

Development of Coating Material from Epoxidized Unripe Fresh Fruit Bunch Palm Oil

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Abstract

Unripe fresh fruit bunches (FFB) are typically rejected as it results in inferior quality palm oil, thus leading to wastage and losses. Repurposing the bunches to be used as a replacement for diglycidyl ether of bisphenol A (DGEBA) in a new epoxy coating formulation can increase the sustainability of paint industries. This also reduces the dependency on DGEBA as the conventional epoxy resin which has shown to cause adverse effects on human health based on past researches. Unripe fresh fruit bunch palm oil undergoes epoxidation process which adds an oxirane ring to the fatty acid (FA) structure. The oxirane ring provides a reactive site for the crosslinking reaction during the coating formulation. The formulated coating was tested for its chemical resistance through chemical immersion and solvent rub tests to determine the best coating formulation that can withstand various environments with minimal thickness loss. Fourier Transform Infrared (FTIR) Spectroscopy analysis showed the presence of oxirane ring after the epoxidation process. Results from the chemical immersion test and solvent rub test showed that the developed coating material experienced little to no corrosion and also minimal thickness loss when exposed to organic solvents such as acetone and toluene.

Article Info

Article history:

Received date: 6 December 2019

Accepted date: 6 December 2019

Keywords:

Chemical resistance performance
Epoxidation
Epoxidized unripe palm oil
Epoxy coating

1.0 Introduction

1.1 Epoxy coating

Epoxy coating is a thermoset with superior properties that is used as a coating or paint for metals and floor [1]. Epoxy coatings also provide protection against corrosion and gives an attractive finish. Epoxy coatings are produced from a chemical reaction between a polyamine hardener and an epoxy resin [2].

A common epoxy resin used in the production of conventional epoxy coatings is diglycidyl ether of bisphenol A (DGEBA) which is created by combining bisphenol A and epichlorohydrin through a condensation reaction (BPA) [3, 4]. DGEBA is used due to its high adhesion characteristic, its heat and corrosion resistance as well as its mechanical strength [5]. Although the usage of DGEBA in epoxy coatings results in satisfactory performance, DGEBA poses a health hazard to humans as it is an endocrine disruptor and has antiandrogen [6]. Besides that, exposure to DGEBA for men may result in

gonadotrophic hormone disruption [7]. Figure 1 shows the chemical structure of DGEBA.

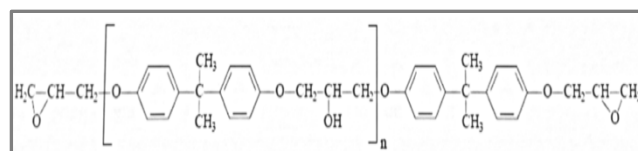


Fig. 1: Chemical structure of DGEBA [4].

Epoxy resin can be used for various applications such as automotive primer, coatings, printed circuit boards and adhesives [8]. The dependency of the paint industry on DGEBA as the conventional epoxy resin in the formulation of epoxy coatings can be greatly minimized or eliminated altogether through the introduction of a safer material.

Materials derived from renewable resources such as vegetable oils (VOs) like soybean oil [9], castor oil [10], sunflower oil [11] and hemp oil [12] are the best options to use in the formulation of new epoxy coatings due to the presence of unsaturated fatty acids (FAs) in the chemical structure which can be converted to epoxy

group during epoxidation reaction.

1.2 Unripe fresh fruit bunch palm oil

Palm oil is obtained via extraction from the ripened mesocarp of fresh fruit bunches (FFB) of the oil palm tree known as *Elaeis guineensis* [13]. Palm oil is made up of vitamin E, free fatty acids (FFAs), triglycerides, oxidation products, phytosterols, carotenoids (coloring pigments), phospholipids and gums [14, 15].

The uses of palm oil predominantly extend to the edible food industry [16] and less predominantly other non-food applications [15]. The oil palm tree bears fruit in the form of bunches which are categorized into unripe, under ripe, ripe and overripe [17, 18].

The Malaysian Palm Oil Board (MPOB) encourages millers and dealers to turn down unripe bunches (poor quality fruit bunches) as it produces inferior quality palm oil [19]. Unripe FFB has less than 14 percent oil content as compared to ripe bunches which has oil content of around 24 to 25 percent [19]. MPOB recommends a minimum oil extraction rate (OER) of 20% [19]. Harvesting unripe bunches results in high FFAs and low OER, thus leading to wastage and losses [20].

In order to reduce wastage of the unripe FFBs, the bunches can be repurposed to be used as a replacement for DGEBA as epoxy resin in a new epoxy coating formulation.

1.3 Epoxidation

Epoxidation is the process of chemically modifying the unsaturated bonds present in crude palm oil (CPO) to a value-added product by the addition of an oxirane ring to the structure [21]. Opening of the oxirane ring occurs at the cleavage of one of the carbon-oxygen bonds [22]. Figure 2 shows the structure of an oxirane ring.

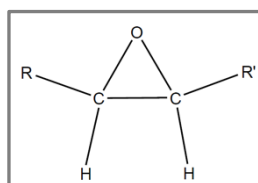


Fig. 2: Structure of an oxirane ring [23].

The epoxidation process happens in the unsaturated bonds of VOs with the aid of peroxy acids ($R-CO_3H$) such as peroxy-formic (performic acid) or peroxy-acetic [24]. Epoxidation leads to the formation of an oxirane (epoxide) functional group and the release of organic acid [21]. Figure 3 shows the mechanism for

oxirane formation. The formation of oxirane ring at the unsaturated bonds ($C=C$) of the unripe FFB palm oil produces epoxidized unripe palm oil (EUPO) as shown in Figure 4.

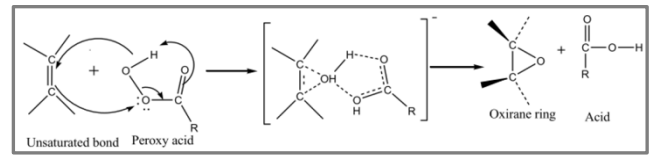


Fig. 3: Mechanism for oxirane formation [25].

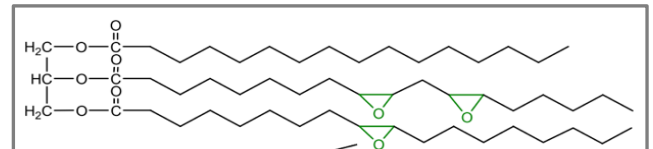


Fig. 4: Structure of triglyceride molecule after addition of oxirane ring through epoxidation process (highlighted in green) [25].

Epoxidation is an important process as the epoxides acquired from VOs can be used as raw materials of high temperature for alcohol or glycols. Epoxidation also improves the stability of the oil and provides adequate reactivity to form chemical linkages with other polymers [26].

This reaction results in new products with superior properties [27]. Soybean oil [9], castor oil [10], sunflower oil [11] and hemp oil [12] are examples of VOs that can undergo epoxidation process based on previous researches.

A coating material based on epoxidized unripe palm oil (EUPO) and DGEBA was developed by using formulation ratios of 10:90, 20:80 and 30:70 (EUPO:DGEBA). The formulated coatings were tested and rated for its chemical resistance through chemical immersion test and solvent rub test.

According to a study conducted by Taharim (2012), the optimum amount of epoxidized palm oil (EPO) in a blend of EPO and epoxy is 30 wt% [23]. Higher amount of EPO (>30 wt%) in the blend results in decreasing tensile strength, Young's modulus, elongation at break, toughness and flexural strength [28].

2.0 Methodology

2.1 Materials

Titanium (IV) oxide powder and formic acid ($HCOOH$) (98-100% purity) were purchased from Merck. Cycloaliphatic amine adduct which includes isophorone diamine (IPDA) ($\geq 99\%$ purity) and

DGEBA were purchased from Sigma-Aldrich. The crude palm oil that originated from unripe FFB was obtained from the Faculty of Plantation and Agrotechnology in Universiti Teknologi MARA (UiTM) Shah Alam. Hydrogen peroxide (H_2O_2) ($\geq 99\%$ purity), sulphuric acid, sodium chloride, sodium hydroxide, acetone and toluene were supplied by the Faculty of Chemical Engineering (FKK) in UiTM Shah Alam. Other chemicals such as sodium carbonate powder and sodium chloride crystals were supplied by the Geology and Drilling as well as Flow Assurance Laboratory, respectively, in FKK, UiTM Shah Alam. Mild steel coupons (test panels) with dimensions of 30 mm x 40 mm x 3 mm were purchased from a hardware store in Section 7, Shah Alam, Selangor.

2.2 Equipment

Riken sandpaper of grade 60, 80 and 120 as well as plastic containers were purchased from a hardware store in Section 7, Shah Alam, Selangor. Fourier Transform Infrared (FTIR) Spectrometer was provided by Instrumentation II Laboratory in FKK, UiTM Shah Alam. Mechanical mixer, clear glass vials, beakers, drilling machine, DPM-816 digital coating thickness tester and TQC sheen wet film thickness gauge (follows ISO 2008, ASTM D4414, ASTM D1212) were provided by Geology and Drilling Laboratory in FKK, UiTM Shah Alam. Polishing machine was provided by Flow Assurance Laboratory in FKK, UiTM Shah Alam.

2.3 Methodology

Figure 5 shows the general procedure for the development and testing of the coating material. The first step was to epoxidize the unripe FFB palm oil to produce EUPO, followed by conducting FTIR analysis on the EUPO sample. Formulation of the coating material was carried out simultaneously with the preparation of the coupons. The formulated coating was applied on the coupons. Chemical resistance testing was carried out after the test panels were completely dried and cured. The procedures starting from the formulation of the coating material were repeated if the results obtained was not satisfactory.

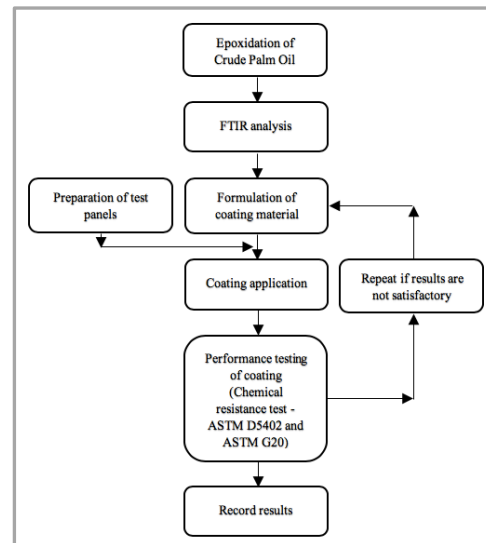


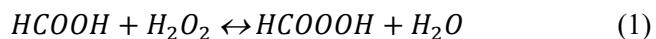
Fig. 5: General procedures for the development and testing of the coating material.

A. Epoxidation of unripe palm oil

Unripe FFB palm oil was epoxidized to add oxirane ring to its fatty acid chain structure. An unchanging molar ratio of EUPO:HCOOH: H_2O_2 (1:5:4 mole/mole) was used and the epoxidation process was carried out under continuous magnetic stirring at a constant temperature of 45°C for 150 minutes [25].

Formic acid and hydrogen peroxide were mixed in a separate beaker before it was added to the oil reagent contained in a round-bottomed flask. The flask was submerged in a beaker filled with water, whereby a reflux condenser was connected to the mouth of the flask.

Hydrogen peroxide was slowly added drop wise into the acidic medium. Equation (1) shows the generation reaction of *in situ* performic acid (HCOOOH) during epoxidation. This reaction is a reversible reaction.



where: HCOOH is formic acid, H_2O_2 is hydrogen peroxide, HCOOOH is performic acid, and H_2O is water.

The completion of the reaction was followed by washing of the sample using distilled water, sodium chloride crystals (5 wt%) and sodium bicarbonate

powder (5 wt%) to separate the organic layer from the mixture. The sodium bicarbonate powder and sodium chloride crystals were mixed and diluted in the distilled water.

B. FTIR analysis

Samples from both unepoxidized unripe palm oil and EUPO were extracted from the washed solution and transferred to clear glass vials for analysis using FTIR Spectrometer. The spectrum of both samples was analyzed and compared for the presence of oxirane ring in the chemical structure before and after epoxidation process.

C. Formulation of coating material

Epoxy resin consisting of a blend of EUPO and DGEBA were prepared using three different weight ratios of 10:90, 20:80 and 30:70 (EUPO:DGEBA). Each mixture was mixed under continuous magnetic stirring at 400 rpm until no phase separation could be observed.

Titanium (IV) oxide powder as pigment was added to the EUPO and DGEBA mixture with a weight ratio of 1:0.25 (EUPO+DGEBA:TiO₂) and mixed under continuous magnetic stirring at 1010 rpm until a homogenous phase was observed which was at the 1 hour mark.

Cycloaliphatic amine adduct as hardener was added to the resulting mixture at weight ratios between the ranges of 1:0.25 to 1:0.29 (EUPO+DGEBA+TiO₂:Amine). The mixture was mixed under continuous magnetic stirring at 400 rpm until no phase separation could be observed which was at the 5 minutes mark.

D. Preparation of test panels

Mild steel coupons with dimensions of 30 mm x 40 mm x 3 mm were used as test panels. Sandpaper grade 120, 80 followed by grade 60 were used to polish the coupons. Acetone was used to wipe down the coupon surface after polishing to remove any remaining metal dust. A small hole was drilled at the top of each coupon to hang strings for the curing process.

E. Coating application

The coating was applied on the steel coupons by dipping the coupons into a plastic container containing the formulated coating. The wet film thickness of the coating material was measured using TQC sheen wet film thickness gauge after the coating application.

Three readings were recorded at each surface of the coupons. The coupons were left to cure overnight at room temperature and standard conditions [23] in a fume hood after the desired wet film thickness was obtained.

F. Coating performance test

The coating performance of the coating was evaluated based on its chemical resistance. The chemical resistance test consists of two parts: chemical immersion test and solvent rub test. These tests follow the standards outlined in ASTM G20-88 [29] and ASTM D5402-93 [30]. The degree of blistering and rusting of the coating were evaluated based on the standards defined in ASTM D714-87 [31] and ASTM D610-01 [32].

The chemical immersion test consisted of fully immersing the coated coupons in three different solutions: sulphuric acid (acidic environment), sodium chloride (salt water environment) and sodium hydroxide (alkaline environment). Observation of the effect of immersion time on the coated coupons at different formulation ratios (EUPO:DGEBA) were made on day 1, day 5, day 10 and day 15 [29].

The solvent rub technique consisted of performing double rubs on the test panels 25 times using cotton cloth dipped in organic solvents such as acetone and toluene [30]. The thickness before and after performing the solvent rub test was recorded using DPM-816 digital coating thickness tester.

Figure 6 shows the degree of rusting as defined in ASTM D610-01. Table 1 shows the scale and description of rust ratings as outlined in ASTM D610-01.

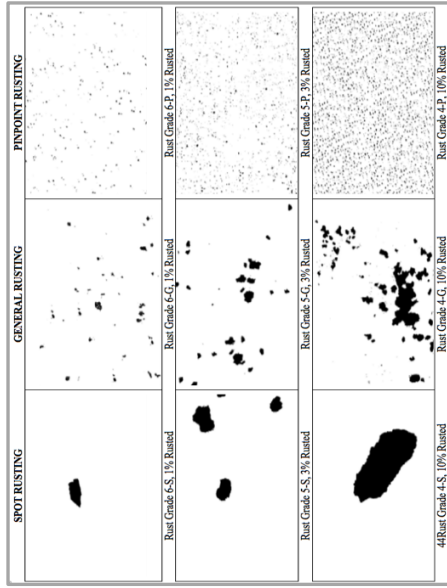


Fig. 6: Examples of degree of rusting [32].

Table 1: Scale and description of rust ratings [32].

Rust Grade	Percent of Surface Rusted	Visual Examples		
		Spot(s)	General (G)	Pinpoint (P)
10	Less than or equal to 0.01 percent		None	
9	Greater than 0.01 percent and up to 0.03 percent	9-S	9-G	9-P
8	Greater than 0.03 percent and up to 0.1 percent	8-S	8-G	8-P
7	Greater than 0.1 percent and up to 0.3 percent	7-S	7-G	7-P
6	Greater than 0.3 percent and up to 1.0 percent	6-S	6-G	6-P
5	Greater than 1.0 percent and up to 3.0 percent	5-S	5-G	5-P
4	Greater than 3.0 percent and up to 10.0 percent	4-S	4-G	4-P
3	Greater than 10.0 percent and up to 16.0 percent	3-S	3-G	3-P
2	Greater than 16.0 percent and up to 33.0 percent	2-S	2-G	2-P
1	Greater than 33.0 percent and up to 50.0 percent	1-S	1-G	1-P
0	Greater than 50 percent		None	

3.0 Results and discussion

3.1 Epoxidation of unripe palm oil

Figure 7 shows the spectrum profile of unripe FFB palm oil prior to epoxidation. The spectrum shows the presence of a carbon-carbon double bond (unsaturated fatty acid group) at wavenumber 1650 cm^{-1} . A stretch of C-H is observed between wavenumber 2921.80 cm^{-1} to 2852.82 cm^{-1} . A carbonyl stretch is observed at wavenumber 1743.86 cm^{-1} and methyl bending at wavenumber 1457.13 cm^{-1} . Presence of nitrogenous compound is also observed at wavenumber 1541.53 cm^{-1} and a methylene group at wavenumber 721.22 cm^{-1} . Bending of C-H is shown at wavenumber 1377.81 cm^{-1} . The existence of the stretching of C-O and the

bending of O-H are observed at wavenumber 1159.70 cm^{-1} and 1116.43 cm^{-1} , respectively.

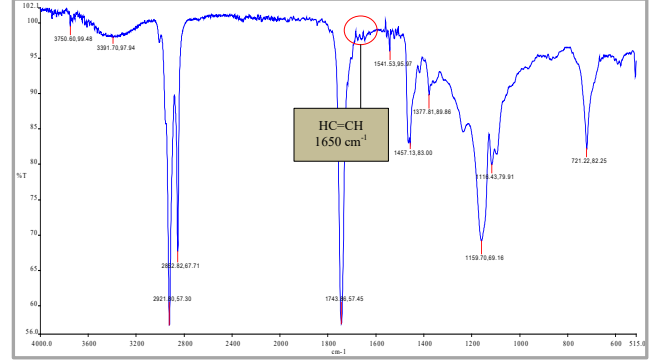


Fig. 7: Spectral analysis of unripe FFB palm oil (before epoxidation).

Figure 8 shows the spectrum profile of EUPO after epoxidation. Based on this spectrum profile of EUPO, it is observed that the unsaturated fatty acid group disappeared after the epoxidation process but the presence of oxirane ring in the structure is observed at wavenumber 844.42 cm^{-1} . The detection of oxirane ring is between wavenumber 750 cm^{-1} to 880 cm^{-1} [33].

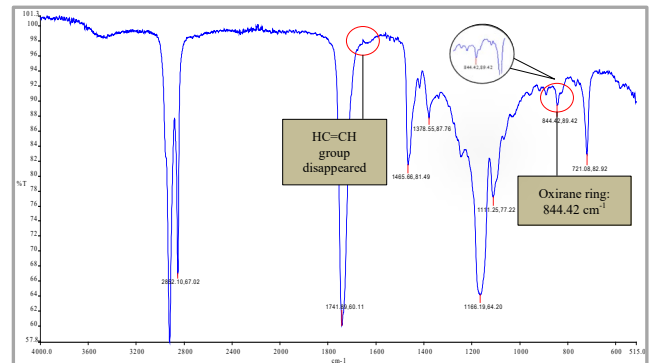


Fig. 8: Spectral analysis of epoxidized unripe palm oil (EUPO) (after epoxidation).

Palm oil is made up of almost equivalent parts of both saturated and unsaturated fatty acids (FAs) [34]. This enables palm oil to undergo epoxidation process as the ethylenic FAs present in palm oil serves as the reactive site for the crosslinking reaction in the epoxidation process which introduces the oxirane (epoxide) functional group into the chemical structure of palm oil via acid catalysis [25, 35].

In terms of curing and aging, shrinkage is reduced when cationic types cross-link by the opening of epoxy

rings. Ring-opening moieties (SOE) are included in this system, which contributes to lower shrinkage, and thus creates a product that is longer-lasting with better adhesion. Furthermore, cationic cures are not air-sensitive [36].

3.2 Chemical resistance test (ASTM G20-88 and ASTM D5402-93)

The chemical resistance of the coating for each formulation was tested via two tests, which were chemical immersion test and solvent rub test.

A. Chemical immersion test

Table 2 (i)–(iii) shows the conditions of the metal coupons during full immersion in sulphuric acid (acidic environment), sodium chloride (salt water environment) and sodium hydroxide (alkaline environment).

Based on Table 2 (i), it can be observed that on the first day of immersion, the steel coupons did not react with the sulphuric acid solution as there were no changes to the surfaces of the coupons. However, on the fifth day of immersion, bubble formation was observed on the surface of the coupons for all the coating formulations, whereby bubble formation on the coupon with formulation 10:90 was the most visible. The bubble formation for formulations 20:80 and 30:70 were similar. This phenomenon continued until the fifteenth day of immersion. Formulation 10:90 created a lower viscosity coating which resulted in a thinner passivation film (protective film), thus increasing the rate of bubble formation [37].

The formation of bubbles showed that the metal reacted with the sulphuric acid solution and the bubble formation phenomenon is known as hydrogen evolution reaction (HER). HER is an indication of the eventual occurrence of corrosion. HER typically occurs in very acidic environments and the reaction involves the production of hydrogen through water hydrolysis process [38, 39]. Figure 9 shows the mechanism of HER.

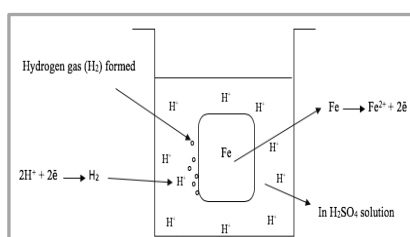


Fig. 9: Hydrogen evolution reaction (HER)

mechanism [40].

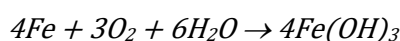
Based on Figure 9, the anodic iron (Fe) metal undergoes oxidation process (loss of electrons) whereas the hydrogen ions (H^+) in the sulphuric acid solution (electrolyte) undergoes reduction process. Reaction with electrons occurs at the surface of metal which acts as the cathodic surface. The H^+ ions receive electrons from the Fe atom which reduces the ions to hydrogen atoms. Hydrogen gas (H_2) is produced when the hydrogen atom combines with other hydrogen atoms present. The evolution of the hydrogen gas through the cathodic surface results in the formation of bubbles on the steel coupon surface [38, 39].

On the first day of immersion, the coupons for all the coating formulations were observed to have no changes as there were no changes to the surfaces of the coupons. On the fifth day of immersion, the coupons for coating formulation 10:90 and 20:80 were observed to have localized corrosion or spot corrosion on one edge of the coupon for both formulations. The corrosion area progressively increased until the fifteenth day of immersion.

The coupons for coating formulation 30:70 experienced bubble formation on the surface of the coupons on the fifth day of immersion and this continued until the fifteenth day of immersion. This shows that HER took place in the sodium chloride solution. The coupon with formulation 30:70 did not experience spot corrosion which may be attributed to the higher viscosity coating which forms a thick and durable protective layer on the metal surface [41].

Continual occurrence of HER will lead to corrosion as the loss of electrons from the metal results in the breaking down of the passive film (protective layer), thus rendering the metal vulnerable to corrosion [42]. The rate of corrosion in salt water is typically higher as versus in fresh water as a result of the presence of chloride ions which significantly decrease the capability of the passive film to withstand dissolution or negative metal oxidation [43].

The presence of chloride ions in the solution increases the rate of redox reaction (acceptance of electrons) which increases the conductivity of the solution, thus effectively increasing the concentration of chloride ions in the solution. This increases the rate of corrosion of the metal. The metal that rusts is iron (Fe) and it oxidizes to iron (II) oxide (Fe_2O_3) in the presence of water (H_2O) and oxygen (O_2) as shown in Equation (2) [43]:



Based on Table 2 (iii), the coupons did not undergo any reaction in the sodium hydroxide solution throughout the fifteen days period of immersion. The possibility of

Table 2 (i): Full immersion of steel coupons in sulphuric acid (bubbles highlighted in red)

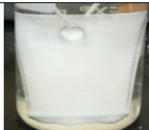
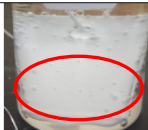
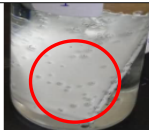
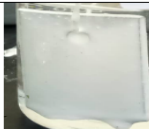
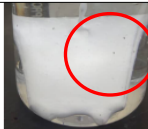
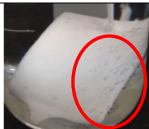
EUPO/DGEBA	1 st Day	5 th Day	10 th Day	15 th Day
10:90				
20:80				
30:70				

Table 2 (ii): Full immersion of steel coupons in sodium chloride (rust highlighted in blue; bubbles highlighted in red)

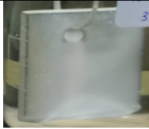
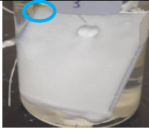
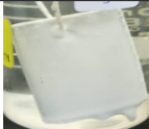
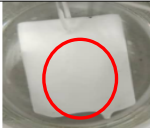
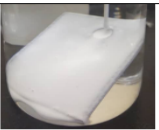
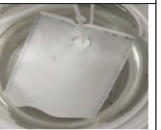
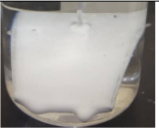
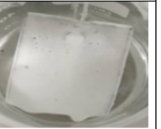
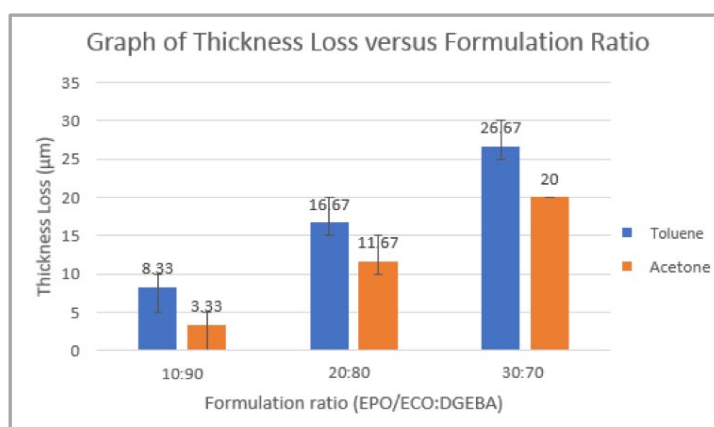
EUPO/DGEBA	1 st Day	5 th Day	10 th Day	15 th Day
10:90				
20:80				
30:70				

Table 2 (iii): Full immersion of steel coupons in sodium hydroxide

EUPO/DGEBA	1 st Day	5 th Day	10 th Day	15 th Day
10:90				
20:80				
30:70				

**Fig. 10:** Thickness loss of the coating after solvent rub for every formulation ratio.

solvent rub was performed for coating formulation 10:90, 20:80 and 30:70.

Based on Figure 10, it can be observed that coating formulation 30:70 had the highest thickness loss for both solvents (acetone and toluene) after the solvent rub test was performed, followed by coating formulation 20:80 and finally 10:90 which had the lowest thickness loss. Minimal thickness loss for coating formulation 10:90 was attributed to the lower dry thickness of the coating. The oxirane ring present in the chemical structure of EUPO increases the hydrophilicity of EUPO, thus making it more resistant to organic solvents [41]. The chemical resistance of the coating is increased with increasing EUPO content in the coating formulation.

From Figure 10, it can be observed that toluene was able to reduce the thickness of the coating at a higher rate compared to acetone. Toluene as an organic solvent is a little more than half as strong as acetone [45]. It is more aggressive and a stronger cleaning agent compared to acetone. Toluene is also one of the main ingredients in paint thinner which functions to reduce

coupons after fifteen days of immersion in sodium chloride solution which represents a salt water environment. Signs of rusting were visible on one edge of the coupon for coating formulations 10:90 and 20:80. However, the degree of rusting for these coating formulations are classified as “0” as the rusting were only results of “edge effects” and no rusting was found on the flat surfaces of the steel coupons. No sign of rusting was visible on the steel coupon for coating formulation 30:70. Therefore, the degree of rusting for the coupons are classified as “0”.

Table 4 (iii) shows the final conditions of the steel coupons after fifteen days of immersion in sodium hydroxide solution which represents an alkaline environment. No signs of rusting were visible on the coupons for all the coating formulations. Therefore, the degree of rusting for the coupons are classified as “0”.

the thickness of paints or coatings after application [45].

C. Degree of rusting (ASTM D610-01)

The degree of rusting on the surfaces of the coupons can be referred in Figure 6. Figure 6 shows the visual examples of different rust grades as outlined in ASTM D610-01. Table 4 (i) shows the final conditions of the steel coupons after fifteen days of immersion in sulphuric acid solution which represents an acidic environment. No signs of rusting were visible on the test panels for all the coating formulations. Therefore, the degree of rusting for the coupons are classified as “0”.

Table 4 (ii) shows the final conditions of the

Table 4 (iii): Degree of rusting of steel coupons immersed in sodium hydroxide solution.

10:90	20:80	30:70
		
Degree of rusting: 0	Degree of rusting: 0	Degree of rusting: 0

4.0 Conclusions

A coating material based on epoxidized unripe palm oil (EUPO) and diglycidyl ether of bisphenol A (DGEBA) was developed using using formulation ratios of 10:90, 20:80 and 30:70 (EUPO:DGEBA). The formulated coatings were tested and rated for its chemical resistance through chemical immersion test and solvent rub test. The comparison between unepoxidized unripe palm oil and epoxidized unripe palm oil (EUPO) showed that the unripe palm oil contains a carbon-carbon double bond (unsaturated bond) which enabled the oxirane ring formation in the chemical structure of EUPO. The results obtained from the chemical immersion test showed that the steel

Table 4 (i): Degree of rusting of steel coupons immersed in sulphuric acid solution.

10:90	20:80	30:70
		
Degree of rusting: 0	Degree of rusting: 0	Degree of rusting: 0

Table 4 (ii): Degree of rusting of steel coupons immersed in sodium chloride solution.

10:90	20:80	30:70
		
Degree of rusting: 0	Degree of rusting: 0	Degree of rusting: 0

coupons experienced minor to no corrosion, with the minor corrosion resulting from “edge effects” only. The best coating formulation that was able to protect the steel coupons against corrosion is 30:70 (EUPO:DGEBA). The results obtained from the solvent rub test showed that the coating experienced minimal thickness loss against organic solvents.

Acknowledgment

My sincere gratitude and thanks is extended to my supervisor, Madam Fuzieah binti Subari, as well as my co-supervisor, Dr Najmiddin Yaakob for their immeasurable amount of support and guidance that they have given throughout this research.

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