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Synthesis and Performance Evaluation of Degradable Sorbitol-derived Epoxy Resins

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Electronic Supplementary Information

Abstract The introduction of dynamic covalent bonds into the structure of epoxy resins can improve the degradation performance of the materials. But to a certain extent, it will affect the insulating properties of the resin, and how to balance the insulating properties and degradation performance has become an urgent problem. In this paper, the effects of different catalysts on the thermal-force-electrical properties of sorbitol-based resins were systematically investigated based on the dynamic ester bonding to construct the resin crosslinking network, and the biobased sorbitol glycidyl ether was used as the resin matrix. The experiments show that the resin system catalyzed by triethanolamine (TEOA) exhibits excellent comprehensive performance, which combines good thermal stability and mechanical properties with excellent electrical properties (breakdown field strength of 44.21 kV/mm and dielectric loss factor of 0.29%). In addition, chemical degradation tests were conducted on the resin systems with different catalysts, and the experiments showed that the produced resins could be degraded in benzyl alcohol and exhibited good degradation performance. This study provides a theoretical basis and technical path for the development of new bio-based electrical insulating materials with both high insulation and degradation properties, which is conducive to the popularization and application of bio-based resins in the field of electrical equipment.

Keywords Bio-based; Catalyst; Degradation; Epoxy resins

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INTRODUCTION

Epoxy resins have a stable three-dimensional crosslinked structure and excellent thermal stability and electrical properties; therefore, they are widely used in a variety of fields, such as aerospace, electronics manufacturing, sporting goods, and automotive manufacturing.[1] Currently, bisphenol A glycidyl ether-based resins (DGEBA) still dominate the market. DGEBA is obtained by epoxidizing bisphenol A (BPA) with epichlorohydrin, both of which are poisonous chemical reagents that can release harmful substances over a long period of time, thus endangering human health. Moreover, polymer products containing BPA are prone to releasing BPA into the surrounding environment. BPA is structurally and functionally similar to estrogen and can interact with estrogen receptors, leading to changes in human hormone levels and a variety of related health problems.[2,3] In addition, BPA is prepared from fossil raw materials such as acetone and phenol, and its long-term use in large quantities exacerbates the consumption of non-renewable resources. Although the traditional epoxy resin has a stable three-dimensional crosslinking structure that makes it chemically stable and has excellent performance, it also causes problems of difficult solubility, degradation and recycling. When the electrical equipment is decommissioned, the waste resin cannot be recycled, and it also causes problems of resource waste and environmental pollution. To solve these problems and realize green sustainable development, it is important to develop a bio-based degradable epoxy resin with excellent performance.

To solve the difficult problem of resin degradation, researchers have introduced dynamic covalent bonds into the resin structure to improve the degradation performance of the resin and realize the degradation and recycling of the waste resin. Commonly used dynamic bonds include ester bonds,[4–8] imine bonds,[9–12] disulfide bonds[13–15] and acetal bonds.[16–18] Liu *et al.*[19] investigated the effects of different ester bond and catalyst contents on the mechanical and electrical properties of epoxy resin systems. When the ratio of the substance amounts of epoxy groups, glutaric anhydride, and zinc acetylacetonate of the resin was 1:1:0.05, the system combined better mechanical and electrical properties. Sun *et al.*[20] synthesized a degradable epoxy resin based on vanillin using a one-pot method, which introduces a dynamic Schiff base bond in the conventional epoxy-amine system, thus realizing the modification of the resin. The modified resin possessed electrical-thermal-force properties compara-

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ble to those of the conventional resin while combining good degradation properties. Li *et al*.[21] prepared a resin containing an asymmetric acetal structure using 1,4 cyclohexanedimethanol divinyl ether and phenolic resin as raw materials. The resin exhibited excellent creep resistance and reprocessing properties, and could be degraded and recycled under weakly acidic conditions.

Finding renewable bio-based materials to replace traditional petroleum-based resins is important for achieving sustainable development. Currently, commonly used bio-based materials include lignin,[22–26] vanillin,[27–31] itaconic acid,[32–38] sorbitol,[39,40] and cashew nut phenol,[41–44], *etc*. Zhou *et al*.[45] synthesized fully bio-based epoxy resins by modifying lignin to obtain multicarboxylic lignin (MGL), which has high heat resistance and good mechanical properties. Wang *et al*.[46] synthesized bio-based flame retardant epoxy resins with vanillin, diamine, and diethyl phosphite as raw materials with very high T_g (214 °C), which are expected to replace the traditional resins as new flame retardant epoxy materials. Bai *et al*.[47] prepared bio-based flame-retardant materials based on sorbitol glycidyl ether, which were highly efficiently flame-retarded by utilizing ultralow levels of phosphorus.

Although several bio-based materials have been developed, there is still a gap in research on the degradation properties of bio-based materials. Therefore, in this paper, based on sorbitol glycidyl ether to prepare bio-based resin matrix, and also based on epoxy-anhydride curing system, three common ester exchange catalysts were added to the system to prepare and obtain the degradable epoxy resin based on dynamic ester bonding, and the effects of different catalysts on the thermal-force-electrical properties of the resins were systematically investigated, and the performance was compared with that of resin systems cured by traditional catalysts. In addition, degradation experiments of each resin system were conducted using benzyl alcohol to explore the effect of different catalysts on the degradation rate of the resin system.

EXPERIMENTAL

Experimental Materials

The main raw materials used in this study were sorbitol polyglycidyl ether (SPE), 98% purity, with an average epoxy equivalent of 180 g/equiv., Hubei Shineng Chemical Science and Technology Co., Ltd; 2,4,6-triphenol (DMP-30), analytically pure, Aladdin's Reagent (Shanghai) Co; 1,5,7-triaza-bicyclo [4.4.0] deca -5-ene (TBD), purity 98%, Aladdin's Reagent (Shanghai) Co., Ltd; Triethanolamine (TEOA), purity ≥ 98%, Aladdin's Reagent (Shanghai) Co., Ltd; Zinc acetylacetonate, purity ≥ 98%, Aladdin's Reagent (Shanghai) Co., Ltd; Methylhexahydrophthalic anhydride (MHHPA), purity ≥98%, Aladdin Reagent (Shanghai) Co., Ltd. Among them, DMP-30 is a traditional catalyst, while TBD, TEOA, and zinc acetylacetonate are ester exchange catalysts. The molecular structures of SPE and MHHPA are shown in Fig. 1.

Preparation of Resin Samples

Preparation of bio-based resin system based on sorbitol: the ratio of the substance amounts of epoxy base, hardener, and catalyst was 1:1:0.05. Considering the complete dissolution of the catalyst in the resin, samples were prepared by preheating the hardener, adding the catalyst, and stirring until the catalyst was

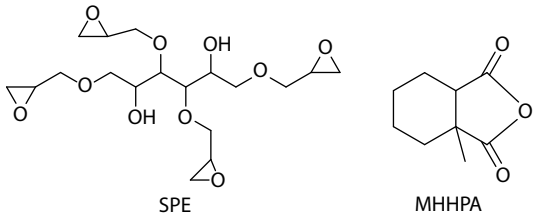

Fig. 1 Molecular structures of SPE and MHHPA.

completely dissolved. It is worth noting that DMP-30 and TEOA are liquid catalysts that can be well dissolved in the curing agent at 70 °C, whereas TBD and zinc acetylacetonate need to be dissolved better in the curing agent at 120 °C because they are solid catalysts. After the catalyst and curing agent are fully dissolved, they are cooled to a certain temperature, then added to the resin matrix and stirred thoroughly, then defoamed, and finally poured into the preheated mold for curing, and the operation flow chart is shown in Fig. 2. The curing process was determined using the exothermic curve of differential scanning calorimetry. S-DMP, S-TBD, S-TEOA, and S-Zn represent the sorbitol-based resins obtained by catalytic curing with DMP-30, TBD, TEOA, and zinc acetylacetonate, respectively.

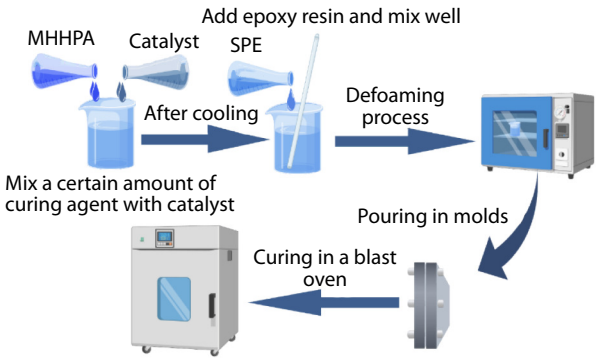

Fig. 2 Operational flow chart of resin preparation (Created with BioGDP.com).

Testing and Structural Characterization

(1) Curing reaction analysis: a differential scanning calorimeter was used to collect and analyze the DSC data of the reaction mixture. After mixing the reactants according to the proportion, 5–15 mg of the sample was placed in a crucible, and under nitrogen atmosphere, the sample was heated from 25 °C to 280 °C with the heating rate of 5, 10, 15, 20 °C/min for testing, and the heating and curing curves of the resin were recorded under different heating rates respectively, and then the curing heating curves were analysed, and the curing process of the resin was determined by using the extrapolation method.

(2) Fourier transform infrared spectroscopy (FTIR): infrared spectra of each sample were recorded using a TENSOR II (255) spectrometer. The samples were prepared by pressing potassium bromide, and the number of sample scans was set to 32, the resolution was set to 0.4 cm^{-1}, and the wavenumber scanning range was from 400 cm^{-1} to 4000 cm^{-1}.

Dynamic Thermal Performance Testing

(1) Dynamic Mechanical Analysis (DMA): the test was carried out using a Dynamic Thermo-Mechanical Analyzer (TA-Q800) under

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the following conditions: frequency of 10 Hz, heating rate of 10 °C/min, temperature range of 30–200 °C, and single cantilever mode.

(2) Thermogravimetric analysis (TGA): different resin systems were tested using a thermogravimetric analyzer (TGA 4000) under nitrogen atmosphere with a heating rate of 10 °C/min and a temperature range of 30–800 °C.

Mechanical Performance Testing

The tensile and flexural properties of each resin sample were tested using an EL104 microcomputer-controlled electronic universal testing machine in accordance with the methods and requirements specified in standards IOS 527-2-2012 and ISO 178-2010, respectively.

Electrical Performance Testing

(1) Industrial frequency breakdown field strength test: according to the requirements of the standard GB/T 24344-2009 "Specification for Voltage Resistance Tests of Industrial Machinery and Electrical Equipment," the breakdown field strength of different resin samples is tested, in which the thickness of the samples is 1 mm. Two copper spherical electrodes submerged in insulating oil were used to conduct the breakdown test of the samples, and the samples were pressurized at a constant 2 kV/s boosting rate until they broke down. The samples were pressurized at a constant rate of 2 kV/s until they penetrated, and the pressurized value of the samples at the time of penetration was recorded. Fifteen valid data points were recorded, and the results were statistically analyzed using the Weibull distribution to obtain the breakdown field strength of the sample.

(2) Dielectric loss factor test: in accordance with the requirements of GB/T 1409-2006 "Recommended Methods for Measuring the Capacitance and Dielectric Loss Factor of Electrical Insulating Materials at Industrial Frequency, Audio, and High Frequency (Including Mi-Wave Wavelengths)," the test is conducted by using the YG9100 Automatic Anti-Interference Precision Dielectric Loss Tester, with a test voltage of 2 kV, a voltage of 50 Hz, and a specimen of 50 Hz, with a test voltage of 2 kV and a voltage of 50 Hz. frequency was 50 Hz, the specimen had the shape of a cake, the radius of the bottom circle was 20 mm, and the thickness was 3 mm.

Chemical Degradation Performance

In order to avoid the influence of other interfering factors on the experiment, the size of the sample for degradation performance test was uniformly set to 40 mm (length) × 10 mm (width) × 4 mm (thickness), and the samples of different resin systems were immersed in a mixture of 120 g benzyl alcohol and 12 g tripotassium phosphate. The degradation test was carried out at 190 °C, and the samples were taken out and weighed every one hour, and the change in the mass of the samples was observed and recorded.

RESULTS AND DISCUSSION

Kinetic Analysis of Curing Reactions

The curing reaction conditions of different system resins are different, and the design of the curing process is of great significance in the practical application of resins in engineering. Different curing processes will have an impact on the performance of the resin. In this paper differential scanning calorimetry was used to study the curing reaction kinetics of the sorbitol-based bio-based resin system and to determine the optimal curing process of the resin. The curves of the curing reaction of the SPE/MHHPA/catalyst mixture are shown in Fig. 3, from which it can be seen that the exothermic curves of the four resin systems contain only a single exothermic peak. The curing reaction curves of the four bio-based resin systems with different catalysts, and the data of the corresponding three characteristic temperatures of the curing — the onset curing temperature T_i, the peak curing temperature T_p, and the termination curing temperature T_e are shown in electronic supplementary information. And it can be seen that the data show that the characteristic temperatures of the four resin systems are similar to each other, and therefore, for the four resin systems, there are two exothermic peaks. Therefore, the same curing conditions were used for the four resin systems, and the curing process was determined from the linear fitting results as 145 °C/2 h+170 °C/5 h.

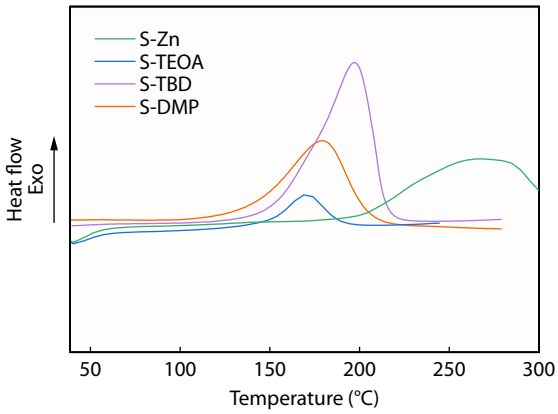

Fig. 3 Exothermic curves of different resin systems at 20 °C/min.

FTIR Spectroscopy

The infrared spectra of the bio-based resin systems with the four different catalysts before and after curing are shown in Fig. 4. The peak at 910 cm^{-1} corresponds to the absorption peaks of epoxy groups, 1850 cm^{-1} corresponds to the absorption peaks of anhydride groups, 1736 cm^{-1} corresponds to the absorption peaks of carbonyl groups in ester bonds (C═O), and 1084 cm^{-1} corresponds to the absorption peaks of ether bonds in ester bonds (R—O—R).

As can be seen from Fig. 4, the mixture of resin and curing agent has obvious absorption peaks of epoxy groups and anhydride groups in the system before curing. After the mixture is cured by the programmed temperature, the absorption peaks of the epoxy and anhydride groups are greatly reduced, and the absorption peaks of the ester bond appear in the system, which indicates that the epoxy groups react with the anhydride groups, and the epoxy groups are opened to form an ester bond, and the molecular crosslinking is completed to form an epoxy polymer.

Dynamic Mechanical Analysis

Under the action of a sinusoidal alternating stress field, epoxy resins exhibit typical viscoelastic behavior with a phase lag angle δ ($0°<\delta<90°$) between the strain response and stress excitation. Dynamic thermomechanical analysis (DMA) can be used to obtain the characteristic parameters of the dynamic mechanical

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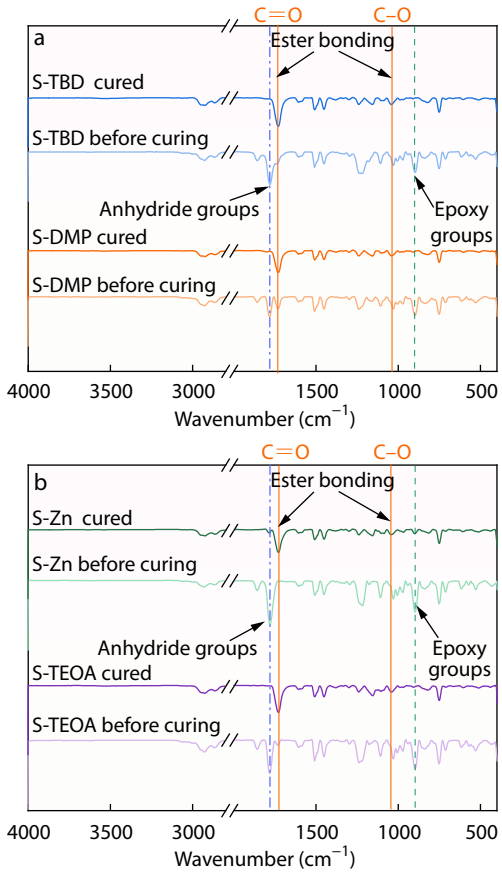

Fig. 4 Infrared spectra of bio-based resin systems with different catalysts before and after curing: (a) S-TBD and S-DMP systems; (b) S-Zn and S-TEOA systems.

properties that characterize the intrinsic dissipative properties of the material: the energy storage modulus (E') and loss factor (tanδ).

The energy storage modulus and loss factor versus temperature curves for the bio-based resin systems with the four different catalysts are shown in Fig. 5 and Fig. 6, respectively.

The glass transition temperature of a resin material is an

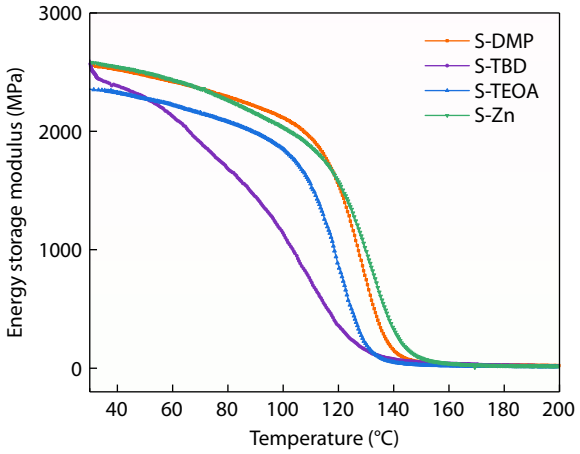

Fig. 5 Variation curves of energy storage modulus with temperature for bio-based resin systems with different catalysts.

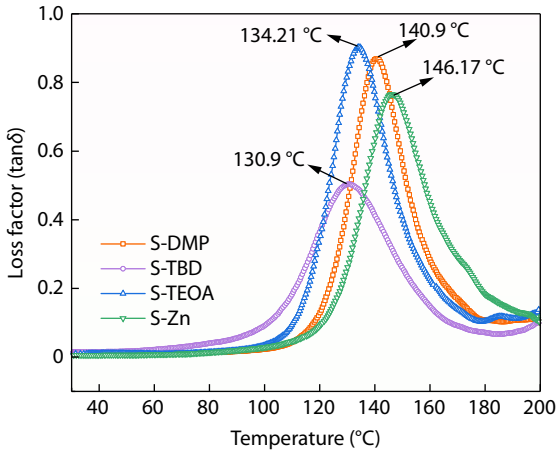

Fig. 6 Variation curves of loss factor with temperature for bio-based resin systems with different catalysts.

important parameter for assessing its heat resistance, and the peak point temperature of the loss factor is considered as T_g. The crosslinking density of the resin system and the structure of the rigid chain segments can have a significant effect on T_g, and according to the theory of rubber elasticity, the crosslinking density υ_e can be expressed as[48]

$$\upsilon_e = \frac{E'}{3RT} \quad (1)$$

where E' is the energy storage modulus of the rubber state, generally taken as the energy storage modulus corresponding to T_g+30 °C in Pa; R is the gas constant, the value of which is taken as 8.314 J/(mol·K); and T is the thermodynamic temperature in K.

The crosslink density and glass transition temperature (T_g) of the bio-based resin systems with four different catalysts are listed in Table 1, and the variation rule of the loss factor (tan δ) with temperature for the resin systems with different catalysts is shown in Fig. 6. From the data in Table 1, the T_gs of the four resin systems, S-DMP, S-TBD, S-TEOA, and S-Zn, were 140.9, 130.9, 134.2, and 146.2 °C, respectively. Overall, the addition of the ester-exchange catalyst resulted in a large number of dynamic ester bonds in the system, leading to a decrease in T_g. Among them, the S-TBD resin system has the lowest T_g and the largest crosslink density, which is due to the fact that TBD is a kind of catalyst that rapidly catalyzes the ester exchange reaction, on the one hand, the structure of TBD has a strongly basic bicyclic guanidinium group, and on the other hand, it has a suspending hydrogen that exhibits a weak acidic nature, so it has a dual catalytic effect and the reaction activity is higher than that of the remaining three catalysts. Therefore, in the process of resin curing, TBD can accelerate the reaction rate of the ester exchange reaction in molecules to a greater extent and promote the generation of a polyester-type crosslinking network in the system, so that the resin curing is more complete and the crosslinking density of the resin system is increased. Simultaneously, because of the high catalytic reaction activity of TBD, the resin system is transformed from a glassy state to a rubbery state at a lower temperature, resulting in a lower glass transition temperature of the resin system. In addition, the S-Zn resin system has the largest T_g, which is due to the higher crosslink density of the S-Zn system on the one hand, and the lower reactivity of

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Table 1 Crosslinking density of bio-based resin systems with distinct catalytic mechanisms.

Resin type	T_g (°C)	T_g+30 (°C)	T (K)	E' (MPa)	Crosslinking density (mol/m^3)
S-DMP	140.9	170.9	443.9	27.4	2473.0
S-TBD	130.9	160.9	433.9	37.8	3489.1
S-TEOA	134.2	164.2	437.2	18.7	1718.5
S-Zn	146.2	176.2	449.2	22.7	2028.9

zinc acetylacetonate as a catalyst, so that the transition from the glassy to the rubbery state is realized only at higher temperatures, thus leading to a high T_g of the S-Zn system.

Thermal Stability

Thermogravimetric analysis can be used to determine the initial decomposition temperature ($T_{5\%}$), maximum decomposition rate temperature (T_{max}), and residual carbon rate of the materials, and then quantitatively evaluate their thermal stability performance. Fig. 7 shows the evolution of the thermogravimetric curves of the bio-based resin systems with different catalysts, and the characteristic thermal decomposition parameters of each resin system are summarized in Table 2 after systematic characterization. As can be seen in Fig. 7, the thermal decomposition temperatures of the four resin systems with different catalysts were in the range of 280–450 °C, among which the S-TBD system had the lowest initial decomposition temperature of 297.3 °C and the temperature of maximum decomposition rate

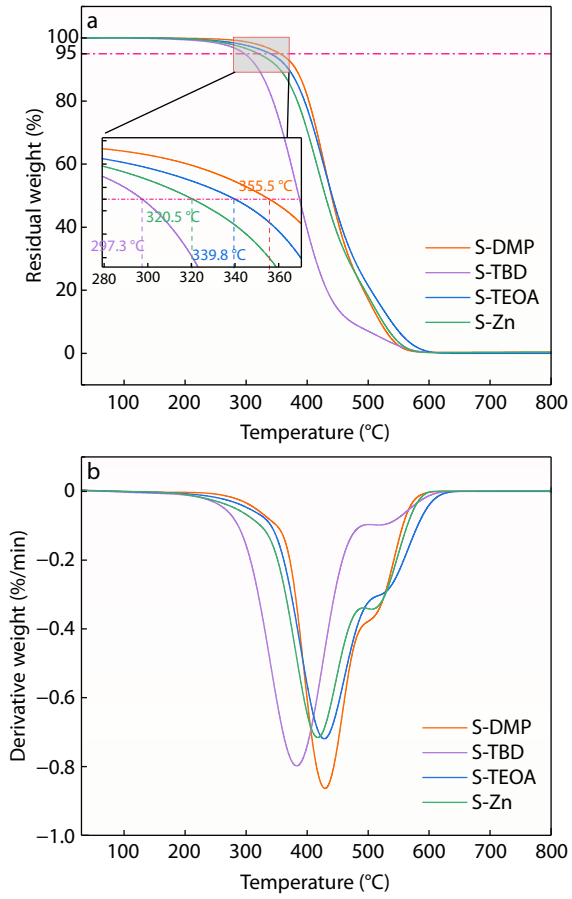

Fig. 7 TGA test results of bio-based resin systems with different catalysts: (a) TG test curves; (b) DTG test curves.

was also the lowest, which was only 386.2 °C. The low thermal decomposition temperature of the S-TBD system may be related to the high activity of the TBD-catalyzed transesterification reaction. From the previous DSC exothermic curves, the highest exothermic peak of the S-TBD system was observed at a heating rate of 20 °C/min, indicating the highest reactivity of TBD. For a fully cured resin, the catalyst is dispersed in the resin structure. When the sample was warmed up for TGA testing, the high temperature activated the dynamic ester bonds in the molecular structure. Under the combined effect of high temperature and catalyst, dynamic esterification reactions occur in the resin structure, and molecular chain segments are rearranged, a process that produces free groups and small molecule chain segments. At the same time, the high temperature leads to volatilization of small molecules, which decreases the quality of the sample. The higher the catalyst activity, the faster the rate of the dynamic transesterification reaction, the more free groups and small molecule chain segments are generated, and the faster the sample quality decreases under high temperature, which ultimately leads to a decrease in the decomposition temperature of the sample, and thus the thermal decomposition temperature is lower for the S-TBD system.

Table 2 TGA parameters of bio-based resin systems with distinct catalytic mechanisms.

Resin type	$T_{5\%}$ (°C)	T_{max} (°C)
S-DMP	355.5	429.3
V-TBD	297.3	382.8
V-TEOA	339.8	428.2
V-Zn	320.5	417.5

Mechanical Performance

Mechanical performance, as a key evaluation index of intrinsic material properties, has a direct impact on the long-term reliable operation of epoxy-based cast electrical equipment. The tensile and bending strengths, as core evaluation parameters of material deformation resistance, have important characterization values in engineering application scenarios. The tensile and bending strengths as well as the elongation at break of the bio-based resin systems with different catalysts are shown in Fig. 8.

A comparison of the mechanical properties of S-DMP and the remaining three resin systems in Fig. 8 shows that the resin system cured by conventional catalysts had high flexural and tensile strengths, in which the values of S-TEOA were similar to those of S-DMP, and the mechanical properties of S-Zn showed the worst performance. No ester exchange reaction occurred in the resin structure at room temperature, so the difference in the mechanical properties of the resins may be related to the crosslinked network structure of the resin. TEOA, as a reactive catalyst, has higher activity in catalyzing the ester exchange reaction; therefore, the content of dynam-

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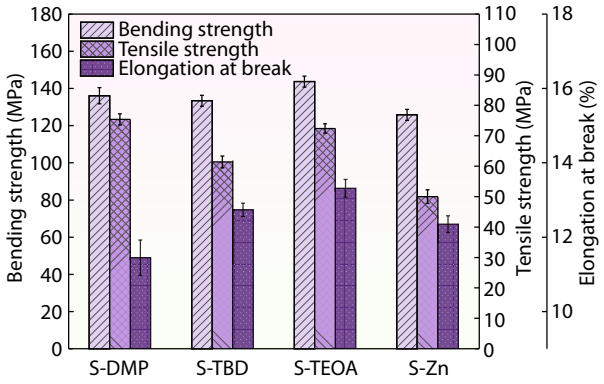

Fig. 8 Bending strength of bio-based resin systems with different catalysts.

ic ester bonds in the molecular structure of the resin is also higher, and the percentage of the flexible ester bonding group is increased, so the bending strength of the system also increases accordingly. In addition, from the previous analysis, it can be seen that TBD is a catalyst with high reactivity, because its catalytic reaction rate is too fast, resulting in the rapid occurrence of the crosslinking reaction, the reaction exothermic exacerbation, the local reaction is too fast, and it will be introduced in the crosslinking network more defects, so that the mechanical properties of the S-TBD resin system are not as good as those of the S-DMP. For the zinc acetylacetonate catalyst, its catalytic activity is relatively low, so the proportion of flexible groups in the structure is relatively low, resulting in a slightly worse bending strength.

However, as can be seen from Fig. 8, the elongations at break of the resin systems based on the ester exchange catalysts are all higher than that of the S-DMP system, indicating that the introduction of dynamic ester bonding enhanced the toughness of the resin systems. The use of an ester exchange catalyst increases the flexible molecular chain segments in the resin structure, so the systems have a higher elongation at break, with the S-TEOA system having the highest elongation at break of 13.32%. The high toughness of the resin material helps improve the structural stability of the epoxy cast electrical equipment and maintains the long-term stable operation of the equipment.

Electrical Performance Testing

Industrial frequency breakdown field strength

The breakdown field strengths of the bio-based resin systems with different catalysts are shown in Fig. 9. As shown in the figure, the breakdown field strengths of the four resin systems, S-DMP, S-TBD, S-TEOA, and S-Zn, were 44.2, 42.1, 43.3, and 40.71 kV/mm, respectively. This result shows that the introduction of dynamic ester bonding in the resin system affects the electrical properties of the system to a certain extent, and the breakdown field strength of the resin is reduced. Dynamic covalent bonding is different from covalent bonding in a traditional resin system, which undergoes a dynamic exchange reaction under certain conditions, affecting the crosslinking density of the resin structure and leading to the movement of carriers, resulting in a decrease in the electrical properties. From the results of the previous dynamic thermo-mechanical analysis, it can be seen that the S-TBD resin system has the highest crosslinking density and the catalyst has the highest reactivity; therefore, on the one hand, the molecular structure of high crosslinking density leads to the difficulty of carrier movement in the resin structure, and the breakdown field strength is correspondingly larger; on the other hand, due to the high reactivity of this catalyst, TBD, the high crosslinking density of the structure also results in a more likely occurrence in the dynamic ester exchange reaction, resulting in reduced electrical properties. On the other hand, owing to the high reactivity of TBD as a catalyst, the high crosslinking density structure also leads to easier dynamic ester exchange, which leads to a decrease in the electrical properties. Therefore, the breakdown field strength of the S-TBD system with a high crosslinking density was lower than those of the S-DMP and S-TEOA resin systems. In addition, the S-Zn resin system had the lowest breakdown field strength, as shown in Fig. 9. Although the crosslinking density of this system is higher, the catalyst produces metal ions synchronously in the process of promoting the ester exchange reaction, which influences the conductive ability of the resin system and decreases the insulation strength. At the same time, owing to the small amount of catalyst added, the introduction of zinc ions in the system is small, which has little effect on the insulation properties of the system, and the S-Zn resin system still has a high breakdown field strength.

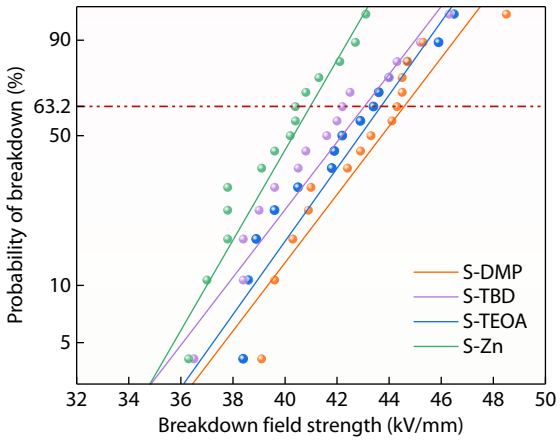

Fig. 9 Breakdown field strength of bio-based resin systems with different catalysts.

Dielectric loss factor

The measured dielectric loss factors of the bio-based resin systems with different catalysts are shown in Fig. 10. The dielectric loss factors of the four resin systems, S-DMP, S-TBD, S-TEOA, and S-Zn, are 0.32%, 0.33%, 0.29%, and 0.36%, respectively. Under an alternating electric field, the polar groups in the resin structure undergo steering polarization to produce loss. From the structure of SPE in Fig. 1, it can be seen that there are strong polar hydroxyl groups in SPE, and the resin crosslinking network in this study is based on the "epoxy-anhydride" curing system. There are a large number of ester bonds in the system, and the presence of these polar groups will increase the loss of the system.

Among them, the dielectric loss factor of the S-TEOA system was smaller, although hydroxyl groups also existed in the structure of triethanolamine; however, because triethanolamine was a reactive catalyst, the hydroxyl groups in its structure would participate in the crosslinking reaction, re-

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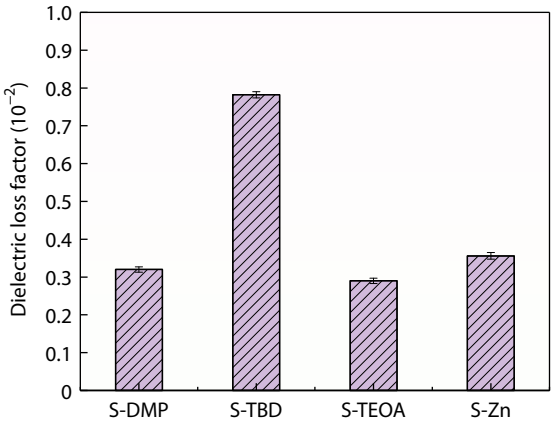

Fig. 10 Dielectric loss factor of bio-based resin systems with different catalysts.

sulting in the number of hydroxyl groups in the system instead of decreasing. The reaction of triethanolamine as a catalyst was not as violent as that of TBD, and the defects introduced into the system were relatively few, resulting in a lower dielectric loss. The dielectric loss factor of the S-Zn system is larger, which may be due to the introduction of zinc acetylacetonate as a catalyst, which increases the metal ion content of the crosslinking system and increases the conductivity of the system, thereby increasing the conductivity loss of the system. Thus, the dielectric loss is larger, which is similar to the conclusions of the previous analysis of the breakdown field strength.

Chemical Degradation Performance

The ester exchange reaction refers to the dynamic reorganization between ester groups and hydroxyl groups through the reversible intermolecular exchange of ester groups between esters and alcohols under the condition of a catalyst or thermal activation to produce new esters and corresponding alcohols (or acid-ester complexes). According to this reaction principle, benzyl alcohol was chosen as the alcohol solvent and tripotassium phosphate was chosen as the catalyst for the chemical degradation of the resin in this study. Under the joint action of high temperature and catalyst, benzyl alcohol as a solvent can play the role of "swelling," making the resin structure loose, which is conducive to the subsequent ester exchange reaction in the molecular chain. On the other hand, the hydroxyl nucleophilic attack of benzyl alcohol on the ester bonding sites in the molecular chain of the resin rearranges the ester bonding of the resin macromolecule structure and breaks the molecular chain. However, the hydroxyl nucleophilic group of benzyl alcohol attacks the ester bond site in the molecular chain of the resin, causing the ester bond in the macromolecular structure of the resin to be rearranged, the molecular chain to be broken, and the polymer to be decomposed into epoxy oligomers, which are dispersed in the solvent of alcohols,[34] ultimately realizing the

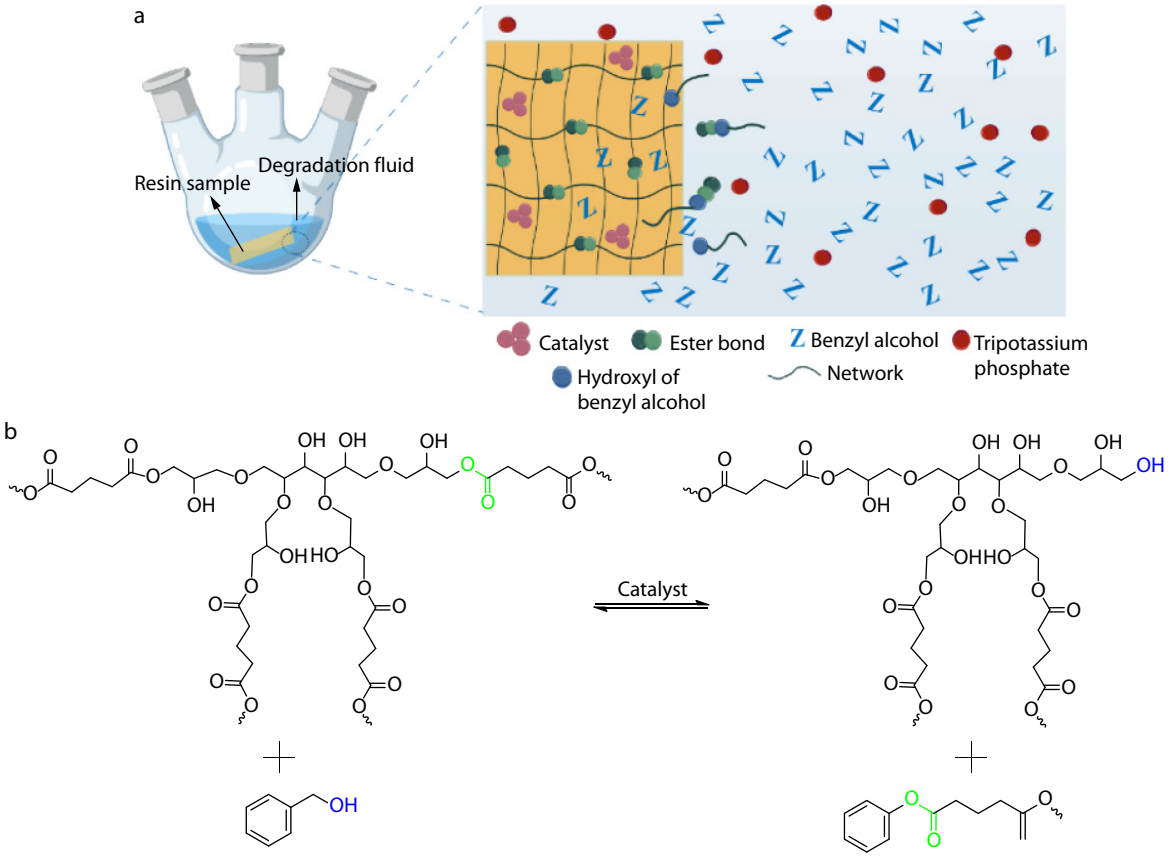

Fig. 11 Degradation process of epoxy resin in ethylene glycol solution and principle of ester exchange reaction: (a) schematic representation of the degradation process of an ester exchange-based epoxy resin (Created with BioGDP.com); (b) principles of ester exchange reactions in resin systems (As an example, one ester bond is involved in the reaction).

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degradation of the epoxy resin network. The degradation process of the resin system based on this reaction principle and a schematic diagram are shown in Fig. 11.

Chemical degradation experiments were carried out on samples of four different resin systems in a mixed solution containing 120 g of benzyl alcohol and 12 g of tripotassium phosphate at 190 °C. The appearance change diagrams of the different resin systems and the change curves of the residual mass of the samples were obtained, as shown in Fig. 12 and Fig. 13. From the appearance change diagrams during resin degradation in Fig. 12, it can be seen that in the process of resin degradation, the resin cannot maintain its original shape, the corners of the resin gradually disappear, and the surface becomes uneven. This is because in the degradation process of the resin sample, owing to the catalyst and high temperature, the crosslinked dense structure of the resin is "dissolved" by the solvent, the molecular structure becomes loose, and a large number of hydroxyl groups in the solvent undergo a dynamic ester exchange reaction with the ester bond in the crosslinked structure. The ester bond is rearranged, and the macromolecular structure of the resin is destroyed, the molecular chain is broken, and the macromolecular structure is damaged. The molecular structure of the resin is destroyed, the molecular chain is broken, and the macromolecular polymer becomes an epoxy oligomer, which macroscopically shows that the overall volume of the resin is reduced, thus realizing degradation of the resin.

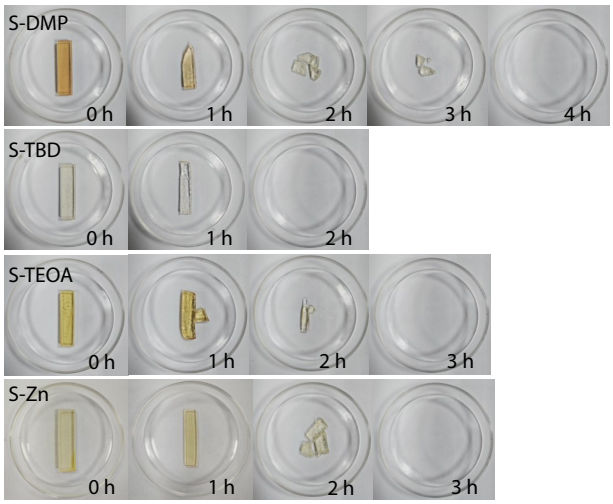

Fig. 12 Changes in sample appearance during degradation of different resin systems.

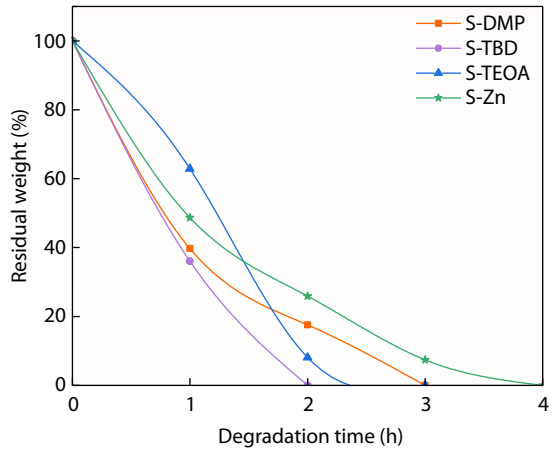

Fig. 13 Changes in residual mass upon degradation of different resin systems.

As shown in Fig. 13, the degradation rate of the S-TBD resin system was the fastest, and the degradation was complete within 2 h. As mentioned before, TBD is a type of ester exchange reaction catalyst with a double catalytic effect, and its catalytic ester exchange reaction has high activity and violent reaction; therefore, in the degradation process, the rate of ester bond rearrangement is also accelerated, which leads to an accelerated rate of resin degradation. For the S-TEOA and S-Zn resin systems, because of the relatively low reactivity of these two types of catalysts, the rate of ester bond rearrangement during degradation was also lower, the rate of molecular chain breakage was slower, and the rate of resin degradation was correspondingly slower.

After the degradation solution of the resin was subjected to filtration and spin-evaporation treatment, an IR test was performed, and the curve was obtained, as shown in Fig. 14. As shown in the figure, the absorption peak of the hydroxyl group was added to the degraded product. In addition, a small deviation in the absorption peak of the ester bond occurred, and the absorption peak of the mono-substituted benzene ring also appeared, indicating that the ester bond in the structure of the resin opened to generate hydroxyl groups during the degradation process, and benzyl alcohol was involved in the transesterification reaction to generate benzyl alcohol esters, which is in accordance with the degradation principle shown in Fig. 11(b).

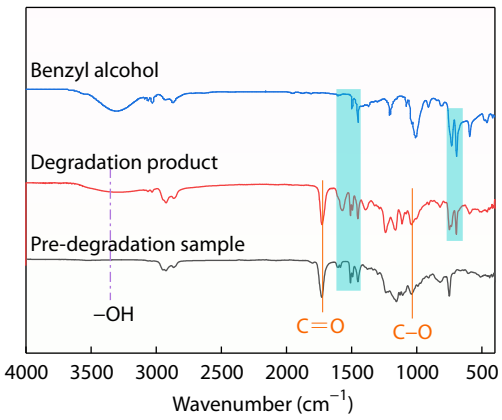

Fig. 14 Infrared characterization of degradation product.

CONCLUSIONS

In this study, four different catalysts were used to construct a bio-based resin system based on sorbitol, and bio-based resins with different catalyst systems were tested for thermal, mechanical, and electrical properties. The structure of the resin was characterized by infrared spectroscopy, and the chemical degradation properties of the four resin systems were also investigated. The S-TEOA resin system had the best comprehensive performance, which provided a theoretical basis for the en-

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gineering of sorbitol-based resin. Among them, the S-TEOA resin system had the best overall performance, which can provide a theoretical basis for the engineering application of sorbitol-based resins:

(1) The thermal decomposition temperatures of the four different catalyst systems of bio-based resins were in the range of 280–450 °C, except for the S-TBD system. The other three resin systems had higher thermal decomposition temperatures, among which the onset of decomposition temperature ($T_{5\%}$) of S-TEOA was as high as 355 °C, the maximum degradation rate temperature (T_{max}) was as high as 440.8 °C. Thus, the S-TEOA resin system had good thermal properties.

(2) The electrical properties of the bio-based resins with four different catalyst systems were tested, in which the breakdown field strength of the S-TEOA resin system was the largest, 44.21 kV/mm, and the breakdown field strength of S-Zn was the lowest, 40.71 kV/mm. In addition, the dielectric loss factor of S-TEOA was the lowest at only 0.29%. Therefore, the S-TEOA resin system exhibited the best overall electrical performance.

(3) The bending performance test results showed that the four bio-based resins with different catalyst systems exhibited good bending performances. Among them, the bending strength of the S-TEOA resin system was the largest, up to 142.77 MPa, and the bending strength of the S-Zn resin system was the smallest (120.82 MPa. Therefore, the mechanical properties of the S-TEOA resin system are optimal.

(4) In the mixed solution of benzyl alcohol and tripotassium phosphate, the bio-based resins with four different catalyst systems could be degraded, and the degradation performance of the S-TBD resin system was the best, and could be completely degraded within 2 h under the experimental conditions. Under the experimental conditions, this system achieved complete degradation within 2 h, while the S-DMP resin system had the slowest degradation rate, and the degradation was complete within 4 h.

The experimental results showed that the sorbitol-based bio-based resin system catalyzed by triethanolamine (TEOA) exhibited excellent comprehensive performance with good thermal-electrical-force properties. In addition, the resin system can be degraded in a compound system of benzyl alcohol/tripotassium phosphate, which combines the dual properties of high performance and controlled degradation, providing a new strategy for the development of environmentally friendly alternative materials. Bio-based resins are expected to serve as substitutes for traditional petroleum-based materials and become a new insulating material matrix in the field of green electrical equipment.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at http://doi.org/10.1007/s10118-025-3417-4.

Data Availability Statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request. Author's contact information: 15274692771@163.com.

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