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Synthesis, Spectral and Thermal Characterization of Epoxy Resin Containing Azomethine Moiety

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ABSTRACT

Azomethine epoxy resin were synthesized from epichlorohydrin and Bis(3-methoxy-4-hydroxy benzylidene) hydrazine and Bis (4-hydroxy benzylidene) hydrazine and its chemical structure was characterized by ^{1}H nuclear magnetic resonance(^{1}H-NMR), infrared(IR), ^{13}CNMR and UV-VIS spectroscopy. The cure behaviors of these epoxy resin were studied by dynamic differential scanning colorimeter and thermal stability determined by TGA. The resultant epoxy thermosets had high thermal stability (upto $200^{0}C$).

KEYWORDS

Synthesis, Thermal Analysis,Spectral analysis, Azomethine epoxy resin, Thermal resistance.

1. INTRODUCTION

Polyazomethines shows greater interest because of their attractive properties such as high thermal stability, chelate forming ability, semiconducting property, fiber formingproperty, electronic, optoelectronic, electrochemical and non-linear optical applications. Studies in the synthesis of conjugated polymers and investigation of their properties constitute a large area of research in recent polymer science. By adding flexible side chain onto polyazomethines which changes the base properties because of the C=N linkage in backbone. Strong protonic acid or Lewis acids in aprotic solvents were used to achieve processability and solubility of rigid chain polymers. A series of epoxy derivatives of thermosets are prepared. The epoxy compounds were characterized by elemental analyses as well as by IR and ^{1}H NMR and ^{13}C NMR spectroscopy. All epoxy compounds were thermally polymerized and the thermal stabilities of the resulting polymers were evaluated by dynamic TG , DTA and DSC techniques.

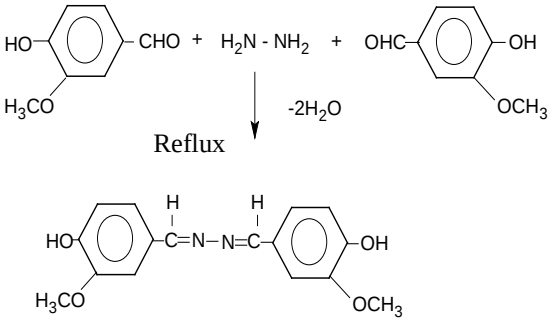
2.EXPERIMENTAL METHODS

The materials employed are p-hydroxy benzaldehyde, vanillin, hydrazine hydrate, epichlorohydrin, ethanol were used as received. The other reagents and solvents were either used as received or purified by adopting standard procedure.

3.SYNTHESIS OF MONOMERS

3.1 Synthesis of bis (3-methoxy-4-hydroxy benzylidene) hydrazine (BMHBH)

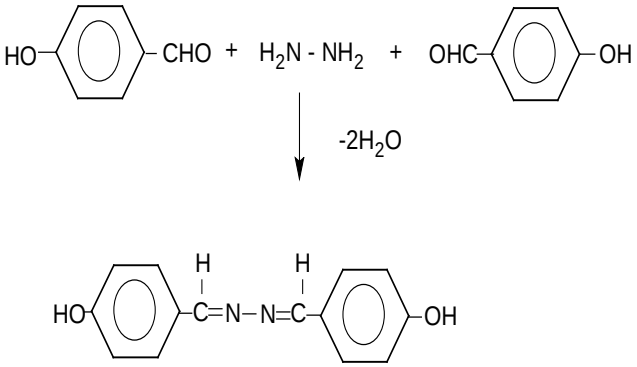
To a solution of vanillin in ethanol was added solution of hydrazine hydrate. The colour of the reaction mixture gradually changed to yellow that was heated to reflux with continuous stirring at 60^{o} C for 3 hours to complete reaction and a yellow precipitate was obtained. The precipitate was filtered and dried in oven.



Bis (3-methoxy-4-hydroxy benzylidene) hydrazine (BMHBH)

3.2 Synthesis of (4-hydroxy benzylidene) hydrazine(BHBH)

To a solution of p-hydroxy benzaldehyde in ethanol was added solution of hydrazine hydrate . The colour of the reaction mixture gradually changed to yellow that was heated and a yellow precipitate was obtained. The precipitate was filtered and washed with ethanol and dried in oven.

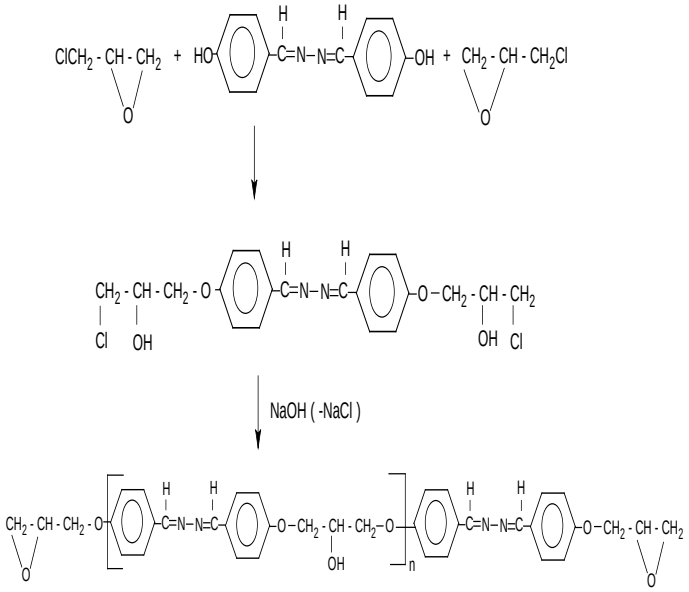


Bis (4-hydroxy benzylidene) hydrazine (BHBH)

4. SYNTHESIS OF POLYMER

4.1 Synthesis of azomethine epoxy resin (BMHBH resin)

5g of BMHBH is taken in 250ml double neck RB flask and 1.25g of NaOH and water was added. Then epichlorohydrin was added to the BMHBH and stirred with continuous stirring at 90°C for 5 hours using mechanical stirrer. The pale yellow precipitate was obtained. It was filtered and washed with water and diethyl ether.



Azomethine epoxy resin (BMHBH resin)

4.2 Synthesis of azomethine epoxy resin (BHBH resin)

5g of BHBH is taken in 250ml double neck RB flask and NaOH and water was added. Then epichlorohydrin was addd to the BHBH and stirred with continuous stirring at 90°C for 5 hours using mechanical stirrer. The obtained pale precipitate was filtered and washed with water and diethyl ether.

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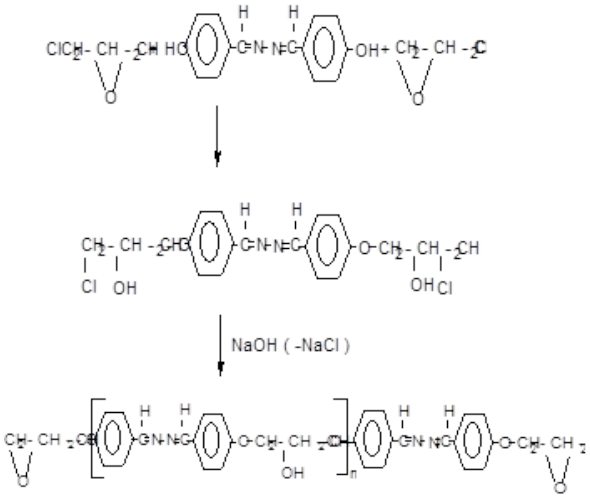
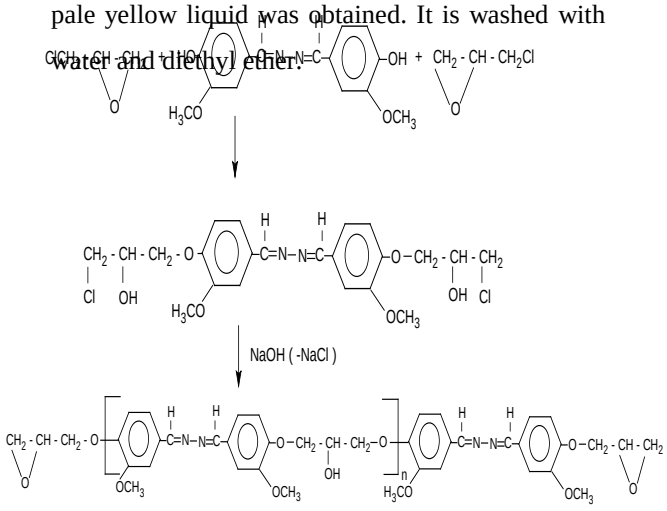


Azomethine epoxy resin (BHBH resin)

4.3 SYNTHESIS OF LIQUID EPOXY RESIN (BMHBH)

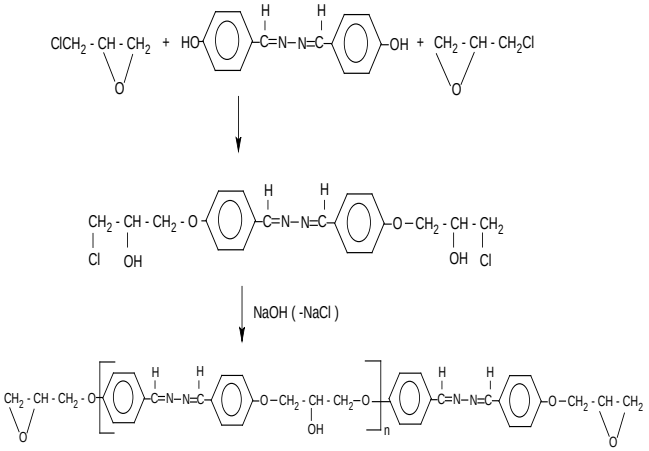
5g of BMHBH is taken in 250ml double neck RB flask and 1.25g of NaOH in 12.5ml water was added. Then 100ml of epichlorohydrin was added to the BMHBH and stirred with continuous stirring at 90°C for 7 hours using mechanical stirrer. The pale yellow liquid was obtained. It is washed with water and diethyl ether.



LIQUID EPOXY RESIN (BMHBH)

4.4 SYNTHESIS OF LIQUID EPOXY RESIN (BHBH)

5g of BHBH is taken in 250ml double neck RB flask and 1.56g of NaOH in 15.6ml water was added. Then 100ml of epichlorohydrin was added to the BHBH and stirred with continuous stirring at 90°C for 7 hours using mechanical stirrer. The pale yellow liquid was obtained. It is washed with water and diethyl ether.



LIQUID EPOXY RESIN (BHBH)

5. TECHNIQUES FOR THE STUDY

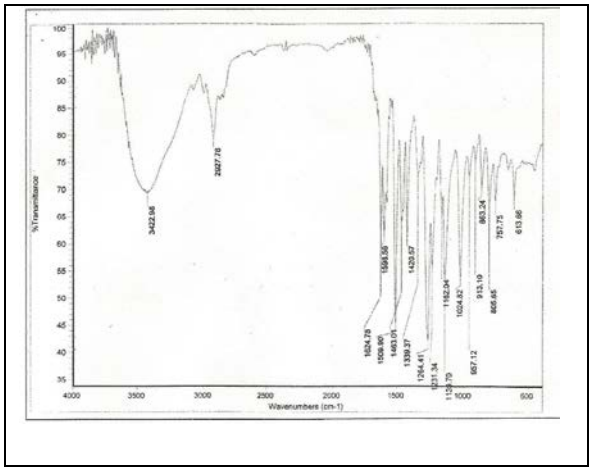


Fig.1- FTIR Spectrum of Epoxy Resin of BHBH

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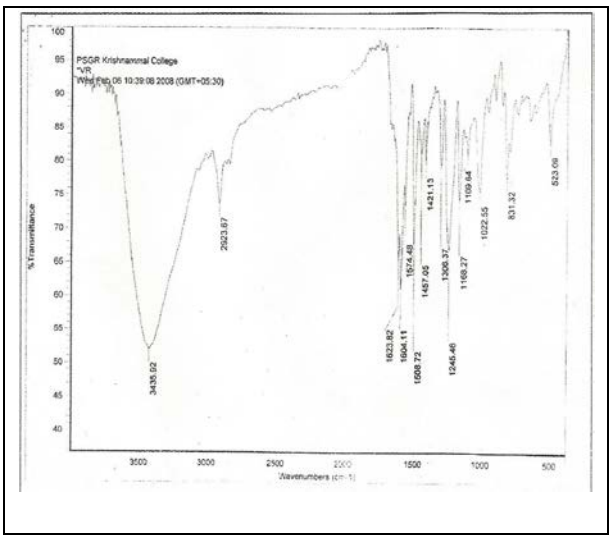

Fig.2 - FTIR Spectrum of Epoxy Resin of BMHBH

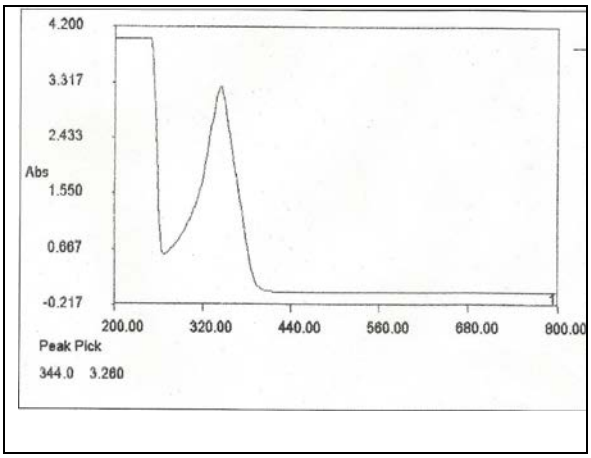

Fig.3 - UV-Vis Spectrum of Epoxy Resin of BMHBH

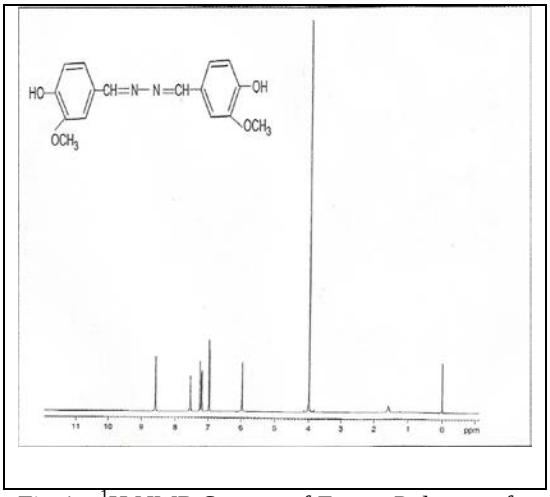

Fig.4 – ^{1}H NMR Spectra of Epoxy Polymer of BMHBH

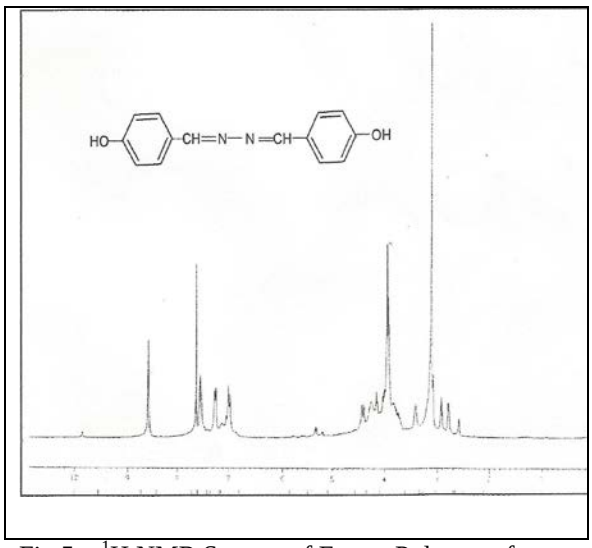

Fig.5 – ^{1}H NMR Spectra of Epoxy Polymer of BHBH

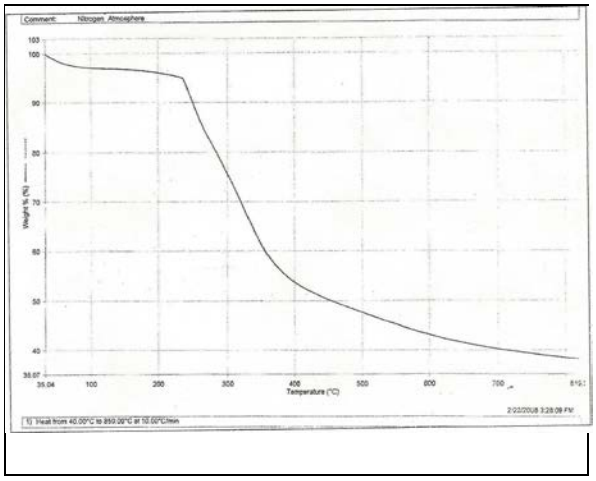

Fig.6- TG Curve of Epoxy Polymer of BMHBH

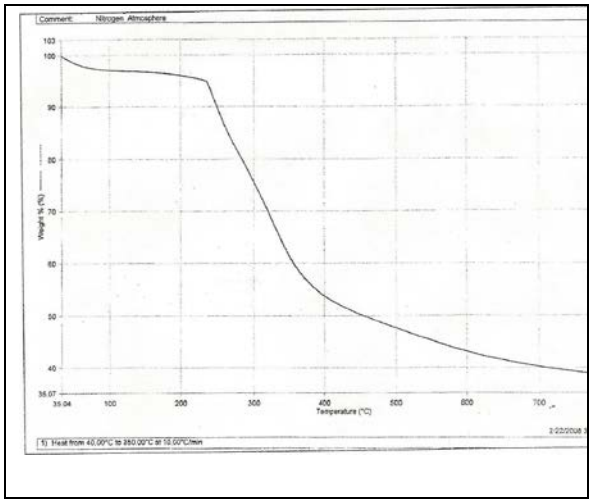

Fig.7 - TG Curve of Epoxy Polymer of BHBH

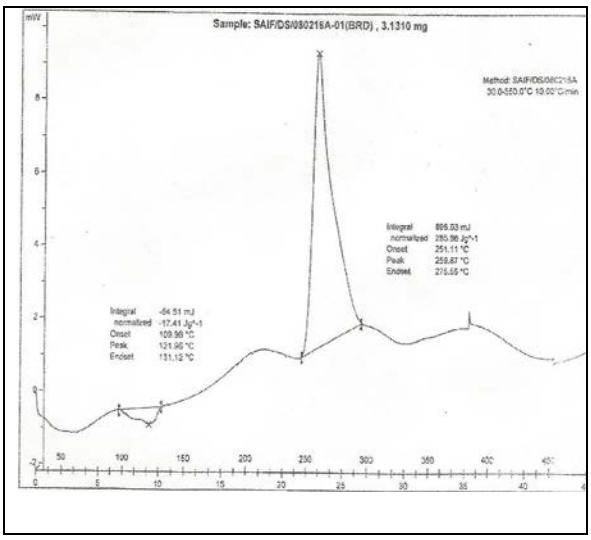

Fig.8 - DSC Curve of Epoxy Polymer of BMHBH

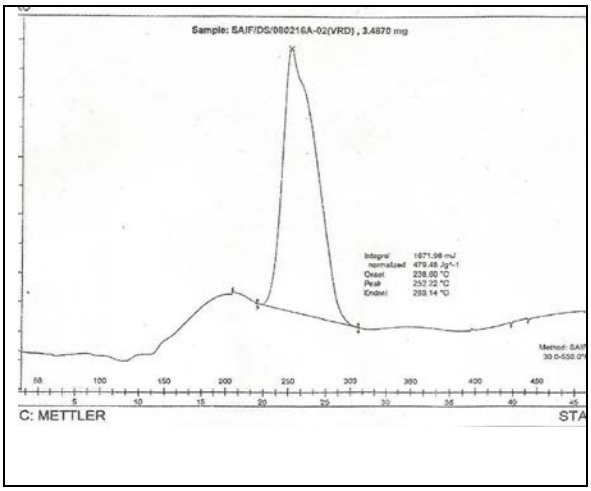

Fig.9 - DSC Curve of Epoxy Polymer of BHBH

SUMMARY

An azomethine bisphenol (BMHBH) and (BHBH) were synthesized by condensing vanillin and p-hydroxy benzaldhyde with hydrazine hydrate respectively in ethanolic medium.

A solid azomethine epoxy resins were obtained by treating the diol with epichlorohydrin in presence of aq.NaOH.

The liquid epoxy resins were obtained by treating of diol with excess of epi chlorohydrin in presence of aq.NaOH.

The synthesized epoxy resins is soluble in highly polar solvents like DMSO, NMP and Con.H_2SO_4.

The synthesized diol and epoxy resins have been confirmed by Spectral analysis like FT-IR, UV-Vis and NMR.

TGA and DSC determined the thermal behavior of epoxy resins.

The epoxy resins were stable up to 200˚C and then it disintegrate, which is confirmed by both TGA and DSC.

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