IOSR Journal of Applied Chemistry (IOSR-JAC)
e-ISSN: 2278-5736.Volume 16, Issue 10 Ser. I (October 2023), PP 47-50
www.iosrjournals.org

Effect of Diluent Concentration on Thermal and Mechanical Properties of Modified Epoxy Novalac Resin

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Abstract: *Four sets containing five samples, in each, of epoxidizednovalac resin with varying amounts of diluent concentration ranging between 5-25 percent having a fixed percentage of hardners with respect to total amount of epoxy and diluent. The cure time of resin and hardner system was found to be 16 hours at room temperature and 2 hours at 100°C. It was found that the bending and tensile strength increased while compression strength decreased containing the hardners, diethyl triamine (DETA) (11%) and triethyltetraamine (TETA) (13%), with the increase of diluent concentration from 5-25 percent. The pot-life improved appreciably whereas the glass-transition temperature (T) decreased significantly upto 25 percent addition of diluent in epoxy resin. With the hardner containing reactive polyamino amide, all three mechanical properties decreased with diluent concentration. The pot-life, in this case, increased significantly whereas the T'S value decreased appreciably with the increase of concentration of diluent. The hardner containing the mixture of modified aromatic amine adduct and its accelerated version gave pot-life of the resin much higher as compared with other hardner systems.*

Date of Submission: 17-10-2023 Date of Acceptance: 27-10-2023

I. Introduction

The epoxidizednovalc resin are characterized by the possession of more than one group per molecule. This group may lie within the body of the molecule but it is usually terminal Lee and Neville,. The three membered epoxy ring is highly strained and reactive to many substances particularly by with proton donors. Such reaction allows chain extension and/or cross- linking to occur without the elimination of small molecules such as water. In consequence, these materials exhibit a lower shrinkage than many other types of thermosetting plastics. The non-epoxy part of the molecule may be aliphatic, cyclo-aliphatic or highly aromatic or it may be non-hydrocarbon and possibly polar and hence, giving a very wide range of epoxy resins having very diverse applications in protectice coatings, adhesives, as body solders, dipping, potting and encapsulation compounds, low-pressure molding resins, and glass reinforced plastics, Potter; May and Tanaka,; Woo et al, Qin, Y. This wide spectrum applications area are due to their better mechanical, electrical, thermal and chemical properties than any other thermosetting resins. The varied performance behaviour of epoxy resins is mainly due to the functionality of the base resin and that of the hardner used for curing purpose Bauer Ika et al. Gupta and Raymond; Michael, Bryan, Rudawska, A.

For a number of purpose the unmodified epoxy resins may be considered to have a certain disadvantages which include high viscosity, high cost and too great rigidity for specific application areas Pearson and Manas, Srivastava and Mathur ; Brun and Prime, Sankaran and Manas . These are, therefore, often modified by the incorporation of diluents, flexiblizers, fillers, etc. Diluents are free flowing liquids incorporated to reduce the resin viscosity and thus, simplifying handling of the resin Lee and Neville, Potter, Baker M, May and Tanaka,.

In the present article, the effect of various diluents on various thermal and mechanical characteristics of various epoxy/hardner systems have been investigated.

II. Experimiental

The epoxidizednovalac resin (Viscosity: 11700 cps; Epoxy value:5.2), procured from M/s Atul Ltd., Valsad, Gujarat, was physically mixed with varying amounts of reactive diluents ranging from 5-25 percent having a fixed percentage of hardners viz. diethyl triamine (DETA) (11%), triethyltetraamine (TETA) (13%), a reactive polyamino amide (50%) and a mixture of modified aromatic amine adduct and its acceleratedersion (50%) with respect to total amount of epoxy and diluent. Pot-life of all the samples was determined at 25°C. The glass-transition temperature (T) was evaluated from the DSC thermograms (TA instruments, USA) taken at a heating rate of 10°C/ mm in the temperature range between 30-200°C. Brookfield viscometer was used to measure the viscosity of the resin. Different spindles were used to measure the viscosity of the resin. The dial reading when multiplied by spindle's factor gave the viscosity of the resin in centipoise.

The tensile, bending and compression strengths were measured on Universal Testing Machine (M/s STARTEST, Mumbai) at speeds of 10, 7.5 and 7.5 mm/min, respectively. All the results obtained during the study have been summarized in Tables 1-3.

III. Results And Discussion

Table 1 depicts the, effect of diluent concentration on the viscosity of the epoxy resin, for which, theviscosity has been found to be 11700 ep. The viscosity of the epoxy resin decreased about 67.4 percent only by adding 5 percent diluent in the epoxy resin and then it decreased at a slower rate upto 25 percent addition of diluent. The addition of diluent from 5-25 percent brought a very little change in the epoxy value of the base epoxy resin.

Table 1: Variation of viscosity and epoxy value with diluent concentration of epoxy resin.

Epoxy (E) (%)	Diluent (D) (%)	Viscosity at 25°C (cp)	Epoxy Value
100	0	11700	5.2
95	05	3814	5.1
90	10	1740	5.1
85	15	1108	5.0
80	20	591	4.9
75	25	416	4.8

Table 2: Variation of pot-life and glass-transition temperature (T) with diluent concentration of various epoxidizednovalac systems.

	Epoxy Conc. (%)	Diluet Conc. (%)	Hardner Conc. (%)	Sample designation	Pot-life at 25°C (min)	T_g (°C)
Set-A (Hardner DETA[a])	95	05	11	EDH_{111}	20	106.1
	90	10	11	EDH_{211}	22	86.5
	85	15	11	EDH_{311}	24	77.9
	80	20	11	EDH_{411}	25	60.4
	75	25	11	EDH_{511}	26	52.2
Set-B (Hardner TETA[b])	95	05	13	EDH_{113}	20	97.5
	90	10	13	EDH_{213}	22	80.4
	85	15	13	EDH_{313}	25	67.7
	80	20	13	EDH_{413}	26	58.7
	75	25	13	EDH_{513}	28	53.8
Set-C (Hardner RPAA[c])	95	05	50	EDH_{150}	81	84.1
	90	10	50	EDH_{250}	84	81.2
	85	15	50	EDH_{350}	112	76.8
	80	20	50	EDH_{450}	117	N.S.
	75	25	50	EDH_{550}	121	N.S
Set-D (Hardner MAAA[d])	95	05	50	EDH_{141}	161	80.0
	90	10	50	EDH_{241}	172	77.1
	85	15	50	EDH_{341}	193	65.2
	80	20	50	EDH_{441}	251	N.S.
	75	25	50	EDH_{541}	270	N.S

The variation on pot-life and glass-transition temperature (T) with diluent concentration on epoxy and different hardner systems has been summarized in Table 2. With hardner, diethyl triamine (DETA) (11%), as the concentration of diluent is increased from 5-25 percent, the pot-life increased appreciably whereas the T value decreased in the range between 18-50 percent with diluent concentration when compared with the T, value of epoxy having 5 percent diluent concentration (Sample EDH,,,). The T value for sample EDH was found to be 97.5°C with TETA hardner which wasabout 8 percent lower than the value for DETA hardner. Again, the pot-life showed a similar trend with TETA hardner to that for DETA hardner.

The pot-life of epoxy/hardner system increased significantly with modified aromatic amine hardner (MAAA) (50%) with diluent concentration. The increase in case of sample EDH,50 was found to be 38.3 percent with that of epoxy having 5 percent hardner (EDH150). The pot-life for sample EDH was found to be increased by 49.4% compared with EDH, 150*

Table 3: Variation of mechanical properties diluent concentration of various epoxy/hardner systems.

Sample	C.S.[a](Kgf/mm^2)	T.S.[b](Kgf/mm^2)	B.S.[c](Kgf/mm^2)
EDH_{111}	11.53	4.67	0.53
EDH_{211}	10.26	4.89	0.66
EDH_{311}	9.95	6.36	0.83
EDH_{411}	7.72	6.66	0.90
EDH_{511}	5.81	7.52	0.96
EDH_{113}	11.56	3.41	0.70
EDH_{213}	10.58	4.76	0.79
EDH_{313}	9.97	5.52	0.85
EDH_{413}	8.01	5.89	0.90
EDH_{513}	6.33	6.40	0.96
EDH_{150}	8.54	4.56	0.47
EDH_{250}	8.16	4.21	0.35
EDH_{350}	7.52	3.83	0.41
EDH_{450}	6.22	3.32	0.30
EDH_{550}	4.57	2.93	0.21
EDH_{141}	10.21	5.61	1.12
EDH_{241}	8.87	5.28	1.01
EDH_{341}	8.32	4.87	0.94
EDH_{441}	6.06	3.20	0.65
EDH_{551}	4.41	1.96	0.46

The T's values for sample EDH450 and EDHsso were very difficult to detect as the peaks in DSC scans were not very sharp. At lower diluent concentration i.e. upto 15 percent, the T's values decreased by approximately 3 percent and 9 percent when compared with the T value for sample EDH150. When the mixture of accelerated version of modified aromatic amine and modified aromatic amine (ratio 45:15) was used as hardner, there occurred a very high increase in pot-life when compared with DETA and TETA hardners. The pot-life of this epoxy/hardner system was found to be 161 mm at 25°C. Upto 15 percent diluent addition, the increase was found to be slowbut, after this the value increased about 56 percent with 20 percent diluent addition (EDH) as compared to sample EDH,, whereas for 20 and 25 percent diluent addition, T values could not be detected. The T's values upto 15 percent addition have been found to be comparable with other system of hardners.

The data related to mechanical properties viz. compression, tensile and bending strengths of epoxy/ hardner systems with diluent concentrations are presented in Table 3. It is clear from the data that the compression strength decreased whereas the tensile and bending strengths increased with diluent concentrations with DETA and TETA hardners. Thehardners used in Set-C and Set-D samples decreased the tensile, compression and bending strengths. The increase or decrease was found to be appreciably in all sets of samples. The increase in percentage of reactive diluent impacts flexibility of the epoxy system which might lower crosslink density to final cured product and hence, decreased or increased the mechanical properties.

IV. Conclusions

From the above results and discussion, the following conclusions may be drawn -

1. The pot-life of epoxidizednovalac containing mixture of MAAA and its accelerated version of hardner was much higher that with other systems of hardners such as DETA, TETA, etc.
2. The T's of all sets of samples of epoxidizednovalac resin decreased appreciably with the addition of diluent.
3. The tensile and bending strengths increased with DETA and TETA hardners whereas for other hardners (used in Set-C & Set-D), their value decreased.
4. The compression strength decreased for all sets of samples of epoxy/hardners with diluent **concentrations.**

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