ВОЛАСТОНІТУ ЗА ДВОСТАДІЙНОЮ ТЕХНОЛОГІЄЮ

Анотація. *Досліджено вплив мінералогічної форми та хімічного складу кремнеземистих і вапняних компонентів на перебіг фізико-хімічних процесів утворення гідросилікатів і силікатів кальцію в умовах двостадійної технології синтезу воластоніту. Встановлено, що ступінь кристалічності SiO_2 та наявність домішок у сировині визначально впливають на формування тоберморитової фази під час гідротермального оброблення. Вивчено закономірності формування мікроструктури синтезованих продуктів. Показано, що аморфні різновиди кремнезему забезпечують максимальне утворення закристалізованих гідросилікатів кальцію порівняно з кварцом. Методами РФА та ЕМА встановлено, що перетворення тоберморіту на β-воластоніт відбувається в інтервалі температур 900–1100 °С. Зі збільшенням температури випалювання підвищується ступінь кристалічності воластоніту та збільшується розмір кристалів. Оптимальною температурою для отримання β-воластоніту з виразною стовпчастою морфологією та розмірами кристалів 2–5 мкм є 1100 °С. Запропонована технологія забезпечує синтез воластоніту з контрольованими структурними характеристиками за знижених температур, а також можливість зменшити енергетичні витрати порівняно з класичними методами твердофазового синтезу та використовувати доступну вторинну сировину.*

Ключові слова: *гідросилікат кальцію, тоберморит, воластоніт, гідротермальне оброблення, хімічні технології.*