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Investigation of influence alumina-containing waste on the boiling process and the properties of soil glass-enamel

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Abstract. In the article by using the physic-chemical, electron microscopy and infrared spectroscopic methods the alternation of phase separation and quasi-homogeneous structure in the microstructure of glass enamel, depending on the content of alumina-containing waste has been studied. It has been also detected the appearance of a small peak on the leaching curve in the vitreous containing 2.5 wt. % of alumina-containing waste. When you increase the amount of waste by adding from 5.0 to 7.5 wt. %, this rate is decreased. Containing 10% of alumina-containing waste of GChC Shurtan no significant change of physic-chemical properties observed in the condition of glass enamel.

Keywords. glass enamel, alumina-containing waste, chemical resistance, liquation structures, exothermic effect, damping of enamels, microstructure.

Introduction.

Specialists involved in the development of glass enamel materials technology are increasingly attracted to alumina-containing glasses, which are characterized by high chemical resistance. In the scientific and technical literature, there is information about the composition of glasses containing up to 20% alumina [1, 2]. When alumina is introduced into alkali-silicate glass, the degree of cohesion of the structural network increases, as it is embedded in the silicon-oxygen framework and changes the structural role of the alkaline component.

The formation of aluminum ions in the structure of glass in sixfold coordination is most likely in alkali-free and low-alkaline compositions with a pronounced acid character. Small-radius alkaline earth cations are most conducive to the stabilization of the sixfold coordination of aluminum in glass. In the literature, data have been published according to which, depending on the chemical LOVcomposition, in glasses of the R_2O-RO-B_2O_3-Al_2O_3-SiO_2 system, there are two regions of liquation [3]. It is known that Al_2O_3 can contribute to the damping of titanium-calcium enamels, provided that the TiO_2 content is in the range of 4-5% [4].

Research methodology

In this work, the DTA method was used to clarify the endothermic and exothermic effects arising from the presence of inorganic and organic components in the composition of raw materials, as well as possible modification phase changes.

DTA was carried out on a Q-1500D derivatograph according to the system of F. Paulik, W. Paulik, L. Erdey. The heating rate was 20 deg / min. The interpretation of the thermogram was carried out on the basis of the data given in [5].

The infrared absorption spectra of the samples were recorded on an AVATAR-360 spectrometer (Nicolet) in the transmission range 400□4000 cm □1. KB2 tablets were used as a disk.

Spectra, EPR of some compositions of glass enamels were obtained for the same sample weights on a JES-FA200 spectrometer made in Japan (at RTU, Latvia) at a field stability of 1x10-6, a generation frequency from 8.8 to 9.6 GHz, a maximum power of 1 kW, a band range pulses from 10 to 100 s (in order to assess changes in the intensity of the EPR signal containing lines with spectroscopic splitting factors g).

Determination of the chemical stability of glass enamel in water, acid and alkali was determined by the simplified German method (DIN No. 12111) [6] and according to GOST 22291-83 [7].

Results and discussion

This article presents the results of studying the effect of alumina-containing waste on the cooking process and the properties of ground glass enamel. Alumina-containing waste - spent catalyst is formed during polymerization due to adsorption of titanium and vanadium salts by an aluminum-containing catalyst, absorption of volatile components of HCl, CH3COOH and some other compounds. In appearance, granules of alumina-containing waste (aluminum oxide) have the shape of a cylinder or a ball and are not flammable. The chemical composition of the spent catalyst includes (wt%): Al2O3□75.00; Na_2O - 1.40; Fe2O3-0.16; FeO-0.14; TiO2-0.14; CaO 0.90; MgO 0.48; pp-19.7 and a small amount of other impurities.

The composition containing the following ingredients (wt.%) Was taken as the initial glass: SiO_2 – 47.0; B_2O_3 □ 15.0; Na_2O □ 15.0; K_2O □ 2.7; Fe_2O_3 □4.0; CaF_2 – 4.0; $P2O_5$□ 1.5; MgO □ 1.5; CaO □ 1.5; CoO □ 0.6; NiO □ 1.7. In this case, the compositions of the test glasses were changed so that the total amount of Na_2O + B_2O_3 remained constant, equal to 30.0%, and the amount of Al_2O_3 varied from 2.5 to 10.0% with an interval of 2.5%. The contents of the other components of the original glass also remained unchanged (Table 1).

Table 1. Composition of test glasses with alumina-containing component

Compositions	Relation		Components, wt. %		
	Na_2O: B_2O_3	(Na_2O+B_2O_3): Al_2O_3	Na_2O	Al_2O_3	B_2O_3
AG - 1	1:1	12:1	15.00	2.50	15.00
AG □ 2	1:1	6:1	15.00	5.00	15.00
AG □ 3	1:1	4:1	15.00	7.50	15.00
AG □ 4	1:1	3:1	15.00	10.00	15.00

The melting of test glasses and samples was carried out according to the traditional methodology generally accepted in glass-making technology. At the same time, it was noted that the compositions of glasses with a high content of aluminum oxide were obtained more refractory, although the

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quality of the glass fiber had no signs of non-penetration.

Alumina-containing waste causes a sharp change in the microstructure of the glass: instead of the liquation structure, a quasi-homogeneous structure appears. With an increase in alumina content, a new type of liquation structure appears, which noticeably weakens at the ratio Al2O3> (B2O3 + Na2O). Delamination of enamel glass into very small droplets contributes to the uniform distribution of the titanium-containing phase in the glass structure and the growth of small crystals, which provide a high degree of damping and gloss of the coating, which is consistent with the data of works [3-8].

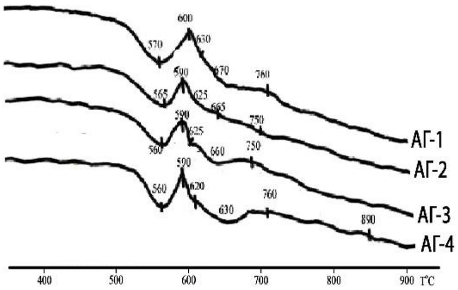
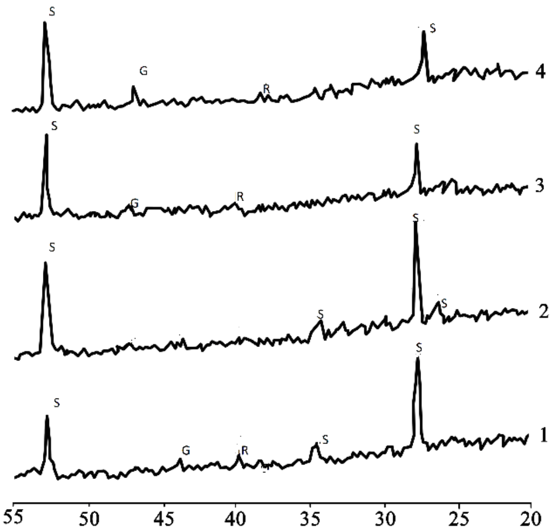


Figure 1. Fragments of DTA test glass enamels with alumina-containing waste.

In accordance with fig. 1, the intensity of exothermic effects decreases not only with an increase in aluminum oxide and a decrease in the content of B_2O_3, but also with a low content of B_2O3. There are several exothermic and endothermic effects on the thermal curves of heating glass enamels, which indicates the presence of several crystalline and glassy phases. To determine the composition of the crystalline phases, the enamel coatings were kept for an hour at the temperatures of occurrence of exothermic effects, after which they were subjected to X-ray phase analysis. The diffraction patterns of enamel coatings (Fig. 2) with alumina-containing waste revealed mainly the presence of crystalline phases of sphene, rutile and gehlenite.

The whiteness of enamel coatings muffled with sphene does not exceed 75%.



S-crystalline phase of sphene; G-crystalline phase of gehlenite; R-crystalline rutile phase. 1) AG-1; 2) AG-2; 3) AG-3; 4) AG-4.

Figure 2. Diffraction patterns of experimental glass enamel coatings with different contents of alumina-containing waste.

IR absorption spectra of glass enamel coatings made it possible to reveal that when 2.5% alumina-containing waste is introduced into the enamel composition, absorption bands with diffuse maxima appear, which may be the result of the disordered structure of the formed phases (Fig. 3). An increase in the amount of waste up to 7.5 mass. % enhances the intensity of the absorption maxima of the highly cationic silicate phase (880, 575 cm-1), which, when fused, provides damping of enamels. In addition to the high-cationic silicate phase, the IR spectra also show absorption bands characteristic of the highly silicate (1040, 720, 455 cm-1) and high-borate (1350-1450 cm-1) phases. Further substitution of (B_2O_3 + Na_2O) for alumina-containing waste causes a change in the configuration of the IR spectra: the intensities of the absorption bands at 880 and 575 cm-1 weaken and disappear, at the same time the intensity of the bands at 1040 and 720 cm-1 increases, and in the high-frequency part of the spectrum the absorption decreases at 1350 - 1450 cm-1 and a new band appears at a wavenumber of 1200 cm-1.

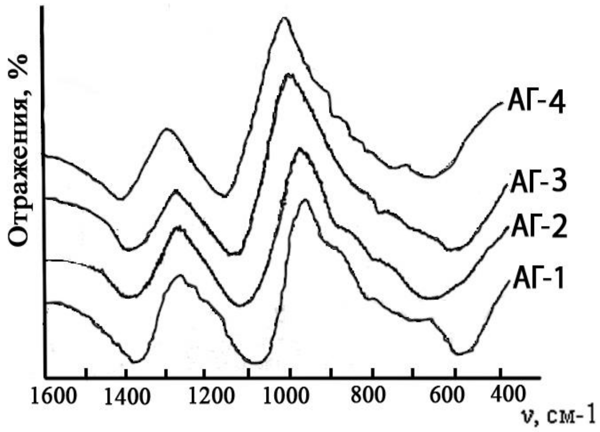
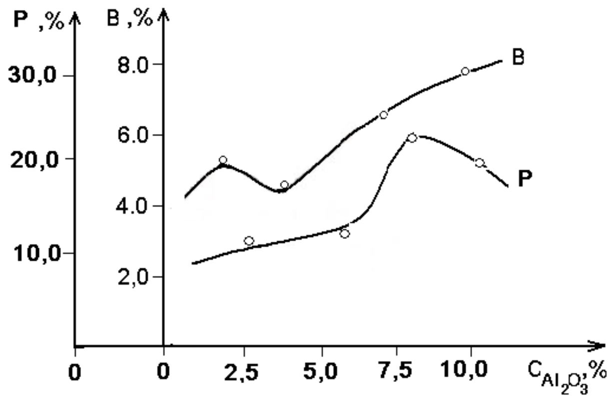


Figure 3. IR absorption spectra of experimental ground glass enamels

According to the data of works [9], these changes can be caused by the formation of a single quartz-like framework ⁅Si (Al) O_4⁆. Changes that occur in the structure of enamels with the gradual replacement of one component by another, are reflected in their properties.



mass, %

Figure 4. Dependence of the leachability (B) and spreadability (P) of the experimental ground glass enamels on the concentration of alumina-containing waste.

With the help of electron microscopic and IR spectroscopic analyzes in the studied range of compositions, the alternation of liquation and quasi-homogeneous microstructures was established depending on the Al_2O_3 content. The leaching and spreading of glasses change in the same relationship (see Figure 4). Introduction of 2.5 masses. % alumina-containing waste causes the appearance of a small maximum on the leachability curve, the range of compositions with alumina-containing waste 5.0-7.5 wt.% is characterized first by a decrease and then by an increase in the value of leaching. The spreadability of the compositions first increases, and then, when the content of alumina-containing waste reaches 7.5%, a decline begins. Therefore, we can conclude that the decisive factor is not always the chemical composition of the enamels, but their microstructure, in which certain crystalline phases are formed.

Using modern physicochemical methods of analysis, we studied the basic properties of the obtained glasses. The study was based on the analysis methods proposed in [10]. The results of the study are presented in Table 2. Analysis of the data obtained shows that with an increase in the content of alumina-containing waste, the indicators of physical and mechanical properties increase.

Table 2. Main physical and mechanical properties of ground glass enamels with alumina-containing waste

Properties	Compositions, mass %:				
	Original composition	AG - 1	AG -2	AG -3	AG - 4
TCLE, $\alpha \cdot 10^{-7}$, deg^{-1}	115.1	116.2	119.4	120.6	126.2
Surface tension, n / m	0.020	0.023	0.027	0.029	0.035
Softening onset temperature, 0C	540	545	555	560	570
Density, kg / m3	2310	2328	2375	2385	2398

Microhardness, kg / mm2	398.2	399.0	405.6	416.5	422.0
Heat capacity, J / (kg · k)	780	785	790	792	795
Thermal conductivity, W / (m · k)	0.96	0.98	0.96	0.97	0.98
Heat resistance, number of heat cycles, 400-20-400 0C	6	6	6	7	7
Contact angle of wetting, degrees	52.5	51.2	51.4	51.3	51.2

Conclusions

Thus, on the basis of the data obtained, it can be assumed that with the introduction of alumina-containing waste of Shurtangsky MCC into the composition of glass enamel up to 10%, no significant negative changes in physical and chemical properties are observed, while the optimal amount of alumina-containing waste introduced into the composition of the glass enamel mixture is 7.5 %.

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