**Research Paper**

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# Applications of IR Spectroscopy for Two Component Polyurethane Coatings

**Science**

**KEYWORDS :** Epoxy polyol, Isocyanate hardener, FT IR, Properties,

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## ABSTRACT

*Polyurethane coatings are formed from a chemical reaction between a polyol and a polyisocyanate hardener. Two component solvent based polyurethane coatings were prepared by blending of epoxy polyol and Toluene Di Isocyanate (TDI) hardener. The composition was based on the solid contents of the hydroxyl equivalent weights of epoxy polyol and NCO equivalent weight of TDI. After the film coated with the prepared polyurethane coatings, the various physical properties and mechanical properties were measured. The formation of the various functional groups like Hydroxyl group,methylene group was supported by FTIR spectral data. The IR data showed the conformation for the formation of polyurethane and in the thermal product formation.*

## Introduction

The chemical structure of epoxy resins gives them high chemical resistance against a wide range of severe corrosive conditions [1]. These properties are derived from the aromatic nature of the backbone and good chemical stability of the phenolic ether linkage. Two component polyurethanes (PU) have found extensive applications in the coating industry mainly because they exhibit excellent abrasion resistance, toughness, low temperature flexibility, chemical and corrosion resistance and a wide range of mechanical strength[2-4]. Polyurethane coatings are formed from a chemical reaction between a polyol and a polyisocyanate hardener[5]. Among the common commercially available polyols for two components PU systems are hydroxyl functional polyester, acrylics, polyether and polyols. Hydroxyl-terminated polyurethane (HTPU) prepolymer and crystalline polymer particles were used to modify the toughness of diglicidyl ether of bisphenol-A (DGEBA)epoxy cured with diaminodiphenyl sulphone(DDS) having the improved toughness property as reported by Huei-Hsiung Wang et al[6]. Lizhi-hua et all study the structural characteristics and properties of polyurethane modified TDE85 (Diglycidyl-4,5-epoxy cyclohexane-1,2-dicarboxylate)/ MeTHPA (Methyl tetrahydrophalic anhydride) epoxy resin with interpenetrating networks[7]. Jong-Shin Park [8] prepared and investigates the mechanical properties PU films using starch as main polyol component with Toluene Diisocyanate (TDI) for various polyol starch content and NCO/OH molar ratio. Cardopolyester polyol has been synthesized by reacting epoxy resin of 1, 1 bis (3Methyl 4 Hydroxy phenyl) cyclohexane and recinoleic acid by PH Parsania[9]) for their industrial importance as coating and adhesive materials. We have synthesised hydroxyl terminated epoxy systems as epoxy polyol resin[10]. Two component polyurethane coatings were prepared by blending of epoxy polyol and Toluene diisocyanate (TDI) hardener and the ratio of blending were optimised by using the physical,chenical and electrochemical studies[11]. In the present work we have conformed the presence of various functional groups and formation of the product for air dry applications and hot cure applications by using the IR studies and the mechanical properties.

## EXPERIMENTAL ANALYSIS

*Materials* - Epoxy resin GT 6071-X75(Diglycidyl ether of Bisphenol-A Type, Epoxy equivalent weight 501 g/Eq and solids 75% in Xylene) from M/S Ciba Gaey – Mumbai, Diethanol amine from Spectrum Baroda, Xylene, cellosolve acetate and butyl acetate from E-Merck, Toluene diisocyanate (TDI) Desmodur L75% from M/S Bayer chemicals. A three neck flask (500 ml), equipped with a mechanical stirrer, condenser and a Thermometer was placed in a water bath. One mole of di-epoxy resin GT 6071-X75-(266.67g), and two moles of Diethanolamine (42 g) were placed in a three neck flask. Then Cellosolve acetate (40 g) and Xylene (93.33 g) was added as solvents to carry out the reaction and stirred well at room temperature for 5 minutes. Gradually the temperature increases from 60°C to 70°C to open up the oxirane ring of epoxy resin to generate secondary alcohol. The reaction mixture was refluxed for 6 Hrs with stirring The transparent secondary hydroxyl terminated epoxy polyol resin was transferred to air tight bottle[10]. To a 100 ml beaker containing Epoxy polyol resin (33.26g), TDI hardener (42.57 g), Butyl acetate (5.00g),Cellosolve acetate(3.00g) and Xylene(16.17g) (Xylene, Butyl acetate and Cellosolve acetate used as solvents for PU coatings) at room temperature and stirred manually for ten minutes as the optimised ratio 1 : 1.10 ratio (OH/NCO ratio)[11]. The test solution was thoroughly mixed just before the application of their films on to mild steel panels. Films were applied with varying wet film thickness, such that the dried films of all the compositions had a thickness of approximately 50 microns measured by using a magnetic thickness gauge. The films were allowed to cure at ambient conditions (Room temperatue30 °C and relative humidity of approximately 50-60%) for at least 48 Hrs before tests for mechanical and chemical properties were carried out. Compositions were tested for their pot life by checking for the rise in their viscosity after mixing the two components at an interval of 20 min until the value approximately double the initial value at ambient temperature. Films were tested for drying time (ASTM D 5895), Micro Gloss (IS 101- Testing of paints – Part 4 – Optical test Section 4-Gloss), Scratch hardness (IS 101 – Testing of paints – Part 5-Mechanical test on paint films Section 1 – Hardness test, Pencil hardness [19], Cross hatch adhesion (ASTM D 4752), impact resistance (ASTM D 2794-92) and salt spray test (ASTM B 117). Solvent resistant and Chemical resistance of the films was studied by the spot test for 30min under 2" watch glass. The FT IR spectra were taken and interpreted for Epoxy polyol resin on KBr pellets and recorded using BomemMichelson series Spectrophotometer.

## RESULTS AND DISCUSSION

### IR Specturm of Epoxy polyol:

**IR spectrum of Epoxy polyol in Figure 1 was showed the following bands:**

**3389.23 cm$^{-1}$ – OH – Stretching[12],**

**3036.45 cm$^{-1}$ – C-H bonding for aromatic carbon[13],**

1475.60 cm$^{-1}$ – Tertiary –C-C- stretching[14] due to the presence of the above absorption bands in the spectra. It was confirmed that, the formation of the epoxy polyol resin shown in the reaction scheme[15] (Figure 2).

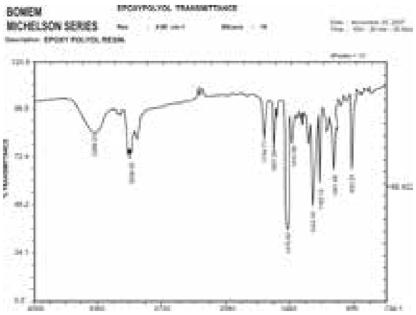
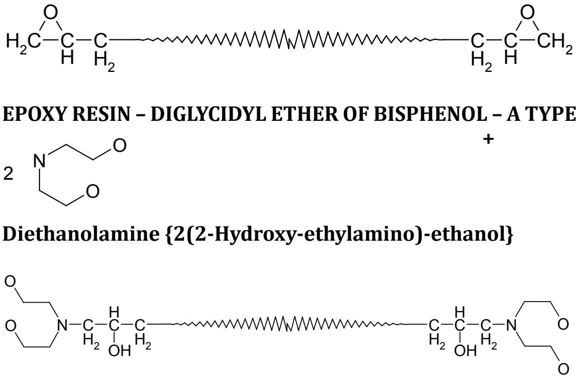


Figure 1- IR Spctrum of Epoxy polyol



**EPOXY RESIN – DIGLYCIDYL ETHER OF BISPHENOL – A TYPE**

+

**Diethanolamine {2(2-Hydroxy-ethylamino)-ethanol}**

**Epoxy polyol**

**Figure 2 – Reaction scheme of Epoxy Polyol**

**IR Specturm of TDI:**
FTIR Spectrum of TDI in Figure 3, showed the following bands,

A peak at 3020.80 cm$^{-1}$-C-H stretching in aromatic carbon and

2960 cm$^{-1}$ C-H stretching in methyl group[16]

2219.24 cm$^{-1}$– N-C Stretching and 1680.94 cm$^{-1}$ –C=O Stretching[15]

From the above peaks, it was confirmed that the tested sample contain NCO groups to react with the epoxy polyol as a hardener.

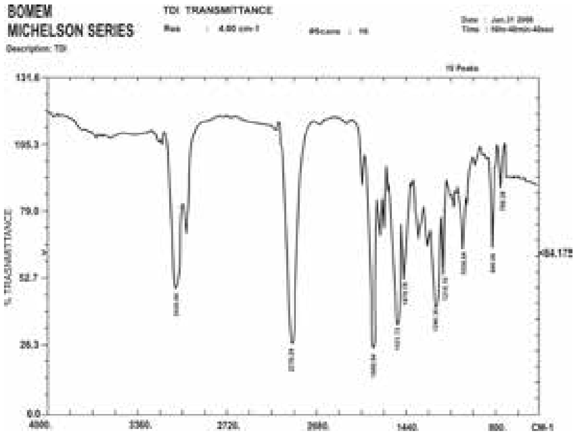


**Figure 3- FT IR of TDI**

**FTIR Spectra of polyurethane – Air drying**
IR Spectrum of the polyurethane is shown in Figure 4. The test solution prepared as per the procedure given above in chemical composition section 4.2.1. Epoxy polyol and TDI are taken in the ratio of 1: 1.10 as a test solution, showed the following bands, The broad peak appeared at 3380.00 cm$^{-1}$- was resulting from the formation of NH of the urethane linkage[17] ,3020 cm$^{-1}$ - C-H Stretching for aromatic[18], 2220 cm$^{-1}$– N-C Stretching, 1720 cm$^{-1}$ –C=O Stretching (for the formation of urethane linkage)[19], 1520 cm$^{-1}$ - Tertiary –C-C- Stretching. From the above bands the formation of polyurethane product has been confirmed.

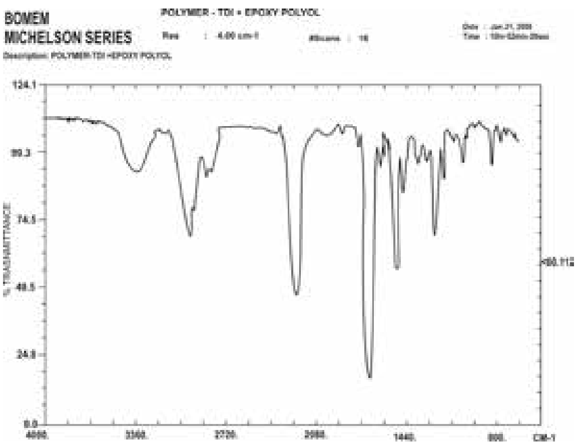


**Figure 4 - FT IR of PU**

**FTIR spectra of polyurethane- Heat cured sample**
FTIR Spectrum of polymer-H is shown in Figue 5; the preparation of the sample for the test was the same as above. The sample was kept in hot air oven for 80°C at 30 min. The spectrum was taken after the sample cooled to room temperature. The observed bands were not much varied from the FTIR Spectra for polyurethane. The FTIR spectra for both of the PU systems cured under different conditions, it was concluded that ambient cure is equivalent to curing at 80°C at 30 min[9].

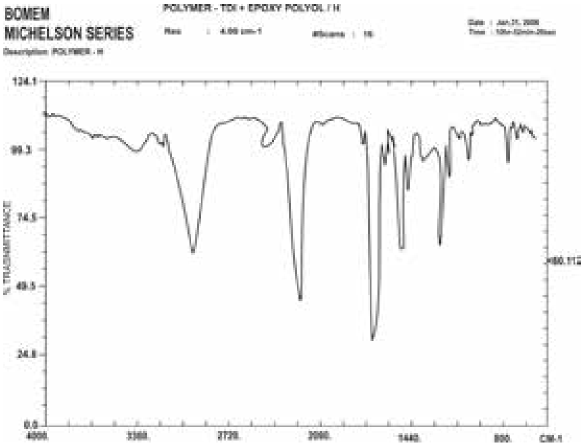


**Figure 5 – FT IR of Hot cure**

The preparation of the test solution was the same as above. The test solution was applied on a mild steel panel. Let the panel was exposed in an open air for 10 Min as a flash-off time, i.e. the time allowed for the bulk of the solvent before entering a stoving oven. Then the panel was kept in the hot air oven 120°C at 30 min. After the cooling of the coated film the dry film properties were compared and tabulated (Table-1).

From the Table1, the physical properties of the coated film from the stoving application showed better than the film formed from the air drying application. The diisocyanate becomes effectively active at the curing temperature. Hot air oven cure improves physical properties of the network[20,21]. Hence, epoxy polyol and TDI composition can be used for air drying cum stoving applications.

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**Table 1 – Dry film characteristics of ambient cured film and hot cured film**

| Dry film characteristics | Hot cure film | Ambient cure film |
|---|---|---|
| (EP : TDI) | 1.00 : 1.10 | 1.00 : 1.10 |
| Micro gloss@20° Angle<br>Micro gloss@60° Angle | 90<br>100 | 94<br>102 |
| Impact test<br>- Direct(60')<br>- Indirect(60') | Passes<br>passes | Passes<br>Passes |
| Scratch hardness | 2.3 Kgs | 2.1 Kgs |
| Cross hatch test | 100% | 100 % |
| Pencil hardness(2HB) | Passes | Passes |
| Conical mandrill test | Passes | Passes |
| Chemical resistance<br>- Acetic acid<br>- Sodium chloride<br>- Hydrochloric acid<br>(10% in water 8 Hrs) | P(12H)<br>P(12H)<br>P(12H) | P(10H)<br>P(10H)<br>P(10H) |
| Solvent resistance<br>- O-xylene<br>- MEK<br>(30 Min under 2" watch glass) | P(70M)<br>P(70M) | P(45M)<br>P(45M) |
| Salt spray test(5% salt soln) | 450 Hrs | 440 Hrs |

## Conclusion

Epoxy polyol can be synthesized by the reaction of epoxy resin and diethanolamine. The two component polyurethane coatings were prepared by blending with polyisocyanate, Toluene diisocyanate with these epoxy polyol resin. The prepared coatings showed a rapid drying character with 90-120 Min of pot life and good gloss level.The physical and mechanical properties of the coated film from the stoving application showed better than the film formed from the air drying application. The diisocyanate becomes effectively active at the curing temperature. Hot air oven cure improves physical properties of the network· Hence, epoxy polyol and TDI composition can be used for air drying cum stoving applications. The FTIR studies have helped in the conformation of the formation of the polyurethane by NH stretching (3360 cm$^{-1}$) and CO stretching (1730 cm$^{-1}$) frequencies. The FTIR spectra for air drying and hot cure PU systems it was concluded that ambient cure is equivalent to curing at 80°C for 30 min.

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