

# Synthesis of Hexafunctional Epoxide Resin and Application on Jute and Glass Reinforced Composites

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**ABSTRACT:** *Bisphenol-A, formaldehyde, and epichlorohydrin form hexafunctional epoxy resin. The curing behavior of resin has been evaluated by using five different hardeners viz. diethyl triamine, triethyl tetraamine, phenalkamine, polyamido amines, and polyamides. The resin was further characterized by epoxy equivalent weight, hydrolyzable chlorine content, volatile content, viscosity and rise in viscosity, weight average molecular weight, and Fourier Transform InfraRed (FT-IR) spectroscopy. The hexafunctional epoxy resin was used for the preparation of jute and glass-reinforced composites. All composites were characterized by their mechanical properties, thermal properties, and chemical resistance.*

**KEYWORDS:** *Epoxy resin; Fiber; Composites; FT-IR; Hexafunctional.*

## INTRODUCTION

Epoxy resins are widely used in composites, electronics & electrical, adhesives & coatings, aerospace, building, automobile, and many other areas due to their excellent mechanical properties such as tunable mechanical properties, good electrical properties, high bonding strength, excellent dimensional stability, and good processing abilities [1-3]. Nowadays, epoxy resins are increasingly popular in the fiber composite field, and the excellent mechanical properties of epoxy resins (e.g. high strength and modulus) draw tremendous attention from scientists and researchers [4-6]. The properties of cured epoxy resins are highly related to their network structure, which is critically influenced by the functionality of the epoxy resin. One effective method to achieve excellent epoxy resin

systems with high strength and high modulus are to increase the functionality of the epoxy resin [7-11]. Multifunctional epoxy resin is a way to enhance heat-resistant properties because of its higher curing density [12, 13]. Multifunctional epoxy resins are well known for their improved mechanical, chemical, thermodynamic, and electrical properties [14-17]. Other advantages of multifunctional epoxy resins are their high glass transition temperatures, high decomposition temperatures, long-term high-temperature performance, and good wet strength performance. Multifunctional epoxy resins have two important limitations because of their intrinsic brittle nature and considerable moisture absorption tendency from the environment. It adversely affects on most of the physicochemical properties

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of the fabricated articles. These drawbacks increase by enhancing the cross-link density of the network. *Hourston et al.* studied the dynamic, mechanical, and fracture properties of three types of epoxy resins with different functionalities and concluded that glass transition temperature ( $T_g$ ) varied with functionality, whereas the strain energy release rate and the stress intensity factor varied insignificantly with functionality [10]. Epoxy-based fiber composites have become more commonly used in automobile, electronic devices, construction, and aerospace industries. This is attributed to the attractive mechanical properties, dimensional stability, and corrosion resistance of the composites [18-21]. Recently epoxy-based hybrid composites have been extensively used in many engineering and industrial applications. Load bearing engineering applications, superior adhesive properties, and mechanical strength are the key features of the composite material. In comparison with synthetic fiber composites; natural fibers are characterized by their attractive price, low density, and lower abrasion. The energy consumption needed for the production of synthetic fibers is much more than that needed for a similar quantity of natural fibers [22]. The synthetic fibers and natural fibers have a wide variation in diameter and length, which in turn affects the composite expected mechanical behavior. The variation in dimensions depends on fiber type, fiber maturity, harvesting time as well as processing methods adopted for the extraction of fibers, which all affect the diameter and stability of the fiber. Source, age, separating techniques, moisture content, and the history of fiber also play an important role in the filament and individual fiber properties. The implementation of natural fibers in composites is attractive for different industrial sectors like automobiles and construction [23, 24].

The main aim of the present work is to synthesize bisphenol-A-based Hexa-functional epoxy resin and determine different properties of resin such as epoxy equivalent weight, hydrolyzable chlorine, viscosity, volatile content, FT-IR, and weight average molecular weight. The curing kinetics of resin was analyzed by various hardeners. Mechanical, chemical, and thermal properties of jute and glass fiber reinforced composites were also studied.

## EXPERIMENTAL SECTION

### Raw Materials

Solvents and chemicals used were of laboratory grade and purified prior to their use. Bisphenol-A, formaldehyde,

epichlorohydrine, diethyl triamine, triethyl tetraamine, methyl ethyl ketone, perchloric acid, acetic acid, tetraethylammoniumbromide, and sodium hydroxide were supplied by Sigma-Aldrich. Aromatic hardeners phenalkamine, polyamido amines, and polyamides were obtained from Admark Polycoats Pvt. Ltd., Vadodara. Glass fiber and jute fiber were purchased from Composites Tomorrow, Vadodara.

### Synthesis of hexafunctional epoxy resin based on bisphenol-A

A 2 L three-neck flask equipped with a mechanical stirrer and condenser was placed into a thermostat bath. To this flask add 1.0 mole of (228 g) bisphenol-A and 5.0 mole of (324 mL of 37-41 %) formaldehyde were added under alkaline conditions (pH 8.5) and stirred at 55°C for 1 h. Then 6 mole of (470 mL) epichlorohydrine was added to the reaction mixture and finally, 85mL 35% aqueous NaOH solution (based on the weight of bisphenol-A) was added in a dropwise manner. This is an exothermic reaction so the temperature was maintained at 50°C during the addition process. After that, the temperature was raised to 80°C and the reaction was stirred for 1 hour. Methyl Ethyl Ketone is used as a solvent. The resin first underwent vacuum distillation for the removal of salt and then distilled at 118°C temperature for the removal of unreacted water, methyl ethyl ketone, and epichlorohydrine. Finally, the resin obtained was cooled at room temperature. The obtained resin has a dark yellow color. The probable reaction mechanism is given in Fig. 1.

### Fabrication of hexafunctional resin - jute fiber- diethyl triamine composite (BJD)

Resin and the required amount of hardener was taken for composites preparation. The ratio of jute fiber to resin is 30:70 and the diethyl triamine is taken according to their amine hydroxide equivalent weight. Resin and hardener were mixed in a beaker and applied on a sheet of jute fiber by hand layup. The sheet size is 15 x 12 cm. All fiber were stacked one over the other between two Teflon sheets which were kept between the compression molding machine at 80°C for 1 h and 30 min and then the pressure of 50 psi was applied for 2 minutes. Finally, composites were cooled at room temperature.

Similarly, other composites systems were prepared with the use of different hardeners. Hexafunctional resin- jute fiber-

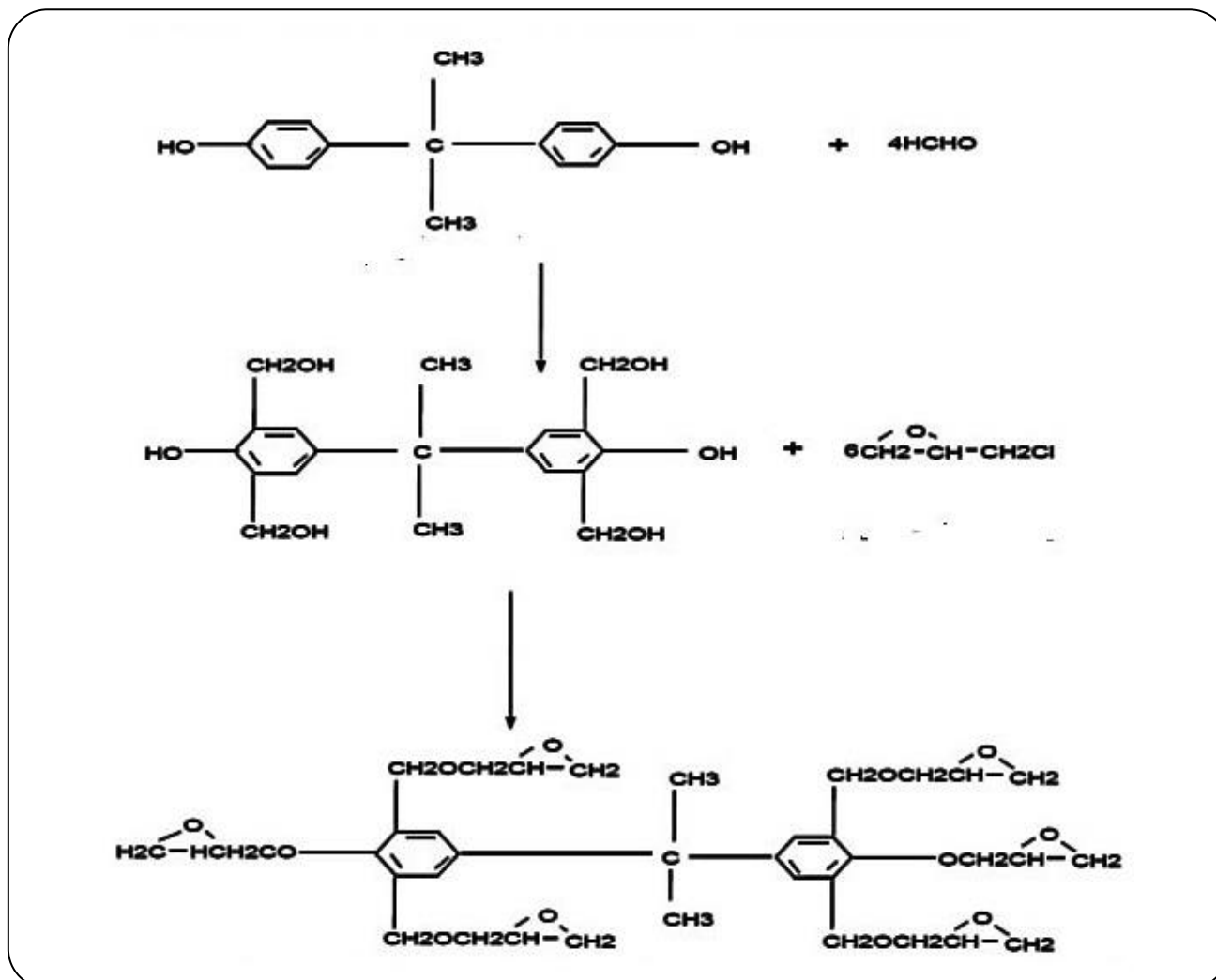


Fig. 1: Probable reaction of hexafunctional epoxy resin based on Bisphenol-A.

triethyl tetramine composite (BJT), hexafunctional resin- jute fiber- phenalkamine composite (BJP), hexafunctional resin- jute fiber- polyamido amines composite (BJPA), and hexafunctional resin- jute fiber- polyamides composite (BJPD) prepared.

#### **Fabrication of hexafunctional resin- glass fiber- diethyl triamine composite (BGD)**

To produce glass fiber based composites the ratio of fiber to resin is 40:60. hexafunctional resin- glass fiber- diethyl triamine composite (BGD) are produced the same way as the BJD composite and similarly, hexafunctional resin- glass fiber- triethyl tetramine composite (BGT), hexafunctional resin- glass fiber- phenalkamine composite (BGP), hexafunctional resin- glass fiber- polyamido amines composite (BGPA) and hexafunctional resin- glass fiber- polyamides composite (BGPD) were prepared.

## **CHARACTERIZATION**

### **Epoxy Equivalent Weight (EEW) (ASTM D1652)**

The accurate weight of the resin was dissolved in tetraethylammoniumbromide solution, which is titrated against 0.5N perchloric acid solution. Metroham Auto Titrator was used to carry out the titration.

### **Viscosity measurement (ASTM D789)**

Brookfield viscometer model no. RV digital viscometer was used to determine the viscosity of the resin at 25°C temperature.

### **Volatile content (ASTM D1259)**

The accurate weight of the resin was poured into a petri dish, which was kept in an oven at 110°C temperature for 30 minutes. Volatile content was determined by the percentage change in the initial and final weight.

**Hydrolysable chlorine content (Hycl) (ASTM D1726)**

2 gm. of resin was dissolved in a mixture of 15ml toluene and 25 ml alcoholic KOH solution. The reaction mixture was refluxed for 20 min at 300°C temperature. Blank reading is taken without a sample. Blank and sample were titrated against 0.5N HCl solution. Metroham Auto Titrator was used to carry out the titration.

**Gel Permeation Chromatography (GPC)**

The weight average molecular weight of the resins was measured on Turbo Matrix-40 of Perkin-Elmer, USA, using tetrahydrofuran (THF) as a solvent.

**FT-IR**

The FT-IR spectra were measured on a Spectrum GX of Perkin-Elmer, USA. The range of FT-IR spectrophotometer was between 10,000–370  $\text{cm}^{-1}$ . Absorbance spectra were acquired at 0.15  $\text{cm}^{-1}$  resolution and signal-averaged over 20 scans.

**MECHANICAL PROPERTIES OF COMPOSITES****Tensile strength (ASTM D 638)**

Tensile strength was performed on Universal Testing Machine (UTM) Shimadzu AG 100. The test was performed as per ASTM D 638 at 100% strain rate and a crosshead speed of 50 mm/min. This test gives an idea about the material's extent of stretching towards the applied force, which tends to pull it apart.

**Izod impact strength (ASTM D 256)**

Izod impact strength was measured on a CEAST Izod tester as per ASTM D 256. All the specimens were notched with a standard notch cutter. The Izod impact test determines the kinetic energy consumed by the pendulum in order to break the specimen.

**Flexural strength (ASTM D 790)**

Flexural strength is the ability of the material to withstand bending forces applied at the outside surface of the test bar. The test was performed on an UTM Shimadzu AG 100 ASTM D 790, at a crosshead speed of 1.2 mm/min.

**Rockwell hardness (ASTM D 785)**

The information from a hardness test can be used to provide critical material performance information and insight into the durability, strength, flexibility, and

capabilities of a variety of component types from raw materials to the prepared specimens and finished goods. Digital Rockwell hardness tester with HRL indenter is used to measure the hardness value of the prepared test specimen. An average value of three repeated analyses is denoted and all the mechanical properties were measured at room temperature.

**Thermogravimetric analysis (TGA)**

A Pyris-1 series thermal analyzer (Pyris-1 TGA, PerkinElmer, USA) was used to investigate the thermal stability of composites. For the analysis, composites were ground into a fine powder and about 5–10 mg of samples was taken and heated in nitrogen from 50°C to 1000°C temperature at the heating rate of 10°C  $\text{min}^{-1}$  in all cases.

**Chemical resistance test (ASTM D 543 - 87)**

The chemical resistance of the composites is studied as per ASTM D 543-87 method. This method covers the chemical resistance testing of all the composites for reporting changes in weight, dimensions, appearance and strength properties by the action of chemical reagents. In the present work, all test specimens' dimensions were 1.5 × 1.5 cm and immersed in suitable uniform containers with six different chemicals. Concentrated sulfuric acid (10% wt/wt), aqueous sodium hydroxide (10% wt/wt), sodium chloride (10% wt/wt), tetrahydrofuran, and methanol were used as chemical media. The water absorption by the composites was also studied. For each specimen, 250 ml of solution was introduced into the container. They were removed after 7-day exposure to each chemical and immediately washed with distilled water. Specimens were dried by pressing them on both sides with filter paper at room temperature. Before and after each test cycle, all specimens were weighed in a precision electronic balance, and the percentage of weight loss/gain was determined. Percentage change in thickness is also determined with the aid of a digital vernier caliper. In each case, two samples were tested and their average values are reported.

**RESULTS AND DISCUSSION**

The synthesized resin is characterized by epoxy equivalent weight, weight average molecular weight determination, volatile content, viscosity determination, hydrolysable chlorine content, FT-IR, and its curing mechanism with different hardeners. Weight average

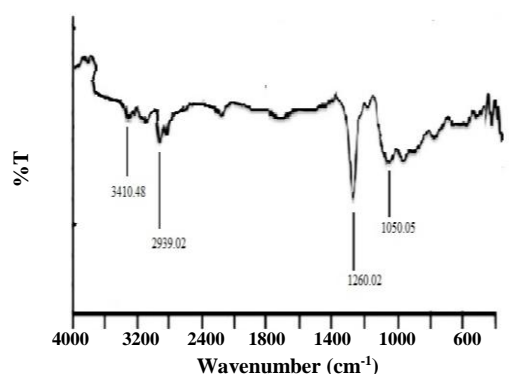
the molecular weight of the resin is 2620 while the epoxy equivalent weight of the resin is 537 gm, which accounts for the functionality of the synthesized resin of 5.83. The resin has a small number of impurities of solvent which conformed by volatile content which is 0.98%. The viscosity of resin determined by Brookfield RV viscometer was 20625cps. To ensure that the synthesized resin is useful for higher temperature applications, a rise in viscosity was determined by putting the resin in an oven for 110°C temperature for 24 hours. The change in viscosity to original viscosity is only 1.3% so the resin is useful to apply in the high-temperature application. Hydrolysable chlorine content of the resin is 1.5%, which indicates a lower amount of hydrolysable chlorine present in resin so it cannot affect the reactivity of resin. Gel time of resin using different hardeners is given in Table 1. Diethyl triamine required 54 minutes to convert resin into gel form meanwhile polyamido amines work six times faster than diethyl triamine and convert the resin to gel form in around 9 minutes. The FT-IR spectrum of the synthesized resin is shown in Fig. 2. In FT-IR spectra the peak at 3410.48  $\text{cm}^{-1}$  is due to the stretching of -OH groups. The peak at 2939.02  $\text{cm}^{-1}$  indicated C-H stretching vibration. Two strong peaks appeared at 1050.05  $\text{cm}^{-1}$  and at 1260.02  $\text{cm}^{-1}$  indicating ether linkage which confirmed the synthesis of resin.

Mechanical properties (Tensile strength, Flexural strength, Impact strength, and Rockwell hardness) data are given in Table 2. The tensile strength of jute fiber composites ranges from 34.4-38.8 MPa. Increasing order of tensile strength of jute fiber composites were BJPA > BJP > BJPD > BJD > BJT. The tensile strength of glass fiber composites ranges from 60.1-64.9 Mpa. Polymeric hardeners have higher molecular weight so they provide additional strength to composites and because of this, BJPA and BGPA composites have higher tensile strength than the rest of jute and glass fiber composites, respectively.

The flexural strength of composites is almost 2 to 2.5 times higher than their tensile strength for both jute and glass fiber-based composites. BJPA composite has higher flexural strength at 81.6 Mpa while BJT composite has the lowest flexural strength at 73.2 MPa. For glass fiber-based composites higher flexural strength is shown in BGPA at 139.2 Mpa which is 5.25% higher than BGT composite. Polymeric hardeners provide strength to composites because of their higher molecular weight as well as

**Table 1: Gel time of resin with different hardeners.**

Hardener Name	Gel time of Resin (Minutes)
Diethylene triamine	54.09
Triethylene tetraamine	49.38
Phenalkamine	11.50
Polyamido amines	9.06
Polyamides	46.32



**Fig. 2: FT-IR of hexafunctional epoxy resin based on Bisphenol-A.**

because their adhesion property is superior to the aliphatic hardeners. Therefore, BJPA and BGPA have higher flexural strength than BJT and BGT respectively.

Izod impact strength of all the particulate composites was measured to study the resistance of the specimen against the sudden impact force applied to it from a specific direction. Aliphatic hardeners-based composites have lower Izod impact strength than aromatic hardeners-based composites. BJT composite has 29.4% lower Izod impact strength than BJPA composite while for glass fiber the BGT composite has 22.4% lower zod impact strength than BGPA composite.

Rockwell hardness was analyzed to check the rigidity of composites. Increasing order of Rockwell hardness of jute fiber-based composites were BJPA > BJP > BJPD > BJD > BJT. BJPA composite has higher Rockwell hardness at 49.2 which is almost 13.9% higher than BJT composite. Rockwell hardness of glass fiber-based composites is almost 1.5 times higher than that of jute fiber-based composites. Rockwell hardness of glass fiber composites is 32.8, 31.7, 35.2, 37.1, and 33.7 for BGD, BGT, BGP, BGPA, and BGPD, respectively.

All mechanical properties results were taken three times and the mean of it is given in Table 2. All the results

**Table 2: Mechanical properties of Jute and Glass fiber-based composites.**

Sr.no.	Sample Code	Tensile Strength (MPa)	Flexural Strength (MPa)	Impact Strength (J/cm)	Rockwell Hardness
1	BJD	35.3 (1.2)	74.5 (1.8)	2.75 (0.3)	25.3 (0.9)
2	BJT	34.4 (1.3)	73.2 (1.8)	2.42 (0.3)	24.2 (1.0)
3	BJP	37.6 (1.2)	78.9 (1.8)	2.98 (0.3)	27.3 (0.9)
4	BJPA	38.8 (1.2)	81.6 (1.7)	3.43 (0.4)	28.1 (1.0)
5	BJPD	35.8 (1.3)	75.2 (1.7)	2.87 (0.4)	26.1 (0.9)
6	BGD	60.7 (1.6)	132.8 (2.1)	4.03 (0.4)	32.8 (1.3)
7	BGT	60.1 (1.3)	131.9 (2.1)	3.89 (0.4)	31.7 (1.2)
8	BGP	63.2 (1.4)	135.9 (1.7)	5.01 (0.5)	35.2 (1.4)
9	BGPA	64.9 (1.5)	139.2 (1.7)	5.13 (0.5)	37.1 (1.4)
10	BGPD	61.4 (1.6)	134.1 (1.9)	4.25 (0.4)	33.7 (1.3)

**Table 3: Chemical resistance of Jute and Glass fiber based composites.**

Sr. no.	Sample code	Water		10%NaOH		10%H <sub>2</sub> SO <sub>4</sub>		10%NaCl		Methanol		THF	
		%A	%B	%A	%B	%A	%B	%A	%B	%A	%B	%A	%B
1	BJD	0.9	0.4	1.6	0.8	1.9	0.8	1.3	0.4	1.5	0.6	2.9	1.3
2	BJT	1.1	0.5	1.8	0.8	2.2	0.9	1.6	0.4	1.6	0.7	3.6	1.6
3	BJP	0.6	0.2	1.1	0.7	1.3	0.6	0.8	0.3	1.1	0.4	2.5	1.1
4	BJPA	0.5	0.2	0.8	0.6	1.2	0.6	0.7	0.3	0.9	0.4	2.1	0.9
5	BJPD	0.8	0.4	1.4	0.8	1.7	0.7	1.1	0.4	1.4	0.5	2.8	1.3
6	BGD	NC	NC	NC	NC	NC	NC	0.6	0.2	NC	NC	1.9	1.0
7	BGT	NC	NC	NC	NC	NC	NC	0.8	0.2	NC	NC	2.4	1.1
8	BGP	NC	NC	NC	NC	NC	NC	0.3	0.1	NC	NC	1.4	0.7
9	BGPA	NC	NC	NC	NC	NC	NC	0.3	0.1	NC	NC	1.1	0.7
10	BGPD	NC	NC	NC	NC	NC	NC	0.4	0.2	NC	NC	1.6	0.9

%A = Change in weight, %B = Change in thickness, NC = No change.

of mechanical properties were statistically analyzed with data variance and it is given in brackets of Table 2.

The chemical resistance of all composites was measured against six different chemicals. It was observed that the composites have gained weight and therefore no erosion has taken place due to the exposure to chemicals. The results of chemical resistance are given in Table 3. All glass fiber-based composites showed excellent chemical resistance and there were no changes in composites against water, 10% NaOH, 10% H<sub>2</sub>SO<sub>4</sub>, and methanol. In 10% NaCl solution 0.8% and 0.3% weight losses were observed for BGT and BGPA composites and in THF solution 2.4% and 1.1% weight losses were observed. Jute fiber is a natural polymer and due to the polar

nature of natural polymers, weight loss was observed against all six chemicals. In all chemical reagents the highest chemical resistance was observed for the BJPA composite, while poor performance was given by BJT composites. The highest weight loss data were shown in THF solution, which are 2.9%, 3.6%, 2.5%, 2.1%, and 2.8% for BJD, BJT, BJP, BJPA, and BJPD composites, respectively.

TGA i.e. weight loss as a function of temperature was done in order to understand the thermal stability of the composites. Mass loss curves for all jute and glass fiber-based are shown in Fig. 3 and Fig. 4, respectively. The decomposition rate of the composites associated with various temperature intervals is calculated and shown

Table 4: Thermal kinetics parameters of jute and glass fiber based composites.

Sample code	T <sub>0</sub> (°C)	PDT (°C)	E <sub>a</sub>
BJD	50	374	5.32
BJT	50	371	5.28
BJP	50	403	5.42
BJPA	50	405	5.46
BJPD	50	384	5.36
BGD	50	457	5.78
BGT	50	445	5.73
BGP	50	497	5.97
BGPA	50	502	6.02
BGPD	50	465	5.82

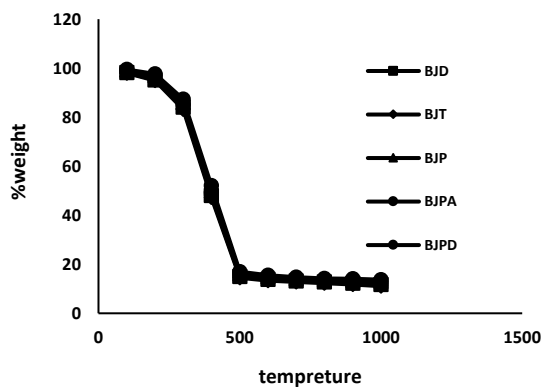


Fig. 3: TGA curve (mass loss curve) corresponds to temperature for jute fiber based composites.

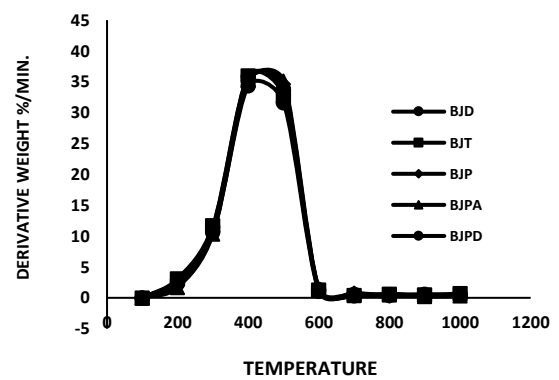


Fig. 5: DTGA graph of BJD, BJT, BJP, BJPA and BJPD.

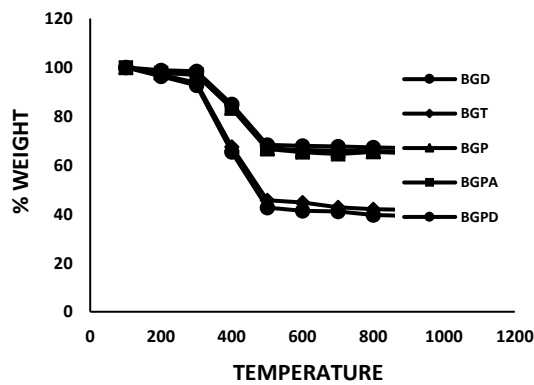


Fig. 4: TGA curve (mass loss curve) corresponds to temperature for glass fiber-based composites.

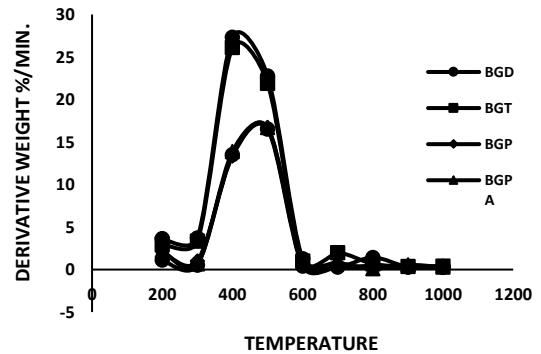


Fig. 6: DTGA graph of BGD, BGT, BGP, BGPA, and BGPD.

Fig. 5 and Fig. 6 are for jute and glass fiber-based composites, respectively. Thermal kinetic parameters like initial system temperature (T<sub>0</sub>), Procedural Decomposition Temperature (PDT), and activation energy (E<sub>a</sub>) were derived from TGA and DTGA curves

and are tabulated in Table 4. The activation energy of the particulate composites was measured as per Broido's method. The highest thermal stability was achieved for BJPA and BGPA composites for jute and glass fiber, respectively, according to their E<sub>a</sub>.

## CONCLUSIONS

In the present study, Hexa functional epoxide resin based on Bisphenol-A was synthesized using formaldehyde and epichlorohydrine. The resin was characterized by epoxy equivalent weight, molecular weight measurement, hydrolysable chlorine content, viscosity, and volatile content. Furthermore, the curing behavior of the resin was measured by using five different types of hardeners. Resin applied with different curing agents to produce jute and glass fiber-based composites. Mechanical properties, chemical resistance, and thermal properties of all composites were measured. After analysis of all data, it was found that polymeric hardeners-based composites are highly superior in gel time, mechanical properties, chemical resistance, and thermal properties than aliphatic hardeners-based composites for both jute and glass fiber-based composites. Glass fiber-based composites have overall better properties than jute fiber-based composites.

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