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Characteristic Studies on Novel Biodegradable Polyurethane Thin Films from Soyabean Oil.

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Abstract

Novel bio-polyurethane thin film have been synthesised from epoxidised soyabean oil, ethylene glycol and isophorone diisocyanate. The biodegradability of the polyurethane film was studied by soil burial test . The chemical resistance of the polyurethane was analysed with different solvents. The polyurethane was characterised with respect to their mechanical properties such as hardness, tensile strength, percentage of elongation and young's modulus. These studies revealed that the rigid polyurethane possess good tensile strength and biodegradability. The characteristic studies such as FTIR and NMR were carried out to identify the nature of the polymer formed.

Keywords: Isophorone diisocyanate, young's modulus , elasticity, rigid polyurethane , tensile strength.

1. Introduction

Soyabean seed contains 18-19 % oil. To extract soyabean oil from seed, the soyabeans are cracked adjusted for moisture content, rolled into flakes and solvent extracted with commercial hexane. The oil is then refined , blended for different applications. Soyabean oil is partially hydrogenated and sold as vegetable oil in a wide variety of processed foods. [1]

Vegetable oils are composed of triglycerides of long chain fatty acids. The most common chain lengths in these fatty acids are 18 (or) 20 carbon atoms which can be saturated (or) unsaturated, where unsaturation of the double bonds are located at 9,12 and 15 carbon. Polyurethanes produced from vegetable oils are rigid and used as foams in different fields. [2] [3] [4]. Since, Bayer synthesised polyurethanes in 1937, its usage has become omnipresent in the biomedical field [5] [6].

Polyurethanes are first synthesised from polyol and diisocyanate by Otto Bayer in the year 1940. Polyurethanes are most versatile polymers due to the flexibility of structure . They are mainly used in rigid and flexible foams, coatings, adhesives, sealants, elastomers and binders [7].

2. Materials and Methods:

The Soyabean oil was purchased from local market, Hydrogen peroxide (30%), (Rankem), Isophorone diisocyanate (99%) (Sigma-Aldrich), Ethyleneglycol (Rankem), Glacial acetic acid (Rankem).

2.1 Method

Synthesis of Epoxidised soyabean oil:

Soyabean oil was purchased from the local market. 100g of soyabean oil was taken in a three necked flask fitted with condenser and thermometer, hydrogen peroxide was added in dropwise for two hours. The setup was kept for ten hours at 60 ^{0} C. Finally the epoxidised resin was taken out and washed with warm water and dried at 60 ^{0} C for half an hour in hot air oven.

Synthesis of soyabean polyol:

The epoxidised resin was taken in a three necked flask and ethylene glycol was added in 1:2 ratio. The set up was kept at 250 ^{0} C for five hours. After five hours a brown viscous liquid formed and it indicate the formation of polyol.

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Manufacture of polyurethane film from soyabean polyol and isophorone diisocyanate:

Polyurethane thin film was manufactured by using isophorone diisocyanate and soyabean polyol at different concentration at 60 ^{0} C. The polyurethane film was removed from the mould after 48 hours. Polyurethane thin film at 1:2, 1:3 and 1:4 were synthesised from polyol and ISPDI. Polyurethane film of 0.1mm thickness is synthesised by this method.

3. Result and Discussion:

3.1 FTIR spectral analysis

The FTIR spectrum of soyabean oil, resin and polyol are recorded between 500 to 4500 cm^{-1}. The Infrared spectrum of soyabean oil showed a strong absorption band at 3009 cm^{-1} due to the presence of non conjugated linoleic acid. A pair of peaks observed at 2924 cm^{-1} and 2852 cm^{-1} is due to the symmetric stretching- vibration of the aliphatic – CH_2 groups. A strong and sharp band at 1743 $cm^{-1}$is due to the ester carbonyl group.[8]

The absorption band at 1462 cm^{-1}revealed the presence of C-H bending of unsaturated methylene groups. The disappearance of 3009 cm^{-1} band in epoxidised soyabean oil shows (-C=C-) bond. The appearance of band at 922 $cm^{-1}$is due to the presence of epoxy group confirmed the epoxidation process. The FTIR spectrum of soyabean polyol indicate the absorption band at 3401 cm^{-1} attribute to hydroxyl group indicate the opening of epoxide ring.

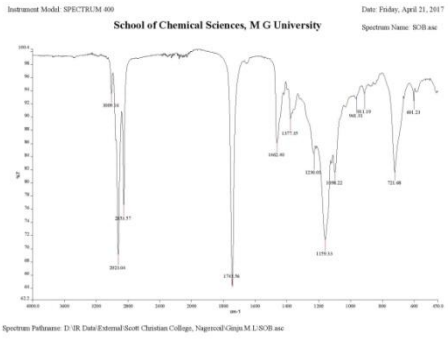

Figure.1. FTIR spectrum of soyabean oil.

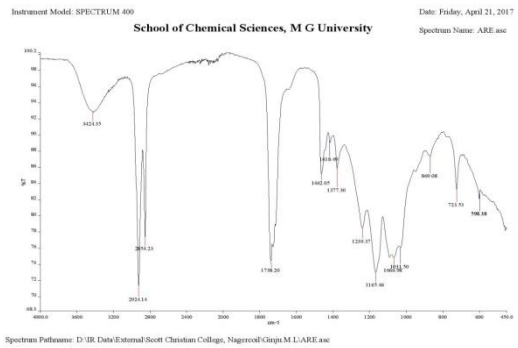

Figure.2. FTIR spectrum of epoxidised soyabean oil.

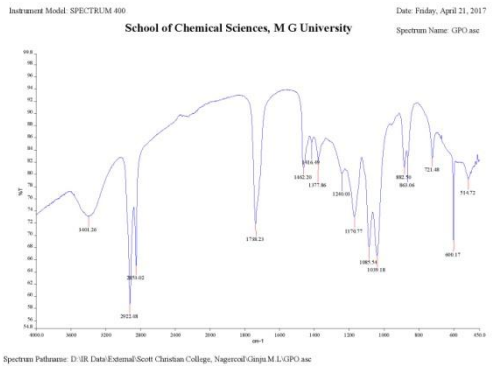

Figure.3. FTIR spectrum of soyabean polyol.

3.2 H^1 NMR spectral studies:

In this study the NMR spectrum of soyabean oil the peak at 4.3 to 5.4 ppm shows the presence of methylene proton. The peak at 2.8 to 2.77 ppm indicates the presence of hydrogen in ethylenic bond. The peak at 5.2 to 5.4 ppm shows the olefinic proton present in soyabean oil. The chemical shift at 4.2 to 3.8 ppm shows methylene protons of ester associated with triglyceride molecule. [10] [11]

In the FTIR spectrum of epoxy resin shows the presence of epoxy linkage at 6.7 ppm. The peak appeared at 5.1 ppm in soyabean polyol shows the presence of hydroxyl group. Thus the formation of polyol confirmed from these studies.The chemical shift at lower region from 5.3 to 0.8 ppm support oleic, lonoleic and saturated fatty acids respectively. [12]

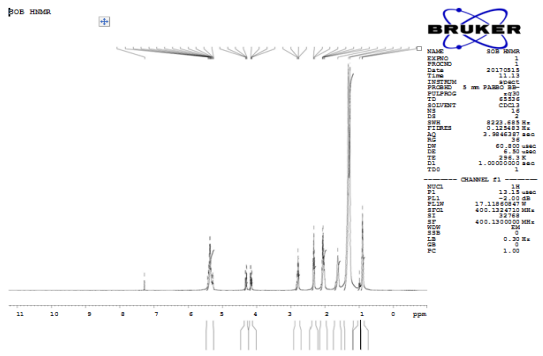

Figure.4. H^1 NMR spectrum of soyabean oil.

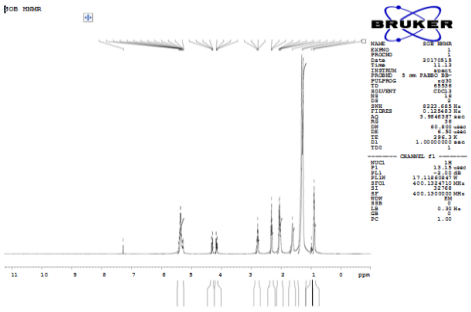

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Figure.5 H^1 NMR spectrum of epoxidised soyabean oil.

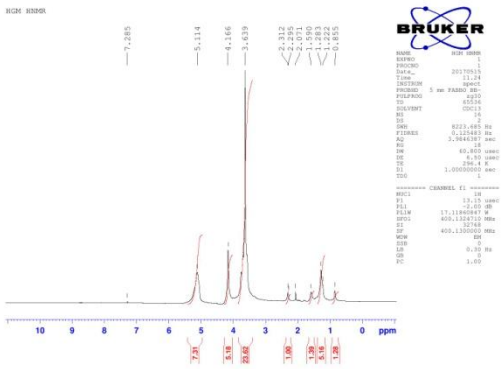

Figure.6. FTIR spectrum of soyabean polyol.

3.3. Mechanical properties:

The mechanical properties of polyurethane film are analysed. The tensile strength, percentage of elongation and young's modulus is evaluated from the table.1.

Table. 1. Mechanical properties of Polyurethane thin film.

Serial number	Polyurethane (NCO/OH)	Hardness	Tensile strength	Elongation (%)	Young's modulus
1.	PU (1:2)	70.8	1.51	11.83	0.075
2.	PU (1:3)	71.4	2.75	15.84	0.081
3.	PU (1:4)	9.8	2.89	24.7	0.09

The polyurethane film of different concentration is synthesised by varying with (NCO/OH) . They possess good tensile strength and strength increases with increase in concentration. This is due to high crosslink density of the rigid polyurethane. In the present work, the hardness of the polyurethane film increases with increase in concentration. This is due to the fact that as the chain length decreases the cross link density increases which results into more rigid network with better mechanical properties. The percentage of elongation is increases with increase in concentration . The increase in elongation is due to the increase in length of soft segment that imparts flexibility.[13]

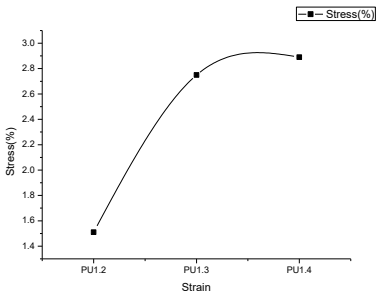

Figure. 7. Tensile strength

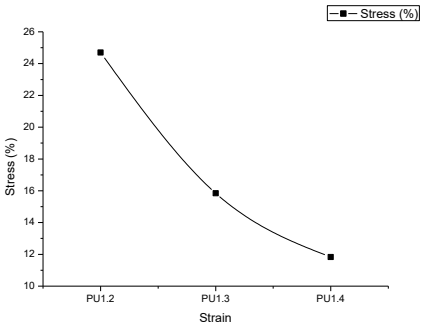

Figure. 8. Elasticity Properties

The tensile strength of polyurethane based on IPDI possess better mechanical properties than those of the HDI based polyurethane. This may be due to the fact that the aliphatic HDI is flexible and symmetric open chain structure with two primary isocyanate groups whereas the cyclo aliphatic IPDI possess unsymmetric structure in which one of the isocyanate group is secondary in nature. [15]

3.4. Chemical Resistance

The thin film of polyurethane is tested under different solvents at regular interval of time. The polar solvents such as sodium hydroxide (2%), sodium chloride (20 %) and sulphuric acid used for swelling analysis. The polyurethane film was unaffected with water while slightly soluble in 20 % sodium chloride and 0.1N sulphuric acid. Polyurethane possess greater effect in 2% sodium hydroxide.

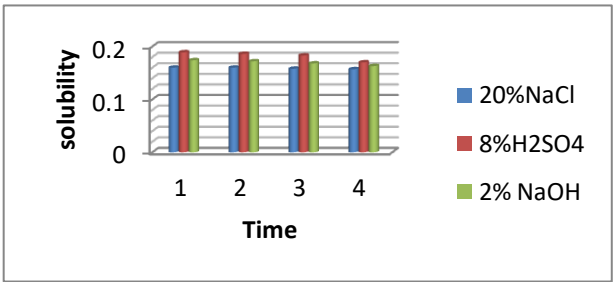

Figure.9. Swelling of polyurethane against polar solvents.

The degree of swelling and sol content is analysed using ether, chloroform, acetone, ethanol, toluene and DMSO. The swelling percentage was calculated by using the formulae,

Swelling percentage= $[(Ws - W_0)/W_0] \times 100$.

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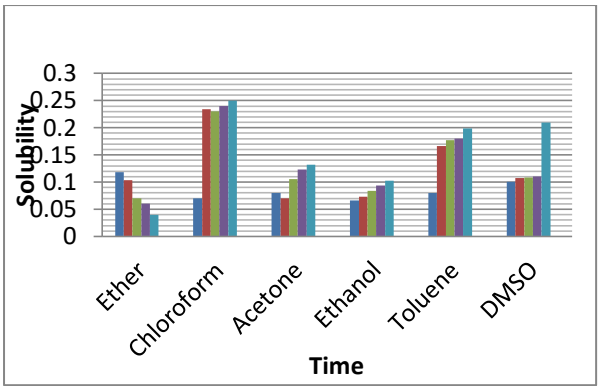

Figure.10. Swelling behaviour of polyurethane against organic solvents

The polyurethane showed good resistance towards acids, alkalies and solvents. It is also found to be the alkali resistance of samples is more than acid resistance. [14]

3.5. Soil burial test

Soil burial test helps to identify the environmental resistance of the polymer. The polyurethane film of 1cm x 2cm dimension is buried into the soil and water is sprayed over the soil for bacterial attack. The weight loss of the polymer was calculated after 10 days. Similarly the weight loss is taken out between four weeks. After 72 days the polymer film is removed from the soil. Then the percentage of weight loss is calculated by using the formulae.

Percentage of weight loss = W_0-Ws / W_0 x 100

Where, Wo = Initial weight of the polymer, Ws = weight of the polymer after 10 days.

Sem analysis is carried out to find out the biodegradability of the polymer

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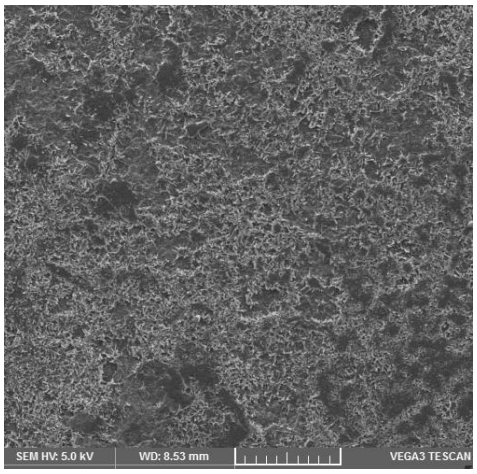

Figure.11.SEM image of Polyurethane

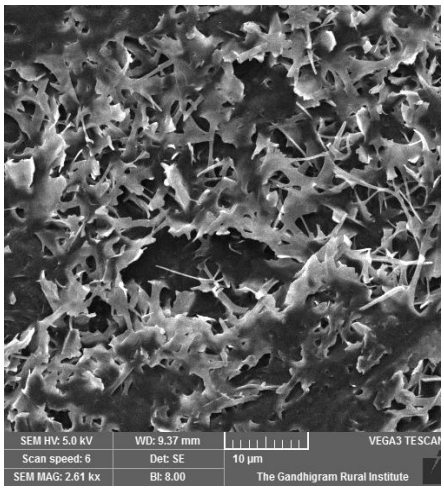

Figure.12.SEM image of Polyurethane before soil burial test after soil burial test.

4. Conclusion

The soyabean oil consist of unsaturated fatty acids such as oleic acid and linoleic acid. The characteristic studies such as FTIR and NMR shows the formation of intermediate products at different stages. The swelling analysis is carried out for polar and organic solvents at different time intervals. The polymer is unaffected by polar solvents and slightly affected by organic solvents. The biodegradability is confirmed by soil burial test. The percentage of weight loss is decreased with different time intervals. The polyurethane film possess good tensile strength and hardness. The tensile strength increases with increase in concentration. The hardness of the polymer is increases with increase in concentration. The conclusion made on these studies are the synthesised polyurethane film is biodegradable and possess good mechanical strength and hardness. The rigid polyurethane is more stable than tough polyurethane.

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