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The Effect of the Hardener on the Characteristics of the Polyester-Based Coating †

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Abstract: In order to reduce negative technological factors when using epoxy resin as a thermosetting binder for the composition, a less viscous epoxyamine resin was studied. To impart elastic properties, the resin was modified with two components: polyester based on sebacic acid and a polyamide hardener. The compositions were cured under various temperature conditions: without heating and with maximum heating at a temperature of 120 °C. The formation of a crosslinked polymer using the IR spectrum is shown. The influence of temperature conditions on the degree of curing of polymer films was determined: the largest amount of gel fraction was formed with gradual heating of the mixture. However, the best physical, mechanical, and operational characteristics were obtained for the sample, which was cured at maximum temperature.

Keywords: polyester; hardener; coating; polycondensation; polyamine; adhesion

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1. Introduction

Polymer composite materials are widely used in various fields of industry. Most of them contain epoxy resins as a binder [1,2]. The use of epoxy binders is determined by the combination of their unique characteristics: low shrinkage, high reactivity, water and chemical resistance, heat resistance, and manufacturability [3,4].

However, the processing of high-viscosity diane resins is associated with some technological problems [5]. Thinners are usually used for this purpose. The introduction of non-reactive diluents into the composition leads to a decrease in the physical and mechanical properties and chemical resistance of the resulting materials. Therefore, in order to reduce the viscosity of compositions, reactive diluents are introduced into the composition or the same effect is achieved by choosing a low-viscosity hardener [6–8].

Epoxyamine resin, which is a low-viscosity product of the reaction of aniline with epichlorohydrin, is known for its use as an active bifunctional diluent. A high content of epoxy groups can impart improved physicochemical properties in the cured composition. In addition, EA resin is perfectly compatible with polyester resins. The dielectric and physical-mechanical properties of the resin during curing are similar to those of diane resins.

Condensation products of polyalcohols with unsaturated and saturated aliphatic acids, as well as with aromatic acids, are used as polyester modifiers [9,10]. Aliphatic polyesters are widely used in environmental applications due to their high biodegradability and low cost [11]. They are obtained from a wide range of monomers and with a wide range of properties. Glycerin is the most important building block obtained from biomass, and industrially it is obtained as a by-product in the production of biofuels [12]. Hyperbranched aliphatic glycerol polyesters are a new class of stable promising polymers with low costs and toxicity [13,14]. Through functionalization, free hydroxyl groups in their structure provide ample opportunities for varying properties and applications [15,16]. In this class, polyglycerol sebacate is the most studied polyester, and various strategies for its synthesis

with control of its structure and properties have been developed. Currently, a wide range of block copolymers of glycerin aliphatic polyesters has been studied and effective copolymers with improved properties have been obtained.

In this case, the main purpose of polyester modifiers is to improve the technological properties of epoxy compositions. Strength and dielectric properties of polymers based on diane resins, modified with polyesters in an amount of 15–20 wt. parts per 100 wt. parts, including epoxy resin and unmodified resins, do not differ significantly [5]. Plastics based on epoxy-polyester resins have high strength properties and increased resistance to weathering and solar radiation [17,18].

The use of various hardeners and the use of diane resins with different molecular chain lengths ensures a change in the physical and mechanical properties of the resulting polymers over a wide range: from rubber-like materials under normal conditions to hard, high-strength, and high-modulus materials with preservation of strength characteristics under conditions of prolonged exposure to temperatures up to 200 °C and above [19–21]. Due to the presence of a large number of hydroxyl groups in some brands of diane resins, compounds containing active hydrogen can be used for curing and thus obtain polymers with valuable properties [22–24]. Thus, the influence of the chemical structure of hardeners on the physical and mechanical properties of polymers is of decisive importance.

At room temperature, diane resins are usually cured with aliphatic amines or their derivatives [25,26]. The most widely used primary aliphatic amines are ethylenediamine, diethylenetriamine, triethylenetetraamine, and hexamethylenediamine, as well as a technical mixture of amine–polyethylene polyamines, consisting of a mixture of 25 compounds, which includes the first 4. All of the listed amines are quite volatile and toxic substances, providing limited viability of compositions based on diane resins and satisfactory physical and mechanical properties.

Hardeners have a significant impact on the rate of crosslinking reaction, curing mode, and characteristics of the target product [27–30]. There are several technologies for forming a network polymer, but hardeners improve the polymerization mechanism and allow maximum product properties to be achieved. As post-curing occurs in the polymer structure, the crosslinker ensures completion of polymerization by reacting with the polymer. The development of high-performance and environmentally friendly epoxy thermoset materials remains a challenging task, which requires searching for and determining a hardener to obtain a three-dimensional polymer network with the best characteristics.

In this regard, in the work to obtain films, polyamide was studied as a hardener—a product of the interaction of polymerized fatty acids of vegetable oils and polyethylene polyamines, which is characterized by low toxicity, good chemical resistance of the resulting polymers, and resistance to the effects of minerals acids, aqueous solutions of alkalis, water, and hydrocarbons. In order to modify the epoxy resin, a saturated polyester based on sebacic acid was used. The novelty of this work lies in the study of the possibility of modifying epoxyamine resin with industrial polyester based on sebacic acid in order to increase the plasticity of the resulting polymer.

2. Materials and Methods

2.1. Materials

Polyester 24K is a polycondensation product of ethylene glycol and glycerin with sebacic acid, and was purchased from SPE Abika, Moscow, Russia. Polyester 24K is a paraffin-like mass, ranging from gray to dark gray or brown in color. The initial components for the production of 24K polyester are shown in Figure 1.

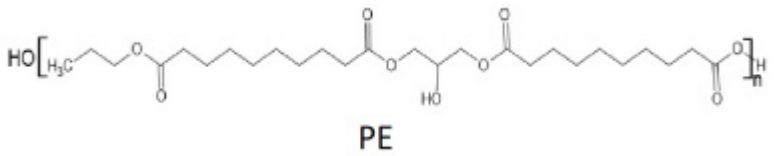


Figure 1. Polyester 24K.

The main characteristics of the PE resin are as follows: density of 50% polyester solution in acetone at 20 °C, g/cm^3—0.93–0.94; dynamic viscosity of a polyester solution in acetone with a mass fraction of 50% at 20 °C, MPa·s—20–30; acid number, mg KOH per 1 g of polyester—8–18; mass fraction of hydroxyl groups, %—5.2–8.0; and mass fraction of water, %—not more than 0.3.

Epoxyamine resin (EAR, Figure 2) is a product of the reaction of aniline with epichlorohydrin, and is produced by Kurskkhimprom LLC, Kursk, Russia. The resin is a liquid ranging from yellow-brown to dark red in color. Properties of EA resin: mass fraction of epoxy groups, %—not less than 31.2; mass fraction of volatile substances, %—not more than 1.2; mass fraction of chlorine ions, %—no more than 0.035; mass fraction of saponified chlorine, %—no more than 1.5; dynamic viscosity at 25 °C, Pa·s—no more than 0.35.

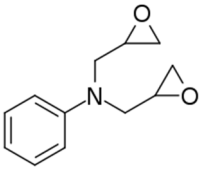


Figure 2. Epoxyamine resin.

The hardener (PAH, Figure 3) is a product of the interaction of polymerized fatty acids vegetable oils and polyethylene polyamines, and was purchased from Kurskkhimprom LLC, Kursk, Russia. The hardener is a homogeneous transparent viscous liquid ranging from yellow to dark brown color. The main characteristics of the hardener: amine number, mg HCl/g—90–120; amine number, mg KOH/g—139–185; mass fraction of non-volatile substances, %—100; dynamic viscosity at 20 °C, Pa·s—no more than 10–50.

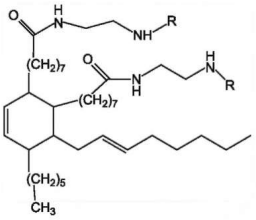


Figure 3. The hardener.

2.2. Method of Obtaining Samples

Samples of polymer films were obtained by mixing the components polyester and epoxyamine resin, and a hardener, in certain proportions. The compositions were poured into silicone molds and kept according to the selected conditions. The film curing modes are described below. For physical and mechanical tests, blades were cut out of the films (Figure 4) using a pneumatic punching press.

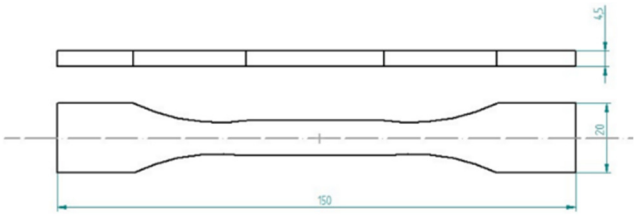


Figure 4. Test specimen type and dimensions.

2.3. Methods for the Analysis of the Samples

IR spectra. Samples of the obtained products were analyzed using the FTIR spectroscopy method with KBr tablets, which were prepared according to the standard procedure. IR absorption spectra were recorded in the range 450–3700 cm^{-1} using a Fourier

spectrometer IRAffinity-1S (Shimadzu, Kyoto, Japan) at room temperature. The resolution was 4 cm^{-1}, and the number of scans was 20.

The degree of curing of the polymer was determined by extracting the films for 24 h in acetone. The mass of the sample was 1 g. The sample was weighed to the nearest 0.001 g and placed in a solvent. After 24 h, a sample of the polymer film was weighed with an accuracy of 0.001 g. Then the sample was dried in a vacuum oven for 24 h. The degree of curing was calculated using Formulas (1) and (2):

$$S\ (\%) = (m - m_1)/m \cdot 100 \quad (1)$$

$$G = 100 - S \quad (2)$$

where m_1—mass of dried sample, g; m—mass of sample, g.

Physical and mechanical tests were carried out on an INSTRON 5900R universal testing machine (Instron, Norwood, MA, USA). A video extensometer was used to measure the longitudinal deformation of the samples, as well as determine the elastic modulus. The elastic modulus was determined in the 0.05–0.25% section of the deformation curve. The fracture strain was recorded at maximum load.

Tensile strength was measured at T = 23 ± 2 °C. Samples of the composition were pre-conditioned. The arithmetic mean value of the thickness and width measured in three places was used as the value of the initial cross-section of the samples. Specimens with applied marks were fixed into the testing machine in such a way that the direction of movement of the movable clamp corresponded to the direction of the axes of the clamps and the sample.

The tensile strength of films (MPa) was determined on cut-out samples and calculated using Formula (3):

$$P = F/S \quad (3)$$

where F—load under which the sample failed, H; S—initial cross-sectional area of the sample, mm^2.

Elongation at break was determined at T = 23 ± 2 °C. The samples were placed in a testing machine in a similar manner to the determination of tensile strength. To eliminate sample displacement during testing, the clamps of the testing machine were tightened evenly. The value of relative elongation at break of a polymer sample (L, %) was calculated using Formula (4):

$$L = ((l_1 - l_2)/l_1) \cdot 100 \quad (4)$$

where l_1—initial sample length, mm; l_2—sample length at rupture, mm.

Thermogravimetric analysis of polymers was carried out on a Netzsch STA 449 F3 Jupiter instrument (Netzsch, Selb, Germany). For this purpose, a sample weighing 5 ÷ 20 mg was placed in a platinum crucible. The experiment temperature in the device is from 25 °C to 500 °C, and the heating rate is 10 K/min in an air atmosphere with a blowing rate of 30 mL/min. During the experiment, dependences of sample mass loss on temperature were obtained.

3. Results and Discussion

The scheme shown in Figure 5 was chosen for the study.

Based on preliminary experiments, the ratios of components for obtaining polymer samples were established as follows: EA:PE:Res = 3:2:4. The designations of the samples and their curing modes are presented in the Table 1.

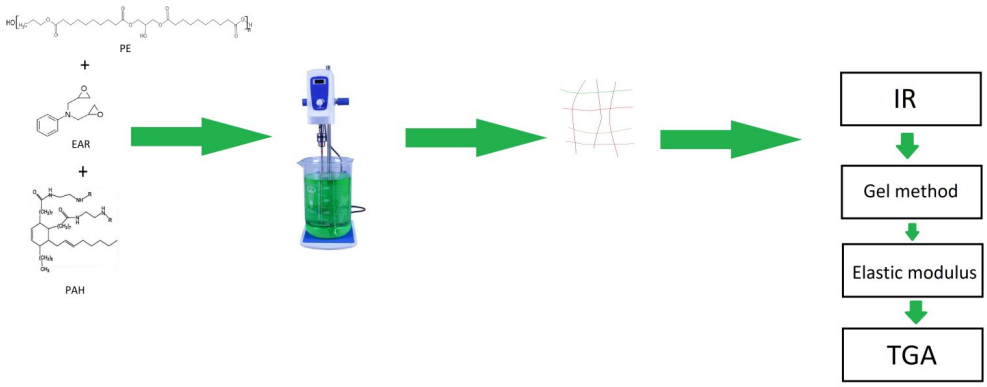


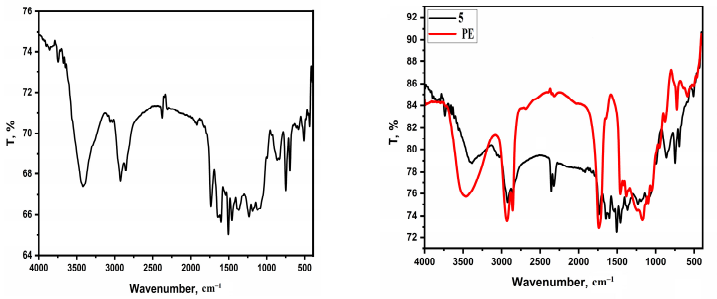
Figure 5. Research flowchart.

Table 1. Designations of samples and their curing modes.

Sample	Curing Modes
1	22 °C/56 h
2	22 °C/24 h, 60 °C/1 h, 80 °C/1 h
3	22 °C/24 h, 80 °C/1 h, 100 °C/1 h, 120 °C/1 h
4	60 °C/1 h, 80 °C/1 h, 120 °C/1 h
5	22 °C/24 h, 80 °C/1 h, 120 °C/3 h

The resulting plates, approximately 3 mm thick, were plastic polymers.

The occurrence of the reaction between the components of the mixture and the production of the polymer was confirmed by IR spectra (Figure 6). In the obtained IR spectra of the samples and polyester, signals of the OH group appear in the region of 3480 cm^{-1}. In sample 1 and the original polyester, a fairly large signal is observed. Although in sample 5 the signal becomes less intense, it is important to take into account that in both obtained samples, in contrast to the initial PE, a peak of the NH group appears in this region. Thus, a certain part of the hydroxo groups interacted. In the region of 1720 cm^{-1}, a characteristic signal of the carbonyl group C=O of carboxylic acids is observed. This peak in crosslinked samples is much less intense than in PE. The peak in the region of 1200–1350 cm^{-1} in the spectra of the samples, characteristic of the carbonyl group C=O of esters, is absent in the spectrum of polyester. The spectra of the films contain peaks in the region of 2800 cm^{-1}—CH_2—terminal epoxy group.



Sample 1. Sample 5 and PE.

Figure 6. IR spectra of samples.

The degree of curing of samples is determined by the amount of sol fraction (Table 2). The amount of sol fraction in the crosslinked polymer decreases with increasing processing temperature.

Table 2. Percentage of sol fractions in samples.

Sample	Percentage of Sol Fractions, %
1	21.78
2	27.54
3	20.69
4	27.15
5	24.21

However, the sample without heating (1) is not characterized by the largest amount of uncrosslinked prepolymer. Probably, the curing processes in the sample proceed for a long time even without heat treatment. Heating immediately after mixing the components does not improve the quality of the sample (4) in terms of this indicator. The highest degree of curing was shown by polymer 3, which was heated gradually to the maximum temperature of all the samples presented, in contrast to sample 5, which was heated less smoothly.

Determination of the physical and mechanical characteristics of the obtained samples was carried out on cut-out blades according to Figure 4 (Table 3). As a comparison, the indicators of a polymer sample without the addition of polyester are given (standard).

Table 3. Physical and mechanical characteristics of films.

Sample	Modulus, MPa	Tensile Strength, MPa	Elongation at Breaking, %	Elongation, mm	Maximum Strength, N
Standard	37.57	2.54	72.05	44.83	54.79
1	2.56	0.43	34.99	21.77	6.08
2	2.67	0.59	42.27	22.65	6.66
3	2.75	0.72	48.76	23.53	9.67
4	3.62	0.89	54.28	26.35	14.89
5	4.36	1.12	59.10	30.05	24.12

Elastomeric materials usually show low tensile strength (<1 MPa) [14]. Currently, the copolymerization of polyglycerol sebacate has been well studied by other forporlimers. The obtained results of the physico-mechanical characteristics are in good agreement with the literature data on copolymerization with other prepolymers. In our case, an increase in temperature during sample processing provides an increase in the indicators of all the studied characteristics. A characteristic feature can be identified—a decrease in ductility with increasing curing temperature, which is confirmed by the obtained modulus values. It is important to note that the introduction of polyester into the formulation leads to a decrease in the strength characteristics of the composite compared to the base formulation.

The thermal stability of polymers is important for establishing the manufacturability of their processing. The results of thermal analysis of copolymers are given in Table 4. Thermogravimetric curves are presented in Figure 7.

Table 4. Results of thermal analysis of copolymers.

Sample	Δm (0–150 °C), %	Δm (150–300 °C), %	Δm (300–450 °C), %	Δm (450–600 °C), %	m_f, g
1	5.11	15.79	58.27	17.29	4.42
2	1.77	15.46	61.79	16.52	4.46
3	1.87	11.38	65.84	16.53	4.38
4	1.67	10.87	63.77	18.57	5.12
5	0.23	7.39	67.22	19.76	5.40

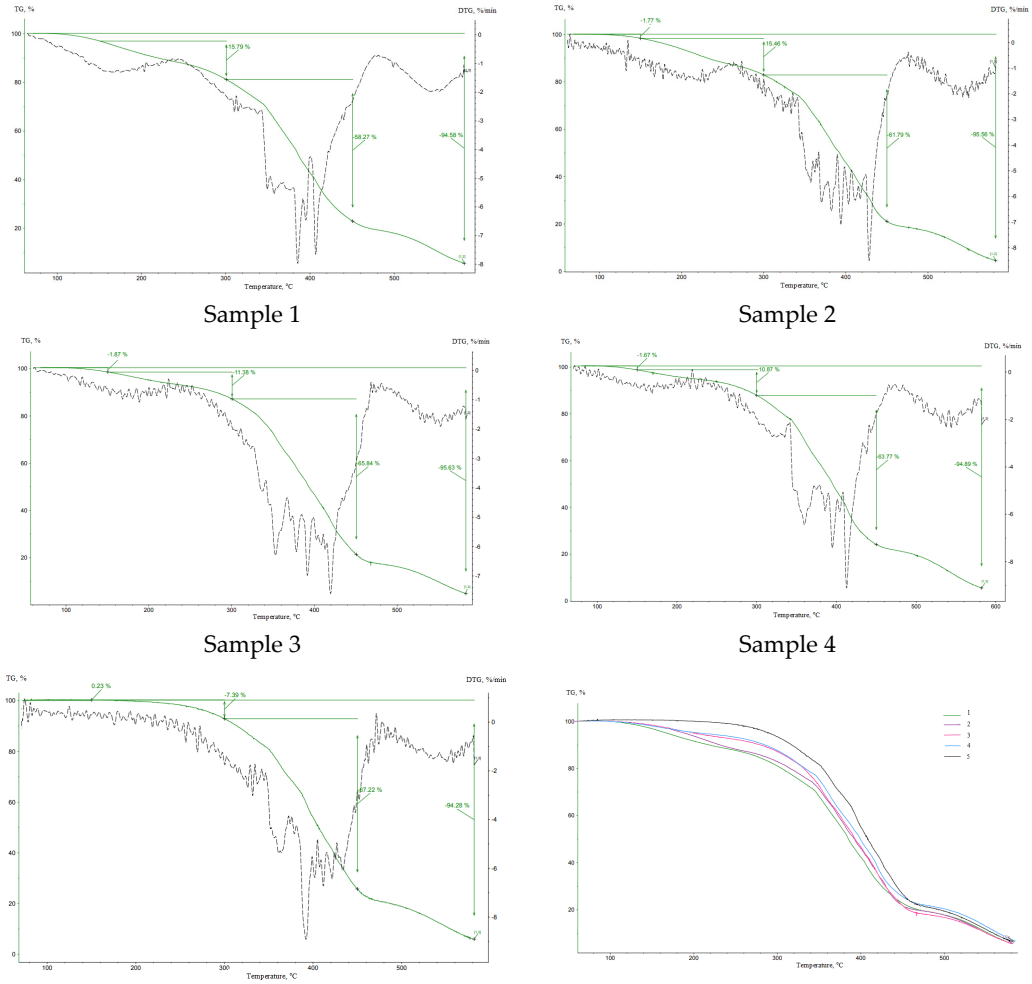


Figure 7. TG curves of copolymers.

Thermogravimetry of the samples showed that sample 5, which was subjected to the highest temperature treatment, has the greatest heat resistance. On the DTG curve of sample 2, the largest number of peaks are observed, corresponding to post-curing processes in the region of 250–350 °C. The least intensive post-curing processes occur in the sample without heat treatment. It is likely that crosslinking of the components occurred during long-term storage. However, this sample is characterized by the presence of the largest quantity of highly volatile components, which is reflected in the TG curve in the form of the greatest mass loss in the region up to 150 °C. In general, the peaks on the DTG curve, when moving from sample 1 to sample 5, gradually shift to the region of high temperatures (more than 400 °C), where decomposition and oxidation processes occur (sample 5). In the general thermogram of the samples, the pattern of increasing thermal stability is clearly visible for samples in row 1–5.

4. Conclusions

The developed composition is not characterized by a maximum degree of curing under cold curing conditions. The introduction of a polyester based on sebacic acid, which has free hydroxyl groups in its structure, requires heating to ensure the maximum crosslinking of the polymer. Accordingly, temperature treatment of the composite clearly contributes to an increase in the level of physical, mechanical, and operational characteristics. Additionally, in order to reduce the amount of sol fraction, it is probably necessary to change the amount of crosslinking agent hardener and, possibly, it is necessary to increase the curing temperature.

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