

## Synthesis of Diethylamine-Functionalized Soybean Oil

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Specialty chemicals based on renewable resources are desirable commodities due to their eco-friendly nature and “green” product characteristics. These chemicals can demonstrate physical and chemical properties comparable to those of conventional petroleum-based products. Suitably functionalized amines in the triacylglycerol structure can function as an antioxidant, as well as an antiwear/antifriction agent. In addition, the amphiphilic nature of seed oils makes them an excellent candidate as base fluid. The reaction of amine and epoxidized seed oils in the presence of a catalyst almost always leads to different intra/intermolecular cross-linked products. In most cases, the triacylglycerol structure is lost due to disruption of the ester linkage. Currently, there is no reported literature describing the aminolysis of vegetable oil without cross-linking. Here the epoxy group of the epoxidized soybean oil has been selectively reacted with amines to give amine-functionalized soybean oil. The optimization procedure involved various amines and catalysts for maximum aminolysis, without cross-linking and disruption of the ester linkage. Diethylamine and ZnCl<sub>2</sub> were found to be the best. NMR, IR, and nitrogen analysis were used to characterize the products.

**KEYWORDS:** Vegetable oils; epoxidized soybean oil; epoxy ring opening; diethylamine; zinc chloride; aminol preparation

### INTRODUCTION

Due to growing public concern and strict government regulations over oil pollution during the past decade, there has been a constant demand for environmentally friendly lubricants (1). Most lubricants originate from petroleum stock, which is toxic to the environment and difficult to dispose of. Consequently, lubricants prepared from biodegradable resources such as vegetable oils are receiving considerable interest as natural alternatives to synthetic lubricants, and the development and application of biobased lubricants in the industrial and automotive sectors are rapidly increasing.

Vegetable oils with high oleic content are potential substitutes for conventional petroleum-based lubricating oils and synthetic esters (2, 3). Vegetable oils are preferred as lubricants over synthetics because, unlike mineral-based oils, they are biodegradable, nontoxic, renewable, and relatively inexpensive (2, 4).

As lubricants, vegetable oils have very low volatility due to the high molecular weight of the triacylglycerol molecule, and they have a narrow range of viscosity changes with temperature. Polar ester groups enable the molecules to adhere to metal

surfaces, and thereby impart good boundary lubrication to machined parts. In addition, vegetable oils have high solubilizing power for polar contaminants and additive molecules. For example, a biobased lubricant with improved high- and low-temperature stability was prepared by chemical modification of epoxidized soybean oil (ESBO) (5).

On the other hand, vegetable oils have poor oxidative stability (5, 6), primarily due to the presence of bisallylic protons. They are highly susceptible to radical attack and subsequently undergo oxidative degradation to form polar oxy compounds. This phenomenon often leads to an increase in oil insoluble deposits, oxygenates (containing acidic functionality) and oil viscosity. Vegetable oils also show poor corrosion protection (7). The presence of an ester functionality renders these oils susceptible to hydrolytic breakdown (8). Therefore, contamination with water in the form of emulsion must be prevented at every stage. Low-temperature studies have also shown that most vegetable oils undergo cloudiness, precipitation, poor flow, and solidification at  $-10\text{ }^{\circ}\text{C}$  upon long-term exposure to cold (9, 10), in sharp contrast to mineral oil-based fluids.

We report a novel synthetic approach for the chemical modification of vegetable oils to improve their thermo-oxidative and low-temperature stabilities. The chemical modification is carried out by epoxy ring opening of the epoxidized soybean oil by organic amines. Separate studies are in progress to determine the physical and performance properties of the products developed with optimized process. Scale-up process

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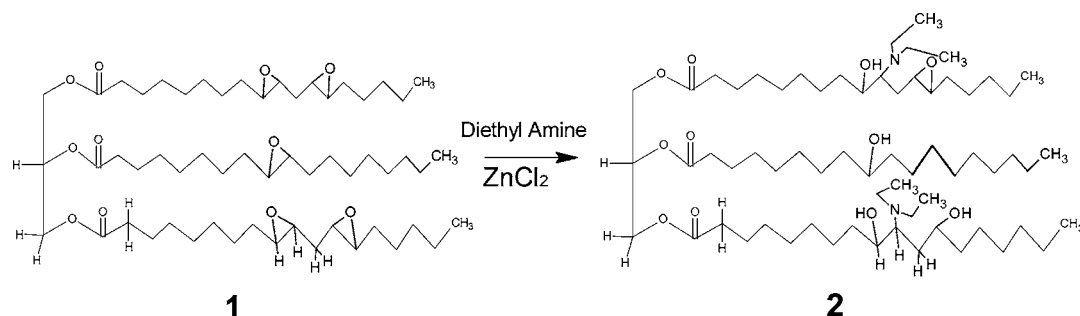


Figure 1. Reaction of ESBO with diethylamine.

and cost calculations are outside the scope of this paper. These amine derivatives may serve the dual function of antioxidant and antiwear specialty chemicals in automotive and industrial applications.

## MATERIALS AND METHODS

**Synthesis of Diethylamine-Functionalized Soybean Oil from Epoxidized Soybean Oil.** Epoxidized soybean oil (1.9 g, 2 mmol of Vikoflex 7170 Epoxidized Soybean Oil, Atofina Chemicals, Inc.) was dissolved in 1.5 mL (14.3 mmol) of diethylamine (Sigma-Aldrich, 99.5+%) in a round-bottom flask (100 mL, Kontes), and then 0.3 g (2.2 mmol) of zinc chloride (Sigma, 98%) was added. The temperature of the solution was kept constant at 80 °C by means of a Therm-O-Watch (I2R, L14-1800) and was stirred while heated for 4 h. After heating, the solution was allowed to cool to room temperature and evaporated in a rotovap (Büchi, R-200) at 60 °C. Ethyl acetate (50 mL, Fisher Scientific) was used to dissolve the oil, and then the solution was transferred into a 125 mL separatory funnel. The zinc chloride was removed with three washes, each of 50 mL of saturated sodium bicarbonate solution. Finally, the organic layer was washed three times each with 50 mL of Nanopure water and saturated sodium chloride solution (J. T. Baker). The organic layer was dried with  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (Fisher Scientific) and filtered (Whatman, no. 1, 55 mm  $\varnothing$ ), and the effluent was evaporated for a minimum of 30 min at 60 °C under vacuum to give oil in 80% yield. Any unreacted diethylamine was distilled off under this condition.

**NMR Analysis.** All of the  $^1\text{H}$  NMR spectra were recorded quantitatively using a Bruker ARX-400 spectrometer (Bruker, Rheinstetten, Germany) at a frequency of 400 using a 5 mm dual probe. Sample solutions were prepared in deuterated chloroform ( $\text{CDCl}_3$ , 99.8% D, Aldrich, Milwaukee, WI) in 15 v/v concentrations. Each spectrum was aligned internally with respect to TMS at 0.0 ppm. Fourier transformation, phase correction, peak picking, and integration were all performed with MestRe-C NMR software (MestRe-C, Santiago de Compostela). After the integration was performed, the integration values were referenced to 4.00 between 4.29 and 4.15 ppm.

**FTIR Analysis. Preparation of FTIR Test Samples.** Test samples were prepared to obtain infrared spectra of reagents and products of the reaction between diethylamine and epoxy soybean oil at various levels of catalyst. Reaction products were viscous yellow/amber oils extracted from the reaction mixtures by washing with Nanopure water and ethyl acetate as described above. Diethylamine was tested as received in reagent grade. The  $\text{ZnCl}_2$  catalyst was dried and ground to a fine powder by ball-milling (Brinkman Instruments, Inc., Westbury, NY) in sealed stainless steel vials under liquid nitrogen.

Test samples of the reaction products were pressed between two KBr disks (25 mm  $\times$  5 mm) to give thin transparent oil films for analysis by FTIR spectrometry. Diethylamine was tested neat in sealed liquid cells with KBr windows with 0.05 mm spacing. The  $\text{ZnCl}_2$  powder was pulverized with KBr and pressed into transparent disks for FTIR analysis. A sample of ground powder (2.66 mg) was mixed with KBr (798 mg) and pulverized at liquid nitrogen temperature in a sealed stainless steel vial containing a stainless steel ball bearing for 10 s on a Wig-L-Bug amalgamator (Crescent Dental Manufacturing, Lyons, IL). The vial was allowed to warm to room temperature before 300 mg of the pulverized  $\text{ZnCl}_2$ -KBr mixture was transferred to a

KBr die (Perkin-Elmer Corp., Norwalk, CT) and pressed for 30–60 s under vacuum at 110 MPa on a laboratory press (Fred S. Carver, Menominee Falls, WI).

FTIR spectra were measured on a Bomem Arid Zone spectrometer (Bomem, Cambridge, CT) equipped with an air-cooled detector. Absorbance spectra were acquired at 4  $\text{cm}^{-1}$  resolution and signal-averaged over 32 scans. Interferograms were Fourier transformed using cosine apodization for optimum linear response. All spectra were baseline corrected and normalized to the average of the methylene peaks at 2927 and 2855  $\text{cm}^{-1}$ . An FTIR spectrum typical of the reaction products is shown in Figure 5. FTIR spectra of the diethylamine reagent and  $\text{ZnCl}_2$  catalyst (data not shown) were used as controls in interpreting the spectra of reaction products.

**Spectral Peak Integration.** Peak integration was performed with a routine provided in GRAMS/AI software (Galactic Industries, Salem, NH). The area of the epoxy (oxirane) absorbance band, which existed in the oils as two overlapping peaks centered at 846 and 824  $\text{cm}^{-1}$ , was measured above a drawn baseline connecting the two minimums between 880 and 790  $\text{cm}^{-1}$ . The inset in Figure 5 shows the difference between the integrated band areas of the unreacted epoxy soybean oil and the reacted oil.

**Pressurized Differential Scanning Calorimetry (PDSC).** A DSC 2910 thermal analyzer from TA Instruments (New Castle, DE) was used for the experiments. A 2  $\mu\text{L}$  sample (resulting in a <1 mm film thickness) was taken in an aluminum pan hermetically sealed with a pinhole lid and oxidized in the presence of air. Dry air (Gateway Airgas, St. Louis, MO) was pressurized in the module at a constant pressure of 1378.95 kPa (200 psi), and a 10 °C/min heating rate was used during the test duration. The onset temperature (OT) and signal maximum temperature (SM) of oxidation were calculated from the exotherm in each case. Subambient DSC experiments were done using the same above apparatus. Before each experiment, the DSC cell was purged with low-pressure nitrogen gas. A 10 mg oil sample was accurately weighed in an open aluminum pan and placed in the DSC module with a similar empty pan as reference. The procedure involved rapidly heating the sample to 80 °C and then holding the sample under isothermal condition for 10 min. This helps in dissolving and homogenizing any waxy material present in the oil, which may inadvertently act as seed to accelerate wax crystal formation during cooling. The system was then cooled to -40 °C at a steady rate of 5 °C/min using liquid nitrogen as the cooling medium. The heat flow (W/g) versus temperature for each experiment was analyzed to determine wax appearance temperature (WAT, °C). The average value of three independent measurements was taken in each case.

Nitrogen contents were measured in duplicate with a Leco CHN-2000 analyzer, using EDTA as a standard.

## RESULTS AND DISCUSSION

The removal of unsaturation in the soybean oil by converting it to epoxy groups, **1**, significantly improves the thermal and oxidative stability of the oil (*11*, *12*). It has already been established that the presence of multiple double bonds in the vegetable oil fatty acid chains accelerates oxidative degradation (*11*). Our goal was to open the epoxy ring of **1** of ESBO by various amines without cross-linking. As amine groups are

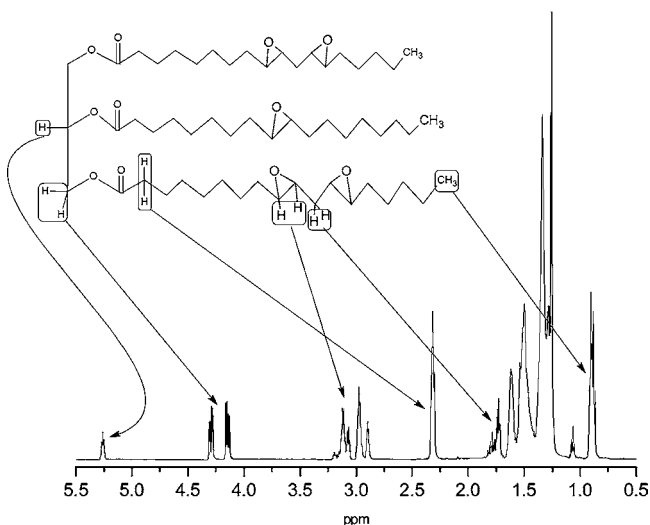


Figure 2. Proton NMR of ESBO.

effective antioxidants, amine functionalization may enhance the oxidative stability and lubrication properties of soybean oil (13–15). Thus, an epoxy ring opening by amine is an effective way of introducing branching on the fatty acid chain of vegetable oils and the resultant aminol, **2**, may possess significantly improved thermal, oxidative, and low-temperature stability compared to unmodified soybean oil.

Moreover, reaction of the amine group with the epoxy group is much more facile as compared with the acid-catalyzed ring opening of epoxy. This higher reactivity of amines is due to the higher nucleophilicity of amine as compared to the hydroxyl group of alcohol or water. Curing of epoxides by amines is a

very useful reaction, and the cross-linked products have found major commercial applications as epoxy glues in the adhesive industry and composites in the aerospace industry (16). However, for our purpose cross-linking is undesirable. Unless we control the reaction of amines with ESBO, the cross-linked product will be useless as a grease or lubricant.

Initial attempts to use other Lewis acid catalysts such as  $\text{BF}_3$  with sterically hindered amines at elevated temperature have resulted in high-viscosity polymeric products, which were a result of amine-initiated cross-linking reactions. These catalysts, which are stronger Lewis acids than  $\text{ZnCl}_2$ , complexed with the epoxy oxygen too strongly and, thus, activated the epoxy group more than desired. This enhanced electrophilicity of the two carbons of epoxy rings led to higher reactivity and cross-linking reaction. It was found (17) that  $\text{ZnCl}_2$  was the right type of catalyst for our purpose to carry out the desired reaction as drawn in Figure 1. There are reports (18, 19) of ESBO epoxy group opening by amines. However, in both of these reports it was unclear whether the amine reacted with epoxy or the ester groups. The ester groups are equally reactive, and the product may have been amide. Conclusions were based on IR spectra, which are not able to unequivocally distinguish between amide and aminol when both are present in the product.

To optimize the reaction conditions, five reaction parameters were varied. The parameters were catalysts, amines, reaction time, reaction temperature, and concentration. We found that  $\text{ZnCl}_2$  was the most effective catalyst in opening the epoxy rings of ESBO. We used diethylamine, *n*-propylamine, *tert*-butylamine, and isobutylamine. Diethylamine gave the best result. Initially, ESBO was reacted with diethylamine/ $\text{ZnCl}_2$  in refluxing acetonitrile and methylene chloride for 5 h at 82 °C.

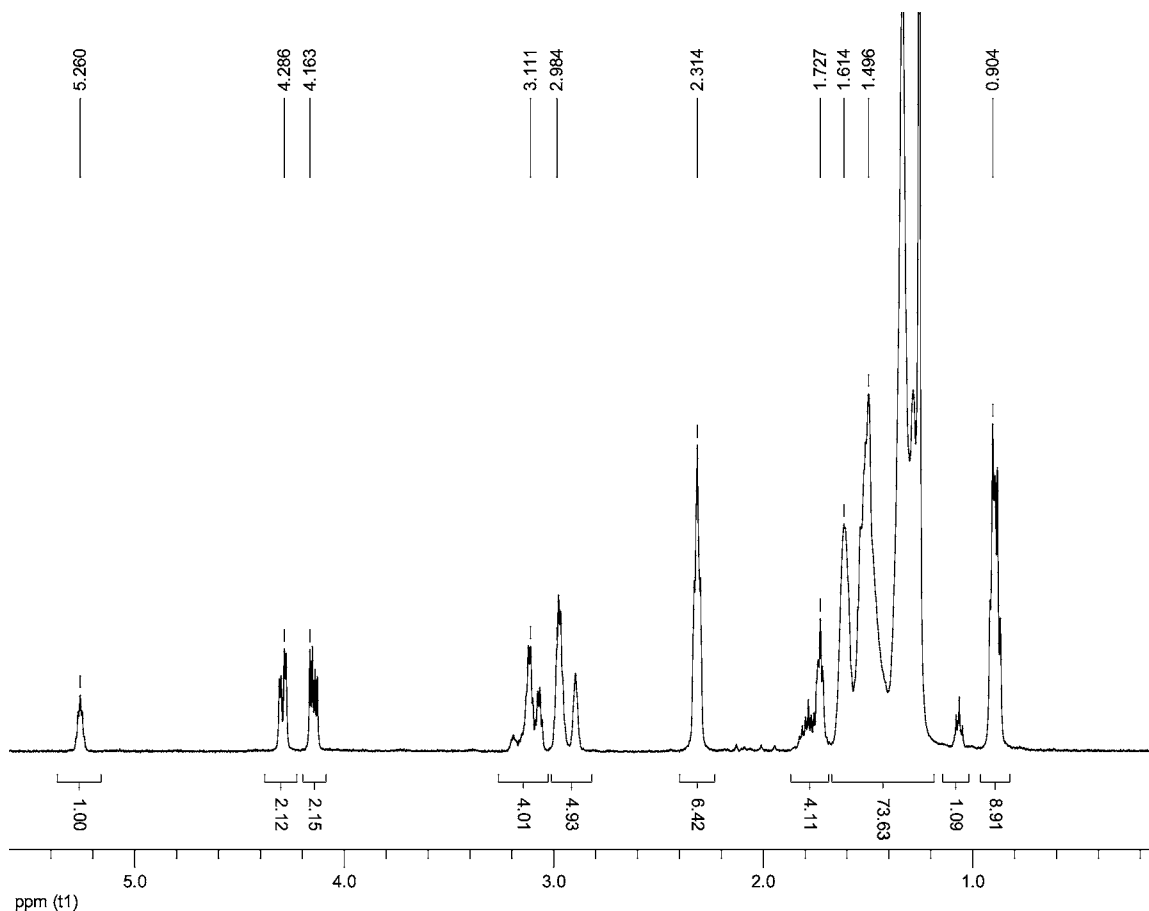


Figure 3. Proton NMR of ESBO with proton area integration.

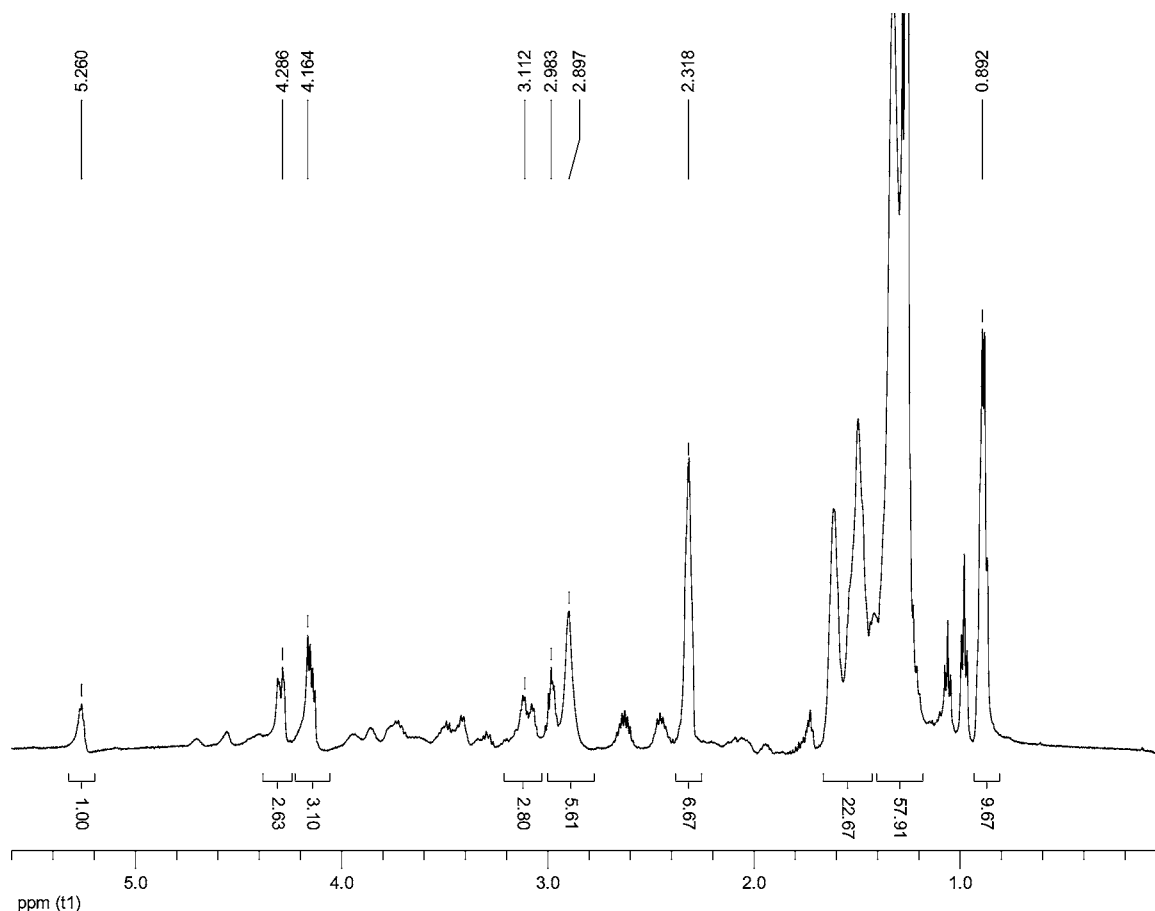


Figure 4. Proton NMR of amine-functionalized soybean oil.

However, those conditions were proved to be too mild, and the ESBO did not react. We turned our attention to a more practical solventless method to carry out the reaction neat, without the acetonitrile or methylene chloride as solvent. Thus, ESBO, diethylamine, and  $\text{ZnCl}_2$  were reacted at  $90^\circ\text{C}$  for 4 h. On average, each mole of ESBO has 4.5 mol of epoxy groups. Thus, in principle, the maximum amount of amine that should be used is 4.5 mol per mole of ESBO. We varied the amine amount from 5 to 8 mol for each mole of ESBO. Thus, 8 mol of diethylamine and 1.1 mol of  $\text{ZnCl}_2$  for each mole of ESBO was optimum and opened about 1.5 epoxy groups. However, residual epoxy group and hydroxyl remained in the product. The nitrogen content of 1.97% corresponds to opening of about 1.5 epoxy groups. Thus, only 1.5 of 8 mol of amine reacted.

$^1\text{H}$  nuclear magnetic resonance measurements on **1** indicate that the epoxy group protons appear in the  $\delta$  3.0–3.2 region (Figures 2–4), the methane proton of the  $-\text{CH}_2\text{CHCH}_2-$  backbone at  $\delta$  5.1–5.3, the ethylene proton of the  $-\text{CH}_2\text{CHCH}_2-$  backbone at  $\delta$  4.2–4.7, the  $\text{CH}_2$  proton adjacent to two epoxy groups at  $\delta$  2.8–3.0 ppm, the  $-\text{CH}-$  protons of the epoxy ring at  $\delta$  3.0–3.2, the  $\alpha\text{-CH}_2$  to  $>\text{C}=\text{O}$  at  $\delta$  2.2–2.4, the  $\alpha\text{-CH}_2$  to epoxy group at  $\delta$  1.7–1.9, the  $\beta\text{-CH}_2$  to  $>\text{C}=\text{O}$  at  $\delta$  1.55–1.75, the  $\beta\text{-CH}_2$  to epoxy group at  $\delta$  1.4–1.55, the saturated ethylene groups at  $\delta$  1.1–1.4, and the terminal  $-\text{CH}_3$  groups at  $\delta$  0.8–1.0 region. Figures 3 and 4 show that product **2** retains most of the characteristic peaks of ESBO **1** except the protons at the  $\delta$  2.8–3.2 and 1.4–1.55 regions, corresponding to protons attached to epoxy groups and ethylene groups adjacent to the epoxy group, respectively. We also see two multiplet peaks at  $\delta$  0.9–1.2 corresponding to the ethyl groups of diethylamine. As the diethylamine opened the epoxy ring we saw reduction of protons at  $\delta$  3.2 corresponding to the

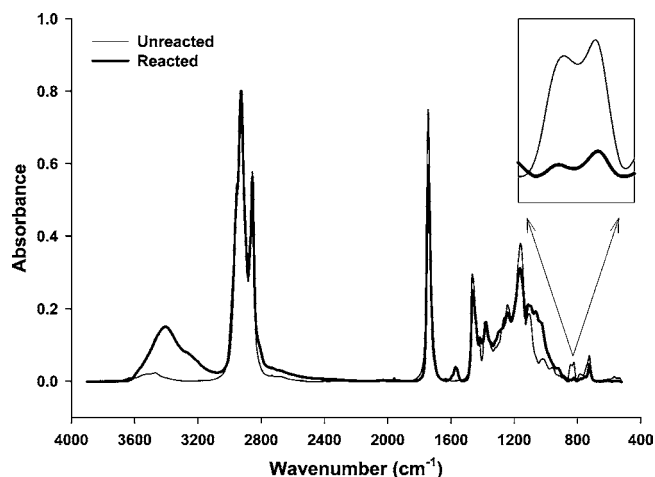


Figure 5. IR spectra of the ESBO and amine-functionalized soybean oil.

protons attached to the epoxy groups. By comparing the proton count (by integration) of this peak to the protons at  $\delta$  4.2–4.7 for the terminal ethylene group of the backbone, we determined the extent of epoxy ring opening.

The FTIR spectra confirmed the  $\text{ZnCl}_2$ -catalyzed reaction of diethylamine with epoxy soybean oil. As seen in Figure 5, the reacted product spectrum shows a decrease in the epoxy (oxirane) band ( $846\text{--}824\text{ cm}^{-1}$ ) compared to the unreacted oil, and a new broad band appears (centered at  $3250\text{ cm}^{-1}$ ), attributed to the addition of hydroxyl groups in the product. These changes are due to opening and elimination of oxirane rings by diethylamine. A new peak at  $1570\text{ cm}^{-1}$  indicated possible carboxyl salt formation in this reaction. Also, the spectrum of the reaction product shows a concomitant decrease

**Table 1.** PDSC Data of SBO, ESBO, and Amine-Functionalized Products at 10 °C/min in Air under 200 psi Pressure

test fluid	onset temp (OT), °C	signal max temp (SM), °C	wax appearance temp (WAT), °C
SBO	134	180	-12
ESBO	198	223	-10
amine functionalized-1	182	242	-3
amine functionalized-2	184	213	-7

<sup>a</sup> SBO, soybean oil; ESBO, epoxidized soybean oil; OT and SM values are average of three experiments with standard error of  $\pm 1$  °C.

in the height of the ester carbonyl band at 1743  $\text{cm}^{-1}$ . The broadening of the band around 2730  $\text{cm}^{-1}$  is the result of intermolecular H-bonding involving hydroxyl and amine functional groups. This phenomenon also causes an increase in viscosity of the reaction product as obtained.

In addition to confirming the reaction occurred, an objective of FTIR analysis was to measure the reaction efficiency by estimating the fraction of unreacted epoxy groups remaining in the product oil. From the spectral peak integrations in **Figure 5** it was observed that the area of epoxy band in the spectrum of reaction product is markedly reduced. The product oil **2** retained 11.6% (area ratio = 0.2131/1.8351) of the original epoxy groups. Therefore, the oxirane opening reaction was evidently 88.4% complete. However, due to competing hydrolysis of the epoxy groups, the aminolysis reaction efficiency could not be estimated from the FTIR spectral integrations. This was accomplished instead by elemental nitrogen analysis, which indicated  $\sim 33\%$  of the epoxy groups were converted to aminol, which is in good agreement with the extent of epoxy ring opening determined by  $^1\text{H}$  NMR. The total acid number (TAN) of the starting material and the product mixture indicates the presence of free fatty acids and carboxylic acids generated during the reaction. Soybean oil has a TAN value of 0.57 mg of KOH/g of oil, whereas ESBO and the reaction mixture showed 0 TAN. Due to the steric geometry of the ESBO molecule, complete conversion to aminol product could not be achieved. Proton NMR spectra also illustrate the fact that unreacted epoxy groups are retained in the product mixture.

PDSC measured the oxidation stability of amine-functionalized oil samples. Data presented in **Table 1** illustrate the improvement in oxidation stability of amine derivative compared to ESBO and soybean oil. DSC is widely used in the lubricant industry and is considered to be a reliable method for evaluating base oils as well as finished lubricants (20). The PDSC results, OT and SM, for modified oil samples are shown in **Table 1**. The amine-functionalized derivatives show higher oxidation stability than SBO as a result of the removal of poly-unsaturation from the SBO molecular structure. During epoxidation (98% + epoxy), three-membered oxirane rings are formed at the unsaturated sites of SBO. Such epoxidized oils are fairly stable to oxidation. At higher temperatures ( $>250$  °C), rapid degradation sets in when the oxirane ring is broken under oxidizing environment. Such degradation often follows a radical pathway to give polymeric oxidation products. The oxidation behaviors of ESBO and amine derivatives are comparable on the basis of the OT and SM data. However, due to the presence of amine groups, the antioxidant characteristic of the molecule is significantly improved. Sterically hindered amine compounds are often used as radical trapping agents to arrest oxidation process. Furthermore, greater affinity of amines for metal surfaces will improve the tribochemical behavior through lubricity improvement, otherwise unattainable with SBO and ESBO.

In conclusion, we have developed a simple solvent-free method to prepare amine-functionalized soybean oil. The epoxy groups of the ESBO with amines were selectively reacted with amines to give amine-functionalized soybean oil from various amines and catalysts used for maximum aminolysis of epoxy group, without cross-linking and with little disruption of the ester linkage. Diethylamine and  $\text{ZnCl}_2$  were found to be the best. Aggressive conditions or catalysts led to cleavage of the ester groups. Optimally, each mole of ESBO when reacted with 8 molar equiv of diethylamine with 1.1 mol of  $\text{ZnCl}_2$  opened up about 1.5 epoxy groups of ESBO.

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