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Study of Physico-Chemical Properties of Sulfur-Containing Epoxy Resin

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Abstract: The preparation of a sulfur-containing epoxy resin was studied by analyzing its compositions based on polyethylene fillers and the structure and composition of a sulfur-containing epoxy resin. According to the results of the presented studies, it was shown that the resulting compositions are sealed materials with high physical and mechanical properties used in construction.

Keywords: sulfur and nitrogen containing oligomer, filler, composition, optical microscopy, and epoxy resin.

In the world, for the creation of sealing and protective coatings, polysulfide oligomers are of undoubted interest. The consumption of curable sealants in construction today reaches more than half of their total production. The share of consumption of sealants based on polysulfide oligomers in the construction sector in the world is 70%. This is due to the invariance of their curing methods, acceptable physical and mechanical properties and aggressiveness, a wide range of operating temperatures for vulcanizates.

Today, all over the world, attention is paid to research aimed at improving the quality and efficiency of the use of phosphorus- and sulfur-containing oligomers and polymeric materials. In this aspect, reactive oligomers containing functional groups of phosphorus a and sulfur are of certain scientific and practical interest. At the same time, phosphorus and sulfur-containing oligomers can be effectively used to obtain highly effective sealants.[1.2]

Among the numerous ways to modify the structure and properties of polyolefins, chemical modification occupies a special place. As a way to create polyolefins, with an improved complex of specific properties, this method has now been widely developed. Achievements in this area are reflected in numerous patents and publications, including a number of detailed reviews and monographs. An analysis of these data shows that the success of this area of research is largely related to the physicochemical approach to the evaluation and generalization of the realized results from the point of view of the polymeric nature of the reacting particles, i.e. under conditions that take into account the initial state of macromolecular structures, the nature of intermolecular interactions, and various kinds of conformational and supramolecular effects.

This work is devoted to the development of new effective local resources and environmentally friendly sulfur-containing epoxy binders and cold-curing composites with improved performance properties based on nitrogen and sulfur-containing epoxy oligomer (ETEP), intended for restoration, repair and corrosion protection of metal, concrete, reinforced concrete and other building structures, as well as for the manufacture of filled building composites.

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The studied compositions in the work served to obtain compounded polyolefins (PO) based on high-pressure polyethylene (LDPE) brand F-0220S of the Shurtan gas chemical plant and calcium hydroxide filler. Sulfur-containing epoxy resin based on epichlorohydrin (ECH) and thiourea was used as modifiers. Samples were modified in solution and in melt. Changes in the fine chemical structure of modified software were studied by IR spectroscopy, optical microscopy, and using an electric microscope with elemental analysis.

We have synthesized new sulfur-containing epoxy oligomers, while studying the optimal conditions for obtaining oligomers, such as temperature, viscosity and the ratio of the initial components, and studying their IR spectra. For comparison, some data from the results obtained are given. Table 1 shows the results of viscosity measurements for dilute solutions of sulfur-containing oligomers. To determine the viscosity of oligomer solutions, an Ubbelohde capillary viscometer and a method based on measuring the outflow time of a pure solvent and solutions of various concentrations were used (started with a concentration of a 1% solution with the following dilution of the solution to a concentration of 1; 0.5; 0.25), at a constant temperature ^{of} 22 ° C. According to the readings of table 1, a diagram was built (Figure 1) and the characteristic viscosity of the oligomers was determined from the diagram.

Table 1. Measurement of the Viscosity of Dilute Solutions of Sulfur-Containing Oligomers .

No.	The name of the oligomer.	Solution concentration , %	η_{rel}	η_{beat}	η_{pr}	η_{log}	η_{xv}
one	Sulfur-containing epoxy resin	1	1.10	0.1	0.1	0.095	0.07
		0.5	1.04	0.04	0.08	0.73	
		0.25	1.01	0.01	0.05	1.3	

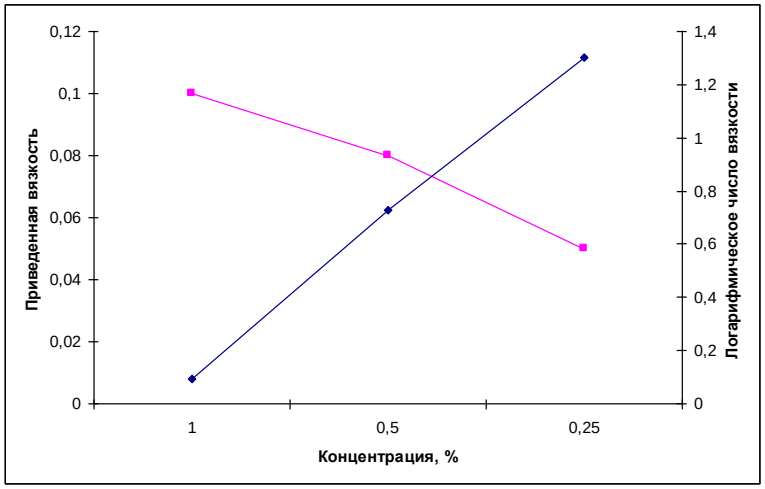

Figure 1. Dependence of η_{sp} /C or In η_{rel} /C on the concentration of sulfur-containing epoxy resin.

As can be seen from table 2, under optimal conditions (T= 90°C, τ=3h), a high yield of the oligomeric compound is obtained at a ratio of epichlorohydrin: thiourea 1:1. The yield is 78%. The resulting oligomeric compound is a viscous substance of the ETEp brand

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Table 2. Physical and chemical characteristics of the ETEp oligomer

1	Appearance	Brown color.
2	pH	7
3	Density (25°C), g/ ^{sm3}	1.2
4	Mass fraction of volatile substances, %, no more	1.4
5	Mass fraction of chlorine ion, %, no more	0.11
6	Mass fraction of total chlorine, %, max	5
7	Solubility	Soluble in organic matter

The IR spectrum shows that the bands of the sulfur-containing oligomer correspond to absorption bands corresponding to stretching vibrations of bonds in the regions of 2850 cm $^{-1}$, there are absorption bands confirming the presence of -CH $_2$ - groups, and absorption bands in the region of 1340 cm $^{-1}$, corresponding carbon and hydrogen containing groups.

The IR spectrum contains absorption bands in the 1650cm $^{-1 \text{ region}}$, confirming the presence of –CONH $_2$ groups in the Free State, and absorption bands in the 3300-3440cm $^{-1 \text{ region}}$, corresponding to secondary –CONHR groups. The IR spectrum contains absorption bands in the region of 3000-3050 cm $^{-1}$ corresponding to the epoxy ring and absorption bands in the region of 750-950 cm $^{-1}$ asymmetric to the stretching vibrations of the ring. Absorption bands in the areas of 800-600cm $^{-1}$, confirming the presence of carbon and sulfur-containing (- C - S) groups and absorption bands in the area of 450-550cm $^{-1}$, corresponding to - S - S - groups (Figure 2).

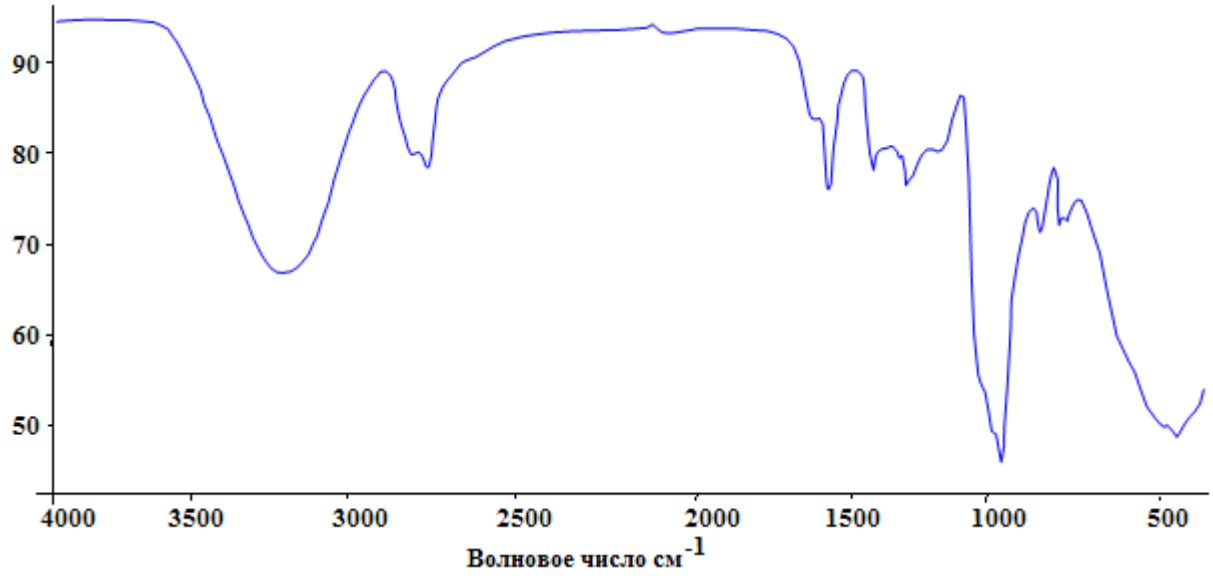

Figure 2. IR spectrum of a sulfur-containing epoxy oligomer grade ETEp

The processes of obtaining an epoxy oligomer proceeding by the interaction of epichlorohydrin (ECH) with thiourea have been studied. To obtain a composite material, calcium hydroxide filler was used, and sulfur- and nitrogen-containing epoxy resin of the ETEp brand was used as a binder. and high-pressure polyethylene brand F -0220 S (LDPE). The amount of filler in the polymer matrix was 40 wt. hours, this amount is optimal for compositions based on calcium hydroxide filler with ETEp and polyethylene, which is proved in these works. The effect of the filler was evaluated by the change in physical and mechanical characteristics.

The study of the fillers used by optical microscopy showed that the filler particles are characterized by some agglomeration of particles, which indicates a high activity of their surface (Figure 3).

Figure 3. Optical microscopy of the ETEp composition

The results of optical microscopy show that the compositions have the best miscibility with polymers. Studies of the polyethylene composition obtained with the addition of nitrogen and sulfur-containing epoxy resins showed that these compositions have the best results compared to other compositions.

Conclusion. Therefore, the characteristic properties of epoxy resin, sulfur-, phosphorus- and nitrogen-containing oligomer were the oligomer can be used as a sealant of building polymer materials. The physicochemical properties were studied: determined by IR spectroscopy, SEM and DSC, as a result of laboratory tests it was proved that the density, melting point, solubility, IR spectroscopy and DSC in sulfur-, nitrogen- and phosphorus-containing oligomers.

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