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Research of Formal-Kinetic Regularities of the Curing Process of Low-Viscosity Epoxy Binder

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Abstract*: The expanding range of components intended for manufacturing products of polymer composite materials is often not accompanied by comprehensive information on the characteristics and recommended technological curing modes. The relevance and practical significance of this investigation are based on the need to predict rational technological modes for curing products of polymer composite materials based on the low-viscosity epoxy binder Etal-Inject-SLM, which is in demand in the manufacture of products from polymer composite materials by cold curing. The purpose of this investigation is to determine kinetic patterns, which allow for predicting the time and degree of curing of the low-viscosity epoxy binder Etal-Inject-SLM for various isothermal curing conditions. The method of differential scanning calorimetry was used to measure the thermal effect of the curing reaction of the Etal-Inject-SLM epoxy binder at various heating rates. Based on the experimental calorimetric data and using the model-free (non-prior) Friedman method, using the specialized software Netzsch Kinetics Neo, the parameters characterizing the formal kinetic laws of the curing process were calculated. This allowed for plotting isothermal and iso-conversion thermokinetic diagrams necessary for predicting the time and degree of curing of the Etal-Inject-SLM binder at different temperatures and curing times. This results can be useful for optimizing technological processes in which the low-viscosity epoxy binder Etal-Inject-SLM is used.

***Keywords*: curing degree; differential scanning calorimetry; epoxy oligomers; kinetics; thermokinetic diagram.**

I. INTRODUCTION

Industrial development makes ever-increasing demands on the level of properties and the variety of materials used. In this regard, in addition to traditional metal, ceramic and polymeric materials, composite materials have become increasingly common. In particular, due to its unique properties, polymer composite materials with a thermo-setting matrix are widely used. For more than half a century, the main types of thermosetting resins and binders based on them have been phenol and amine formaldehyde condensates. However, when using phenol - and amino-formaldehyde binders, a significant drawback was the need to use a high curing temperature (above 130-170ºC) and high pressure during molding (200 to 500 kg/cm2), and the curing process was accompanied by the release of toxic volatile components. The later development of epoxy and polyester binders made it possible to almost completely replace the amino and phenol-formaldehyde compositions [19]. In addition to the existing technologies for molding composite products using high-temperature hot curing, the rapid growth of autoclaveless technologies using low viscosity types of binder followed in turn, the range of technical tasks in which cold-curing compositions were in demand was significantly expanded. The improvement of the compositions and properties of low-viscosity cold-cured epoxy compositions as a result ensured their demand for the manufacture of polymer composite materials for structural purposes [30] and made it possible to use them in construction [8, 14, 23, 24] in various, including difficult climatic conditions. Modern materials obtained using low-viscosity cold-cured epoxy binders have sufficient strength and rigidity with a high level of viscoplastic properties [30]. Nevertheless, the molding of products from polymer composite materials with a thermoplastic matrix is associated with a number of undesirable features of the exothermicity of the curing process of the binder, such as, for example, the appearance of temperature gradients and related curing rate gradients in the material of the molded product, leading to heterogeneity of the structure and properties of the final material and the occurrence of distributed residual stresses. The main way to deal with these imperfections is to optimize the temperature-time conditions of molding, taking into account the geometry of the product and taking into account the curing kinetics of the formed material. Thus, for technologists, the choice of rational temperature-time regimes for curing composite products is an urgent and practically significant task. However, the expanding range of binders intended for the manufacture of products from polymer composite materials with a thermosetting matrix is often not accompanied by comprehensive information on the characteristics and recommended technological curing modes.

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The purpose is to study kinetic patterns, which allow for predicting the time and degree of curing of the low-viscosity epoxy binder Etal-Inject-SLM, which is in demand in the manufacture of products from polymer composite materials by cold curing, for isothermal curing conditions.

II. LITERATURE REVIEW

Currently, epoxy resins are one of the most used types of binders that are used in the manufacture of reinforced plastics, casting and impregnating compositions, paints, and varnishes [22]. Advantages of epoxy binders are as follows [38]: good adhesion to most reinforcing fillers, low shrinkage during curing, the absence of emission of volatile products during curing, high chemical resistance, good electrical insulation properties [39]. Depending on the type of curative, the heat resistance of epoxy matrices can vary over a wide range from 90 to 200°C [21].

Epoxy monomers and oligomers include compounds containing in the molecules of at least two epoxy or glycidyl groups. During curing, block copolymers or mesh homopolymers are formed. The class of epoxy resins used in the technique is very wide. However, more than 70% of their world production is occupied by diane resins based on diphenylol propane and epichlorohydrin [13]. The dominant position of diane epoxy resins in the world and Russian markets is explained not only by the unique properties of diphenylolpropane as a raw material for their synthesis, but also by the presence of cost-effective large automated industries with continuously improving technology for the production of starting monomers and low molecular weight epoxydianine resins [2, 25, 29, 33].

Various grades of diane resins are characterized by molecular weight distribution and distribution according to the type and number of functional groups. Diane resins are generally viscous liquids and glassy substances. Industrial diane resins contain high molecular weight fractions that prevent their crystallization. The reactivity of diane resins depends on the content of epoxy and hydroxyl groups, and the distance between them determines the density of the mesh in the cured polymer. Solid resins of high molecular weight are the basis for powder paints, press powders, paint, and varnish materials. The resin of medium molecular weight is usually cured at high temperatures (hot curing mode) by dicarboxylic acid anhydrides, aromatic amines, phenol-formaldehyde resins. Such resins are used for manufacturing enamels, varnishes, and adhesives. Low-molecular-weight epoxy resins are mainly used for manufacturing impregnation and pouring compounds, coatings and binders for reinforced plastics. They have high reactivity, which allows for curing aliphatic amines at both room and raised to 60 – 100°C temperatures [2, 25, 29, 33].

Epoxy resins curing occurs as a result of polycondensation reactions or polymerization reactions with epoxy groups opening. Block copolymers are formed in the first case, and homopolymers of mesh structure are formed in the second case. In poly-coupling, the chemical structure and functionality of the curative, as an equal component of the resulting copolymer mesh, affects both the technological properties of the reactive mixture and the performance of the cured material. The number of industrial brands of curatives is more than 100, however, they all fall into several main classes that differ in the chemical nature of the main block of the molecule, the type and number of reactive groups. The main industrial classes of epoxy oligomer curatives are primary aliphatic, aromatic diamines, and polyamines; simple amides; imidazole polyamides; dicyanamide; diisocyanates, and polyisocyanates; acid anhydrides; Lewis acids; modified aliphatic and aromatic amines. The existing range of curatives for epoxy resins allows for varying the pot life, thermophysical, thermomechanical, and technological parameters of the cured compositions over a wide range. At that, using amine type curatives has a number of advantages, such as high viability, low viscosity of the binder, while ensuring sufficiently high physical and mechanical characteristics of the finished composites. All this has ensured the widespread availability of amine curatives for cold cured epoxies [2, 25, 29, 33].

It should be noted that the properties of net polymers based on diane resins are more dependent on the type of hardener than on the molecular weight of the resin. At that, the curing mode also plays the most important role in producing composite materials. Under-cured material will have an incomplete resource of operational characteristics. High-temperature differences during the curing process can lead to warping and even cracking of molded composite products. Additionally, curing at s high temperature requires using more expensive equipment. All this leads to the need for choosing the curing modes of the composite product, rational for each specific type of thermosetting binder.

III. MATERIALS AND METHODS

The object of the study is a low-viscosity epoxy compound by Etal-Inject-SLM. Low viscosity binder Etal-Inject-SLM is well suited for vacuum infusion, manual molding, and RTM technology. Compound by Etal-Inject-SLM has high viability and a quick set of strength at room temperature; it soaks well multilayer composite preforms based on carbon fabric, fiberglass and glass cloth. Composite products made using Etal-Inject-SLM can cure at room temperature. Additional heat treatment, post-curing, can be used to achieve enhanced physical and mechanical properties and heat resistance. The temperature of thermal deformation Etal-Inject-SLM after post-cure exceeds 90°C. Currently, the Etal-Inject-SLM compound is widely used for the manufacture of various large-sized composite cold-cured products, in particular, hulls of boats and boats. The curing mode recommended by the manufacturer of the Etal-Injection-SLM compound corresponds to 24 hours at a temperature of 23°C and four hours at a temperature of 75°C. However, there is no information necessary to predict the time and degree of curing of the Etal-Inject-SLM binder under other temperature-time conditions. The authors carried out their experimental study of the thermal effects of the Etal-Injection-SLM binder curing process by the differential scanning calorimetry (DSC) method. The DSC method implies that the heat is determined through the heat flux - the derivative of heat with respect to time.

Therefore, the method is called differential [40, 41]. Heat fluxes are measured by the difference in temperature at two points of the measuring system at one point in time. The time dependence of the temperature difference between the cell with the sample and the comparison cell is measured experimentally. Measurements can be carried out both in isothermal conditions and in dynamic mode with a programmable change in temperature, calorimeters of this type are called scanning calorimeters [3, 4, 11, 18, 20, 31]. The authors used Netzsch DSC 204F1 for measuring the specific heat flux. The measurements were carried out at a purge speed of the measuring cell of 50 ml/min with argon; the weight of the binder sample in all measurements was in the range from 9 to 11 mg. The samples of the studied Etal-Inject-SLM compound were cured at four different heating rates to perform subsequent kinetic calculations: 20, 10, 5 and 2.5 K/min. Netzsch Kinetics Neo software processed the experimental data and modeled the kinetics of binder curing.

Thanks to modern software, the DSC method allows for determining the kinetic parameters of the curing reaction of an epoxy binder of various chemical compositions [7, 9, 36, 37], which, together with thermo-analytical studies, become the basis for modeling the curing processes of thermosetting binders in arbitrarily given temperature-time conditions. The kinetic analysis methods, included in the Netzsch Kinetics Neo software, can be divided into two groups [26]: non-prior (model-free) and model-based. The model-based methods require the value and sequence of the reaction stages, as well as the type of kinetic equations describing them to be set initially. The reaction independence principle is taken as a prior and the system of differential equations, describing the reaction rate at all stages, is made up based on the chosen kinetic model. Each reaction stage has the parameters of the kinetic equation (the reaction order, diffusion rate, etc.), the frequency factor and the activation energy defined by means of optimizing the coefficients of the regression equation. However, the number and the sequence of the reaction stages and the type of kinetic equations describing them are not known in advance for many of the studied materials. Therefore, in this manuscript, non-prior analysis methods were used for kinetic analysis [6, 27], which allow for estimating the activation energy of the curing reaction from the slope of iso-conversion lines (lines of equal curing degree) obtained at different heating rates of the samples. The disadvantages of isoconversion methods include the inability to uniquely determine the type of kinetic equations of elementary stages. At the same time, the main advantage of isoconversion analysis methods is that the calculation result does not depend on the type of kinetic equation, and the values of the kinetic model parameters obtained using various isoconversion methods are in good agreement both with each other and with the results of other physicochemical analysis methods [12]. The experimental and calculated results obtained in this manuscript are presented below.

IV. RESULTS

The baseline-adjusted calorimetric curing curves of the Etal-Inject-SLM binder are shown in Fig. 1. Main characteristics of the calorimetric curves are described in Table- I. A tangential curve was used as a baseline to compensate for the temperature difference in the value of the specific heat capacity of the reaction products and the initial substances.

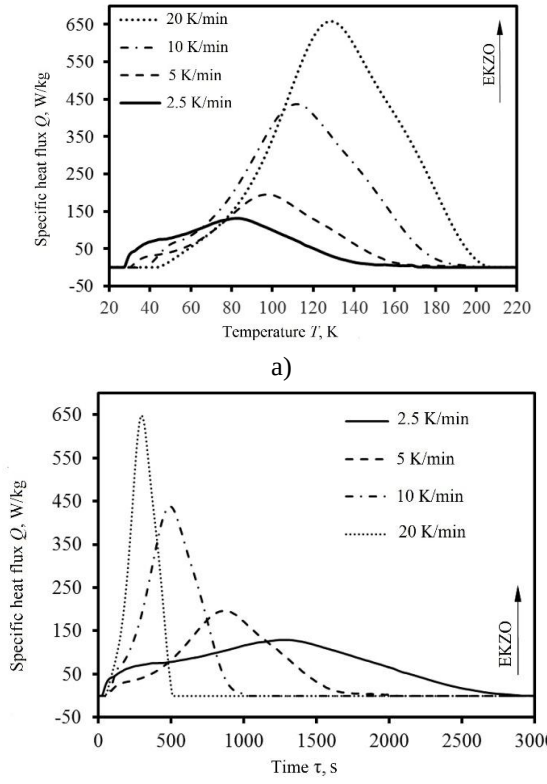


Fig. 1. Dependencies of the specific heat flux on the temperature (a) and time (b) at curing the Etal-Inject-SLM binder at various heating rates

Table- I: Main characteristics of the calorimetric curves

Heating Rate, K/min	20	10	5	2.5
Exothermic reaction peak, °C	128.7	112.3	98.3	84.2
Enthalpy of the curing process, J/g	137.2	182.1	152.7	207.6

The heat release at curing the binder depends on the heating rate, which allows, in accordance with the expression (1), for researching the formal-kinetic dependencies of the curing process based on the calorimetric curves measured at various heating rates [15]. The calorimetric curves were analyzed using Netzsch Kinetics Neo software, the use principles of which are shown in [1, 26].

$$\frac{D\alpha}{d\tau} = \frac{1}{H_T} \cdot \frac{dH}{d\tau}, \qquad (1)$$

where α *is* the curing (conversion) degree; τ *is* the processing time; H_T is the total enthalpy of the curing process.

Testing of various models included in Netzsch Kinetics Neo showed that the best approximation of the calculated and experimental results is achieved at describing the curing kinetics of Etal-Inject-SLM binder using the model-free Friedman test [28],

which uses the following mathematical formalization of the process' kinetics (2):

$$\frac{d\alpha}{d\tau} = A_\alpha(\alpha) \cdot f(\alpha) \cdot exp\left(-\frac{E_\alpha(\alpha)}{R \cdot T}\right), \qquad (2)$$

where R is the universal gas constant; T is the absolute temperature; E_α is the energy for activating the curing reaction; A_α is the proportionality factor; $f(\alpha)$ is the function, which form is not specified as there are no set forth assumptions on the mechanisms and stages of the process when describing them following the Friedman test. and all the calculations are made isoconversionally.

The values of the activation energy, identified using the Netzsch Kinetics Neo software under the Friedman test $E_\alpha(\alpha)$ and the $A_\alpha(\alpha)$ factor, defining, following the expression (2), the curing process of Etal-Inject-SLM epoxy compound, which, in turn, depends on the curing degree α, are shown at Fig. 2.

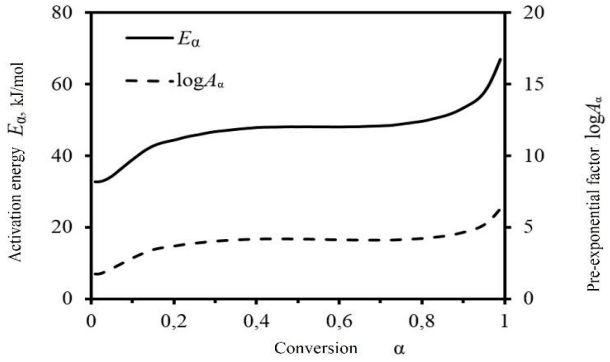
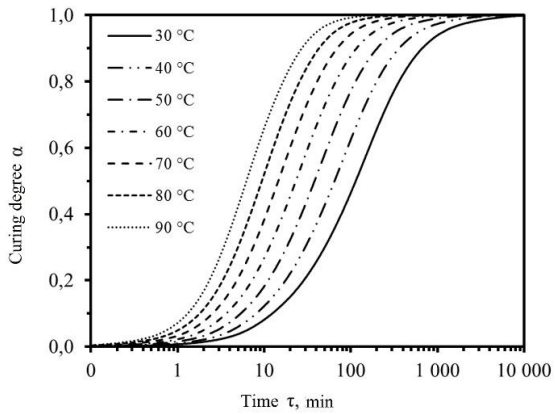
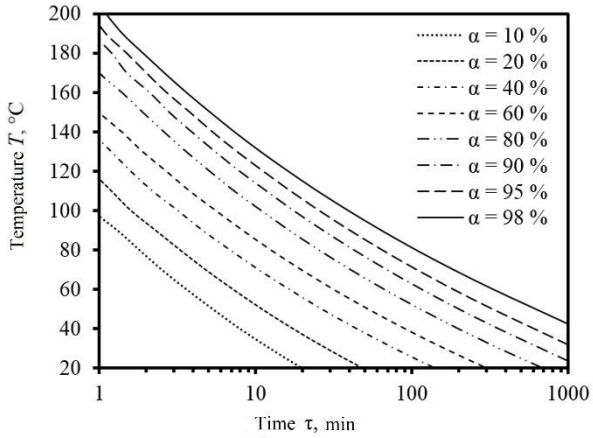


Fig. 2. The change in the activation energy E_α and the pre-exponential factor A_α in the curing process of Etal-Inject-SLM epoxy binder

The dependencies, shown in Fig. 2, were used in the calculation of the thermokinetic diagram of curing the binder, which allows for forecasting the time and curing degree of the binder for various isothermal modes. A detailed description of the calculation procedure and the features of using thermokinetic temperature-time-transformation diagrams for thermoset materials are described in [10, 16, 17, 34, 35]. The thermokinetic diagram of the Etal-Inject-SLM binder obtained in this work is shown in Fig. 3a and 3b as a family of isothermal and isoconversion curves.



a)



b)

Fig. 3. Isothermal (a) and isoconversion (b) thermokinetic diagrams of curing of the epoxy binder Etal-Inject-SLM

V. DISCUSSION

Analysis of the data presented in Fig. 2 allows for concluding that the activation energy of the curing reaction of the Etal-Inject-SLM epoxy compound depends on the cure degree. In turn, a change in the values of the activation energy during the curing reaction means the presence of several stages of the curing process. Apparently, at least three dominant stages of the process can be distinguished. The initial stage of the Etal-Inject-SLM compound curing process is characterized by the lowest value of activation energy equal to 35-45 kJ/mol. This stage corresponds to the appearance of primary curing centers in the material, which are then combined into a branched network of the cured epoxy polymer. Then, in the next stage, the energy of 45-55 kJ/mol is needed to activate the curing reaction of most of the reactive material, but already upon reaching a conversion of approximately 80%, large energy is required to cure the remaining unreacted material - up to 70 kJ/mol. Apparently, this fact is explained by a decrease in diffusion mobility [5] of molecules due to an increase in the density of the cured epoxy polymer network.

VI. CONCLUSION

The expanding range of components intended for manufacturing products of polymer composite materials is often not accompanied by comprehensive information on the characteristics and recommended technological curing modes. The relevance and practical significance of this investigation are based on the need to predict rational technological modes for curing products of polymer composite materials based on the low-viscosity epoxy binder Etal-Inject-SLM, which is in demand in the manufacture of products from polymer composite materials by cold curing. The thermal effect of the curing reaction of the epoxy compound Etal-Inject-SLM was measured using differential scanning calorimetry at various heating rates.

Based on the experimental data, the parameters characterizing the formal-kinetic laws of the curing process are determined.

The ultimate goal of kinetic calculations in the study of curing processes is to predict the degree of completion of the reaction (curing degree) depending on the temperature and holding time at the selected temperature, or vice versa - to determine the curing duration at a given temperature and the degree of conversion [32]. The practical possibility of such a forecast for the Etal-Inject-SLM epoxy compound was first provided by the thermokinetic diagrams obtained in this work, which are presented in Fig. 3. This results can be useful for optimizing technological processes in which the low-viscosity epoxy binder Etal-Inject-SLM is used.

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