

## **Epoxidation and Acrylation of Syrian Cottonseeds oil**

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**Abstract:** Epoxidized vegetable oils are promising candidates as a substitute for petroleum oil based plasticizer, lubricants and adhesives. Chemical modification of fatty acid chain of triglyceride appears to be one route towards this objective substitution of petroleum product. In this study, the epoxidation of cottonseeds oil has been carried out via catalytic acidic ion exchange resin method using 30% hydrogen peroxide as oxygen donor and acetic acid as oxygen carrier in presence of sulphuric acid as a catalyst. Further, an epoxy acrylate resin was synthesized from the epoxidized cottonseeds oil (ECISO) by using methylmethacrylate monomer, and triethylamine as a catalyst, and hydroquinone as an inhibitor respectively. The acrylation of epoxidized cottonseeds oil (MMAECISO) is done by introducing methylmethacrylate, which is an unsaturated monomer into oxirane groups of the epoxidized oil (ECISO). This reaction was confirmed by analytical data in terms of Acid Value, Iodine Value, Oxiran content %, Viscosity, Differential Scanning Calorimetry (DSC), Scanning Electron Microscope (SEM), and spectral analysis (FTIR).

**Keywords:** Epoxidation, acrylation, MMAECISO, cottonseeds oil, DSC.

### **Introduction**

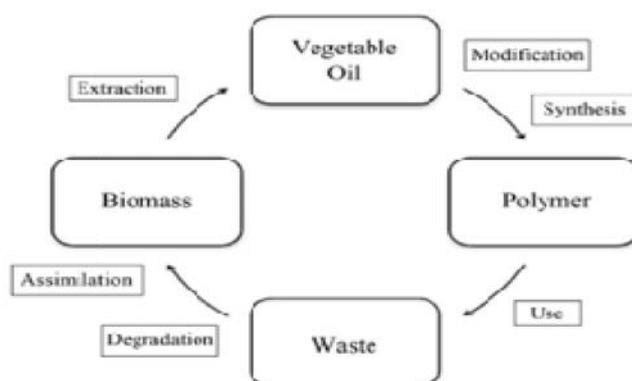
In recent years, there has been a growing trend in using vegetable oils as renewable resources especially in oleo chemical productions. Several derivatives of vegetable oils are used as polymerizable monomers in a radiation of curable system due to their environmentally friendly character and low cost when compared with products from petroleum. Moreover, the long fatty acid chains of vegetable oils impart desirable flexibility and toughness to some brittle resin systems such as epoxy, urethane and polyester resins [1]. Vegetable oils have a number of excellent properties which could be utilized in producing valuable polymeric materials such as epoxy, polyester, amide, alkyd and polyurethane, in addition to its many applications in other areas[2]. Epoxidized vegetable oils, for example epoxidized palm oil and epoxidized soybean oil were utilized in UV curable coating systems [3]. Vernonia oil, natural oil containing epoxide groups, was utilized as a polymerizable monomer in cationic UV-cured coatings [4].

Nowadays, there is a growing interest to produce biopolymers. The environmental problems generated by disposal of and other related polymers have intensified researches with polymers that have their origin from renewable resources such as plant oil, cellulose and lactic acid [5]. One of the current priorities in polymer chemistry is to explore biobased polymers from renewable resources. Among those polymers, vegetable oils are expected to be an ideal alternative of chemical feedstock, owing to their low cost and environmental friendliness [6,7]. Triglyceride oils are one of the most important sources for biopolymers. Triglycerides from plants, such as soybean, palm, rapeseed or sun flower, can be utilized. The triglyceride compound must be isolated and purified, and also functionalized to obtain the requested reactivity. Various chemical modification reactions are possible; the most common ones proceed via an epoxidation reaction. Therefore, the use of plant oil based resins

in liquid molding resins not only would reduce volatile organic compounds emissions, thereby reducing health and environmental risks, but it also would promote global sustainability [8]. Triglycerides are composed of three fatty acid chains joined by a glycerol center. Oil-based biopolymers have many advantages compared with polymers prepared from petroleum-based monomers. They are biodegradable and, in many cases, cheaper than petroleum polymers. The life cycle of polymers based on triglyceride oils is given in (Scheme 1).

Vegetable oil constituents are complex multicomponent mixtures of different triglycerols that are an ester of glycerol and fatty acids. Recently biobased, thermosetting polymers from vegetable oils such as an epoxy soybean oil, epoxy castor oil, epoxy palm oil, epoxy linseed oil and epoxy canola oil have been synthesized in several ways.

Bio-based polymers and resins are currently attracting a great deal of interest in both research and industrial applications. Such polymers have many advantages compared to conventional petroleum-based polymers. They are biodegradable, and can be obtained from renewable resources at lower cost.



**Scheme(1):** Life cycle of polymers based on triglyceride oils

Vegetable oils are composed of complex mixtures of different triglycerides, which are esters of glycerol and fatty acids [9]. Triglycerides are one of the most important raw materials for bio-based thermosetting polymers [10]. One of the most effective modifications of triglycerides is the epoxidation reaction, in which the C=C double bonds are converted to oxirane (epoxide) rings [11] which are highly strained and very reactive. They can easily undergo variety of reactions to give important chemical compounds for polymer synthesis, such as polyols [12], and can undergo cationic polymerization to form polymeric networks [13], as well as be used in UV curable systems [14]. Epoxidized oil acrylate is synthesized by several methods; Habib and Bajpai reported the synthesis of epoxidized soybean acrylate using acrylic acid in the acrylation reaction. Analytical and spectroscopic characterization of the product revealed that the epoxy rings were opened and the epoxy acrylate was obtained successfully [15]. Pelletier and Gandini synthesized epoxy acrylates by reacting the hydroxyl and oxirane functional groups in the triglyceride molecules with acryloyl chloride [16]. Castor oil was found to be a good renewable source for polymeric material [17]. Vernonia oil is a naturally epoxidized oil. It was used to prepare epoxy acrylates by reacting the oxirane rings with acrylic and methacrylic acids and acrylation yields were up to 85%–98% [18].

This paper describes the development of the acrylated cottonseeds oil based. Acrylated epoxidized cottonseeds oil (MMAECSO) has been prepared using the epoxidized cottonseeds oil, methyl methacrylate, triethylamine as a catalyst, and hydroquinone as a gelling inhibitor.

## **Materials and Methods**

### **2.1. Materials**

Refined cottonseeds oil (CSO) was obtained from alshahbaa factory. Hydrogen peroxide (30 wt%) and acetic acid. Methyl methacrylate and triethyl amine, hydroquinone, sulphuric acid (98 wt%),  $\text{BF}_3$  in methanol were obtained from Merck. For analytical purpose, hydrogen bromide, crystal violet indicator, potassium hydroxide, sodium hydroxide, Wij's solution, sodium thiosulphate, potassium iodide.

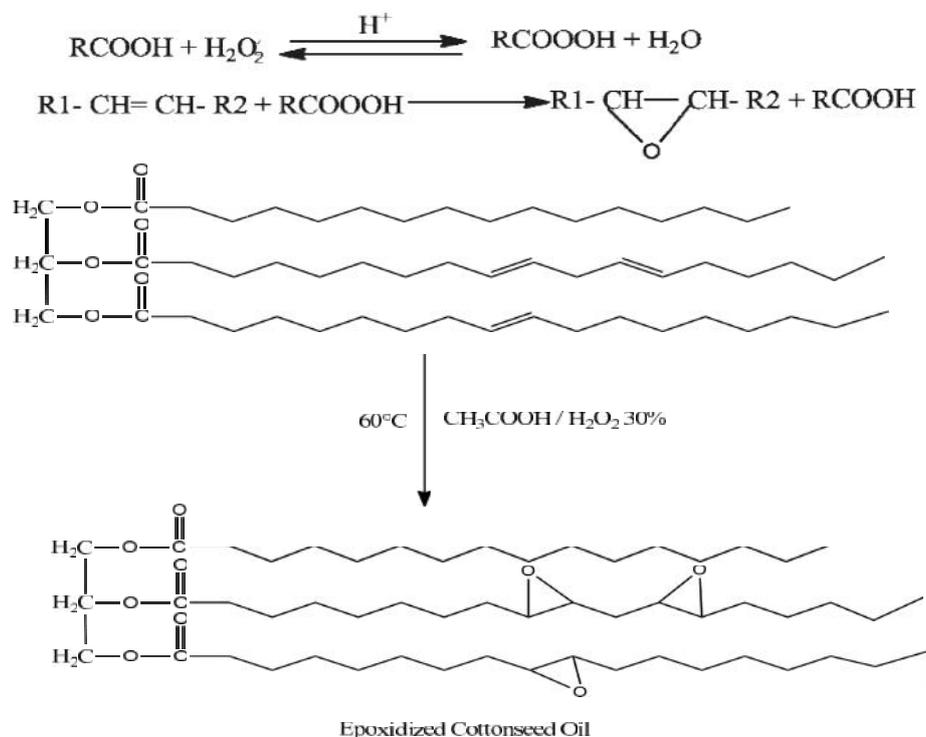
### **2.2. Fatty Acid Composition of Cottonseed Oil**

Many naturally occurring fats are containing fatty acids with variation in chain length between 14-22 carbon atoms and single to triple bonds between two carbon atoms. The unsaturated fatty acids present in cottonseeds oil are oleic acid, linoleic acid and linolenic acid containing one, two and three double bonds between two carbon atoms respectively.

The functionality present in cottonseed oil is in terms of double bonds thus, the  $\text{C}=\text{C}$  acts as a reaction site for chemical modification in vegetable oil. Before epoxidation, cottonseeds oil was run on a Mass-gas chromatograph using  $\text{BF}_3$  methylation method following AOCS official method 996.01, Sec. E (35). The (Table 1) gives the fatty acid composition of cottonseed oil, and (Fig 1) shows the composition of cottonseeds oil by gas chromatography[19].

### **2.3. Epoxidation of Cottonseeds Oil ECSO**

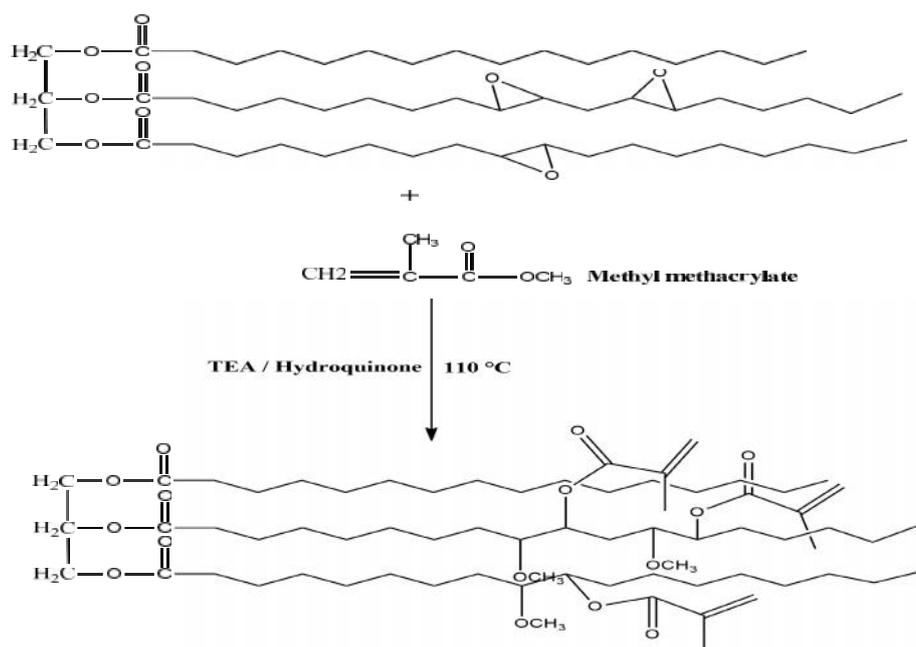
The epoxidation reaction is carried out in a 1-liter three-neck flask equipped with glass mechanical stirrer. The whole assembly was immersed in a water bath at ( $60^\circ\text{C}$ ). 200ml of cottonseeds oil was taken in the reactor and with respect to this, calculated amount of acetic acid ( $\text{CH}_3\text{COOH}$  to double bond mole ratio: 0.5:1), and was added to the reactor and the mixture was stirred for about 30 minutes. Then the required amount of 30 % aqueous hydrogen peroxide ( $\text{H}_2\text{O}_2$  to double bond mole ratio: 2:1) was added drop wise in such a way that the addition was completed in half an hour and the reaction was continued further for the required time duration. After the addition of  $\text{H}_2\text{O}_2$ , 3% sulfuric acid was added to the mixture as an acid medium (3% of the total added weight of acetic acid and oxygen water), and the mixture was continue stirred for 6 hours (scheme2), then the collected samples were filtered and extracted with diethyl ether in a separating funnel, after that washed with cold and slightly hot water successively to remove free acid. The final product pH of product was checked to have a value of 7.0 and then analyzed for iodine value, oxirane content% [20].



**Scheme (2):** Synthesis of epoxidized cottonseed oil

#### 2.4. Acrylation of Epoxidized Cottonseed Oil MMAEC SO

The apparatus used for acrylation of epoxidized cottonseed oil is consisted of 500 ml three-neck round bottom flask equipped with a mechanical stirrer, a reflux condenser and a dropping funnel. A mixture containing (0.2 mol) epoxidized cottonseeds oil, 0.5 %hydroquinone (on the weight of reactants)as inhibitor and 1.0 % triethylamine (on the weight of reactants) as a catalyst were charged into three necked flask. While stirring the mixture, (0.8 mol) methyl methacrylate was introduced to the mixture through the dropping funnel. After the addition of methyl methacrylate was completed, the mixture was heated up to(110 °C).The product is collected and saved for analysis (scheme3) and (Fig 2)[21].



**Scheme (3):** Synthesis of acrylated epoxidized cottonseed oil

## 2.5. Characterization of the synthesized ECSO

Epoxidation of fatty acids is a reaction of a carbon carbon double bond with an active oxygen, usually from a peracid, which results in the addition of an oxygen atom, converting the original  $-C=C-$  bond into a three-membered epoxide (oxirane) ring. Epoxy content is the most important property of epoxy materials. Samples of ECSO were analyzed for their percents (by weight) of epoxy functional groups by an AOCS official method Cd9-57 (Oxirane oxygen in epoxidized materials). Iodine value was determined by method according to AOCS official method Cd 1d-92. Viscosity was measured on a Digital Viscometer at 25 °C (Table 2). Fourier Transform Infrared (FTIR) spectra were recorded by Jasco FT-IR 4100 Spectrophotometer. FTIR spectra were obtained by coating the samples on potassium bromide (KBr) pellet (Fig 3). <sup>1</sup>H NMR spectra of the prepared ECSO were obtained using Bruker Bio spin 400 MHz using CHCl<sub>3</sub> as a solvent (Fig 4).

## 2.6. Characterization of the synthesized MMAECSO

The extent of reaction was determined by calculating the iodine value which determined by method according to AOCS official method Cd 1d-92. Oxiran oxygen content was determined by method according to AOCS official method Cd 9-57. Viscosity was measured on a Digital Viscometer at 25 °C (Table 2). Fourier Transform Infrared (FTIR) spectra were recorded by Jasco FT-IR 4100 Spectrophotometer. FTIR spectra were obtained by coating the samples on the potassium bromide (KBr) pellet (Fig 5). Differential scanning calorimetry (DSC) was carried out using a DSC20- (Mettler Toledo) Samples were scanned at 10 °C/min, in the range from 25 °C to 450 °C (Fig 6), and it was observed the glass transition temperature (T<sub>g</sub>) and Crystallization transition temperature (T<sub>c</sub>) and melting transition temperature (T<sub>m</sub>) (Table 4). The morphological microstructures from scanning electron microscopy using Vega Tescan Scanning Electron Microscope were used to examine the structure of the studied sample MMAECSO (Fig 8) shows the morphologies of bioplastics from studied epoxidized oils.

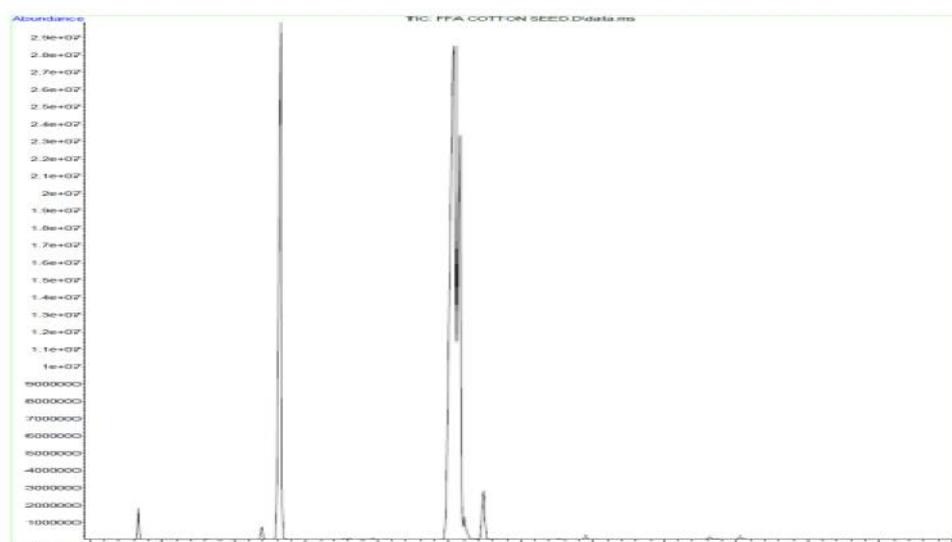
## **Results and Discussion**

### **3.1. Analysis of synthesized ECSO**

The iodine value and the oxirane oxygen content are important properties in the characterization of epoxidized vegetable oils. While the iodine value indicates to the unsaturated residue remained after the epoxidation reaction, the oxirane oxygen content indicates of epoxy groups to presence in the products. In the preparation of polymers, epoxy resins with a lower iodine value and higher oxirane oxygen content are desired (Fig 7). The reductions in iodine values indicated the consumption of unsaturation during epoxidation, but they did not represent solely conversion to epoxy groups because the epoxy ring degradation generates side products. The determined iodine value of ECSO is 20.5. The highest epoxy content in synthesized epoxidized cottonseed oil (ECSO) was 3.65 % and it is almost comparable to the commercial ESO (6.3 %). The unsaturation and epoxy groups can also be monitored by the Fourier transform infrared (FTIR) spectroscopy. The FTIR spectroscopy is a rapid, non-destructive technique that has been widely applied in the characterization of lipids because lipids have functional groups with characteristic absorption bands in the infrared region of the electromagnetic spectrum [22, 23]. In FTIR spectrum of ECSO (Fig 3), C–H stretching ( $\text{CH}_3$ ) is observed at  $2926\text{ cm}^{-1}$ , and C–H stretching ( $\text{CH}_2$ ) is observed at  $2856\text{ cm}^{-1}$ . C–O–C stretching from oxirane vibration appears at  $1242\text{ cm}^{-1}$  and  $868\text{ cm}^{-1}$ . C=O stretching is observed at  $1742\text{ cm}^{-1}$ . Only CSO showed a peak at  $1652\text{ cm}^{-1}$  representing the double bond (C=C). This peak disappeared in the ECSO.  $^1\text{H-NMR}$  spectra were recorded by Bruker Bio spin 400 MHz using  $\text{CHCl}_3$  as a solvent with. All the data are given as chemical shifts (ppm). The resonance D between 3.8 and 4.4 indicated the glycerol center in the triglycerides. A multiplet in the region between of F ( $=2.51\text{ ppm}$ ) and E ( $=2.72\text{ ppm}$ ) corresponds to the protons of the epoxy group (Fig 4).

### **3.2. Analysis of synthesized MMAECSO**

The esterification reaction takes place between the epoxidized cottonseed oil and methyl methacrylate in the presence of triethyl amine, the epoxy group reacts with the carboxyl ionic group to form additional polymeric methoxy ester. Epoxide resins react with carboxylic group to form esters. The reaction takes place between the carboxyl group of the methyl methacrylate and both the epoxy and hydroxyl groups of the epoxides. Tertiary amine interacts with methyl methacrylate and removes its methyl group as cation thus producing a carboxylate anion, this carboxylate anion undergoes nucleophilic addition to electron deficient carbon atom of epoxy group and produces an alcoholate anion the alcoholate anion abstracts the proton from amine and completes the reaction with formation of ester and regeneration of amine. The carboxylic anion then attacks the other unreacted epoxy group till all epoxy and methyl methacrylate get exhausted, which results in the formation of epoxy methyl methacrylate (Fig 2). The degree of esterification is an important parameter that may affect the polymer property. The iodine value was higher, and the oxirane oxygen content was lower after the acrylation reaction accomplished (Fig 8). The FTIR spectroscopy of MMAECSO shows C=O of the acrylated compound at  $1743\text{ cm}^{-1}$ , and C–O distinctive for ester at  $1241\text{ cm}^{-1}$ . Another significantly different absorption band is observed in the spectrum at  $1632\text{ cm}^{-1}$  indicating the presence of vinyl functionality ( $-\text{C}(\text{CH}_3)=\text{CH}_2-$ ). The presence of vinyl functionality of the acrylated polymer which also supported by the absorption peak at  $965\text{ cm}^{-1}$  as this indicates the presence of vinyl group,  $[\text{CH}_2=\text{C}(\text{CH}_3)(\text{CO})-\text{O}]$  depicted in (Fig 5).



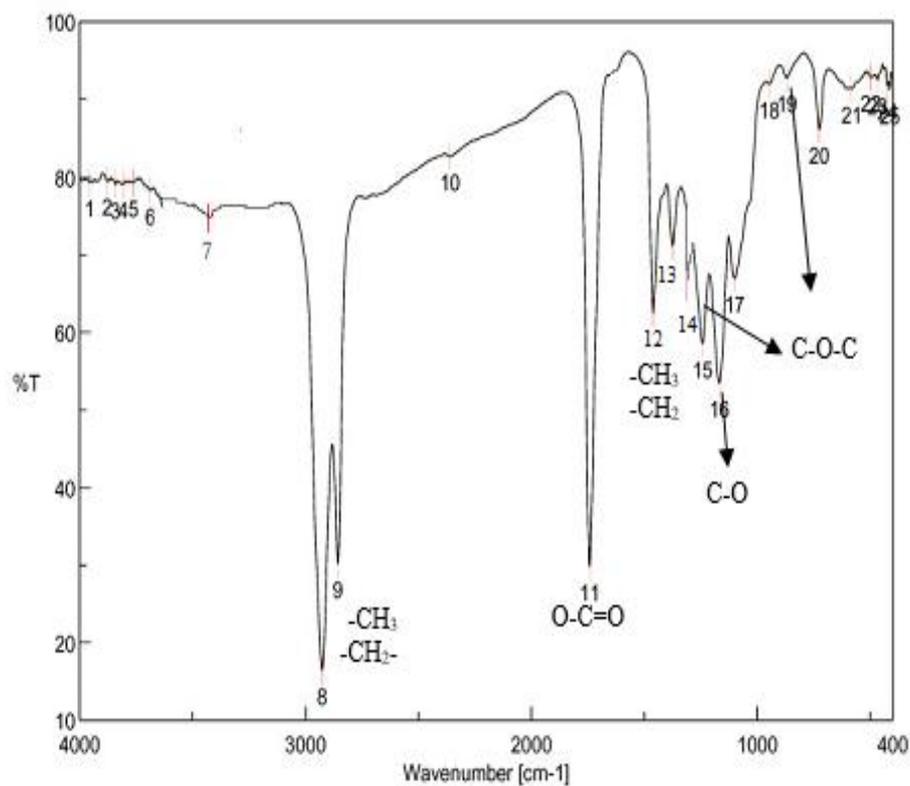
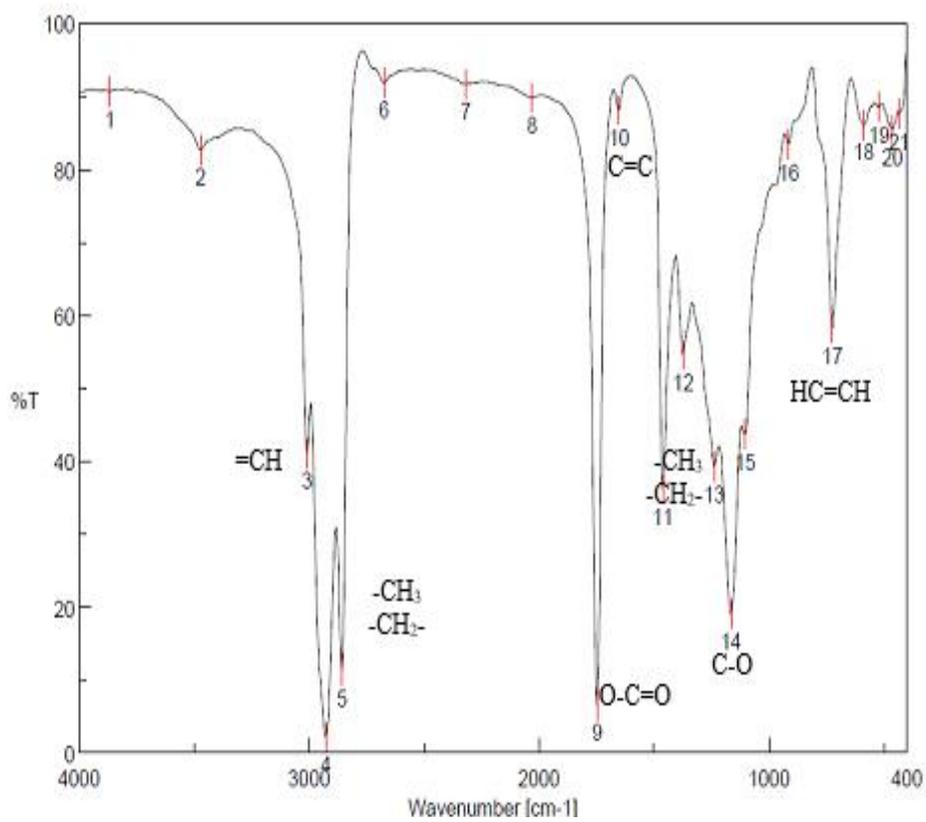
**Figure (1):** The composition of fatty acids in cottonseeds oil by gas chromatography

**Table (1):** The fatty acid composition of cottonseed oil

Serial Number	Retention Time	Fatty Acids	Composition (wt%)	Molecularweight
1	3.688	Myristic Acid (C <sub>14</sub> H <sub>28</sub> O <sub>2</sub> )	0.90	228.37
2	5.414	Palmitoleic Acid (C <sub>16</sub> H <sub>30</sub> O <sub>2</sub> )	0.55	254.4
3	5.712	Palmitic Acid (C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> )	26.79	256.42
4	8.233	Linoleic Acid (C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> )	52.25	280.45
5	8.396	Oleic Acid (C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> )	16.54	282.46
6	8.611	Stearic Acid (C <sub>18</sub> H <sub>36</sub> O <sub>2</sub> )	2.75	284.48
7	9.902	Linolenic Acid (C <sub>18</sub> H <sub>30</sub> O <sub>2</sub> )	0.12	278.4
8	12.036	Arachidic Acid (C <sub>22</sub> H <sub>42</sub> O <sub>2</sub> )	0.1	312.53



**Figure (2):** The product MMAECSO



(b)  
Figure (3):FTIR Spectrum of CSO (a), and ECSO (b)

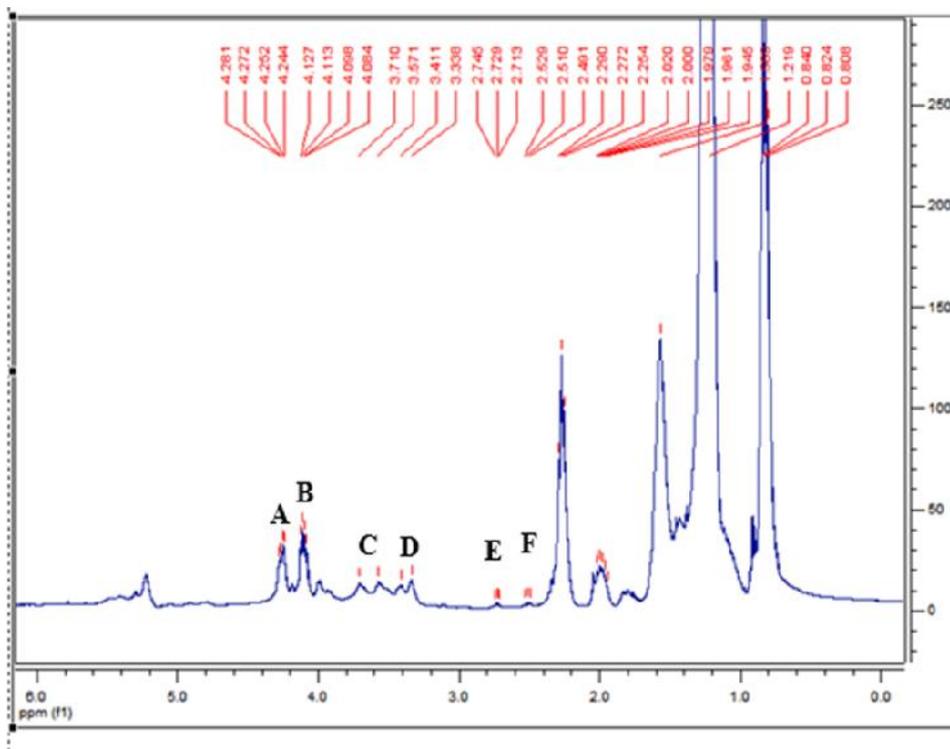


Figure 4. <sup>1</sup>H-NMR of ECSO

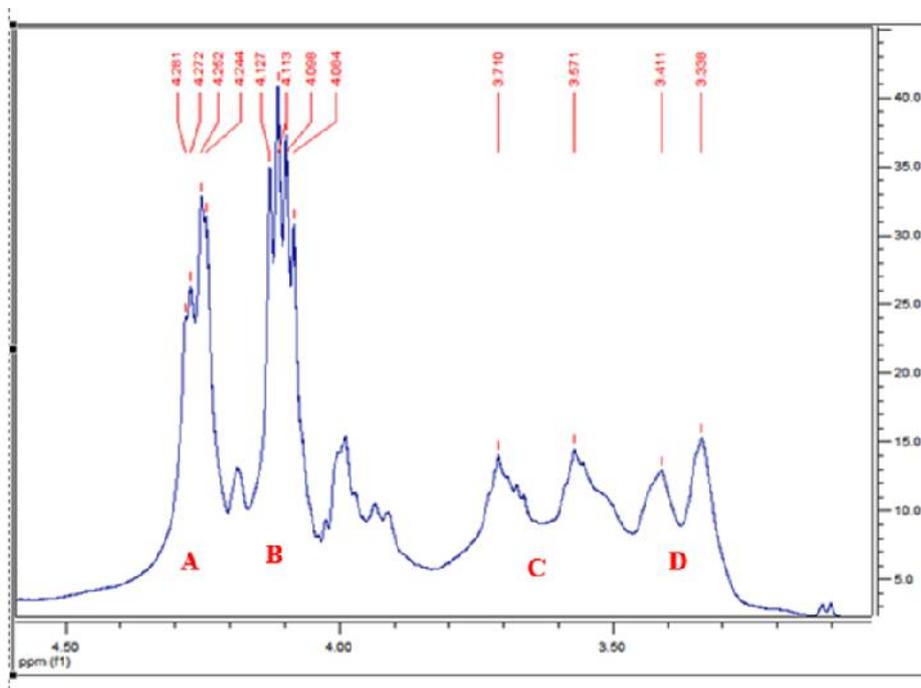


Figure 4. (continued)

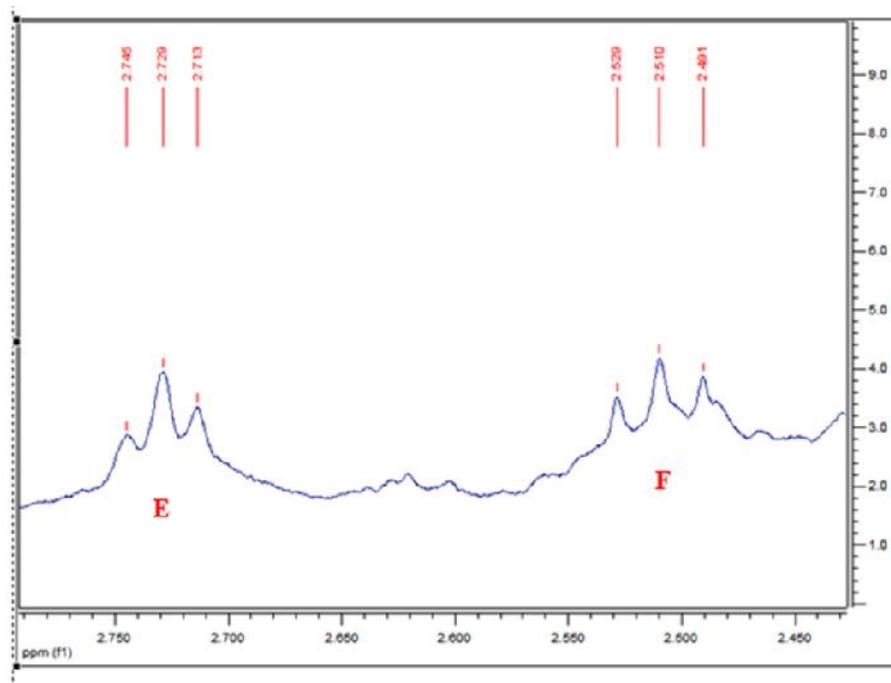


Figure 4. (continued)

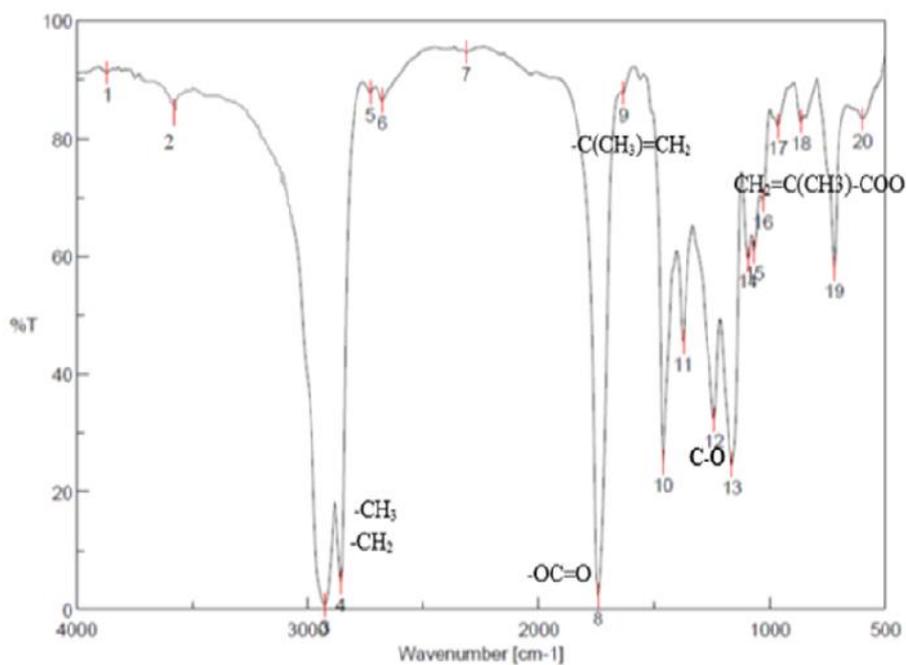


Figure (5):FTIR Spectrum of MMAEC SO

The physical and analytical characteristics of ECSO and MMAECSO are presented in (Table 2). The decrease of the percentage of oxirane rings reveals the consumption of epoxy groups as a result of the acrylation process. On the other hand, the addition of the acrylate groups to the epoxy chain resulted in a higher molecular weight product (MMAECSO), with a higher viscosity compared to the starting reactant ECSO. The molecular weight and molecular weight distribution of ECSO and MMAECSO were determined using Equation (1) and (2):

$$M_n = X_n \cdot M_0 \quad (1)$$

$$M_w = X_w \cdot M_0 \quad (2)$$

and the polydispersity index (PDI) was calculated using Equation (3) (Table 3):

$$(3) \quad PD = \frac{M_w}{M_n}$$

the molecular weight of ECSO was increased due to the extent of the chains increasing by grafting of acrylate groups onto the triglyceride backbone. These results are in good agreement with the increase of the viscosity, as it could be seen in (Tables 2) and (Table 3). the viscosity of epoxy acrylate adduct is very high, it is difficult to measure at room temperature by normal viscometers, (Fig 6) shows the DSC Curve of MMAECSO It can be clearly observed that the peak temperature, and were determined the glass transition temperature  $T_g$ , crystallization transition temperature  $T_c$ , and melting transition temperature  $T_m$  (Table 4).

**Table 2.** Physio-chemical characteristics of ECSO and MMAECSO

Resin	Iodine Value I <sub>2</sub> /100g	Oxiran content %/100g	viscosity (cPs)
ECSO	20.50	3.65	465
MMAECSO	45	0.5	-

**Table (3):** Molecular weights of ECSO and MMAECSO

Resin	M <sub>w</sub> g/mol	M <sub>n</sub> g/mol	p	PD M <sub>w</sub> /M <sub>n</sub>
ECSO	1427	922	0.78	1.54
MMAECSO	5124.16	2987.58	0.7	1.71

**Table (4):** Thermal data of MMAECSO

sample	T <sub>g</sub> °C	T <sub>c</sub> °C	T <sub>m</sub> °C	H <sub>m</sub> j/g	S <sub>m</sub> j/g
MMAECSO	64.89	255.84	379.19	-211.39	0.5574

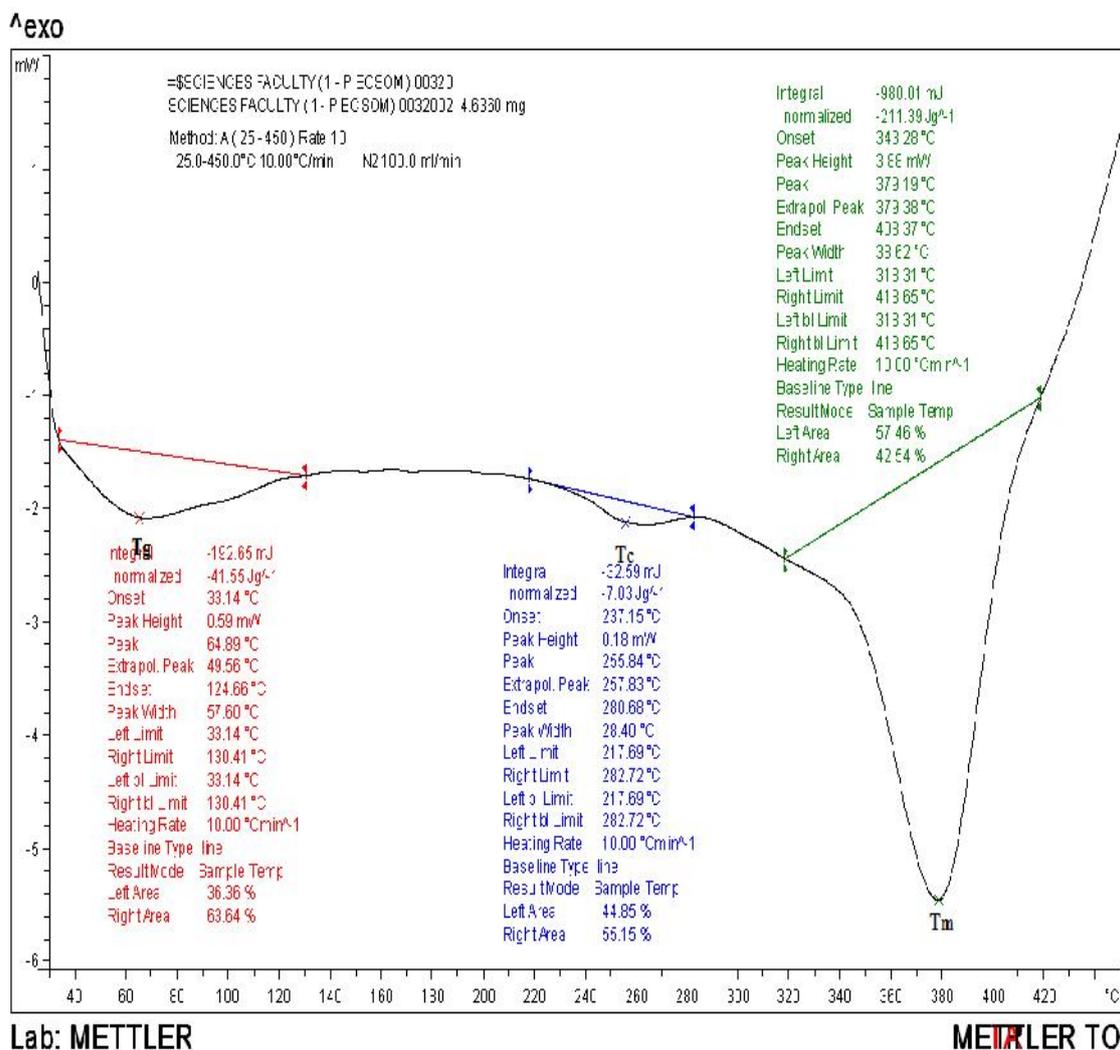
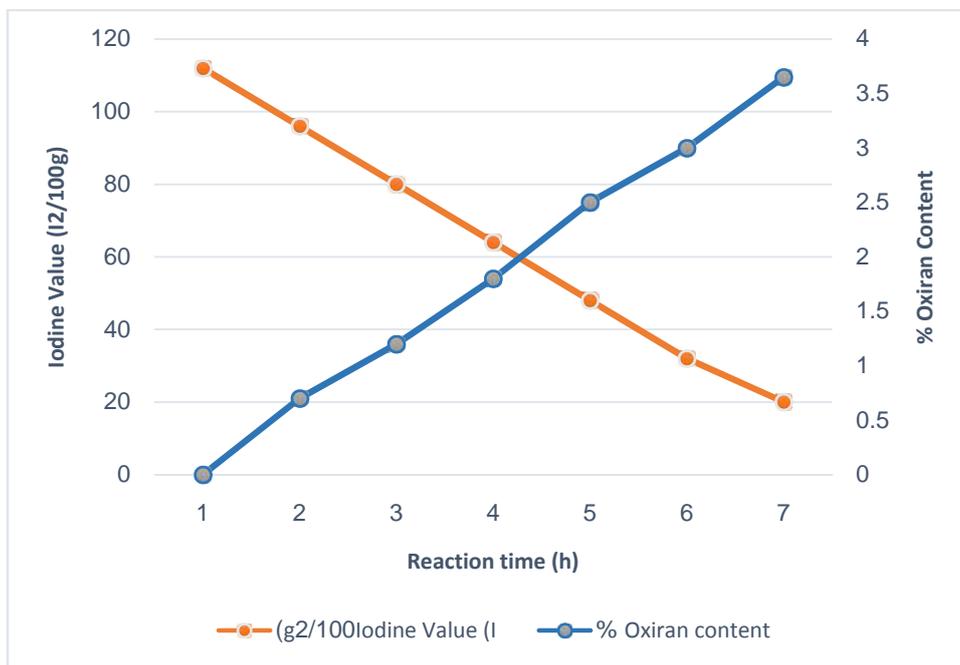
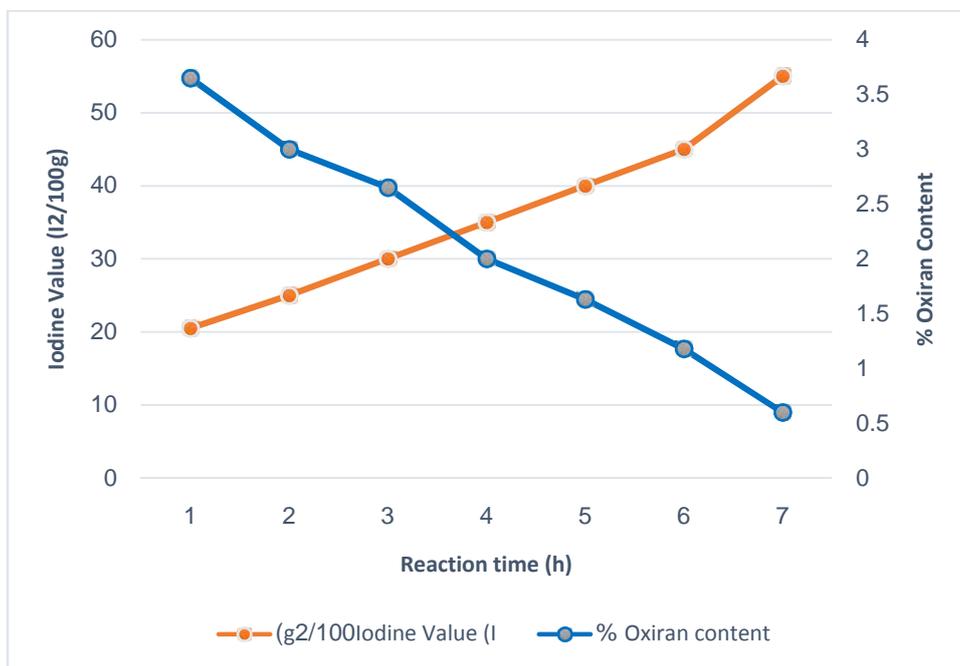


Figure (6):DSC traces of MMAEC SO



**Figure (7):** Iodine Value and Oxiran Content (%) during Epoxidation Reaction Time



**Figure (8):** Iodine Value, and Oxiran Content (%) during Acrylation Reaction Time

(Fig 9) shows a SEM micrograph of a MMAECSO sample indifferent magnifications, Where appears the network structure for polymer.

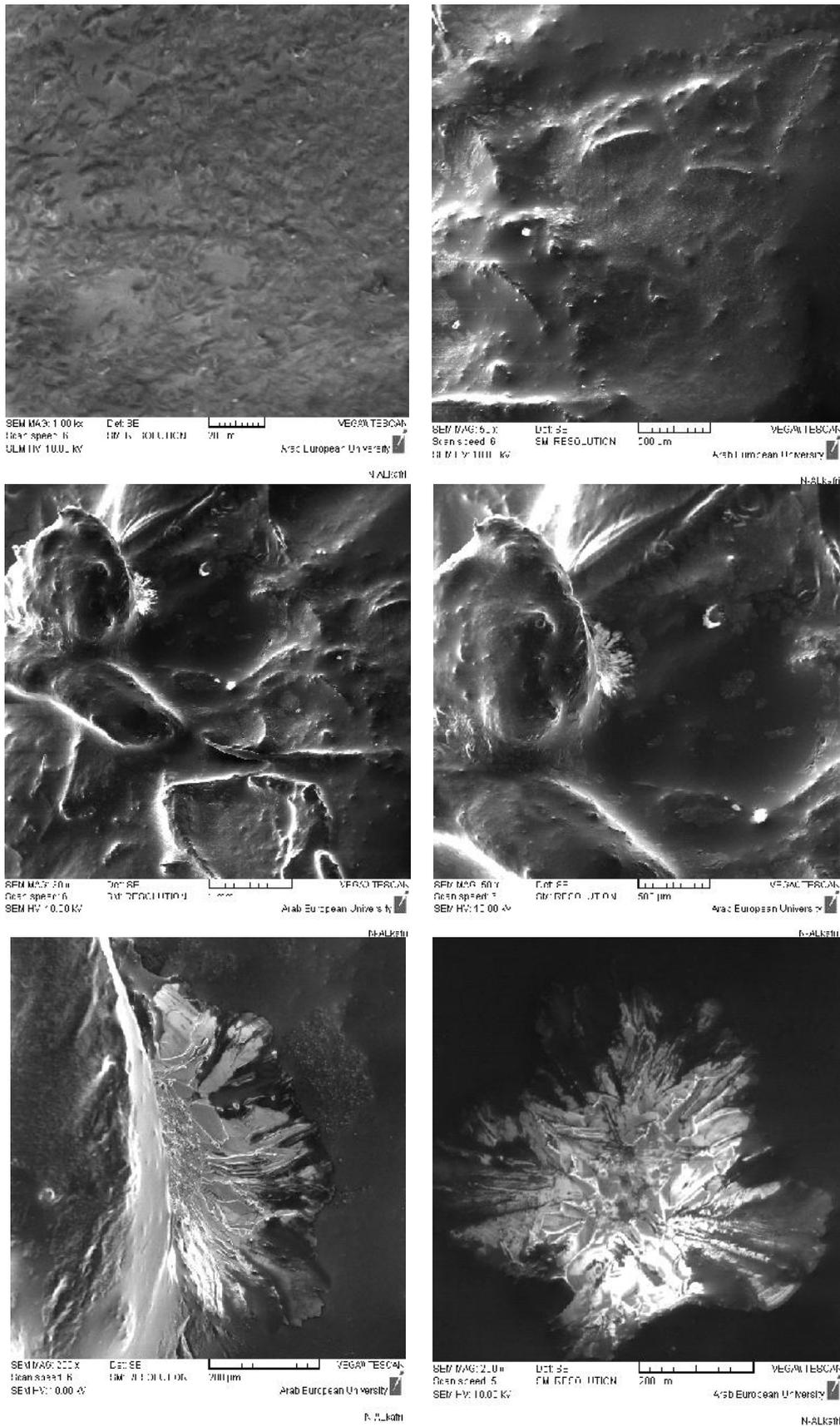


Figure (9): SEM micrographs of network system for MMAECSO

## Conclusions

In this article, the acrylation of the epoxidized cottonseed oil to produce MMAECSO in a yield of about 96% was successfully accomplished. Spectroscopic investigations by means of FTIR and NMR revealed that the oxirane rings were opened, and the acrylate groups were introduced onto the triglyceride molecules. Accordingly, the molecular weight of ECSO was increased as well as its viscosity. Moreover, the thermal properties of this product showed superior results compared to ECSO.

## Acknowledgements

We greatly acknowledge the research laboratories of Chemistry Departments - Aleppo University- Syria, for the technical and financial support of the research.

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