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# *Acid-Resistant Liquid Glass Coatings Using Soda Ash Production Waste*

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**Abstract – The article presents the results of research on the topical problems of protection of building structures, structures and products of various purposes operated in aggressive, mainly acidic environments. The existing scientific bases of obtaining high-performance acid-proof cements on the basis of liquid glass with the use of local raw materials and secondary resources of various industries are developed. Questions of increasing and improving the properties of acid-resistant liquid glass compositions by changing the input components: a primer, a filler and a hardener by introducing various active and inert additives, modifying the liquid glass, optimizing curing regimes, selection of compositions for specific operating conditions are considered.**

**Keywords – Acid-Resistant Materials, Porphyrite, Wollastonite, Liquid Glass, Chemical Stability, Microstructure, Aggressive Medium, Optimal Composition, Liquid Phase, Chemical Compounds, Silicic Acid Gel.**

## I. INTRODUCTION

In recent years more and more plastic self-hardening liquid glass mixtures with calcium-containing and other substances as a hardener are used [1,2,3]. The results of studies of the processes leading to hardening of dry quartz sand and liquid glass masses when impregnating them with magnesium, calcium and barium chloride solutions and NH4Cl and HCl solutions are given in the authors' work [4,5]. Based on the unequal strength of the masses impregnated with different electrolytes and taking into account the notion that the coagulating force of cations increases with increasing cation radius, the authors conclude that the hardening of masses treated with alkaline-earth metal chlorides is coagulative in nature. This point of view is confirmed by the dependence of mechanical properties of hardening masses on the viscosity of liquid glass, with a decrease in which the strength decreases when the chemical composition remains unchanged.

Another work by the authors [6] shows that the interaction of calcium chloride with liquid glass produces SiO2 gel and calcium oxide adsorbed on silica gel. In dilute solutions, the interaction of concentrated sodium silicate with calcium chloride is characterized by very rapid formation of a silica film separating CaCl2 from soluble glass. The authors admit the possibility of two parallel processes - the adsorption of calcium oxide on the SiO2-gel formed by the excess against metasilicate and disilicate silica, and the formation of calcium silicate as a result of the exchange reaction between CaCl2 and silica, which is part of the meta- and disilicate sodium . The interaction of liquid glass with alkaline earth metal chlorides has also been studied by a number of other researchers. The purpose of our work consists firstly: in modifying of liquid sodium glass by wastes of soda production; distillery liquid; secondly: in application of new local raw materials as fillers basalt of Karakiya deposit and diabase flour of Balpantaus deposit; thirdly: in modification of fillers by natural wollastonite; fourthly: on the basis of received results of researches to make selection of compositions of acid-proof compositions for working conditions of chemical equipment in aggressive environments.

## II. Methodology

We used the method of V.V. Moskvin [7] to determine the acid resistance of the studied compositions. Samples were formed into cubes with rib size of 1.41 mm from plastic dough. One day later the samples were unmolded and stored for 10 days in air conditions. Acid resistance was determined in sulfuric acid and hydrochloric acid concentrations - 0.5 n and 5.6 n, 6.1 n . The solution was poured at the rate of 100 ml per sample. In parallel, the samples were stored in tap water in closed desiccators. Solutions were replaced every two months. Compression tests were performed after 28, 180, and 360 days. X-ray phase analysis was performed on a modern computer-controlled XRD-6100 diffractometer (Shimadzu, Japan). We used CuKα radiation (β-filter, Ni, 1.54178. Current mode 30 mA and tube voltage 30 kV), constant detector rotation speed 4 deg/min with 0.02 deg step. (ω/2θ-coupling), the scanning angle was varied 4 to 80o. The IR spectra of the tested samples were studied using a NicoletI S-50 FTIRA dvanced KBr Gold spectrometer+Nicolet Continuum, manufacturer: Thermo Scientific (USA).

## III. Results and discussion

Creation of wasteless technological processes in the processing of raw materials is currently an acute socio-economic problem. In this regard, soda production is accompanied by accumulation of large quantities of liquid wastes which are difficult to utilize. The distillation slurry, the content of which is 9-10 m3 /t of soda, is dumped into storage tanks, the so-called "white sea". When storing these discharges, liquid filtration occurs through the bottom and walls of the storage tanks, which leads to salinization of adjacent land and groundwater. In experiments we used liquid sodium glass with a density of 1.49 g/cm3 with a silicate modulus of 2.8, pH - 10.3 sodium silica, mineral fillers, distillation liquid - brine treatment sludge CaCl2 and NaCl in the ratio 2:1.

In this work the mechanism of interaction of liquid glass with distiller's liquid, sodium fluoride, calcium containing silicates waste from soda production in the presence of monomineral fine fillers is considered. Distiller's liquid in the amount of 25, 50% of the volume of liquid glass was added in the role of a charger. Chemical composition of initial materials is given in table 1.

***Таблица 1. Химический состав исходных материалов.***

| № | Сырьевые материалы | Содержание оксидов, масс.% | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | MnO | $Na_2O$ | $R_2O$ | Cl | $SO_3$ | П.п.п | Σ |
| | | | | | | | | | | | | | |
| 2 | Базальт | 49.1 | 14.48 | 9.33 | 13.2 | 7.5 | 0.15 | 2.23 | 0.32 | 0.003 | 1.55 | - | 97.86 |
| 3 | Диабаз | 58.28 | 18.27 | 7.39 | 5.32 | 4.70 | 0.001 | 1.74 | 0.10 | 0.05 | 3.51 | - | 99.17 |
| 4 | Волластонит | 37.22 | 1.00 | 0.80 | 42.83 | 3.95 | 0.07 | 0.1 | 0.19 | 0.06 | 12.50 | - | 98.72 |
| 2. | $Na_2O{\cdot}SiO_2$ | 33.24 | | | | | | 11.65 | | | | - | 44.89 |
| 3. | $Na_2SiF_6$ | 27.9 | 0.10 | 0.12 | 0.30 | 0.30 | 0.01 | - | 31.03 | | 0.10 | - | 59.86 |
| 4. | Шлам очистки рассола | 0.10 | 0.01 | 0.15 | 22.29 | 1.61 | 0.01 | 9.44 | | 10.92 | 2.91 | 54.91 | 102.35 |

Four compositions of the following compositions were prepared:

1 composition: diabase - 63% - filler, Na2SiF6 - 4% - hardening gas pedal, liquid glass(Na2O-Si2O ) - 35ml.

Composition 2: Diabase - 56% - filler, Na2SiF6 - 4% - hardening gas pedal, liquid glass - 35 ml, wollastonite - 11%.

3 composition: Basalt - 63% - filler, Na2SiF6 - 4% - hardening gas pedal, liquid glass - 35 ml.

Composition 4: Basalt - 56% - filler, wollastonite - 11%, Na2SiF6 - 4% - hardening gas pedal, liquid glass - 30 ml.

Table 2 shows the results of mechanical strength and chemical resistance coefficients of samples of compositions of 4 compositions using liquid glass and distiller fluid

***Таблица 2. Механическая прочность и коэффициенты кислотостойкости образцов кислотоупорных композиций на основе жидкого стекла и дистиллерной жидкости.***

| Составы | Предел прочности образцов МПа, хранившихся в агрессивных средах и КС* | | | | |
|---|---|---|---|---|---|
| | $H_2O$ | 0.5 н $H_2SO_4$ | 6.1 н $H_2SO_4$ | 0.5 н HCl | 5.6 н HCl |
| 28 суточного твердения | | | | | |
| 1 | 7.1/0.67 | 17.9/1.5 | 13.6/1.92 | 9.3/ 1.31 | 7.9/1.11 |
| 2 | 17.1/0.97 | 29/1.7 | 33.36/1.97 | 24.62/1.44 | 21.03/1.23 |
| 3 | 8.1 /1.0 | 14.3/1.76 | 21.42/2.64 | 11.34/1.40 | 13.09/1.72 |
| 4 | 30.81/1.20 | 56.38/1.83 | 83.71/72 | 45.29/1.47 | 59.84/1.78 |
| 180 суточного твердения | | | | | |
| 1 | 14.8/1.59 | 17.9/1.21 | 28.56/1.93 | 19.83/1.34 | 17.46/1.18 |
| 2 | 14.2/1.62 | 24.42/1.72 | 28.26/1.99 | 21.00/1.48 | 18.03/1.27 |
| 3 | 12.5/1.00 | 22.88/1.83 | 33.36/2.67 | 18.75/1.50 | 22.251.78 |
| 4 | 11.14/1.23 | 25.89/1.89 | 37.54/2.74 | 20.96/1.53 | 24.80/1.81 |
| 360 суточного твердения | | | | | |
| 1 | 25.76/1.59 | 32.38/1.26 | 50.63/1.97 | 36.24/1.41 | 31.10/1.21 |
| 2 | 23.00/1.64 | 40.94/1.78 | 46.00/2.00 | 34.50/1.50 | 29.90/1.30 |
| 3 | 22.1/1.20 | 41.99/1.90 | 62.10/2.81 | 34.03/1.54 | 40.00/1.81 |
| 4 | 32.2/1.25 | 62.15/1.93 | 88.87/2.76 | 50.23/1.56 | 60.21/1.87 |

*в числителе прочночсть образцов при сжатии, в знаменатели КС, определяемый отношением Rсж. в среде / Rсж.в воде.

The results of the table show that on the basis of diabase and basalt it is possible to obtain acid-proof cements with not very high strength indicators in water and in acid solutions. With the introduction of wollastonite in the composition of diabase flour mechanical strength of cement at 28 days of age is doubled. Composition 1 strength in water, 0.5N-sulfuric acid, 0.5N-chloric acid is 7.1 MPa, 10.7 MPa, 9.3 MPa, respectively. With introduction of wollastonite mechanical strength increases and makes respectively -17,1 MPa,16,8 MPa,15,7 MPa.

At use in a role of filler of basalt flour mechanical strength of samples is characterized by high indicators. Introduction of wollastonite into basalt flour composition increases chemical resistance in water and in concentrated acid solutions. Thus, strength in water is increased from 8.1 MPa to 21 MPa, in 6.1 n - from 21.4 MPa to 23.7 MPa, in 5.6 n HCl from 13.9 to 15.2 MPa.

At partial replacement of liquid glass by distiller's solution in an amount of 25% very high results were received, especially at introduction in compositions 1,3 of natural wollastonite. So at composition 2 strength indicators are following: in water-17,1 MPa; in 0,5H2 04 29MPa; in 6,1H2 04-33,68 MPa, at composition 4 following results are received accordingly: in water -39.88 MPa; in 0.5 n-sulfuric acid - 56.38 MPa; in 6.1 n-sulfuric acid -83.71 MPa; in 0.5 n-hloric acid - 45.29 MPa and in 5.6 n-hloric acid -59.84 MPa. The obtained results indicate the previously stated positive effect of calcium and chloride-containing compounds on the hardening processes of acid-proof binders on the basis of liquid glass.

Thus, the partial replacement of liquid glass with distiller's liquid increases the chemical stability of samples depending on their composition. The best composition is the 4th composition based on basalt and wollastonite.

Chemical stability of the obtained diabase and basalt acid-proof cements can be judged by the calculated coefficients of resistance.

Table 2 summarizes the results of stability coefficients of 1,2,3,4 compositions on the basis of liquid glass and distillate of 28-day, 180-day and 360-day hardening ages in water, in weak and concentrated solutions of hydrochloric and sulfuric acids. The data in the table show a smooth increase in the KC of all the given compositions. With the increase of operation time in water and aggressive media the introduction of wollastonite gives higher coefficients of resistance, especially in concentrated sulfuric and hydrochloric acids. The highest results are observed for the 4th composition. So, at 360-days hardening the coefficient of resistance in water is 1,25; in 0,5n-sulfuric acid -1,93; in 6,1n-sulfuric acid -2,76; in 0,5n-hloric acid -1,58; in 5,6n-hloric acid -1,87.

The research of adhesion ability of putties to different materials: metal, ceramics and rubber (Table 3) has shown that the samples stored both in air-dry conditions and in acids have the lowest adhesive properties to rubber, and the highest - to metal. Moreover, the adhesion ability of diabase-wallastonite composition in the aggressive environment is higher than that of diabase - 2 MPa.

Table 3. Results of research of adhesive properties of diabase and diabase-wallastonite putties

Name of compositions Materials Tensile strength of samples, MPa, in environments

| Compositions name | Materials | Tensile strength of samples, MPa, in media | |
|---|---|---|---|
| | | In the air | In a corrosive environment |
| Diabase | Rubber<br>Ceramics Metal | 0,96<br>1,4<br>1,93 | 0,85<br>1,08<br>1,145 |
| Diabase-wollastonite | Rubber<br>Ceramics Metal | 0,99<br>2,1<br>2,4 | 0,87<br>1,9<br>2,00 |

The phase composition and microstructure of liquid cement compositions based on diabase and wollastonite, basalt and wollastonite in aggressive media have been studied using complex physical and chemical analysis methods.

X-ray diffraction patterns of basalt cement composition samples cured in 6.1n H2SO4 at 28-day hardening show many lines characteristic of the minerals contained in basalt: anorthite (d=0.320; 0.269; 0.403 nm), augite (d=0.295; 0.255; 0.162 nm), divalent gypsum (d=0.716; 0.355 nm), sodium carbonate (d=0.253), sodium silica (d=0.334; 0.177). By 360 days the lines of the above minerals weaken, the lines of gypsum disappear, which indicates its binding in ettringite with d=0.981; 0.559; 0.466; 0.386 nm. Lines characteristic of calcium hydroxychloride appear with d=0.823 nm.

In concentrated hydrochloric acid at 28 days of age intensify lines of quartz d=0.445; 0.412; 0.188 nm, pyroxenes d=0.312; 0.324 nm. The lines of NaF d=0.232; 0.166 nm and CaF2 d=0.314; 0.193 nm appear. Emerging neoplasms are in a state of high dispersion. They confirm about full course of reaction between liquid glass and sodium silica[ 8 ]

2Na2SiO3+ Na2SiF6+6H2O=6NaF+3Si(OH)4

Crystalline phase of calcium hydroxychloride d=0,734; 0,555; 0,383 nm appears, which condition the formation of structure, providing cement stability in aggressive media.

In weak acid solutions (HCl and H2SO4), X-ray patterns of basalt cement composition samples show mainly peaks characteristic of basalt flour minerals - quartz, pyroxene. Blurred peaks indicate the formation of silicic acid gel and tobermoritic gel. A high peak is characteristic of calcium hydroxychloride (d=0.832 nm) .

Modification of basalt composition with natural wollastonite indicates the intensity of diffraction maxima, diffraction lines are blurred, only at the end of the radiographs appear high peaks characteristic of calcium hydrocarbonates and hydroxychlorides. In the initial periods of hardening interplanar distances characteristic of the silica phases are more intense, the lines Na2SiF6 d=0.455; 0.307 nm, which disappear by 360 days, appear villionite and fluorite lines, indicating a fine-crystalline structure of compositions blurred lines characterizes the increase in silicic acid gels.

Introduction of distiller's liquid, soda production waste leads to X-ray amorphous structure and formation of new compounds of calcium hydrosilicates and calcium oxychlorides. Given the good solubility in water of calcium oxychlorides and poor calcium hydroxide, portlandite lines are fixed in the clusters of amorphous silica on X-rays. The formation of crystalline hydrates shifts this reaction to the right with the resultant enrichment of the liquid glass with silica and the appearance of SiO2-gel.[ 9 ]

Thus, during the course of chemical reactions in liquid glass compositions made on a soda waste solution, there is a binding of alkaline cations into compounds, the composition of which is largely determined by the nature of the initial substances. The transformation of liquid glass into $SiO_2$-gel is caused by the formation of calcium oxychlorides and, probably, hydrochlorides, as well as sodium chloride.

The rate of hydrolysis of sodium silica fluoride slows down, but the mechanism of interaction and the nature of the products formed remains unchanged[ 10,11,12 ].

The test results showed high chemical stability of distiller's liquid samples depending on its amount, as well as the type and concentration of acids. Increasing the amount of distiller's liquid up to 50% with respect to the liquid glass leads to a decrease in the resistance coefficient, which probably indicates a weak crystallization ability of high silica sodium silicates.

An intermediate zone of whitish, cloudy $SiO_2$-gel is instantly formed on contact with liquid glass of distiller's liquid. A common feature in the interaction of sodium chloride and sodium silica with liquid glass is a zonal structure of the interaction boundary of the solutions of the starting substances. In the process of chemical reactions alkaline cations bind into compounds whose composition is largely determined by the nature of the starting substances. The interaction of calcium chloride with liquid glass produces $SiO_2$ gel and calcium oxide adsorbed on silica gel. In dilute solutions, the interaction of concentrated sodium silicate with calcium chloride is characterized by very rapid formation of a silica film that separates $CaCl_2$ from soluble glass. Two parallel processes may occur - adsorption of calcium oxide on silica gel, formed due to excess against metasilicate and disilicate silica, and formation of calcium silicate as a result of exchange reaction between $CaCl_2$ and silica, which is part of sodium meta and disilicate by the total equation.[ 13,14]

$$Na_2O \cdot SiO_2 + CaCl_2 + nH_2O \rightarrow 2NaCl + CaSiO_3 + nH_2O$$

The introduction of $CaCl_2$, NaCl electrolyte additives into cement batter contributes to changing the conditions of crystallization of hydrate phases, increasing due to increase of ionic force of solution the degree of oversaturation of liquid phase in relation to hydration products.[ 15 ]

In the case of distiller fluid containing $CaCl_2$ and NaCl, the transformation of liquid glass into $SiO_2$-gel is due to the formation of calcium oxychlorides and probably calcium hydrochlorides. At maintenance of samples in a corrosive environment radiographically the improvement of a degree of crystallization of the formed phases - $CaSiF_6$, NaF, $CaF_2$ with occurrence of weak intensities of the strongest lines of hydrosilicates of calcium peculiar tobermorite $Ca_5(Si_6O_{18}H_2)4H_2O$ - with diffraction maximums d = 0.307; 0.215; 0.206; 0.200 nm is noted.

Thus, hardening of liquid glass compositions set with distiller's liquid occurs as a result of chemical reactions of alkaline cations binding into compounds, the phase composition of which depends on the nature of the initial substances.[ 16,17,18 ] X-ray patterns indicate crystalline phase formation in weak and concentrated HCl solutions. There are peaks typical for anorthite, augite, pyroxenes, which indicates incomplete decomposition of basalt. In concentrated and weak $H_2SO_4$ solutions, complete decomposition of basalt and wollastonite is observed. The peak characteristic of $Na_2SiF_6$ is absent, which confirms the full course of reaction between sodium fluoride and liquid glass. As the hardening time increases, new formations characteristic of portlandite, calcium hydrocarbonates, ettringite and calcium oxychloride appear. Blurred peaks are characteristic of tobermorite gels and silicic acid gels.

It is possible to judge about composition of formed products of interaction of liquid glass with calcium silicates by the type of IR-spectroscopy curves. Fig. 1 shows infrared spectra of samples of the 4th composition: basalt, wollastonite, sodium silicate, liquid glass diluted with distilled liquid, stored in water; 0.5n and 5.6n solutions of hydrochloric acid. IR spectra of samples stored in water differ slightly from IR spectra of samples stored in acid solutions. The strain vibration interval in the 790-500 cm-1 region has longer peaks. The sharpness of the peak bands indicates an increase in the degree of crystallization of the hydrate neoplasms at 785-722-648 cm-1. The 1450-1460 cm-1 band is characteristic of the bonding of sodium cations with $CO_2$ groups. Absorption band of Ca-O bond vibration falls in the interval 1400-1500. A small band at 1430 cm-1 is characteristic of $Na_2CO_3$ spectrum, indicating the presence of $Na_2CO_3$-$nH_2O$ sodium carbonates and hydrocarbonates.

The spectra of samples stored in solutions of HC1 are almost identical, the only difference is a more blurred band of strain vibrations at 3500 cm-1. Broad bands of hydroxyl vibrations associated with silicon atoms at 2600-3600 cm-1 probably correspond to the OH vibration band of SiOH groups in the silicate composition $CaNaHSiO_4$. Broad bands show the basic vibrations of silicon and oxygen Si-O-Si atoms in SiO4-4 groups. Lines 785-722 indicate the presence of Al occurring as [AlO4]-4 groups. The 1110-1000 and 500 cm-1 vibrational bands found in all silicate spectra belong to the basic vibrations of SiO4-4, which are elementary structural units of silica and all silicates. The band with a maximum at 1638 cm-1 indicates the presence of crystallization water in the liquid glass and distillation liquid - H-O-H deformation vibrations. Thus, the IR spectroscopic analysis confirms that the hardening of the structure of the acid-resistant composition is due to chemical and intermolecular interaction of the components.

## IV. Summary

On the basis of the studies performed it is possible to draw a conclusion about the possibility of using diabase and basalt flours as a filler for acid-proof cements and partial replacement of liquid glass by distiller's solution - a waste of the Kungrad soda plant significantly increases both mechanical strength of samples and chemical stability in aggressive environments.

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