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Carbamide Composition With Quartz Fillers And Hydrogel Additives

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Abstract

The paper presents the results of experimental studies of the effect of hydrogel additives on the physical-mechanical and structural properties of carbamide compositions with quartz filler. The aim of the study was to determine the optimal hydrogel content, which ensures maximum strength and thermal stability of the composition.

The samples were made with a different content of hydrogel additives (0.05–0.17% by weight of the filler), taking into account the amount of free water released during polycondensation of carbamide-formaldehyde resin. The optimal compressive strength (88.0 MPa) was achieved with a hydrogel content of 0.13%, which corresponds to the binding of 75% free water. A further increase in dosage led to a decrease in strength due to excessive moisture binding and changes in hardening conditions.

IR spectroscopic analysis showed that the introduction of a hydrogel additive promotes the formation of stronger hydrogen bonds between the components of the composition and reduces the hydrophilicity of the quartz filler. Electron microscopic studies have revealed an ordered microstructure with a decrease in interfacial stresses and porosity. Based on the results of thermogravimetric analysis, it was found that hydrogel compositions have a lower mass loss when heated to 830 ° C, which indicates an increase in their heat resistance.

Thus, it was found that the optimal content of the hydrogel additive is 0.13% of the mass of the filler, which increases the strength of the carbamide composition by 20-25%, reduces microporosity and improves the thermal stability of the material.

Keywords: Carbamide composition, urea-formaldehyde resin, hydrogel polymer additive, compressive strength, water binding, IR spectroscopy, microstructure, thermal stability.

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1. Introduction

With the development of material science and technology, polymer materials exhibit application potentials in the building industry attributed to their excellent properties compared with inorganic materials, such as waterproof, anticorrosion, wear resistance, antiseismic, lightweight, good strength, sound insulation, heat insulation, good electrical insulation, and bright colors. Due to their superior properties, polymer materials have been widely used in the building industry, such as the insulation layer of the water supply pipe, drainage pipe, wire and cable, and wall insulation material[1][2][3].

Synthetic binders are special adhering materials manufactured from pure polymers, polymeric blends, resins, and oils[4][5].

Ever since they were first introduced in 1884, urea-formaldehyde resins have been used extensively as adhesives in the wood sector [6]. Favorable properties of the resins—high reactivity, quick gelation, economy, nondiscoloration, and long-term durability make UF resins some of the most common formaldehyde-based adhesives in the world[6] [7].

One of the relevant areas of application of polymer composite materials is their use as protective coatings that protect the elements of concrete structures of buildings and structures from the effects of adverse natural and man-made factors, primarily from moisture saturation[8].

It should be noted that along with such valuable properties as incombustibility, colorlessness, good miscibility with water, etc. urea-formaldehyde resins and compositions based on them have a number of disadvantages: relatively low physical and mechanical properties (lower than 0.1 Pa·s at 25 ◦C)[9], the formation of high internal stress during hardening, brittleness, a large amount of chemically unrelated water, low heat resistance, which complicate their use in construction on a large scale[10][11].

The strength and chemical resistance of carbamide compositions are significantly improved if free water is bound, which is released by side during resin polycondensation and accounts for about 35-40% of the binder weight. It is known that the intensive release of free water during polycondensation of the carbamide oligomer is accompanied by the formation of high internal stresses, which leads to a decrease in the crack resistance of composites based on them. The choice of effective water-binding additives is very difficult due to the acidic nature of carbamide composites hardening. Therefore, the search for effective acid-resistant water-binding additives is an important step in the development of carbamide composite materials. This can be achieved by using artificial and natural zeolites, polymer and other substances, the products of which are water- and chemically resistant[12][13].

Therefore, the issues of binding of free water released during polycondensation of carbamide resin, as well as improving the physical-mechanical properties and chemical resistance of carbamide compositions require additional research.

Polymer building compositions are materials obtained by hardening combined synthetic polymer binders and mineral fillers[9]. As a result of this combination, polymer compositions have high density, strength, chemical resistance, and many other positive properties[14].

The porosity of carbamide compositions is increased by the release of free water of the order of 35-40% by weight during resin polycondensation. With further polycondensation, the amount of free water increases. In this regard, during polycondensation of carbamide resins, numerous pores form in the hardened composite, which negatively affect their density, strength, and chemical resistance.

Water, which accounts for 35-40% of the carbamide resin, has a significant effect on the structural and strength properties of the composition. Therefore, the main direction in the technology of carbamide compositions is the development of effective methods for binding free water to a hardening mixture. The water contained in the resin, as well as released as a result of the polycondensation reaction, envelops the particles of the mineral filler and prevents the formation of strong adhesive bonds in the contact zone between the resin and the filler. In addition, dehydration causes a decrease in the strength and density of binders and initiates shrinkage of the system. Therefore, in order to increase the strength and reduce shrinkage deformations of the carbamide composition, it is necessary to chemically bind part of the water into structuring compounds.

In order to bind free water, polyisocyanates were introduced into the composition. Due to the reactive isocyanate groups (23-25%), polyisocyanates interact very vigorously with water. At the same time, the physical-technical properties of the composition are significantly improved.

Phosphogypsum was used as a water-binding additive and a hardener for carbamide compositions. Although this increases the strength and chemical resistance, however, the problem of creating a stable structure of the latter is not completely solved. Thus, hydrated gypsum and phosphogypsum themselves are not water- and chemical-resistant in aggressive solutions. Consequently, the strength and chemical resistance of carbamide compositions decrease during operation in aggressive environments.

The introduction of phosphogypsum into liquid and cast formulations with N/A = 0.4-0.6 causes a slight increase in strength and even a decrease in it. At the same time, phosphogypsum, introduced in an amount of 50% by weight of resin, binds about 21% of free water (or increases the concentration of resin by about 9%). Binding of water with gypsum or phosphogypsum reduces the mobility and viability of carbamide composites. Thus, the thickness of the cone of the carbamide composition with a thickness of 0.5 without phosphogypsum was 291 mm, and with the addition of 20 and 50% phosphogypsum by resin weight - 249 and 222 mm, respectively. The presence of 0.6-1.0% phosphoric acid in the phosphogypsum composition also contributes to a decrease in the viability of the carbamide composition[15].

It is known that the binding of 7-8% of free water by introducing up to 50% gypsum or phosphogypsum by weight of resin leads to an increase in the strength and water resistance of carbamide binders. Phosphogypsum, which is a waste product of the chemical industry, is traditionally used as a water-binding additive[16].

One of the effective ways to strengthen the structure of polymer composites is to modify carbamide compositions with water-binding additives, which, in our opinion, should lead to an increase in the strength of the composition. In this case, the effectiveness of the modification method is determined by the condition of compatibility of the constituent compositions and the nature of the adsorption-chemical activity of the filler to the binder.

However, experimental and theoretical data predict that the presence of water in small amounts is necessary because it serves as a co-catalyst for polymerization. The total amount of it introduced and formed in the binder as a result of polycondensation is calculated, it is 3-4% of the total mass.

In this scientific work, polymer hydrogel additives were used as a water-binding additive. Hydrogels are three-dimensional, cross-linked polymer networks known for their ability to absorb and retain large amounts of water, exhibiting significant swelling capacity. Over the years, advancements in polymer cross-linking have made hydrogels highly tunable materials for a wide array of applications[17].

2. Methods

The following materials were used to prepare the carbamide composition:

Binder. Urea-formaldehyde resin is a product of polycondensation of urea with formaldehyde. A resin of the KF-J brand (increased viability) was used, which is a homogeneous suspension of white and light-yellow color.

Hardener. Aniline hydrochloride, a light green powder with a density of 1.2 g/cm3, aniline hydrochloride content of 98.9%, moisture content of 0.5-1.5% and 0.1% insoluble substances were used to cure carbamide resin; it has the chemical formula C6H5NH2CL.

Filler. Ground quartz sand from the Volsky deposit with a density of 2.6-2.7 g/cm3 was used as a filler.

The water-binding additive hydrogel is a highly swelling polymer hydrogel synthesized on the basis of polymerization of saponified acrylic acid with a sodium salt of carboxylmethyl cellulose in the presence of the initiator potassium persulfate.

Hydrogel has the ability to absorb and retain very large amounts of moisture, sometimes hundreds of times their own weight. They are obtained from substances that do not dissolve in water, but form a polymer network. The additional moisture does not disrupt their mesh structure and does not dilute them. Hydrogels are cross-linked polymers and in their initial state before hydration they are similar to rigid polymers, inflexible, brittle and rigid. When immersed in water, the hydroxyl groups of the dry polymer attract water molecules and the polymer absorbs water. The volume of absorbed water depends on the amount of hydroxyl components in its structure. When saturated with water, the polymer becomes soft and flexible. They have an amorphous structure. The structure of the hydrogel is permeated with numerous pores, the size and number of which differ greatly from substance to substance.

The determination of the physical-mechanical properties of the carbamide composition was carried out in accordance with the "Guidelines on Polymer Concrete Testing methods" and "Recommendations on the methodology for determining the strength and deformation characteristics of polymer concrete under short-term and prolonged loads", as well as standard methods.

To determine the compressive strength, samples were made from a polymer composition with dimensions of 30x30x30 mm. The strength of the carbamide composition during bending was determined by breaking beams with dimensions of 40x40x160 mm. 6 samples were prepared from each composition.

The production of samples from the carbamide composition was carried out in accordance with CMC 3.03.08-98 "Instructions for the technology of preparation of polymer concrete and products from them".

The polymer composition was prepared manually. The samples were molded on a laboratory vibrating pad with a frequency of 2800-3000 rpm and an amplitude of 0.5 mm. After heat treatment, the samples were removed from the molds, exposed to air under normal conditions for 24 hours, and then subjected to testing.

Ground quartz sand up to the specific surface area under study was obtained in a laboratory porcelain mill. The raw materials were ground in batches of 500 g. Upon receipt of the filler, hydrogel powder was added at the last stage of quartz pitch grinding in order to uniformly distribute the polymer additive in it. The amount of the additive was calculated from the calculation of free water of 33-34% released during polycondensation of carbamide resin. At the same time, part of the free water in the amount of 3-4% was left in the polymer system to ensure favorable conditions for the curing of the composition. Given the high hygroscopicity of the sand filler, including hydrogel, individual crushed batches were stored in plastic bags to avoid contact with air.

The specific surface area of the filler was determined using a T-3 pneumatic surface meter.

Physical-chemical research methods.

The method of differential thermal analysis (DTA) was used to determine the heat resistance of a carbamide composition with a quartz additive and a hydrogel filler.

The infrared spectroscopy method was used to study the composition structure formation process.

The morphology of the structure of carbamide compositions was studied using the electron microscopy method, more precisely, the method of carbon enveloping replicas on a transmission electron microscope.

The method of X-ray phase analysis was used to qualitatively and quantitatively evaluate the structure of polymer compositions.

3. Results And Discussion

To study the effect of hydrogel additives on the basic physical and mechanical properties and determine their optimal amount, samples from a carbamide composition were made. When adding hydrogel components to the composition, the amount of free water released during polycondensation of carbamide-formaldehyde resin was taken into account. It is known that the maximum released free water during resin polycondensation is 35-40% by weight of the latter. If we consider that 1 g of hydrogel has the ability to absorb 100 g of water, then for the maximum degree of absorption, it is necessary to add 0.175% of the hydrogel additive by weight of the filler. This composition can be considered a 100% absorbed composition.

To absorb 25, 50, 75 and 100% of free water, the amount of hydrogel additive should be 0.05, 0.09, 0.13 and 0.17% by weight of the filler, respectively.

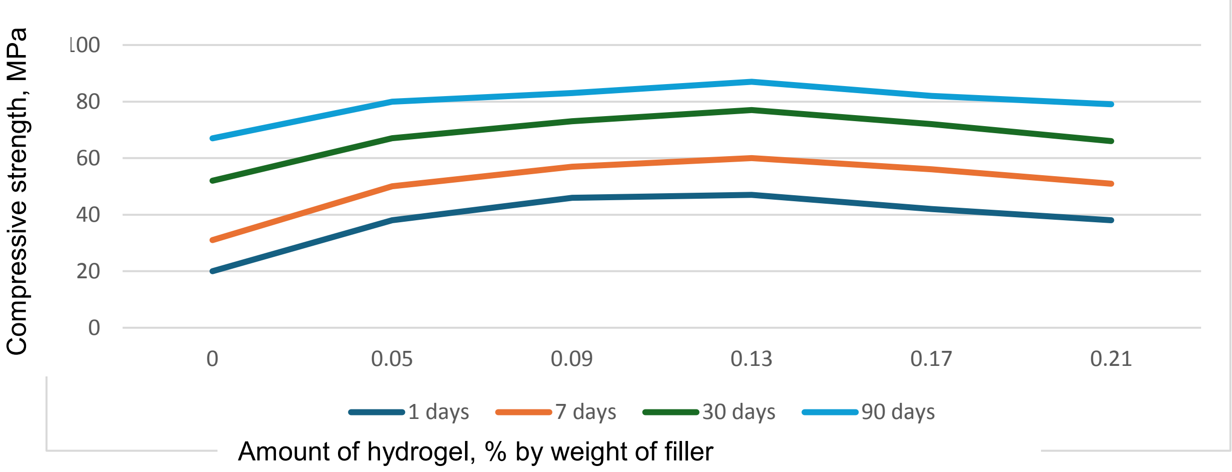
Hydrochloric acid aniline (SKA), which is a traditional hardener of carbamide compositions, was used as a hardener. Its consumption was in the range of 2.5... 3.5% by weight of resin.

Quartz flour from the Volsky deposit was used as a filler, the degree of filling in the composition is Cv = 2, and Sd = 0.2 m2/g. After pouring the polymer composition, the samples were compacted on a vibrating pad.

The amount of hydrogel additive was determined depending on the compressive strength of the carbamide composition samples. For the experiments, a carbamide composition was prepared with a different content (0.05...0.17%) of hydrogel from the mass of the filler.

The change in the compressive strength of the developed carbamide composition depending on the amount of hydrogel additive is shown in Fig.1. As can be seen from Fig.1, the compressive strength of the composition reaches its maximum values with a 0.13% addition of hydrogel by weight of filler at the age of 90 days (88.0 MPa).

Figure 1. Dependence of the strength of the carbamide composition on the amount of hydrogel



It was found that with an increase in the amount of hydrogel additive, the curve of compressive strength has an extreme maximum character at 0.13% of the additive. With a hydrogel additive consumption of 0.05, 0.09, 0.13 and 0.17% by weight of the filler, the compressive strength values are 80.5, 84.5, 88 and 84 MPa, respectively.

The extreme change in the compressive strength of the carbamide composition at constant values of N/A and Sd of the sand filler from the hydrogel content is explained by several reasons. Firstly, the water-binding effect naturally depends on the amount of hydrogel additive. However, the positive effect of this effect is maximal with a 0.13% additive content. A further increase in the dosage of the hydrogel additive leads to a decrease in strength, although the values remain higher than the control composition. This is probably due to the amount of free water that must remain in the polymer system to ensure favorable hardening conditions.

Therefore, if calculations are performed, when 0,05; 0,09; 0,13 and 0,17% of the hydrogel are introduced, the latter binds 25; 50; 75 and 100% respectively, of free water released during polycondensation of carbamide resin. At the same time, the maximum strength of the carbamide composition with a sand filler and the addition of 0,13% hydrogel is observed when binding 75% free water. This means that in order to create favorable hardening conditions and reduce internal stresses, 25% of free water must remain unbound in the modified carbamide composition. This trend is independent of the age of the carbamide composition.

Secondly, it is likely that the addition of hydrogel has a plasticizing effect on the polymer system. As the content of the hydrogel additive in the carbamide composition increases, the role of plasticization at the polymer-filler interface gradually decreases, and plasticization of the carbamide oligomer itself begins to play a more significant role, i.e., disruption of intermolecular bonds in the polymer itself. However, since the interaction of the carbamide oligomer with the surface also occurs with a relatively high content of the hydrogel additive, the further weakening of the bonds with the surface is superimposed on the actual plasticization of the oligomer and the overall decrease in strength in the presence of the hydrogel additive becomes greater.

Thirdly, due to the absorption of 75% of free water, the hydrophilicity of the sand filler particles is significantly reduced, which can positively affect the adhesion to the binder.

Thus, the research results indicate that the use of a hydrogel additive as a water-binding component increases the strength of the carbamide composition by 20-25%.

The method of infrared spectroscopy was used to study the nature of the bonds between the components in the proposed composition. In this regard, IR spectra of the initial components of the carbamide composition and their mixtures were obtained (Fig.2).

To study the structure of the hydrogel, IR spectra taken in the range of 400-4000 cm-1 were studied.

Infrared absorption spectra of the hydrogel make it possible to draw a conclusion about the structure and identify changes occurring in the chain of macromolecules of the hydrogel.

When a hydrogel additive is introduced into the carbamide composition, the following absorption bands appear in the IR spectra: in the region of 3000-3600 cm-1 - valence vibrations (v) of OH and NH groups associated with hydrogen bonds, 1620-1640 cm -1 - deformation vibrations (δ) of hydrated water, 1537 cm -1 - skeletal vibrations of the benzene ring, 1378 cm-1 is the valence vibrations of the carboxylate ion (COO-), the range of 1250-900 cm-1 corresponds to the absorption of asymmetric valence vibrations of SiO2, the maximum of 774 cm -1 characterizes symmetrical valence vibrations of SiO2.

It can be seen from Fig. 1 that the introduction of hydrogel additives in the amount of 0.05, 0.09, 0.13 and 0.17% by weight of the filler, as shown by IR spectra, does not significantly affect changes in the structure of the carbamide composition. However, in the spectrum of compositions with the content hydrogel supplements are 0.05, 0.09 and 0.13 percent by weight of the filler is observed shift of the absorption bands in the low frequency region is related to the absorption of OH — groups, associated hydrogen bonds (V=3283 cm-1) and characterized by deformation vibrations of water (V=1628 cm-1±3 cm-1) compared with the composition with the content of 0.17% hydrogel supplements (ѴOH = 3306 cm-1, δOH = 1631 cm-1). At the same time, there is a higher frequency of asymmetric valence vibrations of SiO2, which are part of quartz sand (Vas=1030 cm -1 ± 2 cm -1) in compositions with a hydrogel additive content of 0.05-0.13% compared with a composition with a hydrogel additive content of 0.17% by weight of filler (Vas =1027 cm -1).

Based on the above, it can be concluded that with the maximum absorption of free water by the hydrogel additive, less strong hydrogen bonds are formed with the participation of OH groups of water and stronger hydrogen bonds with the participation of oxygen atoms of silicon dioxide.

For polymer composite materials, it is important not only the nature of the bond between the components, but also the morphology of the structure, including the supramolecular one. Therefore, we have carried out studies of the morphology of the structure of a carbamide composition with a hydrogel additive.

The electron microscopy technique is the most suitable for studying the structure of filled compositions. The electron microscopy technique is necessary because when thin sections are obtained on an ultramicrotome, the structure of the filled polymer is distorted, the filler is discolored and, in addition, the structure of the thin films may differ from the structure of the filled polymer in the block.

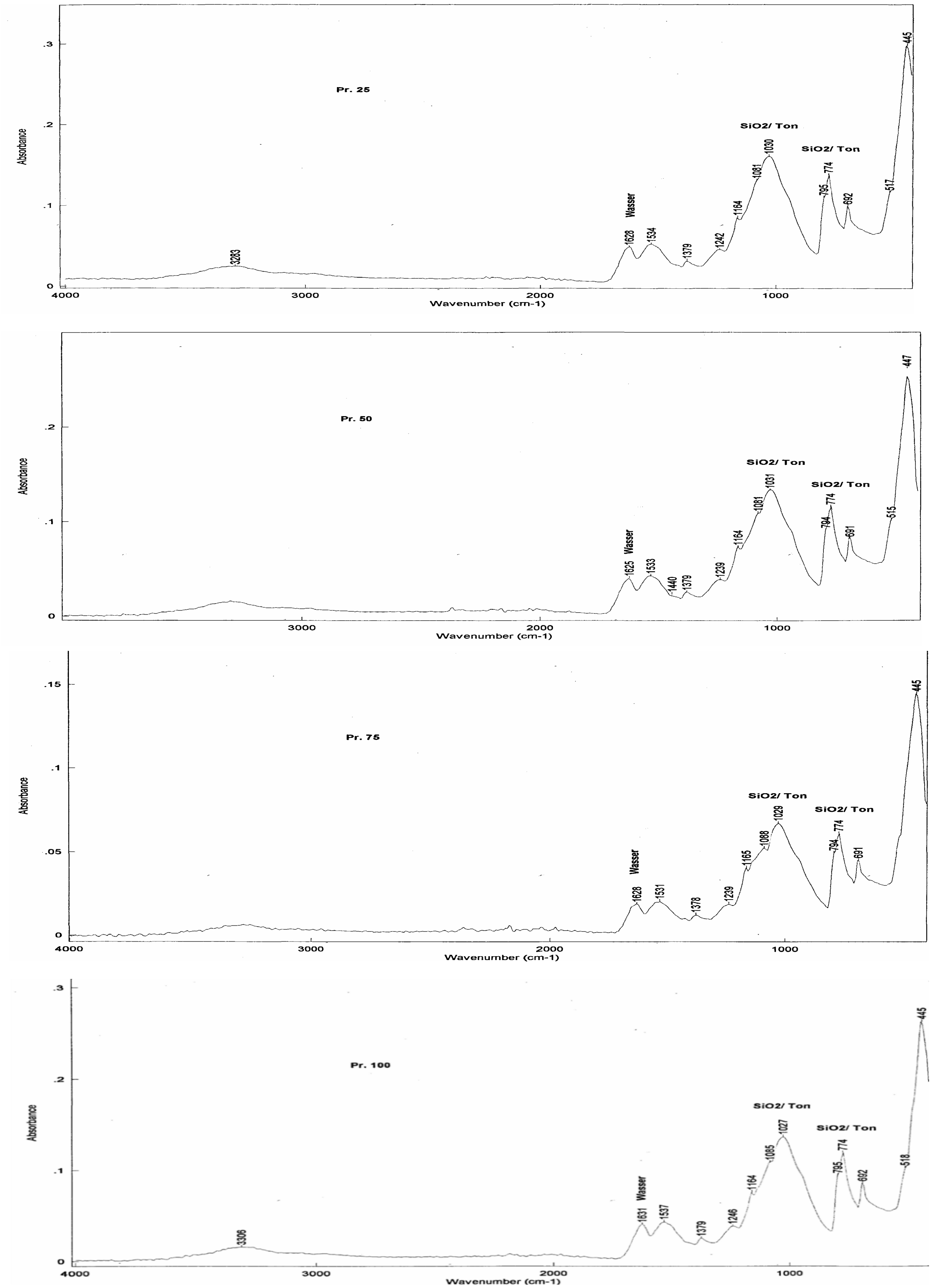


Figure 2. IR spectra of Carbamide compositions

An electronic micrograph taken from the surface of a carbamide resin sample filled with quartz filler indicates pronounced interfaces between the particles of the complex and carbamide resin (Fig.3).

The appearance of a pronounced interface is apparently explained by the stresses resulting from the different coefficients of linear expansion of the filler and carbamide resin, as well as the shrinkage of the latter during the curing process. The introduction of hydrogel additives into the carbamide composition leads to a drastic change in the structure, makes the surface of the chip relatively smooth, absorbing and dissecting the internal stresses arising between the phases. Due to the presence of water-binding additives in the carbamide composition, the surface of the composite is represented by globular lumpy-fine formations on relatively round grains with low stepped sections. It is especially clearly seen that when magnified 500x and 2000x, fragments 0.13-04 and 0.13-01 grow into a mass, creating an ordered crystal with a completely parallel microstructure, block-decorative in nature. In addition, when magnified 4000x and 8000x in fragments 0.13-02 and 0.13-03, it can be seen that the viscous mass is continuous, in which single large quartz crystals are embedded, creating an isomorphic folded microstructure of the surface.

It is known that the thermal behavior of compositions depends on the composition, the nature of the constituent components, the number of components, the reactivity of individual compounds, and many other factors. Figure 4 shows the results of DTG and TGA carbamide compositions based on carbamide resins at the age of 28 days of hardening, which show that the DTA curve of compositions without hydrogel additives shows endothermic effects at 125.9, 251.2, 288.3, 307.6 ° C and exothermic effects at 497.9, 527.2, and 585.5 °C. The nature of the first endothermic effect corresponds to the removal of water molecules and the beginning of decomposition of the composition. The weight loss along the TG curve is 4.12%. The nature of the next three endothermic effects is due to the intense decomposition of the composition, which accounts for a decrease in weight along the TG curve of 18.11%. The three following exothermic effects relate to the burning of the organic fragment of the compositions. Gorenje The mass loss in the temperature range of 420-830 °C according to the TG curve is 7.23%. The total weight loss is 29.46%, 0.63% more than the previous sample.

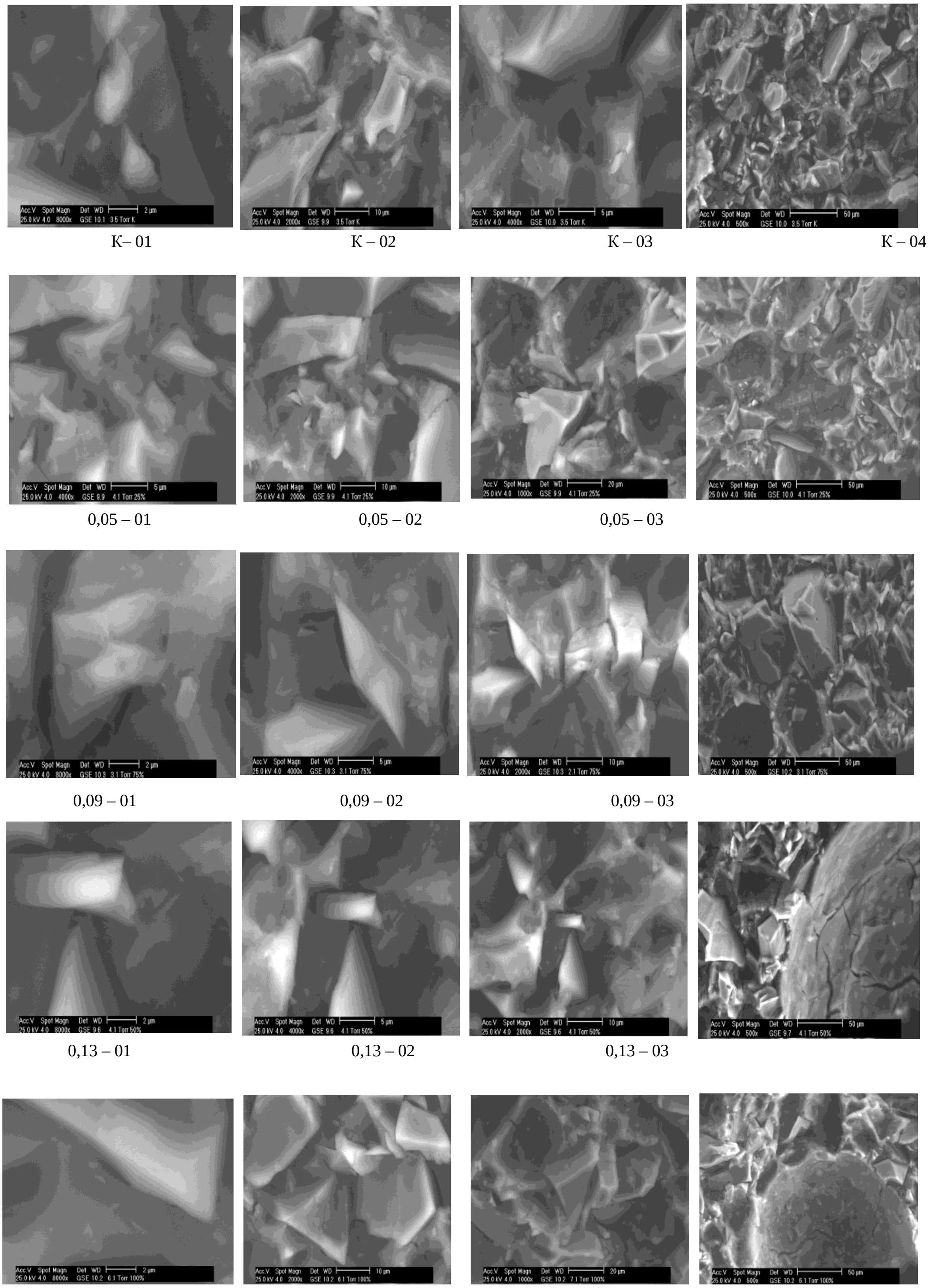
The heating curve of a composition containing 0.05% hydrogel additive by weight of filler is characterized by four endothermic effects at 125.5, 250, 290.9, 310 ° C and four exothermic effects at 464, 490, 528.8 and 586 °C. The nature of the noted thermal effects remains the same as in the previous composition. Differences are observed in the temperature values of the effects, their intensities and mass losses. If three exothermic effects were found in the previous compositions, then the DTA curve shows an inflection of the exoeffect at 464 ° C. The total weight loss on the TG curve in the temperature range 30-830 ° C is 29.46%.

Four endothermic effects were found on the heating curve of the composition with a content of 0.09% hydrogel additive by weight of filler at 125.5, 253.2, 289.6, 310.7 ° C and three exothermic effects at 485.5, 522.5 and 583.7 ° C. The first endothermic effect is accompanied by a 4.31% weight loss. The sum of the mass loss with subsequent endoeffects is 17.60%.

A further increase in temperature leads to a smooth decomposition of the composition with a weight loss of 6.92%. Total weight loss is 28.83%.

The heating curve of a composition containing 0.13% hydrogel additive by weight of filler is characterized by four endothermic effects at 124.7, 250.2, 291, 310 °C and exothermic effects at 472.9, 499.6, 524.8 and 586.2 °C. The nature of the thermal effects remains the same as in the previous compositions. A significant difference is the appearance of a new exothermic effect at 472.9 °C. The total weight loss in the temperature range 30-830 ° C according to the TG curve is 27.88%, which is 1.58% less than in the composition without hydrogel additive and with a content of 0.05% hydrogel additive by weight of filler.

The heating curve of a composition containing 0.17% hydrogel additive by weight of filler is characterized by four endothermic effects at 129.8, 258.3, 292.5, 307.5 ° C and three exothermic and form effects at 454.5, 499.8 and 586.1 ° C. The intensity of the endothermic effects is identical, as for compositions containing 0.05, 0.09 and 0.13% hydrogel additives by weight of filler. However, the areas of exothermic effects differ significantly from the previous samples. The total weight loss of TG in the temperature range of 30-830 ° C is 27.50%, which is less than in the previous composition.



К– 01 К – 02 К – 03 К – 04

0,05 – 01 0,05 – 02 0,05 – 03

0,09 – 01 0,09 – 02 0,09 – 03

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Figure 3. Microstructure of the carbamide compositions

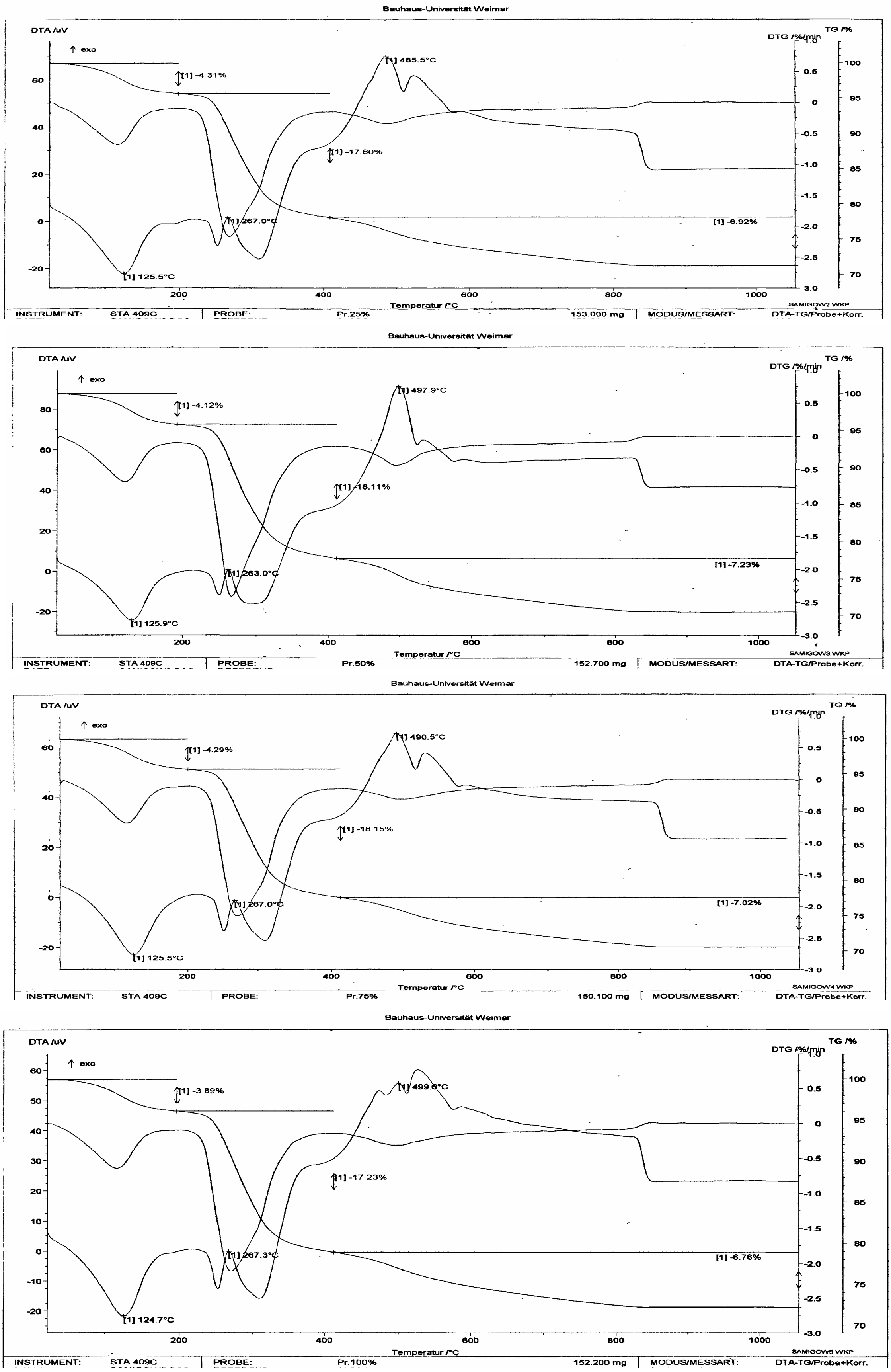

Figure 4. Derivatograms of carbamide compositions

An analysis of the derivatographic data of the studied compositions shows that the change in weight loss after the first effect depends on the amount of hydrogel administered. The greatest weight loss is observed in a composition with a content of 0.09% hydrogel additive by weight of filler, while the highest loss of the second decomposition corresponds to a composition with a content of 0.09% hydrogel additive by weight of filler. In the case of the third stage of decomposition, the greatest weight loss is a composition with a content of 0.09% hydrogel additive by weight of filler.

As can be seen from Fig.4, when the cured carbamide compositions are heated to 830 ° C, a mass loss of 29.46% occurs. The carbamide composition containing 0.05, 0.09, 0.13 and 0.17% of the hydrogel additive by weight of the filler at the same temperature has a weight loss of 29.46, 28.83, 27.88 and 27.5%, respectively. In other words, the more the hydrogel additive binds free water, the less the weight loss of the carbamide composition.

Thus, it should be concluded that the thermal behavior of the compositions depends on the amount of hydrogel added. A special place is given to comparing the nature of the exothermic effects of the second stage, where the shape and temperature values of the effects differ significantly.

Thus, we have established that hydrogen bonds of varying strengths can form at the polymer-filler phase interface. However, their role in the reinforcement process has not yet been definitively established. Based on the ideas about the importance of adhesive phenomena in the reinforcement mechanism, it can be assumed that the formation of a certain number of sufficiently strong bonds is essential here, regardless of their nature.

4. Conclusion

The possibility of obtaining a carbamide composition with quartz filler with improved structure, increased technological, physico-mechanical properties and chemical resistance by using a hydrogel additive in an amount that binds 75% of free water released during resin polycondensation is substantiated.

The positive effect of the hydrogel additive is due to a decrease in the hydrophilicity of the sand filler, a water-binding effect, increased adhesion in the contact zone, plasticization of the polymer system, and improved morphology of the structure and its porosity.

The role of a hydrogel additive in the structure formation of a carbamide composition with a sand filler has been revealed. It is shown that the hydrogel is a modifier of the plasticizing effect, which forms a chemical bond with the components due to the water-binding effect of sealing the structure of the carbamide composition with quartz filler.

A quantitative dependence of the strength of the carbamide composition on the content of hydrogel and hardener, as well as the degree of filling, was obtained, which made it possible to optimize its composition. It was found that the hydrogel content and a slightly lower amount of hardener and the degree of filling have the greatest effect on the strength of the carbamide composition with quartz filler.

Physico-chemical studies have shown that the addition of hydrogel has a positive effect on the formation of the structure. The presence of chemical bonds between the components, a change in the morphology of the structure, a decrease in microporosity, an increase in density and thermal stability of the carbamide composition are shown.

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