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## SYNTHESIS AND CHARACTERISATION OF BIODEGRADABLE RAPESEED OIL-BASED POLYURETHANE & POLYURETHANE/SiO<sub>2</sub> NANOCOMPOSITE FOR BIODIESEL PURIFICATION

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### ABSTRACT

This project describes the creation of rapeseed oil-based polyurethane and its nanocomposites utilising rapeseed oil and several isocyanates, particularly metaxylene di-isocyanate (MXD) and isophorone di-isocyanate (IPDI). This synthesis was performed in large amounts without the need of a catalyst by a singular reactive step. For polyurethane nanocomposites based on Metaxylene diisocyanate, Silicon dioxide nanoparticles were used and PU-SiO<sub>2</sub> Nanocomposites was Synthesized. The purification of the bio-diesel by Polyurethane and polyurethane Nanocomposites also performed in the research.

**Keywords:** • Rapeseed oil based Polyurethane • Metaxylene Diisocyanate • Isophorone Diisocyanate • Silicon dioxide (SiO<sub>2</sub>) Nano – particles • Nanocomposites • Bio-diesel Purification

### INTRODUCTION

#### NANOMATERIAL:

Traditional construction and structural materials, polymeric and composite materials are examples of common materials. It should be emphasised that a

nanoparticle, nanocrystal, or nanomaterial might have other properties as well outside its size. The majority of the atoms in many nanomaterials localise on the surface of a particle, as the authors believe, as opposed

to typical materials where atoms are scattered throughout the volume of a particle [1].

#### **NANOCOMPOSITES:**

The nanocomposite material exhibits the basic characteristics of non-metallic, metallic, and polymeric materials, which has the extra benefit of assisting in overcoming shortcomings and expressing some current features. Such a material symbolises the multiphase crossing of matrix and reinforcing elements. The continuous phase that is the matrix material is composed of metallic, inorganic non-metal, and polymeric substances. Future materials will likely be made of nanocomposites, which offer a fresh alternative to the current constraints of microcomposites and monolithics [2].

#### **POLYURETHANE/SIO<sub>2</sub>**

#### **NANOCOMPOSITES:**

Today, researchers are looking for PUs with improved performance to expand the application domains for PU. It is well known that polymer-based nanocomposites have significant advantages in physical, electromagnetic, thermal, photonic, and sonic properties when compared to pure biopolymers [3-8]. Additionally, significant research has been done on the interactions at the interface between fillers and polymer matrix. As a result, adding nanoparticles to PU is a viable strategy to enhance its qualities [9-12]. SiO<sub>2</sub> Nps have been

frequently incorporated into polymers during the last few years to enhance their mechanical, electrical, and radiation resistance, as well as their heat and radiation resistance [13, 14].

#### **EXPERIMENTAL SECTION**

#### **MATERIALS:**

The Rapeseed oil had a saponification value of 194.7 mg KOH/g and an acid value of 2.7 mg KOH/g. The Metaxylene diisocyanate (MXD) and isophorone diisocyanate (IPDI), which were used as received, were supplied from TCI. Mueller hinton agar was used for biodegradation was obtained from Laboratory. Acetone was purchased from Merck Chemical Co. Ad-Nanotechnologies delivered silicon dioxide (SiO<sub>2</sub>) nanoparticles with an average dimension of particles of 20 to 50 nm. Karanja oil was obtained from the local ayurvedic Shop. Methanol and ethyl acetate Was purchased from LOBA.

#### **METHOD:**

#### **Synthesis of Rapeseed oil-Based PU(Polyurethane):**

Prior to usage, weighed amounts of Rapeseed oil were dried with a water bath at 60 °C for 24 hours in a vacuum. By adding the di-isocyanate and rousing at 75 °C for 24 minutes in a dry nitrogen ambience, the dry Rapeseed oil's reaction was carried out. To finish forming the network, the bulk was then maintained in an oven at 80 C for 20 hours.

**Preparation of the polyurethane/SiO<sub>2</sub> Nanocomposites:**

SiO<sub>2</sub> nanoparticles were added in amounts of 0.3, 0.6 and 0.9 (w/w)% to PU Nano composites based on MXD with  $r = 1$ . A suspension of Silicon Dioxide in acetone was used to make polyurethane Nano composites. Acetone was used because to its low temperature of boiling (56 °C). The initial process involved vacuum-drying the raw Rapeseed oil at a temperature of 70°C. The acetone suspension was subjected to an ultrasonic bath treatment during this time to prevent the nano filler from clumping together.

The second stage involved vigorously swirling the suspension and Rapeseed oil for 15 minutes. The reaction mixture was then given the required amount of MXD, and it was agitated for an additional 15 minutes. After that, it was kept into hot air oven at 75°C for 22 hours.

**Synthesis of Biodiesel:**

Biodiesel is produced by mixing karanja oil and methanol. Then, put 50 ml of karanja oil in a dry RBF. Methanol, 14 ml, is then added. Following that, combine it while gradually adding 4 cc of sulfuric acid. For 24 hours, it was swirled at 60 °C. To remove glycerol and other impurities, water and ethyl acetate were utilised. Using sodium sulphate, the moisture was removed. To get rid of any colour impurities, activated charcoal was added to the mixture.

Application of Purification of Bio-Diesel: Take 2 ml of biodiesel precisely into a smaller RBF. To that, add 0.40 mg of polyurethane. At room temperature, it was stirred for an hour. Then used Watt Man filter paper to remove it. Consider the filter and record it on paper. This is the method used to purify biodiesel by polyurethane. The similar process should be followed to purify biodiesel utilizing SiO<sub>2</sub> nanocomposites.

**Fourier transform infrared spectroscopy (FTIR) analysis:**

The infrared spectrum of absorption in solid, liquid and gas can be obtained using the analysis method known as Fourier-transform infrared spectroscopy (FTIR). High-resolution spectral data are simultaneously run up over a wide spectrum range by an FTIR. That would appear from the requirement of a Fourier transform to turn the raw data into the real spectra (a mathematical procedure). FTIR uses an infrared absorption spectrum to examine chemical bonding in molecules. The sample's outline may be seen in the spectra, and we can filter and scan samples by using a common molecular fingerprint. The fundamental idea behind FTIR is that these frequencies can be compared to quantum physics' ground state. FTIR analysis of raw rapeseed oil, metaxylene diisocyanate (MXD), Polyurethane sample which was made by using the MXD, Isophorone

diisocyanate (IPDI), polyurethane sample which was made by using the IPDI, these samples were analysed using the FTIR machine from the CR4D of the Parul University.

### **<sup>1</sup>H NMR Analysis (Nuclear Magnetic Resonance):**

Proton nuclear magnetic resonance employs hydrogen-1 nuclei within a substance's molecules to analyse the structure of those molecules using nuclear magnetic resonance. When pure hydrogen (H) is used in samples, the isotope <sup>1</sup>H compensates nearly all of the hydrogen. Polyurethane which was made by using metaxylene

Diisocyanate, that sample was analysed by using this machine from the M. S. University.

### **RESULTS AND DISCUSSION:**

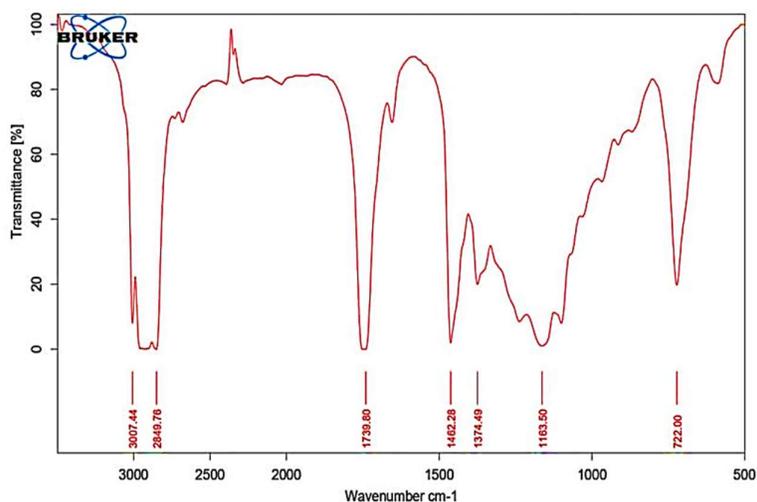
In this study, we used FTIR & NMR analysis to evaluate the polyurethane samples. The following reports all analysis findings.

#### **Characterisation:**

Here, the Characterisation of Polyurethane Samples with use of different diisocyanate is described with FTIR report and NMR report.

#### **Characterisation By FTIR Analysis:**

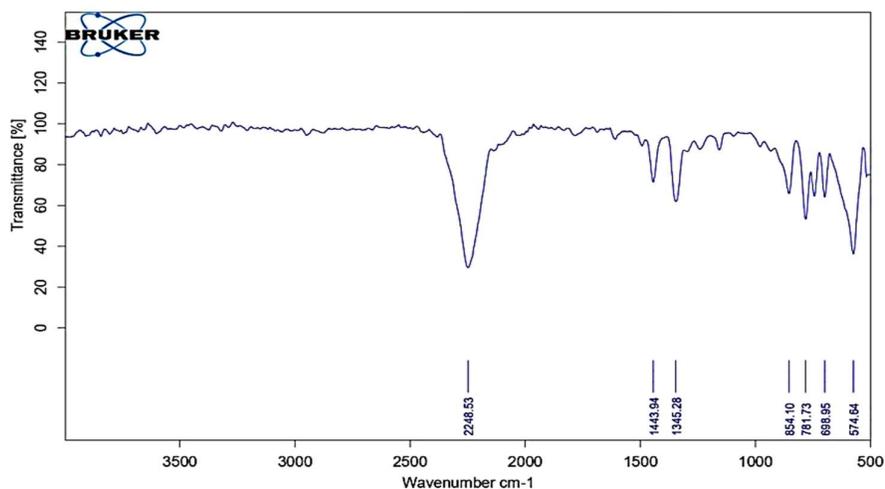
##### **FTIR of Raw Rapeseed Oil**



The trans C=C bond's C-H bending and the cis C=C bond's CH<sub>2</sub> bending were each attributed to the peak m at 1163 cm<sup>-1</sup>, while the extremely faint peak e, around 1739 cm<sup>-1</sup>

<sup>1</sup>, was attributed to the cis C=C elongation. In conclusion, a shoulder peak, at 722 cm<sup>-1</sup>, revealed the cis C=C bonds' CH bending.

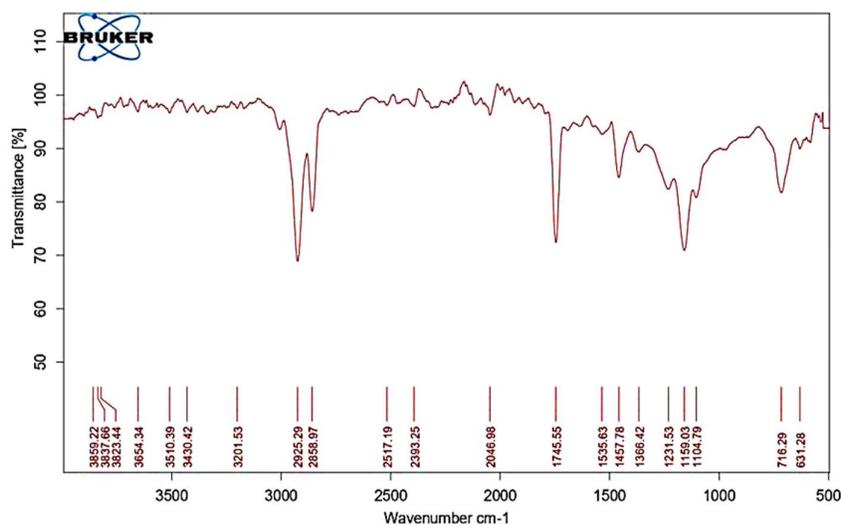
##### **FTIR of Metaxylene Diisocyanate**



The isocyanates group with a C=N stretch exhibits the peak between 2240 and 2275  $\text{cm}^{-1}$ . MXD diisocyanate, which ranges between 2248.53  $\text{cm}^{-1}$  and the observed peak, is located below it. The Amine group C = N stretch peak is located between the

1345.28  $\text{cm}^{-1}$  range. The range of the alkynes group is 800 to 860  $\text{cm}^{-1}$ , and the measured range is 854  $\text{cm}^{-1}$  with a C-H bend.

#### FTIR of Polyurethane Using MXD

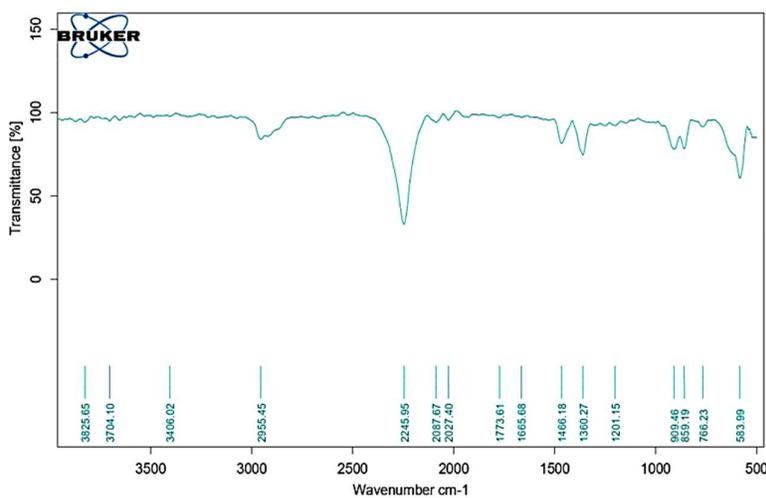


The unique carbonyl elongation at 1745  $\text{cm}^{-1}$ , which denotes the existence of a urethane linkage, was observed. The lack of the NCO bending and resonance peak at 2248  $\text{cm}^{-1}$

demonstrated that all of the isocyanate molecules had reacted with all of the OH groups during the synthesis polymerization step. The band observed at 2925 and 2858

$\text{cm}^{-1}$  indicated the presence of  $-\text{CH}_2$  and  $-\text{CH}$  elongation.

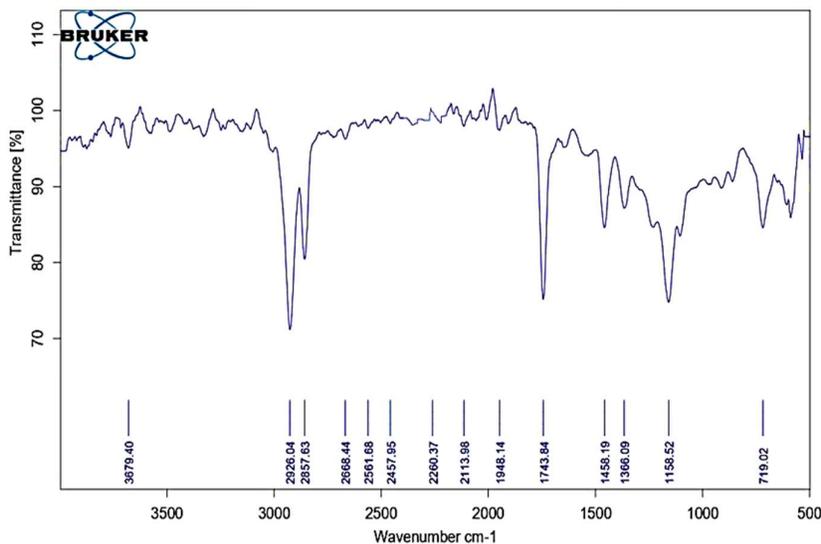
### FTIR of Isophorone Diisocyanate



Due to distinctive isocyanate ( $-\text{NCO}$ ) groups connected to the isophorone diisocyanate, FTIR spectra of isophorone diisocyanate (IPDI) reveal a prominent

signal. Due to IPDI's C-H stretching, the FT-IR spectra exhibits strong peaks at  $2245.95 \text{ cm}^{-1}$ .

### FTIR of Polyurethane Using IPDI



The distinctive carbonyl stretching led to the identification of the urethane link at  $1743 \text{ cm}^{-1}$ . Every isocyanate group had

interacted for every OH group throughout the synthesis polymerization phase, as evidenced by the lack of the NCO



Acid value of pure biodi-esel	Acid value of biodiesel After treatment of Rapeseed oil based polyurethane (MXD)	% Acid value of biodiesel After treatment of SiO <sub>2</sub> nanocomposite (MXD)	% of Acid value efficiency of Rapeseed oil-based polyurethane (MXD)	% of Acid value efficiency of SiO <sub>2</sub> nanocomposite (MXD)
46.25	30.85	31.87	33.29%	31.09%

## CONCLUSION:

Without using a catalyst, PU containing rapeseed oil as a polyol component was produced. A variety of SiO<sub>2</sub> nanoparticle contents 0.3, 0.6, and 0.9(w/w) % were used to create nanocomposites utilising aromatic diisocyanate or MXD. The application of biodiesel purification was also done by the proper method with the help of polyurethane Nanocomposites. After being purified by polyurethane and SiO<sub>2</sub> Nanocomposites, biodiesel has an efficiency of 33.29% & 31.09%, respectively. Therefore, the Purification of Bio-diesel Mentioned Above Can Used to Perform.

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