DOI: 10.6060/ivkkt.20246704.6947

УДК: 666.189

ФИЗИКО-ХИМИЧЕСКИЕ ПРОЦЕССЫ ЩЕЛОЧНОГО АКТИВИРОВАНИЯ КРЕМНЕЗЕМА ПРИ ТЕРМООБРАБОТКЕ В СИСТЕМЕ SiO_2-NaOH-H_2O

К.В. Скирдин, О.В. Казьмина, В.И. Верещагин, И.Е. Рыманова

Кирилл Вячеславович Скирдин (ORCID 0000-0002-6564-3280)*, Ольга Викторовна Казьмина (ORCID 0000-0001-5285-3329), Владимир Иванович Верещагин, Ирина Евгеньевна Рыманова (ORCID 0000-0002-7780-5419)

Томский политехнический университет, ул. Ленина, 30, Томск, Российская Федерация, 634034
E-mail: skirlin.kirill@mail.ru*, kazmina@tpu.ru, vver@tpu.ru, rymanova@tpu.ru

Исследованы физико-химические процессы, протекающие при щелочном активировании композиции системы SiO_2-NaOH-H_2O с кристаллическим кремнеземом. Исследования проводились с помощью термогравиметрического, рентгенофазового методов анализа и данных ИК-спектроскопии. Установлены зависимости свойств композиции от величины силикатного модуля (SiO_2/Na_2O 4-7), количества микрокремнезема (10-30%), вводимого взамен маршалита, и концентрации раствора гидроксида натрия (30-60 мас.%). По результатам исследования предложены реакции взаимодействия компонентов и модель фазовых превращений в композиции при ее нагревании до 850 °С. На этапе взаимодействия компонентов (30-130 °С) происходят процессы гидратации с образованием гидросиликата натрия $Na_2SiO_3(OH)$, на поверхности которого формируется слой кристаллогидратной воды и водной оболочки. При этом свободная вода отсутствует. При нагревании композиции до температур 130-300 °С удаляется водная оболочка и далее кристаллогидратная вода. Удаляемая вода вступает во взаимодействие с непрореагировавшим кремнеземом с образованием гидратированных форм кремнезема. При дальнейшем нагревании до температур 310-800 °С гидросиликаты натрия и гидратированные формы кремнезема переходят в безводные силикаты. Нагрев композиции до 850 °С приводит к образованию пиропластичной массы из эвтектического расплава ($Na_2O\cdot 2SiO_2$–SiO_2) и остаточного кремнезема. Установлен двухстадийный механизм формирования пористого каркаса композиции. На первой стадии (температуры 80-200 °С) происходит разложение кристаллогидратов гидросиликата натрия, и на второй (температуры 788-850 °С) - вспенивание расплава. Вспенивание происходит за счет удаления паров воды (80-200 °С) и расширения объема газов (788-850 °С) в пористой структуре, образованной на первом этапе вспенивания. Разработанный состав высокомодульной композиции (SiO_2/Na_2O 5,7) служит в качестве основы для получения пористого стеклокомпозита по одностадийной щелочной технологии, при введении дополнительных оксидов, повышающих химическую стойкость. Состав включает следующие компоненты, мас.%: маршалит – 50, микрокремнезем – 23, гидроксид натрия – 16, вода – 11.

Ключевые слова: пористый стеклокомпозит, маршалит, микрокремнезем, гидроксид натрия, гидросиликаты, процессы силикатообразования

PHYSICO-CHEMICAL PROCESSES OF ALKALINE ACTIVATION OF SILICA DURING HEAT TREATMENT IN THE SiO_2-NaOH-H_2O SYSTEM

K.V. Skirdin, O.V. Kazmina, V.I. Vereshchagin, I.E. Rymanova

Kirill V. Skirdin (ORCID 0000-0002-6564-3280)*, Olga V. Kazmina (ORCID 0000-0001-5285-3329), Vladimir I. Vereshchagin, Irina E. Rymanova (ORCID 0000-0002-7780-5419)

Tomsk Polytechnic University, Lenin st., 30, Tomsk, 634034, Russia
E-mail: skirlin.kirill@mail.ru*, kazmina@tpu.ru, vver@tpu.ru, rymanova@tpu.ru

The physicochemical processes during alkaline activation of the SiO_2-NaOH-H_2O system with crystalline silica have been investigated. The studies were carried out using thermogravimetric, X-ray phase analysis methods and IR spectroscopy data. Compositions with different silicate modulus (SiO_2/Na_2O 4-7), the amount of the introduced silica fume (10-30%) in substitution of marshalite and the concentration of sodium hydroxide solution (30-60 wt.%) are considered. According to the results of the study, the reactions of interaction of the components and a model of phase transformations in the composition when it is heated to 850 °C are proposed. At the stage of interaction of the components (30-130 °C), hydration processes occur with the formation of sodium hydrosilicate $Na_2SiO_3(OH)$, with on the surface of which a layer of crystallohydrate water and a water layer to be formed without formation of free water. When the composition is heated to temperatures of 130-300 °C the water layer and then the crystallohydrate water are removed. The removed water interacts with unreacted silica and forms hydrated forms of silica. Upon further heating to temperatures of 310-800 °C, OH groups are removed from sodium hydrosilicates and hydrated forms of silica and it turns into anhydrous silicates. Heating the composition to 850 °C leads to the formation of a pyroplastic mass from a eutectic melt ($Na_2O{\cdot}2SiO_2$–SiO_2) and residual silica. The two-stage mechanism of formation of a porous frame is established at the stage of decomposition of sodium hydrosilicate crystallohydrates (80-200 °C) and at the stage of melt foaming at high temperatures (790-850 °C). Foaming occurs due to the removal of water vapor (80-200 °C) and the expansion of the volume of gases (790-850 °C) in the porous structure formed at the first stage of foaming. The developed compound of the high-modulus composition (SiO_2/Na_2O 5,7) is a basis to obtain a porous glass composite using a two-stage alkaline technology with the introduction of additional oxides that increase chemical resistance. The composition includes the following components, wt.%: marshalite – 50, silica fume – 23, sodium hydroxide – 16, water – 11.

Key words: porous glass composite, marshalite, silica fume, sodium hydroxide, hydrosilicates, silicate formation

Для цитирования:
Скирдин К.В., Казьмина О.В., Верещагин В.И., Рыманова И.Е. Физико-химические процессы щелочного активирования кремнезема при термообработке в системе SiO_2-NaOH-H_2O. *Изв. вузов. Химия и хим. технология*. 2024. Т. 67. Вып. 4. С. 108–114. DOI: 10.6060/ivkkt.20246704.6947.

For citation:
Skirdin K.V., Kazmina O.V., Vereshchagin V.I., Rymanova I.E. Physico-chemical processes of alkaline activation of silica during heat treatment in the SiO_2-NaOH-H_2O system. *ChemChemTech [Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.]*. 2024. V. 67. N 4. P. 108–114. DOI: 10.6060/ivkkt.20246704.6947.

INTRODUCTION

Current studies in the field of porous fiberglass composites are in focus to find available, reasonable raw materials and less energy consuming synthesis methods. Recently silica melt foaming with sodium hydroxide (alkaline technology) has been studing. This is due to high reactivity of alkali which actively reacts with silica and decreases the melting point of the composition [1-9].

A method of obtaining of porous materials with crystalline silica raw materials on a two-stage alkaline technology is known [10-11]. Silicate formation is based on the interaction of silica with soda ash and synthesis of glass granulate. Foaming of the mixture obtained from glass granulate occurs at the second stage.

An alkaline technology refers to a single-stage process being its advantage and allows us to reduce energy consumption. At the same time this technology is used only with amorphous silica raw materials. Physico-chemical processes of alkali activation of amorphous raw materials with SiO_2 70-87% (diatomite, tripolith, gaize) have been described in literature [12-16].

However, the processes occurring during alkali activation of crystalline silica raw materials, such as marshlite (96% SiO_2) have not been studied yet. Consequently, the study of the processes of alkali activation of silica raw materials while heat treatment is crucial. The obtained results allow us to simulate physico-chemical processes to obtain porous fiber-glass composite using the single-stage alkali technology.

The aim of this study is to establish the consistent pattern of physico-chemical processes occurring By heating the marshalite-sodium hydroxide-water composition up to 850 °C to develop a composition suitable for obtaining porous fiber-glass composite from quartzous raw material bases using a single-stage technology.

MATERIALS AND METODS

In the study silica materials in the form of natural marshalite and manmade silica fume as a by-product from the ferro-alloy production were used. Marshalite and silica fume with small amount of impurities refers to highly dispersive silica materials. An average particle size of marshalite is 10 μm, an average particle size of silica fume is 20 μm.

Table

Chemical composition of silica components

***Таблица.* Химический состав кремнеземистых компонентов**

Raw materials	Content of oxides, wt.%					
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Δm
marshalite	95,7	2,1	0,3	1,0	0,4	0,5
silica fume	91,7	0,9	0,5	1,2	0,9	4,8

Note: Δm – mass loss ignition

Примечание: Δm – потеря массы при прокаливании

The relative intensity of the basic reflex (I_{rel}) of quartz was estimated in relation to the intensity of the basic reflex in the material and in the raw material, according to the formula:

$$I_{rel}=\frac{I_{mat}}{I_{source}}\cdot 100\%,$$

where I_{rel} is the relative intensity, %; I_{mat} and I_{source} are the intensities of quartz in the foamed material and the initial marshalite, imp./sec.

Properties of raw materials and processes in the composition during alkaline activation were analyzed using as follows: thermal gravitational analysis (thermoanalyzer «SDT Q600»), laser analyzer («Shimadzu SALD-7101») and IR spectrophotometry (spectrophotometer «IRPrestige-21»).

RESULTS AND DISCUSSION

Recently the positive impact of an active agent on obtaining porous glass composite at the single-stage alkali technology [17] has been shown. Consequently, the study focused on investigation of the effect of different parameters on physico-chemical processes of sodium silicate formation from hydrosilicate obtained by alkali activation was considered on marshalite and silica fume compositions. The controlled parameters were chosen as follows: SiO_2/Na_2O ratio (the silicate module) in the range of 4 to 7, the concentration of sodium hydroxide (30-60 wt.%), the amount of silica fume (10-30 wt.%), the solid and liquid phase in the initial stage (from 3 to 8). The influence of these parameters on the processes while obtaining porous material was evaluated according the thermogravimetric analysis results.

There are three specific sectors on the thermogravimetric curves obtained for the tested compositions (Fig. 1). Every sector corresponds to definite processes occurring when heated the composition, is characterized by mass loss (Δm_2 and Δm_3) and the temperature corresponding to the process, i.e. T_1- the temperature of beginning of the sample mass loss, T_2 – the minimum temperature of the second sector, at which the mass loss is not more than 1% at temperature fluctuation on 10 °C, T_3 – the temperature of completion of silication (when the curve reaches the straight line).

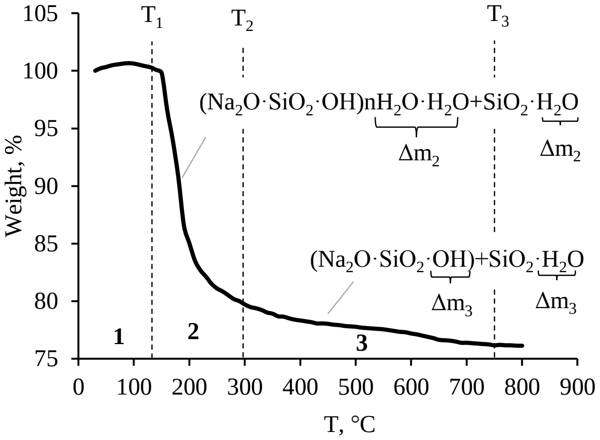


Fig. 1. Thermogravimetric curve of the composition with SiO_2/Na_2O=5.7 and 30 wt.% of silica fume and solution concentration of 60 wt.% NaOH

Рис. 1. Термогравиметрическая кривая композиции с SiO_2/Na_2O=5,7 и 30 мас.% микрокремнезема и концентрации раствора 60 мас.% NaOH

At the first sector (30-130 °C) the interaction reaction of alkaline solution with particles of silica fume and the surface of marshalite particles occurs. As a result, crystallohydrates of hydrosilicate are formed according to Scheme 1.

$$mSiO_2+2nNaOH+pH_2O\rightarrow$$
$$\rightarrow n(Na_2O\cdot SiO_2\cdot OH)\cdot xH_2O+(m-n)SiO_2\cdot(p-x)H_2O \quad (1)$$

The water introduced into the composition has been established to be completely bound into sodium hydrosilicates ($Na_2O\cdot SiO_2\cdot OH$) on the surface of which a layer of water is formed in a crystalline form (xH_2O). A water layer is formed on the surface of crystalline hydrates and unreacted silica ($(p-x)H_2O$) in the case of the presence of water in excess of a whole number of moles.

At a temperature of 80-130 °C, the crystalline hydrates of sodium hydrosilicate melt to form a viscous plastic mass. In parallel, foaming processes occur due to the removed water up to 180-200 °C at which the porous base gets solid.

The second sector (130-310 °C) has a maximum mass loss. It happens due to removal of the water layer and the water in crystallohydrate form Scheme 2:

$$n(Na_2O\cdot SiO_2\cdot OH)\cdot xH_2O\cdot H_2O\uparrow + SiO_2\cdot H_2O\uparrow \rightarrow$$
$$\rightarrow n(Na_2O\cdot SiO_2\cdot OH)\cdot xH_2O\uparrow + SiO_2 \qquad (2)$$

At the same time, hydration of unreacted silica occurs by the removed water occurs to form hydrated silica forms.

The mass loss in the third sector in the temperature range from 310 to 800 °C is associated with removal of OH groups from sodium hydrosilicate and hydrated silica forms with further completion of silication and the curve becoming direct. When the temperature reaches 788 °C, melting of a eutectic composition occurs according to the Scheme 3:

$$Na_2O\cdot 2SiO_2 + SiO_2 \xrightarrow{788^{o}C} \text{eutectic melt} + SiO_2 \qquad (3)$$

At the temperature 788-850 °C silica partially dissolves in the obtained eutectic melt, with the viscosity of pyroplastic mass to increase. The second stage of foaming is carried out owing to thermal expansion of gases being in the pores of the material.

$$\text{melt}+SiO_2 \xrightarrow{800\text{-}850^{o}C} \text{melt}+SiO_2 \xrightarrow{\text{cooling}}$$
$$\xrightarrow{\text{cooling}} \text{glass phase}+SiO_2 \qquad (4)$$

Thus, mass loss in the second sector (130-310 °C) corresponds to the removal of water layer and the water from crystallohydrates of sodium hydrosilicate. The mass loss in the third sector (310-800 °C) is due to removal of OH groups from sodium hydrosilicate and hydrated silica forms. Consequently, the value of mass loss in the third sector also is determined by the amount of the removed water in crystallohydrated form, which later interacts with silica to form hydrated forms of silica.

Comparative analysis of the thermal gravitational curves of the investigated composition has shown:

1. While increasing the SiO_2/Na_2O ratio from 4 to 7 the temperature (T_2) values decrease by 10-60 °C in the second sector of the thermal gravitational curves due to decrease of amount of crystallohydrates of sodium hydrosilicate.

2. While increasing the amount of silica fume from 10% to 30% the temperature value (T_2) decreases by 30-50 °C, with the mass loss of the second sector (Δm_2) to decrease by 1-3%. The established dependences of are connected with intensification of interaction of amorpous silica leading to decrease of the amount of water in a crystallohydrate form.

3. The influence of the concentration of sodium hydroxide solution is revealed in the second sector of the composition treatment. With an increase in the concentration of sodium hydroxide solution by 20%, the mass loss (Δm_2) decreases on average by 6-10%, which is associated with a decrease in the amount of water in a crystalline form.

To sum up the results of thermal gravitational analysis, the content of silica fume has been established to have the biggest effect from all considered factors, then silica module, and further the concentration of sodium hydroxide solution. Heating the composition up to 800 °C leads to not only foaming the mixture, but the decrease of the remaining unreacted silica at the silica formation stage and being undissolved in the obtaining melt.

Structural transition in the composition while heating are confirmed with IR spectrophotometry (Fig. 2) and explained the parameter dependency T_2-T_3, Δm_2-Δm_3 (Fig. 4-5). According to the IR spectrophotometry parameters, the water in the composition was determined to be in valency (3620 cm^{-1}) and deformation forms (1450 cm^{-1}). Presence of water in crystalline deformation form confirms the presence of sodium hydrosilicate crystallohydrates. A valency form indicates the presence of small amount of water with weak (3620 cm^{-1}) hydrogen bonds. Absence of specific band (1620 cm^{-1}) denotes the absence of water in a free form in the composition. IR spectrum 470 cm^{-1} (Si-O), 695 cm^{-1} (O-Si-O) with the stretch vibration of siloxane linkages 780 and 1080 cm^{-1} corresponds to the strained vibration of siloxane linkages. Availability of the spectrum (2370 cm^{-1}) of the strong strained hydrogen bond of hydroxygroups with nonbridging oxygen of tetrahedral units Q_3 (or Q_2) confirms the presence of the hydrated silica forms [18-20].

The decrease in intensity of transmission spectrum corresponding to siloxane linkages (470 cm^{-1}, 695 cm^{-1}, 780 cm^{-1}, 1080 cm^{-1}) while heating up to 220 °C indicates that the reactions of silica interaction are carried out. The decrease in transmission spectrum intensity (1450 cm^{-1}) specific for crystallohydrated water on heating confirms that mass loss at the second sector happens due to removal of crystallohydrated water. The increase in intensity of transmission spectrum 2370 cm^{-1} on heating informs about the temperature intensification of the processes to obtain hydrated silica forms.

According to the established dependences the phase transformation model in the marshalite-silica fume-sodium hydroxide-water system on heating was offered (Fig. 3). Initially (30-130 °C), interaction of sodium hydroxide solution with silica fume and the surface of marshalite particles is carried out to form hydrosilicates and crystallohydrates of sodium, a water layer on their surfaces and unreacted silica (Fig. 3, a). When the temperature rises to 130-300 °C dehydratation first of water layer and then of the crystalline hydrates of sodium hydrosilicate (Fig. 3, b) occurs. On the heating process a part of crystallohydrated water reacts with unreacted silica to form hydrated silica.

At a temperature from 300 °C to 850 °C OH groups are removed from sodium hydrosilicate and hydrated silica forms. On reaching the eutectic point the eutectic composition melts and forms foaming pyroplastic mass (Fig. 3, в).

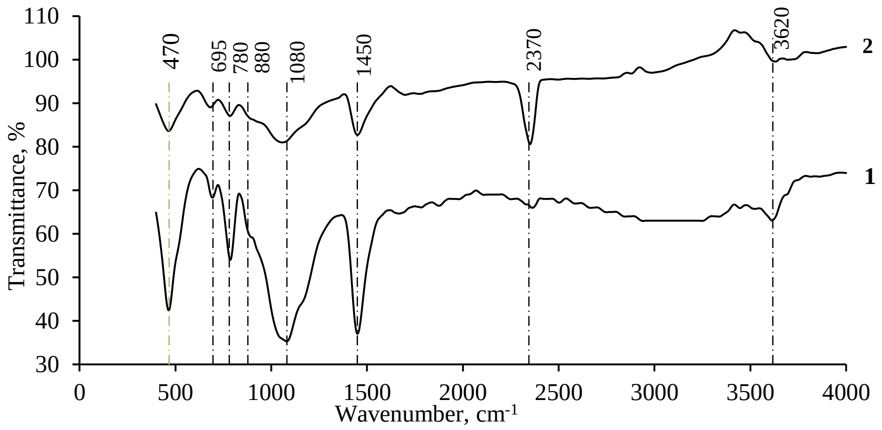
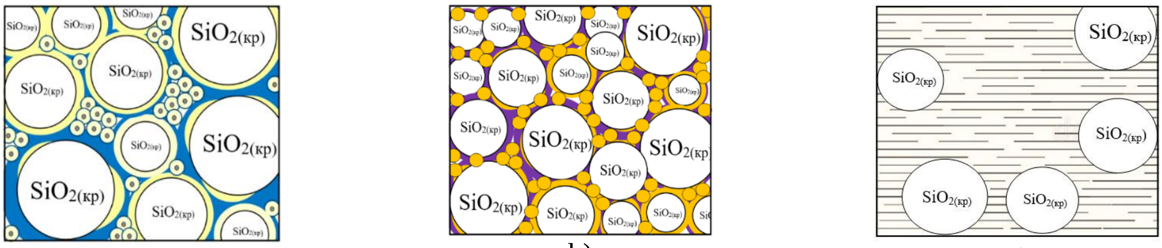


Fig. 2. IR spectra heat-treated at 100 °C (1) and 220 °C (2) samples obtained from a composition with SiO_2/Na_2O 5,7 , 30 wt. % microsilicon, 60 wt.% concentration of NaOH solution
Рис. 2. ИК спектры термообработанных при 100 °C (1) и 220 °C (2) образцов, полученных из композиции с SiO_2/Na_2O 5,7 , 30 мас.% микрокремнезема, 60 мас.% концентрации раствора NaOH



a) b) c)

Fig. 3. Model of phase transformations in the marshalite-silica fume-NaOH-H_2O composition at temperatures, °C:
a – 80 (($Na_2SiO_3(OH)$)·nH_2O water layer); б – 300 ($Na_2SiO_3(OH)$ hydrated SiO_2); c – 800 (eutectic melt residual SiO_2)
Рис. 3. Модель фазовых превращений в композиции маршалит-микрокремнезем-NaOH-H_2O при температурах, °C:
а – 80 (($Na_2SiO_3(OH)$)·nH_2O водный слой); б – 300 ($Na_2SiO_3(OH)$ гидратированный SiO_2); с – 800 (остаточный эвтектический расплав SiO_2)

The reliability of the proposed model of phase transformation is confirmed by the determined dependences, the results of IR spectrometry and X-ray analysis.

CONCLUSION

According to the research results of physico–chemical processes during alkiline activation of the compositions based on marshalite the following dependences were found:

- alkaline acivation of marshalite by means of solution of sodium hydroxide with partial substitution on silica fume leads to the formation of sodium hydrosilicate $Na_2SiO_3(OH)$ on the surface of which the layer of crystallohydrated water is formed at the component mixing stage, as confirmed by the data of thermal gravitational analysis and IR spectroscopy;

- a model of phase transformations that occur while heating in the marshalite–silica fume–NaOH–H_2O system is offered, according to which a frame formation of a porous structure is on the foaming stage (130-200 °C) due to water removal from crystallohydrates of sodium hydrosilicate, their transformation into silicates and pyroplastic mass formation;

- during heating the composition up to 800 °C the stage of silicate formation is completed and pyroplastic mass consisted from an eutectic melt ($Na_2O\cdot 2SiO_2$–SiO_2) and remained silica as unreacted and non-dissolved particles of marshalite is formed;

The composition of the high modulus compound composition (SiO_2/Na_2O 5,7) in the system Na_2O–SiO_2–H_2O was determined suitable to obtain a porous fiber-glass composite from quartzous raw material (marshalite) during one stage, including mass. %: marshalite 50, silica fume 23, sodium hydroxide 16, water 11. From the obtained composite under a temperature of 800 °C the porous fiber-glass composite with a density of 650 kg/m^3 and a compressive strength of 6 mPa was obtained that meets all requirements for heat insulating material (such as foam glass). The determined composition was taken as a basis, with the of the additives for water resistance increase and its density decrease to be introduced.

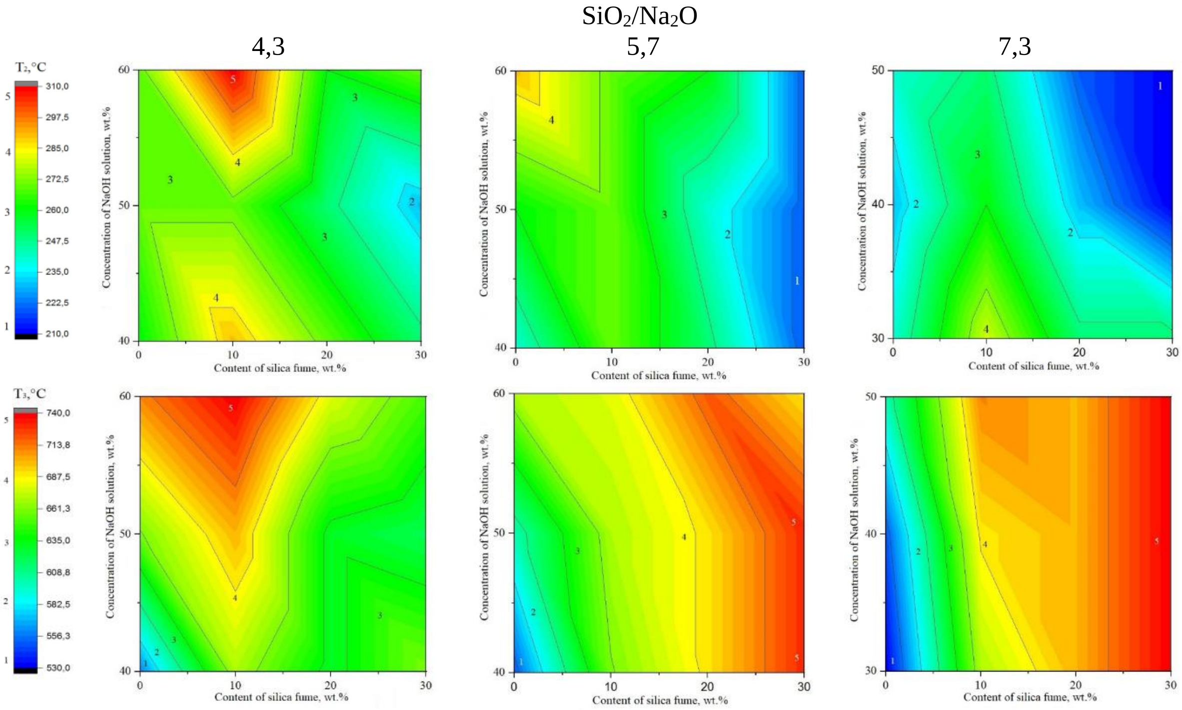


Fig. 4. Response planes of T_2-T_3 values as a function of the silica fume content and NaOH solution concentration when SiO_2/Na_2O 4.3; 5.7; 7.3

Рис. 4. Плоскости отклика значений T_2- T_3, от содержания микрокремнезема и концентрации раствора NaOH, при SiO_2/Na_2O 4,3; 5,7; 7,3

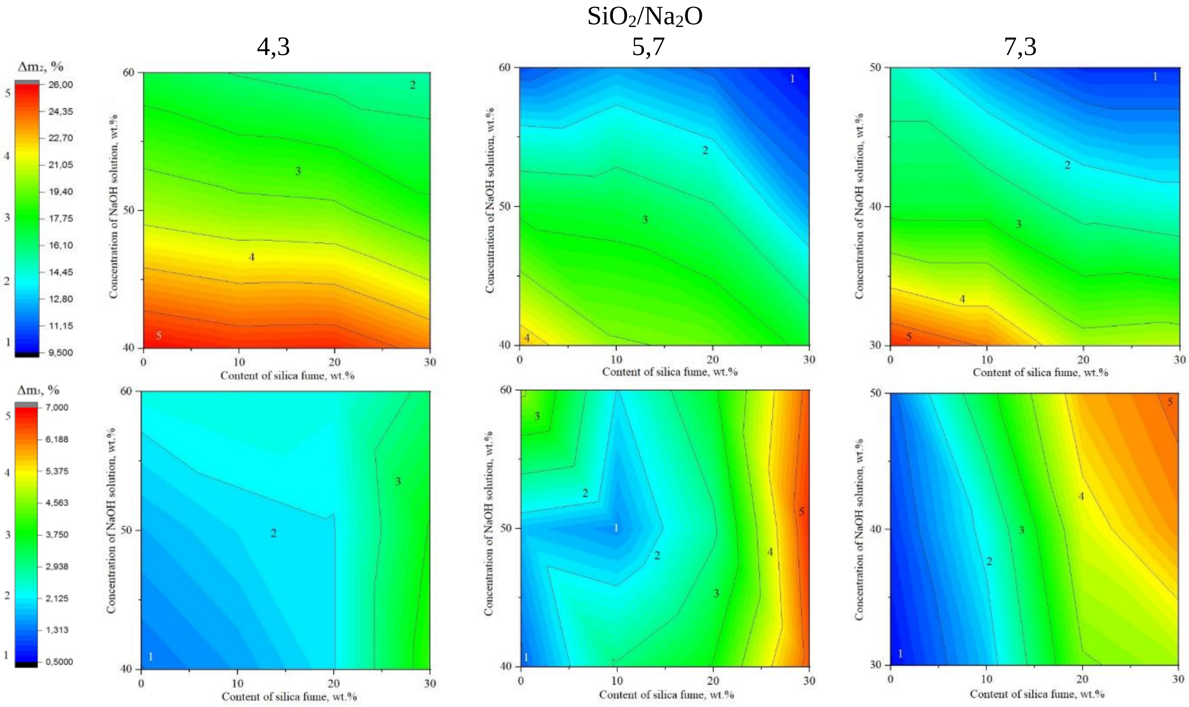


Fig.5. Response planes of Δm_2-Δm_3 values, as a function of the silica fume content and NaOH solution concentration when SiO_2/Na_2O 4.3; 5.7; 7.3

Рис. 5. Плоскости отклика значений Δm_2-Δm_3, от содержания микрокремнезема и концентрации раствора NaOH, при SiO_2/Na_2O 4,3; 5,7; 7,3

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

REFERENCES
ЛИТЕРАТУРА

1. **Goltsman B.M., Yatsenko L.A., Goltsman N.S.** Production of foam glass materials from silicate raw materials by hydrate mechanism. *Solid State Phenom.* 2020. V. 299. P. 293-298. DOI: 10.4028/www.scientific.net/SSP.299.293.
2. **Bajare D., Bumanis G., Korjakins A.** New porous material made from industrial and municipal waste for building application. *Constr. Mater.* 2014. V. 20. N 3. P. 333-338. DOI: 10.5755/j01.ms.20.3.4330.
3. **Simonsen M.E., Sønderby C., Søgaard E.G.** Synthesis and characterization of silicate polymers. *J. Sol-Gel Sci. Technol.* 2009. V. 50. P. 372-382. DOI: 10.1007/s10971-009-1907-4.
4. **Luukkonen T., Heponiemi A., Runtti H., Pesonen J., Yliniemi J., Lassi U.** Application of alkali-activated materials for water and wastewater treatment: a review. *Rev. Environ. Sci. Biotechnol.* 2019. V. 18. P. 271-297. DOI: 10.1007/s11157-019-09494-0.
5. **Manakova N.K., Suvorava O.V., Semushin V.V.** Physico-chemical substantiation of obtaining porous glass materials from silica-containing raw materials. *Glass Phys. Chem.* 2023. V. 49. N 2. P. 193-198. DOI: 10.1134/S108765962260106X.
6. **Kutugin V.A., Lotov V.A., Gubanov A.V., Kursilev K.V.** Porous articles with rigid structure based on natural amorphous silica. *Glass Ceram.* 2018. V. 75. N 1-2. P. 12-16. DOI: 10.1007/s10717-018-0019-4.
7. **Reka A.A., Pavlovski B., Makreski P.** New optimized method for low-temperature hydrothermal production of porous ceramics using diatomaceous earth. *Ceram. Int.* 2017. V. 43. N 15. P. 12572-12578. DOI: 10.1016/j.ceramint.2017.06.132.
8. **Makarov D.V., Manakova N.K., Suvorova O.V.** Production of rock-based foam-glass materials (Review). *Glass Ceram.* 2023. V. 79. P. 411–417. DOI: 10.1007/s10717-023-00522-8.
9. **Miryuk O.A.** Influence of fillers on properties of liquid-glass compositions. *ChemChemTech [Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.].* 2019. V. 62. N 12. P. 51–56. DOI: 10.6060/ivkkt.20196212.5915.

   Мирюк О.А.Влияние наполнителей на свойства жидкостекольных композиций. *Изв. вузов. Химия и хим. технология.* 2019. Т. 62. Вып. 12. С. 51-56. DOI: 10.6060/ivkkt.20196212.5915.
10. **Kazmina O.V., Dushkina M.A., Vereshchagin V.I., Voland S.N.** The use of dispersed screenings of construction sands for the production of foam glass materials. *Stroit. Mater.* 2014. N 1-2. P. 93-97 (in Russian).

    Казьмина О.В., Душкина М.А., Верещагин В.И., Волланд С.Н. Использование дисперсных отсевов строительных песков для получения пеностекольных материалов. *Строит. материалы.* 2014. № 1-2. С. 93-97.
11. **Volland S.** Influence of the mechanical activation of raw mixes on the properties of foam glass from sand sludge. *Constr. Building Mater.* 2016. V. 125. N 30. P. 119-126. DOI: 10.1016/j.conbuildmat.2016.07.116.
12. **Ivanov K.S.** Influence of the methods of preparing a silicate-sodium mixture on the formation of the structure of foam glass ceramics. *Glass Phys Chem.* 2019. V. 45. P. 60-65. DOI: 10.1134/S1087659619010048.
13. **Makarov D.V., Manakova N.K., Suvorova O.V.** Production of rock-based foam-glass materials (review). *Glass Ceram.* 2023. V. 79. P. 411–417. DOI: 10.1007/s10717-023-00522-8.
14. **Yatsenko E.A., Goltsman B.M., Klimova L.V., Yatsenko L.A.** Peculiarities of foam glass synthesis from natural silica-containing raw materials. *J. Therm. Anal. Calorim.* 2020. V. 142. P. 119–127. DOI: 10.1007/s10973-020-10015-3.
15. **Ivanov K.S.** Preparation and properties of foam glass-ceramic from diatomite. *J. Wuhan Univ. Technol.-Mat. Sci. Edit.* 2018. V. 33. P. 273–277. DOI: 10.1007/s11595-018-1817-8.
16. **Da Silva R.C., Kubaski E.T., Tenório-Neto E.T., Lima-Tenório M.K., Tebcherani S.M.** Foam glass using sodium hydroxide as foaming agent: Study on the reaction mechanism in soda-lime glass matrix. *J. Non-Cryst. Solids.* 2019. V. 511. P. 177-182. DOI: 10.1016/j.jnoncrysol.2019.02.003.
17. **Skirdin K.V., Miskovets A. Yu., Kazmina O.V.** Influence of silica fume on the production process and properties of porous glass composite. *ChemChemTech [Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.].* 2023. V. 66. N 1. P. 84-92. DOI 10.6060/ivkkt.20236601.6607.

    Скирдин К.В., Мисковец А.Ю., Казьмина О.В. Влияние кремнезема на процесс производства и свойства пористого стеклокомпозита. *Изв. вузов. Химия и хим. технология.* 2023. Т. 66. Вып. 1. С. 84-92. DOI: 10.6060/ivkkt.20236601.6607.
18. **Kiselev A.V., Lygin V.I.** Infrared spectra of surface compounds. M.: Nauka. 1972. 459 p.

    Киселев А.В., Лыгин В.И. Инфракрасные спектры поверхностных соединений. М.: Наука. 1972. 459 с.
19. **Efimov A.M., Pogareva V.G.** IR absorption spectra of vitreous silica and silicate glasses: The nature of bands in the 1300 to 5000 cm^{-1} region. *Chem. Geol.* 2006. V. 229. N. 1–3. P. 198-217. DOI: 10.1016/j.chemgeo.2006.01.022.
20. **Zhai C., Zhong Y., Zhang J., Wang M., Yu Y., Zhu Y.** Enhancing the foaming effects and mechanical strength of foam glasses sintered at low temperatures. *J. Phys. Chem. Solids.* 2022. V. 165. N 110698. DOI: 10.1016/j.jpcs.2022.110698.

Поступила в редакцию 21.08.2023
Принята к опубликованию 26.12.2023

Received 21.08.2023
Accepted 26.12.2023