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Synthesis and Characterization of Olive Oil based Biodegradable Polymers

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Abstract: Similar to olive oil, plant oil is a fat extracted from the fruit of the olea europaco family oleaceae, a typical Mediterranean crop. It is used to make polymeric materials. These are derived from naturally occurring renewable resources. The purpose of this work was to determine the composition and characteristics of a biodegradable polymer derived from a non-volatile oil such as olive oil. The influence of olive oil-based acrylated epoxidized resin on monomers such as methyl methacrylate and vinyl acetate was investigated in this study using the thermal polycondensation process with the inclusion of a catalyst. Additionally, solubility tests, TG-DTA (Thermo Gravimetric – Differential Thermal Examination) and mechanical analysis, SEM analysis, and chemical investigations were performed on newly produced copolymers. These polymers displayed a broad variety of mechanical and degrading properties that may be tailored by monomer selection.

Keywords: Biodegradation, Epoxidization, Methyl methacrylate (MMA), Vinyl acetate (VA), Olive Oil.

1. INTRODUCTION

Vegetable oils are fatty acid triglycerides. Olive oil has a variety of advantageous qualities that enable it to be used in the production of important polymeric materials such as epoxy, polyester amides alkyds, and polyurethane, in addition to its many other uses. 1,2,3 The acrylated epoxidized resins were made mostly from olive oil. The manufacturing technique that incorporates stoned olives seems to be a technical advancement in the industrial production of olive oil. 4,5 Several studies have shown the advantages of the destoning method for phenolic compounds. 6

Nowadays, there is a rising interest in biopolymer production. Biopolymers derived from oil offer a number of benefits over polymers derived from petroleum monomers. Recently, bio-based thermosetting polymers derived from vegetable oils, such as epoxy olive oil and epoxy sunflower oil, have been produced in many formulations. These polymers include a functional epoxie group that may be reacted with appropriate curing agents to form an elastomeric network. 7 Epoxidized flaxseed oil and its derivatives have shown promise as reactive resins due to the oil's unsaturation, which may be chemically changed by simple processes.

2. EXPERIMENTAL

Materials Used

Olive oil that is commercially accessible was utilised.

Hydrogen peroxide (H2O2), glacial acetic acid, sulphuric acid, acrylic acid, triethyl amine, and benzene were all utilised as compounds.

Sigma-Aldrich Chemical Company provided the monomers methyl methacrylate and vinyl acetate.

3. RESULT AND DISCUSSION

Polymer Preparation

Olive oil was epoxidized with glacial acetic acid and H2O2 in a 250 ml round bottomed flask for 12 hours at 80°C. Separating the mixture using a separating funnel and collecting the epoxy resin in a beaker.

After epoxidation, the olive oil was acrylated using acrylic acid. The catalyst was triethylamine, while the solvent was benzene. The reaction mixture was then refluxed for about 20 minutes at 80°C–100°C with steady agitation in a nitrogen environment and collected in the beaker to yield acrylated epoxidized olive oil (AEOO). The olive oil acrylated epoxidized resin was co-polymerized with co-monomers such as Methyl methacrylate (MMA) and Vinyl acetate [VA]. The initiator was benzoyl peroxide, while the accelerator was N,N1 dimethyl aniline. The slurry was cast onto a clean silicon oil-spread glass plate and cured at 100oC for 1 hour. All cured materials demonstrated a high degree of toughness, elastometry, and transparency. The acrylated epoxidized olive oil (AEOO) was made as illustrated in Figure 1.

Figure 1: Synthesis of Acrylated epoxidized olive oil resin

Thermal analysis

Thermal studies of the polymers was determined by TG-DTA analysis. Thermo gravimetric analysis (TGA) was performed by perkins elimer thermo gravimetric analyzer over the temperature ranging from 30oC to 700oC at a heating rate of 10oC/min under nitrogen gas atmosphere.

Thermogravimetric analysis (TGA) was utilised to determine the polymers' heat stability. The TG-DTA curves of OLIAEMMA and OLIAEVA in Figures 2 and 3 illustrate the breakdown behaviour of the polymers samples in a nitrogen environment. The bulk polymer is thermally stable below 100oC and decomposes between 100oC and 250oC. Stage [1], stage [2] between 250oC and 450oC, and stage [3] over 450oC. The bulk polymer's initial breakdown stage [stage 1] is mostly due to evaporation and decomposition of unreacted free oil. At a heating rate of 20oC/min, the decomposition temperature of the strongly crosslinked polymer exceeds 400oC, which is about the temperature at which the bulk polymer decomposes in stage 2. This procedure results in the degradation and development of char on the cross linking polymer network. Above 460oC, the char residues progressively oxidise and release oxygen into the air. As a result, the final temperature region is same for all polymers.

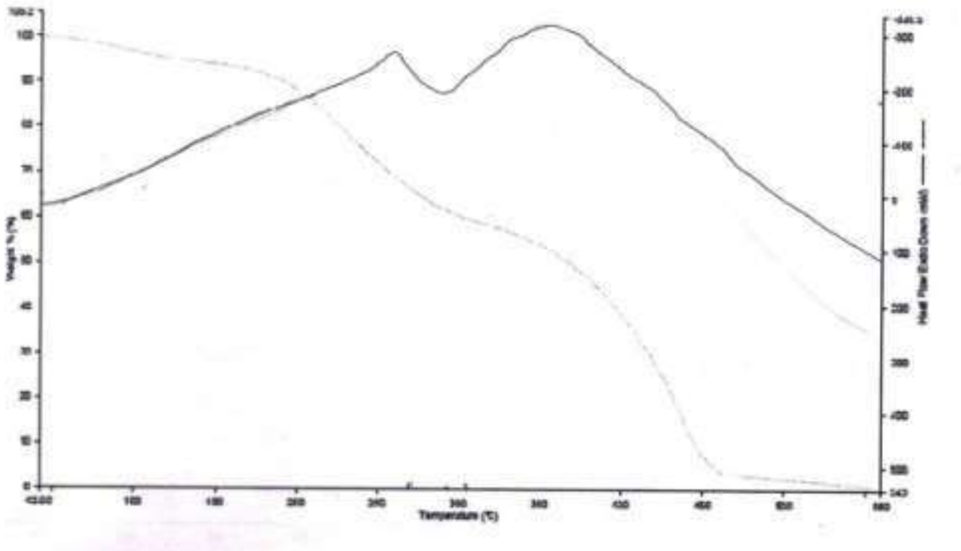

Figure 2: TG-DTA curve of olive oil methyl methacrylate

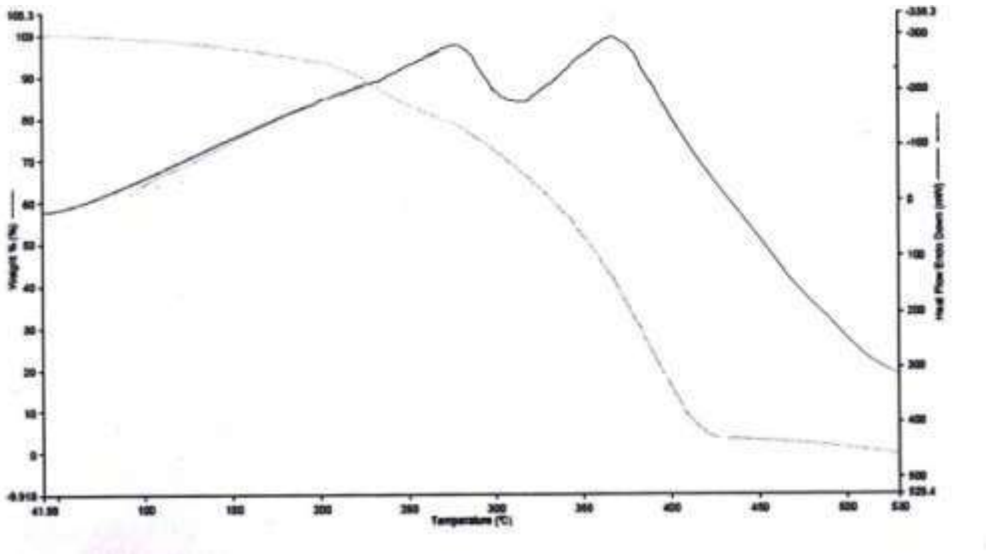

Figure 3: TG-DTA curve of olive oil vinyl acetate

The DTA curve of an OLIAEMMA polymer sample exhibits an endothermic peak about 290°C, which corresponds to the polymer network's melting point, and two exotherms. The first exotherm occurs at around 260°C and is caused by the cleavage of a long alkyl side chain. The second exotherm in the sample occurs strongly at 370°C and corresponds to the breakdown and synthesis of char from the cross linking polymer network.

Similarly, the DTA curve of the OLIAEVA polymer sample exhibits an endothermic peak at 300°C, which corresponds to the polymer network's melting point, and two exotherms at 260°C and 370°C. When compared to DTA curves, OLIAEVA polymer had a slightly higher melting point than OLIAEMMA polymer.

Mechanical Studies

Tensile strength of the produced polymers was evaluated using a dumb-bell-shaped cut from the specimen using an Instron UTM, a ten-ton static universal testing machine. The valves depicted were the mean of around three to four samples. In contrast to plastics, the novel polymeric material displayed tensile stress strain behaviour. According to the rubber elasticity theory E1=3veRT, the cross link densities re were estimated from the rubbery modulus plateau. Where E1 denotes the number of cross-linked co-polymers in the plateau area with a storage modulus of 1. The universal constant (8.314 I-mol-1.k-1) is denoted by R, while the absolute temperature is denoted by T.

Table 1 summarises the mechanical parameters of thermosetting polymers, including tensile strength, percentage of elongation, Youngs modulus, and shore 'D' hardness

.

Table 1: Mechanical Properties of polymers

Polymer Sample	Cross link density ($X10^{-3}$)	Mol. Wt. between cross links (mol^{-1})	Tensile Strength $\times 10^5$Pa	% of elongation	Young's Modulus $\times 10^5$Pa	Shore D hardness
OLIAE50MMA50	1.15	867	3.10	0.62	80	52.5
OLIAE75MMA25	1.01	988	3.25	0.67	99	56.4
OLIAE25MMA75	1.23	812	3.01	0.60	103	57.2
OLIAE50VA50	3.72	269	9.29	2.51	258	59.3
OLIAE75VA25	3.63	275	9.42	2.67	244	58.2
OLIAE25VA75	3.73	268	9.12	2.43	267	58.7

In the preceding Table 1, polymer samples such as MMA and VA are listed at various concentrations.

The findings indicate that the polymer samples created from these polymers have a high tensile strength and a high young's modulus, but the polymer samples made from OLIAEMMA have a low tensile strength and a low young's modulus. The tensile strength and youthful modulus of elasticity of polymers rise as the cross link density increases.

Biodegradation – Soil burial Tests

We explored the biodegradation of polymers using a soil burial approach. For the soil burial test, copies of the sample (5x3cm) were buried 30cm below the ground surface in garden soil and injected with sewage sludge capable of sticking to and degrading the polymer film for three months. 10 The test specimen was obtained from the soil on a regular basis and meticulously cleaned to eliminate any related debris or dust before being vacuum dried. The connection was used to evaluate weight reduction after 30 and 60 days. 11

Degree of biodegradation, D = Wo−Wt × 100

Wo

Where, how much weight does the original film have?

Wt - weight of leftover film after various times of deterioration.

Table 2 summarises the biodegradation of polymer samples generated by free radical copolymerization of olive oil acrylated epoxidized resin with varied concentration monomers such as MMA and VA.

Table 2: Percentage of Biodegradation of polymer sample from olive oil

Polymer Sample	Degree of Biodegradation %	
	30 days	60 days
OLIAEMMA	10.8	26.48
OLIAEVA	3.7	8.37

The findings obtained from the soil burial test indicate that the amount of biodegradation rises as the monomer concentration drops. According to this research, biodegradation is rapid in OLIAEMMA and slow in OLIAEVA polymer.

SEM Analysis

SEM is commonly used for studying both the surface morphology, and cellular response of bio materials. Figure 4 shows the SEM micrographs of the polymer OLIAEMMA, OLIAEVA.

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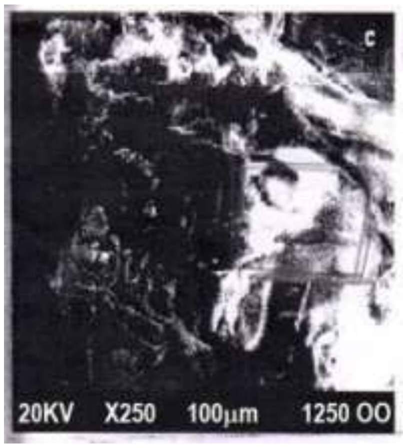
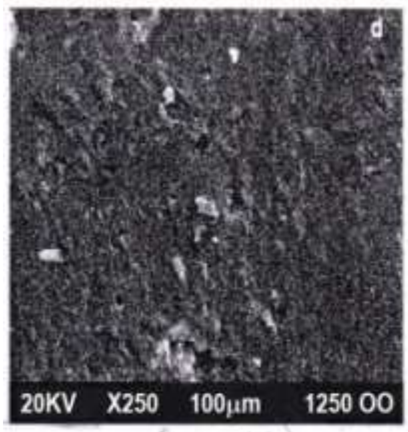
SEM Micrograph of OLIAEMMA (Before)



SEM Micrograph of OLIAEMMA (After)

SEM Micrograph of OLIAEVA (Before)



SEM Micrograph of OLIAEVA (After)

Figure 4: SEM Micrographs of Polymers before and after soil burial test

Chemical Resistance

Chemical resistance of freshly synthesised polymer samples was investigated by immersing them in different solvents such as CCl4, CHCl3 diethyl ether, toluene, and DMSO for one week and four weeks and evaluating the dimensional changes. Weight loss was also determined after 45 days.

The degree to which polymers are attacked by chemicals is governed by a variety of characteristics, both chemical and polymer-specific. Chemical resistance of freshly

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manufactured polymer samples was investigated by immersing them in different solvents such as CCl4, Toluene, CHCl3, diethyl ether, and DMSO for one week and four weeks and evaluating the dimensional changes. Weight loss was also determined after 45 days. All polymeric samples generated are quite stable, however owing to their fragility, they degrade somewhat in Toluene, CHCl3, diethyl ether, DMSO, and CCl4 (Table 3). Polymer samples exhibit increased chemical resistance and are biodegradable.

Table 3: Weight loss of polymers in various solvents

Solvents	Weight loss % in 45 days	
	OLIAEMMA	OLIAEVA
Toluene	6.3	3.4
Diethyl ether	4.5	3.7
CCl_4	6.3	4.5
$CHCl_3$	6.2	4.1
DMSO	7.2	3.5

Figure 5 compares the stability of the polymers OLIAEMMA and OLIAEVA to a variety of chemicals. According to the research, olive oil polymers (OLIAEMMA) have a lower chemical resistance than the other polymer, OLIAEVA.

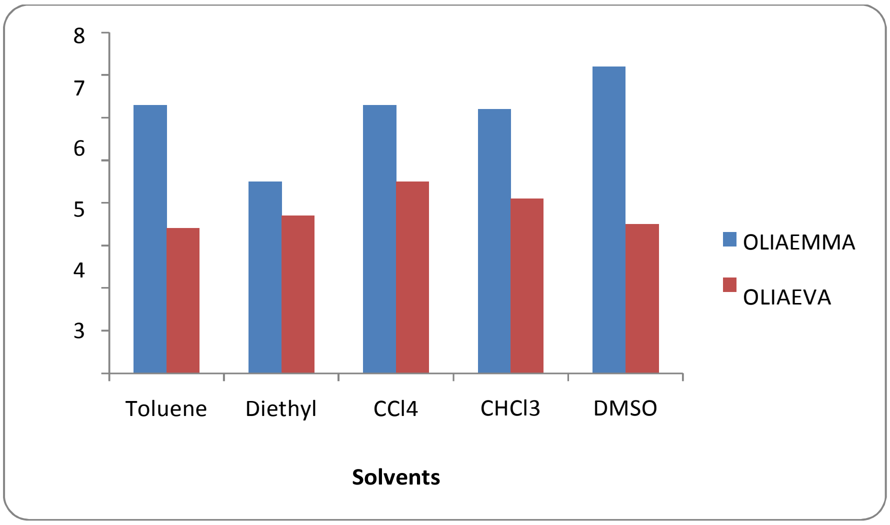

Figure 5: Chemical resistance of polymers in various chemicals

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CONCLUSION

Edible oils originating from plants, such as olive oil, that have been employed in the manufacture of acrylated epoxidized resin. These resins are used to create a broad variety of polymers with varying mechanical characteristics. These resins are very durable and robust.

The newly synthesised polymeric samples, such as OLIAEVA, have a high tensile strength and a low modulus in comparison to conventional thermosetting polymers. This demonstrates that the polymer samples are very rigid and flexible, similar to plastics. The low tensile strength and low young modulus of samples such as OLIAEMMA show that the sample is soft and rubbery. The soil burial test indicates that the polymer samples created are only slightly biodegradable and are very stable, although they degrade in chemicals such as toluene, CHCl, CCl, dietherether, and DMSO, indicating that the polymer samples exhibit a high level of chemical resistance.

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