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HYDROTHERMAL TREATMENT OF THE CHARGE USING HIGH-SILICA RAW MATERIALS IN GLASS PRODUCTION

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The paper substantiates the possibility of expanding the raw material base of glass production through the use of high-silica raw materials, specifically tripoli. The essence of the study lies in the fact that the main glass-forming component, SiO_2, is introduced at the expense of amorphous silica contained in tripoli. The charge was prepared using a hydrothermal method. Autoclave treatment of the fired mixture was carried out at 150^{0}C for 10 hours under a pressure of 0.5 MPa in the presence of NaOH. The content of sodium hydroxide in the mixture for hydrothermal treatment was 25% calculated in terms of Na_2O. Thermal analysis of the ordinary charge showed that dehydration reactions last up to 200^{0}C, whereas for the hydrothermal charge dehydration occurs up to 500^{0}C. This is explained by the release of chemically bound water from siloxane, silanol, and silandiol groups. For the ordinary charge, the next endothermic effect begins at 500^{0}C and reaches its maximum at 540^{0}C, which corresponds to the dehydration of kaolin and is 40^{0}C higher compared to the similar effect in the hydrothermal charge. In the ordinary charge, the following exothermic effects are observed at 610^{0}C, 650^{0}C, 830^{0}C, and 940^{0}C, which are associated with silicate formation. In contrast, for the hydrothermal charge, only two corresponding exothermic effects are recorded at 580^{0}C and 880^{0}C. Other exothermic effects occur at lower temperatures and overlap with the pronounced endothermic effect of water release from siloxane groups. XRD data confirm that the hydrothermal charge becomes almost completely amorphous at 1000^{0}C, and at 1200^{0}C all diffraction peaks disappear, indicating a fully glassy state. In the ordinary charge, even at 1200^{0}C, peaks corresponding to crystalline silica remain. Visual observation of the stages of glass formation revealed significant differences in the temperature intervals of silicate and glass formation between the hydrothermal and ordinary charges. The use of a hydrothermal charge allows the glass melting temperature to be reduced by 200–250^{0}C.

Keywords: hydrothermal charge, tripoli, glass, thermal analysis, XRD.

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Introduction

Given that the reserves of high-quality quartz sands required for glassmaking are limited, there is a need to find alternative raw materials for introducing SiO_2 into silicate glass. Such materials can be low-grade sands or high-silica rocks [1–4]. Researchers also pay much attention to unconventional methods of glass charge preparation [4–6]. These include the precipitation method, hydrothermal charge production, and chemically activated charge production [7]. The charge produced in this way has a number of advantages: high homogeneity, increased reactivity and low-temperature melting [1,8]. They are produced using alkali-silicate solutions or liquid glass [5,6]. At

present, natural quartz sand containing 98–99% SiO_2 and a minimum amount of impurities is mainly used as a raw material for the production of alkaline-silicate solution by the hydrothermal method [9]. The use of high-silica rocks for the introduction of SiO_2 into glass has been little studied.

Ukraine has large deposits of high-silica rocks [10]. Leaching of SiO_2 from the tripoli by the hydrothermal alkaline processing yields alkali metal silicates. Further, these solutions can be used to produce a complex glass charge by co-precipitation, which will ensure homogeneity and a significant reduction in the glass melting temperature [4].

The purpose of this paper was to study the processes of leaching SiO_2 from the tripoli and fabricate the charge by the hydrothermal method. In addition, we compared the samples prepared from the hydrothermal charge in terms of heat treatment, structure and thermal properties with those prepared from the traditional charge.

The object of experimental research was the phase transformations and kinetics of glasses based on the amorphous high-silica rock, the tripoli.

Given that the processes that occur during the hydrothermal method of rock-based charge preparation are poorly understood, it is important to study the physicochemical transformations that occur during the heating of hydrothermal and conventional charges.

Experimental

The charge for glass production was prepared using two methods: hydrothermal and conventional. The composition of the charge was selected so that the welded glass corresponded to conventional container glass in terms of its composition [7]. The chemical composition of the resulting glasses corresponds to the average composition of container glass (Table 1).

Kaolin, chalk and dolomite are used for both charges. The sodium-silicate component of the traditional charge is provided by sand and soda, while the hydrothermal charge is provided by tripoli and sodium hydroxide. The chemicals used for glassmaking are sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3), as well as natural raw materials: tripoli from the Nemiia deposit (Vinnytsia region), sand from Novoselivka deposit (Kharkiv region), chalk from Zdolbuniv deposit (Rivne region), dolomite from Zavadivka deposit (Ternopil region), and kaolin from Hlukhivske deposit (Vinnytsia region).

Table 1

Chemical composition of container glass

Oxide	SiO_2	Na_2O	CaO	MgO	Al_2O_3
Oxide content in glass, %	71.5	15.3	8.3	1.9	3.0

To calculate the chemical composition of the blends, we took into account the composition of raw materials (Table 2).

The conventional charge was prepared by grinding and sieving the raw materials. The fraction with a size of no more than 0.5 mm was used in the experiments.

To prepare the hydrothermal charge, the above two preparatory operations are supplemented by hydrothermal autoclave treatment of raw materials. After mixing, the hydrothermal charge was processed in a laboratory autoclave. The charge was mixed with water in a 1:1 ratio. The pressure was 0.5 MPa, the processing temperature was 150^{0}C, and the processing time was 10 hours. The content of sodium hydroxide in the mixture for hydrothermal treatment was 25% in terms of Na_2O.

The resulting product, colloidal alkaline solutions and suspensions of unreacted particles, was subjected to drying in an oven at 120^{0}C. However, even without hygroscopic moisture, the charge still contains a large amount of chemically bound water [6]. Laboratory experiments have confirmed that the resulting hydrothermal charge based on tripoli is capable of swelling when heated up to 500^{0}C. The dehydration and decarbonization of the hydrothermal charge were also confirmed by the results of thermal analysis. Therefore, the pre-dried hydrothermal charge was subjected to additional thermal treatment: firing in a muffle furnace at a temperature of 800^{0}C. The charge treated in this way was solid with a glassy phase content due to the melting of low-temperature eutectics.

Thermal studies of the charge samples were carried out using Derivatograph Q-1500 (Hungary) of the Paulik-Paulik-Erdey system. The samples were analyzed in a dynamic mode at a heating rate of 10^{0}C/min to 1000^{0}C in an air atmosphere. The weight of the samples was 1500 mg. Al_2O_3 was taken as a reference substance. A platinum crucible was used in the experiments. The sensitivity was 50 mg and 250 μV on the TH scale and on the DTA scale, respectively. For the thermogravimetric analysis, a hydrothermal charge was used, which had undergone preliminary dehydration at a temperature of 120^{0}C. The conventional charge was used without drying.

The X-ray phase analysis (XRD) of the obtained samples was performed on a DRON 3M diffractometer (CuK_α radiation, U=30 kV). The identification of phases was carried out using the ICDD card index. The samples were taken from a conventional and a hydrothermal charge at 20^{0}C, as well as charges heated

Table 2

Chemical composition of charge components

Charge components	Chemical composition of glass, %						Mass loss on ignition, %
	SiO_2	Na_2O	CaO	MgO	Al_2O_3	Fe_2O_3	
tripoli	82	0.2	5.7	0.5	3	2	6.6
sand	97.7	0.2	0.56	0.12	0.56	0.2	0.66
soda	–	58.5	–	–	–	–	41.5
sodium hydroxide	–	77.5	–	–	–	–	22.5
chalk	0.95	–	54.65	0.07	0.5	0.1	43.73
dolomite	2.1	–	31.9	20.5	0.5	0.35	44.65
kaolin	46.5	–	0.8	–	39.1	0.4	13.2

to 600, 800, 1000, and 1200^{0}C and kept at each temperature for 1 hour.

The morphology of the prepared materials was analyzed visually and by microphotography.

Results and discussion

Both chemical reagents and natural raw materials were used to prepare the charge. Taking into account the composition of the raw materials, a formulation of hydrothermal blends was developed to produce glass of a given composition (Table 3).

Given that the raw materials contained some components that were not part of the glass matrix, the calculated composition differs slightly from the specified one. However, this deviation is insignificant (Table 4) and cannot affect the final result.

The formulation for the conventional charge was calculated taking into account all raw materials (Table 5).

Since the raw materials contain some components that were not part of the glass matrix, the calculated composition slightly differs from the given one. However, this deviation is insignificant and cannot significantly affect the final result (Table 6).

Table 3

Formulation for hydrothermal charge

Charge components	Component content, %
tripoli	74.514
sodium hydroxide	16.821
chalk	1.707
dolomite	6.164
kaolin	0.794
total	100.000

Figure 1 shows the curves of relative mass loss (TGA) of hydrothermal and conventional charges when heated to 1000^{0}C. The conventional charge undergoes a significant mass loss when heated to 200^{0}C. This is due to the fact that it was not subjected to preliminary drying, and this moisture loss is explained by the release of adsorption moisture. In this case, 4–5% is lost, which corresponds to the natural moisture content of the air-dry charge. With an increase in temperature above 200^{0}C, the TG curve stabilizes somewhat up to 500^{0}C, but there is a slight constant mass loss. By 500^{0}C, the total mass loss is about 0.5%.

When heated above 500^{0}C, the ordinary charge again loses significant weight, the rate of which increases to 600^{0}C. In this temperature range, the charge loses more than 2%. This is primarily due to dehydration of kaolinite:

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O = Al_2O_3 \cdot 2SiO_2 + 2H_2O$$

After the mass loss rate decreases to 700^{0}C, it increases rapidly again and reaches a maximum at 900^{0}C.

The maximum losses in this temperature range (900^{0}C) correspond to the following reaction:

$$CaCO_3 + SiO_2 = CaSiO_3 + CO_2$$

Table 4

Comparison of the specified and calculated composition of glass for hydrothermal charge

Component of glass	SiO_2	Na_2O	CaO	MgO	Al_2O_3	Fe_2O_3
Given, %	71.50	15.30	8.30	1.90	3.00	–
By calculation, %	70.26	15.04	8.16	1.87	2.95	1.73
Imbalance, %	–1.24	–0.26	–0.14	–0.03	–0.05	1.73

When the temperature reaches 1000°C, all dehydration and decarbonization reactions are completed [9].

The dynamics of mass loss with a hydrothermal charge is significantly different from the mass loss with a traditional charge. Thus, the initial mass loss (≈7%) at a temperature of 200°C is significantly higher than in the previous case. This can be interpreted as the result of silica dehydration due to desorption of physically (25–150°C) and chemically bound water (150–300°C). This stage persists up to significantly higher temperatures (up to 500°C). This is undoubtedly due to the presence of siloxane, silanol, and silandiol groups in the hydrothermal charge of chemically bound water [10]. Literature data from spectroscopic analyses and studies of the chemical properties of silica at different temperatures describe its general formula, which takes into account the presence of these groups and describes the relationship between them [50]: $(SiO_2)_{1-5.5x}(SiO_{5/2}H)_{4.5x}$ $(SiO_3H_2)_x$. This formula describes the chemical composition of dehydrated silica up to 800°C [11]. In this temperature range, the formation of silicates with the release of carbon dioxide shall also be present. Thus, in this temperature range, we obtain the superposition of exo- and endo-effects of carbonate decomposition and dehydration of the charge components. Subsequently, the rate of mass loss of the sample gradually decreases and practically stops at a temperature of 900°C. The decrease in the rate of mass loss at the final stage of heat treatment and its practical cessation indicates that the main processes of silicate formation in the hydrothermal charge occurred at significantly lower temperatures. This confirms the higher reactivity of the hydrothermal charge compared to the ordinary one [10].

Figure 2 shows the results of differential thermal analysis (DTA) of the samples. In both cases, pronounced end-effects are observed, the maximum of which corresponds to a temperature of 120°C. For the ordinary charge, this is the evaporation of adsorption water, for the hydrothermal charge, it is the evaporation of adsorption water, to which is added an intense end-effect of water release from siloxane groups [11].

For the ordinary charge, the following thermal effect (end effect) begins at a temperature of 500°C and reaches a maximum at 540°C; it is caused by the dehydration of kaolin. A similar effect is observed for the hydrothermal charge sample, which begins at 460°C and reaches a maximum at 500°C. That is, it is shifted by 40°C to the low-temperature region compared to the ordinary charge. For the ordinary charge, the following thermal effects (exo-effects) are observed at temperatures: 610°C, 650°C, 830°C, and 940°C. They are caused by the formation of various silicates. For the hydrothermal charge sample, we observe only two corresponding exo-effects at temperatures of 580°C and 880°C. This is due to the fact that the other exo-effects occurred at lower temperatures. They are superimposed on the pronounced end-effect of water dehydration.

Thus, the results of the differential thermal analysis of the charges indicate that phase transformations in the hydrothermal charge occur and end at significantly lower temperatures than in the ordinary charge.

X-ray phase analysis was performed for samples of ordinary (Fig. 3) and hydrothermal (Fig. 4) charges heated to a certain set temperature. This made it possible to trace their transformation during the heat treatment process. For the hydrothermal charge, its diffractogram before heat treatment is also shown.

The XRD data confirm that tripoli exhibits a high degree of amorphousness, unlike quartz sand. In the hydrothermal charge, the rate of all reactions is much higher, as evidenced by the decrease in reflexes at lower temperatures compared to the ordinary charge. Thus, the hydrothermal charge at a temperature of 1000°C is almost amorphous, and at 1200°C all reflexes

Table 5

Formulation for conventional charge

Charge components	Component content, %
sand	57.681
soda	21.404
chalk	7.619
dolomite	7.291
kaolin	5.320
total	100.000

Table 6

Comparison of the given and calculated glass composition for conventional charge

Component of glass	SiO_2	Na_2O	CaO	MgO	Al_2O_3	Fe_2O_3
Given, %	71.50	15.30	8.30	1.90	3.00	–
By calculation, %	71.35	15.27	8.28	1.90	2.99	0.21
Imbalance, %	–0.15	–0.03	–0.02	0.00	–0.01	0.21

are absent, which indicates a 100% glassy state. It can be predicted that the amorphization of this charge ended around 1100°C. In the ordinary charge, even at 1200°C, diffraction peaks corresponding to crystalline silica are observed [3,12].

Visual observation of the stages of glass formation showed that the heat treatment of hydrothermal and ordinary charges at temperatures of 600°C, 800°C, 1000°C, and 1200°C has significant differences in the temperature intervals of the silicate and glass formation stages (Fig. 5). Thus, at a temperature of 600°C, the hydrothermal charge is represented by a sintered product, which indicates the appearance of a liquid phase caused by the melting of eutectics. At the same time, no changes have occurred in the ordinary charge. When the hydrothermal charge is heated to 800°C, its active melting with rapid gas release can be observed. This is evidenced by large cavities from gas bubbles. The ordinary charge at this temperature is a partially sintered product.

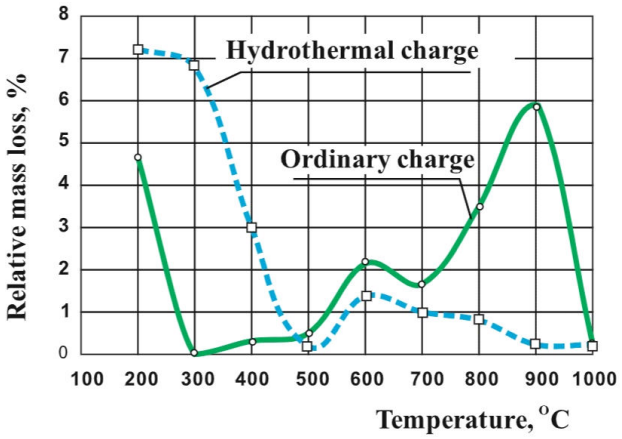

Fig. 1. Relative weight loss of samples during the heat treatment of charges

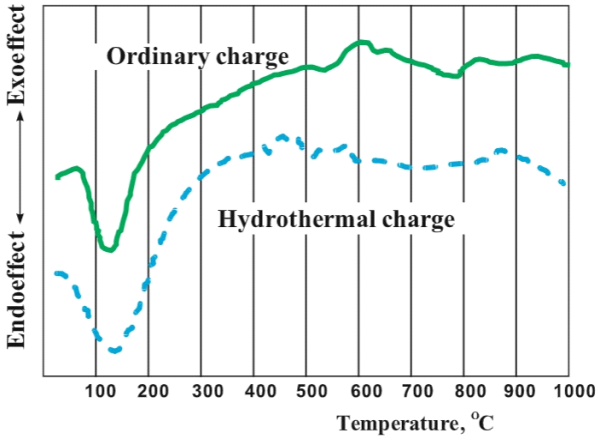

Fig. 2. Differential thermal analysis of charges

At a temperature of 1000°C, a glassy phase has already formed from the hydrothermal charge. The clarification process is nearing its completion. For the ordinary charge, we observe active melting with rapid gas emission. This is evidenced by large gas bubble cavities in the melt. At 1200°C, the hydrothermal charge is practically welded, with only residual «gnats» observed. For the ordinary charge, we observe the partial appearance of a vitreous phase with a large amount of foam in the surface layers and significant bubbles.

Thus, visual observations indicate that when using a hydrothermal charge, all stages of silicate and glass formation are approximately 200°C ahead of those for an ordinary charge.

Conclusions

The studies confirmed the advantages of using a hydrothermal charge with tripoli compared to an ordinary charge for the production of container glass. The hydrothermal method applied for making the charge using the high-silica mineral tripoli allows us to achieve a number of technological advantages. Specifically, these are the use of local mineral raw materials and reduced emissions of dusty components of the charge during preparation, transportation and loading into the glass furnace. Such a charge is characterized by high homogeneity. In addition, the use of this method allows reducing the glass melting temperature by 200–250°C compared to ordinary melting. In addition, the hydrothermal charge can be compacted and produced in the form of granules, briquettes or pressings, which will significantly improve the environmental friendliness of the glassmaking process.

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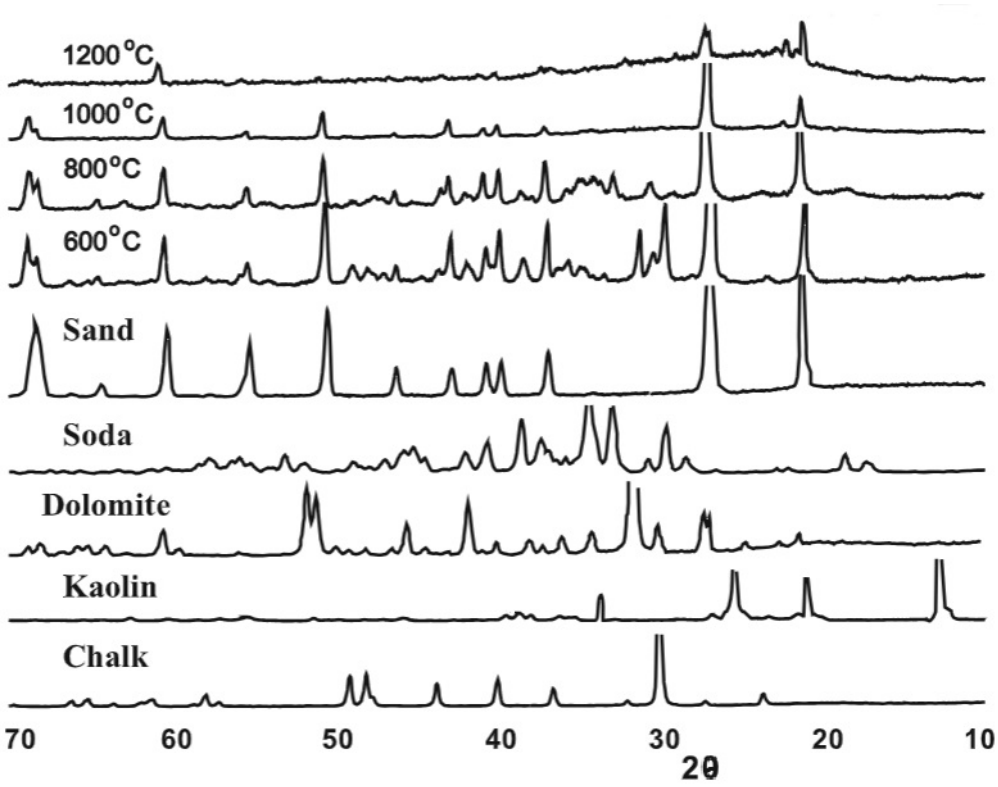

Fig. 3. XRD patterns of raw materials and ordinary charge

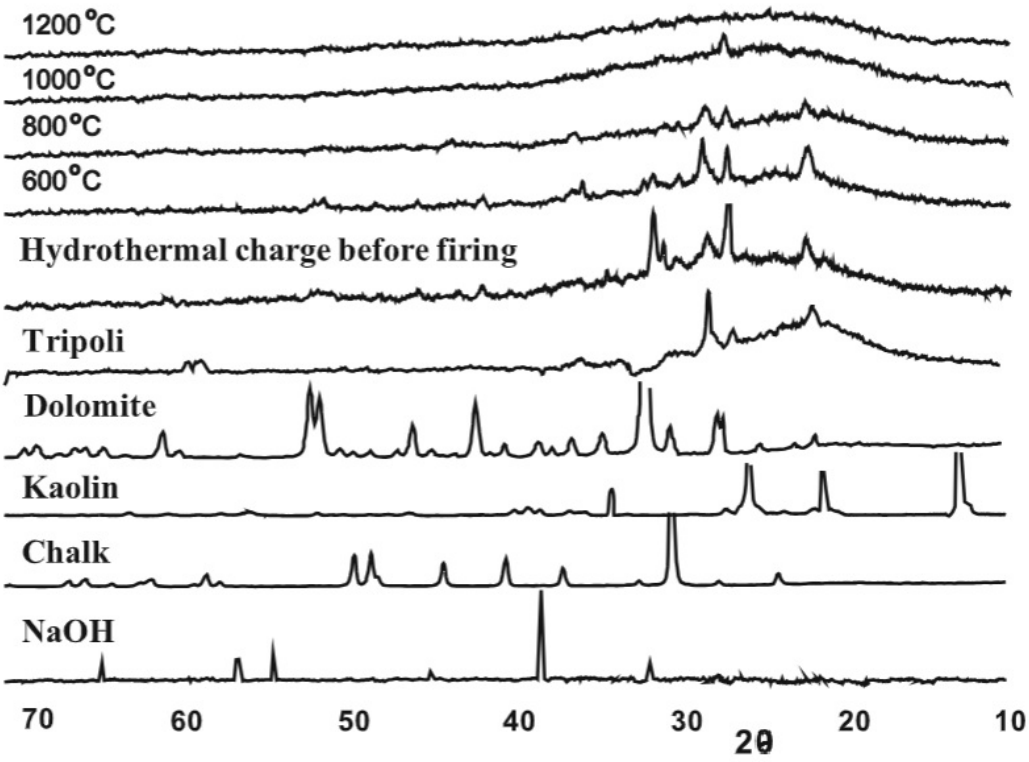

Fig. 4. XRD patterns of raw materials and hydrothermal charge

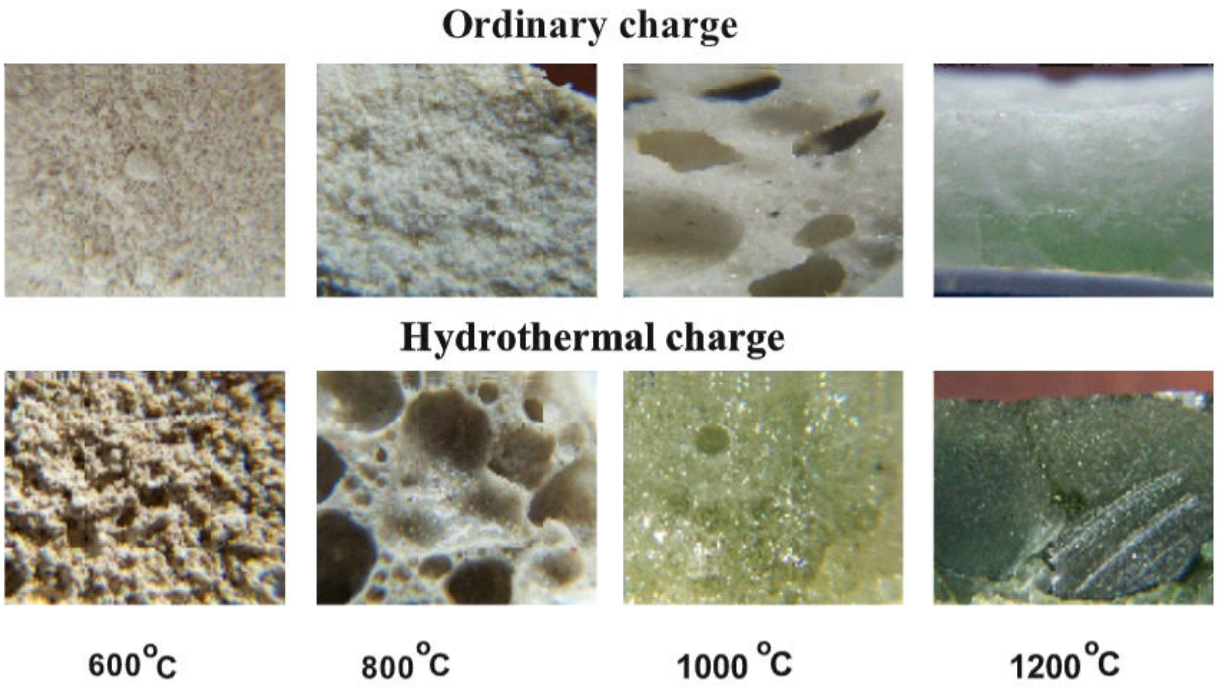

Fig. 5. Glassmaking kinetics of ordinary and hydrothermal charge

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ГІДРОТЕРМАЛЬНА ОБРОБКА ШИХТИ ІЗ ВИКОРИСТАННЯМ ВИСОКОКРЕМНЕЗЕМИСТОЇ СИРОВИНИ ПРИ ВИРОБНИЦТВІ СКЛА

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У роботі обґрунтована можливість розширення сировинної бази скляного виробництва за рахунок використання висококремнеземистої сировини – трепелу. Суть дослідження полягає в тому, що введення основного компоненту скла SiO_2 відбувається за рахунок аморфного кремнезему, що міститься у трепелі. Шихту готували гідротермальним способом. Автоклавне оброблення відпаленої суміші здійснювали за температури 150^{0}C протягом 10 годин при тиску 0,5 МПа з використанням NaOH. Вміст натрій гідроксиду у суміші для гідротермальної обробки у перерахунку на Na_2O складав 25%. Термічний аналіз традиційної шихти показав, що реакції дегідратації тривають до 200^{0}C, тоді як для гідротермальної шихти дегідратація відбувається до 500^{0}C, що пояснюється виділенням хімічно зв'язаної води силоксанових, силанольних і силандіольних груп. Для традиційної шихти наступний ендоефект починається при 500^{0}C і досягає максимуму при 540^{0}C, що обумовлений дегідратацією каоліну та на 40^{0}C вище у порівнянні з аналогічними ендоефектом гідротермальної шихти. Для традиційної шихти наступні екзоефекти спостерігаються при температурах: 610^{0}C, 650^{0}C, 830^{0}C, 940^{0}C. Вони обумовлені утворенням силікатів. Відповідні екзоефекти для зразка гідротермальної шихти спостерігаються лише два при температурах 580^{0}C і 880^{0}C. Інші екзоефекти відбуваються за нижчих температур та накладаються на виражений ендоефект дегідратації води силоксанових груп. Дані рентгенофазового аналізу підтверджують, що гідротермальна шихта за температури 1000^{0}C практично аморфізована, а при 1200^{0}C всі рефлекси відсутні, що свідчить про стовідсотковий скловидний стан. У традиційній шихті навіть за температури 1200^{0}C спостерігаються піки, що відповідають кристалічному кремнезему. Візуальне спостереження стадій скловаріння показало, що термообробка гідротермальної та традиційної шихт має суттєві різниці температурних інтервалів стадій силікато- і склоутворення. Використання гідротермальної шихти дозволяє знизити температуру варіння скла на 200–250^{0}C.

Ключові слова: гідротермальна шихта, трепел, скло, термічний аналіз; рентгенофазовий аналіз.

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Keywords: hydrothermal charge; tripoli; glass; thermal analysis; XRD.

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