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Physicochemical Analysis of Water-Soluble Sodium Silicate Obtained from Modified Raw Materials

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ABSTRACT

This article investigates the physicochemical properties of water-soluble sodium silicate synthesized from modified raw materials. During the research process, several compositions of soluble glass samples were prepared, and their structural characteristics were analyzed using infrared (IR) spectroscopy. In addition, X-ray fluorescence (XRF) analysis was employed to determine the chemical composition of the products and the distribution of their components. The results demonstrated that two of the studied compositions are considered promising in terms of technological and physicochemical parameters. In particular, the third composition provided the most optimal outcome, exhibiting superior stability, structural homogeneity, and practical applicability of the soluble glass. The findings of this study highlight the potential for developing energy-efficient technologies for the production of water-soluble sodium silicate based on local and modified raw materials. The results of infrared spectroscopic analysis showed that the BMK-3 sample exhibited significantly more stable and intense Si–O–Na bonds compared to other analyzed samples and compositions. This phenomenon is explained by the structural uniformity of the composition, the balance of cation–anion interactions, and the strength of the glass network structure. Therefore, the third composition is recommended as the most promising sample.

Keywords: water-soluble sodium silicate, modification of raw materials, IR analysis, X-ray fluorescence, optimal composition, physicochemical properties.

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Introduction

Water-soluble sodium silicates belong to the group of alkali metal silicates and are primarily obtained from a mixture of silicon dioxide and sodium oxide. Many researchers believe that the terms *"soluble glass"* and *"liquid glass"* are interchangeable. However, the concept of *"liquid glass"* is much broader, as it refers to all aqueous solutions of alkali silicates, regardless of their method of production, type of cation, concentration, or polymeric structure of silica [[1], [2]].

While soluble glass serves as a raw material for the production of liquid glass, the latter can also be synthesized through the dissolution of silica in alkalis, as well as by dissolving amorphous or crystalline, hydrated or anhydrous powdered alkali silicates. Liquid glass may be potassium-based, sodium-based, lithium-based, or ammonium-based.

Both substances are considered large-tonnage products of inorganic synthesis [[3], [4], [5], [6]].

A key feature of liquid glass production lies in its environmental friendliness, the availability of raw materials, and its low cost. The composition of soluble glass is expressed as $R_2O{\cdot}SiO_2$. In terms of physical properties, it exists as silicate lumps (solid pieces) and as an aqueous solution, which represents the actual liquid glass. The molar ratio between alkali metal oxides and silica, known as the silicate modulus (*n*), ranges from 2.0 to 4.0. Depending on the type of raw material used, soluble glasses are categorized as soda, soda-sulfate, or sulfate-based, while by composition they are classified as sodium, potassium, mixed, or double. In construction, sodium-based liquid glasses are most widely applied, followed to a lesser extent by potassium-based glasses [7].

Liquid glass is classified according to the following parameters:

1. type of cation: sodium, potassium, lithium, or organic bases;
2. silicate modulus;
3. absolute content of R_2O and SiO_2;
4. content of impurities such as oxides of iron, aluminum, calcium, magnesium, and sulfur;
5. density of the solution.

The density of sodium liquid glass ranges from ρ = 1.30–1.60 g/cm³ with a silicate modulus *n* = 2.0–3.5, while potassium liquid glass typically has a density of ρ = 1.25–1.40 g/cm³ and *n* = 2.8–4.0 [8]. Due to their wide compositional range, liquid glasses may contain various cations, silicate anions ranging from monomeric to highly polymerized forms, and silica in different structural and aggregate states. Consequently, their properties also vary within a wide range [9].

A specific feature of soluble glass lies in the gradual change of its chemical composition during operation. With decreasing alkalinity, transformation toward silica sols occurs, accompanied by changes in the physicochemical nature of the solution. Conventional sodium and potassium glasses occupy only a narrow segment within this diversity of systems [10].

The process of silicate formation from glass melt is multistage in nature, proceeding sequentially and often simultaneously through high-temperature reactions between components in both solid and liquid phases. The main transformations occurring during heating include: removal of hygroscopic moisture at 110–120 °C; elimination of crystallohydrate moisture, which may form upon moistening of the raw mixture, at 200 °C; polymorphic transitions of sodium carbonate at 235 °C; polymorphic transformations of quartz at 575 °C; and dissociation of potassium carbonate. At 800–900 °C, solid-phase silicate formation reactions begin, while at 855 °C, melting of mixture components is initiated. In the range of 900–1400 °C, eutectic phases are formed within the $R_2O–SiO_2$ system, followed by the melting of alkali–quartz compounds and the dissolution of silica. At 1400 °C, glass mass formation occurs, after which the melt undergoes cooling. In the case of sodium soluble glass, the reactions typically begin at approximately 380 °C [[11], [12], [13], [14]].

$$Na_2CO_3 + SiO_2 \xrightarrow{®} Na_2O \cdot nSiO_2 + CO_2. \quad (1)$$

The complete binding of sodium carbonate is observed at temperatures of 920–950 °C. During this process, the product consists of sodium metasilicate, silica, and alkali silicate, forming a glass-like solid structure. With increasing temperature, this mass undergoes a series of physicochemical transformations associated with the dissolution of silica, which leads to an increase in the melt volume and the formation of an excess amount of material of up to approximately 30% [[15], [16], [17]].

The minimum temperature for the formation of an alkali silicate melt is 780 °C; however, to obtain a homogeneous melt of industrial composition, a temperature of 1250 °C is required. During the glass-forming process, quartz residues gradually dissolve in the viscous sodium silicate melt, resulting in the formation of a zone enriched with SiO_2. As saturation of this zone increases, the solubility of sand decreases, while the excess amount of silica is removed through diffusion driven by the concentration gradient [18].

The diffusion rate determines the rate of glass formation and depends on temperature, viscosity, surface tension of the melt, as well as the intensity of mixing. To clarify the glass mass—removing visible gas inclusions and achieving homogenization to obtain a uniform mass—a temperature of 1400 °C is required [[19], [20]].

Experimental part

In this study, the chemical structure of sodium silicate samples was investigated using infrared (IR)

spectral analysis. The analyses were performed on a *Shimadzu Fourier-transform IR spectrometer* within the range of 4000–400 cm^{-1}, with a resolution of 4 cm^{-1}. Sample preparation was carried out using the potassium bromide (KBr) pellet technique: pre-dried and finely ground samples were uniformly mixed with KBr and compressed into transparent pellets using a specialized press. The characteristic absorption bands observed in the spectra were used to identify functional groups present in the sodium silicate structure. The elemental composition of the synthesized glass samples was analyzed using X-ray fluorescence spectroscopy (XRF). This method enables the accurate detection of both major and minor components in glass without destruction of the sample. The analyses were conducted with a modern XRF analyzer equipped with an Rh-anode X-ray tube, operating at 50 kV and 40 mA. The samples were prepared either in pressed pellet form or fused with a flux. The identified elements included the main oxides - SiO_2, Na_2O, Al_2O_3, Fe_2O_3, MgO, and CaO - as well as minor components such as K_2O, TiO_2, and P_2O_5. The analytical results were presented in the form of spectra, images, and tables. Data processing and phase interpretation were carried out using specialized software packages such as *Spectra Plus* and *Shimadzu XRF Manager*. Calibration of the instruments was performed using international standard reference materials (SRM, NIST). The applied analytical methods demonstrated several advantages, including rapid analysis, high accuracy, non-destructive sample preparation, and broad elemental coverage. These features are of critical importance for the comprehensive evaluation of glass composition and the analysis of technological processes.

Results and Discussion

The infrared spectroscopic (IR) analysis of the sample was carried out, and the obtained IR spectra are presented in Figures 1, 2, 3, and 4. In the spectrum of the sample shown in Figure 1, several distinct absorption bands were observed. The high-frequency sharp peaks at 3732, 3691, 3667, and 3610 cm^{-1} correspond to the Si–OH hydroxyl groups. The broad absorption band in the range of 3519–3193 cm^{-1} is attributed to hydrogen-bonded –OH groups and molecular water.

The characteristic deformations related to silicate structures were identified at 1245 and 1139 cm^{-1}, which correspond to Si–O–Si linkages in sodium silicate. Additionally, the absorption band at 1022 cm^{-1} indicates the presence of Si–O^-–Na vibrational groups coordinated with sodium, which is a distinctive feature of sodium silicate.

In the spectrum of the sample shown in Figure 2, a broad absorption band at 3436 cm^{-1} was identified, corresponding to –OH groups and molecular water, although present in very small amounts. The absorption bands at 2361 and 2342 cm^{-1} are typically associated with the vibrational modes of residual CO_2 carbonates, which may indicate that the sample absorbed CO_2 from the atmosphere. A strong absorption peak observed at 1033 cm^{-1} is attributed to the Si–O–Si stretching vibrations and, to some extent, to Si–O^-–Na vibrations, which are characteristic for sodium silicate. The band at 912 cm^{-1} corresponds to Si–O^- groups coordinated with sodium, confirming features typical of soluble glass. Furthermore, the absorption peaks at 777 and 687 cm^{-1} represent the bending vibrations of Si–O–Si bonds, while the bands at 581 and 454 cm^{-1} correspond to the skeletal vibrations of the silicate framework, indicating the structural stability of the silicate network.

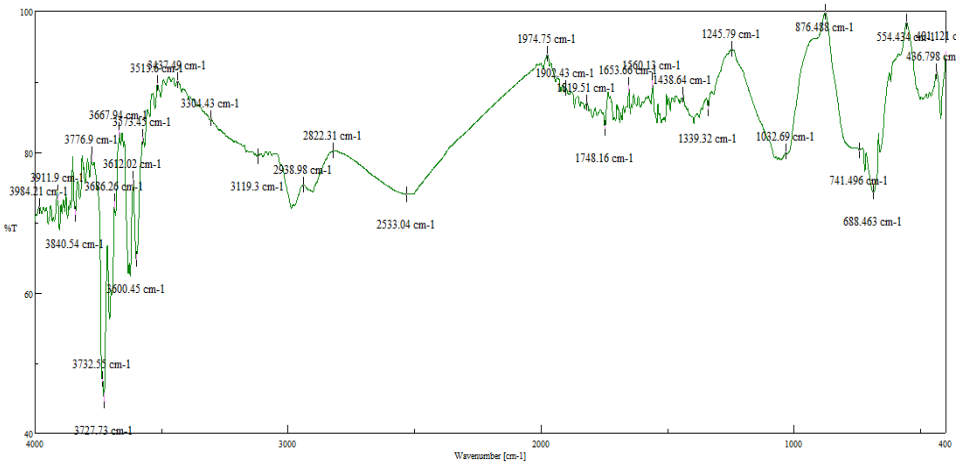

Figure 1 - IR spectroscopic analysis of Sample 1 of soluble sodium silicate

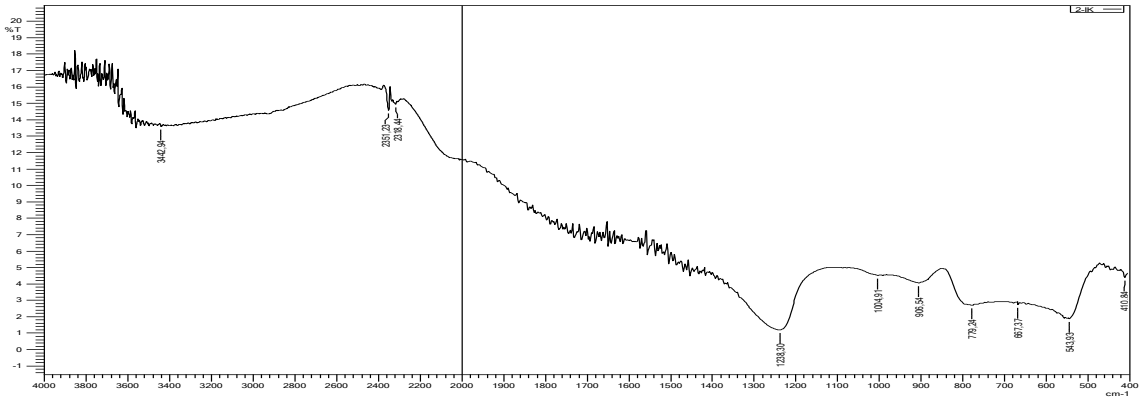

Figure 2 - IR spectroscopic analysis of Sample 2 of soluble sodium silicate

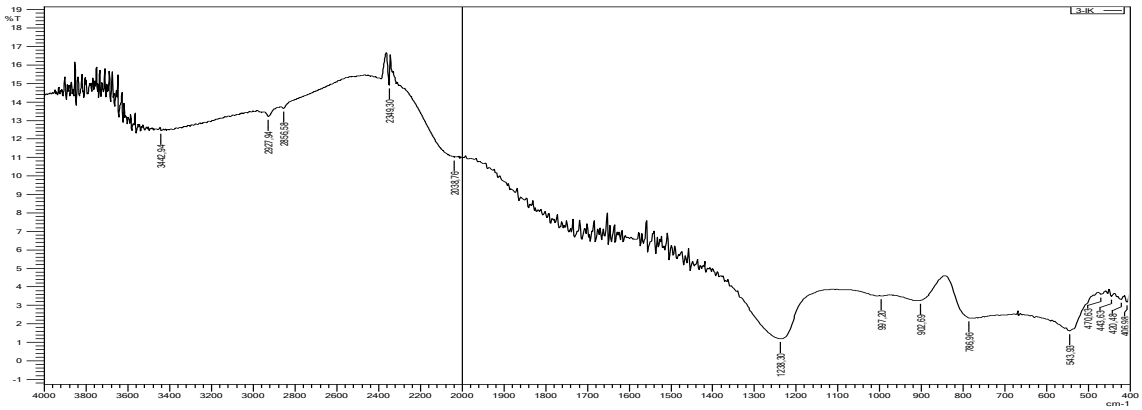

Figure 3 - IR spectroscopic analysis of Sample 3 of soluble sodium silicate

In the spectrum of the sample shown in Figure 3, a broad absorption band at 3434 cm^{-1} was observed, corresponding to the –OH groups and molecular water. The absorption peaks at 2361 and 2336 cm^{-1} are typically attributed to the vibrational modes of CO_2 carbonate groups. A strong and sharp band at 1030 cm^{-1} is assigned to the Si–O–Si stretching vibrations, while the absorption band at 912 cm^{-1} indicates the presence of $Si–O^-$ groups coordinated with sodium, a characteristic feature of soluble glass.

Additionally, the absorption bands at 787 and 719 cm^{-1} correspond to the bending vibrations of Si–O–Si bonds, whereas the peaks at 654, 614, and 454 cm^{-1} are associated with the skeletal vibrations of the silicate framework, reflecting the stability and integrity of the silicate network structure.

According to the results of the spectrum shown in Figure 4, the broad absorption bands in the range 3957–3610 cm^{-1} correspond to the O–H stretching vibrations of hydroxyl groups. The peaks at 2929 cm^{-1} and 2854 cm^{-1} are attributed to the C–H stretching vibrations of methyl or methylene groups. The absorption at 2358 cm^{-1} is assigned to CO_2 molecules, which may have been absorbed from the surrounding atmosphere. The band at 1994 cm^{-1} can be related to complex Si–O–Si vibrations or mixed vibrational modes.

The absorption range 1732–1566 cm^{-1} indicates the presence of C=O groups, which may belong to carboxyl or carbonate compounds, as well as possible H–O–H bending vibrations of molecular water. A very strong absorption band between 1054–995 cm^{-1} is attributed to the Si–O–Si stretching vibrations of the silicate framework. The bands at 823–761 cm^{-1} correspond to Si–O deformation vibrations within the silicate structure, while the absorptions at 665–472 cm^{-1} are associated with the Si–O bending vibrations characteristic of sodium silicates.

The spectrum clearly demonstrates that sodium silicate is the main component. The presence of organic residues (C–H, C=O bands) suggests that the sample was derived from a waste-based or mixed raw material system. In the lower-frequency region (400–800 cm^{-1}), the presence of distinct absorption bands further confirms the inorganic silicate network structure. Overall, this spectrum is typical of soluble sodium silicate synthesized from waste-containing or mixed-component systems.

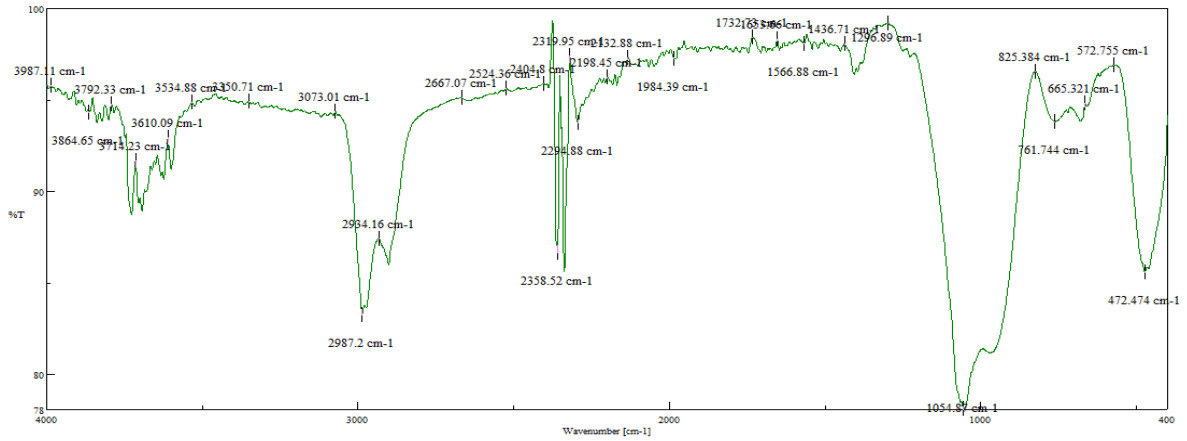

Figure 4 - IR spectroscopic analysis of Sample 4 of soluble sodium silicate

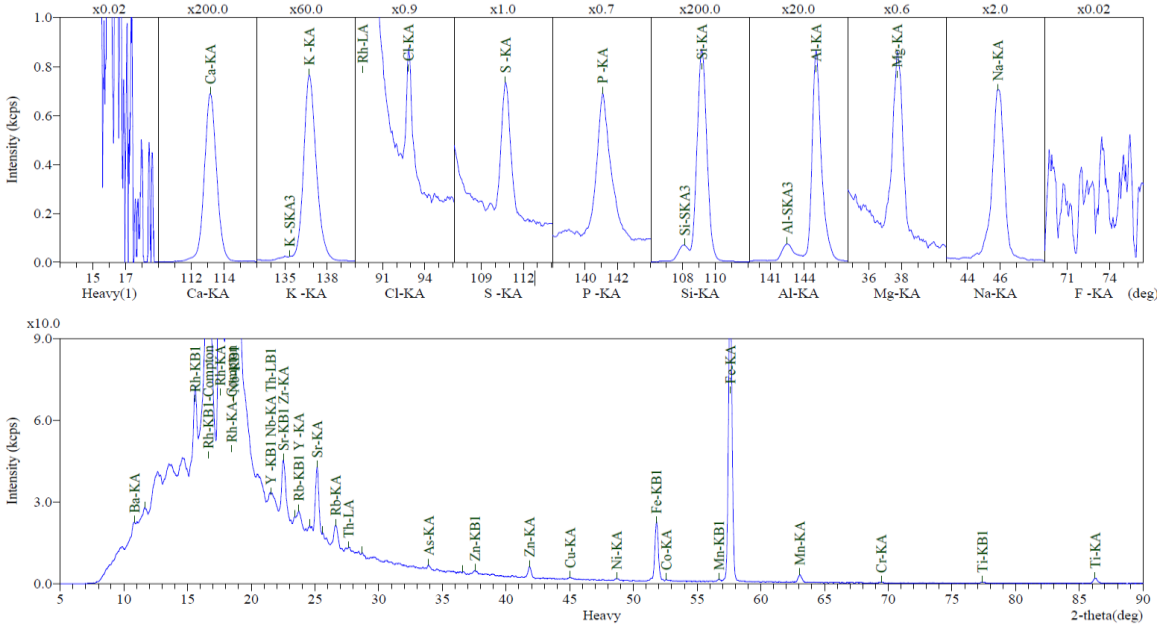

Figure 5 - X-ray fluorescence (XRF) analysis of soluble sodium silicate, Sample 3

Table 1 - Chemical composition of soluble sodium silicate

Sample	Oxide content, wt.%									LOI, wt.%
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	P_2O_5	
Sample - 3	67.9	1.74	1.86	1.77	0.844	1.79	23.0	0.2	0.02	0.876

Based on the results of the IR spectroscopic analysis, the characteristics of Sample 3 revealed properties and features typical of soluble glass.

To further verify these findings, an X-ray fluorescence (XRF) analysis was carried out in order to determine the oxide composition of Sample 3 and to evaluate its compliance with the requirements of GOST 13079–2021, as well as to calculate the silicate modulus (fig. 5 and Tab. 1).

The chemical composition of the obtained samples was analyzed based on Table 1, and it was determined that their main components are SiO_2, Na_2O, CaO, Al_2O_3, and Fe_2O_3 oxides. The SiO_2 content of 67.9% plays an important role in ensuring the structural stability of the glass. Na_2O (23.0%) is the main compound required for the synthesis of soluble glass. Its sufficient amount accelerates the bonding process with SiO_2 and increases solubility. CaO (1.77%) and MgO (0.844%) serve as additional modifier oxides that improve the mechanical strength and thermal resistance of the glass mass. However, their excessive content may reduce the solubility of the glass. Fe_2O_3 was found in a concentration of 1.86%, and a higher amount of this oxide can negatively affect the quality of the glass. In addition, other impurities such as Cl^- and SO_4^{2-}, if

present in significant amounts, can also have a detrimental effect. Therefore, technological regulation of their concentration is of great importance.

Conclusions

In all spectra, broad peaks were observed in the range of 3200–3600 cm^{-1}, indicating the presence of hydroxyl groups. Higher peak intensity suggests that the sample has better potential for dissolution during liquid glass production. Strong Si–O–Si stretching vibrations were observed in the range of 1000–1200 cm^{-1} across all four spectra. To determine the most suitable composition, the peaks should be sharp and well-defined, without additional side signals, as this indicates a well-developed silicate network. Si–O bending vibrations were present in the range of 450–800 cm^{-1} in all samples. In the spectra of Figures 3 and 4, a distinct peak around 470 cm^{-1} was observed, confirming the presence of a fully polymerized silicate structure. In Figures 2 and 3, weak peaks in the range of 1450–1500 cm^{-1} were detected, suggesting the presence of small amounts of carbonates and organic impurities, which can lower the quality of the solution. In this study, a peak at 2358 cm^{-1} was detected, corresponding to absorbed CO_2, likely introduced from the surrounding atmosphere. Although this can be removed during processing, excessive amounts may negatively influence product quality. Based on these observations, the first sample revealed OH groups but also noticeable carbonate impurities. The second sample showed strong Si–O peaks, but additional signals in the range of 1400–1500 cm^{-1} were present. The third sample exhibited distinct silicate peaks, with a strong peak at 470 cm^{-1}, indicating a well-polymerized structure. The fourth sample contained Si–O peaks but also CO_2 traces at 2358 cm^{-1}. Overall, the third sample spectrum was determined to be the most suitable for liquid glass production. This is attributed to its sharp and strong Si–O–Si peaks, high degree of polymerization, lower carbonate (CO_2) impurities, and balanced OH content related to moisture. Comparative analysis of the chemical composition confirmed that the third sample was optimal for liquid glass synthesis. This is due to the balanced ratio of SiO_2 and Na_2O, along with relatively lower levels of harmful impurities such as Fe_2O_3 and SO_4^{2-}. These findings demonstrate the potential of producing liquid glass through energy-efficient technologies based on waste-derived raw materials.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Модификацияланған шикізаттан алынған суда еритін натрий силикатының физика-химиялық талдауы

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ТҮЙІНДЕМЕ
Мақалада модификациялық шикізаттар негізінде синтезделген еритін натрий силикатының үлгілерінің физика-химиялық қасиеттері зерттелді. Зерттеу барысында әртүрлі құрамдағы еритін шыны үлгілері дайындалып, олардың құрылымы инфрақызыл (ИҚ) спектроскопия әдісі арқылы талданды. Сонымен қатар, өнімдердің химиялық құрамын және компоненттердің таралуын анықтау мақсатында рентгенофлуоресценттік (РФ) талдау әдісі қолданылды. Алынған нәтижелер зерттелген құрамдардың екеуінің технологиялық және физика-химиялық көрсеткіштері бойынша болашағы бар екенін көрсетті. Ал үшінші құрам еритін шынының жоғары тұрақтылығын, құрылымдық консистенциясын және практикалық қолданудағы тиімділігін қамтамасыз ететін оңтайлы нәтижелер көрсетті. Зерттеу нәтижелері жергілікті және модификацияланған шикізат негізінде еритін натрий силикатын өндіру үшін энергия үнемдейтін технологияларды әзірлеу мүмкіндігін анықтайды. ИК-спектроскопиялық талдау нәтижелері бойынша үшінші үлгінің спектрограммасында Si–O–Na байланыстары басқа зерттелген үлгілер мен құрамдармен салыстырғанда анағұрлым тұрақты және жоғары интенсивтілік байқалды. Бұл құбылыс құрамның құрылымдық біртектілігімен, катион-анион байланыстарының тепе-теңдігімен және шыны торының беріктігімен түсіндіріледі. Осыған байланысты ВМК-3 құрамы ең болашағы зор үлгі ретінде ұсынылады.

Түйін сөздер: еритін натрий силикаты, модификациялық шикізат, ИҚ талдау, рентген флуоресценция, оңтайлы құрам, физика-химиялық қасиеттер.

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Физико-химический анализ водорастворимого силиката натрия, полученного из модификационного сырья

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АННОТАЦИЯ
В данной статье изучены физико-химические свойства образцов растворимого силиката натрия, синтезированных на основе модификационного сырья. В процессе исследования были подготовлены образцы растворимого стекла различного состава, структура которых проанализирована методом инфракрасной (ИК) спектроскопии. Кроме того, для определения химического состава продуктов и распределения компонентов был применён метод рентгенофлуоресцентного (РФ) анализа. Полученные результаты показали, что два из исследованных составов являются перспективными по своим технологическим и физико-химическим показателям. В частности, третий состав продемонстрировал оптимальные результаты, обеспечив высокую стабильность растворимого стекла, структурную согласованность и эффективность в практическом применении. Результаты исследования свидетельствуют о возможности разработки энергосберегающих технологий производства растворимого силиката натрия на основе местного и модификационного сырья. Результаты ИК-спектроскопического анализа показали, что в спектрограмме образца ВМК-3 связи Si–O–Na проявляются значительно более устойчивыми и интенсивными по сравнению с другими исследованными образцами и составами. Данное явление объясняется структурной однородностью состава, сбалансированностью катион-анионных взаимодействий и прочностью стеклосеточной структуры. В связи с этим третий состав рекомендуется как наиболее перспективный образец.

Ключевые слова: растворимый силикат натрия, модификационное сырьё, ИК-анализ, рентгенофлуоресценция, оптимальный состав, физико-химические свойства.

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