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The effect of curing modes on the parameters of molecular meshes of epoxy and polyester copolymers

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INTRODUCTION & AIM

Aliphatic polyesters are an attractive class of biologically based polymers. How-ever, the thermal and mechanical properties, as well as the service life of products based on them, in particular, and polysebacinates, are unsatisfactory for certain applications. The introduction of modifying resins as hardeners promotes the formation of mesh polymers. Mesh polymers of various nature and crosslinking densities are widely used in modern composite materials. The scope of application of the copolymers obtained depends on the degree of crosslinking of the components and the parameters of the molecular grids, which can be adjusted during the curing process and which determine their physical, mechanical and operational parameters. This way is universal and allows to obtain mesh polymers with any properties. Currently, methods of direct investigation of the structural characteristics of mesh polymers are actively developing. In this regard, it is relevant to study the regularities of the formation of polymer grids in the interaction of multifunctional compounds, as well as the ways of their directed synthesis with a given structure and properties.

METHOD

Sebacic acid polyester with ethylene glycol and glycerin was used to produce polymer films (Figure 1). Polyester is a paraffin-like mass of light gray color. The main characteristics of polyester: the acid number (mg KOH per 1 g of polyester) is 10, the mass fraction of hydroxyl groups (%) is 7.

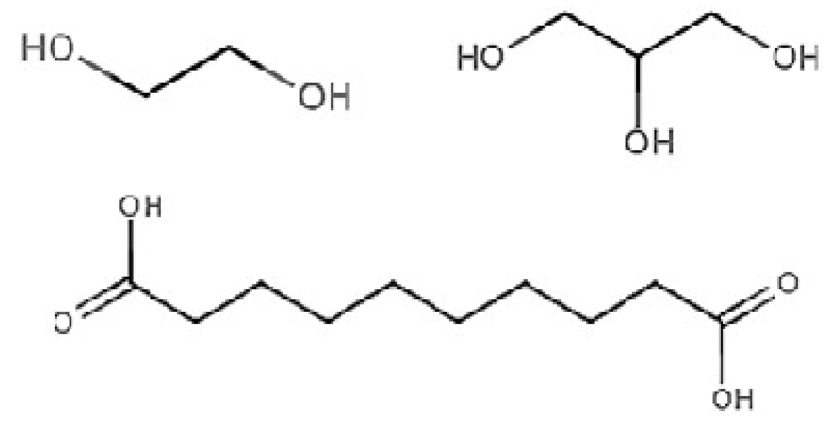

Figure 1. The initial components for the production of polyester

To develop a formulation for a cold-cured polymer coating, compositions based on the obtained polyester (PE) and epoxyamine resins (EAR, Fig. 2) were composed. A polyamide hardener was tested as a hardener – a product of the interaction of polymerized fatty acids of vegetable oils and polyethylene polyamines (PAH, Fig.3).

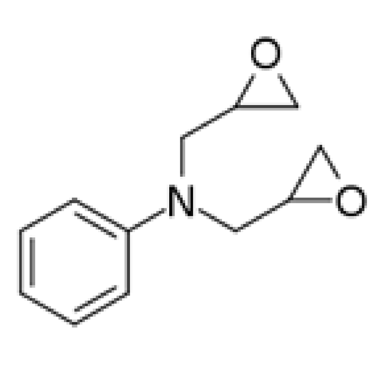

Figure 2. EAR

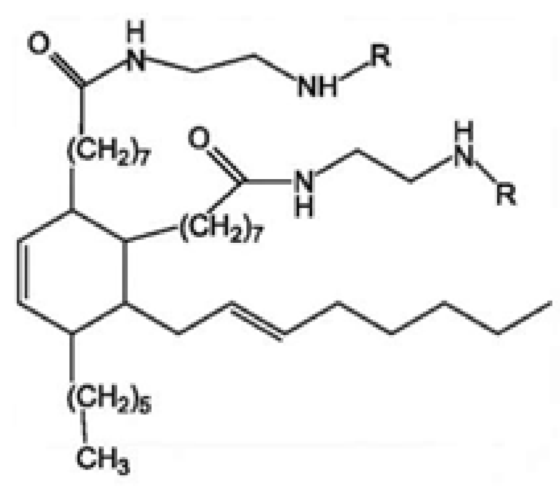

Figure 3. PAH

The components of the formulation were mixed in the ratio PE : EAR : PAH = 2 : 3 : 4. After 24 hours, the film was presented with burgundy glossy polymer.

RESULTS & DISCUSSION

The designations of the samples and their curing modes are presented in the table 1. The degree of curing of samples is determined by the amount of sol fraction (Table 2). Based on this indicator, other parameters of the molecular grid of the obtained copolymers were calculated: j – the degree of crosslinking, the average number of crosslinking links per molecule; V_a – share of active circuits; α – branching factor; γ – crosslinking density.

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

Table 2. The main indicators of copolymers.

Sample	S, %	j, %	Va, %	α	γ
1	21.78	3.78	60.66	0.264	1.46
2	27.54	3.04	52.05	0.249	1.25
3	20.69	3.96	62.37	0.269	1.51
4	22.56	3.66	59.47	0.263	1.43
5	15.99	5.00	70.01	0.287	1.79

To determine the structural parameters of the polymer crosslinking, the swelling of copolymer samples was studied. A sample of copolymer 3, characterized by the lowest value of the sol fraction, was used as the object of the study.

Таблица 3. Calculated values of the Huggins constant.

Sample	χ			
	chloroform	xylene	dimethylformamide	acetone
3	0.839	0.721	0,882	0.737

The best solvent for determining the structural parameters of the polymer crosslinking is the solvent with the lowest value of the Huggins constant. In this case, xylene. According to the experimental data obtained, the following indicators were calculated: Ds – the degree of swelling, Ms – the molecular weight of the chain segment enclosed between the nodes; Nc – the number of chains between the nodes per unit volume; nc – the number of moles of chains enclosed between the nodes (Table 4).

Table 4. Calculated characteristics of copolymers.

Sample	Ds, %	M_c, g/mol	Nc • 10^{23}, 1/cm³	n_c, mol/cm³
1	44.44	74.26	0.0980	0.016
2	43.09	67.67	0.1156	0.019
3	52.84	76.09	0.0965	0.016
4	67.98	79.69	0.1012	0.017
5	46.92	77.01	0.0860	0.014

CONCLUSION

As a result of modification of the epoxyamine resin with polyester based on sebacic acid using an amide hardener, a highly cross-linked copolymer was obtained. The amount of gel fraction and, accordingly, the degree of curing of the resulting product in-creases with an increase in the temperature regime of curing. However, in order to achieve the maximum degree of curing, it is necessary to carry out cold curing at the initial stage of formation of the crosslinked polymer. Otherwise, it is likely that with an increase in temperature, the supramolecular cohesion is disrupted and the interaction of prepolymer molecules is fluctuating, which leads to a decrease in the amount of gel fraction. Experimentally found degrees of swelling of copolymers confirm that heating of the prepolymer mixture immediately after mixing accelerates the crosslinking processes and bonds are formed along all possible interaction centers. Accordingly, relaxation processes do not have time to go through in the system, and fluctuations contribute to a low degree of curing.