Development of an Analytical and Graphic Method for Determining the Compositions of Epoxy Compounds with Increased Chemical and Biological Resistance

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Abstract. The task of developing durable and effective building materials for protection against aggressive environments is urgent. One of the ways to increase the durability of buildings and structures is the use of polymer composite materials during their construction. In addition, the urgent task of recycling waste from construction and chemical industries also has to be considered. Determination of rational compositions of composite materials manufactured by compression and vibrocompression methods is of considerable interest. The problems of obtaining compositions of polymer composites based on the use of a modifying additive - a plasticizer (organosilicon varnish KO-922) and asbestos-containing fillers based on waste from construction and chemical industries are addressed. An analytical and graphical method for calculating the compositions of epoxy composite materials based on production wastes has been developed. Rational formulations based on modified epoxy binder have been obtained. The nomograms make it possible to make a practical choice of one or another filler composition for given conditions.

1. Introduction

At present, new chemical and biological technologies are widely used in industry, and the number of enterprises with aggressive production environments is growing. Therefore, the task of increasing the volume of production of durable and effective building materials capable of ensuring long-term and reliable operation of structures in corrosive environments becomes extremely urgent.

One of the ways to increase the durability of buildings and structures is the use of polymer composite materials (PCMs) during their construction. The scope of these materials in construction is steadily expanding. In connection with the emergence of new chemically resistant materials, it is proposed to use them to protect against harmful natural and man-made influences [1, 2, 3, 4, 5, 6, 7].

A rational technology for obtaining materials and products from composite materials (CMs) should be based on the following basic principles: preparation and use of fillers of the required dispersion and physical and chemical activity; widespread use of multifunctional additives, plasticizers, surfactants; clear fractionation of aggregates and the use of their mixtures, selected according to the rules of discontinuous granulometry; optimal filling of binders in order to provide a set of required properties of CM; compaction of mixtures and the appointment of modes of heat treatment of products, taking into...
account the provision of optimal conditions for structure formation at all levels of structure for a given CM. Taking into account the consideration of the last principle, it is important to formulate the prerequisites for obtaining materials by compression method and the manufacture of frame polymer concretes based on epoxy matrices filled with production waste.

When creating composite materials, a set of tasks is solved: achieving high operational and technical indicators, ensuring the manufacturability of the compositions themselves and the process of their manufacture, efficiency, environmental value. Depending on the specific task, the technologist finds one or another optimal value. Having obtained the information on the technical, economic, and environmental performance of the components that form the composite, the researcher must narrow the area of optimum to the level of a consistent relationship: from composition to technology then structure and properties. Following this chain, the optimal composition of the material is determined.

Despite the increasing rates of use of composite polymer binder-based materials in construction, some problems of their structure formation and durability in chemical and biological aggressive environments, unfavorable climatic factors remain poorly understood. In addition, polymer composites have a high cost, sometimes many times higher than the cost of materials based on cement, gypsum and other mineral binders. This problem is solved by various methods, for example, through the use of modifying additives and aggregates based on local raw materials and industrial waste in the manufacture of polymer concretes [8, 9, 10, 11, 12, 13, 14].

2. Purpose of research
The problems of obtaining compositions of polymer composites based on the use of a modifying additive - a plasticizer (organosilicon varnish KO-922) and asbestos-containing fillers based on waste from construction and chemical industries is thus addressed.

At the first stage, the areas of variation of recipe factors were determined, taking into account the following boundaries:
- for the plasticizer: plasticization, significantly improving the technological properties, can lead to a decrease in strength, elastic modulus and other performance indicators of structural composites;
- by filler: it is known that for each type of thermosetting synthetic resin and filler there is a strictly defined degree of filling, which ensures the maximum strength of the composite. A decrease or increase in the degree of filling leads to a significant drop in the strength of the system. In this case, the optimal amount of filler is determined not so much by the nature of the synthetic resin and filler, but also by the dispersion of the latter.

3. Methods of research
Determination of rational compositions of composite materials manufactured by compression and vibrocompression methods is of considerable interest. For the manufacture of epoxy composites, following epoxy binders, hardeners, fillers, and modifying additives were used.

Diane epoxy resin ED-20 in accordance with GOST 10587-84 was used as an epoxy binder. Polyethylene polyamine (PEPA) (TU 6 02 594 85) was used as a curing agent. Polysiloxane - organosilicon varnish KO-922 was used as a modifying additive for the epoxy binder. As fillers were used: quartz sand, crushed to a specific surface of 2000 cm²/g; fibrous waste from the chemical industry (FWCI) with a specific surface area of 4000 cm²/g; finely dispersed construction waste (FDCW) with a specific surface area of 5100 cm²/g. FWCI are finely dispersed asbestos fibers, which contain the main minerals of cement clinker in an amount from 0% to 10%. FDCW are finely dispersed asbestos fibers (percentage about 10-12%) in combination with finely dispersed Portland cement (about 88-90%). Quartz sand was used as a filler for the reference composition.

Two series of epoxy composite materials (ECMs) were produced by compression methods. Series 1 is made on a binary filler, including ground quartz sand and fibrous chemical waste (FWCI); series 2 - on a filler, including quartz sand and finely dispersed construction waste (FDCW). Silicone varnish KO-922 was used as a plasticizer for the compositions of all series.

The initial data for calculating the composition of EC are:
– true densities of fillers, modifier, resin and binding agent;
– composition volume;
– volume fraction of FWCI или FDCW \( v_b \);
– volume fraction of quartz sand \( \text{SiO}_2 \) \( v_p \);
– volume fraction \( \omega_a = \frac{m_a}{m_r} \) of plasticizer in relation to resin volume;
– volume fraction \( \omega_h = \frac{m_h}{m_r} \) of crosslinking agent in relation to resin volume.

The calculation scheme is based on the equation of the absolute volumes of the five-component system:

\[
V = \frac{m_p}{\rho_p} + \frac{m_b}{\rho_b} + \frac{m_r}{\rho_r} + \frac{m_a}{\rho_a} + \frac{m_h}{\rho_h},
\]

where \( V \) is the volume of composition; \( m_p, m_b, m_r, m_a \) and \( m_h \) are the mass of filler \( \text{SiO}_2 \), mass of FWCI and FDCW, mass of resin, mass of plasticizer and mass of binding agent respectively; \( \rho_p, \rho_b, \rho_r, \rho_a \) and \( \rho_h \) are their densities.

The methods for calculating the volumetric and mass content of components (determined characteristics and design formulas) are given in Tables 1 and 2, respectively.

### Table 1. Determination of the volumetric content of components.

| №  | Determinable characteristic | Design formula | Note |
|----|----------------------------|----------------|------|
| 1  | Volume of filler           | \( V_f = V \nu_f \) | \( V \) – volume of composition; \( \nu_f \) – total fraction of disperse phase |
| 2  | Volume of FWCI or FDCW     | \( V_b = c_r \nu_f \) | \( c_r \) – reinforcement ratio; \( \nu_f \) – total fraction of disperse phase |
| 3  | Volume of \( \text{SiO}_2 \) | \( V_p = (1 - c_r) \nu_f \) | \( c_r \) – reinforcement ratio; \( \nu_f \) – total fraction of disperse phase |
| 4  | Volume of binder           | \( V_m = V(1 - \nu_f) \) | \( V \) – volume of composition; \( \nu_f \) – total fraction of disperse phase |

### Table 2. Determination of the mass content of components.

| №  | Determinable characteristic | Design formula | Note |
|----|----------------------------|----------------|------|
| 1  | Mass of resin              | \( m_r = V_m \left( \frac{1}{\rho_r} + \frac{\omega_a}{\rho_a} + \frac{\omega_h}{\rho_h} \right)^{-1} \) | \( V_m \) – volume of binder; \( \omega_a \) and \( \omega_h \) – concentration of modifier and hardener |
| 2  | Mass of hardener           | \( m_h = \omega_h m_r \) | \( \omega_h \) – concentration of hardener; \( m_r \) – mass of resin |
| 3  | Mass of modifier           | \( m_a = \omega_a m_r \) | \( \omega_a \) – concentration of modifier; \( m_r \) – mass of resin |
| 4  | Mass of FWCI or FDCW       | \( M_b = V_b \rho_b \) | \( V_b \) – volume of FWCI or FDCW; \( \rho_b \) – density of FWCI or FDCW |
| 5  | Mass of \( \text{SiO}_2 \)  | \( M_p = V_p \rho_p \) | \( V_p \) – volume of \( \text{SiO}_2 \); \( \rho_p \) – density of \( \text{SiO}_2 \) |

Within each series, the true densities of the individual components are constant. Composition volume values for six samples \( 6-0,01-0,01-0,05-1,1 = 3,3\times10^{-5} \text{ m}^3 \) and mass fraction of binding agent \( (\omega_h=0,135) \) for all compositions are fixed. Valid variables are:
– volume fraction of FWCI or FDCW \( v_b \);
– volume fraction of quartz sand (\( \text{SiO}_2 \)) \( v_p \);
– concentration \( \omega_a \).

The levels and intervals of variation of the operational variables are shown in Table 3.

The values shown in Table 3 were determined based on the results of preliminary studies, during which it was found that an increase in the volume fraction of FWCI above 0.12 leads to a sharp deterioration in the rheological properties of the composition and a decrease in all physical property values.

**Table 3.** The investigated area of the factor space for compositions filled with quartz sand, FWCI or FDCW.

| Filler                     | Factors | Intervals of variation | Levels of variation |
|----------------------------|---------|------------------------|--------------------|
|                            |         | -1        | 0         | +1        |
| quartz sand + FWCI         | \( v_b \) | 0,06       | 0         | 0,06      | 0,12      |
|                            | \( v_p \) | 0,14       | 0,2       | 0,34      | 0,48      |
|                            | \( \omega_a \) | 0,02       | 0         | 0,02      | 0,04      |
|                            | \( v_b \) | 0,0594     | 0         | 0,0594    | 0,1188    |
| quartz sand + FDCW         | \( v_p \) | 0,022      | 0,022     | 0,044     | 0,066     |
|                            | \( \omega_a \) | 0,02       | 0         | 0,02      | 0,04      |

4. **Experimental results and analysis**

The design scheme includes the following steps (design formulas are given in Tables 1 and 2):

1. Determination of the volume of FWCI or FDCW (1);
2. Determination of the volume of \( \text{SiO}_2 \) (2);
3. Determination of the mass of FWCI or FDCW (3);
4. Determination of the mass of \( \text{SiO}_2 \) (4);
5. Determination of the volume of the binder (5);
6. Determination of the resin mass (6);
7. Determination of the mass of the plasticizer (7);
8. Determination of the weight of the binding agent (8).

The planning matrix and calculation results for compositions with FWCI are shown in Table 4. After processing the results, the regression equations are obtained:

\[ M_r = 19.909 - 1.990 \cdot X_1 - 4.644 \cdot X_2 - 0.471 \cdot X_3 - 0.001 \cdot X_1^2 + 0.047 \cdot X_1 \cdot X_3 - 0.002 \cdot X_2^2 + 0.111 \cdot X_2 \cdot X_3 + 0.011 \cdot X_3^2 - 0.002 \cdot X_1 \cdot X_2 - 0.001 \cdot X_1 \cdot X_3^2 - 0.004 \cdot X_1 \cdot X_3^2 \]

\[ M_h = 2.687 - 0.269 \cdot X_1 - 0.627 \cdot X_2 - 0.062 \cdot X_3 + 0.002 \cdot X_1^2 - 0.002 \cdot X_1 \cdot X_3 + 0.006 \cdot X_1 \cdot X_3 - 0.001 \cdot X_2^2 + 0.013 \cdot X_2 \cdot X_3 + 0.002 \cdot X_3^2 - 0.002 \cdot X_1 \cdot X_2^2 + 0.002 \cdot X_1 \cdot X_2 \cdot X_3 + 0.000 \cdot X_2 \cdot X_3^2 + 0.000 \cdot X_2 \cdot X_3^2 - 0.001 \cdot X_1 \cdot X_3^2 \]
Table 4. The planning matrix and calculation results for compositions with FWCI (mass of components is given in grams).

| №  | $v_b$ | $v_p$ | $\omega_u$ | $M_b$   | $M_p$   | $M_r$ | $M_a$   | $M_h$ |
|----|-------|-------|------------|---------|---------|-------|---------|-------|
| 1  | -1    | 0     | -1         | 0       | 0       | 16.2175| 27.185989| 0     | 3.670109|
| 2  | 0     | 0.06  | -1         | 0.2     | 0       | 16.2175| 25.14704| 0     | 3.39485 |
| 3  | 1     | 0.12  | -1         | 0.2     | 0       | 5.652  | 23.108091| 0     | 3.119592|
| 4  | -1    | 0     | 0.34       | -1      | 0       | 27.56975| 22.428441| 0     | 3.02784 |
| 5  | 0     | 0.06  | 0.34       | -1      | 0       | 2.826  | 20.389492| 0     | 2.752581|
| 6  | 1     | 0.12  | 0.34       | -1      | 0       | 5.652  | 18.350543| 0     | 2.477323|
| 7  | -1    | 0     | 0.48       | -1      | 0       | 38.922 | 17.670893| 0     | 2.385571|
| 8  | 0     | 0.06  | 0.48       | -1      | 0       | 2.826  | 15.631944| 0     | 2.110312|
| 9  | 1     | 0.12  | 0.48       | -1      | 0       | 5.652  | 13.592995| 0     | 1.835054|
| 10 | -1    | 0     | 0.48       | 0       | 0.02   | 16.2175| 26.542858| 0.5308572| 3.583286|
| 11 | 0     | 0.06  | 0.48       | 0       | 0.02   | 2.826  | 24.552143| 0.4910429| 3.314539|
| 12 | 1     | 0.12  | 0.48       | 0       | 0.02   | 5.652  | 22.561429| 0.4512286| 3.045793|
| 13 | -1    | 0     | 0.34       | 0       | 0.02   | 27.56975| 21.897858| 0.4379572| 2.956211|
| 14 | 0     | 0.06  | 0.34       | 0       | 0.02   | 2.826  | 19.907143| 0.3981429| 2.687464|
| 15 | 1     | 0.12  | 0.34       | 0       | 0.02   | 5.652  | 17.916429| 0.3583286| 2.418718|
| 16 | -1    | 0     | 0.48       | 1       | 0.04   | 38.922 | 17.252858| 0.3450572| 2.329136|
| 17 | 0     | 0.06  | 0.48       | 0       | 0.02   | 2.826  | 15.261243| 0.3052429| 2.060389|
| 18 | 1     | 0.12  | 0.48       | 0       | 0.02   | 5.652  | 13.271429| 0.2654286| 1.791643|
| 19 | -1    | 0     | 0.2        | 0       | 0.04   | 16.2175| 25.929452| 1.0371781| 3.500476|
| 20 | 0     | 0.06  | 0.2        | 1       | 0.04   | 2.826  | 23.984734| 0.9593897| 3.23794 |
| 21 | 1     | 0.12  | -0.2       | 0       | 0.04   | 5.652  | 22.040034| 0.8816014| 2.975405|
| 22 | -1    | 0     | 0.34       | 0       | 0.04   | 27.56975| 21.391798| 0.8556719| 2.887893|
| 23 | 0     | 0.06  | 0.34       | 1       | 0.04   | 2.826  | 19.447089| 0.7778835| 2.625357|
| 24 | 1     | 0.12  | 0.34       | 1       | 0.04   | 5.652  | 17.50238 | 0.700952 | 2.362821|
| 25 | -1    | 0     | 0.48       | 1       | 0.04   | 38.922 | 16.854143| 0.6741657| 2.275309|
| 26 | 0     | 0.06  | 0.48       | 1       | 0.04   | 2.826  | 14.909435| 0.5963774| 2.012774|
| 27 | 1     | 0.12  | 0.48       | 1       | 0.04   | 5.652  | 12.964726| 0.518589 | 1.750238|

Graphical dependencies were obtained according to the equations (figures 1 и 2).
Figure 1. Change dependencies of $M_r$ from the content of FWCI and quartz sand:

a) $\omega_a = 0$; b) $\omega_a = 0,02$; c) $\omega_a = 0,04$
Figure 2. Change dependencies of $M_h$ from the content of FWCI and quartz sand:

a) $\omega_a = 0$; b) $\omega_a = 0.02$; c) $\omega_a = 0.04$

The planning matrix and calculation results for compositions with FDCW are shown in Table 5.
Table 5. The planning matrix and calculation results for compositions with FDCW (mass of components is given in grams).

| $x$ | $v_b$ | $v_p$ | $\omega_a$ | $M_b$ | $M_f$ | $M_a$ | $M_h$ |
|-----|-------|-------|-------------|------|------|------|------|
| 0.1  | 0.22  | -1    | 0.022       | 1.783925 | 33.234872 | 0   | 4.486708 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 31.216312 | 0   | 4.214202 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 29.197753 | 0   | 3.941697 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 32.487257 | 0   | 4.38578  |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 30.468698 | 0   | 4.113274 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 2.784513  | 0   | 3.840769 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 31.739643 | 0   | 4.284852 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.721083 | 0   | 4.012346 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 27.702523 | 0   | 3.739841 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 32.448644 | 0.6489729 | 4.380567 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.47836  | 0.6095567 | 4.114508 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 28.507029 | 0.5701406 | 3.848449 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 31.718715 | 0.6343743 | 4.282027 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.47908  | 0.5949582 | 4.015968 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 27.771101 | 0.555542  | 3.749909 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 30.988786 | 0.6197757 | 4.183486 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.017979 | 0.5803596 | 3.917427 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 27.047172 | 0.5409434 | 3.651368 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 31.698754 | 1.2679502 | 4.279332 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.773493 | 1.1909397 | 4.019422 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 27.842311 | 1.1139292 | 3.759511 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 30.985695 | 1.2394278 | 4.183069 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 29.060433 | 1.1624173 | 3.923158 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 27.135171 | 1.0854068 | 3.663248 |
| -1   | 0     | 0     | 0.044       | 3.56785 | 30.276235 | 1.2109054 | 4.086806 |
| 0.1  | 0.22  | -1    | 0.022       | 2.798   | 3.56785   | 28.347373 | 1.1338949 | 3.826895 |
| 0.1  | 0.22  | -1    | 0.022       | 5.595   | 3.56785   | 26.422111 | 1.0568844 | 3.566985 |

After processing the results, the regression equations are obtained:

$$M_b = 29.751 \cdot X_1 - 1.971 \cdot X_2 - 0.729 \cdot X_3 - 0.704 \cdot X_1 - 0.001 \cdot X_1 X_2 + 0.047 \cdot X_1 X_3 - 0.002 \cdot X_2 X_3 + 0.017 \cdot X_2 X_1 - 0.015 \cdot X_2^2 - 0.01 \cdot X_3 X_2 + 0.002 \cdot X_3 X_1 + 0.001 \cdot X_3 X_2 - 0.001 \cdot X_3 X_3 - 0.002 \cdot X_1 X_2^2 - 0.001 \cdot X_1 X_3^2$$

$$M_h = 4.015 - 0.266 \cdot X_1 - 0.097 \cdot X_2 - 0.096 \cdot X_1 + 0.001 \cdot X_1^2 + 0.001 \cdot X_1 X_2 + 0.007 \cdot X_1 X_3 + 0.001 \cdot X_2^2 + 0.003 \cdot X_2 X_3 + 0.002 \cdot X_2^2 - 0.003 \cdot X_1 X_2 + 0.002 \cdot X_1 X_2 + 0.001 \cdot X_1 X_3 + 0.003 \cdot X_2 X_3 + 0.003 \cdot X_3 X_2 - 0.002 \cdot X_1 X_3^2$$

Graphical dependencies were obtained according to the equations (figures 3 and 4).
Figure 3. Change dependencies of $M_r$ from the content of FDCW and quartz sand:
   a) $\omega_a = 0$; b) $\omega_a = 0,02$; c) $\omega_a = 0,04$
Figure 4. Change dependencies $M_b$ from the content of FDCW and quartz sand:

a) $\omega_a = 0$; b) $\omega_a = 0.02$; c) $\omega_a = 0.04$

5. Conclusions
As a result of the studies performed, an analytical-graphic method for determining the compositions of epoxy compositions with predetermined properties has been developed. Rational compositions of epoxy composites based on FWCI and FDCW were obtained, the samples of which were made by two different methods: compression and vibrocompression.

The nomograms shown in Figures 1–4 make it possible to perform a practical choice of specific filler composition for given conditions.

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