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DEVELOPMENT AND INVESTIGATION OF THE PROPERTIES OF LOW-TEMPERATURE FUSIBLE ENAMEL COMPOSITIONS BASED ON THE “KHIVA QUARTZ SAND – SULTAN UVAYS FELDSPAR – LEAD-CONTAINING WASTE” SYSTEM

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Abstract. *This article presents the results of research on the development of low-temperature fusible glass enamel compositions based on Khiva quartz sand, Sultan Uvays feldspar, and lead-containing industrial waste, as well as the investigation of their physicochemical and technological properties. Based on experimental studies, GEF-1, GEF-2, and GEF-3 compositions were developed, and their coefficient of linear thermal expansion, spreading ability, adhesion strength, density, and softening temperature were determined. The results showed that the GEF-3 composition fully complies with the requirements of GOST 24405-80.*

Keywords: *glass enamel, frit, quartz sand, feldspar, lead-containing waste, coefficient of thermal expansion, physicochemical properties, low-temperature enamel.*

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РАЗРАБОТКА И ИССЛЕДОВАНИЕ СВОЙСТВ ЛЕГКОПЛАВКИХ ЭМАЛЕВЫХ КОМПОЗИЦИЙ НА ОСНОВЕ СИСТЕМЫ «ХИВИНСКИЙ КВАРЦЕВЫЙ ПЕСОК – ПОЛЕВОЙ ШПАТ СУЛТАН-УВАЙС – СВИНЕЦСОДЕРЖАЩИЕ ОТХОДЫ»

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Аннотация. *В данной статье представлены результаты исследований по разработке легкоплавких стеклоэмалевых композиций на основе кварцевого песка Хивинского месторождения, полевого шпата Султан-Увайс и свинецсодержащих промышленных отходов, а также изучению их физико-химических и технологических свойств. На основе экспериментальных исследований были разработаны составы GEF-1, GEF-2 и GEF-3, определены коэффициент линейного термического расширения, растекаемость, прочность*

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сцепления, плотность и температура размягчения. Установлено, что состав GEF-3 полностью соответствует требованиям ГОСТ 24405-80.

Ключевые слова: *стеклоэмаль, фритта, кварцевый песок, полевой шпат, свинецсодержащие отходы, коэффициент термического расширения, физико-химические свойства, легкоплавкая эмаль.*

"XIVA KVARS QUMI – SULTON UVAYS DALA SHPATI – QO'RG'OSHINLI CHIQINDI" TIZIMI ASOSIDA PAST HARORATDA SUYUQLANUVCHI EMAL KOMPOZITSIYALARINI ISHLAB CHIQISH VA XOSSALARINI TADQIQ ETISH

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Annotatsiya. *Mazkur maqolada Xiva koni kvars qumi, Sulton Uvays dala shpati va qo'rg'oshin saqlovchi sanoat chiqindilari asosida past haroratda suyuqlanuvchi shisha emal kompozitsiyalarini ishlab chiqish va ularning fizik-kimyoviy hamda texnologik xossalarini tadqiq etish natijalari keltirilgan. Tajribalar asosida GEF-1, GEF-2 va GEF-3 tarkiblari ishlab chiqilib, ularning termik kengayish koeffitsienti, yoyilish darajasi, yopishish mustahkamligi, zichligi va yumshash harorati aniqlangan. Tadqiqot natijalariga ko'ra, GEF-3 tarkibi GOST 24405-80 talablariga to'liq javob berishi aniqlangan.*

Kalit so'zlar: *shisha emal, fritta, kvars qumi, dala shpati, qo'rg'oshinli chiqindi, termik kengayish koeffitsienti, fizik-kimyoviy xossalar, past haroratli emal.*

Introduction. The glass products manufacturing industry is one of the key sectors of the national economy worldwide. It consumes substantial amounts of raw materials, energy, and labor resources, which in turn determines the development level of the economy's core branches. Accordingly, the efficiency of the glass industry depends directly on the rational and economical use of these resources.

Rapid urban and industrial development has made metal structures among the most widely used materials in construction. Among these, enameled metal products based on iron are of particular importance. As petroleum prices continue to rise, the cost of corrosion-resistant materials derived from it also increases. Glass enamels have thus gained wide adoption as an alternative corrosion-resistant coating. Enameled metal surfaces offer advantages such as smoothness and durability, making them applicable not only in the construction sector but also in the food industry, pharmaceuticals, and other branches of the national economy.

Glass enamel samples were studied in accordance with the technical requirements of GOST R 52569-2018 (Frits. Technical Specifications) [1]. This research aims to make effective use of locally available raw material resources in Uzbekistan and to substitute imported products with domestically manufactured alternatives. In earlier studies [2–10], the authors investigated compositions of glass enamels for glass surfaces using Khiva deposit feldspathic quartz sands [11], Sultan Uvays deposit feldspar [12], lead-containing waste from zinc processing, and other local raw materials [13]. The influence of siliceous raw materials and alkaline earth metal oxides from the Khorezm region was examined in the course of these investigations.

Research Methods. The coefficient of linear thermal expansion (CLTE) of the glass enamel samples was determined by calculation, based on the methods specified in GOST R 52569-2018 and GOST 24405-80. The CLTE was calculated using the following formula:

$$\alpha = \frac{\sum \alpha_i \times m_i}{100}; \quad (1)$$

where α_i is the coefficient of linear thermal expansion of the oxide or component in the frit composition over the temperature range of 20–400 °C (°C⁻¹); m_i is the molar fraction of each component (%).

Surface spreading of the samples was evaluated by selecting a standard enamel frit reference sample. Frit materials were placed on metal plates that had been washed with distilled water and dried, and the spreading lengths were measured before and after holding at 860 ± 5 °C for 15 minutes. The spreading value was calculated using the formula:

$$L = \frac{l_u}{l_{CO} \times l_{\alpha'}}; \quad (2)$$

where l_u – is the spreading length of the test frit sample (mm); l_{CO} – is the spreading length of the standard reference sample (mm); $l_{\alpha'}$ – is the pre-determined spreading length of the standard reference sample (mm).

Chemical resistance of the samples to atmospheric conditions was assessed visually by monitoring changes upon exposure to drops of 10% citric acid solution. Surface microstructure was studied using a EM-5 electron microscope with the carbon plate technique. Microhardness was measured with a PMT-3 instrument at loads of 0.196 and 0.49 N [14].

Chemical Composition of Raw Materials. For developing the experimental glass frit compositions, Khiva deposit feldspathic quartz sand (HFQS), Sultan Uvays deposit feldspar (SUF), and lead waste (LW) were selected as primary raw material components (Table 1). Boron, titanium, and nickel oxides were added in chemically pure reagent form.

Table 1.

Chemical composition of raw materials used for producing enamel coatings on glass surfaces

Sample name	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	PbO	ZnO	SO_3	LOI, wt.%
HFQS	97.04	1.25	0.10	0.21	0.10	0.13	0.40	–	–	–	0.77
SUF	74.08	13.64	0.05	0.55	0.30	9.01	1.56	–	–	–	0.60
LW	2.40	0.68	3.58	1.93	0.49	3.77	36.10	6.10	18.3	26.65	–

Development of Batch Compositions and Sample Preparation. Based on a review of the relevant literature, low-melting glass enamel compositions in the “quartz sand – feldspar – industrial waste” system were investigated. As a reference, the chemical composition (wt.%) prescribed by GOST 24405-80 for grade “ESG-31” (borate type) was adopted: SiO_2 – 45-52; Al_2O_3 – 5-7; Fe_2O_3– <3; CaO – <7; $Na_2O + K_2O$ – 16-20; B_2O_3 –13-18; TiO_2– <5; NiO – 0.5-3; CoO – 0.2-0.8.

Several compositions were prepared, and following comparative evaluation, three optimal batch compositions GEF-1, GEF-2, and GEF-3 were selected (Table 2).

Table 2.

Batch compositions prepared for producing glass enamel from the studied raw materials (wt.%)

No.	Raw material	GEF-1	GEF-2	GEF-3
1	Khiva quartz sand	31.00	28.00	25.00
2	Sultan Uvays feldspar	29.00	29.00	29.00
3	Lead waste	6.00	8.00	10.00
4	Calcined soda	20.00	20.00	20.00
5	Boron oxide	14.00	15.00	16.00
Total (Σ, wt.%)		100	100	100

Na_2O was introduced into the glass batch through calcined soda. B_2O_3, TiO_2, and NiO were introduced as chemically pure reagents. TiO_2 and NiO were added at 5 wt.% and 2 wt.%, respectively, relative to the total mass. The resulting chemical composition of the developed enamel batches for glass surfaces is presented in Table 3.

Table 3.

Chemical composition of the developed enamel batch compositions for glass surfaces

Sample	SiO_2	Al_2O_3	K_2O	Na_2O	CaO	MgO	Fe_2O_3	ZnO	PbO	B_2O_3	SO_3
GEF-1	51.74	4.45	2.70	12.11	0.37	0.17	0.27	0.37	2.17	13.72	1.10
GEF-2	48.88	4.43	2.69	12.18	0.41	0.18	0.34	0.49	2.89	14.70	1.46
GEF-3	46.02	4.41	2.69	12.24	0.44	0.19	0.41	0.61	3.61	15.68	1.83

Sample Preparation and Test Results. Each component was weighed out at 1 kg and glass enamel batch compositions were prepared according to the specified formulations. Samples were fused in unglazed corundum crucibles at 1200°C with a 45-minute hold. The resulting glass melt was first quenched in a water bath at room temperature, then allowed to cool freely for 12 hours. The visual characteristics of the glass enamel samples are presented in Table 4 and Figure 1.

Table 4.

Visual characteristics of the obtained glass enamel samples

Visual characteristics	GEF-1	GEF-2	GEF-3
Degree of fritting	Moderate	Good	High
Number of bubbles in fused glass, pcs. (per 1 cm^2)	7	4	None
Number of undissolved inclusions, pcs. (per 1 cm^2)	5	3	None

Visual inspection revealed that the sample produced from the GEF-3 experimental composition outperformed the others in terms of fritting degree and visual quality — no undissolved inclusions or bubbles were observed per 1 cm^2 of surface area (Figure 1).

To address the defects observed in GEF-1 and

GEF-2, additional samples were prepared and compared at temperatures above 1200 °C and with firing durations ranging from 45 to 60 minutes. Based on the results, firing at 1200 °C for 45 minutes was identified as the optimal condition.

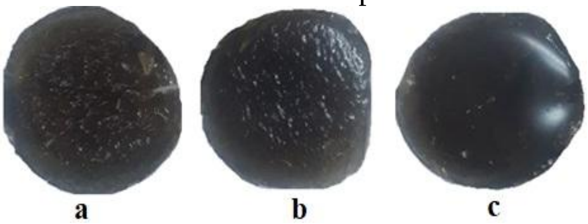

Fig.1. Visual appearance of glass enamel frit samples

a – GEF-1; b – GEF-2; c – GEF-3

Technological and Physical Properties. The technological properties of the glass enamel frit samples for glass surfaces were determined in accordance with GOST 24405-80, and the results are summarized in Table 5.

Table 5.

Technological properties of the obtained samples

No.	Technological property	GOST 24405-80	GEF-1	GEF-2	GEF-3
1	CLTE, $\alpha \times 10^{-7}$, $°C^{-1}$	95–105	91.5	94.2	97.4
2	Spreading on surface, mm	30–40	31	33	36
3	Adhesion strength, score	–	4	4	5

Analysis of the table shows that GEF-3 best satisfies the requirements of GOST 24405-80: CLTE of $97.4·10^{-7}$ $°C^{-1}$, spreading of 36 mm, and an adhesion strength score of 5.

The key physical properties of the glass enamel frit samples were also determined, and the results are given in Table 6.

Table 6.

Physical properties of the experimental glass enamel frit samples

Physical property	GEF-1	GEF-2	GEF-3
Density, g/cm³	2.91	2.94	2.98
Refractive index	1.62	1.66	1.71
Softening temperature, °C	601	579	552
Mass loss in chemical medium, wt.%	0.26	0.27	0.27

Conclusions. Based on the results of the study, the following conclusions were drawn:

1. Three glass enamel frit compositions (GEF-1, GEF-2, GEF-3) were developed within the “Khiva quartz sand - Sultan Uvays feldspar - lead waste” system.

2. The GEF-3 composition (Khiva quartz sand 25 wt.%, Sultan Uvays feldspar 29 wt.%, lead waste 10 wt.%, calcined soda 20 wt.%, boron oxide 16 wt.%) fully complies with the requirements of GOST 24405-80.

3. No visual defects (bubbles or undissolved inclusions) were detected in GEF-3. The sample exhibited a CLTE of $97.4·10^{-7}$ $°C^{-1}$, spreading of 36 mm, density of 2.98 g/cm^3, and a softening temperature of 552 °C.

4. The optimal firing condition was determined to be 1200 °C with a 45-minute hold.

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