

Preparation and Characterization of a Novel Biodegradable Epoxy Resin Modified with Epoxidized Oleic Acid

F. Ravari*, M. Shoghi Toosi, Z. Gordi, R. Rahbarian and S.M. Fazeli

Chemistry Department, Payame Noor University, 19395-4697 Tehran, I. R. of Iran

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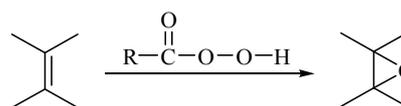
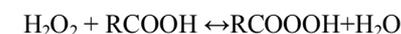
The goal of this research was to study the curing behavior and biodegradability of DER 736 modified with epoxidized oleic acid. DER 736 epoxy resin is a flexible, low viscosity, and light color resin for use in coatings and adhesives. This epoxy resin is a liquid reaction product of epichlorohydrin and dipropylene glycol. In this paper, the efficient epoxidation of oleic acid with performic acid generated *in situ* from formic acid and hydrogen peroxide was demonstrated in the presence of H₂SO₄ as catalyst. The highest relative epoxy yield of 61% was achieved at 40 °C after 10 h. DER 736 modified with epoxidized oleic acid was thermally cured using succinic anhydride as curing agent, in the presence of tri ethyl amine. Also, degradation of composite was studied in the presence of three different loadings of epoxidized oleic acid (0, 20 and 40 wt%) with lipase from porcine pancreas in phosphate buffer. It was found that this agent caused reducing the weight of the samples in 45 days. SEM studies also revealed higher surface erosion phenomenon and structural change of the matrix with increasing epoxidized oleic acid.

Keywords: Biodegradation, DER 736, Epoxidized oleic acid, Epoxy resin, Scanning electron microscope, Succinic anhydride

INTRODUCTION

Epoxidation of oils is a commercially important reaction, since the epoxides have been commercially used as plasticizers and stabilizers in polyvinyl chloride [1,2], lubricants, components in thermosetting resins and composites. Traditionally, these are used in the painting and coating process [3]. Furthermore, epoxides are used as intermediates for alcohols and alkanol amines because of the high reactivity of epoxide ring. Epoxidation is the addition of oxygen to double bond to give epoxide ring (Scheme 1). On the industrial scale, the epoxidation of oil is carried out with percarboxylic acids, such as peracetic acid and performic acid, obtained through the respective organic acid with hydrogen peroxide [4].

Epoxy resins are fragile non-biodegradable materials; therefore, their utilization has been limited. So, addition of some epoxidized oil can improve the toughness and



Scheme 1. Epoxidation reaction

resistance of epoxy resin and increases biodegradability of resin. In recent years, the utilization of hardeners increased to promote the crosslinking of polymer and therefore material with higher heat, chemical and mechanical resistance obtained. Degree of curing has effect on the physical, mechanical and electrical properties of epoxy resin.

Epoxy resins are cured using a wide variety of curing agents as aliphatic and aromatic amines and anhydrides, polyamides, phenol formaldehyde resins, and polysulfides *via* curing reactions [5-10]. Properties of the cured polymer depend on the hardener/epoxy molar ratio, type of epoxy resin and curing agent [11-12]. Recently, anhydrides have

*Corresponding author. E-mail: Fatemeravari@yahoo.com

been reported to be effective hardener and can be used as toughening agent to improve epoxy resin [13-14]. Anhydride cured epoxies provide a lower exotherm during cure and show excellent electrical properties, good electrical insulation and good physical properties and chemical resistance. Usually, curing with anhydride curing agent is catalyzed by amine.

In recent years, biodegradable resins have attracted considerable attention due to the environmental issues. A large number of polymers have been used in biomedical applications such as polylactic acid, nanoclay and polycaprolactone [15-20]. Enzymatic degradation of epoxy resin have been less investigated. Moeseret *et al.* [21] investigated the biological degradation of epoxy resin with microorganisms.

In the present research, we study the effect of epoxidized oleic acid on novel composite. Herein, novel composite based on DER 736 modified with different amounts of epoxidized oleic acid have been synthesized. The composite was cured with succinic anhydride in the presence of triethylamine. The mechanical properties and water uptake of the cured composite were investigated. Also, the biodegradability of the epoxy composite was evaluated by the pig pancreatic lipase.

EXPERIMENTAL

Materials

DER 736 and pig pancreatic lipase were purchased from Sigma-Aldrich. Glacial acetic acid, formic acid, diethyl ether, aqueous hydrogen peroxide (30%), chlorobenzene (99.8%), H₂SO₄, oleic acid, 0.1 N hydrogen bromide and chloroform were procured from Merck.

Characterization Techniques

IR was obtained on a Shimadzu model 408 FT-IR spectrophotometer in 400-4000 cm⁻¹ range using KBr pellet technique.

Analytical techniques. The percentage of oxirane oxygen was determined by titration method with hydrobromide acid solution in acetic acid. About 0.5 g of the epoxidized oleic acid dissolved in 10 ml of chlorobenzene and then titrated with 0.1 N hydrogen bromide in the presence of violet crystal. Epoxy content

was calculated following Eq. (1) [22,23]:

$$\%Epoxy = \frac{V \times N}{n} \times 100 \quad (1)$$

N, V and n are normality of HBr in acetic acid, HBr solution volume used for titration of the sample (ml), and the number of double bond (mmol), respectively.

Morphological studies. Surface morphology of the samples before and after immersion in the presence of enzyme and without it was studied by the scanning electron microscope (SEM, LEO 1450VP).

Mechanical properties. Mechanical properties including modulus, tensile strength and elongation at break were determined with Hounsfield model H50KS at a strain rate of 8 mm min⁻¹. Mitutoyo micrometer with 0.001 mm resolution was used to determine the film thickness.

Water uptake testing. Water uptake values of the films were determined according to ASTM D 570-98. The water uptake percentage (WU%) value was calculated from the following Eq. (2) [24-26]:

$$(\%)WU = \frac{W_t - W_o}{W_o} \times 100 \quad (2)$$

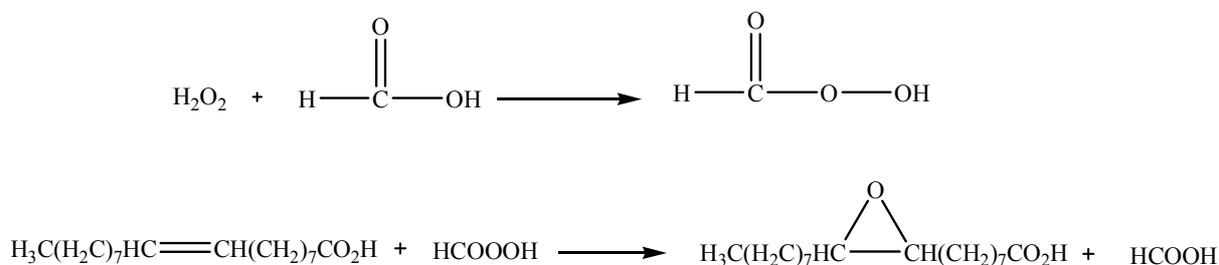
where, W₀ and W_t are the weights of the cured films before and after immersion, respectively.

Epoxidation Procedure

The epoxidation of oleic acid was carried out with performic acid formed *in situ* by reacting H₂O₂ and formic acid in the presence of H₂SO₄ as catalyst. Oleic acid (5 g) and formic acid (0.698 g) were added to a round bottom flask in a water bath equipped with magnetic stirrer, thermometer and reflux condenser. After 30 min, 0.025 ml hydrogen peroxide 30% (w/v) was added dropwise. The reaction was performed at different temperatures (40, 45, 50, 55 and 60 °C) and desired time duration (6, 8, 10, 12 and 14 h). At the end of the required reaction time, the crude product was filtered and washed with distilled water for three times until pH of 7.0 was obtained. Then, diethyl ether was added to the oil phase and was kept in the oven for 12 h at 35 °C. All samples were analyzed for epoxy oxygen content.



Fig. 1. Synthesized epoxy coatings. The sample contains 40% oleic acid that was used for enzymatic degradation.



Scheme 2. Epoxidation of oleic acid

Curing of DER 736 Modified Epoxidized Oleic Acid with Succinic Anhydride

The epoxidized oleic acid (1 eq) was mixed with an epoxy DER 736 (1 eq) in the teflon mold. Afterward, succinic anhydride (2 eq) as curing agent and triethyl amine as initiator were added, and the mixture was heated for 100 min at 130 °C temperature in vacuum oven, and then for 2:30 h at 70 °C. Synthesized epoxy coating was removed from the mold after cooling. Figure 1 shows the sample containing 40% oleic acid used for enzymatic degradation.

Biodegradation of Epoxy Resin

This section accomplished according to previous study [27]. The cured resin films were cut into 10 × 10 × 2 mm cubes, and weighted to an accuracy of 0.33 g. Each film was placed into an individual vial containing 15 ml of a phosphate buffer solutions of pH = 7.5 in the presence of an enzyme lipase from porcine pancreas. The samples were incubated at 30 °C for 45 days. Then, The films were removed, washed with water and dried in an oven to

complete dryness and weighed to determine the degradability using the following Eq. (3):

$$(\%) \text{ degradability} = \frac{m_0 - m_d}{m_0} \times 100 \quad (3)$$

where, m_0 and m_d are the weights of initial and after the destruction and drying sample weight, respectively.

RESULTS AND DISCUSSION

Synthesis and Structural Analysis of Epoxidized Oleic Acid

Oleic acid epoxidation was performed using hydrogen peroxide, as an oxygen donor, and formic acid at 40-60 °C (Scheme 2).

FT-IR spectra analysis was performed to further verify epoxidation reaction. FTIR spectra of oleic acid and epoxidized oleic acid are presented in Figs. 2 and 3, respectively. The spectra of oleic acid and epoxidized oleic

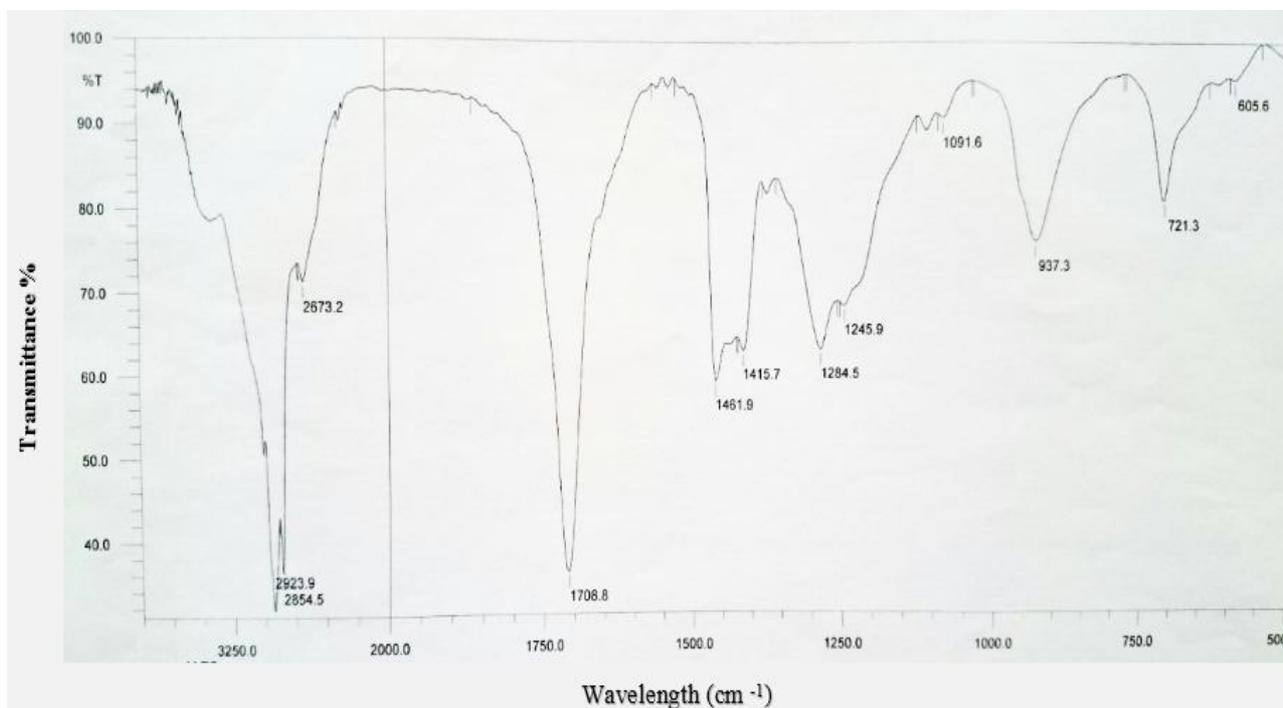


Fig. 2. FTIR spectrum of oleic acid.

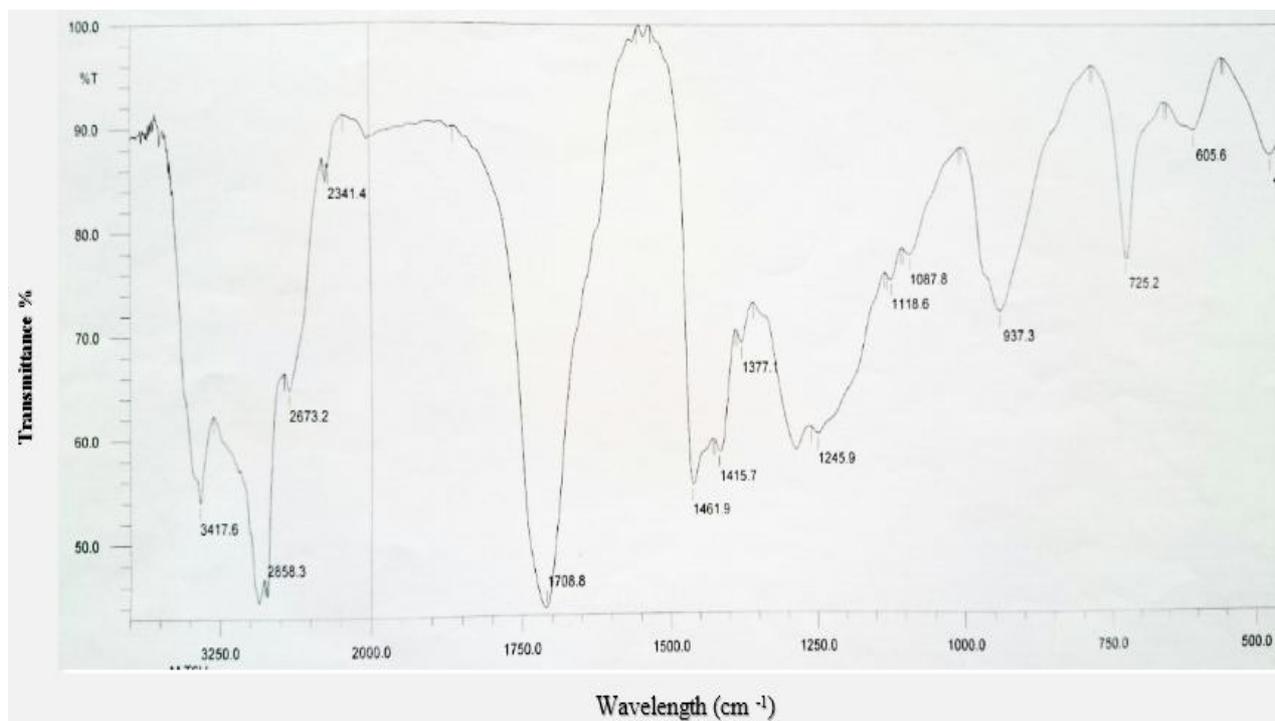


Fig. 3. FTIR spectrum of epoxidized oleic acid.

Table 1. Optimization of Reaction Temperature

Entry	1	2	3	4	5
Temperature (°C)	40	45	50	55	60
Conversion (%)	48.23	36.17	20.56	29.79	41.09

Table 2. Optimization of Reaction Time

Entry	1	2	3	4	5
Time (h)	6	8	10	12	14
Conversion (%)	32.98	48.23	61.00	43.49	37.50

acid were compared and the structures of both oils were investigated. In Fig. 3, absorption bands at 1262, 919 and 846 cm^{-1} were assigned to epoxy group. The appearance of such peaks verified the success of oleic acid epoxidation reaction.

Time and temperature are important factors to determinate the percentage of oxirane. To optimize the reaction conditions with respect to the reaction temperature and time; reactions of oleic acid, formic acid and H_2SO_4 were studied as a model reaction according to the method described in the former part. The effect of temperature on the epoxidation was studied at 40, 45, 50, 55 and 60 °C in 8 h of reaction time (Table 1).

The results show epoxy content is decreased with an increase in the temperature. This indicates that an increase in temperature not only decreased the rate of epoxidation, but increase the rate of side reactions. These results show that the maximum conversion to epoxy (48.23%) has been achieved at 40 °C.

To investigate the effect of reaction time, model reaction were performed in five different times, 6, 8, 10, 12 and 14 h (Table 2). With the increase of reaction time from 6 to 10 h, the oxirane content increased. However when reaction time increased further to 12 and then 14 h, epoxidized oleic acid reduced.

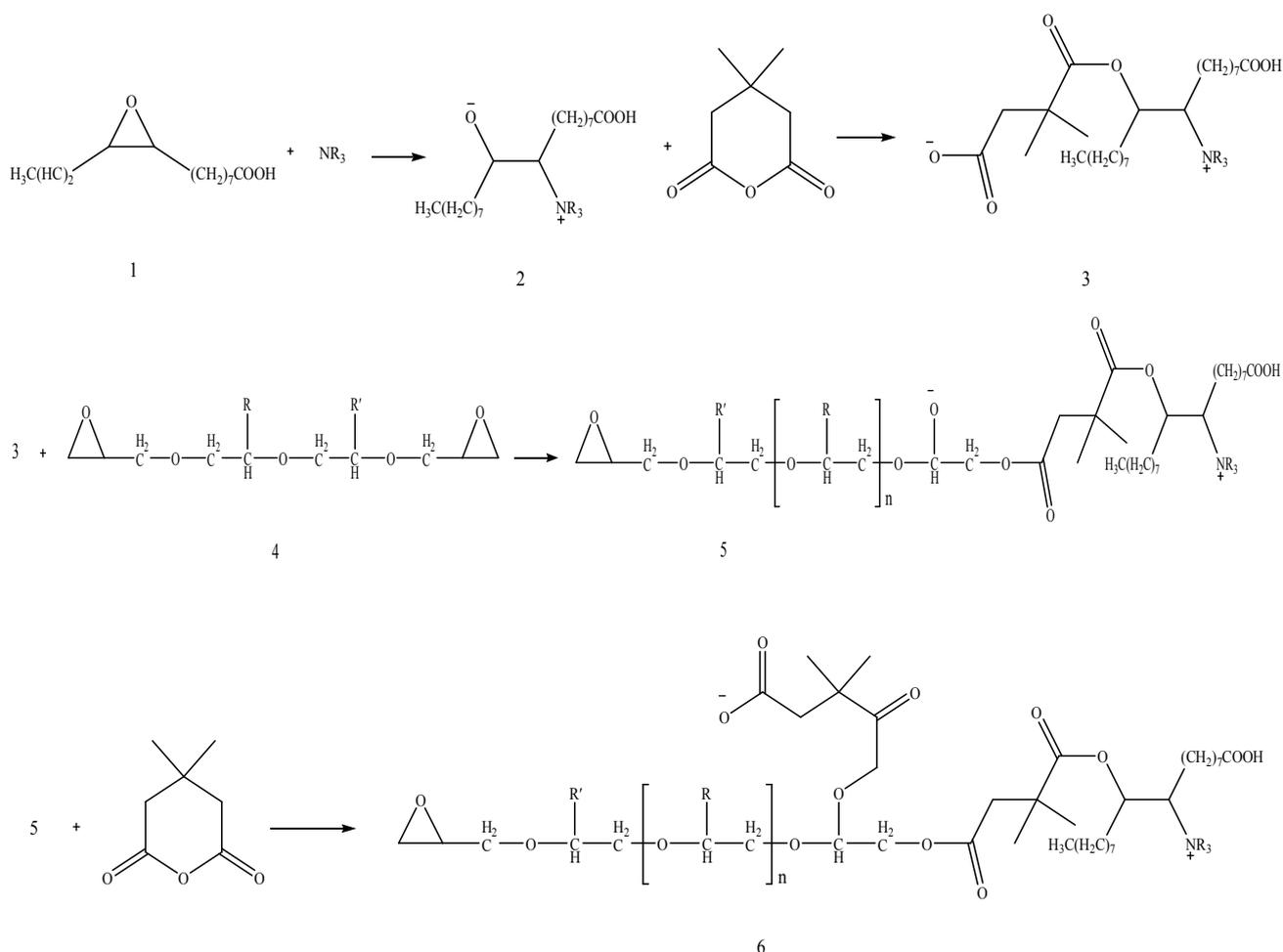
Curing Process

The goal of this study was to prepare novel biodegradable epoxy resin. To this end, DER 736 was modified with three loadings of epoxidized oleic acid (0, 20 and 40 wt%) and then cured with succinic anhydride in the presence of triethyl amine. The synthetic route is outlined in Scheme 3. The reaction of epoxidized oleic acid (one equivalent) with succinic anhydride leads to 3 precomposit, which was subsequently reacted with DER 736 to prepare epoxy-terminated precomposit 5. In the final step, prepolymer 5 was reacted with succinic anhydride (one equivalent) to prepare epoxy-terminated composit 6.

Mechanical Properties of Cured Resin

The mechanical properties are most important properties of polymeric materials. Mechanical properties of epoxy resins are influenced by the cross-linking density. Material selection is based on the properties such as tensile strength, modulus and elongation. Tensile strength test was carried out on the samples from three epoxy coating systems; oleic acid-free, 20% oleic acid and 40% oleic acid. The tensile properties testing was done via three points of film and then averaged. Average thickness was obtained 1.3 mm for all samples (oleic acid-free, 20% and 40%). Figure 4 shows tensile properties of specimens.

Figure 5 shows the oleic acid ratio dependency and



Scheme 3. Chemical route for synthesis of epoxy coating

Young's modulus for all the cured resins. Figure 5 shows that the epoxy resin with 0 and 40% oleic acid have the highest and lowest modulus, respectively. These results show that the hardness decreased with an increase in epoxidized oleic acid.

Mechanical properties of 0%, 20% and 40% oleic acid including tensile strength (MPa), elongation (%) at break, and Young's modulus (MPa) are shown in Table 3.

As shown in Table 3, a pure resin without oleic acid is fairly rigid material with a high modulus (1.5 Mpa), high tensile strength (8.4 MPa) but brittle, with lower elongation at break (4 mm). The result shows that addition of 40 wt% oleic acid in DER 736 leads to increase elongation at break (7.6 mm), reduction modulus, and tensile strength (0.55

Mpa, 6.8 MPa), respectively. This result indicated that the enhancement of epoxy oleic acid increases the flexibility and softening but decrease mechanical properties.

Water Adsorption

One limitation of epoxy resin is water adsorption that can severely weaken the adhesion of the coating to the substrate and reducing its ability to protect. Also, water adsorption reduce mechanical and thermal properties of epoxy resins. Different amounts of epoxidized fatty acids can affect water absorption of polymer chain. Epoxies, depending on the formulation and type, can adsorb various amounts of water. In this part, water adsorption of cured epoxy resin was measured using weight gain. Specimens

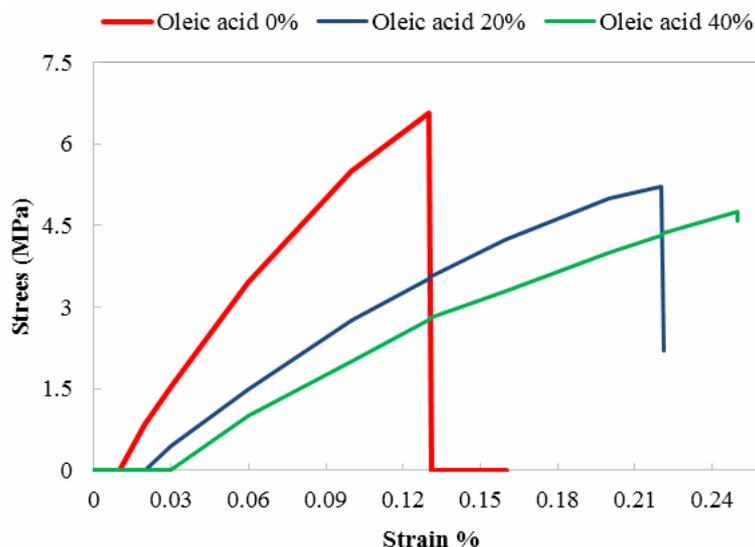


Fig. 4. Tensile curve of cured resin with three loadings of epoxidized oleic acid.

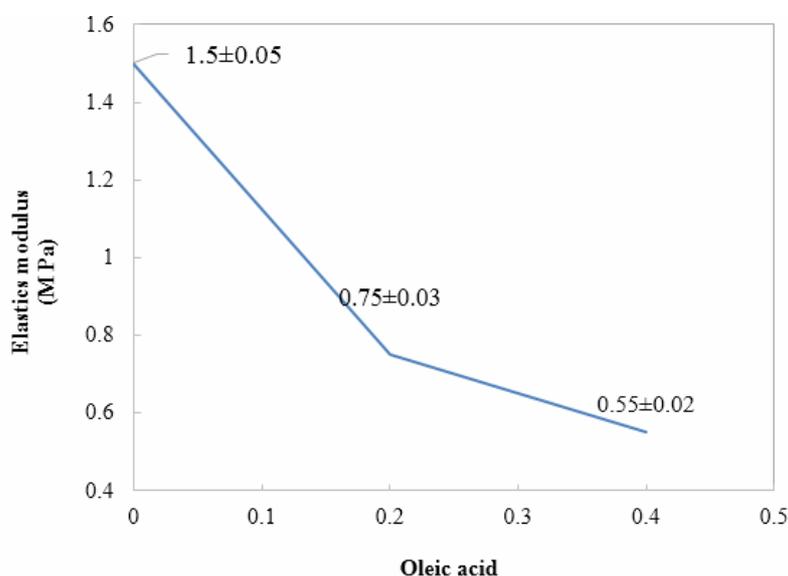


Fig. 5. Modulus curve of cured composite with three loadings of epoxidized oleic acid.

were heated in 40 °C for 2 h and then initial weight was measured (W_0) before placing them in distilled water. The specimens were extracted, dried and weighed again (W_t). The amount of water adsorption was measured each day of first, second and third weeks. The results revealed that in the presence of epoxidized oleic acid, the water adsorption

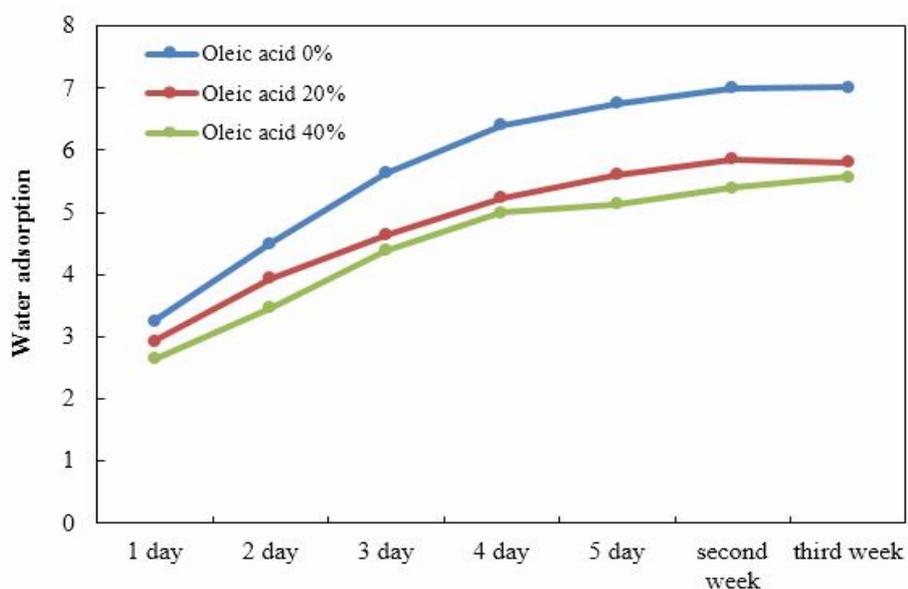
decrease progressively (Fig. 6). So, 40 wt% oleic acid shows the lowest water adsorption. The results show that oleic acid acts as a barrier against moisture.

Enzymatic Degradation

DER coatings were performed with 0, 20 and 40 percent

Table 3. Mechanical Properties Data of Samples

Oleic acid (wt%)	0	20	40
Tensile strength (MPa)	8.4	7.1	6.8
Elongation at break (%)	0.13	0.23	0.25
Modulus (MPa)	1.5	0.75	0.55

**Fig. 6.** Water adsorption of cured composit.

epoxidized oleic acid. After cooling, synthesized samples, were removed from the teflon mold and cut into pieces of $10 \times 10 \times 2$ mm. Enzymatic degradation of the prepared specimens was evaluated by immersing blend films into 15 ml buffer enzyme solution of pH 7.5 for several weeks. Phosphate buffer solutions (0.1 M) were prepared by mixing 6.25 g of Na_2HPO_4 and 1.53 g $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$. Buffered enzyme solution was prepared by adding 1 g of enzyme to these buffer solutions. Enzymatic degradation of epoxy resin is breaking the polymer chains by enzyme. To evaluate the effect of enzyme, it (1 g l^{-1}) was added to buffer solution containing weighed samples (0, 20 and 40 wt% epoxidized oleic acid). Then, the specimens were incubated at 30°C . They were removed after 15, 25, 35 and 45 days and

washed with water and dried in oven (40°C) to complete dryness and were reweighed.

The enzymatic degradation of samples is shown in Fig. 7. These results clearly evidenced an increase of biodegradability by increasing for the time.

The percent of biodegradation of specimens is shown in Table 4. The results indicated that weight loss is increased due to the presence of enzymes. The degradability in phosphate buffer was compared with biodegradability in the presence of enzyme (Table 4). It was observed that degradation in phosphate buffer is lower than that in the case of enzymatic degradation. Accordingly, samples with 40 wt% epoxidized oleic acid revealed 5.09% degradation after 15 days and 27.26% degradation after 45 days, while

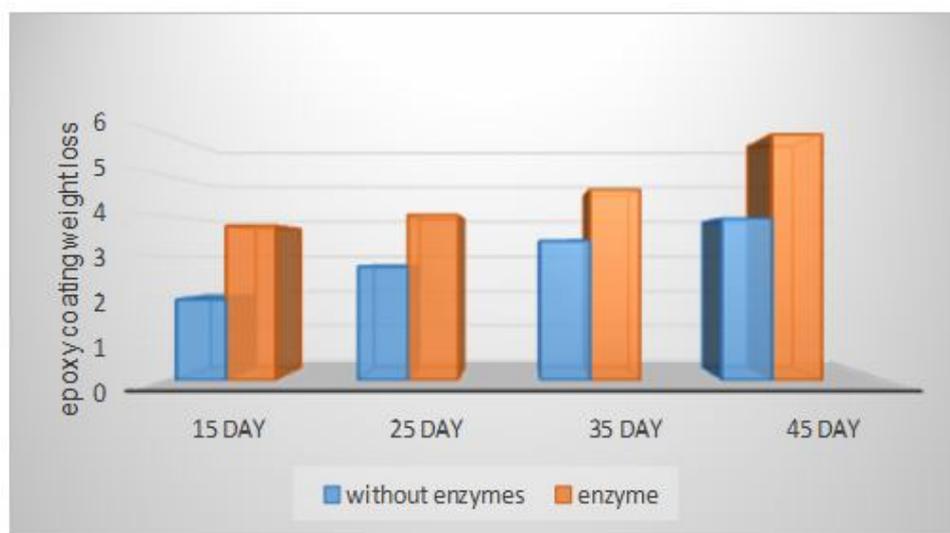


Fig. 7. Degradation of cured composit with 20wt% epoxidized oleic acid.

Table 4. Biodegradation of Cured Composite with Various amount of Epoxidized Oleic Acid in Diffrent Time

Day	Weight loss (%)					
	Oleic acid (0%)		Oleic acid (20%)		Oleic acid (40%)	
	Enzyme	Without enzyme	Enzyme	Without enzyme	Enzyme	Without enzyme
15	1.39	0.94	3.75	2.85	15.53	5.09
25	1.73	1.17	4.01	3.38	24.22	20.66
35	1.85	1.31	4.63	4.01	26.31	25.43
45	3.01	1.40	5.97	4.95	32.19	27.26

15.53% and 32.19% biodegradation was observed for specimens with 40 wt% epoxidized oleic acid after 10 and 45 days, respectively. Table 4 and Fig. 8 showed that the percentage of oleic acid in reducing the weight was statistically significant. In buffer solution without enzyme, the sample free epoxidized oleic acid showed the lowest degradation (1.40%) and epoxy resin with 40 wt% epoxidized oleic acid showed the highest destruction (32.19%) after 45 days.

The degradation curve for epoxy coatings with 0, 20 and 40 percent oleic acid is reported in the Fig. 8. These results

clearly indicate that the enzymatic degradation increase with increment the amount of epoxidized oleic acid, so that the epoxy resin with 40 wt% epoxidized oleic acid have the maximum degradation in 45 days. The enzymatic buffer solution of 40% oleic acid showed the highest amount of degradation compared to epoxy resin with 20% oleic acid and without it on 15, 25, 35 and 45 days.

Morphological Study

The SEM images demonstrated the surface morphologies of epoxy coatings with 40 percent oleic acid

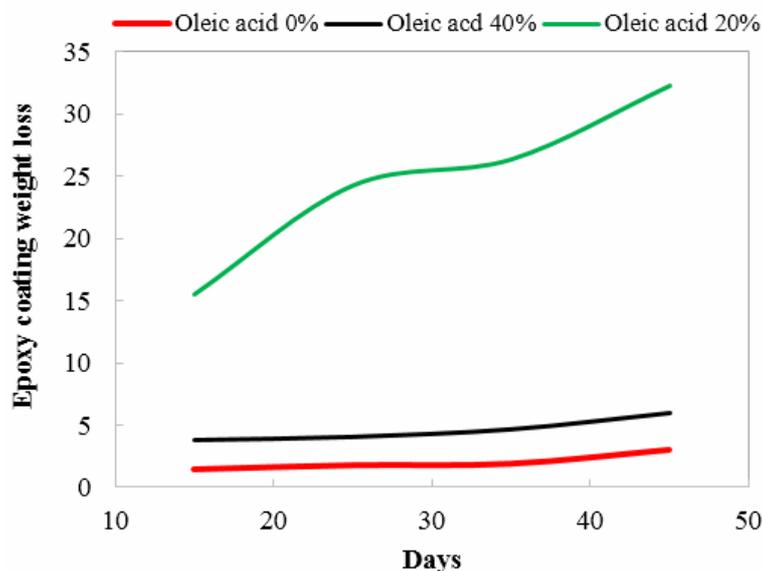


Fig. 8. Enzymatic degradation behavior of composite with three loadings of epoxidized oleic acid.

before biodegradation and 25 days after biodegradation (Fig. 9). The SEM images of epoxy coating 0 wt% oleic acid was observed without significant changes after biodegradation (not shown). However, SEM of the film reveals porous structures due to enzymatic degradation on the surface.

CONCLUSIONS

In the present study, a novel composite containing DER 736 and different loadings of epoxidized oleic acid (0, 20 and 40 wt%) with succinic anhydride as hardener was synthesized. The tensile test of composite showed that the flexibility and softening increased with increasing amount of oleic acid but decreased its mechanical properties. The results of enzymatic degradation test in phosphate buffer and SEM studies revealed that the novel composite is biodegradable. Also, It was found that the highest amount of degradation would be obtained from epoxy resin with 40 wt% oleic acid in 45 days.

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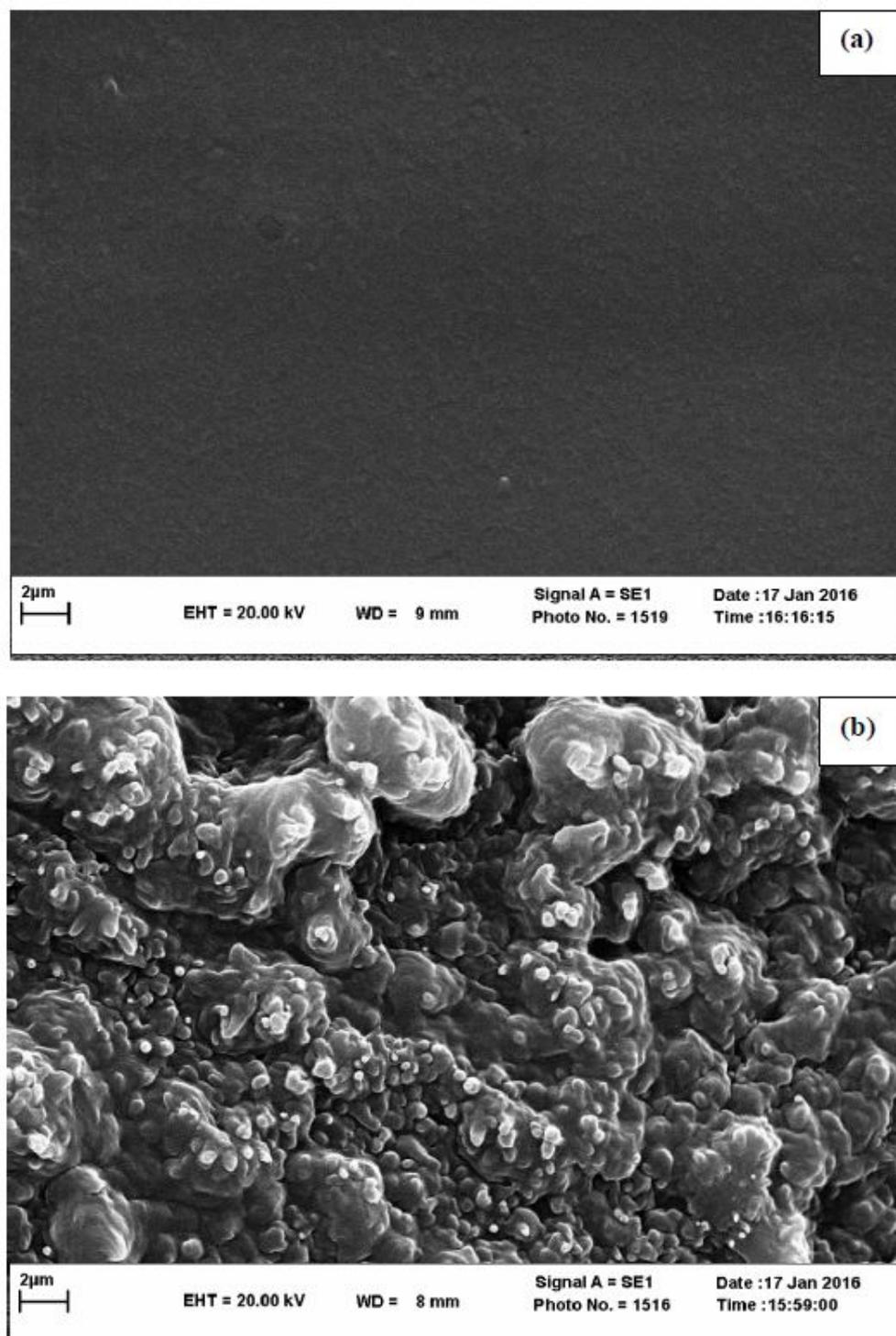


Fig. 9. SEM micrographs of cured composite with 40 wt% oleic acid epoxy (a) before biodegradation (b) 25 days after biodegradation.

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