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SYNTHESIS AND CHARACTERIZATION OF SILICATE GLASS FROM ALTERNATIVE RAW MATERIALS USING HYDROTHERMAL CHARGE

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Abstract

Aim. To study the possibility of expanding the raw material base of glass production due to the use of high-silica raw materials – diatomite and perlite. Thus, the introduction of the main component of silicate glass SiO_2 occurs due to amorphous silica contained in rocks. **Methods.** The batch was prepared by hydrothermal method. Autoclave processing of rocks and silica was carried out for 4 hours at a temperature of 170 °C and a pressure of 0.5 MPa. NaOH was used as an alkaline agent. The molar ratio of SiO_2 to NaOH was equal to 2. The SiO_2 - Na_2O - PbO system and the glass composition in mole fractions were selected for the studies: SiO_2 – 52.4 %; Na_2O – 26.2 %; PbO – 21.4 %. The composition point lies on the liquidus isotherm of about 800 °C. The glass synthesis was carried out at a temperature of 1100 °C. **Results.** The study of the kinetics of glass making showed that when using hydrothermal making batches based on rocks, the stage of obtaining conditioned glass mass occurs at significantly lower temperatures than in the case of glass making from traditional batches and is at least 100 °C. Thus, it can be argued that the use of hydrothermal batches will reduce energy costs at the glass making stage. DTA results confirmed that the physicochemical activity of batches using amorphous silica is lower than for batches made using crystalline silica. X-ray confirmed that all glass samples welded at a temperature of 1000 °C are characterized by high X-ray amorphousness, which indicates the completion of glass formation processes. A visual assessment of the processes of silicate and glass formation showed that the boiling temperature of model glass based on hydrothermal batches is 110–120 °C lower than for glass made on the basis of traditional batches. **Conclusions.** Thus, it can be argued that the use of alternative raw materials, diatomite and perlite, will allow obtaining silicate glass. And the use of the hydrothermal method for obtaining the charge allows for a reduction in energy consumption during the glassmaking stage.

Keywords: glass; hydrothermal batch; diatomite; perlite; thermal analysis; X-ray.

СИНТЕЗ ТА ХАРАКТЕРИСТИКА СИЛІКАТНОГО СКЛА З АЛЬТЕРНАТИВНОЇ СИРОВИНИ З ВИКОРИСТАННЯМ ГІДРОТЕРМАЛЬНОЇ ШИХТИ

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Анотація

Мета. Вивчити можливість розширення сировинної бази виробництва силікатного скла за рахунок використання висококремнеземистих мінералів – діатоміту та перліту, що дозволяє вводити основний компонент силікатного скла SiO_2 за рахунок аморфного кремнезему. **Методи.** Шихту готували гідротермальним способом. Автоклавної обробку гірських порід і кремнезему проводили протягом 4 годин при температурі 170 °C і тиску 0.5 МПа. Як лужний агент використовували NaOH. Молярне відношення SiO_2 до NaOH складало 2. Для досліджень було обрано систему SiO_2 - Na_2O - PbO , із складом скла: SiO_2 – 52.4 %; Na_2O – 26.2 %; PbO – 21.4 % (мольних часток). Точка складу лежить на ізотермі ліквідусу з температурою плавлення близько 800 °C. Максимальна температура нагрівання шихт складала 1100 °C. **Результати.** Дослідження кінетики скловаріння показало, що при використанні гідротермальних шихт на основі гірських порід стадія отримання кондиційної скломаси відбувається при значно нижчих температурах, ніж при використанні традиційних шихт, і становить не менше 100 °C, що дозволяє зменшити витрати енергії на стадії виготовлення скла. Результати ДТА підтвердили, що фізико-хімічна активність скляних шихт з використанням аморфного кремнезему нижча, ніж для шихт, виготовлених із використанням кристалічного кремнезему. Дані РФА підтвердили, що всі зразки скла, синтезовані за температури 1000 °C характеризуються високою рентгеноаморфністю, що свідчить про завершення процесів склоутворення. Візуальна оцінка процесів силікато- і склоутворення показала, що температура синтезу модельного скла на основі гідротермальних шихт на 110–120 °C нижча, ніж у скла, виготовленого на основі традиційних шихт. **Висновки.** Підтверджено можливість використання висококремнеземистих порід – діатоміту та перліту для отримання конденційного силікатного скла, а підготовка шихти гідротермальним методом дає можливість знизити енерговитрати на етапі скловаріння.

Ключові слова: скло; гідротермальна шихта; діатоміт; перліт; термічний аналіз; РФА.

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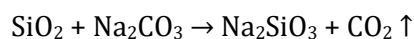
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Introduction

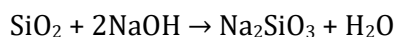
Stocks of high-quality quartz sand, necessary for the synthesis of silicate glass, are quite limited. Therefore, there is a need to find alternative raw materials for introducing SiO₂ into the glass charge. Such materials can be low-grade sand or high-silica rocks [1–4].

Silica (SiO₂) is widely distributed in nature and is presented in crystalline and amorphous forms. The most promising for use in the glass industry are amorphous, highly dispersed forms of silica – diatomite, opoka, trepel, perlite, etc. [1–3]. Due to their unique physical and physicochemical properties, these rocks can be used as raw materials for the production of liquid glass, as well as for the preparation of glass charge by the hydrothermal method [5]. Such charges have a number of advantages: high uniformity, increased reactivity and low-temperature melting [1–4].

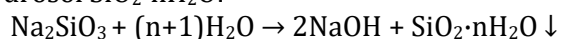
It is possible to compare the methods of obtaining a vitreous phase using traditional and hydrothermal charges. The traditional method of obtaining molten sodium silicate (Na₂SiO₃) occurs during the melting of a mixture of sodium carbonate (Na₂CO₃) and quartz sand (SiO₂) at temperatures >1100 °C [6; 7], based on the reaction:



This method requires high energy costs and complex maintenance. Also, during the cooking of glass, a large amount of carbon(IV) oxide (CO₂) is released, which is a greenhouse gas and pollutes the environment [8–10]. An alternative method is the hydrothermal method, which can be used to produce Na₂SiO₃ by reacting SiO₂ with NaOH. Such a reaction can take place in an autoclave at temperatures up to 250 °C at low pressures [3; 9; 11]. The interaction of reagents takes place according to the reaction:



The liquid phase can be obtained with the help of hydrothermal synthesis at approximately 450–650 °C, a lower temperature than using traditional methods, [12; 13]. As a result of hydrothermal synthesis, sodium silicate is obtained, formed by the anion of a very weak silicate acid, which hydrolyzes in cold water, giving the solution a strongly alkaline reaction. During the process, more complex ions Si₂O₅²⁻, H₂SiO₄²⁻, H₄SiO₇²⁻ [14] are formed. When interacting with hot water, a colloid precipitate falls out of the solution – hydrosol SiO₂·nH₂O:



The availability of the hydrothermal method makes it promising for use in the glass industry [1; 2]. During the hydrothermal synthesis of sodium silicate from inorganic materials, for example, when amorphous silica is used, Na₂CO₃ or NaOH is used as an alkaline agent [8; 15]. However, the use of Na₂CO₃ in the reaction allows extracting up to 70 % of SiO₂ from the rock, and the extraction time of the component can reach up to 48 hours.

Sodium silicate can also be prepared from the activated material by direct action of NaOH. This method of dissolving SiO₂ can be carried out at a temperature of 25–100 °C and atmospheric pressure in a reactor tank equipped with a reflux system [13].

At the same time, hydrothermal treatment of rocks containing SiO₂ in crystalline form requires higher temperatures and pressures than for minerals containing silica in amorphous form [3; 11]. The rate of silica dissolution from rock depends on the crystallinity of the material. Thus, amorphous silica allows its extraction at significantly lower temperatures compared to crystalline silica. Therefore, this method finds wider application due to its advantages in high reactivity. To calculate the required amount of NaOH, the amount of crystalline and amorphous phases of silica contained in the rock should be taken into account [13; 16].

Another factor that largely determines the material conversion factor is the ratio of raw materials. Thus, to dissolve 1 mass fraction of SiO₂, 1.33 and 1.76 mass fractions of NaOH and Na₂CO₃ are required, respectively. Application of the coal ash removal reaction using 10 M NaOH (mass ratio 1 : 4) for 1 hour at a temperature of 95 °C showed extraction of up to 60% of silica [17]. In [18], a ratio of 0.72–2.24 mass of NaOH to silicon waste was used. The process took place in an adiabatic reactor at a temperature of 300 °C, which made it possible to extract >98.5 % of silica. In [4], the mass ratio of NaOH to quartz sand was approximately 1.5–2.5. The reagents were reacted in an open reaction system at a temperature of 150 °C for 45 minutes. As a result of the reaction, a pure silica precipitate was obtained. The extraction was about 80 %. Also, a study was conducted where the ratio of NaOH to quartz sand was 1.1. Heat treatment was carried out at a temperature of 550 °C. Extraction with minimal consumption of NaOH [10]. Owioye et al. [20] in their study synthesized nanosilica by the interaction of NaOH and waste glass containers with a mass ratio of reagents equal to 2. In work [2], the extraction of SiO₂ from trepel using Na₂CO₃

was 74 % at a ratio in the reaction mixture of $T : P = 2.5 : 1$ and soda content in terms of Na_2O in the range of 20–30 %. The obtained solutions of silicates can be used for the preparation of a complex glass charge by the co-precipitation method, which will ensure uniformity and a significant decrease in the melting temperature of glass [2].

The goal of our research was to study the possibility of glass synthesis at a temperature not exceeding 1100 °C, based on amorphous silica-containing rocks – diatomite and perlite. And also to compare the cooking properties, structures and thermal properties of glasses obtained from hydrothermal and traditional charges.

The object of experimental research was the phase transformations and kinetics of glass boiling on the basis of mountain amorphous high-siliceous rocks – diatomite and perlite.

Taking into account that the processes of making glass from hydrothermal charge based on rocks are not sufficiently studied, an urgent task is to study the physicochemical transformations that occur during heating of hydrothermal and traditional charges with the use of alternative raw materials and the use of hydrothermal and traditional charges. Such studies will become the

basis for studying the possibility of cooking glasses based on a hydrothermal charge using silica contained in industrial waste, such as coal fly ash [17], glass waste [15; 17; 19], iron ore enrichment tailings [20], ashes of incineration furnaces [21] and ferronickel slag [22].

Materials and methods

Material characterization

Approbation of this method was performed on model low-melting glasses. This necessity is due to the desire to implement the idea of visual analysis of the kinetics of glass melting with the help of photo and video shooting in real time. At the actual boiling temperatures of industrial silicate glasses, this would be technically more difficult to achieve. The resulting photos and videos clearly illustrate the dynamic processes of sintering, foaming, bubbling, etc.

The SiO_2 – Na_2O – PbO system and the glass composition in mole fractions were selected for the studies: SiO_2 – 52.4 %; Na_2O – 26.2 %; PbO – 21.4 %. The composition point lies on the liquidus isotherm of about 800 °C and is marked on the state diagram of the SiO_2 – Na_2O – PbO system. (Fig. 1).

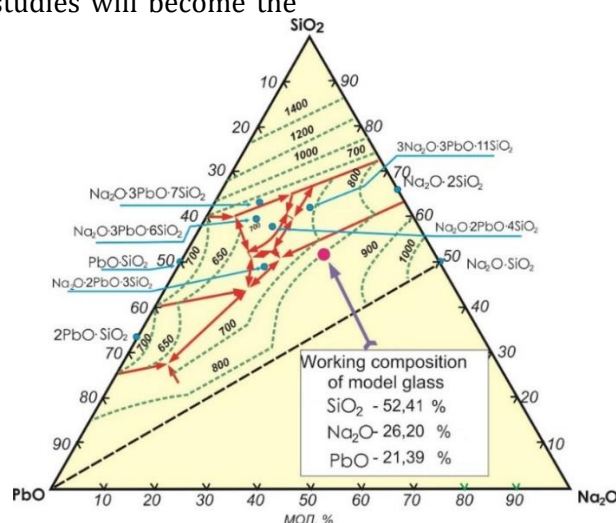


Fig. 1. State diagram of the SiO_2 – Na_2O – PbO system

When converted to mass %, it is possible to obtain the following glass composition: SiO_2 – 32.97 %; Na_2O – 17.03 %; PbO – 50 %.

The glass synthesis was carried out in fireclay crucibles in a silite laboratory furnace at a temperature of 1100 °C.

To introduce SiO_2 into the hydrothermal charge, chemically pure amorphous silica, high-silica rocks – diatomite and perlite were used.

Diatomite from the Velykyi Burluk deposit, Kharkiv region, was used in the work. Diatomite is a highly siliceous amorphous rock containing

about 88 % silica, as well as clay minerals, quartz and glauconite.

For research, perlite from the Fogosh deposit, Transcarpathian region, was also used. Perlite is an acidic aluminosilicate rock of volcanic origin with a SiO_2 content of up to 75 %. It contains impurities of quartz, feldspar, etc. The amount of structural water in the rock is about 4.5 %.

The chemical composition of DE (Table 1) was determined using the classical chemical analysis (for silicates).

Table 1

Instead of component, %	Composition of minerals										loss of ignition, %
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	SO ₂	P ₂ O ₅	
Diatomite	86.03	3.01	2.89	0.20	0.76	0.28	0.75	0.34	-	0.15	5.66
Perlite	74.35	12.94	1.36	0.15	1.17	0.08	3.26	1.36	0.08	-	5.25

Processing of the silica-containing component (amorphous silica, diatomite, perlite) was carried out using NaOH in an autoclave at a temperature of 170 °C for 4 hours, the pressure was 0.5 MPa. The molar ratio of SiO₂ to NaOH was 1:2. As a result, a gel-like homogeneous solution of silicate was obtained. After evaporation and drying, charging with lead(II) oxide was carried out.

Chemically pure amorphous silica and quartz sand from the Novoselivka deposit, Kharkiv region with an SiO₂ content of 99.53 % were used to introduce SiO₂ into the traditional charge.

Chemically pure reagents sodium hydroxide, silicon(IV) oxide and lead(II) oxide manufactured by Merck were used in the work.

To obtain the charge, the components were crushed, sieved and the fraction ≤ 0.5 mm was selected.

Sample preparation

We prepared three mixtures for the preparation of hydrothermal charge using:

sample 1 – chemically pure amorphous silica;

sample 2 – diatomite;

sample 3 – perlite.

To compare the kinetics of glass boiling from hydrothermal preparation charges and from conventional charges, two more traditional charges were prepared using:

sample 4 – chemically pure amorphous silica;

sample 5 – quartz sand.

A Granum Touch laboratory autoclave (China) was used to obtain the hydrothermal charge. A tubular laboratory furnace was used for cooking glasses (Fig. 2).

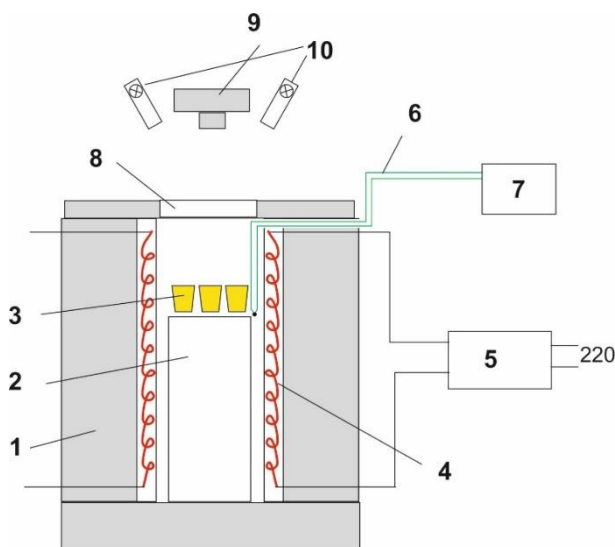


Fig. 2. Unit for boiling glass and visual observation of the process:

1 – tubular furnace; 2 – stand; 3 – crucibles with charge; 4 – heaters; 5 – command electrical device; 6 – thermocouple; 7 – potentiometer; 8 – viewing window made of quartz glass; 9 – photo and video camera; 10 – illuminators

Five crucibles with charges were placed in the cavity of the tubular furnace on a stand. From above, the cavity is closed with a lid with an observation window made of optically transparent quartz glass. On top is a photo and video camera, as well as two illuminators. The oven is heated at a constant speed with the help of a command device. The temperature was measured with a chromel-alumel thermocouple.

Photography was carried out only for certain temperatures, at which it was possible to ascertain certain or other regular changes in the state of the charge or glass mass

The boiling capacity of glass, the aggressiveness of the glass mass in relation to fireclay, the assessment of the surface relief and the assessment of the decorative properties of the obtained materials were performed visually

according to the method described in the article with some changes [23].

Instrumental methods of analysis

The phase composition of vitreous materials was studied by X-ray phase analysis (XRF), which was carried out on a DRON 3M (USSR) diffractometer. X-ray tube: CuK α . U = 30kV. Phases were identified using the ICDD card file. Glass samples were examined after 1-hour exposure at a temperature of 900 °C.

Thermal studies of the samples were carried out on the Derivatograph Q-1500 device (Hungary) of the Paulik-Paulik-Erdey system. The samples were analyzed in dynamic mode with a heating rate of 10 °C/min to 1000 °C in an air atmosphere. The weight of the samples was 1500 mg. Al₂O₃ was taken as the reference substance. A platinum crucible was used in the experiments. Sensitivity according to the DTA scale is 250 μ V.

Visual evaluation of the glassmaking process

Visual assessment of glass synthesis, namely: charge sintering beginning, liquid phase appearance, foam appearance, active gas formation in the melt, gas formation in the melt without foam, illumination process (single bubbles) and full lighting (conditioned glazing), were carried out according to the methods described in the works [24; 25].

Results and discussion

Differential thermal analysis

Physico-chemical processes occurring during the heating of charges were studied with the help of DTA (Fig. 3). As expected, the greatest activity of physicochemical transformations is observed for the traditional charge (sample 5) using quartz sand. On the DTA curve, it is possible to observe two endoeffects with maxima at 130 °C, which corresponds to the removal of physically bound water, and at 840 °C, which corresponds to the processes of melting of the charge and formation of the liquid phase. For sample 4, the corresponding endo-effects are present, but they are less pronounced and shifted towards lower temperatures. For samples of hydrothermal charge (samples 1–3), it is possible to observe extensive endo-effects in the temperature range of 100–500 °C, which is explained by the release of chemically bound water of siloxane, silanol, and silanediol groups. At the same time, they are minimal for a hydrothermal charge based on diatomite, the endow effect is negligible. In the case of using pearlite in the temperature range of 600–1000 °C, there are several thermal effects due to the more complex mineralogical composition of pearlite.

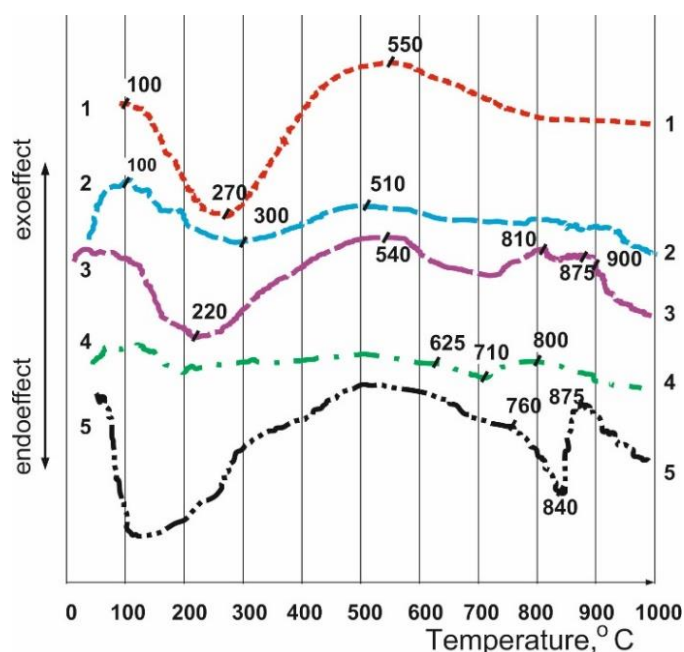


Fig. 3. Thermograms of the studied samples:

1 – hydrothermal charge based on amorphous silica; 2 – hydrothermal charge based on diatomite; 3 – hydrothermal charge based on pearlite; 4 – traditional charge based on amorphous silica; 5 – traditional charge based on quartz sand

Analyzing the DTA results, the following can be stated: the physico-chemical activity of charges using amorphous silica is lower than that of

charges with crystalline silica (quartz sand). Hydrosilicates of sodium and lead will be formed to a greater extent when using amorphous silica

and perlite, to a lesser extent – when using diatomite. This is confirmed by the smaller value of the endoeffect. Therefore, the use of diatomite in the preparation of hydrothermal charge will be more desirable. Since it will require less energy consumption to remove adsorption and gel water from the semi-finished product.

X-ray phase analysis

X-ray phase analysis shows that all samples are characterized by high X-ray amorphousness. It is

known that the presence or absence of reflexes on diffractograms indicates one or another completeness of glass formation processes. The ideal glassy state is at the same time an amorphous state. It is characterized by the absence of peaks and the presence of only blurred halos. The corresponding diffraction patterns are shown in Fig. 4.

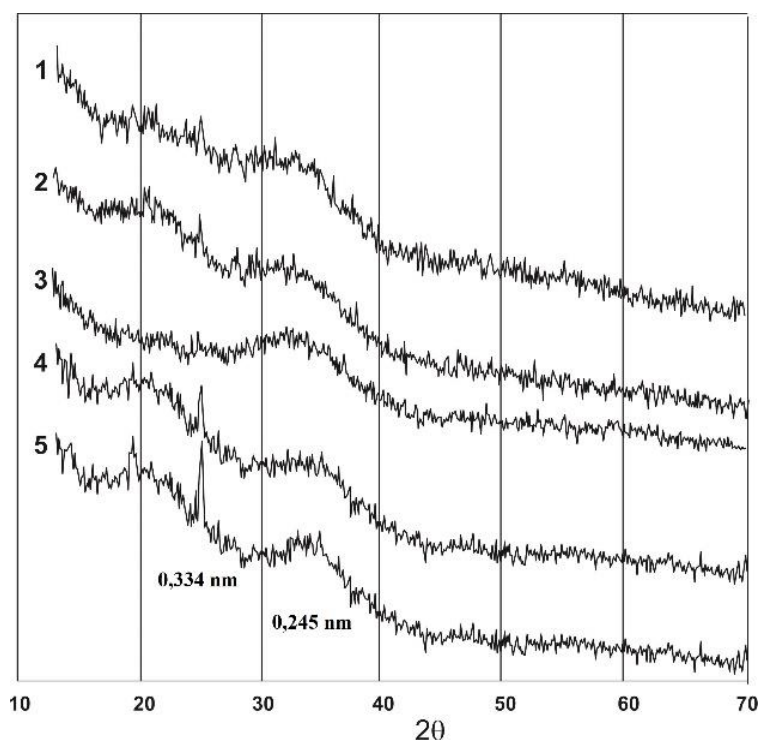


Fig. 4. Diffractograms of model glasses:

1 – hydrothermal charge based on amorphous silica; 2 – hydrothermal charge based on diatomite; 3 – hydrothermal charge based on perlite; 4 – traditional charge based on amorphous silica; 5 – traditional charge based on quartz sand

The XRD results confirm that all glass samples are X-ray amorphous. If to compare the diffractograms of glass samples welded from hydrothermal charges, it should be noted that the most amorphous state is observed in glass welded from hydrothermal charges based on amorphous silica (sample 3). For samples 1 and 2, it is possible to observe the presence of insignificant peaks. Thus, it can be stated that the insignificant presence of the crystalline phase of silica is observed for samples made from hydrothermal charges based on diatomite and perlite. For samples 4 and 5, it is possible to observe insignificant reflexes characteristic of quartz at 0.334 and 0.245 nm [23; 26].

Visual evaluation of the glassmaking process

We conducted an experiment that consisted in recording the image of the processes that take

place in the tubular furnace. It is recorded in real time how the stages of silicate and glass formation take place, starting from the initial charge and ending with the welded conditioned glass mass. This set-up of the experiment is logically justified, since modern glass-making furnaces are equipped with television cameras, the image from which is sent to control points and this provides information about the kinetics of glass-making processes. In the practice of glass melting in bath furnaces, this is the only objective and irreplaceable information that allows to quickly manage the glass melting process. In our work, pictures are presented only at those temperatures at which it was possible to ascertain one or another successive changes in the state of the charge or glass mass (Fig. 5).

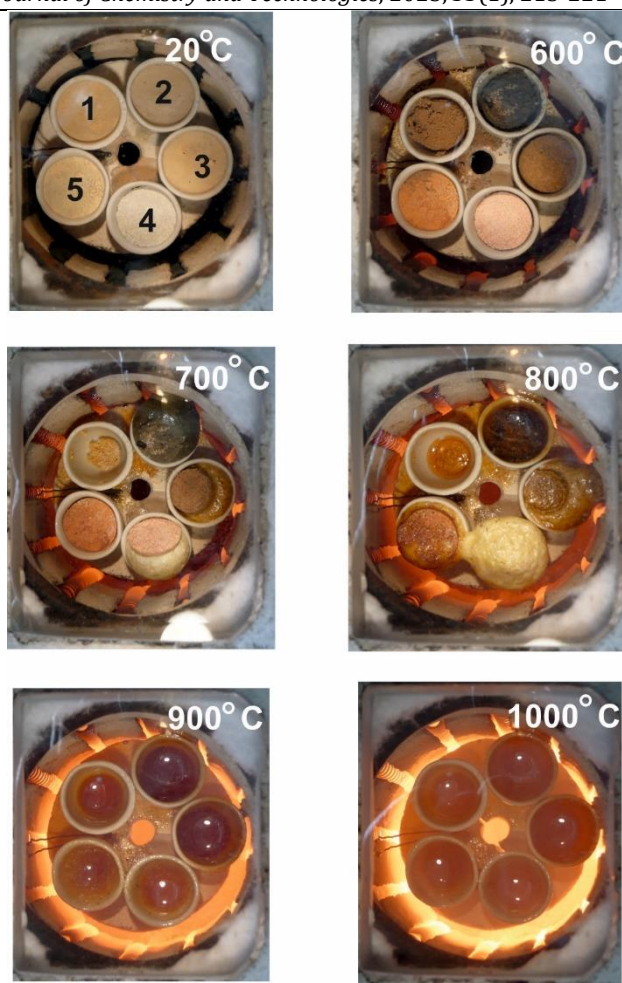


Fig. 5. Condition of the charge and glass mass:

1 – hydrothermal charge based on amorphous silica; 2 – hydrothermal charge based on diatomite; 3 – hydrothermal charge based on perlite; 4 – traditional charge based on amorphous silica; 5 – traditional charge based on quartz sand

The results of the observations are summarized in the Table 2.

Table 2

Glassmaking kinetics of model glasses					
Characteristic stages of glassmaking	Charge type				
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	Temperature, °C				
Charge sintering beginning	675	500	520	520	600
Liquid phase appearance	750	600	600	675	775
Foam appearance	760	700	720	720	785
Active gas formation in the melt	780	750	775	785	825
Gas formation in the melt without foam	825	800	850	870	920
Lightening process (single bubbles)	870	860	870	920	950
Full lightening (conditioned glass mass)	885	875	885	950	1000

For charges of hydrothermal preparation (samples 1–3), the dynamics of the onset and alternation of the characteristic stages of silicate and glass formation are different, but the final temperature of obtaining the conditioned melt is approximately the same and does not exceed 885 °C. In the batch of hydrothermal preparation based on amorphous silica (sample 1), the

temperatures for which the mentioned processes are observed are significantly shifted to the high-temperature region. However, the start of sintering of such a charge begins at a temperature of 675 °C, and for charges of hydrothermal preparation based on diatomite and perlite, these processes begin at temperatures of 500 and 520 °C, respectively. However, the final stage of

glass-making takes place at approximately the same temperatures as in the case of sample 1. Comparing the charges based on diatomite and perlite (samples 2 and 3), it is possible to say that the temperatures of all stages of glass-making in the case of diatomite charge are slightly lower than those of perlite.

The study of the kinetics of glass making showed that the processes of glass full lightening, which are the final stage of glass melting, for hydrothermal batches actively occur at a temperature of 860–885 °C and end approximately 137 minutes after the start of glass melting. The making processes of the ordinary charge based on amorphous silica (sample 4) and on the basis of crystalline silica take place at higher temperatures of 950 and 1000 °C and last about 155 and 170 minutes, respectively. The analysis of the kinetics of glass boiling allows to state that the boiling temperature of model glass based on hydrothermal preparation charges is 110–120 °C lower than for traditional charges based on quartz sand.

Conclusions

The study of hydrothermal charges based on amorphous diatomite and perlite rocks confirmed the possibility of their use for practical vitrification of silicate glasses.

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