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Production of glass-alkali binder for construction purposes by recycling of cullet

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Abstract. Municipal solid waste poses significant environmental challenges due to its wide range and potential contaminating impact. Finding sustainable solutions for its disposal is imperative. Moreover, certain types of municipal solid waste can serve as valuable resources in the construction sector. This study introduces a novel non-firing binder, devoid of cement, crafted from cullet, caustic alkali, water, and a plasticizing additive. These constituents undergo collaborative wet grinding in a ball mill, achieving a specific surface area of 500–550 m^2/kg. Concurrently during milling, glass particles are ground, and amorphous silica is leached with an alkaline solution, yielding a viscous-fluid adhesive mass enriched with siliceous compounds. This mass fills metal mold cells; upon attaining stripping strength, samples undergo heat treatment (drying) up to 90ºC. During this process, sols transform into polysilicic acid gels, which, after 5–6 hours, partially crystallize, achieving requisite strength. The resulting binder, produced without firing, boasts a compressive strength of approximately 25 MPa and a water resistance coefficient of 0.89. Suitable for low-grade concrete production (including glass concrete, fine-grained concrete, and foam concrete), its microstructure was analyzed via scanning electron microscopy, affirming the effective utilization of cullet in construction materials.

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1. Introduction

The work is devoted to the development of the composition and technology for producing glass-alkaline binder, which promotes the development of environmentally friendly building materials at lower hardening temperatures.

Annually, the Russian Federation generates approximately 35–40 million tons of municipal solid waste, with 8–10% comprising glass waste, a valuable but underutilized by-product. Challenges in glass waste utilization include its heterogeneous chemical composition, presence of contaminants, and the high cost associated with its extraction from mixed waste streams. Concurrently, addressing issues related to industrial waste disposal while enhancing resource efficiency and improving the technical properties of construction materials has spurred interest in utilizing cullet from various types of unsorted glass, such as window, container, and household glass.

Cullet is waste glass that is crushed and ready to be remelted. It is an important component in the glass recycling process, used to make new glass bottles and other products. The main consumer for the recycled vitreous material (cullet) is the glass manufactory industry.

Currently, over 2.5 million tons of cullet accumulate in Russian landfills, with volumes steadily increasing. Active research endeavors seek to explore diverse avenues for cullet utilization. Crushed cullet finds application in plastics and various construction materials, including foam glass production, concrete and bitumen additives, and the manufacture of porous facing materials, tiles, panels, artificial slate, and roofing materials, road beds, pavement, trench fill, drainage medium, etc.; and in general use applications including abrasives, fluxes/additives, manufacturing of fiberglass insulation and foam insulation.

When used as sand replacement, there is some indication that there can be a noticeable reduction in compressive strength [1]. This is, however, not consistently observed [2, 3].

Concretes made from alkali-activated fly ash with pulverized glass have also been investigated [4].

Alkali-activated Class F fly ash cement with glass aggregates called “ashcrete” has also been studied [5]. The issue of alkali-silica reaction induced expansion is not a significant problem with alkali-activated fly ash cement mortar in comparison to the use of glass aggregate in normal mortar and concrete. This “ashcrete” has high strength and develops high early strength, which makes it very suitable for the precast concrete industry.

In the work [6], the alkaline solution formed with the glass waste will be use like alkaline activation agent for the blast furnace slag to produce cementitious materials.

Notably, cullet can serve not only as an aggregate, filler or alkaline activator but also as a component of binders, enabling the production of multifunctional materials.

Literature underscores the potential of finely crushed cullet, modified with additives, and subjected to heat and moisture treatment, to serve as a hydraulic binder for diverse construction applications.

Soda-glass cullet powder with specific surface area up to 400 m^2/kg has been used to produce geopolymers [7]. In contrast to fly ash or metakaolin-based geopolymers, water glass is not needed for the setting of glass cullet geopolymers.

Hence, there is a pressing need to develop a binder composition based on cullet capable of achieving strength under ambient conditions or through heat treatment at temperatures up to 100 °C. Grinding methods, including wet grinding, are explored to obtain finely dispersed powders with high specific surface areas, enhancing material chemical activity through surface effects.

When formulating the binder composition, the authors drew upon previous research findings by A.D. Bogatov et al. [8] and S.N. Bogatova et al. [9], who successfully developed non-autoclave composite building materials utilizing glass cullet mixed with alkaline solutions, resulting in enhanced resistance to aqueous acid solutions and biologically active media. However, their approach involved dry grinding of cullet followed by treatment with an alkaline solution with mechanical stirring, leading to partial extraction of amorphous silica solely from the surface of glass cullet particles. In contrast, this study employed grinding of cullet in a concentrated alkaline solution, enabling simultaneous breakdown of glass particles and dissolution of amorphous silica throughout the particle volume. This method ensured a higher concentration of silica gel formation.

The utilization of industrial waste, particularly cullet, in binder and concrete production, represents a promising frontier in modern materials science, gaining significance in recent years. Despite discussions dating back to the 1970s regarding the potential recycling of broken glass in the building materials industry, practical research in this domain has been limited. Presently, several Russian [10] and international universities and research institutes are actively engaged in developing building alkali activated composites (AAC) in general [11], and utilizing waste glass [12–14] and glass cullet [15, 16] in AAC.

The primary objective of this study is to develop effective technologies enabling the use of glass cullet not only as a filler but also as an independent binder, aligning with previous work by Y. Liu et al. [12], J.X. Lu et al. [13], A.B. Pascual et al. [14], R. Vinai et al. [15], J. Giro-Paloma and M. Soutsos [16], N.I. Kozhukhova et al. [17], V.G. Klimenko et al. [18]. Noteworthy contributions from foreign researchers include studies on the effective substitution of cement with finely dispersed glass [19–21].

However, the above-mentioned authors obtained a glass cullet-based binder by dry grinding the glass cullet to a fine dispersion similar to that of Portland cement, and then mixed the grinding product together with an aqueous solution of caustic alkali. In this case, the dissolution of amorphous silica occurred only on the surface of the particles in the glass cullet, which does not ensure its high concentration in the total mass of the binder.

Considering the high energy intensity and associated cost of such technological processes, the development of the use of cullet in the construction industry is relevant – the production of binders and concrete that can harden at temperatures up to 90 °C.

The purpose of this research is to develop a glass-alkali binder using energy-efficient technology: wet grinding of glass cullet in alkaline solution, which allows the use of technogenic raw materials and temperatures below 90 °C while maintaining the performance properties of the final product.

Research objectives: development of glass-alkaline binder compositions based on cullet with improved strength and water resistance; development of compositions of fine-grained concrete based on the obtained binder and glass cullet as a fine aggregate while maintaining strength characteristics; development of technology that allows producing glass-alkaline binder at a temperature up to 90 °C.

2. Materials and Methods

The primary glass component for the binder comprised cullet sourced from window and/or container glass.

The alkaline activator utilized was technical caustic soda (NaOH) conforming to the Russian Standard GOST–R 55064–2012, or potassium hydroxide (KOH) as per Russian Standard GOST 24363–80.

As a plasticizer, Melflux 2651 F manufactured by BASF Construction Additives, Germany, was employed. Melflux 2651 F, a spray-dried powder product based on modified polyester carboxylate, exerted a thinning effect on the binder mass, reducing the required mixing water while simultaneously increasing the alkali concentration in the solution. Tap water served as the solvent [22–24].

Table 1 presents the average chemical composition of the cullet used in the study.

Table 1. Chemical composition of glass cullet (wt. %).

Sourse of glass cullet	Oxide content (wt. %)					
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O+K_2O
Window glass	69.0–72.5	1.5–4.2	0.1–0.8	7.5–8.7	2.5–3.5	13.2–14.0
Container glass	71.5–73.7	0.2–3.3	0.8–1.7	5.2–9.1	0.1–0.6	14.0–14.8

3. Results and Discussion

The cullet sourced from window and/or container glass underwent crushing using a jaw crusher with an outlet size of 2.5–5 mm. Approximately 500 g of crushed glass cullet was then introduced into a porcelain ball mill along with an alkaline solution containing dissolved caustic alkali and a plasticizer. The mixture underwent wet grinding in the designated spherical mill. The components were wet-milled in the specified milling unit for 2–8 h. During this process, the specific surface area of the milled product and the average particle size were measured (Table 2).

Table 2. Change in glass cullet dispersion depending on grinding time.

№	Grinding time, hours	Specific surface area, m^2/kg	Average particle diameter, µm
1	2	386.4	5.9
2	4	516.6	4.6
3	6	541.2	4.3
4	8	550.8	4.2

The results of the experiment (Table 2) showed that an intensive increase in the specific surface area is observed during 6 hours of grinding. Then, the increase slows down, and after 8 hours it becomes ineffective. Thus, the optimal grinding duration was 6 hours. As a result, the specific surface area of the cullet, serving as the primary component of the glass-alkali binder, reached 500–550 m^2/kg, with an average particle size ranging from 4.4–4.6 µm. The resultant milled product exhibited a viscous-fluid adhesive mass, utilized to fill the cubic cells of metal molds [25–27].

Following an exposure period of 16–18 hours under ambient conditions, during which the mixture acquired stripping strength, cube-shaped samples were extracted from the molds. Subsequently, these samples underwent heat treatment in a drying chamber at temperatures ranging from 85–90ºC for 5–6 hours. Post-heat treatment, the cooled samples were measured, weighed, and subjected to tests evaluating density, compressive strength, and water resistance. The outcomes of these tests are detailed in Table 3.

Table 3. Composition of glass-alkali binder and its physical and mechanical characteristics.

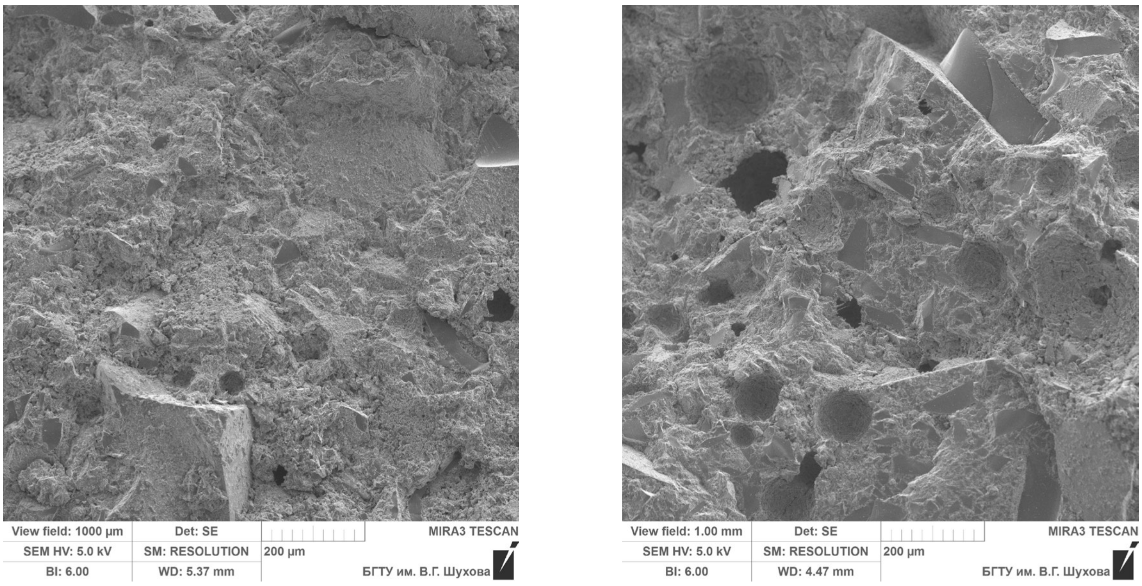
Mix ID	Mixes of glass-alkali binder, wt. %			Physical and mechanical characteristics							
	Glass cullet	Alkali component	W/C ratio	Average density, kg/m^3		Compressive strength, MPa in dry state		Compressive strength, MPa in water saturated state		Water-resistance coefficient	
				NaOH	KOH	NaOH	KOH	NaOH	KOH	NaOH	KOH
1	98.5	1.5	0.2	1802	1800	17.5	17.5	14.7	15.2	0.84	0.87
2	98.3	1.7		1809	1806	20.3	19.6	16.2	15.8	0.80	0.81
3	98.0	2.0		1816	1813	21.6	20.1	18.6	16.8	0.86	0.84
4	97.7	2.3		1823	1819	22.4	20.3	20.2	18.5	0.90	0.91
5	97.5	2.5		1828	1820	23.1	20.4	20.3	17.7	0.88	0.87
6	97.2	2.8		1838	1835	26.4	25.8	23.5	22.9	0.89	0.89
7	97.0	3.0		1823	1818	22.5	21.8	19.8	19.0	0.88	0.87
8	96.7	3.3		1818	1810	21.6	20.3	18.6	18.2	0.86	0.90

The results depicted in Table 3 reveal that samples of the glass-alkali binder, post-drying, exhibit a compressive strength ranging from 17.5 to 26.4 MPa, with their water-resistance coefficient (the ratio of strength in a water-saturated state to the strength of dry samples) varying between 0.80 and 0.91, respectively. Notably, Mix 4 emerges as optimal, displaying the highest compressive strength and water resistance.

Microstructural analysis of fresh specimen fragments, obtained post-strength testing, was conducted utilizing a high-resolution scanning electron microscope TESCAN MIRA 3 LMU. Examination of the microstructure revealed that during cullet grinding in the presence of an alkali solution, finely dispersed amorphous silica dissolves from the glass melt. The resultant polysilicic acid gel, formed through polycondensation reactions, exhibits high adhesion, capable of binding incompletely dissolved glass particles together. Subsequent heat treatment (up to 90°C) induces the crystallization of the polysilicic acid gel, elucidating the relatively elevated values of density, compressive strength, and water resistance observed in glass-alkali binder samples. This phenomenon was documented in SEM images obtained for mixes employing potassium and sodium alkalis [28–30].

As illustrated in micrographs (Fig. 1) captured at various magnifications, the microstructure of the glass-alkali binder resembles a stone-like substance interspersed with partially crystallized silica gel (filamentous and lamellar fragments), with undissolved glass phase particles tightly adjoining them. This configuration facilitates the formation of a compacted and reinforced structure, irrespective of the alkali type used.

a b



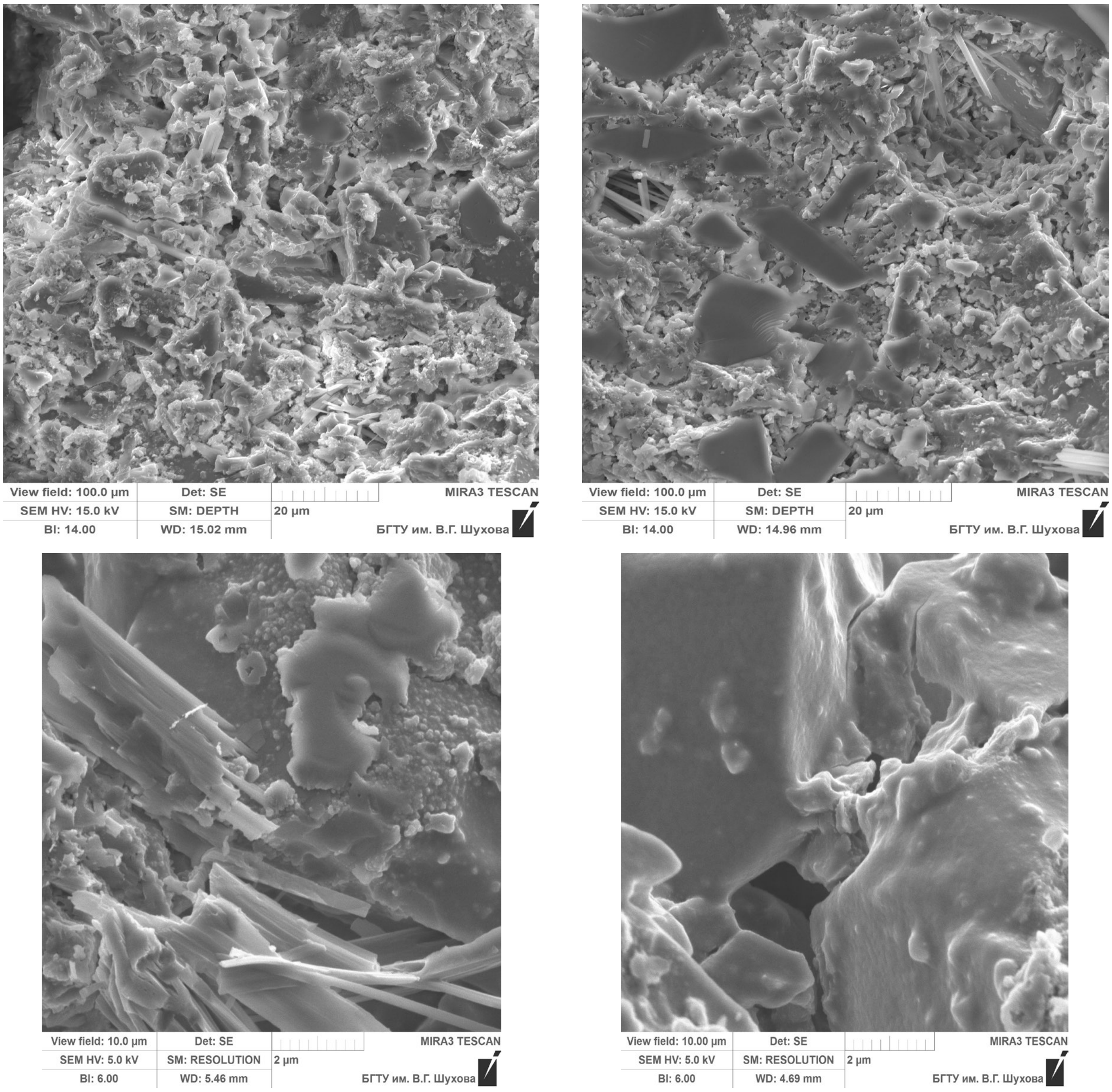


Figure 1. Microstructure of the glass-alkali binder Mixes: a) using KOH; b) using NaOH.

The results of X-ray phase analysis revealed crystalline phases of potassium silicates – $K_6Si_2O_7$, K_4SiO_4, $K_4Si_8O_{18}$, $K_2Si_2O_5$ and K_2SiO_3 – in the hardened binder modified with KOH aqueous solution (Fig. 2).

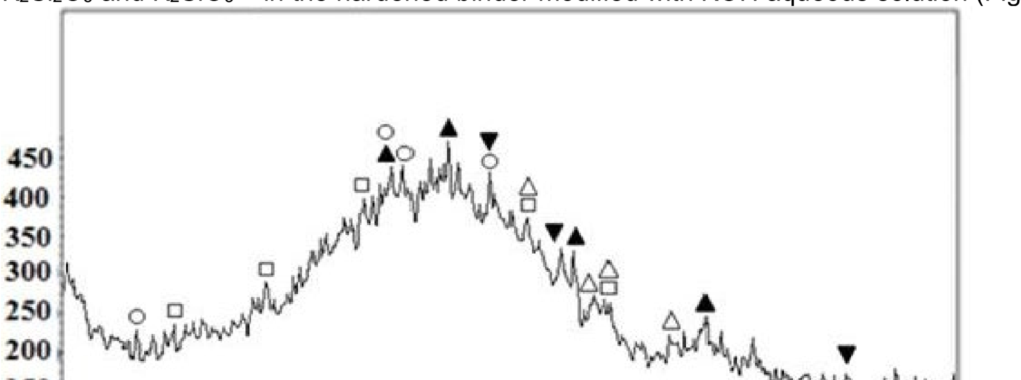


Figure 2. X-ray profile of glass-alkali binder modified with KOH aqueous solution:
∆ – $K_6Si_2O_7$; ▼ – K_4SiO_4; □ – $K_4Si_8O_{18}$; ○ – $K_2Si_2O_5$; ▲ – K_2SiO_3.

In samples modified with NaOH solution, sodium silicates such as the following are observed: Na_2SiO_3, $Na_6Si_{40}O_{83}$, Na_4SiO_4 и $Na_2Si_3O_7$ (Fig. 3).

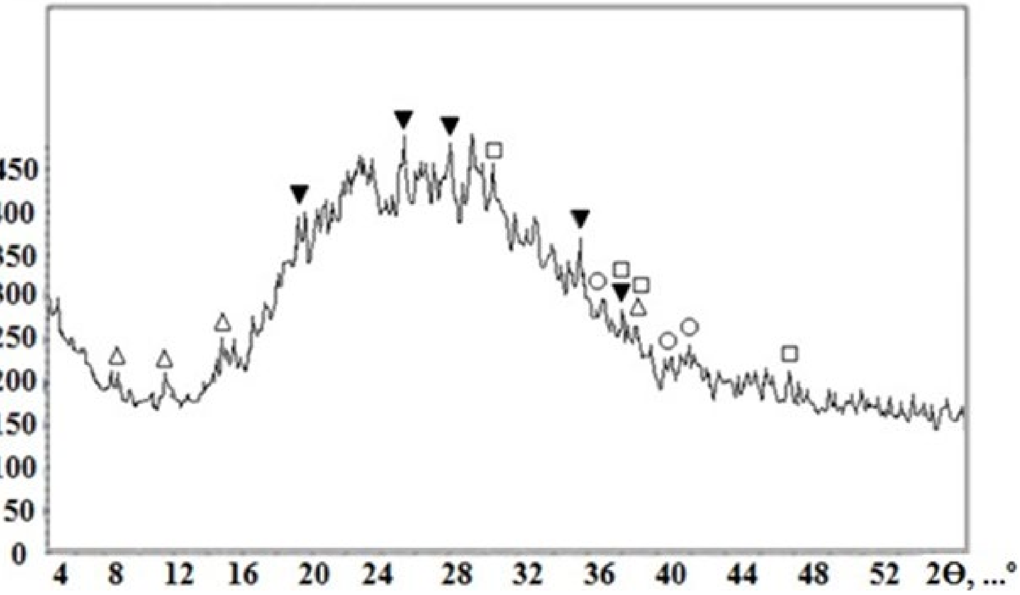


Figure 3. X-ray profile of glass-alkali binder modified with NaOH aqueous solution: □ – Na_2SiO_3; ∆ – $Na_6Si_{40}O_{83}$; ○ – Na_4SiO_4; ▼ – $Na_2Si_3O_7$.

Fine aggregates comprised quartz sand and crushed cullet with possessing a grain size of up to 1.25–0 mm. This fraction was selected considering workability and exhibiting the highest strength indicators in preliminary investigations.

The developed Mixes and the associated manufacturing method offer a pathway to create a binder without reliance on cement, lime, or other calcined binders, thereby circumventing the need for calcination and hydrothermal (autoclave) technologies, while minimizing energy consumption. The total curing time (until maximum strength is achieved) is 24 hours. The proposed glass-alkali binder holds promise for serving as a binder in the production of building materials and products, such as glass concrete, fine-grained concrete, and foam concrete. Utilizing household waste in the form of cullet as a predominant binder component, exceeding 80%, suggests the potential for significant-scale recycling of this waste.

In further studies, glass-alkali binder, Mix 6 (according to Table 3), based on NaOH at different W/C ratios in the range of 0.15–0.25, was used to obtain fine-grained silicate concrete. As a fine aggregate, quartz sand or glass cullet were used. Each aggregate was used separately. Fine aggregate was blended with a specific amount of glass-alkali binder. The resultant mixture was then shaped into metal molds with dimensions of 3×3×3 cm, compacted on a shaking table, and left to set overnight to gain stripping strength. Upon demolding, the samples underwent heat treatment in a drying chamber at temperatures ranging from 85–90ºC for 6 hours. Subsequently, the heat-treated samples underwent physical and mechanical testing, the outcomes of which are outlined in Table 4.

Table 4. Physical and mechanical characteristics of fine-aggregate concrete based on glass-alkali binder.

Mix ID	Solid components, wt. %			W/C ratio	Average density, kg/m^3	Compressive strength, MPa
	Glass-alkali binder	Quartz sand	Glass cullet			
1	75	25	–	0.15	1929	23.31
2	75	–	25		1926	22.44
3	75	25	–	0.18	1943	25.02
4	75	–	25		1938	24.52
5	75	25	–	0.20	1906	20.67
6	75	–	25		1884	18.85
7	75	25	–	0.25	1844	14.41
8	75	–	25		1836	14.08

The experimental findings presented in Table 3 demonstrate that the cement-free glass-alkali binder yields fine-grained concrete with strength properties ranging from 14–25 MPa when employing either quartz sand or crushed cullet as fine aggregates. The subsequent concrete curing process involves drying at temperatures up to 90ºC, eliminating the need for energy-intensive heat, moisture, and autoclave treatments. This innovative approach minimizes energy and steam consumption while achieving robust concrete properties.

4. Conclusion

As a result of the studies, samples of glass-alkaline binder with a compressive strength from 17.5 to 26.4 MPa and a water resistance coefficient from 0.80 to 0.91 were obtained.

Based on the developed glass-alkaline binder, fine-grained concrete mixes with compressive strength values of 14–25 MPa were developed. The results obtained do not depend on the type of fine aggregate: quartz sand or glass waste – crushed glass cullet.

Thus, the conducted research indicates the feasibility of utilizing a cement-free and non-fired binder in the production of fine-grained concrete (utilizing quartz sand as aggregate) and glass concrete (employing cullet as aggregate). The manufacturing process of this binder is characterized by its low energy consumption, offering a sustainable alternative to traditional cement-based binders.

Moreover, the development of the glass-alkali binder facilitates the utilization of a substantial portion of household waste, specifically cullet, exceeding 80%. This presents an opportunity for large-scale waste disposal, thereby significantly mitigating the adverse environmental impact. By incorporating innovative materials and production techniques, this approach aligns with sustain-able development goals, promoting both environmental stewardship and resource efficiency.

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