

## ORIGINAL ARTICLE

## Preparation of Polyurethane-Modified Epoxy Coating Materials Based on Vegetable Oil Derived Ester

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**ABSTRACT** – This study utilizes vegetable oil derivatives as a polyol in polyurethane. A modification of epoxy using polyurethane was done using ester derivative of vegetable oils as polyols, which was polyethylene glycol monooleate (PEGMO). The PEGMO was synthesized via an esterification reaction. The synthesis of polyurethane-modified epoxy using polyethylene glycol monooleate ester (PME-PEGMO) was conducted by reacting epoxy, tolonate, and PEGMO. Analysis results of Fourier transform infrared (FTIR) and proton nuclear magnetic resonance (<sup>1</sup>H-NMR) showed a new absorption peak derived from the urethane bond. The mechanical and thermal properties were characterized by a universal testing machine (UTM) and thermogravimetry analysis (TGA), respectively. According to the analysis results, it is shown that the addition of polyethylene glycol monooleate ester-based polyurethane on epoxy improved the mechanical properties of the epoxy, from 69.61 kgf/cm<sup>2</sup> to 139.80 kgf/cm<sup>2</sup>. However, it decreased the thermal stability of the epoxy. At 500°C, the mass of epoxy was remaining 28%, while the PME-PEGMO was only 13%.

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Epoxy resin and polyurethane are one of the most commonly used materials for coating in the paints and coatings industry [1]–[3]. Epoxy resin is an essential thermosetting polymer and a reactive material with excellent physical-mechanical properties [4],[5]. Epoxy resin has several properties which are very useful for various applications. These properties include high tensile strength, high modulus, low shrinkage, suitable adhesion property, suitable insulation property, good chemical corrosion resistance, good thermal stability, high stiffness, and the ability to dry at low and high temperatures [6]–[8]. Epoxy resin can be used as adhesives, surface coating, coating materials, painting materials, composites, molding compounds, electronic materials, printed circuit boards, and matrices for advanced fiber-reinforced composites [9], [10]. Besides these advantages, epoxy resin has a few drawbacks regarding brittleness and shrinkage during the curing process to degrade the performance and stability of dimension and fragileness when exposed to pressure after curing [3],[11]. These drawbacks lead to limitations in its application and need to be addressed.

Various efforts have been made to improve toughness and reduce the brittleness of epoxy resin. One method to improve the epoxy resin's mechanical properties is chemical modification, which is done by grafting an epoxy resin with polyurethane [2],[11],[12]. Research on epoxy modification using polyurethane has been actively conducted. There are such as the effects of various types of polyurethane on the structure and mechanical properties of the epoxy, evaluation of the nature and structure analysis system of coatings of polyurethane-epoxy, manufacture, and evaluation of properties of nanocomposite epoxy/montmorillonite modified polyurethane, the combination of polyurethane and montmorillonite as a modifier of epoxy and polyurethane [13]–[15]. Polyurethane is one of the most essential and versatile types of polymers. In the coating industry, polyurethane is often used because it has advantages in its properties, such as abrasion resistance, flexibility at low temperatures, toughness, chemical and corrosion resistance, and good mechanical strength [16]–[18]. Polyurethane is obtained by a reaction between isocyanate and polyol. Nowadays, polyols used in the polyurethane industry are mainly derived from non-renewable petrochemical [19]. Vegetable oil as a renewable, non-toxic, non-volatile, and biodegradable resource has been explored to generate a polyol [20]–[22].

Vegetable oil generally has a long hydrocarbon chain, a hydroxyl group, and a double bond and is mostly found in the form of triglycerides and fatty acids [23],[24]. Vegetable oil derivative has several advantages due to their functional groups, such as double bonds, epoxy, hydroxyl, ester, etc. The polar groups derived from vegetable oils in the polymer chain can improve adhesion and flexibility in a polymeric material coating system [25]. The polyols derived from vegetable oil will be used to replace the conventional ones to enter the polyurethane industry [26]. The research regarding polyols from more sustainable and environment-friendly raw materials such as vegetable oil has been actively conducted to reduce the dependence on non-renewable raw materials [27],[28]. Therefore, this research utilized a derivative of vegetable oil, which is oleic acid, to act as a constituent of vegetable oil polyol-based polyurethane. Oleic acid was chosen as a polyol in vegetable oil-based polyurethane because it is an unsaturated fatty acid in liquid form. The liquid form of oleic acid would be easy to process as polyol to produce polyurethane. Oleic

acid was obtained from palm oil as its highest unsaturated component. Unlike this, the highest element of palm kernel oil is lauric acid in the solid form due to saturated fatty acid, so it can be difficult to process when used as polyol to produce polyurethane [29]. The oleic acid was reacted with polyethylene glycol to obtain polyethylene glycol monooleate (PEGMO). Synthesis of vegetable oil-based coating material was conducted by reacting with isocyanates and epoxy.

## EXPERIMENTAL METHOD

### Materials and Instruments

The materials used were oleic acid ( $A_v = 208,72$  mg KOH/g sample), polyethylene glycol 400, potassium hydroxide, and hexane, purchased from Merck (Darmstadt, Germany). Dibutyltin dilaurate was purchased from Sigma-Aldrich (St. Louis, Missouri, United States), while epoxy diglycidyl ether bisphenol A, tolonate HDB 75 (28182-81-2) (%NCO = 32) and versamide 140 were obtained from PT Sigma Utama (Cibinong, Indonesia). Water used was produced by a Millipore water purification system. All chemicals were used without further purification.

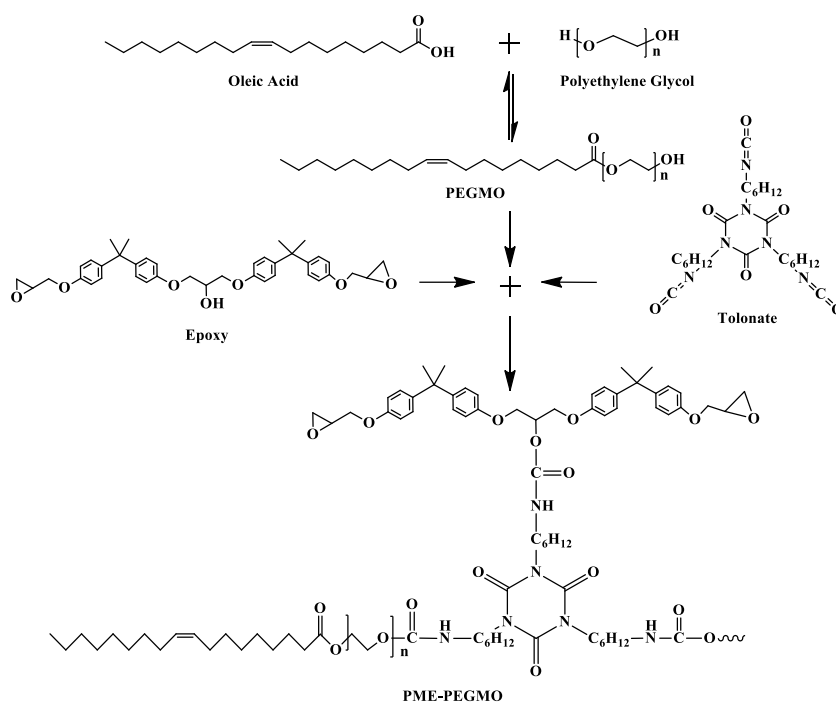
Analysis of Fourier transform infrared (FTIR) was performed using instrument IR Prestige-21 Shimadzu (Kyoto, Japan) to study the changes of functional groups by comparing the spectrum between oleic acid and epoxy PEGMO with PME-PEGMO. The proton chemical structure analysis was performed using a JEOL 500 NMR (Tokyo, Japan). The isocyanate content is determined using the back titration method [30]. The tensile strength measurement from epoxy and PME-PEGMO was conducted according to ASTM D 882 using UTM. The thermal degradation of PME-PEGMO behavior was studied by thermogravimetric analysis (TGA) in TG-HDSC Linseis STA PT 1600 (Selb, Germany). The morphological study was performed by scanning electron microscopy (SEM) Hitachi 3500 at 5 kV (Tokyo, Japan).

### Method and Procedure

Synthesis of PEGMO was done by reacting polyethylene glycol 400 and oleic acid with a mole ratio of 1:1 without excess oleic acid to prevent the formation of diester. 1 wt.% of potassium hydroxide as catalyst was added. Furthermore, the mixture was then refluxed at 180°C for 2.5 hours. The process was done on a three-neck flask equipped with a cooler, a stirrer, and a Dean-Stark apparatus to draw water produced from the esterification reaction. Finally, the PEGMO ester product is washed with water until the pH is neutral and evaporated to remove residual washing water.

PME-PEGMO was synthesized by reacting epoxy, tolonate, and PEGMO in a three-neck flask at 50°C for 30 minutes with the aid of a catalyst dibutyltin dilaurate (DBTL) 0.1 wt.%. The stirring speed was about 200 rpm. According to the previous study, the total weight of tolonate and PEGMO used is 20% of the weight of the epoxy. The comparison used in this study is epoxy: tolonate: PEGMO at 10:1:1 [31].

PME-PEGMO film was synthesized by adding versamide 140 (as curing agent) with a weight ratio between PME-PEGMO and versamide 140 was 2:1, respectively. The films were generated on the surface of a polyethylene board with a film thickness of 0.3 mm and then analyzed using a tensile strength test ultimate testing machine (UTM) Toyo Seiki (Nagano, Japan).



**Figure 1.** The synthesis scheme of PME-PEGMO

## RESULTS AND DISCUSSION

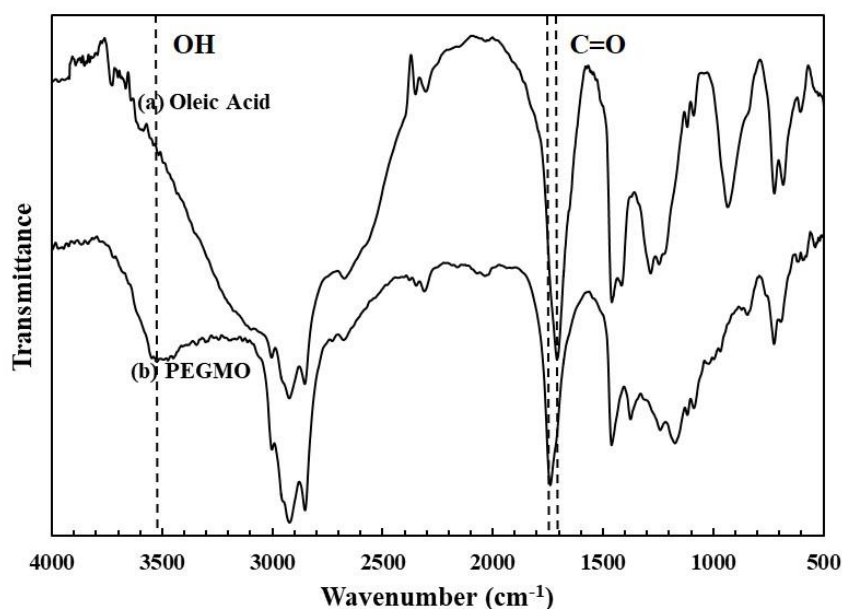
### Synthesis of Polyethylene Glycol Monooleate (PEGMO)

The synthesis of PEGMO was conducted via esterification between polyethylene glycol and oleic acid (Figure 1). PEGMO is monoester. Polyethylene glycol (PEG) has two hydroxyl groups, so the diester may also be formed; it depends on the ratio of reactants (a). In this study, to prevent the formation of diester and make PEGMO maintain a hydroxy group on one side, the ratio of reactant oleic acid and PEG was 1:1 (mol/mol). This aimed to bind one hydroxyl group (OH) of polyethylene glycol to oleic acid. The other side of the hydroxyl group reacts with one isocyanate group (N=C=O) of tolonate forming urethane bonds. Another isocyanate group (N=C=O), aside from tolonate, was expected to react with epoxy to form polyurethane-modified epoxy with polyethylene glycol monooleate (PME-PEGMO), as shown in Figure 1.

Characterization by Fourier transform infrared (FTIR) was performed to detect changes in functional groups that occur in the esterification reaction product of oleic acid with polyethylene glycol. FTIR analysis of oleic acid and PEGMO was done at wavenumbers of 400–4000  $\text{cm}^{-1}$ . The FTIR spectrum of oleic acid and PEGMO is shown in Figure 2.

The FTIR spectrum of oleic acid (Figure 2 (a)) shows an absorption peak at a wavenumber of 1708  $\text{cm}^{-1}$ , indicating the presence of carbonyl groups derived from a carboxylic acid. While in the FTIR spectrum of PEGMO (Figure 2 (b)), it is also shown carbonyl group C=O indicated by the absorption peak at a wavenumber of 1737  $\text{cm}^{-1}$ , which is the carbonyl absorption area for the ester groups. The shift in the absorption peak of the wavenumber of 1708 to 1737  $\text{cm}^{-1}$  indicates that the PEGMO has been successfully synthesized by the esterification reaction. This is also proved by the presence of a new absorption peak at a wavenumber of 1174  $\text{cm}^{-1}$ . This wavenumber indicates a new functional group, the group of C-O-C. The FTIR spectrum of PEGMO also shows absorption peaks at wavenumber 3456–3520  $\text{cm}^{-1}$ , indicating the presence of hydroxyl groups. It shows that the hydroxyl group (OH) remained because polyethylene glycol and oleic acid reacted with a mol ratio 1:1, so only one hydroxyl group of polyethylene glycol reacted with oleic acid. The resulting PEGMO ester product acid number is 37.33 mg KOH/g sample.

The proton nuclear magnetic (H-NMR) analysis was conducted to determine the chemical shifts of protons (H+) in oleic acid and PEGMO. H-NMR spectrums of oleic acid and PEGMO are shown in Figure 3. The  $^1\text{H-NMR}$  spectra of oleic acid and PEGMO is different. The  $^1\text{H-NMR}$  spectra of oleic acid showed no chemical shift at 3–4 ppm, but the  $^1\text{H-NMR}$  spectra of PEGMO showed a new chemical shift at 3–4.5 ppm. However, the new proton chemical shift was observed at 3.64 ppm, indicating H derived from  $-\text{C}(\text{O})-(\text{O}-\text{CH}_2-\text{CH}_2)_n-\text{OH}$  of PEGMO. This chemical shift has the highest integral ratio of the proton, indicating a repeating unit of  $-\text{O}-\text{CH}_2-\text{CH}_2-$  from PEG. A new proton chemical shift also appeared at 4.21 ppm, indicating the H derived from  $-\text{C}(\text{O})-(\text{O}-\text{CH}_2-\text{CH}_2)_n-\text{OH}$  of PEGMO. These new proton chemical shifts proved a reaction between oleic acid and polyethylene glycol, forming PEGMO.

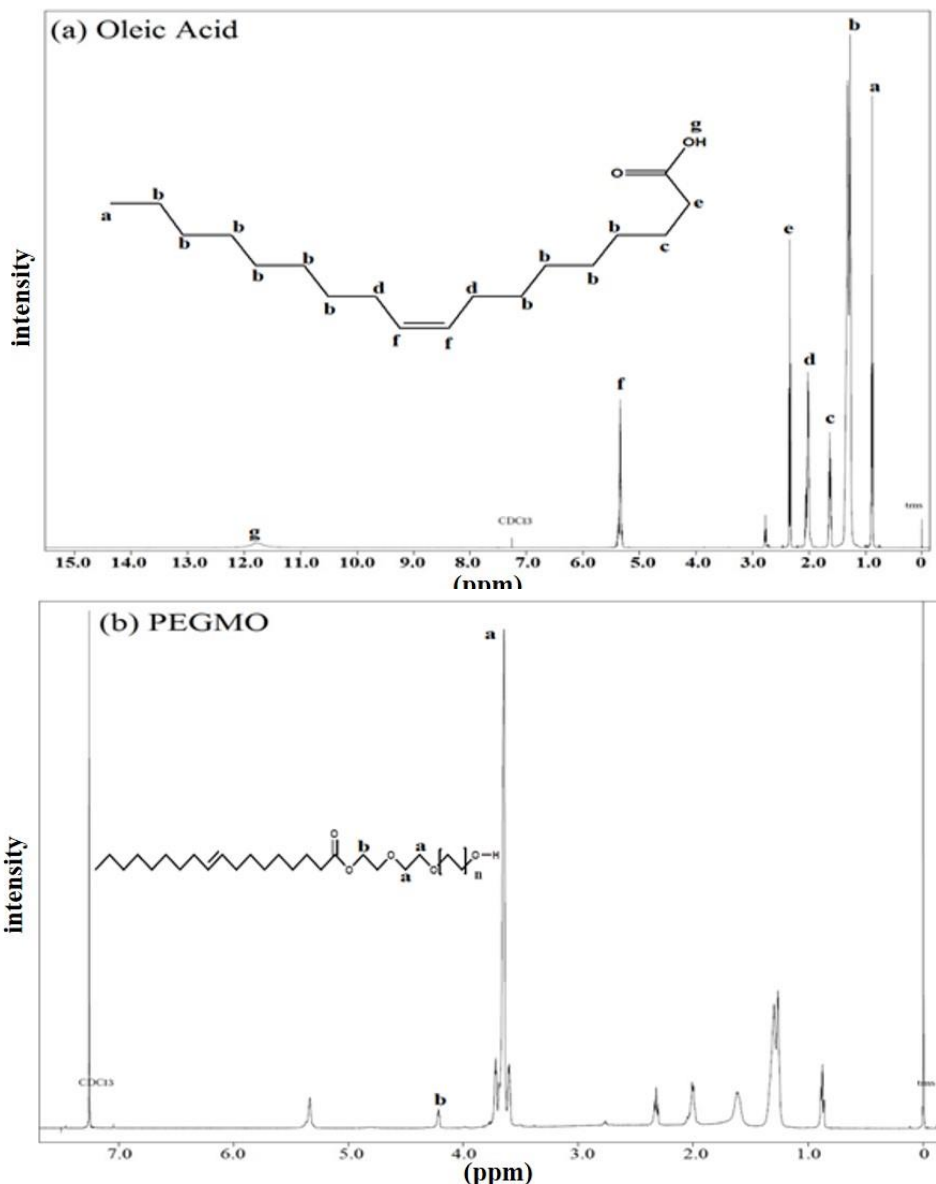


**Figure 2.** FTIR spectrum of (a) oleic acid and (b) PEGMO

### Synthesis of Polyurethane-modified Epoxy with Polyethylene Glycol Monooleate (PME-PEGMO)

The FTIR spectrum of epoxy resin (Figure 4 (a)) shows an absorption peak at a wavenumber of 3506  $\text{cm}^{-1}$ , indicating the hydroxyl group (OH) of the epoxy resin. At the same time, the FTIR spectrum of PEGMO also indicated the OH group. However, in the spectrum of PME-PEGMO (Figure 4 (c)), these absorption peaks disappeared; instead, a new absorption peak emerged at a wavenumber of 3404  $\text{cm}^{-1}$ . This peak indicated the group of NH that was derived from the urethane bond  $-\text{NH}-\text{C}=\text{O}-\text{O}-$ . This showed a reaction between isocyanate (N=C=O) of tolonate with OH of epoxy or PEGMO. The new absorption peaks at a wavenumber of 1689  $\text{cm}^{-1}$  indicated the carbonyl group of COO

derived from urethane bond  $\text{-NH-C=O-O}$ . Both the disappeared and new absorption peaks at the mentioned wavenumbers have proven that urethane bonds have formed. These urethane bonds resulted between the isocyanate ( $\text{N=C=O}$ ) of tolonate and the OH of epoxy and PEGMO. The obtained PME-PEGMO product has an NCO content of 19.29%.

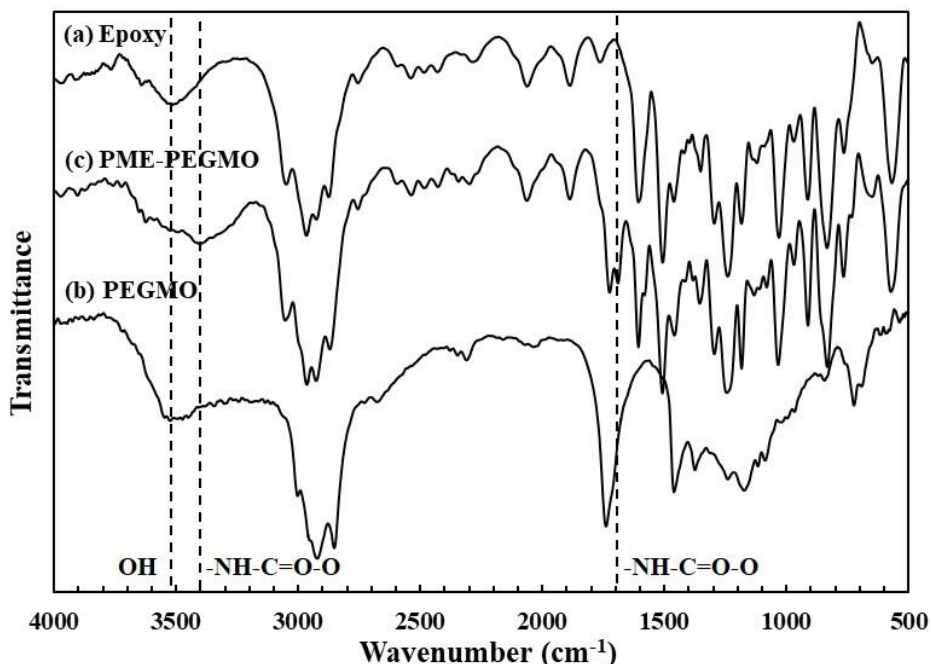


**Figure 3.** H-NMR Spectrum of (a) Oleic Acid and (b) PEGMO

The chemical structure analysis of the epoxy and PME-PEGMO was performed using H-NMR analysis. This analysis was conducted to determine the proton chemical shifts in PME-PEGMO by comparing the proton chemical shifts in epoxy and PME-PEGMO (Figure 5). In the H-NMR spectrum of epoxy (Figure 5 (a)), there is a proton chemical shift at 4.25, which shows the  $\text{-CH-}$  bond derived from H atoms attached to the C atom in the  $\text{-CH-OH}$  bond originated from epoxy. While in the spectrum of PME-PEGMO (Figure 5 (b)), the value of this proton chemical shifts toward a lower field, which is the proton chemical shift at 5.08 ppm showing the  $\text{-CH-}$  bond derived from the H atoms bound to the C atom in  $\text{-CH}_2\text{-CHO-CH}_2\text{-}$  bond. This chemical shift resulted from the reaction between OH hydroxyl groups on the C-OH bond of epoxy with isocyanate group  $\text{N=C=O}$  at tolonate forming urethane bond  $\text{-NH-(C=O)-O-CH-CH}_2\text{-}$ . This forming of urethane is the cause of proton chemical shifts toward the lower field.

In the H-NMR spectrum of PEGMO (Figure 5 (b)), the new proton chemical shifts appeared at 0.88 ppm, 1.26 ppm, 1.30 ppm, 2.06 ppm, 2.31 ppm, 5.08 ppm, and 5.42 ppm. Proton chemical shifts at 0.88 ppm show  $\text{-CH}_3$  bonds derived from the H atoms bonded to  $\text{-CH}_3$  atoms at the end of PEGMO. The shift at 1.26 ppm shows  $\text{-CH}_2\text{-}$  bond derived from the C- $\text{CH}_2\text{-C}$  on PEGMO and tolonate. While the proton chemical shift at 1.30 ppm shows the  $\text{-CH}_2\text{-}$  bond derived from the H atoms bonded to the C atom before the end of the PEGMO ( $\text{-CH}_2\text{-CH}_3$ ), and the proton chemical shift at 2.06 ppm indicates  $\text{-CH}_2\text{-}$  bond originated from H atoms which were bonded to carbon atoms in the bond of  $\text{-CH}_2\text{-CH=CH-CH}_2\text{-}$ .

at PEGMO. The proton chemical shift at 2.31 ppm indicates  $-\text{CH}_2-$  bond originated from H atoms that bonded to carbon atoms in the  $-\text{CH}_2\text{-COO}$  bond. In the spectrum of PME-PEGMO, a new proton chemical shift also emerged at 5.42 ppm, indicating H atoms attached to the C atoms of the double bond of  $-\text{CH}=\text{CH}-$  derived from PEGMO. The proton chemical shifts mentioned above show that there has been a reaction between epoxy, tolonate, and PEGMO,



resulting in the obtained PME-PEGMO.

**Figure 4.** FTIR Spectrum of (a) epoxy, (b) PEGMO, and (c) PME-PEGMO

### Mechanical Properties

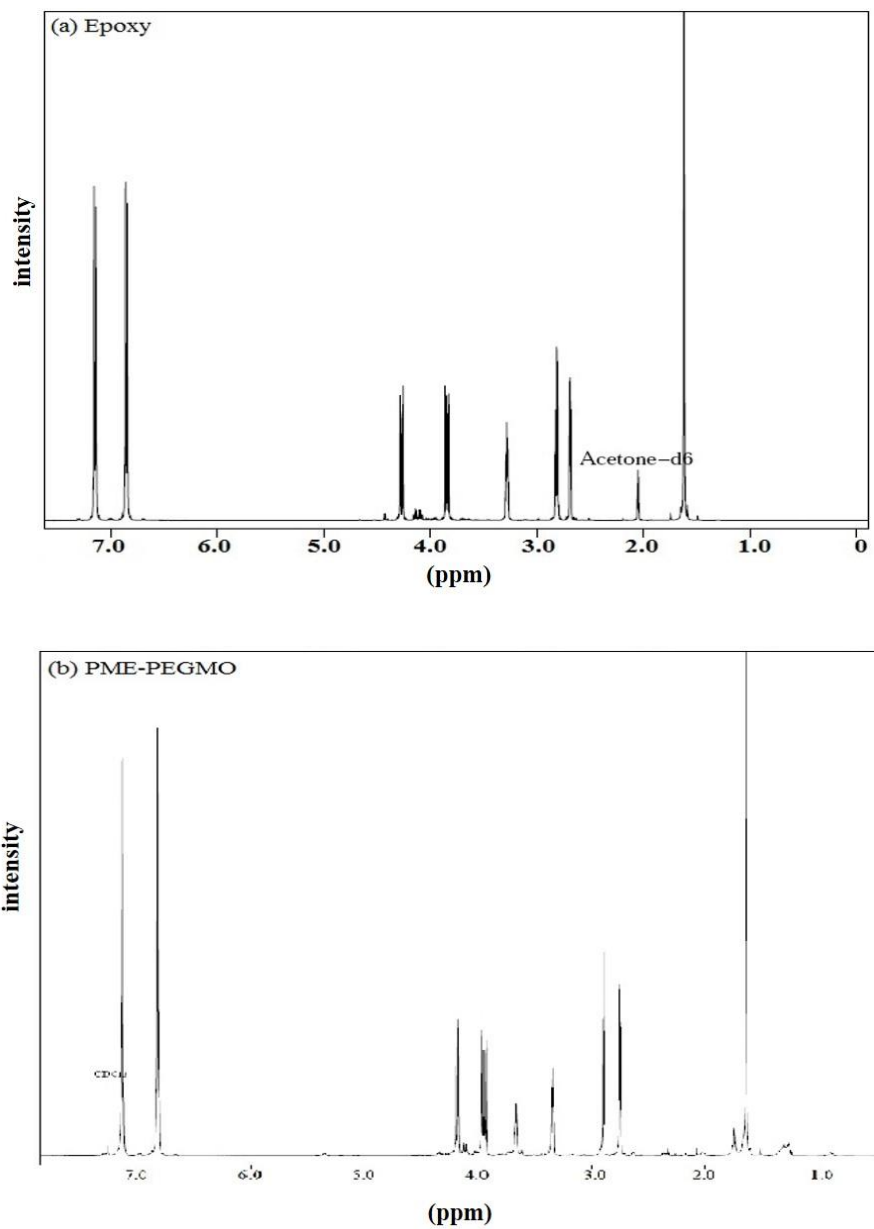
Mechanical properties of epoxy and PME-PEGMO were characterized using a tensile strength test, as shown in Table 1. Epoxy resin has a tensile strength of 69.61 kgf/cm<sup>2</sup>, and PME-PEGMO has a tensile strength of 139.80 kgf/cm<sup>2</sup>, higher than epoxy [32]. The increasing value of tensile strength in the PME-PEGMO was influenced by the presence of polyurethane linkage containing soft segment and hard segment in the structure of PME-PEGMO. During the tensile strength measurement, the PME-PEGMO was exposed to force, resulting in the orientation of the soft segments part and the increase of the tensile strength [30],[32]–[34]. These results indicated that the presence of polyurethane in the structure of PME-PEGMO could increase the tensile strength significantly [34].

**Table 1.** Tensile Strength of Epoxy and PME-PEGMO.

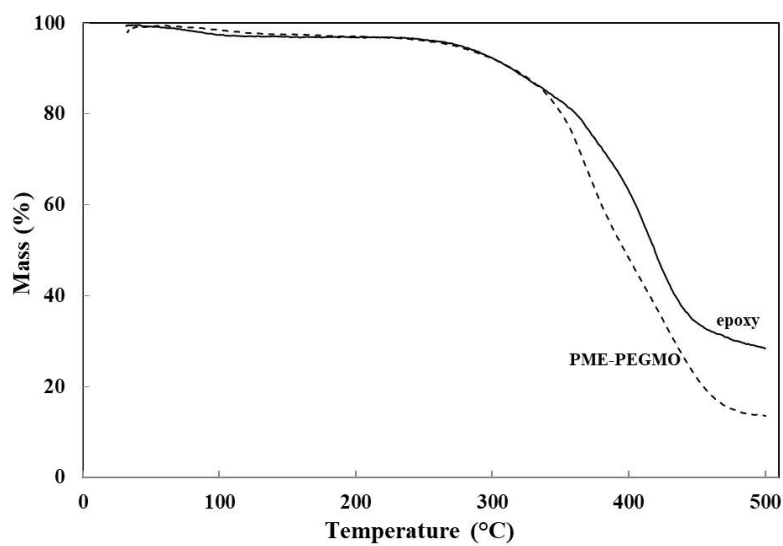
Sample	Tensile Strength (kgf/cm <sup>2</sup> )
Epoxy	69.61
PME-PEGMO	139.80

### Thermal Properties

The thermal properties of PME-PEGMO were studied using thermogravimetric analysis (TGA) by comparing the thermal stability of epoxy to PME-PEGMO. The results of the TGA analysis of epoxy and PME-PEGMO are shown in Figure 6. The thermal stability curve shows that the mass of epoxy and PME-PEGMO decreases similarly until 330°C. Initially, epoxy and PME-PEGMO show a slight mass decrease until 330°C. However, from 330 to 500°C, it shows a significant mass decrease, with the mass reduction of PME-PEGMO was larger than epoxy. At 330°C, both epoxy and PME-PEGMO still had 87% of the mass. However, at 500°C, the mass of epoxy was remaining 28%, while the PME-PEGMO was only 13%. This shows that modification of epoxy with polyurethane based on vegetable oils ester can reduce the thermal stability of the epoxy, which results in more massive mass reduction. This degradation of thermal stability was apparently due to the urethane bond in the PME-PEGMO [23],[28].



**Figure 5.** H-NMR spectrum of (a) Epoxy and (b) PME-PEGMO



**Figure 6.** The thermogravimetric curve of a) epoxy and b) PME-PEGMO

## CONCLUSION

In this study, an epoxy modification with polyurethane was conducted using polyethylene glycol ester monooleate as polyols. An epoxy modification using ester-based polyurethane with polyethylene glycol monooleate (PEGMO) has been successfully carried out. The reaction between epoxy, tolonate, and PEGMO produced PME-PEGMO. The analysis results of Fourier transform infra red (FTIR) and and proton nuclear magnetic resonance (H-NMR) show a new absorption peak derived from the urethane bond. The addition of ester polyethylene glycol monooleate-based polyurethane on the epoxy modification improved the mechanical properties of epoxy but degraded the thermal stability.

## ACKNOWLEDGEMENT

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