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SYNTHESIS AND CHARACTERIZATION OF ACRYLATED EPOXIDIZED SOYBEAN OIL FOR UV CURED COATINGS

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Abstract. This paper investigates the curing of biodegradable polymer films which were synthesized from soybean oil through the ultraviolet radiation and their stability against thermal degradation. In this study the epoxidation of soybean oil has been carried out *via* peracetic method. Further, an epoxy acrylate resin was synthesized from the epoxidized soybean oil (ESO) by using acrylic acid monomer. Triethylamine (TEA) and hydroquinone were used as a catalyst and inhibitor respectively. The acrylation of epoxidized soybean oil (AESO) is done by introducing acrylic acid, which is an unsaturated monomer into oxirane groups of the epoxidized oil (ESO). This reaction was confirmed by analytical data in terms of oxirane oxygen content, acid value, viscosity and spectral analysis. Different UV curable formulations have been investigated using synthesized acrylated resin and trifunctional acrylate monomer used as crosslinkable active diluents with photoinitiator. Monomer used was trimethylolpropane trimethacrylate (TMPTMA) while photoinitiator used was benzophenone along with an activator dimethyl ethanol amine (DEA). The mixtures were cured to make thin polymeric films under UV radiation with optimum irradiation dosing time of 20 min to produce the excellent cured coating which exhibits good thermal stability of about 473 K.

Keywords: epoxidation, acrylation, UV curing, coating.

1. Introduction

UV radiation curing is one of the most effective processes for rapid transforming of a liquid coating film into a solid film. This well proven technology offers a number of advantages making it suitable for preparation of composite polymers [1, 2]. 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 curable system due to their environmentally friendly character and low cost when compared to 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 [3]. 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 [4-6]. Epoxidized vegetable oils, for example epoxidized palm oil and epoxidized soybean oil were utilized in UV-curable coating systems [7, 8]. Vernonia oil, natural oil containing epoxide groups, was utilized as a polymerizable monomer in cationic UV-cured coatings [9].

Nowadays, there is a growing interest to produce biopolymers. The environmental problems generated by disposal of conventional plastics and other related polymers have intensified researches with polymers that have their origin from renewable resources such as plant oil, cellulose and lactic acid [10]. 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 chemical feedstock, owing to their low cost and environmental friendliness [11, 12].

Triglyceride oils are one of the most important sources for biopolymers. Triglycerides from plants, such as soy bean, 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 [13]. 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 [14]. 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. Epoxidized vegetable oil possesses functional epoxide group which can react with suitable curing agents to form an elastomeric network [15], such epoxidized vegetable oils were utilized in UV curable coating system [16, 17]. By using UV oil systems it is possible to save large amounts of organic solvents which are an important aspect with regard to environmental concern. Also the drying process which is known to be a very time-consuming one when conventional oxidative drying oils are used, with UV curing oils it can be done in not more than a fraction of seconds. Radiation curing systems are in general formulated from reactive resin and optionally, a low viscous reactive diluent. A photoinitiator is added to guarantee an immediate curing upon exposure to UV light. Recently epoxidized fatty oils and their derivatives have showed a promising effect when used as reactive resins, since the soybean contains unsaturation that can be chemically modified through simple reactions. These reactions permit to introduce polymerizable groups [18, 19] as epoxy or acrylates which are available to give products with practical useful properties and characteristics [20, 21]. The types of coatings that may be formulated from oleo-resins are clear varnishes, industrial enamels, printing inks, epoxy paints, undercoats, and primers [22].
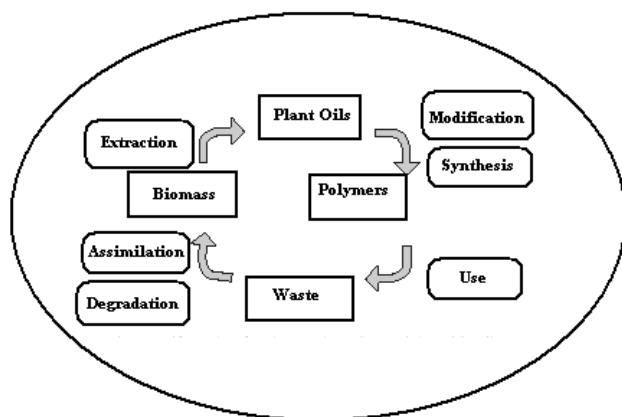
Scheme 1. Life cycle of polymers based on triglyceride oils

Epoxidized soybean oil can be used as a reactive modifier for epoxy resin systems. The epoxy groups are however available for further chemical modification reactions. The introduction of acrylate or methacrylate functions in a polymer or oligomer is generally made with the aim of polymerization or copolymerization of the double bonds leading to network or grafted copolymers. More recently, researchers have begun to explore the feasibility of manufacturing polymer composites from the epoxidized soybean oil and this is the background that motivated these studies, which investigate organic synthetic methods for epoxy functionality. The development of soy-based resins for structural applications is still a challenge for polymer and composite industries.

Radiation curable acrylates can be derived from epoxidized oils by their reacting with the acrylic acid [23]. This paper describes the development of the soybean oil based acrylated UV curable resin which is a potential for surface coating industry. Acrylated epoxidized soybean oil (AESO) has been prepared using the epoxidized soybean oil, acrylic acid, triethylamine as a catalyst, and hydroquinone as a gelling inhibitor. Prepared AESO resin was then incorporated with TMPTMA monomer along with the photoinitiator benzophenone and an activator DEA and subjected to UV irradiation for curing.

2. Experimental

2.1. Materials

Refined soybean oil was purchased from the local market. Hydrogen peroxide and formic acid were obtained from S.D.Fine Chemicals. Diglycidyl ether of bisphenol A (DGEBA) was purchased locally from Resinova. Acrylic acid and triethyl amine were purchased from E. Merck. Trimethylol propane trimethacrylate (TMPTMA) was purchased from Aldrich and benzophenone was produced by Flucka while dimethyl ethanol amine was obtained from S.D.Fine Chemicals.
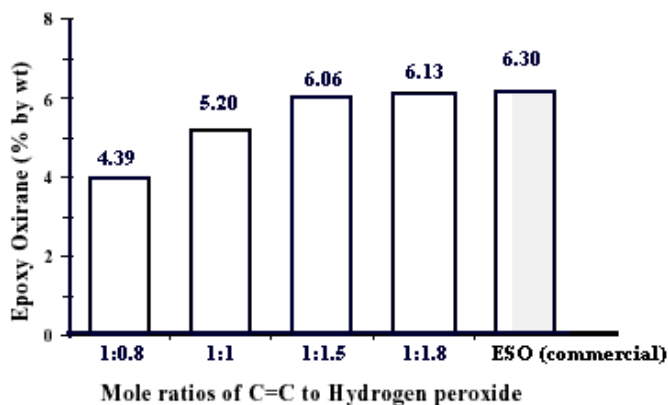
2.2. Epoxidation of Soybean Oil

On industrial scale, the oil epoxidation is carried out by the Prileshjew's reaction, where the oil double bonds react with organic peroxyacids (generally performic and peracetic). For safety reasons peroxyacids are produced in situ in the reaction vessel by acid catalyzed oxidation of the corresponding organic acid with hydrogen peroxide. Refined soybean oil (250 g) and formic acid (75 g) were charged in a three neck flask, equipped with the thermometer and dropping funnel. To start the epoxidation, hydrogen peroxide solution (50 %) was gradually charged into the mixture during first 3 h of reaction (Scheme 2). Four different molar ratios of carbon double bonds to hydrogen peroxide ($C=C:H_2O_2$) were used – 1:0.8, 1:1, 1:1.5 and 1:1.8 (Scheme 3). After the

completion of charging H_2O_2, the reaction was further continued for 4 h maintaining the temperature to 323 K. The crude product was filtered and washed with distilled water repeatedly until pH is equal to 7.0. The oil phase was dried with anhydrous sodium carbonate and then filtered.
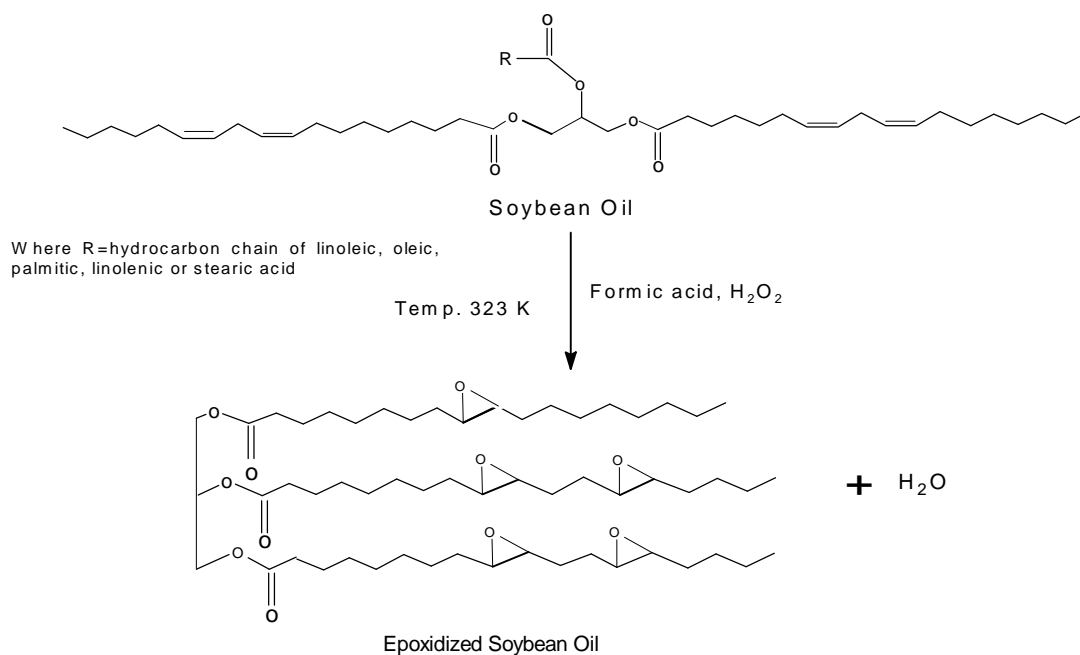
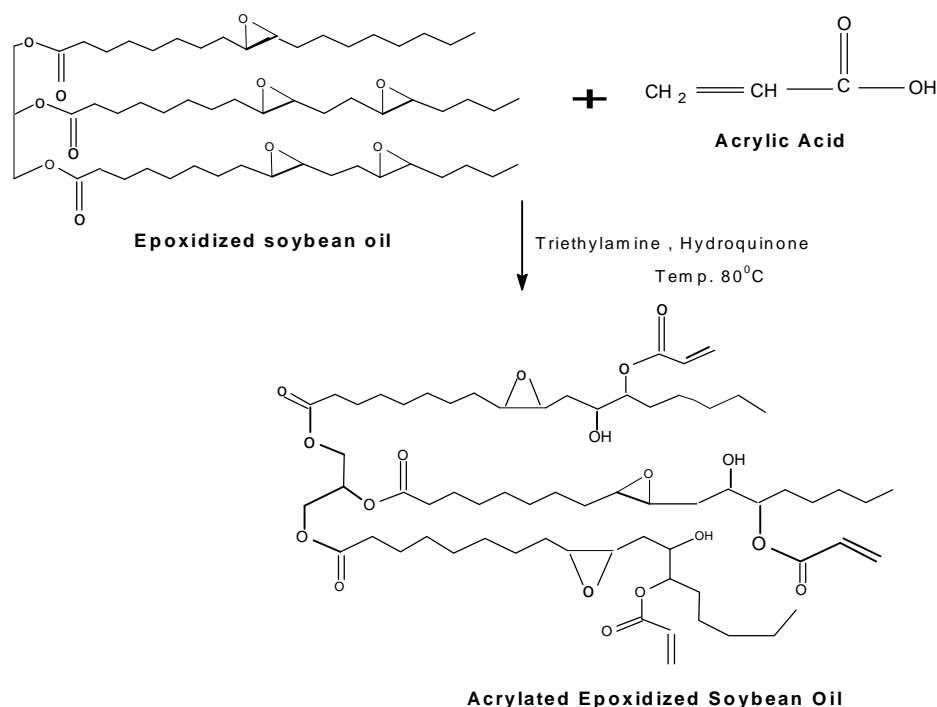
Scheme 2. Synthesis of epoxidized soybean oil

Scheme 3. Percent by weight of epoxy functional group of ESO at four different molar rations of C=C to H_2O_2

2.3. Acrylation of Epoxidized Soybean Oil

A mixture containing 300 parts by weight of prepared epoxidized soybean oil (ESO) and 49.5 parts by weight of acrylic acid was prepared in the present of triethylamine and hydroquinone as a catalyst and inhibitor, respectively. The ESO prepared from 1:1.8 ratio having oxirane oxygen of 6.13 % was used for further acrylation. The epoxidized soybean oil has an average molecular weight of about 1000, oxirane content of about 6 percents by weight and it was the epoxide of the triester of glycerol with soybean oil. The mixture was heated to 353 K for 5 h. The mechanical stirrer was maintained at 400 rpm. The reaction was monitored by measuring acid values every hour for 15 h. After synthesis, unreacted acrylic acid and catalyst were removed through water base extraction and petroleum ether (boiling point is 333–353 K). Synthesized acrylated resin then undergoes the rotary evaporator to remove the excessive petroleum ether (Scheme 4).

Scheme 4. Synthesis of acrylated epoxidized soybean oil

Table 1

Characteristics of epoxidized/acrylated epoxidized soybean oil

Samples	Appearance	Color (Gardner)	Viscosity at 298 K, mPa·s	Oxirane oxygen, %	Iodine number, mgKOH/g	Acid number, mgKOH/g
Epoxidized soybean oil (ESO)	Clear	3	550	6.13	2.5	-
Acrylated epoxidized soybean oil (AESO)	Clear	4	13000	-	-	6.2

2.4. Characterization of the Synthesized ESO

Epoxidation of fatty acids is a reaction of a carbon-carbon double bond with an active oxygen, usually from a peroxide or 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 ESO were analyzed for their percents (by weight) of epoxy functional groups by an AOCS official method Cd 9-57 (Oxirane oxygen in epoxidized materials). Iodine value was determined by cyclohexane-acetic acid method according to AOCS official method Cd 1d-92. Viscosity was measured on a Brookfield Calculating Digital Viscometer, using CP-51 cylindrical spindle at 298 K. (Table 1). Fourier Transform Infrared (FTIR) spectra were recorded by Nicolet, Magna-IR 550 Spectrophotometer.

FTIR spectra were obtained by coating the samples on potassium bromide (KBr) pellet. ^{1}HNMR spectra of the prepared ESO were obtained using Unity Inova spectrometer at 300 MHz using $CDCL_3$ as a solvent.

2.5. Characterization of the Synthesized AESO

The extent of reaction was determined by calculating the acid value [24] at definite time intervals. Viscosity was measured on a Brookfield Calculating Digital Viscometer using CP-51 cylindrical spindle at 298 K. (Table 1). Fourier Transform Infrared (FTIR) spectra were recorded by Nicolet, Magna-IR 550 Spectrophotometer. FTIR spectra were obtained by coating the samples on the potassium bromide (KBr) pellet. ^{1}HNMR spectra of the prepared AESO were obtained using Unity Inova spectrometer at 300 MHz using $CDCL_3$ as a solvent.

2.6. Preparation and Characterization of UV Curing of Coating

Formulation was developed using the DGEBA resin, acrylated epoxidized soybean oil incorporated with the monomer. Since the monomer also acts as active diluents, it is one of the basic compositions used in UV curable formulations. Most diluents used in UV curable formulations are active diluents. Active diluents are used not only to adjust the viscosity of the formulations for applications but also to take part in the UV curing process [25]. TMPTMA was used as an active diluent in this study; the resin DGEBA was mixed with specified amount of AESO, benzophenone photoinitiator was used to initiate free radical polymerization. The formulated solution was coated on clean glass plate (100x100x3 mm) by using a bar coater (RDS 20, Japan) and cured under UV lamp (2 kW; type of lamp: MC200; wavelength range: 180–450 nm) using IST UV machine (U-200M-1-Tr, Germany). The coated plate was irradiated up to 16 passes under UV lamp at a conveyor speed of 4 m/min to achieve maximum crosslinking FTIR spectra of irradiated samples recorded by Perkin-Elmer Fourier Transform Infrared Spectrometer.

3. Results and Discussion

3.1. Analysis of Synthesized ESO

The iodine value and the oxirane oxygen content are important properties in the characterization of epoxidized vegetable oils. While the iodine value indicates the remaining unsaturation after the epoxidation reaction, the oxirane oxygen content indicates the epoxy groups present in the products. In the preparation of polymers, epoxy resins with a lower iodine value and higher oxirane oxygen content are desired. 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 ESO is 2.5. The highest epoxy content synthesized epoxidized soy oil (ESO) was 6.13 % and it is almost comparable to the commercial ESO (6.3 %). Scheme 5 shows the reaction conversion of ESO where the amount of H_2O_2 is varied. Theoretically 1 mole of H_2O_2 will epoxidize one mole of C=C. When the ratio of H_2O_2:C = C is increased from 59.8 to about 80 % the reaction conversion of epoxidized soy oil increases.

The unsaturation and epoxy groups can also be monitored by the Fourier transform infrared (FTIR) spectroscopy. The FTIR spectroscopy is a rapid, nondestructive technique that has been widely applied in the charcterization of lipids because lipids have functional
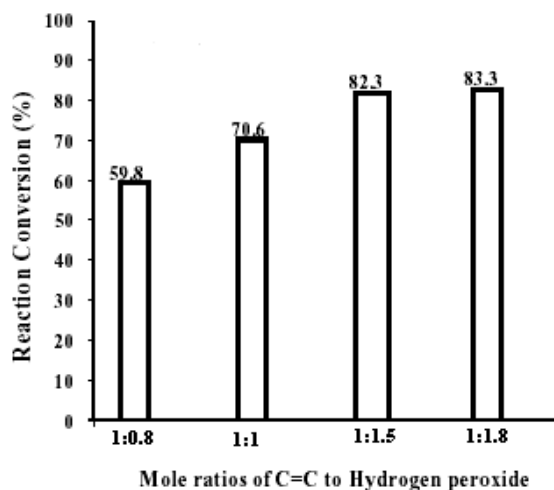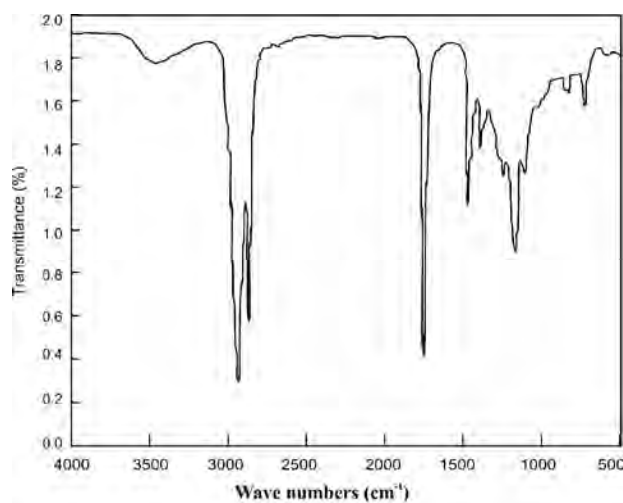

Scheme 5. Conversion of ESO
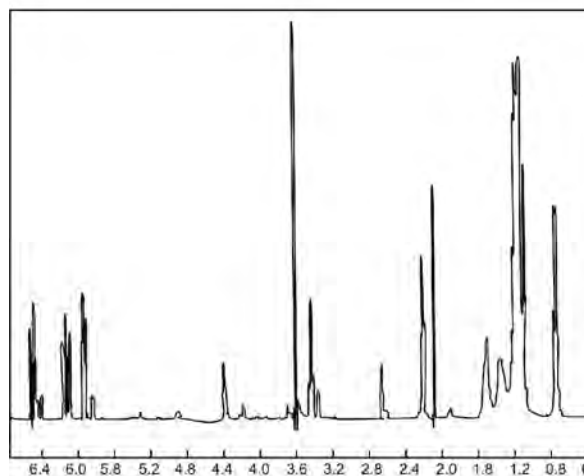

Fig. 1. FTIR Spectrum of ESO

Fig. 2. ^{1}H NMR of ESO

groups with characteristic absorption bands in the infrared region of the electromagnetic spectrum [26, 27]. In FTIR spectrum of ESO (Fig. 1), C–H stretch is observed at 3009 cm^{-1}. C–O–C stretching from oxirane vibration appears at 1250 cm^{-1} and 830–850 cm^{-1}. The most representative signal that evidences the oxirane group is the small intensity one sited at 830 cm^{-1}.

^{1}H-NMR spectra were recorded with Varian Unity Inova spectrometer at 300 MHz using CDCL$_3$ as a solvent. All the data are given as chemical shifts δ (ppm).The signal at 5.25–5.55 ppm corresponding to the protons of the glycerol center (H$_b$) and to the protons of the vinyl groups (H$_8$, H$_9$, H$_{11}$, H$_{12}$, H$_{14}$, H$_{15}$) of soybean oil. A multiplet in the region of 2.92 corresponds to the protons of the epoxy group (H$_8$ and H$_9$) and decreases after epoxidation of all natural oils (Fig. 2).

3.2. Analysis of Synthesized AESO

The esterification reaction takes place between the epoxidized soybean oil and acrylic acid in the presence of triethyl amine, the epoxy group reacts with the carboxyl ionic group to form additional polymeric hydroxy ester. Epoxide resins react with carboxylic acid to form esters. The reaction takes place between the carboxyl group of the acid and both the epoxy and hydroxyl groups of the epoxides. Tertiary amine interacts with acrylic acid and removes its hydrogen as cation thus producing a corboxylate anion, this carboxylate anion undergoes to 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 acrylic acid get exhausted which results in the formation of epoxy acrylate. The degree of esterification is an important parameter that may affect the coating property, and in the present study the reaction was carried out until the acid value reached less than 6 mgKOH/gm. The change in the acid value with the reaction time occurs because of the higher concentration of reactive sites and greater possibility of acid and epoxide groups association.

The FTIR spectroscopy of AESO shows that the hydroxyl group is successfully converted to the acrylated functionality through condensation esterification. This is indicated from the presence of hydroxyl functionality of resin at absorption band of 3412 cm^{-1}, associated with hydrogen bond of –OH and a strong absorption band for the carbonyl group, C=O of the acrylated compound at 1737 cm^{-1}. The tiny absorption band of –OH in the acrylated prepolymers might have come from the minute amount of moisture present in acrylated resin after washing and purification process. Another significantly different absorption band is observed in the spectrum at 1637 cm^{-1} indicating the presence of vinyl functionality

(–CH=CH$_2$–). The presence of vinyl functionality of the acrylated polymer is also supported by the absorption peak at 986 cm^{-1} as this indicates the presence of vinyl group, [CH$_2$=CH (CO)–O] depicted in Fig. 3.

This was also confirmed by ^{1}H-NMR spectrum of the AESO, the epoxy groups give signals around 3 ppm, and in the modified resin samples they disappear, and subsequently new signals are present on its chain confirming modification after acrylation (Fig. 4).
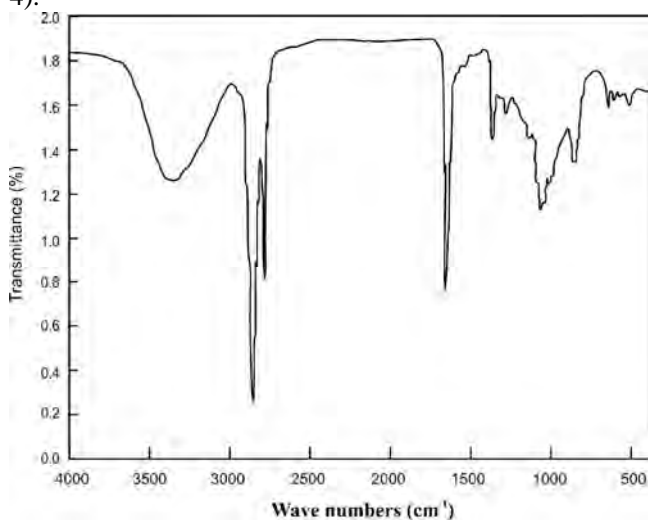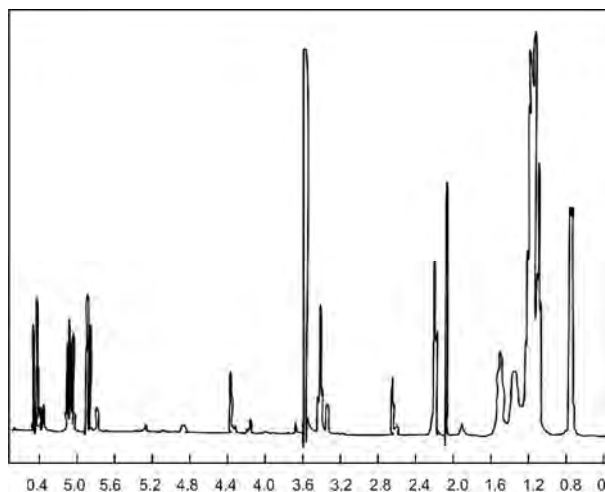

Fig. 3. FTIR Spectrum of AESO

Fig. 4. ^{1}H NMR of AESO

## 3.3. Preparation of UV Curable Coating Composition

The acrylated epoxidized soy oligomer was taken in a beaker and after addition of stoichiometric amount of reactive diluent and photoinitiator it was stirred at room temperature. The amount of each ingredient used in formulation is shown in Table 2.
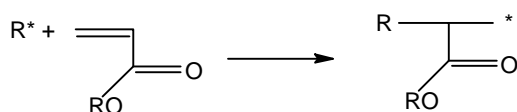
Table 2

Composition of UV Curable Coatings

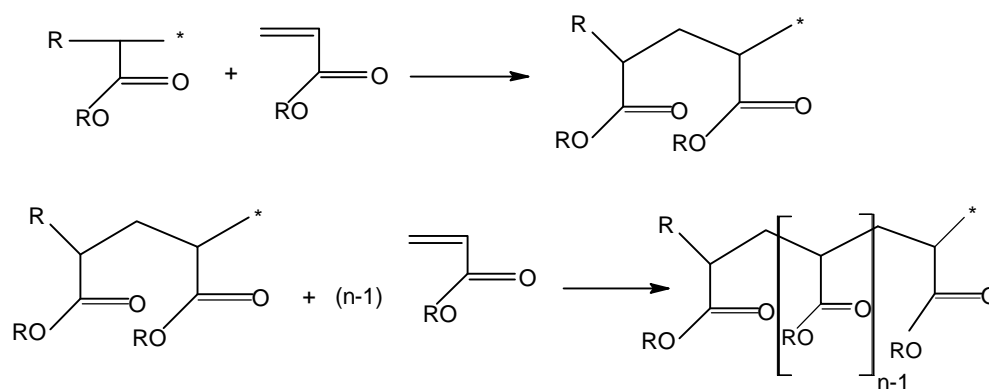| No.	Ingredient	Weight, %	Function
1	DGEBA	50	Oligomer
2	AESO	20	Biobase additive
3	TMPTMA	25	Reactive diluent as crosslinking monomer
4	Benzophenone	3.50	Photoinitiator
5	Dimethyl amino ethanol	1.50	Activator/Catalyst
	Total	**100**	

Initiator radical formation

PI $\xrightarrow{\text{UV light}}$ PI*   PI = photoinitiator

PI* $\longrightarrow$ R_1* + R_2*   R = radical
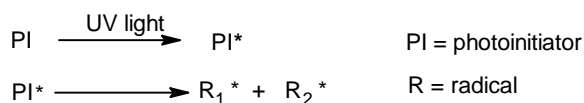
Initiation

Propogation

Termination
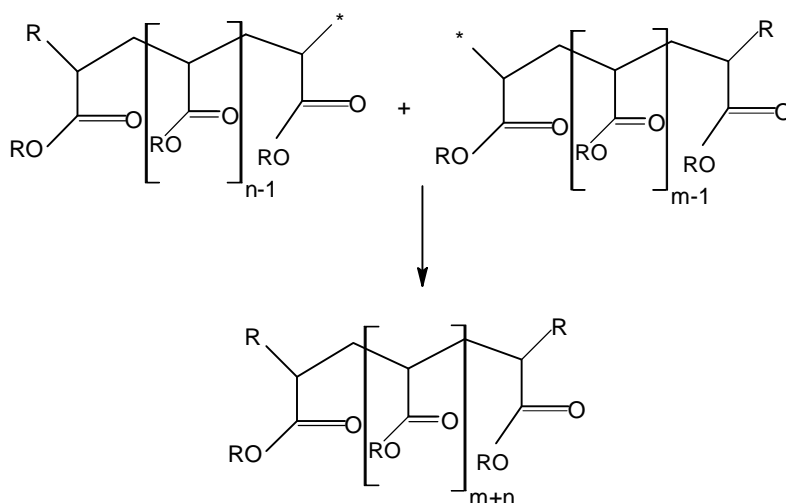
Scheme 6. Mechanism path of free radical polymerization

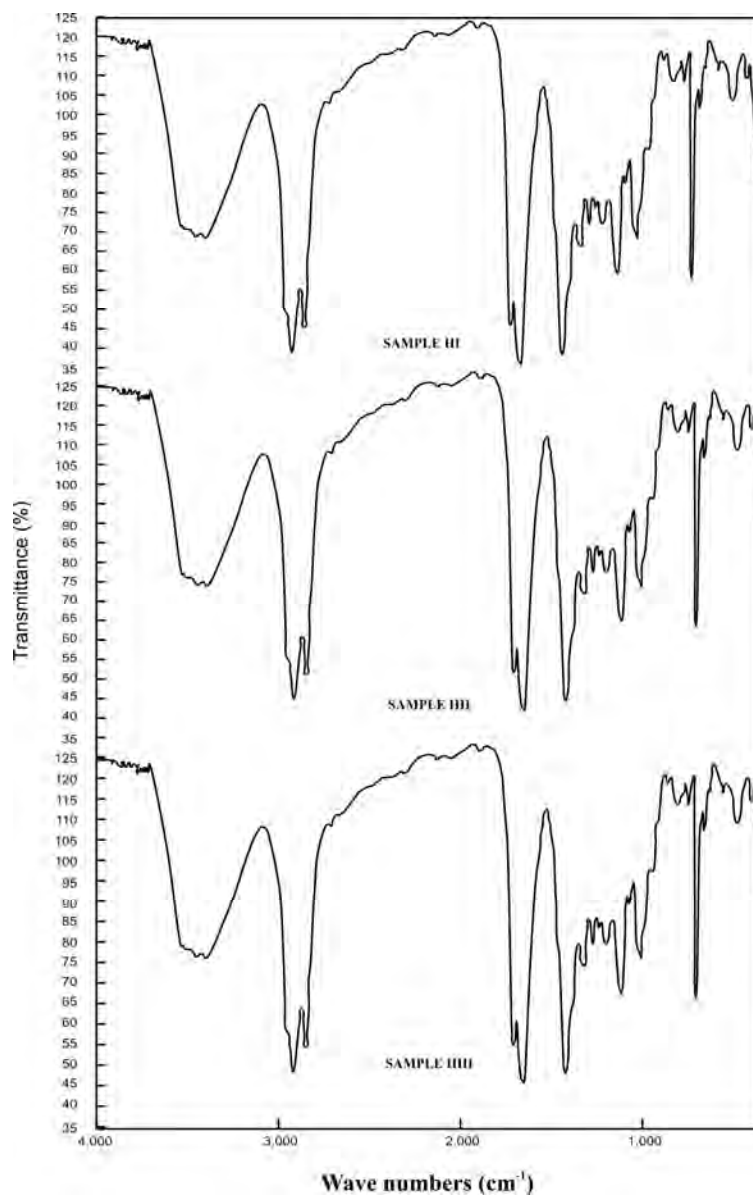*Firdous Habib et al.*

Fig. 5. FTIR spectra of UV cured films

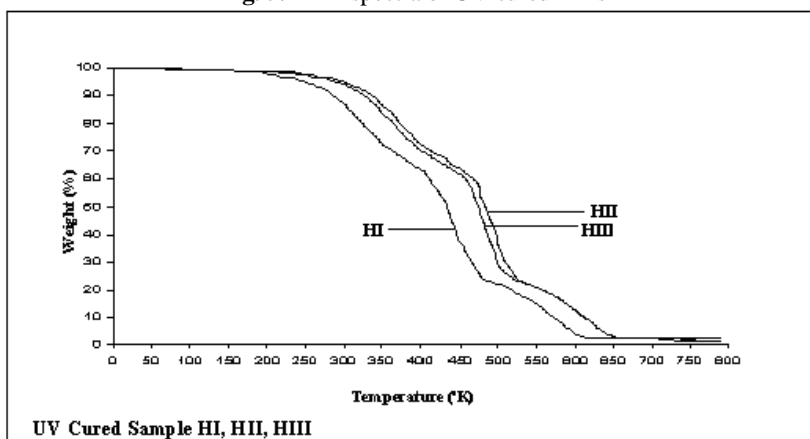UV Cured Sample HI, HII, HIII

Fig. 6. TGA of UV cured AESO films

3.4. Application and Curing of Coating Composition

Three samples, namely HI, HII and HIII were prepared having the formulation depicted in Table 2. Theses samples (HI, HII and HIII) to be tested for UV curing were coated onto MS steel test panel (15x5 cm). An excess of the sample was placed at one end of the test panel and using a rod applicator (K-Bar No.5) drawn across the substrate with even pressure pushing excess material off the edge. The coated panel was exposed to a (200 Watt/inch) (280–360nm) medium pressure mercury lamp of UV-2KW-2-35 curing system. This method produced coating with average film thickness of 23–35 μm.

3.5. Analysis of UV Cured Coating

The extent of UV curing and the liquid-solid phase transition occuring upon UV irradiation via free radical polymerization (Scheme 6) can thus be monitored through FTIR spectra of UV cured films (Fig. 5).

The disappearance of the reactive double bond upon UV radiation can be followed by monitoring the infrared absorption characteristics of the carbon–carbon (C=C) double bond stretching at 1660–1600 cm^{-1}. Before UV radiation, FTIR spectrum of acrylated polymer shows unsaturation at 1637 cm^{-1}. It can be seen after UV irradiation, infrared absorption characteristics of the C=C stretching at around 1660–1600 cm^{-1} disappear although very tiny absorptions can be seen. This may be due to the presence of a certain amount of double bonds still remaining in the film substrate, perhaps from the prepared prepolymers or monomers used in the formulation. It is interpreted to mean that some residual acrylic and vinylene double bonds may be trapped and unable to react in the three-dimensional polymer matrix formed [28, 29]. When crosslinking reaction occurred, the C=C bonds were consumed. With 4 % of photoinitiator all the peaks show the higher level of conversions as the degree of crosslinking density has been increased.

TGA thermograms of cured films are shown in Fig. 6. TGA thermogram clearly indicates good thermal stability of the cured film up to 373 K with a minimum amount of weight loss, which may be due to the minimum amount of unreacted components. The initial decomposition temperatures of all the cured films are about 473 K. This indicates that the cured film has a good thermal stability.

4. Conclusions

The UV curable coating compositions were prepared using Acrylated Epoxy Soybean Oil as a renewable source. The resin was UV curable after incorporation of the photoinitiator and reactive diluent, showing a good rate of conversion which has been confirmed by FTIR spectral analysis. The saturated portion of the triglycerides provides the medium to hold the polymer network and –CH_2CH_3– groups of the end of the fatty acid chains and play a very significant role in delocalization of electrons around the double bond during free radicals production by the impact of radiation thus providing a cured film. UV cured films exhibit good thermal stability with a decomposition rate of about 473 K.

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СИНТЕЗ ТА ХАРАКТЕРИСТИКА АКРИЛОВАНОЇ ЕПОКСИДОВАНОЇ СОЄВОЇ ОЛІЇ ДЛЯ УФ-ЗАТВЕРДЖЕНОГО ПОКРИТТЯ

Анотація. *В роботі досліджено затвердіння біодег-радабельних полімерних плівок, синтезованих з соєвої олії за допомогою ультрафіолетового випромінювання, і їх стійкість до термічного розкладання. Епоксидування соєвої олії проводилось за допомогою надоцтового методу. Синтезовано епоксидну акрилатну смолу з епоксидованої соєвої олії з використанням акрилових мономерів кислоти. Як каталізатор та інгібітор відповідно використано триетиламін і гідрохінон. Акрилування епоксидованої соєвої олії проводилось внаслідок введення акрилової кислоти, яка є ненасиченим мономером, в оксиранові групи олії. Перебіг реакції підтверджено аналітичними даними стосовно вмісту оксиранового кисню, кислотним числом, в'язкістю і спектральними методами аналізу. Досліджено різні УФ-затверджені композиції, використовуючи синтезовані акрилатні смоли і трифункціональні акрилатні мономери як активні активні розчинники, зшиті з фотоініціатором. Як мономер використовували триметилолпропан триметилакрилату, а як фотоініціатор – бензофенон. Активатором слугував диметилетаноламін. Суміші були затверджені під ультрафіолетовим випромінюванням з оптимальним дозуванням опромінення 20 хв. для одержання тонких полімерних плівок, які можуть бути викорситані для виробництва термостійких (вище 473 К) покриттів.*

Ключові слова: *епоксидування, акрилування, УФ-затвердіння, покриття.*