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INVESTIGATION OF THE EFFECT OF REACTIVE DILUENTS ON THE MECHANICAL PROPERTIES OF EPOXY BINDERS

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Abstract. The effect of various types of curing agents and reactive diluents on the rheological and mechanical properties of epoxy binders was investigated. "Etal Inject-T" and "Larit L-285" epoxy resins were used as matrices. "Etal Inject-T", iso-MTHPA, dicyandiamide (DICY), and diaminodiphenylsulfone (DDS) were used as curing agents. The effect of reactive diluents – glycerol triglycidyl ether, pentaerythritol tetraglycidyl ether, and butyl glycidyl ether – was also studied. Maximum strength characteristics were achieved using DDS curing agent at 25%. For the "Etal Inject-T" system, compressive strength values of 140 MPa were obtained. It was shown that the addition of pentaerythritol tetraglycidyl ether at 10% simultaneously reduces the viscosity of the epoxy composition from 400 mPa·s to 280 mPa·s and increases compressive strength from 140 MPa to 155 MPa. The results demonstrate the high effectiveness of multifunctional glycidyl ethers for controlling the structure and mechanical properties of epoxy binders.

Keywords: epoxy resins, reactive diluents, curing agents, mechanical properties, viscosity, epoxy compositions.

INTRODUCTION

Epoxy resins are widely used in the production of composite materials due to their high mechanical strength, chemical resistance, adhesion to various materials, and good dielectric properties. They are used as binders for reinforced composite materials, adhesives, protective coatings, sealants, and electrical insulation [1].

However, one significant drawback of epoxy resins is their relatively high viscosity, which complicates processing, impregnation of reinforcing fillers, and part forming. To improve processing characteristics, various modifying additives — in particular reactive diluents — are introduced into epoxy compositions.

Reactive diluents are low-molecular-weight compounds containing functional groups capable of participating in the epoxy resin curing process and becoming incorporated into the forming three-dimensional polymer network. Unlike inert diluents (solvents), they do not evaporate and become part of the polymer structure, significantly affecting the mechanical and rheological properties of the material.

Such diluents are typically represented by glycidyl ethers, epoxidized oils, olefin oxide compounds, and other functional monomers.

One of the primary functions of reactive diluents is viscosity reduction of the epoxy composition. This is particularly important in the production of composite materials where good impregnation of reinforcing fibers and uniform distribution of the binder are required. The introduction of diluents has been shown to significantly reduce resin viscosity, improving flowability and processability [2, 3].

For example, in study [4], the use of glycidyl methacrylate diluents reduced epoxy resin viscosity from 8592 mPa·s to 1900 mPa·s without the use of organic solvents.

In addition to rheological characteristics, reactive diluents significantly influence the curing process of epoxy systems. The molecular structure of the diluent can alter reaction kinetics and heat release during polymerization. Study [5] established that different types of glycidyl ethers affect the peak exotherm temperature and curing rate of epoxy adhesive compositions differently. Difunctional diluents are capable of increasing crosslink density of the polymer network, whereas monofunctional compounds more often lead to increased structural flexibility.

The introduction of reactive diluents also alters the mechanical properties of epoxy materials. Studies [6, 7] have shown that the addition of diluents leads to a reduction in elastic modulus and strength, while simultaneously increasing polymer plasticity and impact toughness.

Increasing the diluent content promotes a transition in the failure mode from brittle to more ductile, which is associated with increased mobility of macromolecular segments in the three-dimensional network. Additionally, an increase in elongation at break and improved fracture toughness of epoxy systems is observed.

Reactive diluents can also affect other service properties of epoxy compositions, including wear resistance, adhesion, and thermal stability. For example, when modifying wear-resistant epoxy compositions with diluents of various chemical natures, a change in the deformation mechanism and increased resistance to abrasive wear are observed [8].

In some cases, the introduction of an optimal amount of diluent improves adhesion to various substrates through the formation of additional functional groups in the polymer structure.

Study [9] investigated the effect of reactive diluents of various functionalities on the mechanical and thermal properties of an epoxy resin based on D.E.R. 331 cured with methyl tetrahydrophthalic anhydride. XY-633 (trifunctional glycidyl ether) and XY-671 (tetrafunctional glycidyl ether) were used as diluents at 10 parts by weight per 100 parts by weight of resin.

The results showed a significant effect of diluent functionality on the mechanical properties of the epoxy material. The addition of trifunctional diluent XY-633 led to a reduction in tensile strength of approximately 19.5% and a reduction in flexural strength of 5.5% compared to the base resin. Conversely, the use of tetrafunctional diluent XY-671 led to an increase in tensile strength of approximately 18.6% and flexural strength of 7.2%. Thus, increasing the number of reactive groups in the diluent molecule promotes higher crosslink density in the polymer network and improved mechanical properties.

Furthermore, the introduction of the multifunctional diluent significantly improved the epoxy resin's resistance to low-temperature cracking. The crack initiation temperature of the base resin was $-3.9\,^{\circ}$C, whereas the addition of 10 parts by weight of XY-671 lowered it to $-32.0\,^{\circ}$C, indicating a substantial improvement in fracture toughness at low temperatures.

Thus, the results of this study demonstrate that the use of multifunctional reactive diluents simultaneously reduces the viscosity of epoxy systems and improves their mechanical properties without a significant reduction in glass transition temperature.

It should be noted that the effect of diluents on epoxy system properties is determined not only by their chemical nature but also by their concentration in the composition. Small additions may improve both processing and mechanical characteristics, whereas excessive diluent content can lead to reduced strength and thermal stability due to decreased crosslink density [2, 5, 9].

Thus, reactive diluents are an effective tool for controlling the rheological behavior and, to some extent, the mechanical properties of epoxy systems. However, despite their significant influence, the final performance of epoxy binders is largely governed not only by the presence of diluents but also by the characteristics of the curing system. In this context, the type and chemical structure of curing agents play a decisive role in the formation of the three-dimensional polymer network and, consequently, in determining the strength properties of the material.

It has been established that different curing agents lead to substantial variations in mechanical performance due to differences in crosslink density and network rigidity. In particular, aromatic curing agents such as diaminodiphenylsulfone (DDS) promote the formation of rigid and highly crosslinked structures, resulting in increased compressive and tensile strength, whereas aliphatic curing agents tend to form more flexible networks with relatively lower strength but higher toughness. Furthermore, the molecular structure and functionality of curing agents influence the curing kinetics and intermolecular interactions, which directly affect the load-bearing capacity of the polymer matrix [10, 11, 12, 13, 14, 15, 16].

In addition, not only the type but also the content and ratio of curing agent are critical parameters. Deviations from the optimal stoichiometric ratio may lead to incomplete curing or excessive crosslinking, both of which negatively affect the mechanical characteristics of epoxy materials. The importance of optimizing curing systems is also confirmed in applied studies related to composite structures and high-performance materials, where the selection of curing agents directly affects structural integrity and service performance. Therefore, the selection of an appropriate curing agent and its optimal concentration is a key factor in designing epoxy binders with improved strength properties. The relevance of optimizing curing systems is also supported by applied studies in advanced fields, including nanomaterials synthesis and rocket propulsion systems for upper stages, where material structure and performance are critically dependent on processing parameters and composition [17, 18].

Thus, the analysis of literature sources shows that reactive diluents are an effective tool for controlling epoxy resin properties. They allow for significant viscosity reduction, improved processability, and modification of the mechanical characteristics of polymers. However, the effect of diluents on the structure and properties of epoxy materials is complex and requires further investigation to select optimal epoxy composition formulations. Selecting the optimal type and amount of reactive diluent is an important task in the development of epoxy compositions [19].

Moreover, most existing studies consider the effects of reactive diluents and curing agents separately, whereas their combined influence on the rheological, mechanical, and thermal properties has been studied only to a limited extent. Insufficient data are also available for industrial epoxy systems, including Etal Inject-T and Larit L-285, especially under conditions requiring simultaneous viscosity reduction while maintaining high mechanical strength properties.

The objective of this study is to comprehensively investigate the influence of the type of curing agents and reactive diluents on the rheological and mechanical properties of epoxy binders based on Etal Inject and Larit L resins, as well as to establish the relationship between the system composition, the structure of the forming polymer network, and the performance characteristics of the material. This includes evaluating the effect of different types of curing agents on mechanical strength, studying the role of the functionality and concentration of reactive diluents in modifying the viscosity of epoxy compositions, and determining the optimal component ratios that ensure a reduction in viscosity while maintaining or improving the mechanical properties of epoxy materials.

Materials and Methods

Epoxy resins “Etal Inject” (Epital JSC, Russia) and Larit L (Lange + Ritter GmbH, Germany) were used as the base for the binder formulations.

The following curing agents were used: amine curing agent “Etal Inject-T” (Epital JSC, Russia), iso-methyltetrahydrophthalic anhydride (iso-MTHPA) (Zhejiang Heyi Chemical Co., Ltd., China), diaminodiphenylsulfone (DDS) (Huntsman, USA), and dicyandiamide (DICY) (AlzChem Group AG, Germany). Curing agent content varied in the range of 10–45%.

The following reactive diluents were used: glycerol triglycidyl ether (CAS 13236-02-7) (Sigma-Aldrich, Germany), pentaerythritol tetraglycidyl ether (CAS 3126-63-4) (Sigma-Aldrich, Germany), and butyl glycidyl ether (CAS 2426-08-6) (Sigma-Aldrich, Germany). Diluent content in the binder ranged from 5 to 20%.

The following binder characteristics were determined: viscosity of epoxy compositions and compressive strength.

Rheological properties of the epoxy compositions were determined using a RheolabQC rotational rheometer (Anton Paar, Austria). Measurements were conducted at $25 \pm 0.1\,^{\circ}$C using a coaxial cylinder measuring system. The viscosity determination procedure complied with ISO 3219 requirements. Measurements were performed under steady-state shear flow at a shear rate of $10\,s^{-1}$. No fewer than three replicate measurements were conducted for each composition, after which the mean viscosity value was calculated.

Compressive strength of epoxy binders was determined in accordance with GOST 4651–2014. Test specimens with dimensions $30 \times 10 \times 4$ mm were prepared. The loading rate during compression testing was 1 mm/min.

Before curing, all components of the composition were thoroughly mixed until a homogeneous mass was obtained, followed by vacuum degassing to remove entrapped air bubbles. Curing of samples containing the amine hardener Etal Inject-T was carried out at room temperature (25 ś 2 řC) for 24 h, followed by post-curing at 80 řC for 2 h. Systems containing iso-MTHPA were cured according to a stepwise : holding at 80 řC for 2 h, then at 120 řC for 3 h, followed by additional post-curing at 150 řC for 2 h.

For compositions containing diaminodiphenyl sulfone (DDS) and dicyandiamide (DICY), thermal curing was performed in a drying oven with programmable heating. The samples were heated to 120 řC at a rate of 23 řC/min and held for 1 h, after which the temperature was increased to 160180 řC and maintained for 24 h. After curing, the samples were gradually cooled to room temperature inside the oven in order to reduce internal thermal stresses.

Results and Discussion

The investigation of the effect of curing agents on the mechanical properties of the epoxy resin showed that their type significantly influences the strength characteristics of epoxy binders. Figure 1 presents the results of the study of the effect of curing agents on the strength characteristics of “Etal Inject” epoxy resin.

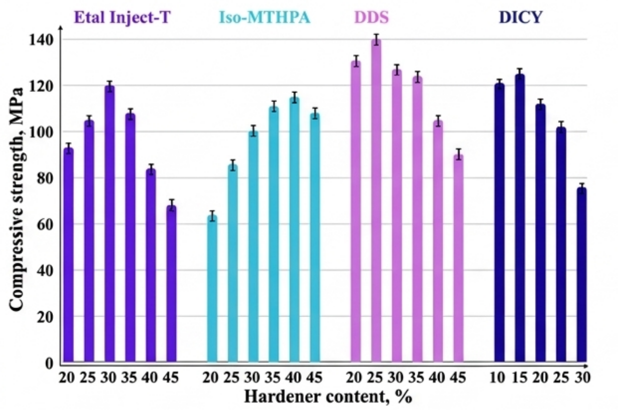

Figure 1 - Investigation of the effect of curing agents on the strength characteristics of “Etal Inject-T” epoxy resin.

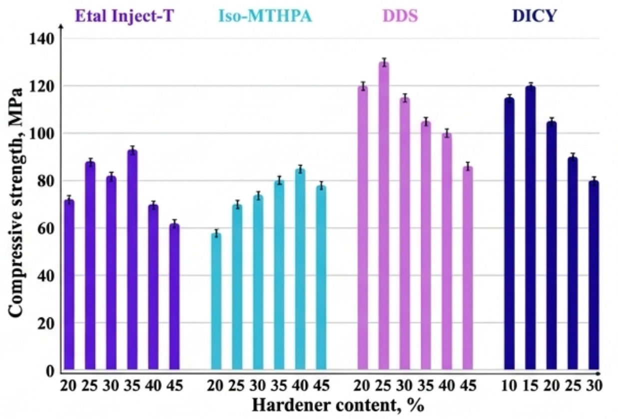

Figure 2 - Investigation of the effect of curing agents on the strength characteristics of “Larit L” epoxy resin.

Figure 2 presents the results of the investigation of the effect of curing agents on the strength characteristics of “Larit L” epoxy resin.

The results showed that the binder composition based on “Etal Inject” epoxy resin with the addition of diaminodiphenylsulfone at 25% exhibits the highest compressive strength of 140 MPa.

This compressive strength value exceeds by 16% the strength characteristics obtained when using the standard amine curing agent “Etal Inject-T” for “Etal Inject” epoxy resin.

For “Larit L” resin, the same trend holds: with 25% diaminodiphenylsulfone, compressive strength is 130 MPa.

The high strength characteristics of these systems are attributed to the specific chemical structure of diaminodiphenylsulfone. The presence of aromatic rings and a sulfone group promotes the formation of a rigid and thermally stable polymer network with high crosslink density. In addition, the aromatic structure of DDS increases polymer chain stiffness and resistance to deformation.

Diaminodiphenylsulfone is an organic compound containing two amine groups ($-NH_2$) located on phenyl rings connected to the carbon chain via a sulfone group ($-SO_2$). These amine groups react with epoxy groups, initiating the curing process. Curing of the epoxy resin begins with the reaction between the epoxy groups of the resin and the amine groups of the DDS curing agent. This reaction proceeds via a nucleophilic substitution mechanism, in which the amine group, acting as a strong nucleophile, attacks the carbon atom of the epoxy group bonded to the oxygen atom.

The conducted study demonstrates the possibility of producing materials with high strength characteristics through optimal combination of epoxy resins and curing agents.

Investigation of the effect of reactive diluents on the viscosity and strength of the binder showed that the introduction of diluents significantly affects both the viscosity and mechanical properties of epoxy compositions. Figures 3 and 4 present data on the effect of reactive diluents on the viscosity and strength of the epoxy binder.

The experimental results showed that increasing the pentaerythritol tetraglycidyl ether content to 20% reduces epoxy resin viscosity from 400 mPa·s to 140 mPa·s. Pentaerythritol tetraglycidyl ether is the most effective reactive diluent for viscosity reduction of the epoxy resin.

The addition of pentaerythritol tetraglycidyl ether reduces the concentration of high-molecular-weight components in the system, leading to a decrease in the average molecular weight per unit volume and a weakening of internal viscous forces due to shortened and more mobile molecules. Pentaerythritol tetraglycidyl ether molecules have comparatively small dimensions and a flexible structure while carrying multiple epoxy groups, which facilitates the movement and interaction of epoxy resin molecules, thereby promoting viscosity reduction.

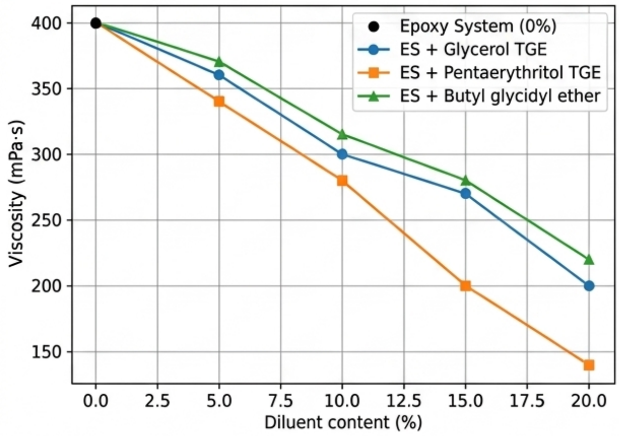

Figure 3 - Dependence of epoxy resin viscosity on diluent content.

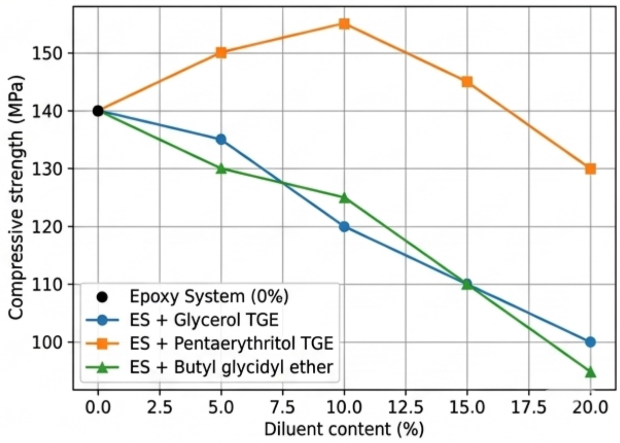

Figure 4 - Dependence of epoxy resin strength on diluent content.

The introduction of pentaerythritol tetraglycidyl ether at 10% simultaneously reduces the viscosity of the epoxy composition from 400 mPa·s to 280 mPa·s and increases compressive strength from 140 MPa to 155 MPa.

The primary effect of pentaerythritol tetraglycidyl ether addition manifests after reaction with the curing agent and consists in improving the material structure. The presence of multiple epoxy groups enables this compound to participate effectively in the formation of the polymer network. The reaction produces a polymer network in which molecules of the epoxy resin, pentaerythritol tetraglycidyl ether, and diaminodiphenylsulfone are interconnected via epoxy and amide groups, forming a strong thermoset structure.

It should also be noted that the effect of the functionality of reactive diluents on the properties of epoxy polymers is determined not only by the number of epoxy groups, but also by their ability to form a spatial network structure. Multifunctional diluents, such as pentaerythritol tetraglycidyl ether, participate in the formation of a larger number of covalent bonds, promoting the development of a denser and more uniformly crosslinked three-dimensional structure. This increases the materials stiffness and improves stress redistribution, thereby reducing the likelihood of defects and local stress concentrations.

At the same time, the high reactivity of such compounds ensures their more complete involvement in the curing process, leading to the formation of a more homogeneous polymer network. In contrast, monofunctional diluents tend to disrupt the regularity of the network by increasing the distance between crosslinking points and enhancing segmental mobility, which results in reduced stiffness and strength of the material.

Due to the reinforcing effect of the tetraglycidyl ether, the polymer acquires improved mechanical properties. The resulting material is a crosslinked polymer with a large number of epoxy and amide bonds that provide its high strength.

CONCLUSION

The conducted research established a significant effect of the type and content of curing agents and reactive diluents on the rheological and mechanical properties of epoxy binders. The choice of curing agent was shown to be one of the key factors determining the strength characteristics of the resulting polymer material. The highest strength values were obtained using diaminodiphenylsulfone (DDS). For "Etal Inject" epoxy resin at a curing agent content of 25%, maximum compressive strength of 140 MPa was achieved, which is 18% higher compared to the system cured with the standard amine curing agent "Etal Inject-T". The same trend is observed for "Larit L" epoxy resin, where the use of DDS also provides the highest strength characteristics.

Investigation of the effect of reactive diluents showed that their introduction enables effective control of the rheological properties of epoxy compositions. The most pronounced viscosity reduction is observed when using pentaerythritol tetraglycidyl ether. Increasing its content to 20% reduces composition viscosity from 400 mPa·s to 140 mPa·s, which substantially improves the processability of epoxy systems. The optimal content of this diluent was determined to be approximately 10%, at which a simultaneous reduction in viscosity from 400 mPa·s to 280 mPa·s and an increase in compressive strength from 140 MPa to 155 MPa are achieved.

Promising directions for further research include the study of the thermal characteristics of epoxy systems, including the glass transition temperature, thermal conductivity, and thermal stability under prolonged thermal loading, as well as the analysis of the combined effect of curing agents and reactive diluents on the kinetics of heat release and the formation of internal stresses during the curing process.

The obtained results demonstrate that a rational combination of epoxy resin, aromatic curing agent, and multifunctional reactive diluent enables simultaneous improvement of both the processing and service characteristics of epoxy binders.

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