

Synthesis and Characterization of Cardanol-Based Polyols

Dr. N. J. Sangeetha

Assistant Professor, Department of Chemistry, Women's Christian College, Nagercoil, Tamil Nadu, India

ABSTRACT

Cardanol-based polyols were synthesized from by condensing cardanol with formaldehyde using malonic acid as catalyst using epichlorohydrin. The polyols were characterized by physico-chemical properties such as specific gravity, viscosity, hydroxyl value, iodine value, moisture content and spectral studies such as FT-IR, NMR. It concludes that the specific gravity, viscosity and hydroxyl value of polyol depicts higher due to intra- and inter-molecular hydrogen bonding through dihydroxypropyl chain ends.

Keywords: Cardanol, Formaldehyde, Malonic Acid, Epichlorohydrin, Methanol.

I. INTRODUCTION

Polyol is one of the essential raw materials in the preparation of polyurethane products using aliphatic and aromatic isocyanates with better thermal stability and mechanical properties. Cardanol-based polyols will have better hydrolytic stability compared to the triglyceride oil based polyols. Cardanol is a phenolic compound with a C₁₅ unsaturated aliphatic side chain in the meta position obtained from cashew nut shell liquid.

Depending upon the hydroxyl value and other characteristics of the polyol, it finds application in the development of adhesives, coatings and foams¹⁻⁶. The prepared polyol has unique structural characteristics such as C₁₅ chain length, which contributes to flexibility, and an aromatic ring, which imparts rigidity in the final application of resulting polyurethane. By choosing optimum ratio of NCO/OH, it is possible to obtain a system, which can be used for the development of a flexible as well as a rigid polymer for suitable application⁷.

II. EXPERIMENTAL

A. Materials

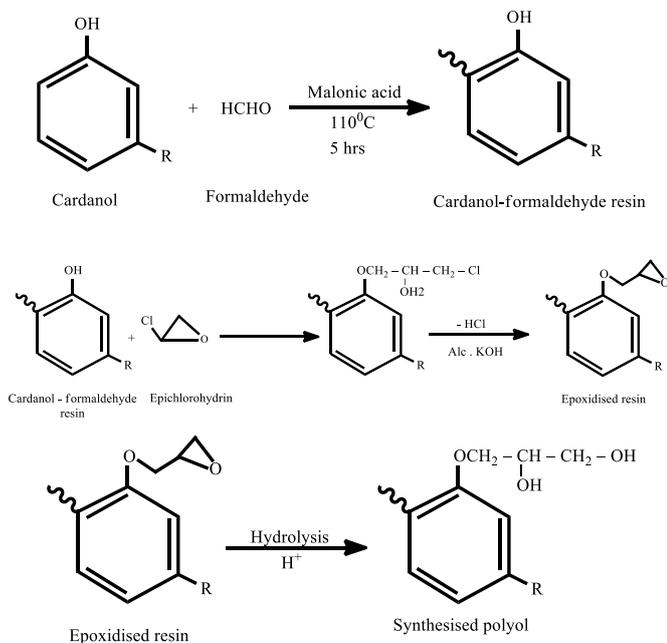
Cardanol was obtained from M/s Satya Cashew Chemicals Pvt. Ltd., Chennai. Formaldehyde (40% solution), and malonic acid and methanol were received from Merck, Mumbai. Epichlorohydrin was obtained from Sisco Research Laboratories Pvt. Ltd., Mumbai.

B. Methods

¹H-NMR spectra are recorded in CDCl₃ with tetramethylsilane as an internal standard. The spectrum was recorded using Bruker Avance H 500 MHz spectrometer. Infrared spectra of the polyurethane and its composites were taken in a Shimadzu FT-IR-8400S spectrometer by KBr pellet method.

C. Synthesis of cardanol-based polyols

Cardanol-based polyols were synthesized by the condensation of cardanol and formaldehyde using malonic acid as catalyst (Scheme 1). Cardanol was taken in a three necked flask equipped with a Liebig condenser, mechanical stirrer and thermometer. Formaldehyde and 1% malonic acid catalyst in methanol was added to the cardanol through a dropping funnel. The reaction was carried out at temperature 110° ± 5°C for 5 h. After, it was reacted with epichlorohydrin at 60°C for 4 h to obtain epoxidized resins. Finally, cardanol-based polyols were synthesized from epoxidized resins using methanol, the epoxide ring was opened at 60°C for 30 min (Scheme 1). Thus, the formed polyols were analysed.



Scheme 1

Formation of cardanol-based polyol

III. RESULTS AND DISCUSSION

The decrease in the iodine value of the polyols may be due to the steric hindrance of adjacent bulky groups to the addition of iodine monochloride. The specific gravity and viscosity of the polyols are found to be greater than that of cardanol-formaldehyde resins. Higher specific gravity of the polyols is due to intra- and inter-molecular hydrogen bonding through dihydroxypropyl units. The determination of molecular weight, hydroxyl number and FT-IR spectral analysis leads to predict the structure of synthesised polyols as presented in Table 1.

Table 1. Physico-chemical properties of cardanol-based polyol

Properties	Cardanol-based polyol
Colour	Yellowish brown
Odour	Phenolic
Specific gravity (g/cc at 30°C)	0.9446
Viscosity at 30°C (cps)	281
Iodine value (Wij's method)	213

Hydroxyl value	229
Molecular weight	1394
Number of hydroxyl groups	6
Moisture content (%)	0.867

FT-IR spectra of the polyol (Fig 1), exhibited the disappearance of peak near 910 cm^{-1} due to oxirane group of epoxide linkage and appearance of a new peak between $1625\text{--}1700\text{ cm}^{-1}$ due to dihydroxypropyl chain ends, confirmed the formation of synthesised polyols.

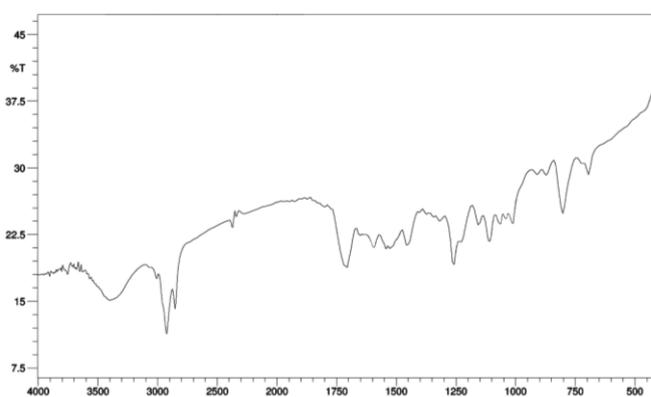


Figure 1. FT-IR spectrum of cardanol-based polyol

$^1\text{H-NMR}$ spectra of the polyol (Fig 2), showed a new peak around $4.18\ \delta$ reveals the presence of methylene protons to $-\text{C-CH}_2\text{-O-}$ of dihydroxypropyl chain ends.

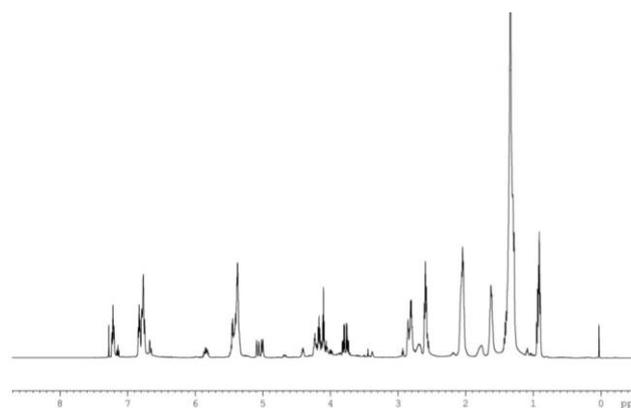


Figure 2. $^1\text{H-NMR}$ spectrum of cardanol-based polyol

IV. CONCLUSION

The specific gravity, viscosity and hydroxyl values of polyol depicts higher due to intra- and inter-molecular hydrogen bonding through dihydroxypropyl chain ends. The low iodine value of the polyol exhibits the steric

hindrance of adjacent bulky groups to the addition of iodine monochloride.

V. REFERENCES

- [1]. P. S. Kumar, N. A. Kumar, R. Sivakumar, C. Kaushik, Experimentation on Solvent Extraction of Polyphenols From Natural Waste, *J. Mater. Sci.*, 44, 5894, 2009.
- [2]. S. Gopalakrishnan, N. T. Nevaditha, C. V. Mythili, Synthesis and characterization of bifunctional monomers for high performance polymers from renewable resource, *Inter. J. ChemTech Res.*, 4(1), 48, 2012.
- [3]. M. Alam, D. Akram, E. Sharmin, F. Zafar, S. Ahmad, Vegetable oil based eco-friendly coating materials: A review article, *Arabian J. Chem.*, 7, 469, 2014.
- [4]. A. Guo, I. Javni, Z. S. Petrovic, Rigid polyurethane foams based on soybean oil, *J. Appl. Polym. Sci.*, 77, 467, 2000.
- [5]. Z. S. Petrovic, A. Guo, W. Zhuang, Structure and properties of polyurethanes based on halogenated and nonhalogenated soy based polyols, *J. Polym. Sci., Part A: Polym. Chem.*, 38, 4062, 2000.
- [6]. I. Javni, Z. S. Petrovic, A. Guo, R. Fuller, Thermal stability of polyurethanes based on vegetable oils, *J. Appl. Polym. Sci.*, 77, 1723, 2000.
- [7]. V. Athawale, N. Shetty, Synthesis and characterization of low cost cardanol polyurethanes, *Pig. Resin Techn.*, 39(1), 9, 2010.