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ORIGINAL CONTRIBUTION

Effect of epoxy resin modifications with industrial fillers on wetting and water absorption

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Abstract

Influence of the composition of the cold cured epoxy diane resin of ED-20 brand and its modifications on the water absorption and wetting properties is studied. Organosilicon additives polydimethyl siloxane (PDMS-5, 2.5 wt%), and nano-dispersed amorphous pyrogenic silica (HDK, 0.5 wt%) were used. Industrial fillers such as Volsky sand (EN 196–1), and the slag Portland cement of grade PC 400-D20 are also applied to the epoxy binder modifications for repair and building composites. The introduction of organosilicon additives reduces significantly the water absorption. In particular, the addition of PDMS-5 reduces the water absorption by 22% in comparison with unmodified samples. Water absorption decreases by more than 30% when (PDMS-5 + HDK) gel modifier is added. The introduction of industrial fillers in various proportions and compositions increases water absorption. It is found that the contact angle of the epoxy binder is linearly dependent on the mass fraction of the curing agent. The equation of the contact angle dependence on the curing agent mass fraction sufficiently fitting the experimental data was obtained. The introduction of PDMS-5 reduces the contact angle by 13% in the case of a metal background and 35% in the case of a concrete background. The contact angle decreases by 34% with the addition of (PDMS-5 + HDK) gel modifier in the case of metal background and decreases by 76% in the case of concrete background.

Keywords Epoxy resin · Water absorption · Wetting angle · Fillers · Repair composites · Building composites

Introduction

Epoxy resins are one of the widely used classes of reactive oligomers [1, 2]. The quality of cured epoxy resins is determined not only by the physical and mechanical properties of a material or product [3, 4], but also by chemical and environmental indicators that significantly affect the areas of application, especially in civil engineering, where environmental requirements are decisive [5].

A building material's water/moisture absorption is one of its most important characteristics [6, 7]. The water absorption of resin-based polymers affects thermal and electrical conductivity, strength, corrosion properties, and especially fungus resistance [8, 9]. The latter plays the role of a sanitary and hygienic indicator that is important during operation and repair. Chemical resistance in aggressive environments also depends directly on the ability of the composite material to absorb or not absorb moisture, etc. [10, 11]. The sorption and diffusion of aggressive media into the materials increase with an increase in water absorption. Therefore, resin composite materials with lower water absorption have better performance [12, 13]. The water absorption of the cured epoxy binder with amine hardeners is usually relatively low, which is a great advantage of this class of materials [14].

The binder must ensure good wetting of the surfaces of fillers, aggregates, as well as the surfaces to be glued or repaired if the binder has an appropriate purpose [15, 16]. Interfacial contact and adsorption interaction at the interface of two phases between the binder and the substrate are of great importance for ensuring the strength of the composition, repair, or glue joint [17, 18]. Many characteristics of resins can be improved by the addition of fillers and additives [19–21] including technogenic fillers [22] such as industrial wastes [23] and steel slag [24]. Therefore, it was important to trace the effect of the composition of the binder and modifying additives on the contact angle.

In this paper, the study of the effect of modifying silicon-containing additives on the water absorption indicators of filled

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epoxy composites is carried out. Despite of the properties of industrial epoxy resin of ED-20 brand were studied well [25, 26], recently we present some newly developed molding, filling, and repair compounds based on it [27]. As noted, the selected components reduce toxicity and improve the environmental properties of the proposed repair and restoration polymer compositions. Moreover, the components of the formulations are widely available and relatively inexpensive, which makes them attractive from an economic point of view.

This article presents new results of the study of wetting properties and water absorption of the proposed new compositions. It should be emphasized that such studies have not been carried out for these compositions. It is shown that water absorption can be improved with the introduction of industrial fillers that reduce the porosity of the material and the imperfection of the structure. Wetting properties of cold curing epoxy binder in relation to changes in the mass fraction of the curing agent are also studied for the first time.

Materials and methods

Materials

Epoxy resin ED-20 (CAS No 25068–38-6) as a polymer base for repair compositions is studied with addition of the curing agent (hardener) L-20 (Technical Conditions 6–06-1123–98) in various mass fractions.

The liquid samples were prepared as follows. First, the polydimethyl siloxane (PDMS-5, CAS No 63148–62-9) of 2.5 wt% in relation to the binder [28, 29] is added to the liquid epoxy ED-20+L-20. Next, the nano-dispersed amorphous pyrogenic silica (HDK, CAS No 67762–90-7) of 0.5 wt% in order to improve the resin properties is added. The chemical structures and properties of the materials used could be found in [27].

The Volsky sand conforming to the European standard EN 196–1 is chosen as a model composition for the repair and restoration of building products and structures containing a significant amount of sand. Sand used for the preparation of composites and polymer solutions must contain at least 98% SiO_2 and be used fractionated. It was used fine sand with the following characteristics: the bulk density is 1.26 g/cm^3; the grain size is less than 1.25 mm; the sand fineness modulus is 1.25; and the humidity is 4%.

A low-grade cement is often used as a filler for structural polymer concrete compositions since cement does not work as a binder in combination with a polymer binder, but performs the function of a finely ground mineral filler. For the repair compositions of building products, we have chosen the Portland cement of grade PC 400-D20, the composition of which contains the addition of slag less than 20%. The mass fraction of sulfuric anhydride SO_3 in cement is about 1–3.5%. Such cement has increased chemical resistance. Its physical and mechanical properties are the following: the flexural strength is 5.4 MPa; the compressive strength is 39.2 MPa.

The amount of mineral filler for polymer concrete composition in relation to the specific task of using the composition was chosen depending on the required viscosity of the system for performing restoration and repair work on sealing joints, cracks, etc. The sample preparation and results of mechanical tests of non-modified epoxy resin could be found in [30]. The preparation features of modified samples during ultrasonic processing could be found in [31]. Kinetics of polymerization and glass transition features of epoxy resin modified by mentioned additives and fillers were reported in [32, 33].

Methods of the water absorption measurement

Water absorption of non-modified ED-20+L-20 samples in a ratio of 1:0.9 is measured. Samples of 5 pieces were dried to constant weight before testing, weighed, and measured. The samples are placed in a container filled with water so that the water level in the container is about 50 mm higher than the upper level of the laid samples. The water temperature is (20±2), °C. Samples are weighed every 24 h of water absorption. They are then taken out of the water, wiped off with a damp cloth, and immediately weighed. The mass of water that flowed out of the sample onto the pan during weighing was included in the mass of the water-saturated sample. The tests are carried out until the results of two successive weightings differ by no more than 0.1%. The water absorption of a separate sample by weight in percent is determined by.

$$W = \frac{m_w - m_d}{m_d} \cdot 100\%, \quad (1)$$

where m_d is the dry sample weight and m_w is the mass of water-saturated sample.

Methods of the contact angle measurement

The contact angle is determined by analyzing the shape of the droplet, which is projected onto a screen or measured when viewed through a microscope MIR-2 with an objective. It is used in experiments the attachment to the microscope MIR-2 (developed at the Department of Physical Colloidal Chemistry in Belgorod V. G. Shukhov State Technological University), which allows measuring directly the contact angle of wetting of solid surfaces with polymers. The scheme of experimental equipment is presented in Fig. 1. The attachment includes a graduated goniometer, rigidly fixed to the horizontal tube, and an arrow indicating the wetting angle, which is rigidly fixed to the microscope eyepiece. This device measures the advancing contact angle of a sufficiently viscous liquid resin [34]. In particular, the apparent or measured contact angle on rough (Wenzel) surface is measured [35].

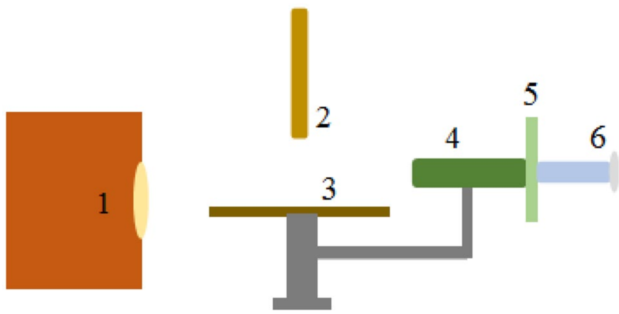

Fig. 1 Scheme of an experimental equipment for measuring the contact angle: 1, light source; 2, pipet; 3, rotating objective table on stand; 4, microscope with an objective; 5, graduated goniometer with an arrow indicating the contact angle; 6, microscope eyepiece

Two different types of surfaces (backgrounds), on which the composite resin samples were applied, are used. The first type of background is a metal surface in order to study wetting properties on the smooth surface of dense material and the second one is concrete grade 300 in order to study wetting properties on a rough surface of a porous material. The roughness of the backgrounds was not measured. Before measurement on a concrete background, it was preliminarily cleaned to visual smoothness.

Various concentrations of the hardener and additives were added to the ED-20 resin, and it was observed how the contact angle changes depending on the concentration of the hardener and additive.

The investigated sample is placed on the rotating objective table on stand. A small drop of sample is carefully applied with a pipette, kept for equilibration for 3 min. The view of the drop and the readings of the goniometer are observed in the eyepiece of the microscope. Then, the eyepiece crosshair is brought to the left edge of the dropped drop, and the goniometer readings are taken. The arithmetic mean of ten measurements in the most stable state on one surface in two mutually perpendicular directions is taken as the result, the discrepancy between which does not exceed 3%.

Results

Influence of modifying silicon-containing micro-additives on water absorption of the epoxy binder

The results of water absorption measurement are presented in Fig. 2. The water absorption of the epoxy binder is significantly reduced with the introduction of silicon-containing modifying additives. The micro-additive PDMS-5 in the composition of the epoxy resin ED-20 in an amount of only 2.5 wt % reduces the water absorption of the binder samples by 22% (from 0.55 to 0.43%). The water absorption of the cured binder samples decreases by more than 30% (from 0.55 to 0.38%) with the addition of the complex gel modifier PDMS-5 (2.5 wt %) + HDK (0.5 wt %).

These results are could be explained by significant changes in the complex of morphological and structural characteristics of the modified binder and are consistent with the conclusions of papers [36–38] on changes in the density and surface hardness of the modified resins.

Liquid monomolecular and oligomeric organic siloxanes play an important role in the structure formation of the epoxy system. In the optimal amount, PDMS-5 fills voids with a disordered zone of the structure, simultaneously affecting the mobility of the polymer chain links at the time of polymerization, facilitating conformational rotations, which helps to overcome steric hindrances during the formation of the initial levels of the structural hierarchy.

The spatial ordering of macromolecules occurs in the interstructural regions because of a decrease in voids and free volume. The regulation of order in amorphous zones occurs because of changes in the conformational set of mobile regions of macromolecules. In any case, filling the resin increases the orderliness

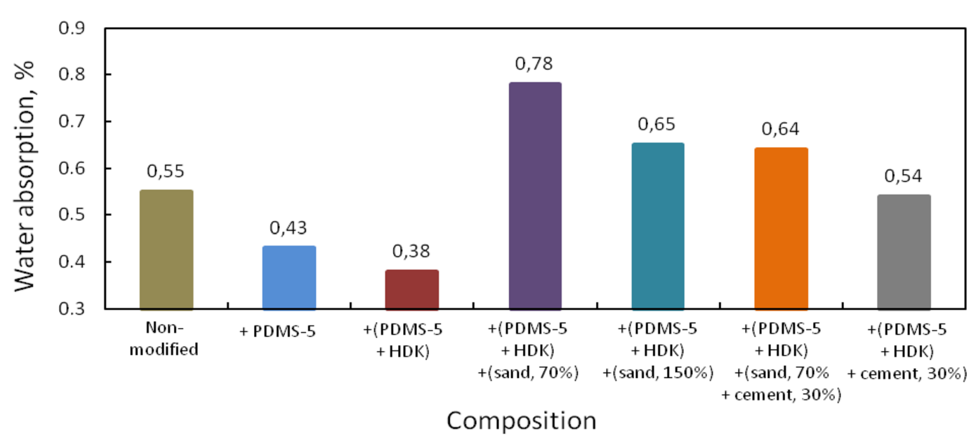

Fig. 2 Water absorption of different compositions based on cold cured resin ED-20 + L-20

of its structure and reduces defectiveness by filling the voids with additives. As a result, due to the difficulty of diffusion of water molecules in the resin with additives, water absorption decreases.

The presence of tightly "cross-linked" globules and a rarely "cross-linked" defective matrix containing unreacted active monomer groups characterizes epoxy polymers. The volume of structural defects does not exceed a few percent. However, they determine the complexity of the most important operational properties and the durability of epoxy materials. During the curing and structuring of the epoxy resin, all foreign substances (additives) are squeezed out of the globules into the defective area of the polymer matrix.

The water absorption is higher for composite materials containing low-profile additives [39]. This behavior is due to the formation of microvoids, which is facilitated by low-profile additives. In our case, the additives represent a highly dispersed mixture with silicon-containing nanoparticles characterized by pronounced hydrophobic properties, which, on the contrary, fill the voids in the resin structure.

Sand-filled composites have slightly higher water absorption than cement-filled composites. This is because, in the case of filling with a more finely dispersed filler with cement, the structure of the composite is denser, that is, it has a denser packing of mineral particles and fewer voids. Sand absorbs moisture to a lesser extent than cement stone. Nevertheless, in our case, as we can see, it was not the nature of the filler that had a greater effect on the water absorption rate, but the nature of the structure of the epoxy binder and the composite as a whole. In our case, it is found that the water absorption of filled epoxy composites is higher and amounts to 0.54–0.78% in comparison with unfilled binder compositions with 0.38–0.55% (see Fig. 2).

The interglobular regions of glass-forming polymers have a lower molecular packing density and a higher defectiveness, including defective regions of the macromolecules themselves. It is these weak zones of the polymer material that need to be strengthened. And since they are the most accessible for diffusion swelling, this creates prerequisites for the modification of polymers by sorbed curing oligomers.

The processes of diffusion and sorption are determined by the structure of the original epoxy resins, since they have a microheterogeneous structure, expressed by topological structure defects with densely reticulated "cores" and rarefied defect zones with an increased concentration of topological defects. In reticulated epoxy polymers, the degree of limited swelling is determined by the parameters of the topology of the molecular network and its defects. The topology of epoxy polymers is changed by adjusting the ratio of the initial reagents, that is, the lack or excess of amine groups. The thickness of the diffusion layer in sparsely reticulated samples is less than in densely reticulated ones, although the degree of swelling by weight is higher in them. This corresponds to a higher concentration of the diffusant in the gradient layers of the samples obtained with an excess of amine groups.

Influence of the composition of the binder and modifying additives on the wetting contact angle of the epoxy binder

The experimental results are presented in Fig. 3 showing the graphical dependence of the influence of the amount of L-20 curing agent on the contact angle of wetting of the binder ED-20 + L-20. The wetting contact angle monotonically decreases with an increase in the content of L-20 curing agent in relation to the epoxy resin ED-20 in the case of concrete background, and it monotonically increases in the case of metal background.

The result is quite obvious and explainable since L-20 curing agent is a product of vegetable oil production and contains organic functional groups in the molecule that reduce the surface tension of the binder. In this case, an increase in strength at the contact boundary-filler (substrate)-binder can be expected.

It is found that experimental dependencies of the contact angle θ on the mass fraction of the curing agent c are sufficiently approximated by a linear equation, which is given by:

$$\theta = s \cdot c + \theta_0, \tag{2}$$

where θ_0 is the contact angle corresponding to zero mass fraction of the curing agent and s is a speed of contact angle change with the mass fraction of the curing agent:

$$s = \frac{d\theta}{dc}. \tag{3}$$

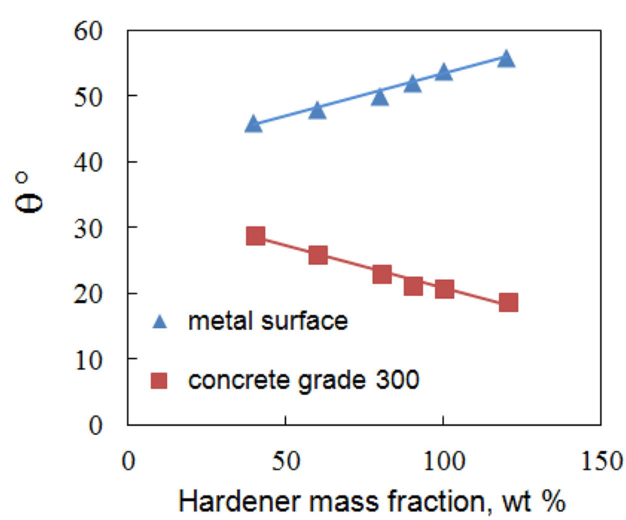

Fig. 3 The experimental (markers) and theoretical (solid line plotted accordingly with Eq. (2)) dependencies of contact angle on the hardener mass fraction of non-modified binder ED-20 + L-20

Equation (2) makes it possible to estimate the contact angle and speed (2) versus the mass fraction of the curing agent. The parameters s and θ_0 are calculated by the least-squares method so that the theoretical Eq. (2) would best fit the experimental data. The following values of parameters of Eq. (2) are calculated in the cases of two backgrounds, which are presented in Table 1.

The functions (2) with calculated parameters for two backgrounds are plotted in Fig. 3 (solid lines), which demonstrate the sufficient fit the experimental data (markers).

The speed (3) is positive ($s > 0$) for metal background (see Table 1), which indicates an increase in contact angle with an increase in the mass fraction of the curing agent. The speed (3) is negative ($s < 0$) for concrete background (see Table 1), which indicates a decrease in contact angle with an increase in the mass fraction of the curing agent. We see that the backgrounds significantly determine the dependence of the wetting contact angle on the mass fraction of the curing agent.

Thus, we find the equation to describe the change in contact angle of prepared fillers and composites versus mass fraction of the curing agent for two backgrounds. The equation can be used to predict the contact angles.

The formation of a porous structure after it has the curing agent for a very long time leads to an increase in the contact angle. The authors of [40] argue that the increase in hydrophobicity occurs due to the formation of porous, tangled irregular microstructures that create air cushions on the surface leading to the transition of the droplet state from Wenzel to Cassie.

Note, that our papers [30, 33] present the results of varying resin properties as a function of the addition of curing agent. In particular, the results of studies of glass transition temperature of ED-20 resin in dependence on the mass fraction of L-20 curing agent were presented in [33]. The data presented in this paper make it possible to establish a relationship between the glass transition temperature T and the contact angle θ (Fig. 4). An analysis of this relationship indicates that contact angles in the range from 48° to 52° correspond to a constant glass transition temperature of about 80 °C. In addition, the empirical equation coupling the glass transition temperature with the contact angle is

$$T = -0.0167 \cdot \theta^4 + 3.4785 \cdot \theta^3 - 270.34 \cdot \theta^2 + 9319.1 \cdot \theta - 120165, \quad (4)$$

which is calculated by the least-squares method in order to the best fit experimental data (calculations show the value of $R^2 = 0.9989$, which indicates that Eq. (4) describes the experimental data well). The graph of Eq. (4) corresponds to the solid line in Fig. 4.

Table 1 Values of parameters of Eq. (2)

Background	s (° per %)	θ_0 (°)	R^2
Metal surface	0.130	40.371	0.983
Concrete grade 300	−0.128	33.696	0.982

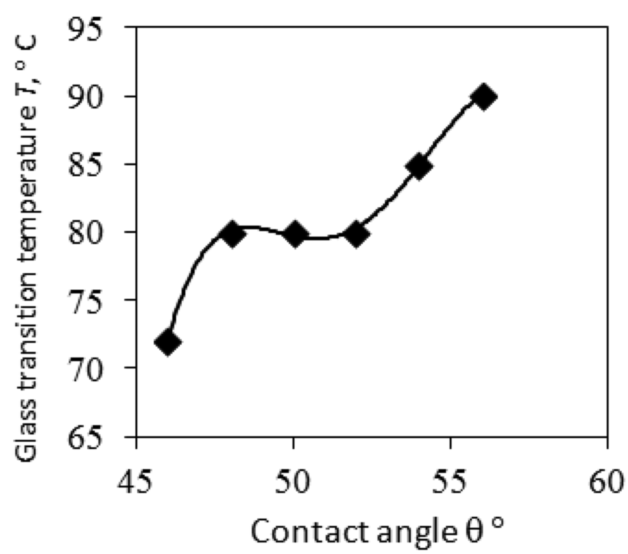

Fig. 4 The experimental (markers) and theoretical (solid line plotted accordingly with Eq. (4)) data of the glass transition temperature versus the contact angle

It has been established in the [30] that the maximum values of mechanical characteristics correspond to the corresponding interval 78–95 wt% of the mass fraction of the curing agent. Therefore, the range 48°–52° of the contact angle corresponds to the maximum values of impact strength and hardness of resin (Fig. 5). Note that the athermal glass transition plateau corresponds to the maximum mechanical characteristics.

The contact angle decreases with the addition of modifying silicon-containing additives containing functional groups (Fig. 6), which is obviously associated with an increase in

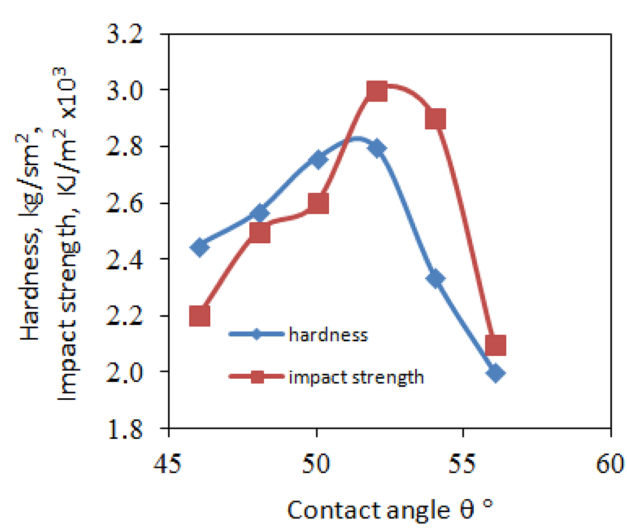

Fig. 5 The resin hardness and impact strength versus the contact angle

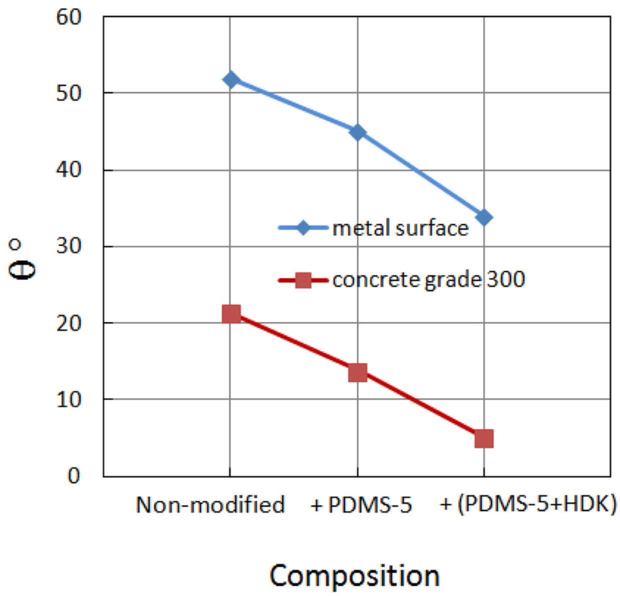

Fig. 6 The contact angle of different compositions based on cold cured resin ED-20 + L-20

the cohesive strength of the binder and an improvement in its adhesion properties.

The micro-additive PDMS-5 in the composition of the binder ED-20 + L-20 (1:0.9) in an amount of only 2.5 wt % reduces the contact angle of the binder samples by 13% (from 51.98° to 45.01°) in the case of metal background, and it reduces the contact angle of the binder samples by 35% (from 21.41° to 13.83°) in the case of concrete background.

The contact angle of the cured binder samples decreases by 34% (from 51.98° to 34.00°) with the addition of the complex gel modifier PDMS-5 (2.5 wt %) + HDK (0.5 wt %) in the case of metal background and decreases by 76% (from 21.41° to 5.13°) in the case of concrete background.

The main factor for creation enhanced adhesion phenomena is the formation of strong bonds and their number, which is necessary to achieve optimal properties. The mobility of macromolecules in the boundary layer decreases with a large number of bonds obtained by increasing the content of the modifying additive. As a result, internal stresses increase and the structure of the surface layer changes. This leads to the appearance of a larger number of defective areas. Such areas are the centers in which the destruction of adhesive bonds begins.

Conclusion

Wetting properties of epoxy resin in dependence on its composition and modification with fillers were described. Water absorption and the contact angle of ED-20 + L-20 for the difference ratio between resin and the curing agent, and with the addition of nanoparticle silicon-containing PDMS-5 and HDK modifiers, were measured. The degree of filling with mineral fillers was determined experimentally, based on the required viscosity and workability for a specific purpose of the composition such as molding, repair, lacquer coating, polymer concrete, primer, putty, etc.

It was found experimentally that the water absorption of the epoxy binder is significantly reduced with the introduction of silicon-containing modifying additives. The addition of mineral fillers, such as sand and cement, leads to an increase in water absorption. The addition of PDMS-5 reduces the water absorption by 22% in comparison with unmodified samples. Water absorption decreases by more than 30% when (PDMS-5 + HDK) gel modifier is added.

The studying of the wetting contact angle shows that it monotonically decreases with an increase in the content of the curing agent in the case of contact the binder with a rough surface of a porous material and it monotonically increases in the case of contact the binder with a smooth surface of dense material. The contact angle decreases with the addition of PDMS-5 and HDK modifiers in both cases of background surfaces.

It was obtained that the linear equation describes the dependence of the contact angle on the mass fraction of the curing agent. The coefficients of the linear equation are calculated to sufficiently fit the experimental data. The proposed equation can be useful for the prediction of the contact angle of the epoxy binder. The introduction of PDMS-5 reduces the contact angle by 13% in the case of a metal background and by 35% in the case of a concrete background. The contact angle decreases by 34% with the addition of (PDMS-5 + HDK) gel modifier in the case of metal background and decreases by 76% in the case of concrete background.

The results presented in this paper expand the studies of water absorption and wetting properties of epoxy modifications [27, 32, 33], and it can be useful for design new perspective polymer composites of repair purposes [41–43].

Declarations

Competing interest The authors declare no competing interests.

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