

**ASSESSMENT OF THE AGGREGATES IMPACT
ON THE PROPERTIES OF RECOVERY POLYMER MORTARS**

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Abstract. The peculiarities of polymer mortar application for renovation and restoration are determined on the basis of priority data integration about the destruction mode of valuable historical buildings. The possibilities of controlling technological, physical-mechanical, and operational properties of recovery polymer mortar due to the aggregates of different nature are shown.

For the analysis and optimisation, the quantitative relations between the structure and property factors of recovery polymer mortars and the factors of recipes and technology determining them were obtained in the form of experimental and statistical models calculated using the COMPEX system. The optimisation methods of recipe and technological solutions based on the use of experimental and statistical models are proposed.

The optimisation of polymer mortar composition according to the package of quality indexes and property stability at high temperature has been carried out. The package of "mixture-technology-properties" models has been obtained, with the help of which the change mechanisms of direct and summarizing indexes of mixture technological properties and mechanical properties of recovery polymer mortars have been established when changing the type of aggregates (ceramics, quartz, carbonates, their binary and triple mixtures).

The influence of aggregates on the durability change of polymer mortar under the influence of various temperature-climatic and operational factors (UV-irradiation, alternate action of temperature and aggressive aqueous solutions) has been studied.

It is recommended to use quantity and type optimal aggregates to provide the complex of technological and operational properties of polymer mortar and to reduce the consumption of imported polymer. The rational compositions of polymer mortar with increased stability of properties under changing temperature and climatic conditions are proposed for different restoration technologies.

The series of nomograms have been developed for the initial selection of the "area" of rational polymer mortar compositions, providing for further correction in relation to a specific repairable object. Technological and marketing analysis according to the research results are carried out.

Key words: recovery polymer mortar, aggregates, rheological properties, strength, optimal compositions, operational properties.

Introduction. The preservation of architectural monuments from further destruction is the goal of a number of international programs performed under UNESCO auspices. One of the main tasks of restoration procedures is to provide the life of buildings and structures while preserving their historical and architectural authority [1]. The great amount of restoration and conservation works, complex nature of structural destruction pose new scientific, methodological, and practical challenges. To solve these problems the special technological methods and materials are required. The composite materials in the form of protective and structural polymer mortars, as the world experience shows, are one of the most long-range materials for repair and restoration of stone and concrete structures. The introduction of the aggregates allows to change the technological and operational properties of polymer mortars in a wide range. The development and introduction of

effective polymer mortars based on the use of epoxy oligomers and aggregates of different nature is an urgent task while improving the technology of restoration work [2, 3].

Analyses of recent studies and publications. By means of research, as well as on the materials of international conferences and literature sources, the causes and classification of the main destructions of stone buildings and structures have been considered [4-8]. The first result was the classification of the most characteristic destructions to be repaired and restored according to the following characteristics: loss of monolithicity, delimitation of stone masonry, deep and through cracks, spalling, weathering, stone failure, etc.

The technological peculiarities of restoration works are determined by the variety of object destructions (which requires the use of different restoration materials with variable technological and physical-mechanical parameters), as well as the climatic pattern. The most important engineering tasks (taking into account the quality of engineering staff) include obtaining materials with the required properties, saving critical imported materials, as well as the related issue of decision validity.

According to the world experience, one of the most effective ways of repair, protection and restoration of building structures is the use of polymer-based composite materials [4-7]. One of the main prerequisites for the effective use of polymer mortars for the restoration purposes is the development of the scientific and practical base of polymer composites (V.V. Paturoev, V.I. Solomatov, V.L. Chernyavsky, V.A. Lisenko, I. Nikolov, R.A. Veselovsky, B.N. Strelenko, I.M. Yeshin, etc.).

One of the long-range ways to improve the quality and durability of composite materials and to save the binder is the use of modifying additives and aggregates, optimal both in concentration and in their physical and mechanical parameters. The use of the aggregates becomes extremely important under the conditions of expanding the requirements to the properties of restoration polymer mortars (RPM) (including the achievement of light and texture similarity), regulating the speed of the technological process and the variety of restoration techniques.

Based on the analysis results of characteristic destructions of buildings and structures, the codes and recommendations in the field of restoration of historical monuments, a possible package of technological solutions for the use of polymer mortars for repair and restoration of structures is proposed. The possible package of restoration works includes: reinforcement of existing foundations; monolithicity restoration of structural elements; replacement of destroyed material with new one; bonding of fallen stones and elements; grouting of delaminated masonry; filling of the cracks with decorative polymer mortar; grouting of the cracks with the injection, etc.

The aim of this study is to develop the compositions of recovery polymer mortar with the required complex of technological, physical and mechanical, and operational properties when using the aggregates of different nature and determination of the range of rational compositions that meet the requirements: effective viscosity within $50 \leq \eta_{\max} \leq 900$ Pa·s, compressive strength $f_{\text{ctfm}} \geq 90$ MPa, bending strength $f_{\text{cm}} \geq 60$ MPa, stability coefficient of RPM properties to temperature $K_t=100$ %.

Objects and methods of research. Epoxy resin ER-20 was used as a binder in the experiments; its parameters are similar to the common foreign analogues (Araldite, Epicol, Epoxy); an amine-type hardener and plasticiser dibutyl phthalate (DBP) were used in the experiments.

The optimisation was carried out using "mixture-technology-properties" models [9]. The properties of filled epoxy polymer mortars were determined by:

V_i – the aggregate fraction of a certain (I-st) kind, with a fractional composition corresponding to fine quartz sand:

V_1 – the ceramic aggregate; V_2 – the carbonate aggregate; V_3 – the quartz aggregate; $\sum V_i = V_1 + V_2 + V_3 = 1$, i.e. the factors are interdependent and form a "mixture";

X_1 – the amount of plasticiser DBP (wt.h) – $X_1=30 \pm 10$ (20, 30, 40);

X_2 – the aggregate degree – $X_2=200 \pm 100$ (100, 200, 300); the factors X_1 and X_2 are interdependent and can be referred to the term "technology". The experiment was implemented according to a specially synthesized plan (Table 1) [9] in the COMREX system.

Table 1 – Experimental plan

№	Fillers, part			The amount of plasticiser DBP, (wt.h)	The filling degree, (wt.h)
	ceramic	carbonate	quartz		
	V ₁	V ₂	V ₃		
1	0.33	0.33	0.33	x ₁	x ₂
2	0	0	1	1	-1
3	0	0	1	-1	0
4	0	0	1	1	1
5	0	1	0	1	-1
6	0	1	0	1	1
7	0	1	0	-1	-1
8	1	0	0	-1	0
9	1	0	0	1	-1
10	1	0	0	1	1
11	0	0.5	0.5	-1	1
12	0	0.5	0.5	0	-1
13	0.5	0	0.5	-1	-1
14	0.5	0	0.5	0	0
15	0.5	0.5	0	0	1

The models in the form of reduced polynomials were obtained for the technological parameters of mixture quality and physical and mechanical properties of hardened polymer mortars, including the effective viscosity, flexural and compressive strength, and dynamic modulus of elasticity. The effective viscosity η was determined with the rotational viscometer "Polymer RPE-1M", which provided viscosity measurement in the range of $1.8 \times 10^{-3} \dots 3.75 \times 10^4$ Pa·s. Bending strength f_{cm} and compression strength f_{ctfm} (MPa) were determined with test beams $4 \times 4 \times 16$ cm; one set of test beams was tested after 10 days after manufacturing, the other one after 60 days stored at the temperature $T=80^\circ\text{C}$. The modulus of elasticity E was determined from the initial linear section of the "load-s" diagram obtained according to step loading with the highest possible average velocity, which provided that strain readings were taken using two lever strain gauges mounted on the opposite edges of the test beams.

Research results. Based on the literature data, the main requirements for recovery polymer mortars were determined. Thus, the viscosity of technological mixtures should vary in a wide range. Its choice depends, first of all, on the method of polymer mortar introduction into the caverns, cracks or open surfaces (injection, hand patching, coating or spraying with the aggregates) [10]. As a rule, the polymer mortars with the bending strength not less than 50-83 MPa and compressive strength 80-100 MPa are used. The important parameters determining the behaviour of polymer mortar in the structure include the bond strength of the RPM with the permanent material, the ratio of elastic moduli and other parameters. The properties of RPM due to the polymer aging can be irreversibly changed under the influence of temperature, humidity, wind loads, ultraviolet irradiation, and other climatic factors [11-14].

Under the conditions of insufficiently studied mechanism of structure formation and destruction of composites and multicriteriality of accepted engineering decisions the most effective approach to the optimisation of their properties is the application of experimental and statistical models (ES-models) [9, 15-17]. There is considerable experience in the application of ES-models in the study and optimisation of composites for various purposes, including polymer mortars for protection, repair and restoration.

At the second stage, the quantitative dependences of the main RPM properties were investigated using ES-models package. Their analysis allowed to choose rational concentrations of the aggregates of different nature for RPM range. The choice of specific types of aggregates (fine brick rubble, limestone grits, fine sands) was determined on the basis of literature data.

The values of effective viscosity η (Pa·s), which characterises the structure-forming ability of the aggregates and was obtained at constant strain ($\dot{\epsilon}=\text{const}=1\text{c}^{-1}$), were comparable characteristic of aggregate surface activity.

The obtained experimental-statistical dependences of the effective viscosity are plotted in Fig. 1 in the form of multicomponent diagrams-tetrahedrons, which were combined with the concentration space "amount of DBP plasticiser – degree of filling".

$$\begin{aligned} \ln \eta = & 6.29V_1 + 2.73V_1V_3 - 1.29V_1x_1 + 1.88V_1x_2 + 0.32x_2^2 \\ & + 4.73V_2 - 0.61V_2x_1 + 1.49V_2x_2 + 0.23x_1x_2 \\ & + 2.67V_3 - 0.58V_3x_1 + 0.63V_3x_2 \end{aligned}$$

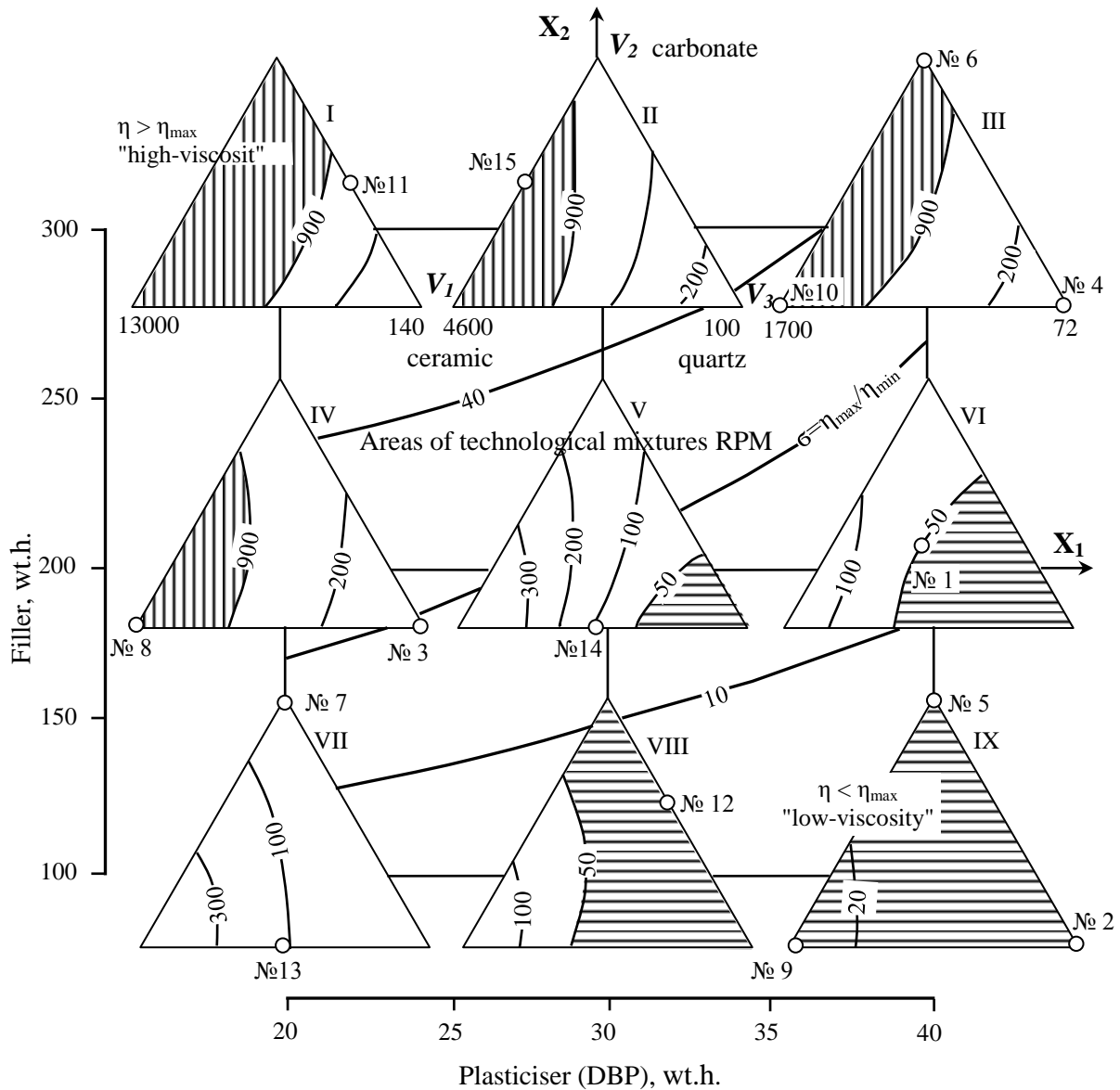


Fig. 1. Nine mixture triangles for the effective viscosity (Pa·s) of the filled polymer mortar constructed according to the model

The base element is a mixture triangle, on which the viscosity isolines η are shown. The "high-viscosity" ($\eta_{max} > 900$ Pa·s) or "low-viscosity" ($\eta_{max} < 50$ Pa·s) areas of polymer mortars are shaded inside each of the nine triangles.

From the analysis of the diagram there are the following technological conclusions. They are necessary for the choice of ways to regulate the technological properties of mixtures:

- to obtain technological mixtures ($50 \leq \eta_{max} \leq 900$ Pa·s) it is necessary, as a rule, to use

average concentrations of aggregates and plasticiser; the working area is limited "at the top" by high-viscosity mixtures with the ceramic aggregate, and "at the top" by low-viscosity mixtures with the quartz aggregate;

- maximum viscosity η_{max} achieved by varying the aggregate type (mixtures do not flow practically) is determined by the ceramic aggregate V_1 , which is explained by high surface activity of this aggregate and, as a consequence, by more complete formation of oligomer supramolecular structures;

- the increase of the quartz aggregate proportion leads to a sharp decrease of the viscosity; for the mixtures V_1+V_2 , the concentration increase of the carbonate aggregate causes a significant viscosity increase of polymer solutions (on the triangles I-III);

- the interaction of ceramic and quartz aggregates makes it possible to regulate the rheological properties of polymer mortars within the wide limits; the analysis of the model coefficients η shows that the greatest effect of structure formation is achieved with the interaction of V_1 and V_3 .

The sensitive shift of viscosity to the variation of aggregate type was investigated using the composite index $=\eta_{max}/\eta_{min}$.

The isolines of the model on the supporting square (Fig. 1) indicate a sharp increase of the system sensitivity to the variations of the concentration and aggregate type – ceramic and quartz.

According to the results of the experiment in 15 experimental points (marked in Fig. 1) the models for the mechanical properties of hardened compositions were calculated. The models described the change of bending strength f_{cm} , the compression f_{ctfm} and the elastic modulus E of polymer mortars depending on the type and amount of considered aggregates. In the whole range of investigated concentrations, with the increase of aggregate degree, the strength factors increase for RPM with ceramic aggregate; they decrease or do not change for RPM with the carbonate, quartz aggregates and their mixture. This difference is explained by the unequal rate of polymer transition from the bulk state to the film state depending on the surface activity of aggregates.

As follows from the analysis of viscosity fluctuation in Fig. 1, the ceramic aggregate has the highest surface activity in the polymer matrix, since $\eta \rightarrow \max$. At the same time, it provides the highest compressive strength. Thus, in comparison with quartz, the introduction of the ceramic aggregate into RPW ($X_1=30$, $X_2=200$ wt.h.) increases the strength by more than 1.3 times, which follows from the analysis of the mixture triangle in Fig. 2, a. Its mixing with the quartz aggregate is very effective to increase the bending loads resistance; in particular, $f_{cm,max}$ for the same compositions corresponds to the binary mixture V_1+V_3 (53% + 47%).

The elasticity modulus decreases with increasing DBP plasticiser amount, firstly for RPW containing carbonate aggregate. It increases when the quartz aggregate is used. The difference between deformative and elastic properties of permanent materials and polymer mortars significantly affects the workability of RPW. Thus, it is necessary to take into account that at $E \rightarrow \min$, the polymer mortars have plastic properties which are necessary to reduce internal stresses. Such stresses arise, for example, at the difference between temperature coefficients of the linear expansion of polymer mortar and the construction material (concrete, stone). The stiffening effect ($E \rightarrow \max$) reduces the difference between the elastic modulus of the materials, which contributes to the reduction of stress concentration in the contact zone "polymer-permanent material".

The secondary analysis allowed to find out the dependence between the recipes and the kinetics of internal stresses development. The studies were carried out on a special measuring complex, which allows to perform simultaneous measurements on 24 samples with the deformation fixation using MI-2 microscope ("cantilever method").

In particular, it was found that regardless of the composition and structure of the polymer mortar, the first 4-6 days from the moment of preparation are characterised by a sharp stress increase. In the following 6-10 days a relative stress stabilisation is observed, after which for different compositions the value of internal stresses decreases by 10-50 %. At the age of polymer mortar more than 25 days the level of stresses remains practically unchanged, which indicates the completion of the process of polymer solution structure formation. The compositions, in which quartz sand was used as an aggregate, are characterised by a lower value of internal stresses than compositions with other aggregates.

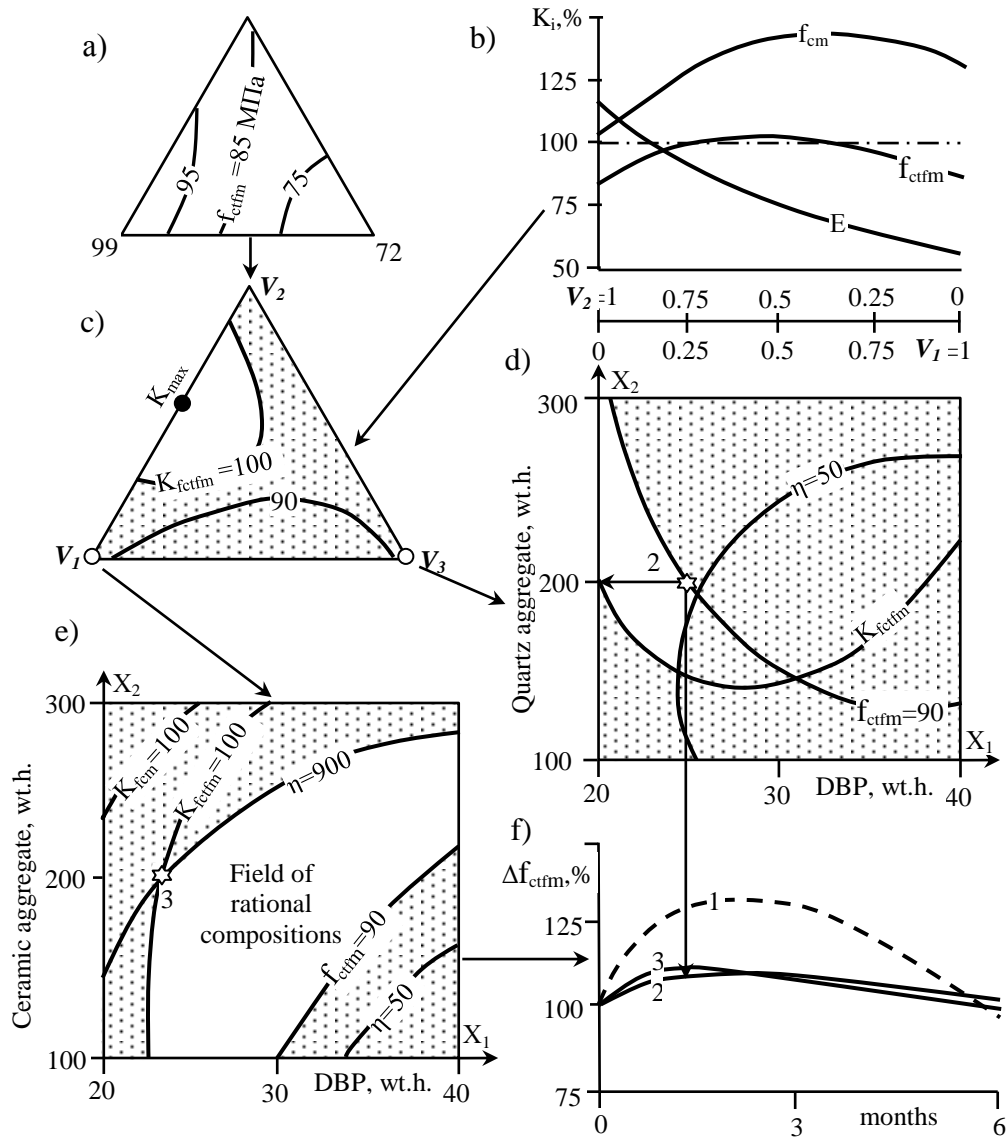


Fig. 2. Complex analysis of aggregates influence on the properties of RPW: (a) compressive strength f_{ctfm} in the mixture diagram; (b) property stability coefficient "K" when going from ceramic (V_1) to carbonate (V_2) aggregate; (c) compressive strength stability coefficient K_{fctfm} for the aggregates V_1 , V_2 and V_3 ; (d) optimal region of quartz-filled PRP; (e) optimal area with ceramic aggregate; (f) fraction change of f_{ctfm} under UV-irradiation (1 – unfilled PRM, 2 – quartz aggregate; 3 – ceramic aggregate)

The properties of restoration polymer mortars must meet the necessary requirements. Since the properties of RPM during the operation can irreversibly change, and this will be reflected on the durability of structures. The most significant external effects include high temperature of the environment. Under the influence of temperature, there are the irreversible physical and chemical transformations, leading to decrease the performance criteria of the material.

In order to assess the resistance of RPM properties to temperature, the comparative analysis of polymer mortars of the initial state (P_0) and after 60 days at the temperature $T=80^\circ\text{C}$ (P_T) was carried out. The stability index was determined as:

$$K = \frac{P_T}{P_0} 100\%.$$

In Fig. 2, b there is K coefficient variation for polymer mortars containing ceramic and carbonate aggregates, as well as their mixtures. The greatest variations of RPM properties under the influence of temperature occurred for those compositions in which the carbonate aggregate was used. The analysis of the isolines of K coefficient on the mixture triangle (Fig. 2, c) allowed to conclude from the effectiveness of mixing ceramic and carbonate aggregates to increase the stability of the compressive strength f_{ctfm} of the polymer mortar under the prolonged temperature effect. In the unshaded area ($K \geq 100\%$), the polymer mortars are characterised by stable strength.

The joint analysis of the "stability" index models f_{cm} и f_{ctfm} allowed to determine the compositions of polymer mortars that keep the required quality level under the influence of temperature. The appropriateness of mixing aggregates, first of all, ceramic and carbonate aggregates, to increase the stability of RPM properties is shown.

In order to select optimal compositions (Fig. 2. d, e), the isolines of "desirable" viscosity, strength and the boundary isolines of stability of strength parameters were superposed on the general diagrams, in particular, for the polymer mortar with quartz (Fig. 2, d) and ceramic (Fig. 2, e) aggregates. In the field of rational compositions the polymer mortars meet the following requirements: technological mixtures – viscosity 50...900 Pa.s; hardened composites: $f_{cm} = 60...80$ MPa, $f_{ctfm} = 80...100$ MPa, $E = 1.3...1.6$ GPa, $K = 100\%$.

The saving of polymer binder due to simultaneous introduction of two aggregates while maintaining the specified quality and reducing the impact on temperature factors is achieved by increasing the amount of quartz aggregate from 150 to 300 wt.h. (Fig. 2, d) or ceramic aggregate from 100 to 280 wt.h (Fig. 2, e). The narrower variation range of quartz and plasticiser aggregate (20...25 wt.h) increases the requirements to materials proportioning. The characteristic variation ranges of RPM compositions are shown in Table 2.

Table 2 – Variation ranges of RPM compositions

Type of filler (equal parts in the mixture)	Mixture viscosity	Content wt.h.	
		filler	plasticiser
carbonate	average	200...300	23...25
ceramic + carbonate	increased	100...200	30...40
carbonate + quartz	reduced	150...250	20...25
ceramic + quartz	average	100...230	20...25

As follows from the performed analysis, the nomograms for the initial reasons of the recovery polymer mortars recipes with complex filling were developed, which are oriented towards further refinement in relation to specific objects.

In addition to high temperature, the most significant factors causing RRM destruction include sun effect (for exposed surfaces), as well as alternating temperature effect and aggressive aqueous solutions (for building pedestals, foundations, etc.).

The experimental studies to assess the effect of aggregates on the durability of polymer mortars with the rational composition (Fig. 2. d, e) were carried out: a) under UV irradiation for 6 months; b) under moistening in aqueous solution (concentration of sulfate ions – 20 g/l.) and drying under hot blast for 300 cycles. To simulate solar radiation, the irradiation stand of the Agrophysical Institute with the ultraviolet and fluorescent lamps was used, providing the irradiation level of the samples up to 160 W/m^2 .

As the conducted researches have shown, under the influence of UV-irradiation the polymer mortars undergo almost the same changes as under the action of repeated temperature and humidity loads. The decrease of the factors is explained by a very significant role of thermo- and photo-oxidative processes in changing the supramolecular and chemical structures of the polymer. After some increasing during the initial exposure period, the bending strength of unfilled polymer mortar decreases sharply thereafter (curve 1, Fig. 2, e). The short-term process of "burst" strength was

explained by the continuing binder hardening processes under the influence of temperature. The rapid strength decrease may be due to the oxidative breakdown of the polymer binder and aging of the polymer. The introduction of ceramic (curve 2) and quartz (curve 3) aggregates into the polymer mortar prolongs to the strength retention under UV irradiation.

At the same time, the lightfastness of the tested samples was investigated. The most noticeable changes of the colour and gloss after UV-irradiation were in the samples containing carbonate aggregate, to a lesser extent – ceramic and quartz. The samples with carbonate aggregate quickly lose their appearance; "spider lines", as well as chalking, appear on the surface, which is due to intensive destruction of polymer bonds in a thin surface layer.

The use of the studied aggregates allows to stabilise the properties of polymer mortars under the combined effect of temperature and aggressive liquid media. Thus, the tests have shown that the introduction of carbonate aggregate stabilises the elastic modulus E more than ceramic aggregate. The process of properties changing also has an extreme character. The appearance of the extremum depends on the type of aggregate. Some strength increase of polymer mortars during the initial period of exposure is explained by temporary relaxation and more uniform distribution of internal stresses in the material amount.

Conclusions. The source and special features of damage have been determined and a possible package of typical technological solutions for the use of polymer mortar for repair and restoration of damaged structures has been considered.

The model of adjustment nomograms have been developed, which are oriented on further specification of rational compositions of recovery polymer mortars in relation to a specific object of repair – renovation.

The influence of aggregates with different nature on the complex of technological and physical-mechanical properties of recovery polymer mortar has been determined; the possibility of controlling the polymer mortar properties by using aggregates of different nature has been shown.

The rational compositions of technological polymer mortars are proposed for restoration works, in particular, with the compressive strength 80-100 MPa and the bending strength 60-80 MPa at minimum internal stresses, with increased stability of mechanical properties under the influence of high environmental temperature.

The positive influence of rational aggregates on the polymer mortar resistance to the temperature and climatic factors, including UV-irradiation, alternating effects of temperature and aggressive aqueous solutions is shown.

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ОЦІНКА ВПЛИВУ НАПОВНЮВАЧІВ НА ВЛАСТИВОСТІ РЕМОНТНО-ВІДНОВЛЮВАЛЬНИХ ПОЛІМЕРРОЗЧИНІВ

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Анотація. На основі пріоритетного узагальнення даних про характер та ступінь пошкодження цінних історичних будівель визначено особливості застосування полімеррозчину для ремонту та реставрації. Показано можливості керування технологічними, фізико-механічними та експлуатаційними властивостями реставраційного

полімеррозчину за рахунок наповнювачів різної природи.

Для аналізу та оптимізації кількісні співвідношення між показниками структури та властивостей ремонтно-відновлювальних полімеррозчинів та визначальними їх факторами рецептури та технології були отримані у вигляді експериментально-статистичних моделей, розрахованих з використанням системи COMPEX. Запропоновано методи оптимізації рецептурно-технологічних рішень, що ґрунтуються на використанні експериментально-статистичних моделей.

Проведено оптимізацію складу полімеррозчину за комплексом показників якості та стабільності властивостей при підвищеній температурі. Отримано комплекс моделей типу "суміш-технологія-властивості", за допомогою яких встановлено закономірності зміни прямих та узагальнюючих показників технологічних властивостей сумішей та механічних властивостей ремонтно-відновлювальних полімеррозчинів при зміні виду наповнювачів (кераміка, кварц, карбонати, їх бінарні та потрійні суміші).

Досліджено вплив наповнювачів на зміну стійкості полімеррозчину при дії різних температурно-кліматичних та експлуатаційних факторів (УФ-опромінення, поперемінний вплив температури та агресивних водних розчинів).

Для забезпечення комплексу технологічних та експлуатаційних властивостей полімеррозчину та зниження витрати імпортованого полімеру рекомендовано використовувати оптимальні за кількістю та видом наповнювачі. Для різних технологій реставраційних робіт запропоновано раціональні склади полімеррозчину з підвищеною стабільністю властивостей за зміни температурно-кліматичних умов.

Розроблено серії номограм для первинного вибору "області" раціональних складів полімеррозчину, що передбачають подальше уточнення стосовно конкретного об'єкта ремонту та реставрації. Проведено технологічні та маркетингові опрацювання за результатами досліджень.

Ключові слова: ремонтно-відновний полімер, наповнювачі, реологічні властивості, міцність, оптимальні склади, експлуатаційні властивості.

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