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Formulation design of a new type of epoxy resin-based insulating adhesive and research on its high temperature resistance

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Abstract. A series of new epoxy resin adhesives were prepared by low viscosity bisphenol A epoxy resin E-44 and E-51as the matrix, low molecular polyamide 650# and phenolic modified JT-3008 as the curing agent, silicon micro powder and calcium carbonate as the fillers. The gelation time, surface drying time and shear strength of the samples were deeply tested. The thermal decomposition temperature and high temperature resistance of the samples were determined by TG analysis. Meanwhile, the surface fracture morphology was observed by SEM. The results showed that a new kind of heat epoxy resin insulation adhesive with long gelation time and convenient construction and meeting the requirements of production adjustment was successfully obtained through the optimization design of the formula,. The new epoxy resin insulation adhesive showed excellent mechanical properties with 18.00 MPa shear strength, which can be used at 380 ℃ for a short period of time and 130 ℃ for a long time. The new adhesive developed in this paper can be widely used in high voltage switchgear and other insulation materials, which has good application prospects.

Keywords: epoxy resin; insulating adhesive; high temperature resistance; room temperature curing

1. Introduction

High-voltage switchgear is a very important power distribution equipment, and insulation capacity is a key factor that determines the service life of high-voltage switchgear, whether it can operate safely and stably, and the probability of accidents [1]. Therefore, the reasonable selection of insulating materials is important for high-voltage switchgear. The performance and comprehensive value of equipment play a crucial role [2, 3]. Among them, insulating adhesive is an indispensable insulating material in the design and installation of high-voltage switchgear equipment. At present, insulating adhesives are mainly used in the fields of main insulation mica tapes of switchgear and electrical equipment, such as bonding and potting [4]. Nowadays, with the rapid development of the power network, higher requirements are also placed on electrical appliances. The development and research of high-voltage, high-power, large-capacity, and high-performance electrical appliances has become an inevitable development trend. The matching insulating adhesive also needs to have the advantages of excellent adhesion, chemical stability, mechanical properties, heat resistance, flame retardancy, low hygroscopicity and thermal conductivity [5, 6]. At present, the main types of commonly used insulating adhesives are: epoxy resin, silicone resin, modified phenolic resin, etc. Among them, epoxy resin is recognized as the main insulation with the most useful value and application prospects for electrical appliances today because of its excellent adhesion, chemical stability, mechanical properties, insulation, easy processing and low cost. one of the materials.

In order to meet the high standard requirements of electrical and electrical insulation adhesives for high temperature resistance, heat dissipation and insulation, the development and development of new high temperature epoxy resin insulation adhesives has become an important topic that many researchers are currently concerned about. In this paper, low-viscosity bisphenol A epoxy resin E-44 and E-51 are used as the matrix, low molecular polyamide 650# and phenolic modified amine JT-3008 are used as the curing agent, and silicon powder and calcium carbonate are used as fillers. A new type of high temperature resistant epoxy resin insulating adhesive with longer gel time,

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convenient construction and meeting the requirements of actual production of glue was obtained through the development of the formula, and its high temperature resistance and mechanical properties were tested. The results show that the new epoxy resin insulating adhesive has excellent mechanical properties, and the shear strength can reach up to 18.00 MPa; it can not only be used for a long time at about 130 ℃, but also has a short-term temperature resistance of up to 380 ℃. It has excellent application prospects in terms of bonding.

2. Experimental part

2.1 Experimental raw materials

Bisphenol A epoxy resin E-44 and E-51, industrial grade, Yueyang Baling Huaxing Petrochemical Co., Ltd.; low molecular polyamide 650#, industrial grade, Shandong Deyuan Epoxy Technology Co., Ltd.; phenolic modified amine JT -3008, industrial grade, Shanghai Jingtian New Material Technology Co., Ltd.; silicon micropowder, 600 mesh, Foshan Yuanlei Powder Co., Ltd.; calcium carbonate, 600 mesh, Foshan Yuanlei Powder Co., Ltd.; toughener DOP, Analytical grade AR, Tianjin Damao Chemical Reagent Factory; Silane Coupling Agent KH550, industrial grade, Jinan Dahui Chemical Technology Co., Ltd.; Acetone, analytical grade AR, Sinopharm Chemical Reagent Co., Ltd.; Anhydrous ethanol, analytical grade AR, Tianjin Kaitong Chemical Reagent Co., Ltd.

Table 1. Reagent parameters

Reagent Type	Color	Epoxy Value mol/(100g)	Amine Value (mgKOH/g)
E-44	light yellow viscous body	0.41-0.47	-
E-51	light yellow viscous body	0.48-0.54	-
JT-3008	orange homogeneous liquid	-	480-520
650#	orange red viscous liquid	-	180-240

2.2 Experimental equipment

CMT4104 Microcomputer Control Electronic Universal Testing Machine, Meters Industrial Systems (China) Co., Ltd.; DHG-9053A Electric Heating Constant Temperature Drying Oven, Shanghai Jinghong Experimental Equipment Co., Ltd.; PTYE5120 Electronic Analytical Balance, Fuzhou Huazhi Scientific Instrument Co., Ltd.; STA409PC comprehensive thermal analyzer, NETZSCH Instrument Manufacturing Co., Ltd., Germany; FTIR-6700 Fourier Transform Infrared Spectrometer, Thermo Fisher Co., Ltd., USA; SU8010 Super Resolution Field Emission Scanning Electron Microscope, Hitachi, Japan.

2.3 Experimental design scheme

Using the single factor experiment method, the amount of fillers such as fixed silica powder, calcium carbonate and toughening agent is unchanged, and only the types of epoxy resin and curing agent are changed in single compounding; in compounding, only epoxy resin and curing agent are changed. The compounding ratio of the curing agent was tested and characterized, and the gel time, surface drying time, shear strength, thermal weight loss decomposition temperature, etc. of each system were tested and characterized, and the surface fracture morphology of the samples was observed by means of SEM, and the comparative analysis was carried out. Thereby the optimal adhesive formulation is obtained. The specific formula design is shown in Table 2.

Table 2. Formula design parameters

Experimental formula	E-44	E-51	JT-3008	650#
1#	100	0	0	100
2#	0	100	0	100
3#	100	0	36	0
4#	0	100	44	0
5#	100	0	18	50
6#	0	100	22	50
7#	30	70	0	100
8#	30	70	40	0
9#	30	70	20	50
10#	50	50	0	100
11#	50	50	42	0
12#	50	50	21	50
13#	70	30	0	100
14#	70	30	38	0
15#	70	30	19	50

3. Results and Discussion

3.1 Gel and surface drying time test

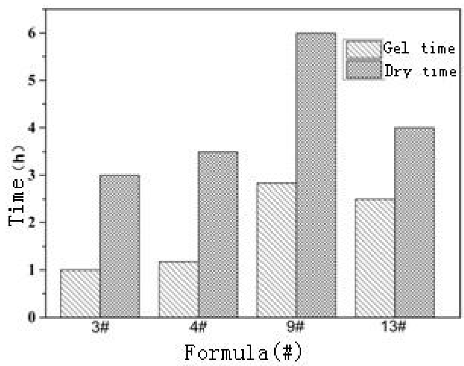

Figure 1. Gelation time and surface drying time

In order to explore the effect of curing agent on the gel time and tack-free time of the samples, the gel time and tack-free time of some formulations were selectively tested in this paper. From the experimental results in Figure 1, it can be seen that the gel time of all formulation systems has reached more than 1 h, compared with the gel time required by Ning Baojiang et al. [7] to use polyene polyamine curing agent for curing at room temperature 25 min, the gel time of the system obtained by the formulation in this paper has increased significantly. A longer gel time is not only conducive to prolonging the service life of the adhesive, but also facilitates subsequent construction, so that it can better meet the actual production requirements for glue adjustment.

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3.2 Shear strength test

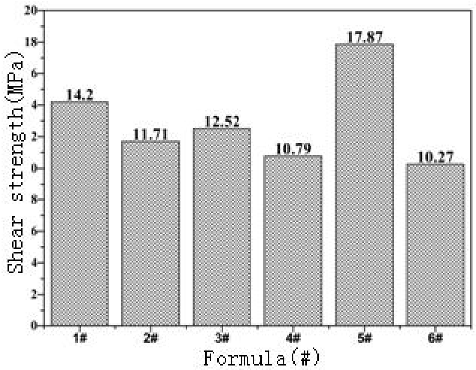

Figure 2. Shear strength of one component cured epoxy resin

From the experimental results of the shear strength of the one-component epoxy resin cured product in Figure 2, it can be seen that the one-component formulation system with E-44 as the main resin has higher shear strength, and the curing agent 650# can significantly improve the formulation compared with JT-3008. E-44 is used as the main resin, and the curing agent is 650# and JT-3008. Its shear strength is the best, reaching 17.87 MPa, which is higher than the results reported in the literature [8], indicating that it has a relatively high shear strength. High shear strength shows better mechanical properties.

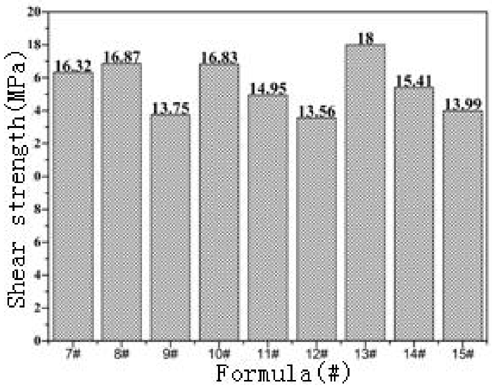

Figure 3. Shear strength of cured epoxy resin E-44 and E-51

Figure 3 shows the experimental results of the shear strength of the cured products after epoxy resin E-44 and E-51 are compounded. It can be seen from the experimental results in Figure 3 that when the curing agent remains unchanged, with the increase of the proportion of E-44 in the formula, the shear strength of the system after curing also increases accordingly. Increasing the amount of E-44 can effectively improve the adhesive. Shear strength after curing. When the epoxy resin remains unchanged, the cured product of the adhesive obtained by using curing agent 650# alone shows higher shear strength than that obtained by compounding curing agent, indicating that it has better mechanical properties. The 9#, 12#, and 15# compound curing agent systems have shear strengths of 13.75 MPa, 13.56 MPa, and 13.99 MPa, respectively, and the shear strengths are relatively low. reactivity, resulting in poor mechanical properties. The experimental results in Figure 3 show that the 13# formula system has the best performance, and its shear strength can reach up to 18.00 MPa, showing the best mechanical properties.

3.3 Scanning electron microscope (SEM) to analyze the microstructure of the fracture

surface In the course of the experiment, it is firstly speculated that there are two failure modes on the fracture surface of the sample: interface failure and cohesive failure. In order to further explore the influence of different ratios on the fracture mechanism, the microcomputer -controlled electronic universal testing machine was used to conduct the tensile fracture treatment of the sample, and the surface morphology of the fracture surface of the adhesive of the test piece was

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characterized in detail with the help of SEM test technology. Analysis, the experimental results are shown in Figure 4.

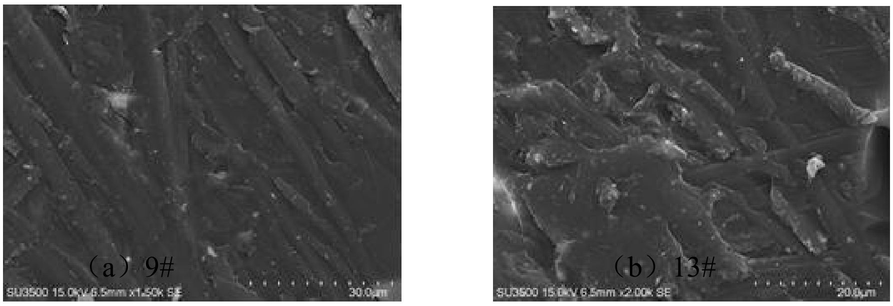

Figure 4. SEM of adhesive surface fracture morphology

Among them, Figure 4(a) shows the SEM image of the 9# formula test piece with low shear strength when epoxy resin E-44 and E-51 are compounded. The results show that the cross-sectional surface is smoother and the crack direction is relatively single. , it is judged that its fracture mode is brittle fracture; Figure 4(b) shows the SEM image of the 13# formula test piece with the highest shear strength. It can be judged that the fracture mode has changed during impact, from the original brittle fracture to ductile fracture. The research of Xie Jianjun et al. [9] showed that the better the bonding of the adhesive at the interface, the higher the corresponding shear strength. When the toughness strength of the adhesive interface is not ideal, the interface is prone to brittle fracture. The fracture becomes a ductile fracture.

3.4 Thermogravimetric (TG) test analysis

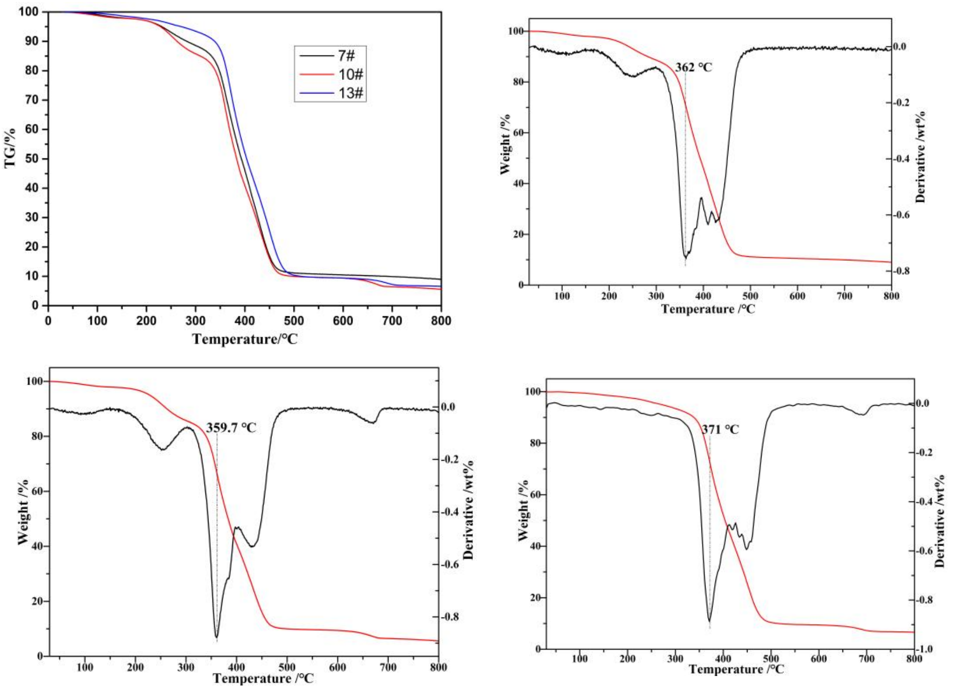

Figure 5. TG curve of experimental formula

Figure 5 shows the TG analysis curves of formulation systems 7#, 10# and 13# with higher shear strength. From the TG experiment results in Figure 5(a), it can be seen that when the temperature is lower than 130 ℃, the three formulations of adhesives did not thermally decompose; and in the temperature range of 130-350 ℃, the three formulations of the adhesives began to decompose slowly. And the weight loss rate of 13# system is obviously smaller than that of formula 7# and 10#; the temperature corresponding to the maximum weight loss rate of formula 7# in Fig. 5(b) is

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362 ℃, and the maximum weight loss rate of formula 10# in Fig. 5(c) corresponds to The temperature of the sample is 359.7 ℃, the temperature corresponding to the maximum weight loss rate of formula 13# in Figure 5(d) is 371 ℃, and the thermal decomposition weight loss rate is 56.27 %; when the temperature is higher than 380 ℃, the samples all decompose rapidly; When the temperature reaches 500 ℃, the decomposition curve tends to be flat. The above experimental results show that the adhesives of the three formulations can be used for a long time at around 130 ℃, but the thermal weight loss rate of the adhesive of the 13# formulation is significantly lower than that of the other two formulations at the same temperature, and the 13# system can withstand 380 in a short time. ℃ high temperature, indicating that the formulation of the adhesive showed good thermal stability.

4. Conclusion

By systematically comparing the mechanical properties, thermal decomposition characteristics and comprehensive evaluation results of SEM characterization of the new epoxy resin-based insulating adhesives with different formulations, it can be seen that the 13# formula new epoxy resin-based insulating adhesives obtained in this paper showed the best thermal performance. It is stable, and has good toughness and mechanical properties; SEM results show that the impact section is crack-like, with the most cracks and more irregular cracks, and the toughness is enhanced. The shear strength of the system reaches 18.00 MPa, which can be used at about 130 ℃ for a long time, and can withstand a high temperature of about 380 ℃ in the short term. And heat resistance and other performance requirements, so it is expected to be used in the bonding and potting of electrical and electrical equipment in practice, and has a good application prospect.

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