

Optimization of the composition and properties of lightweight materials based on liquid glass

Igor V. Bessonov¹ , Alexey D. Zhukov^{1,2} , Andrey A. Medvedev^{2,3} , Elina A. Malova^{1,2*} ,
Ilya S. Govryakov¹ 

¹ Research Institute of Building Physics Russian Academy of Architecture and Construction Sciences, 21, Lokomotivny Proyezd, Moscow, 127238, Russian Federation

² National Research Moscow State University of Civil Engineering, 26, Yaroslavskoye Shosse, Moscow, 129337, Russian Federation

³ Russian State Geological Prospecting University named after Sergo Ordzhonikidze, 23, Ulitsa Miklukho-Maklaya, Moscow, 117485, Russian Federation

* Corresponding author: e-mail: eg15082000@mail.ru

ABSTRACT

Introduction. The use of aluminosilicate microspheres makes it possible to produce lightweight, high-strength fired materials. The use of liquid glass as a binder allows products at a firing temperature of 400 °C, significantly reducing energy consumption compared to ceramic materials. The aim of the research presented in the article was to develop and test a method for assessing the properties of fired products based on liquid glass and silicate microspheres, as well as to select optimal formulation and process parameters.

Methods and materials. Liquid glass with a density of 1400 kg/m³ is used as a binder, and aluminosilicate microspheres are used as a lightweight filler. The properties of the products were studied according to standard methods. The experiment was conducted using statistical and numerical methods. **Results.** A method for predicting the properties of fired products has been developed, and formulation and process problems have been solved based on its application. It has been established that for a product with the highest compressive strength, the most suitable composition would be one containing 34% liquid glass, and the molding pressure should be 4.6 MPa. In this case, the compressive strength will be 16.2 ± 0.6 MPa; the average density is 711 ± 38 kg/m³ and the thermal conductivity is 0.0114 ± 0.0006 W/(m°C). The decrease in compressive strength after 75 freeze-thaw cycles (compared to the strength of dry samples) is 8–11%, and after 100 cycles – 11–14%. **Discussion.** The formation of the properties and structure of the material is based on the processes of uniform distribution of the binder over the surface of the granules with the pressing of the system and crystallization of the binder during the firing process with the formation of strong contact between the granules.

Conclusion. Firing materials based on microspheres have a more developed surface of a single pore space, which affects their water absorption, and, consequently, strength, thermal conductivity and operational durability.

KEYWORDS: liquid glass, aluminosilicate microspheres, compression molding, firing, statistical planning, energy efficiency

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Оптимизация состава и свойств легких материалов на основе жидкого стекла

Игорь Вячеславович Бессонов¹ , Алексей Дмитриевич Жуков^{1,2} , Андрей Александрович Медведев^{2,3} , Элина Александровна Малова^{1,2*} , Илья Сергеевич Говряков¹ 

¹ Научно-исследовательский институт строительной физики Российской академии архитектуры и строительных наук, 127238, Москва, Локомотивный проезд, 21, Российская Федерация

² Национальный исследовательский Московский государственный строительный университет, 129337, Москва, Ярославское шоссе, 26, Российская Федерация

³ Российский государственный геологоразведочный университет имени Серго Орджоникидзе, 117485, Москва, ул. Миклухо-Маклая, 23, Российская Федерация

* Автор, ответственный за переписку: e-mail: eg15082000@mail.ru

АННОТАЦИЯ

Введение. Применение алюмосиликатных микросфер делает возможным получение легких обжиговых материалов повышенной прочности. Использование в качестве связующего жидкого стекла позволяет получать изделия при температуре обжига 400 °С, что значительно снижает энергозатраты по сравнению с керамическими материалами. Цель исследований, изложенных в статье, заключалась в разработке и апробации методики оценки свойств обжиговых изделий на основе жидкого стекла и силикатных микросфер, а также выбора оптимальных рецептурных и технологических параметров. **Методы и материалы.** В качестве связующего используется жидкое стекло плотностью 1400 кг/м³, а в качестве легкого заполнителя – алюмосиликатные микросферы. Исследование свойств изделий осуществлялось по нормативным методикам. Проведение эксперимента осуществлялось с использованием статистических и цифровых методов. **Результаты.** Разработана методика прогнозирования свойств обжиговых изделий, и на основании ее применения решены рецептурные и технологические задачи. Установлено, что если нужно получить изделие с наибольшей прочностью при сжатии, то наиболее приемлемым будет состав, содержащий 34% жидкого стекла, а давление формования должно составлять 4,6 МПа. В этом случае прочность на сжатие составит 16,2 ± 0,6 МПа; средняя плотность 711 ± 38 кг/м³ и теплопроводность 0,0114 ± 0,0006 Вт/(м·°С). Снижение прочности на сжатие при 75 циклах замораживания и оттаивания (по сравнению с прочностью сухих образцов) составляет 8–11%, а при 100 циклах – 11–14%. **Обсуждение.** В основе формирования свойств и структуры материала лежат процессы: равномерного распределения, вяжущего по поверхности гранул с подпрессовкой системы и кристаллизация вяжущего в процессе обжига с формированием прочных контактов между гранулами. **Заключение.** Обжиговые материалы на основе микросфер имеют более развитую поверхность единого порового пространства, что оказывает влияние на их водопоглощение, а следовательно, на прочность, теплопроводность и эксплуатационную стойкость.

КЛЮЧЕВЫЕ СЛОВА: жидкое стекло, алюмосиликатные микросферы, формование с прессованием, обжиг, статистическое планирование, энергетическая эффективность

ИСТОЧНИКИ ФИНАНСИРОВАНИЯ НАУЧНОЙ РАБОТЫ, РЕЗУЛЬТАТОМ КОТОРОЙ СТАЛА ПУБЛИКАЦИЯ: Исследования выполнены в рамках реализации НИР ФНИ Минстроя РФ «Развитие теоретических основ получения особо легких неорганических строительных материалов и исследование влияния пористой структуры на их теплофизические и акустические характеристики», а также в рамках реализации Программы развития НИУ МГСУ «ПРИОРИТЕТ 2030». Проект 3.1 «Научный прорыв в строительной отрасли – новые технологии, новые материалы, новые методы».

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INTRODUCTION

In enclosing structures, including self-supporting or load-bearing structures of buildings, a wide segment is occupied by lightweight materials such as cellular concrete, concrete on lightweight and extra-lightweight aggregates, hollow ceramic bricks and their analogues [1–3]. Products belong to the group of heat-insulating and structural ones having an average density from 400 to 1000 kg/m³

and compressive strength from 2.0 to 8 MPa. Products of this group can be manufactured on various mineral binders (modified gypsum, clay, Portland cement and its analogues, etc.). Stabilization of the structure of these materials can occur under conditions of natural hardening, heat or heat-moisture treatment, firing [4–6]. The materials of this group have relatively low thermal conductivity, which makes it possible to manufacture structures in moderate climate regions with minimal additional insulation, and in

regions with a warm climate – without additional insulation while maintaining a sufficient level of comfort in the premises [7–9]. Ceramic building products (including lightweight or light-weight ones) belong to the group of fired materials, in which the production of ceramic shards occurs at temperatures from 950–1100 °C (depending on the properties of the raw materials used and the type of modifying additives) [10–12]. This predetermines the high energy intensity of technological processes, which affects the energy efficiency of structures. Taking into account the current state of the economy, the main criteria for energy efficiency are the durability of structures, determined by the operational resistance of materials and the correctness of design solutions; ensuring comfortable conditions in the premises, that is, an optimal temperature and humidity regime; reducing the negative impact on the environment and fire safety, as well as reducing the energy costs of manufacturing products [13–15]. One of the ways to reduce these costs is to produce products with a lower firing temperature.

It should also be noted that the use of lightweight porous materials related to thermal insulation and structural materials allows the creation of single-layer structures possessing both sufficient strength characteristics and satisfactory thermal resistance [16–18]. The use of particularly lightweight fillers is promising: lightweight expanded clay, foam glass, mineral capsules and microspheres.

Microspheres are made from various materials; some of them are waste products of other industries. Aluminosilicate microspheres are hollow particles with a predominant diameter of 100–200 μm and a wall thickness of up to 10% of the diameter. The spheres are in close interaction with the substance of the mineral matrix and act as either a fine filler or a filler, as well as a structuring element [19–21]. From the point of view of the possibility of obtaining lightweight materials with reduced energy intensity of manufacture, the use of liquid glass as a substance forming the matrix is promising [22–24]. The formation of porosity of the matrix can be achieved by gas or foam formation, as well as by introducing silicate microspheres selected according to fractional composition [25, 26].

A preliminary analysis of the data revealed that achieving optimal energy efficiency indicators is largely determined by manufacturing costs, as well as the material and labor intensity of producing structures with standard thermal resistance. Based on this analysis, the goal of the study was formulated: to develop and test a methodology for evaluating the properties of fired products based on liquid glass and silicate microspheres, as well as to select optimal formulation and process parameters.

METHODS AND MATERIALS

Liquid glass with a density of 1400 kg/m³ was used as a binder. Aluminosilicate microspheres were used as

a filler. The chemical composition of the microspheres is represented mainly by oxides of silicon, aluminum and iron: SiO₂ – 55–65%; Al₂O₃ – 25–33%; Fe₂O₃ – 1–6%. The bulk density of the microspheres is 360 kg/m³; compressive strength at 50% destruction level is 15.0 MPa, and thermal conductivity is 0.08–0.1 W/(m·°C); water absorption is low; sorption moisture is up to 0.3% by weight.

The technology for producing a building material based on liquid glass and microspheres includes the following operations: preparation of a homogeneous mixture of liquid glass with microspheres; molding under pressure; stripping, and firing. Laboratory samples are produced using molding with pressing. The pressure value is optimized during the experiment. The products are dried to a moisture content of no more than 2% and fired in a laboratory furnace according to the following regime: temperature increase to 100 °C for 15 min, holding at 100 °C for 10 min, temperature increase to 420 °C for 30 min, holding at 420 °C for 60 min. Furnace cooling is normal. The structure of the materials was studied using an electron microscope.

A preliminary calculation of the composition of the material based on liquid glass binder and aluminosilicate microspheres was performed using geometric analogies and the assumed packing of the spherical component. The maximum packing of the microspheres, considering their polydispersity and predominant size of 100–200 μm, was assumed to be 0.73–0.8. The ratio of void volume to microsphere volume was determined:

$$\frac{V_{\text{voids}}}{V_{\text{micro}}} = \frac{0.27}{0.73} \div \frac{0.2}{0.8} = 0.37 \div 0.25. \quad (1)$$

Two possible structure formation options were considered. In the first option (volumetric monolithization), the interspherical voids were filled entirely with liquid glass. The ratio of the liquid glass content to the mass of the microspheres was assumed to be 0.27. In the second option (contact monolithization), this ratio was assumed to be 0.20. Considering dependence (1) and the average density of the components (600 kg/m³ for microspheres and 1400 kg/m³ for liquid glass), the ratio between the masses of liquid glass and microspheres was determined as follows:

$$\frac{M_{\text{lg.}}}{M_{\text{micro}}} = \frac{C_{\text{lg.}} \cdot \rho_{\text{lg.}}}{C_{\text{micro}} \cdot \rho_{\text{micro}}}. \quad (2)$$

The development of a methodology for determining the rational composition of a material based on a liquid glass binder, as well as the prediction of its properties, was carried out using statistical planning methods and digital methods for processing experimental results [27–29]. The “binder-microspheres” system is two-component; the content of these components gives 100%; therefore, these factors are interdependent, and from the point of view of statistical planning, the following factors were adopted:

Table 1. Conditions of the two-factor experiment

Name of the factor	Symbol X_i	Average value of the factor, $\bar{X}_i$	Variation interval, ΔX_i	Factor values at levels	
				–1	+1
Liquid glass content, % by weight	X_1	30	6	24	36
Forming pressure, MPa	X_2	3.4	1.8	1.6	5.2

X_1 – the liquid glass content, % by weight, and X_2 – the pressing pressure. The average density (U_1) and compressive strength (U_2) were adopted as response functions.

The experimental conditions are presented in Table 1. A full quadratic matrix D-optimal design was adopted for the experiment. Each row of the design was repeated five times ($n = 5$). To reduce the likelihood of interference and unaccounted factors, a randomization method was used to randomize the sequence of experiments.

RESULTS AND DISCUSSION

Regression equations were obtained by processing experimental data using the Statistika program. The significance of the polynomial coefficients was established by comparison with confidence intervals: for compressive strength (Δb_2) – 0.2 MPa, for average density (Δb_1) – 3 kg/m³. The following regression equations were obtained, valid within the ranges of variation (domains) of the variable factors (Table 1):

– for medium density

$$Y_1 = 610 + 36X_1 + 68X_2 + 22X_1X_2 - 12X_2^2; \quad (3)$$

– for compressive strength

$$Y_2 = 11,8 + 2,2X_1 + 2,8X_2 + 0,8X_1X_2 - 0,4X_2^2. \quad (4)$$

It was established that, within the range of variable factors under consideration, the average thermal conductivity values of the material are a nearly linear function of the average density. However, deviations from the average values must be taken into account. It was assumed that the distribution of values is close to a unimodal Gaussian distribution, and the significant experimental error does not exceed 10%.

Graphical interpretation of the obtained dependencies consists of constructing, based on algebraic polynomials (3) and (4), graphs of the functions $Y_1 = f_1(X_1, X_2)$, $X_1 = f_1(X_2, Y_2)$ and combining them into a nomogram (Fig. 2). The nomogram consists of three sectors: in the first (I) sector, an assessment is made based on average density; in the second (II) – on compressive strength; in the third (III) – on thermal conductivity.

The direct problem of digital modeling is to predict the properties of products depending on the formula-

tion and process parameters; the inverse problem is to determine the values of the formulation and process parameters depending on the specified product characteristics. An example of solving the direct problem is shown in Fig. 1 with blue lines. We set the liquid glass consumption to 31% and the molding pressure to 3.0 MPa. In sector I, we mark the point 31% on the ordinate axis and draw a straight line through sectors I and II parallel to the abscissa axis. In sector II, at the intersection with the pressure line ($P = 3.0$ MPa), we obtain point “a”, from which we draw a straight line parallel to the ordinate axis in sectors I and III. At the intersection with the thermal conductivity graph, we obtain point “b”, from which we drop a perpendicular to the thermal conductivity axis and obtain a thermal conductivity value of 0.095 W/(m°C). We extend the straight line to the average density axis and obtain the average density value equal to 554 ± 28 kg/m³. In sector II, from point $P = 3.0$ MPa, we draw a straight line parallel to the ordinate axis until it intersects with the line (ac) and obtain the compressive strength value equal to 10.0 ± 0.5 MPa. We obtain that with a liquid glass content of 31% by weight and a molding pressure of 3.0 MPa, the compressive strength of the product will be 10.0 ± 0.5 MPa, the average density is 600 ± 20 kg/m³, and the thermal conductivity is 0.095 ± 0.005 W/(m°C).

The inverse problem of digital modeling is solved by verifying the obtained results in control series of the experiment (Table 2). The average density of the product is used as the evaluation parameter. The experimental conditions are presented in columns 2 and 3. The results of determining and calculating the average density of the samples are presented in columns 4–6; the results of determining and calculating the compressive strength of the samples are presented in columns 7 and 8.

The average experimental error for the average density was 2.6%, which is less than that found using statistical methods (5%). The calculated strength values can be determined either from the nomogram (Fig. 1) or using the algebraic polynomial (4); the calculated thermal conductivity value is determined from the third sector of the nomogram.

If it is necessary to obtain a product with the highest compressive strength, then composition No. 1 will be the most acceptable. In this case, the compressive strength will be 16.2 ± 0.6 MPa; the average density will be $711 \pm$

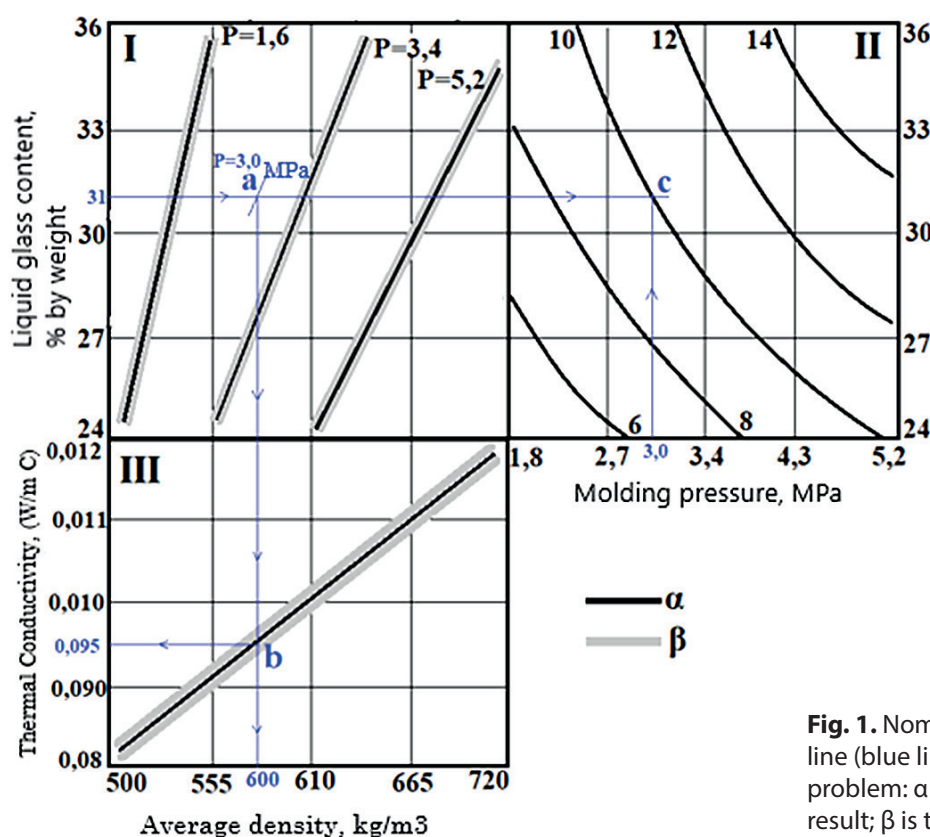


Fig. 1. Nomogram for solving a straight line (blue lines) of a digital modeling problem: α is the average value of the result; β is the deviation from the average value.

Table 2. Results of the control experiment

No.	Experimental conditions		Average density, kg/m ³		Error Δ , %	Compressive strength, MPa	
	Liquid glass content, % by weight	Forming pressure, MPa	Calculated	Experimental		Calculated	Experimental
1	2	3	4	5	6	7	8
1	34	4.6	711	729	2.5	16.6	16.2
2	30	4.6	665	651	2.1	14.4	14.1
3	26	4.6	611	625	2.2	11.4	11.4
4	34	3.4	642	622	3.1	13.6	13.9
5	30	3.4	610	622	1.9	11.8	11.2
6	26	3.4	569	583	2.4	9.8	10.7
7	34	2.2	541	561	3.6	10.4	10.9
8	30	2.2	539	557	3.2	9.0	9.9
9	26	2.2	525	538	2.4	7.6	8.6
			Average Δ , %		2.6		

38 kg/m³ and the thermal conductivity will be 0.0114 ± 0.0006 W/(m°C). If it is necessary to obtain a product with the lowest thermal conductivity, then composition No. 9 will be chosen. In this case, the compressive strength will be 8.6 ± 0.2 MPa; the average density will be 538 ± 26 kg/m³ and the thermal conductivity will be

0.0086 ± 0.0003 W/(m°C). The ratio of the masses of the components (formula 2) for composition No. 1 will be 1.37; and for composition No. 2 – 0.94.

Firing materials based on a liquid glass binder and aluminosilicate microspheres exceed ceramic products with comparable density by more than 2.5 times, while

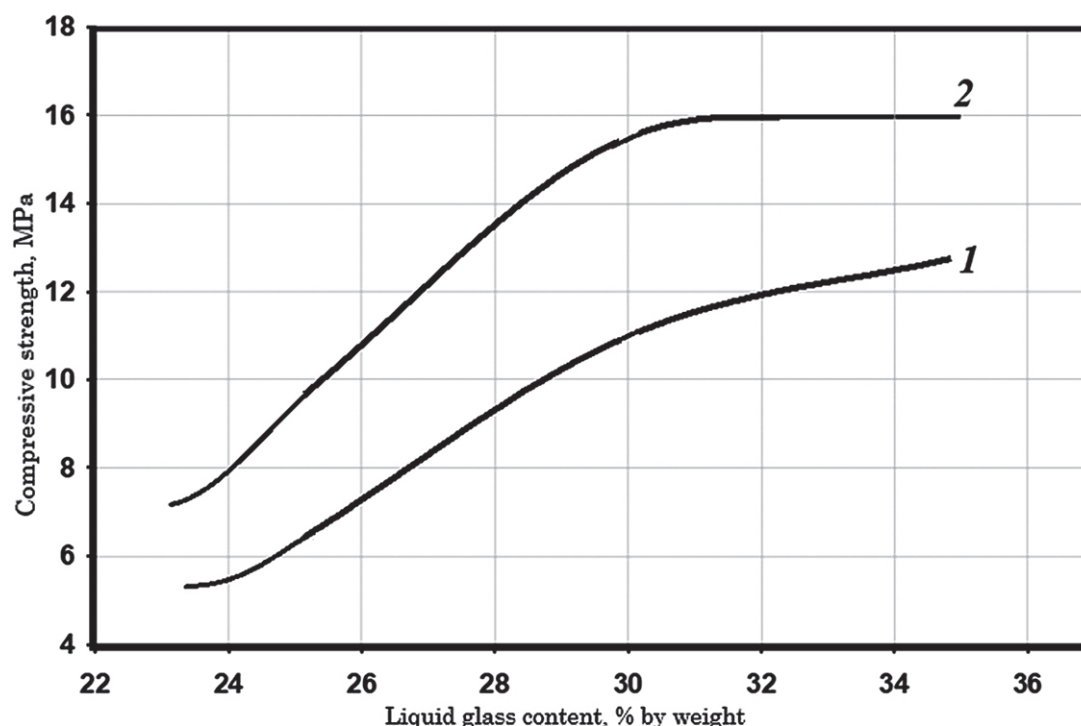


Fig. 2. Compressive strength as a function of binder content at molding pressure: 1 – 3.2 MPa; 2 – 5.0 MPa

the firing temperature decreases from 1050 °C (for wall ceramics) to 400 °C. The change in strength upon wetting, as well as the frost resistance characteristics of these materials, are important for thermal insulation and structural materials. The compressive strength of dry specimens is determined using nomogram data and control series of experiments; the compressive strength of water-saturated specimens decreases by 4–8%. The reduction in compressive strength after 75 freeze-thaw cycles (compared to the strength of dry specimens) is 8–11%, and after 100 cycles, it is 11–14%. The results are quite satisfactory, and a reduction in the loss of strength can be achieved by optimizing the firing conditions of the material.

Depending on the component ratio and molding conditions, the characteristics of products based on liquid glass and aluminosilicate microspheres can vary significantly. The impact of these varying factors on the results can be assessed by analyzing regression equations (3 and 4).

Increasing the liquid glass content of the material and increasing the pressure during sample formation under constant firing conditions predictably contribute to an increase in the strength of the products and an increase in their average density, as confirmed by the coefficients for X_1 and X_2 . The influence of factor X_2 is more pronounced at pressing pressures below average values. As pressing pressures increase above average values, the increase in strength begins to manifest itself to a lesser extent. This is due to changes in the structural characteris-

tics of the molded mixture and the presence of pore space forming between the cells. At low binder consumption rates, molding with pre-pressing compacts the mixture structure and minimizes the free (air-filled) volume of interspherical porosity. As the pressure increases, surface collapse of the microspheres begins, and it can be assumed that at pressures greater than the established variation range, microsphere destruction and a decrease in material strength may occur.

The significant coefficient for X_1X_2 indicates a positive influence of pairwise interaction, suggesting a synergistic effect from the combined effect of liquid glass flow rate and pressing pressure. This effect may be due to adhesion conditions at the granule-mineral matrix contact surfaces.

A relationship was established between the binder content and molding pressure (Fig. 2). At a molding pressure of 5 MPa, a clearly defined plateau is observed. Increasing the binder content above 31% does not increase the compressive strength of the samples. At lower molding pressures, the strength does not stabilize with an increase in the liquid glass dosage, but its increase becomes more gradual. While in the binder content ranges from 24 to 31%, the strength increase rate is 5.4–5.5 MPa/%, then in the range from 31 to 35%, it is 0.4–0.5 MPa/%. Reducing the binder content to less than 31% leads to a decrease in strength. The obtained results correlate well with the results of the statistical analysis and indicate that optimal stability of the “cured liquid glass-aluminosilicate microspheres” system has been achieved.

The structure of the fired product is characterized by a number of features manifested at both the macro- and micro-levels. At the macro-level, the material density in the considered area of factor determination is independent of the amount of binder. This is due to the absence of microfiller grain separation, which can be assessed at the micro-level (Fig. 3). The thickness of the newly formed layers between the aluminosilicate microspheres (microfiller particles) is minimal. This thickness does not impede the formation of a contact structure of microfiller bonding, meaning contact monolithic bonding of the microspheres occurs. Controlling intergranular voids helps optimize the material's structure and, consequently, its properties. This control is achieved both by varying

the binder content and by the pressure generated during molding. Initially, the binder is distributed in thin films across the surface of the microspheres, filling the surface relief of these particles. This can lead to a slight increase in mixture density due to contraction, which is driven by capillary forces. Subsequently, increasing the binder content fills the intercellular voids. Applying external pressure promotes the convergence of the microfiller particles and reduces porosity. The limiting factor for increasing the compaction value is the strength of the microsphere surface layer and the strength of the microspheres themselves, whose destruction is undesirable. The structure of the cured “cured binder-microspheres” system is the basis for the properties of the products. The strength of

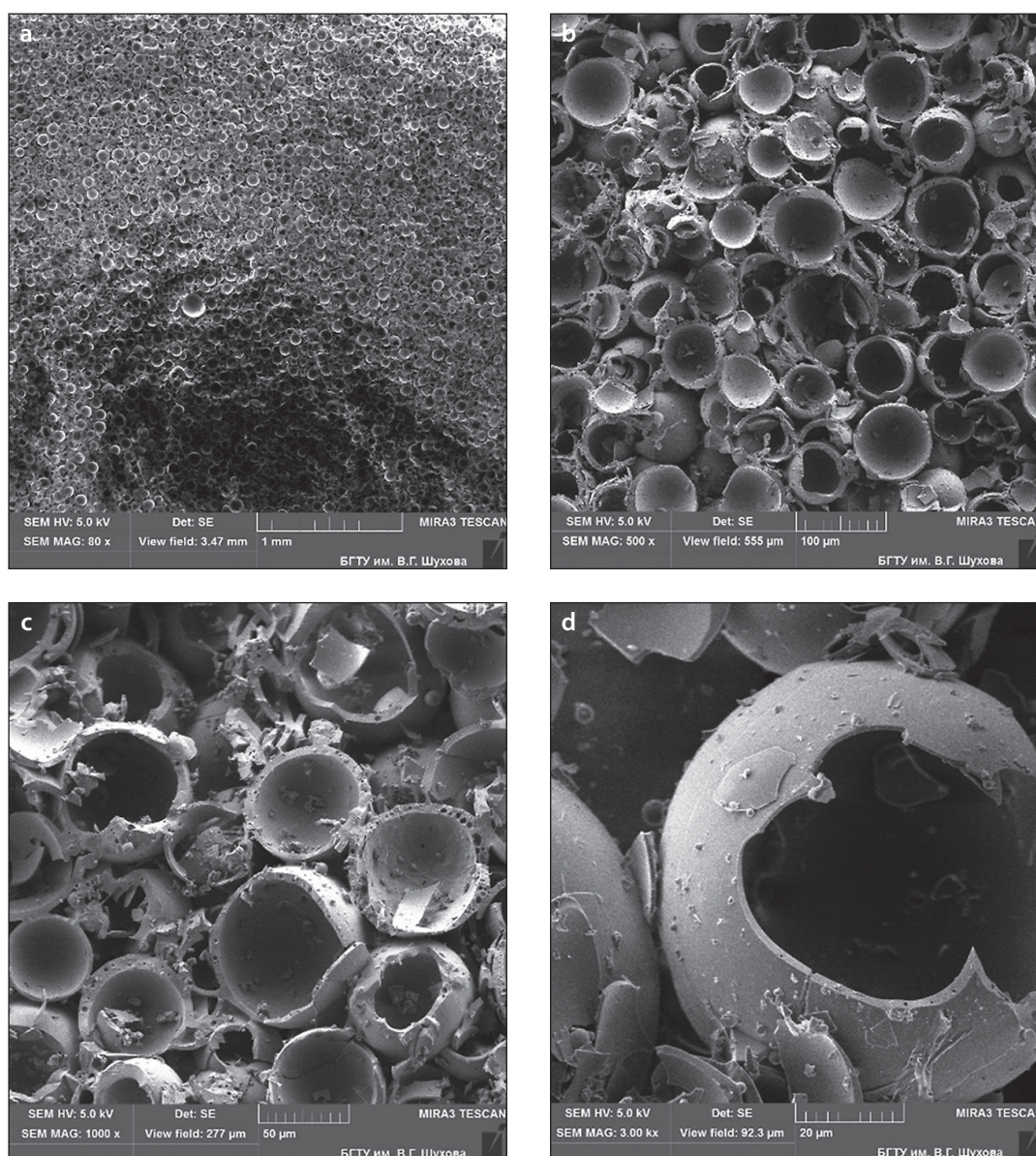


Fig. 3. Microstructure of materials based on microspheres and liquid glass at magnification from 80x to 3000x: a – 80x; b – 500x; c – 1000x; d – 3000x

the products is determined by the strength of the system's components and the strength of the adhesive bond. The newly formed particles from the cured binder are well crystallized and concentrated in the area of contact between the filler particles. Due to the high adhesion, the microsphere shell becomes the weak point in the system, where failure occurs.

The density, thermal conductivity, water absorption, and frost resistance of the material are determined by the structure of its pore space. The pore structure of the material consists of intergranular voids and pore volume hermetically sealed within the microspheres. The porosity of the matrix (hardened by firing liquid glass) is insignificant, and its value does not exceed the statistical error. These factors determine the relatively low density and thermal conductivity of the material. The water absorption of the material is due to the presence of open cells in the interpore space. It is worth noting that moisture penetration into the material increases its thermal conductivity.

The frost resistance of the products is quite high, due to the material's damping properties during freezing. Moisture does not penetrate the microspheres, which are unaffected by frost, but partially fills the macropores. The volume of microspheres in the material is approximately 70–80%, and frost damage occurs only in the binder and the contact surfaces between the binder and the microspheres. Consequently, the overall loss of strength due to frost is insignificant.

Fired products based on liquid glass and aluminosilicate microspheres can be used as structural thermal insulation materials for walls. In regions with moderate and warm climates, additional thermal insulation may not be required, as may multilayer structures. This will allow

for optimization of the construction process in terms of material consumption, labor intensity, and cost. These factors, along with the relatively low firing temperature, also contribute to increased energy efficiency in the structures used.

CONCLUSION

Depending on the component ratio and molding conditions, the characteristics of products based on liquid glass and aluminosilicate microspheres can vary significantly. Increasing the liquid glass content in the material and increasing the molding pressure under constant firing conditions predictably contribute to increased product strength. The frost resistance of the products is explained by the material's damping properties during freezing. The reduction in compressive strength after 75 freeze-thaw cycles (compared to the strength of dry samples) is 8–11%, and after 100 cycles, it is 11–14%. The results are quite satisfactory, and a reduction in strength loss can be achieved by optimizing the material's firing conditions.

Firing materials based on a liquid glass binder and aluminosilicate microspheres exceed comparable ceramic compositions by more than 2.5 times in density. In regions with moderate and warm climates, additional thermal insulation may not be required, nor may multilayer structures. This will allow for optimization of the construction process in terms of material consumption, labor intensity, and cost. It should be noted that for these regions, the decisive factors are comfort: indoor temperature and humidity, and the temperature difference between the interior surface of the enclosing structure and the room.

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ADDITIONAL INFORMATION

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INFORMATION ABOUT THE AUTHORS

Igor V. Bessonov – Can.Sci. (Tech.), Chief Researcher at the Research Institute of Building Physics Russian Academy of Architecture and Construction Sciences, 21, Lokomotivny Proyezd, Moscow, 127238, Russian Federation, Advisor to the Russian Academy of Architecture and Building Sciences, Novy Arbat St., 19, Moscow, 119019, Russian Federation. bessonoviv@mail.ru, <https://orcid.org/0000-0002-9234-4075>

Alexey D. Zhukov – Can.Sci. (Tech.), Associate Professor, Department of Building Materials Science at the National Research Moscow State University of Civil Engineering, 26, Yaroslavskoye Shosse, Moscow, 129337, Russian Federation, Senior Researcher at the Research Institute of Building Physics Russian Academy of Architecture and Construction Sciences, 21, Lokomotivny Proyezd, Moscow, 127238, Russian Federation, Corresponding Member of the Russian Academy of Engineering, Building 4, 9, Gazetny Lane, Moscow, 125009, Russian Federation. lj211@yandex.ru, <https://orcid.org/0000-0003-0593-3259>

Andrey A. Medvedev – Can.Sci. (Tech.), Associate Professor, Department of Higher Mathematics at the National Research University of Pre-University Moscow State University of Civil Engineering (NRU MSUCE), 26, Yaroslavskoye Shosse, Moscow, 129337, Russian Federation, Associate Professor of the Department of Geophysics at the Russian State Geological Prospecting University named after Sergo Ordzhonikidze, (RSGPU), 23, Ulitsa Miklukho-Maklaya, Moscow, 117485, Russian Federation. medvedev747@yandex.ru, <https://orcid.org/0000-0002-2930-2606>

Elina A. Malova – Postgraduate Student, Department of Building Materials Science at the National Research Moscow State University of Civil Engineering 26, Yaroslavskoye Shosse, Moscow, 129337, Russian Federation, Senior Engineer at the Research Institute of Building Physics Russian Academy of Architecture and Construction Sciences, 21, Lokomotivny Proyezd, Moscow, 127238, Russian Federation. eg15082000@mail.ru, <https://orcid.org/0000-0002-7241-4136>

Ilya S. Govryakov – Senior Engineer at the Research Institute of Building Physics Russian Academy of Architecture and Construction Sciences, 21, Lokomotivny Proyezd, Moscow, 127238, Russian Federation. govr190@mail.ru, <https://orcid.org/0000-0001-8327-9980>

CONTRIBUTION OF THE AUTHORS

Igor V. Bessonov – development of methodology and general concept of research.

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Elina A. Malova – conducting experimental studies and interim conclusions.

Ilya S. Govryakov – experimental results processing and conclusion.

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