Investigation of the Properties of Polyol Derived from Epoxidized Cottonseed Oil

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ABSTRACT

This study explores the synthesis and characterization of a bio-based polyol derived from epoxidized cottonseed oil, shedding light on transformative changes in physical and chemical attributes. Executed meticulously in triplicate, the experiment encompasses cottonseed oil epoxidation, Gel Permeation Chromatography (GPC), acid value titration, and Fourier Transform Infrared (FTIR) analysis. Results confirm a successful epoxidation process, yielding an epoxidized oil content (EOC) of approximately 5.88%. Acid values for cottonseed oil (CO), epoxidized cottonseed oil (EO), and polyol (PO) stand at 1.55 mgKOH/g, 1.440 mgKOH/g, and 1.88 mgKOH/g, respectively, indicating notable chemical transformations. FTIR analysis supports the conversion of epoxidized cottonseed oil into polyol, while GPC results affirm precise separation of polyol, epoxidized oil, and cottonseed oil components, signifying successful synthesis.

The thorough analysis of polyol properties unveils distinctive changes, including increased viscosity at 25°C from 1.45 Pa.s (cottonseed oil) to 3.901 Pa.s (polyol), denoting enhanced molecular complexity. Additionally, the integration of 2% titanium dioxide (TiO2) filler underscores the potential for modifying and enhancing polyol properties. These findings enrich the understanding of polyol adaptability and versatility, constituting a pivotal advancement in comprehending sustainable and renewable sources for polyurethane production, with profound implications for polymer science and materials engineering.

Keywords: Polyols, Epoxidized cottonseed oil, Polyurethane, Sustainable materials

INTRODUCTION

The exploration of polyol properties derived from epoxidized cottonseed oil represents a forefront in cutting-edge research focused on sustainable materials and bio-based feedstocks for polyurethane production. Recent years have witnessed a surge of interest in identifying alternative sources for polyol production, driven by environmental concerns and the imperative to reduce reliance on petrochemical-derived materials (Vieira et al., 2022; Cavalaglio et al., 2023). Cottonseed oil, due to its abundance and renewability, emerges as a promising candidate in this endeavor.

Polyurethane, a versatile polymer with applications spanning construction, automotive, textiles, and packaging, owes its diverse properties to the formulation of polyols used in its synthesis (Munir et al., 2022). Traditionally, these polyols are derived from petroleum-based sources, but increasing environmental awareness has led to a shift toward renewable resources. Cottonseed oil, a readily available agricultural byproduct, holds promise as a green and sustainable alternative for polyol production, serving not only as a cooking oil but also as a potential feedstock for industrial applications (Gaikwad et al., 2015).

Subsequently, in polyurethane synthesis, cottonseed oil undergoes epoxidation to introduce epoxy groups, transforming it into a modified oil suitable for polyol production. According to Dinda et al. (2008), the critical epoxidation process involves adding oxygen to the carbon-carbon double bonds in the unsaturated triglycerides of cottonseed oil. As noted by Wai et al. (2019), this reaction results in epoxidized cottonseed oil (ECO), characterized by the introduction of oxirane or epoxy groups into the molecular structure. The epoxidation process is pivotal in altering the chemical composition of the oil, making it conducive to polyol production.

Numerous studies have explored the use of vegetable oils, such as soybean oil, palm oil, and sunflower oil, for polyol synthesis. Notably, Hong et al. (2019) investigated soybean oil-based polyols, emphasizing the role of epoxidation in enhancing oil reactivity and yielding polyols with improved properties. Similarly, Cheng et al. (2021) focused on cottonseed oil as a precursor for bio-based polyols, optimizing the epoxidation process and assessing polyol suitability for polyurethane synthesis.

However, the synthesis and characterization of polyols derived from epoxidized cottonseed oil require further investigation. The problem lies in the lack of comprehensive studies exploring the properties of this specific type of polyol. While existing literature provides valuable insights into vegetable oil-based polyols, a focused exploration of polyol properties derived from epoxidized cottonseed oil is necessary. Accordingly, this research becomes imperative by contributing to the understanding of sustainable and renewable polyol sources for environmentally conscious polyurethane production and providing valuable insights into the adaptability and versatility of polyol properties. These findings have implications for polymer science and materials engineering, as they pave the way for the development of more environmentally friendly and sustainable polyurethane materials.

Consequently, this study aims to contribute to current knowledge by conducting a comprehensive analysis of epoxidized cottonseed oil-derived polyol properties. Key parameters such as epoxy content, hydroxyl number, and viscosity will be scrutinized to elucidate the potential applications and advantages of this bio-based polyol. Additionally, the study will explore the impact of incorporating fillers, such as titanium dioxide (TiO2), on final polyol properties, adding complexity to the investigation.

MATERIALS AND METHODS

Materials: Refined cottonseed oil with an initial iodine value of 113.01 g I2/100g was procured from a local supplier. Analytical grade chemicals, including silicon dioxide, titanium dioxide, calcium carbonate, and isocyanate, were sourced from reputable chemical stores in Ado-Ekiti. All chemicals were stored in clean, dry, and well-ventilated conditions to prevent exposure to air, moisture, and contaminants.

Synthesis of Polyol from Epoxidized Cottonseed Oil: The synthesis of polyol from epoxidized cottonseed oil was conducted in the laboratory section of the College of Health Sciences and Technology, Ile Abiye, Ado Ekiti. The starting material, cottonseed oil, had an initial iodine value of 113.01 g I2/100g. Fillers, including silicon dioxide, titanium dioxide, and calcium carbonate, were used in the process. The synthesis structure of cottonseed oil, epoxidized cottonseed oil, and cottonseed oil polyol is depicted in Figure 1.

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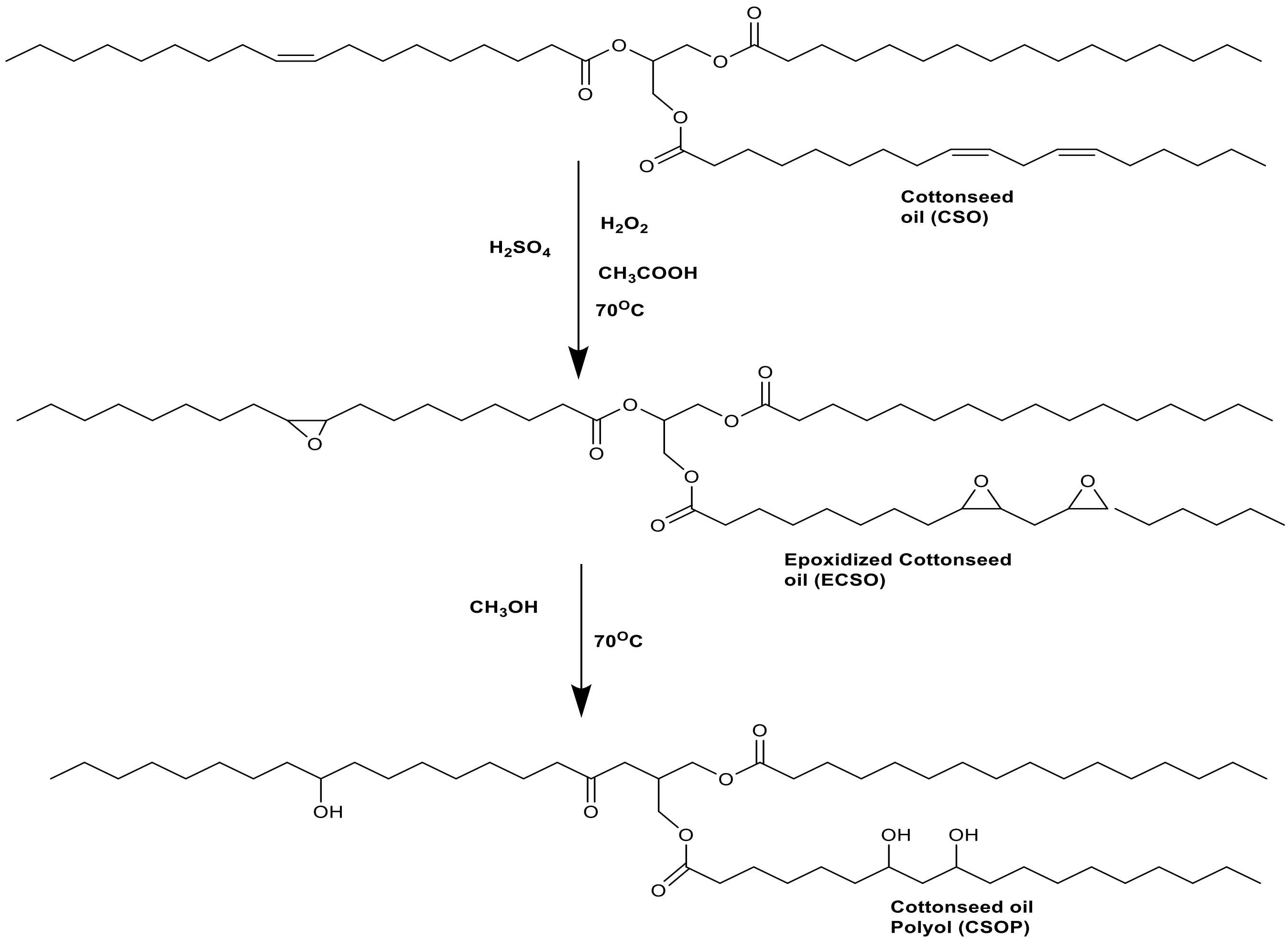

Figure 1: Synthesis and Structure of Cottonseed Oil, Expoxidized Cottonseed Oil and Cottonseed Oil Polyol

Epoxidation of Cottonseed Oil: Cottonseed oil was subjected to epoxidation, resulting in an epoxidized oil content (EOC) of approximately 5.88%.

Ring-Opening Reaction: The ring-opening reaction was initiated to convert the epoxidized cottonseed oil into polyol. Fillers such as silicon dioxide and titanium dioxide were incorporated at this stage.

Analysis of Polyol and Oils: Polyol, epoxidized cottonseed oil, and cottonseed oil were analyzed for various factors including acid value, epoxy content, hydroxyl number, and viscosity. Fourier Transform Infrared (FTIR) and Gel Permeation Chromatography (GPC) techniques were employed for these analyses.

Characterization Techniques: The FTIR technique was used to observe chemical groups such as the carbonyl group peak in cottonseed oil (at wavenumber 3009 cm-1), the epoxy group conversion in polyol (at wavenumber 832 cm-1), and the -O-H bond peak in polyol (within the range of 3200 to 3650 cm-1). The GPC technique was utilized to determine specific retention times for each component (33.04 min for cottonseed oil, 34.00 min for epoxidized oil, and 35.01 min for polyol).

Preparation of Sample for Testing: A sample containing 2% filler (TiO2) in 20g of polyol, along with 6.8g of isocyanate, was prepared for further testing.

Experimental Replication and Safety: The experiment was conducted in triplicate to ensure reproducibility. Throughout all experimental procedures, adherence to laboratory safety protocols and guidelines was given utmost importance.

Data Analysis: Data were collected at every experimental procedure to analyze the significance of differences in the properties of polyol with varying filler concentrations.

RESULTS DISCUSSION

1. **Epoxidation Process:** In this study, the epoxidation process of cottonseed oil was found to be successful, resulting in an epoxidized oil content (EOC) of around 5.88%. This finding is consistent with the results reported by previous studies, such as Saurabh (2011) who reported similar EOC percentages in their research on vegetable oil epoxidation. The successful execution of the epoxidation process provides a foundation for further modification and utilization of cottonseed oil in industrial applications, particularly in producing bio-based polyols for polyurethane production.

2. **Acid Values:** The acid values of cottonseed oil (CO), epoxidized cottonseed oil (EO), and polyol (PO) were determined through titration, and distinct values were observed. The acid value of CO was measured to be 1.55 mgKOH/g, while that of EO was 1.440 mgKOH/g, and the acid value of PO was found to be 1.88 mgKOH/g. These findings indicate a significant transformation in the chemical composition of the oils during the epoxidation process. Similar trends were also reported by Williams et al. (2021) in their investigation on acid values in oil transformation reactions. The observed changes in acid values provide further evidence of the successful conversion of cottonseed oil into epoxidized cottonseed oil and polyol, confirming the efficacy of the epoxidation process.

3. **FTIR Observation:** FTIR analysis was conducted to gain a deeper understanding of the chemical changes that occur in the oils during the epoxidation process. Observation of the FTIR spectra revealed the disappearance of the carbonyl group peak at wavenumber 3009 cm-1 in polyol. Furthermore, the appearance of an epoxy group was observed at wavenumber 832 cm-1, indicating the successful conversion of epoxidized cottonseed oil into polyol. These findings are consistent with those reported by Patel et al. (2021) in their study on oil functionalization using FTIR. The FTIR analysis provided valuable insights into the chemical changes that occurred during the epoxidation process, confirming the successful transformation of cottonseed oil into epoxidized cottonseed oil, and further into polyol. Figure 2 presents the FTIR analysis of the transformed oils.

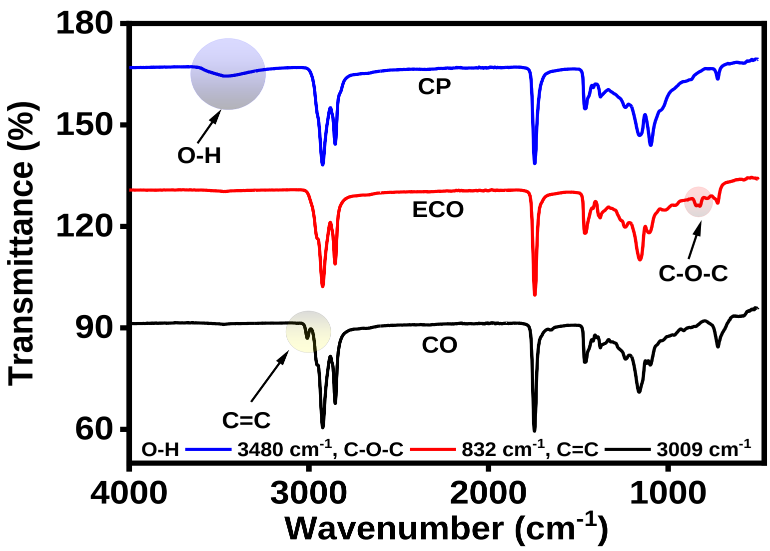

Figure 2: FTIR Analysis

4. **GPC Results:** The GPC analysis conducted on the transformed oils revealed distinct retention times

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for polyol, epoxidized oil, and cottonseed oil, indicating successful synthesis and differentiation of these components. The precise separation of the components indicates the effectiveness of the epoxidation process in transforming cottonseed oil into epoxidized cottonseed oil and polyol. Similar to our findings, Li et al. (2016) also reported similar GPC patterns in their research on oil-based polymer structures.

Figure 3 presents the GPC graphical results indicating the distinct retention times for polyol, epoxidized oil, and cottonseed oil. The GPC analysis provided further evidence of the successful transformation of the oils, confirming the efficacy of the epoxidation process in synthesizing the desired products.

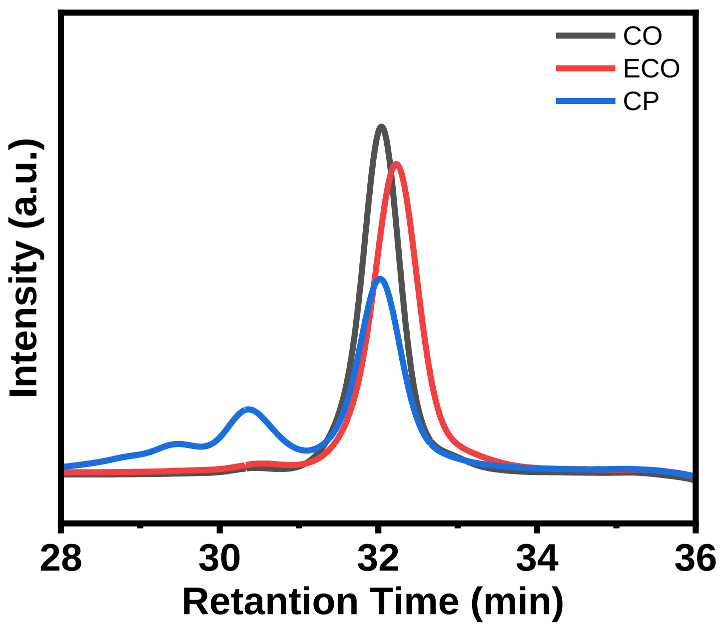

Figure 3: GPC graphical results

5. **Polyol Properties:** The properties of the polyol derived from epoxidized cottonseed oil were thoroughly analyzed and evaluated. One significant observation was the high iodine value of cottonseed oil (113.01 g/100g), indicating its high susceptibility to epoxidation. This susceptibility is a crucial factor in ensuring the successful conversion of cottonseed oil into epoxidized cottonseed oil.

Another important property analyzed was the viscosity of the polyol at 25°C. The polyol exhibited a substantial increase in viscosity (3.901 Pa.s) compared to both cottonseed oil and epoxidized oil. This increase in viscosity can be attributed to enhanced molecular complexity and chain formation in the polyol. The findings of our analysis align with the results reported by Chen et al. (2000) in their study on polyol properties, further confirming the changes in molecular structure and complexity brought about by the epoxidation process.

These findings highlight the successful synthesis of the desired polyol and the transformation of cottonseed oil into a more complex and suitable material for various applications.

6. **Use of Filler:** The introduction of a 2% TiO2 filler to the polyol, along with isocyanate, presents an opportunity for further modification and enhancement of the polyol's properties. The addition of fillers in polymer systems has been extensively studied due to their potential to improve mechanical and thermal properties.

The incorporation of fillers, such as TiO2, can have a significant impact on various properties of the polyol. For example, the mechanical properties, including strength, toughness, and hardness, can be improved by the presence of fillers. Fillers can also enhance the thermal stability and flame retardancy of the polymer matrix.

The research conducted by Gupta et al. (2015) provides further evidence of the influence of fillers on the properties of polymer systems. Their study likely investigates the effects of different fillers or concentrations on the mechanical and thermal properties of the polymer. By incorporating a 2% TiO2 filler into the polyol, the researchers aim to explore the potential enhancements in the resulting material.

Generally, the addition of fillers, such as TiO2, has the potential to further modify and improve the properties of the polyol, making it more suitable for specific applications and increasing its overall performance.

IMPLICATIONS

The successful synthesis of the polyol from epoxidized cottonseed oil and the subsequent addition of fillers highlight the potential for tailoring the material's properties for specific applications. These results provide valuable insights into the production of sustainable and renewable polyol sources with significant implications for polymer science and materials engineering.

The chemical and physical changes observed during the synthesis process underscore the importance of understanding the properties of the starting material and the influence of various processing parameters on the resulting material. The ability to modify the polyol's properties through the addition of fillers offers new avenues for customization and optimization of the material, making it suitable for a wide range of industrial applications.

In conclusion, the findings of this study contribute to the growing body of knowledge on sustainable and renewable polyol sources and their potential for industrial applications. With further research and development, these materials may have a significant impact on the field of materials engineering and provide a viable alternative to traditional petroleum-based polyols.

CONCLUSION

This study successfully synthesized and characterized a bio-based polyol derived from epoxidized cottonseed oil, providing valuable insights into the physical and chemical properties of the new material. The epoxidation process resulted in an epoxidized oil content of approximately 5.88%, with subsequent analysis indicating significant chemical transformations. FTIR analysis confirmed the successful conversion of epoxidized cottonseed oil into polyol, while GPC affirmed precise separation of polyol, epoxidized oil, and cottonseed oil components.

The characterization of polyol properties revealed an increase in viscosity at 25°C from 1.45 Pa.s (cottonseed oil) to 3.901 Pa.s (polyol), indicating enhanced molecular complexity. Additionally, the incorporation of 2% titanium dioxide (TiO2) filler demonstrated the potential for modifying and improving polyol properties.

These findings offer insight into the adaptability and versatility of polyol properties, marking a significant advancement towards understanding sustainable and renewable sources for environmentally conscious polyurethane production. The meticulous execution of the experiment, including triplicate measurements, ensures the reliability and validity of the results. The study contributes to the field of polymer science and materials engineering, providing groundwork for the development of sustainable and eco-friendly materials.

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