



Novel Alkyl Substituted Epoxy Resins Containing Naphthalene Moiety

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Abstract: 2, 6-dimethylol 4-methyl phenol (DMM) and 2, 6-dimethylol 4-tertiary butyl phenol (DMT) were prepared and condensed with 1-Naphthol (N) to synthesis phenolic resin. The resultant resins (DMM-N / DMT-N) were characterized by elemental analysis and IR spectral studies. Further, the phenolic resins were epoxidized and the resultant epoxy resins namely EDMM-N and EDMT-N were characterized duly. The curing of these epoxy resins was monitored on a differential scanning calorimeter (DSC) by using diaminodiphenyl methane (DDM) as a curing agent. Based on DSC data, glass fiber-reinforced composites of epoxy resins have been fabricated and their mechanical, chemical and electrical properties have been evaluated. Unreinforced cured samples of epoxy resins were analyzed by thermogravimetric analysis.

Keywords: Epoxy resin (EDMM-N - EDMT-N) ; Differential Scanning Calorimeter (DSC) ; Infrared Spectra (IR) ; Thermogravimetric analysis (TGA); Composites.

I. Introduction

Epoxy resin is a thermosetting polymer also known as polyepoxide is formed from reaction of an epoxide resin with polyamine which is used as curing agent. Due to the versatile properties of epoxy resin it has found number of applications [1-2] .

Epoxy resins are used in industrial applications due to their attractive properties such as excellent chemical and solvent resistance and good thermal properties [3]. At present, epoxy resins are used in various technical applications too, such as composites, adhesives, coatings, films and many more [4-7]. Due to its large-scale application its properties can be enhanced and improved by modifying its structures [8]. Owing to their superior properties some new epoxy resins have been synthesized in our laboratory [9-10].

The present article comprises synthesis, characterisation and glass reinforcement of alkyl substituted epoxy resins (EDMM-N and EDMT-N). Based on DSC thermograms glass reinforced composites have been laminated and characterized by chemical, mechanical and electrical properties.

II. Experimental

A. Materials

All the chemicals used were of laboratory grade. E-type of glass -woven fabric 0.25 mm thick (Obtained from Unnati Chemicals, India) was used for composite fabrication.

B. Synthesis of Phenolic resins (DMM-N / DMT-N)

p-cresol / p-tert-butyl phenol and formaldehyde in molar ratio of 1:2 were reacted using basic medium. The products formed as 2, 6-dimethylol-4-methyl phenol (DMM) / 2, 6-dimethylol 4-tert -butyl phenol (DMT) and 1-naphthol were charged in a two necked flask equipped with a mechanical stirrer to give phenolic resins

(DMM-N / DMT-N). The reaction mixture was slowly heated to 80°C for 18 hrs with continuous stirring. The resulting phenolic resins were then washed and purified (Scheme-1).

C. Synthesis of Epoxy Resins (EDMM-N / EDTM-N)

Phenolic resins were epoxidised with epichlorohydrin in a three- necked flask equipped with a reflux condenser and mechanical stirrer using basic medium. The process was carried out for 5 hrs. The mixture was then vacuum-distilled in order to remove excess epichlorohydrin. The viscous resin was stored for further analysis (Scheme-1).

III. Characterization and Thermal studies

A. Elemental and Spectral Analysis

The C,H contents of both the epoxy resins was estimated by means of Carlo Erba elemental analyzer. TheFT- IR spectra were recorded in KBR pellets on a Perkin Elmer Spectrophotometer.

B. Thermal Studies

Unreinforced cured samples were subjected to thermogravimetric analysis (TGA) on Perkin Elmer Pyris-1 thermogravimetric analyzer at a heating rate of 10°C/min. The sample weight for investigation used was in the range of 4-5 mg. Curing of Epoxy Resins was carried out on Universal VG03 differential scanning calorimeter (DSC). The instrument was calibrated using standard indium metal with known heat of fusion. Curing was carried out with a heating rate of 10°C/min. 4-5 mg weight sample was taken for investigation.

IV. Composite Fabrication

The composites were prepared by using E-type glass fiber. The ratio of glass fiber to resin is 60-40. A suspension of epoxy resins (EDMM-N / EDTM-N) was prepared in tetrahydrofuran(THF) and 25% diamino diphenyl methane (DDM) was added to it. For 5 minutes the mixture was stirred well. Now with a brush the suspension mixture was applied to 250mm x 250mm glass fiber and the solvent was allowed to evaporate. The dried preprags were stacked on top of one over the another. They were pressed between steel plates coated with a Teflon film release sheet and compressed in flat press under 60-70 psi pressure. The prepreg stack was placed in a air circulating oven for 6 hrs. The composites so obtained were cooled to 50°C before the pressure was released. Finally the composites were then machined to final dimensions.

V. Composite Characterization

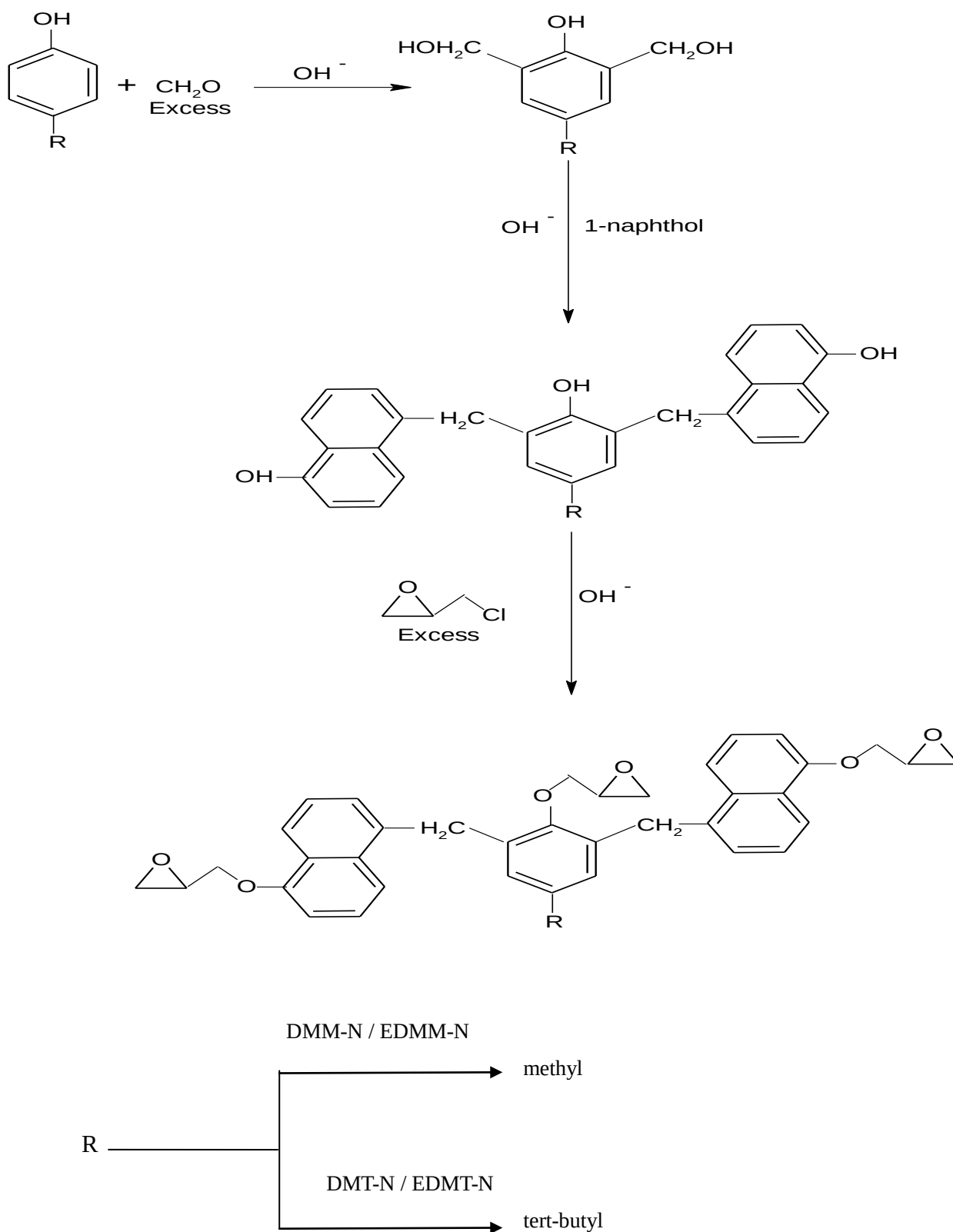
All the mechanical and chemical tests on composites were conducted according to ASTM methods.

Mechanical Testing

1. The compressive strength was measured according to ASTM D 695
2. Impact strength was measured using Izod type impact tester according to ASTM D 256
3. The Rockwell hardness was measured according to the ASTM D 785.
4. Dielectric strength was measured using a high voltage tester machine oil test set.

Chemical Resistance Test

The resistance of the composite samples against chemicals was measured according to ASTM D543



Scheme-1: Synthesis of Phenolic resins and Epoxy resins

VI. Result and Discussion

Novel epoxy resins were prepared by reaction of phenolic resins and epichlorohydrin in basic medium. Epoxy resins so prepared contain naphthalene groups which have not been prepared earlier.

The C,H content of epoxy resins (EDMM-N/EDMT-N) were estimated by Carlo Erba elemental analyzer. Their results are furnished in **(Table 1)**. From the results the values of C and H of both epoxy resins were found consistent with their predicted structures.

The FT-IR spectra of both phenolic resins **(Figure-1)** and the epoxy resins **(Figure-2)** show the prominent positions of important segments in the polymer chain. In the FT-IR spectra of Phenolic resins bands at 3494.27 cm⁻¹ and 3383.86 cm⁻¹ respectively show the presence of OH group. Further the Bands at 910 cm⁻¹ and 914 in EDMM-N and EDTM-N were due to epoxy group. Other bands at different positions confirm the structure of synthesized phenolic resins and Epoxy resins.

The epoxy resins were cured by using DDM as a curing agent. The data show that cured resin systems give a single exothermic peak. The data of DSC thermograms **(Figure-3)** of both the epoxy resins are shown in **(Table 2)**.

The unreinforced cured epoxy resins were analyzed by thermogravimetric analysis (TGA) **(Table 3)**.It was found that the cured samples start their degradation at about 150° C and their initial weight loss is about 0.91 and 0.69%. At 300°C 29.21 and 28.55% weight loss is found. The rate of decomposition increases rapidly between 300°C to 450°C and the products are lost completely beyond 500°C **(Figure-4)**.

Based on DSC data, glass fiber reinforced composites of both the epoxy resins were cured. It is found that mechanical strength of the laminates is good. The laminates also have good chemical resistivity. The results are furnished in **(Tables 4 and 5)**.

VII. Conclusion

The elemental analysis data are consistent with the suggested chemical formulae of the resins. The structure of all synthesized resins (DMM-N,DMT-N,EDMM-N, EDTM-N) is confirmed by their FT-IR spectrum . The resins show good thermal stability property.

The glass fiber reinforced composites of Epoxy resins were characterized for their chemical, Mechanical and Electrical properties and the results shows that composites have good chemical resistant property, good mechanical and electrical strength.

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Table 1: Preliminary Characterization of Epoxy Resins

Epoxy Resin				
EDMM-N	Molecular Formula	$C_{38}H_{36}O_6$		
	Elemental Analysis		%C	%H
		Calc.	77.55	6.12
		Found	78.12	5.92
	Solubility	Soluble in DMF , THF , CH_2Cl_2 , Ether etc . It is Insoluble in Water.		
	Epoxy Equivalent weight	198.01		
EDMT-N	Molecular Formula	$C_{41}H_{42}O_6$		
	Elemental Analysis		%C	%H
		Calc.	78.09	6.66
		Found	76.92	5.98
	Solubility	Soluble in Acetone , THF , Ether , 1,4-Dioxane. It is Insoluble in Water.		
	Epoxy Equivalent weight	206.18		

Table 2 : DSC Curing of Epoxy Resins

Epoxy Resin	Curing Temperature ($^{\circ}C$)		
	Initial Temp. (T_i)	Peak Temp. (T_p)	Final Temp. (T_f)
EDMM-N	112	150	168
EDMT-N	134	156	173

Table 3: TGA of Unreinforced Cured samples of Epoxy Resins

Epoxy Resin	% Weight loss at various temperature $^{\circ}C$ from TGA							
	150	200	250	300	350	400	450	500
EDMM-N	0.91	5.21	15.39	29.21	44.35	61.94	68.54	71.32
EDMT-N	0.69	4.88	16.71	28.55	40.16	57.42	68.09	73.06

Table 4: Mechanical and Electrical Properties of Composites

Composites	Compressive Strength (MPa)	Impact Strength (MPa)	Rockwell Hardness (R)	Electrical Strength in Air (kV/mm)
FM	294	283	110	15.2
FT	256	265	117	21.1

Table 5: Chemical Resistance Properties of Epoxy Resins

Reagents	EDMM-N % Change		EDMT-N % Change	
	Thickness	Weight	Thickness	Weight
H ₂ SO ₄	1.13	1.83	1.81	1.59
HCl	1.09	1.79	0.85	1.89
NaOH	2.1	2.0	1.9	1.4

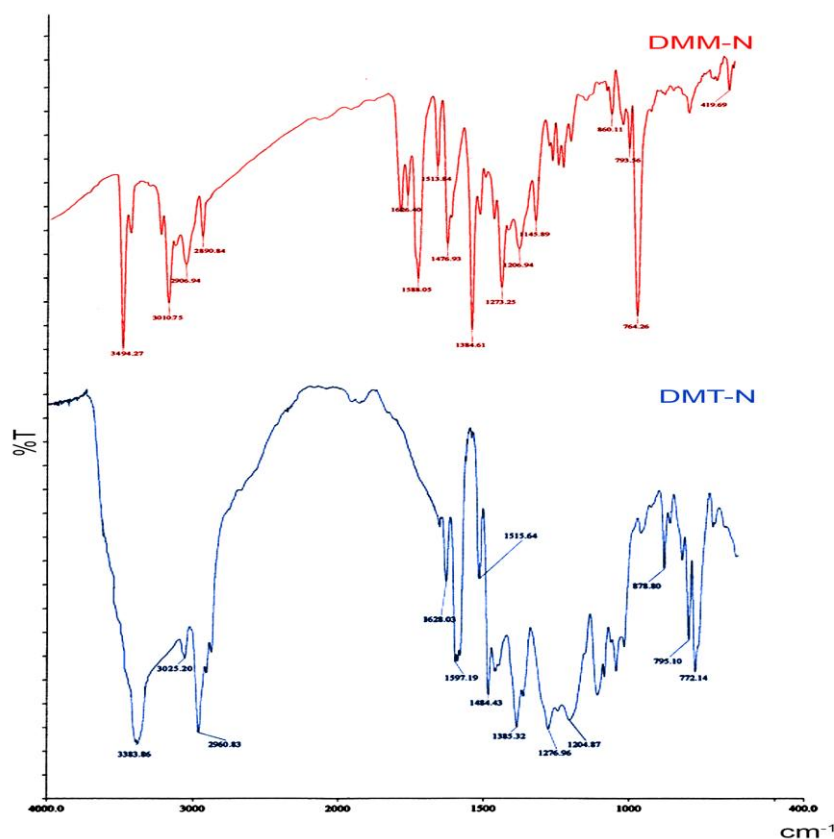


Figure-1: FT-IR Spectra of Phenolic Resins

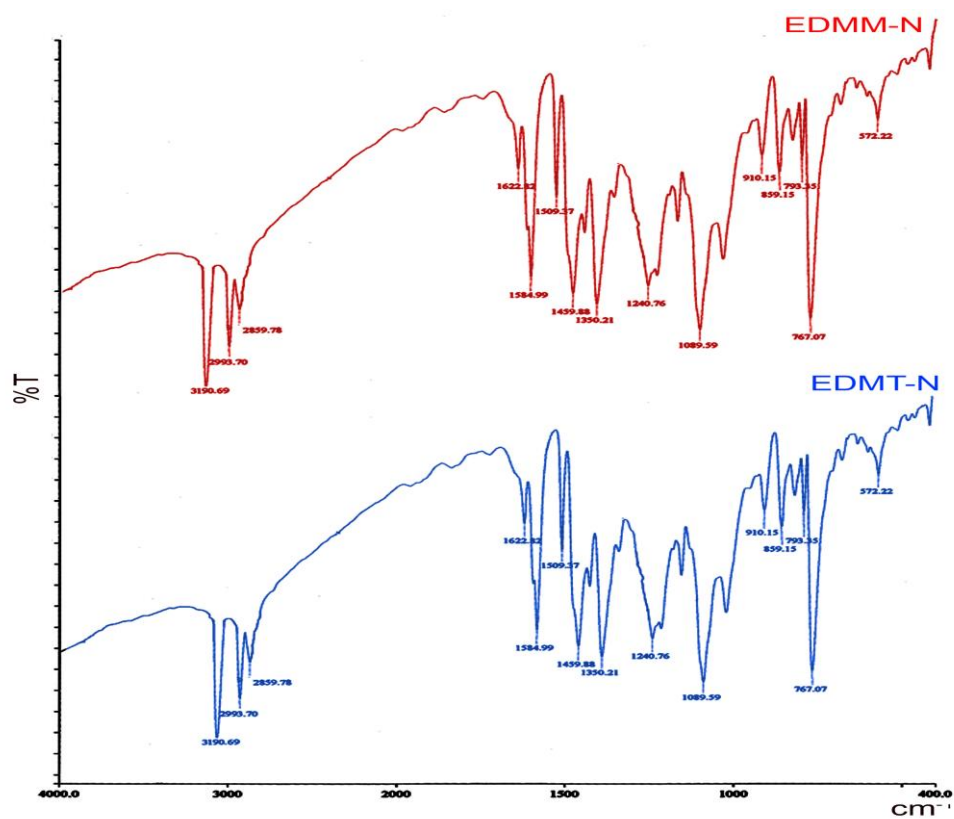


Figure-2: FT-IR Spectra of Epoxy resins

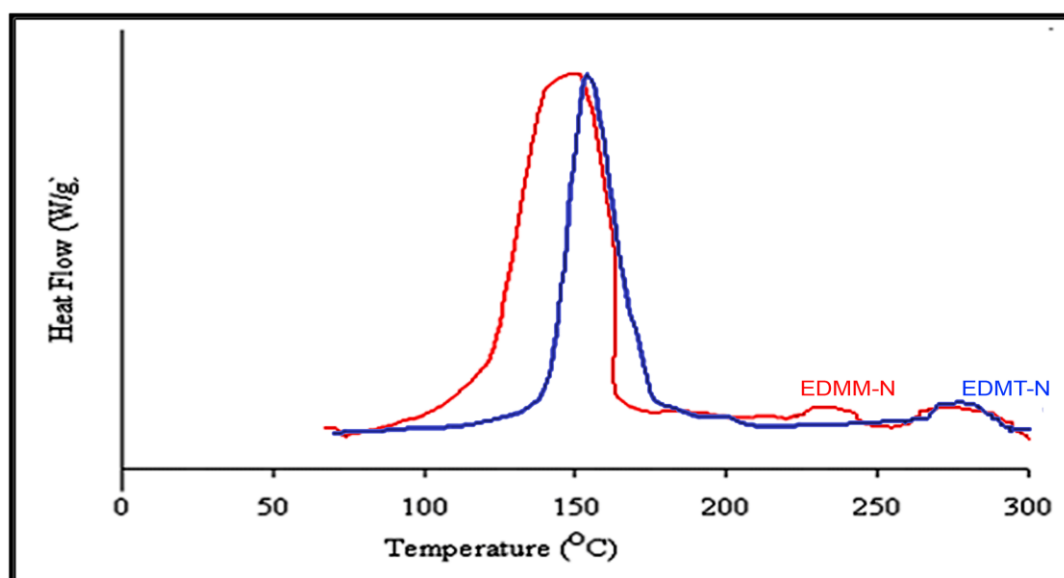


Figure-3: DSC Thermogram of Epoxy resins

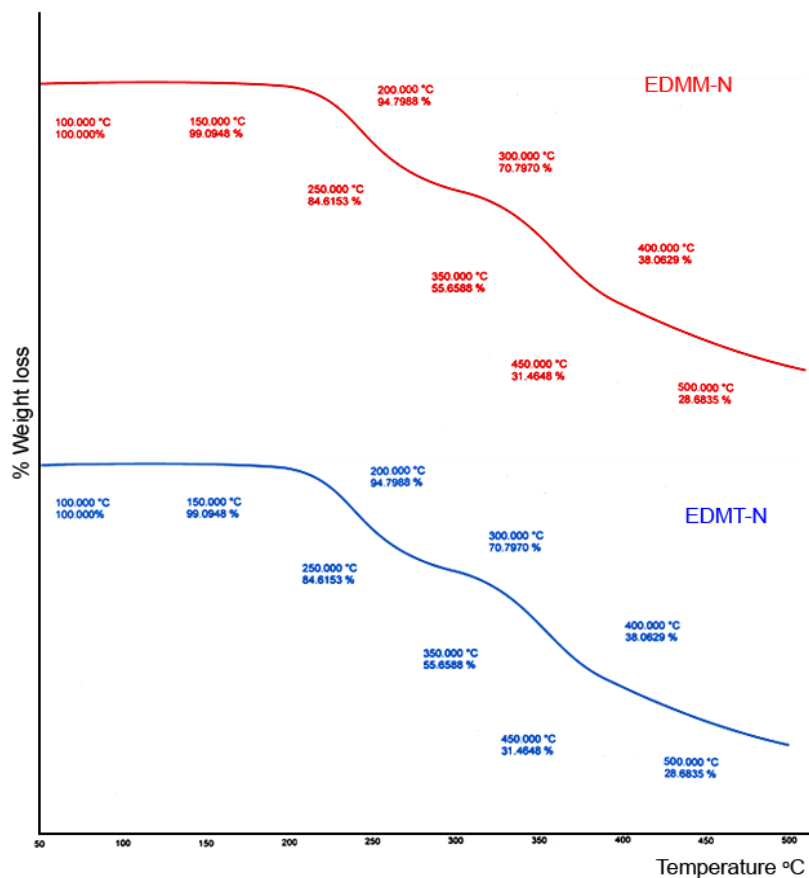


Figure-4 : TGA of Epoxy resins