

The Effects of Increasing Unsaturation in Palm Oil and Incorporation of Carbon Nanotubes on Resinous Properties

Muhammad R. Islam, Mohammad Dalour H. Beg, Saidatul S. Jamari

Abstract—Considering palm oil as non-drying oil owing to its low iodine value, an attempt was taken to increase the unsaturation in the fatty acid chains of palm oil for the preparation of alkyds. To increase the unsaturation in the palm oil, sulphuric acid (SA) and para-toluene sulphonic acid (PTSA) was used prior to alcoholysis for the dehydration process. The iodine number of the oil samples was checked for the unsaturation measurement by Wijs method. Alkyd resin was prepared using the dehydrated palm oil by following alcoholysis and esterification reaction. To improve the film properties 0.5wt.% multi-wall carbon nano tubes (MWCNTs) were used to manufacture polymeric film. The properties of the resins were characterized by various physico-chemical properties such as density, viscosity, iodine value, saponification value, etc. Structural elucidation was confirmed by Fourier transform of infrared spectroscopy and proton nuclear magnetic resonance; surfaces of the films were examined by field-emission scanning electron microscope. In addition, pencil hardness and chemical resistivity was also measured by using standard methods. The effect of enhancement of the unsaturation in the fatty acid chain found significant and motivational. The resin prepared with dehydrated palm oil showed improved properties regarding hardness and chemical resistivity testing. The incorporation of MWCNTs enhanced the thermal stability and hardness of the films as well.

Keywords—Alkyd resin, nano-coatings, dehydration, palm oil.

I. INTRODUCTION

INVENTION of new materials from renewable resources has been given importance nowadays due to the concern of sustainability, scarcity of traditional raw materials and their high price. Polymer, a widely-used material, is majorly being prepared from petroleum-based monomer. Since the reserved feed stocks of petroleum are going to be depleted soon, the alternative of this material should be ready to meet the high demand of polymer [1], [2]. Annually-renewable and plant-based vegetable oils are now being used extensively for polymer preparation instead. They are environmental-friendly and available at low-cost. Various kinds of vegetable oils like sunflower, castor, nahar seed and palm have been utilized to produce different kinds of polymeric resins [3]-[6]. The potential uses of these polymers include as adhesives, surface coatings, insulators, binders and composites preparation.

Alkyds are important materials and extensively being utilized in coating industry, and as a precursor for domestic and industrial products. These can be synthesized from vegetable oil-based triglycerides or fatty acids. Alkyds have some advantages like good film properties, fast-drying and versatility of applications [7]. There are many useful applications of alkyds which include coatings (for electronics and wood surfaces) and graphical designs [8]. Synthesis and preparation of triglyceride oil-based polymers were discussed earlier, where various kinds of alkyd resins and their methods of preparation with property analysis were also highlighted [9]. Two common reactions are involved for the preparation of alkyds: alcoholysis and esterification [10]. In alcoholysis, a reaction between polyol and triglyceride is taken place in the presence of acid or base catalyst to produce mono- or/and diglycerides, which further reacts with dicarboxylic acid to prepare alkyds, known as esterification [10]. The fatty acid chain in the polymer contributes improved flexibility, adhesion and chemical resistivity. Previously, nahar seed oil was studied to produce three different types of polyesters by using phthalic and maleic anhydride [5]. The produced polymeric films were found highly chemical resistant, and can be used as non-polluting coating materials. In another work, water-borne coatings were prepared from rubber seed oil [11]. Different amount of maleic anhydride (MA) were used to treat the rubber seed oil for preparing non-polluting coatings and alkyd resins. It was found that addition of MA improved the acid and saponification value, whereas iodine value found to be decreased. The produced film showed high chemical resistivity to acid and brine solution. In another work, synthesis and characterization of alkyds were studied from jatropha and rapeseed oil to prepare varnish for electrical insulating purpose [12]. The level of standard was achieved by resins prepared from the oils. Among the non-drying oils, palm oil was considered to prepare alkyd, polyester acrylate and water-reducible acrylic alkyd in different studies [13], [15]. Reports on those stated positive effect of using palm oil as surface coating materials. Issam and co-workers reported in their work that without using drying agent, the curing process found difficulties, but using of drying agent, the produced resins showed excellent adhesion onto aluminium surface as a substrate [15]. Ali and co-workers reported the preparation and characteristics of UV-curable acrylate polyester pre-polymers, which were synthesized from palm oil for wood coating application [13]. Different diluents were used to prepare different types of films. Different characterization

Muhammad R. Islam, Muhammad Dalour H. Beg, and Saidatul S. Jamari are with the Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Gambang-26300, Kuantan, Malaysia (e-mail: remanraju@yahoo.com, dhbeg@yahoo.com, sshima@ump.edu.my).

techniques such as pendulum hardness, gloss and impact testing were performed to evaluate the properties of the polymers. Excellent adhesion property with gloss (at 60°) ranged from 58 to 74 were reported in the discussion. In addition, pre-polymer prepared from refined, bleached and deodorized palm oil, which showed better performance than that of crude palm oil-based one.

Among the nanoparticles, multi-wall carbon nanotubes (MWCNTs) are important as filler, which can improve the properties of the polymer matrix, at very small amount of loading [16], [17].

Palm oil is the number one in position regarding the world-wide annual productivity. The huge amount of oil is being utilized mostly as cooking oil, and the commercial applications in non-food sectors are still rare. This oil can be utilized successfully for polymer preparation. Earlier, alkyd-type resins were prepared from palm oil, but a detailed report was not found in the previous document. Moreover, the limited unsaturation in the fatty acid chains of this oil was found to be responsible for inferior curing properties compared to other drying oil based resins. Therefore, in the curing process, curing agent and dryer are needed additionally with heating. Palm oil was used to produce alkyd-based film with good physical properties such as high gloss, toughness and durability [18], [19]. Although the iodine value of palm oil is low, the abundant availability of this oil and comparatively low cost motivates researchers to work on it for biopolymer synthesis. Due to low iodine value and non-drying property, the drying time of palm oil based resins have been experienced longer drying time compared to other vegetable oil based resins, such as castor oil and rapeseed oil [20]. In this study, different types of anhydrides of dibasic acid including aromatic and aliphatic category were used to prepare alkyds, and their properties were analyzed in detailed. In addition, the unsaturation in the fatty acid was improved by dehydration process, which was found significant to improve resinous properties. Moreover, the effects on the properties due to the incorporation multi-wall carbon nanotubes (MWCNTs) were also discussed.

II. EXPERIMENTAL

A. Materials

Palm oil was kindly supplied by Malaysian Palm Oil Board (MPOB). Phthalic anhydride (PA) and methyl ethyl ketone peroxide (MEKP) was purchased from Aladdin Industrial Corporation, Shanghai, China; glycerol and calcium oxide (CaO) were purchased from Aladdin Chemistry Co. Ltd., Shanghai, China. Cobalt-naphthenate was purchased from Sigma Aldrich, USA. Maleic anhydride (MA) was purchased from Acros Organics, New Jersey, USA. Multi-walled carbon nanotubes (MWCNTs) of purity 95%, diameter less than 8 nm and length ranges between 10 to 30 μm was purchased from Timesnano, China. Para-toluene sulphonic acid (PTSA) was purchased from Shanghai Lingfeng Chemical Reagent Company Ltd., China. Sulphuric acid (98%) was purchased

from Fisher Scientific, USA. Styrene was purchased from Sigma Aldrich, USA.

B. Dehydration Process

In the process of dehydration of triglyceride molecule, 2% of sulphuric acid was used in the presence of 2% pumice for the dehydration process. Nearly 50 g of oil sample was taken in a reactor with the said amount of sulphuric acid and pumice. The reaction time was maintained for 15 mins and an inert atmosphere was ensured by following nitrogen gas. The water generated from the reaction was collected through a dean stark apparatus. Finally, the iodine value of the sample was measured to calculate the unsaturation present in it. The same process was repeated for 4% PTSA.

C. Preparation of Resin

A four-necked glass vessel was used for the reactions. The vessel was placed on a magnetic heater, which was fixed with an automatic stirrer. An inert atmosphere was confirmed in the reaction chamber by a continuous flow of nitrogen gas. In the alcoholysis process, oil, glycerol and catalyst (CaO) were taken in the reaction chamber, and heated for about 1.5 h at 230°C. The end point of the reaction was confirmed by the complete dissolution of the reaction products into methanol at a ratio of 1:3. After that, the reaction products were cooled down to 120°C. In esterification process, PA and MA were used at the said ratio with the reaction products of the alcoholysis. The temperature was maintained at 230°C for nearly 2 h. The end point of the reaction was confirmed by measuring the acid value of the products. The reaction steps are illustrated in Fig. 1. After esterification, the reaction products were considered as liquid resin, and kept for further testing. Table I represents the name tags of different formulation of resins.

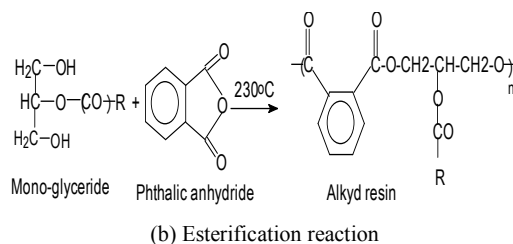
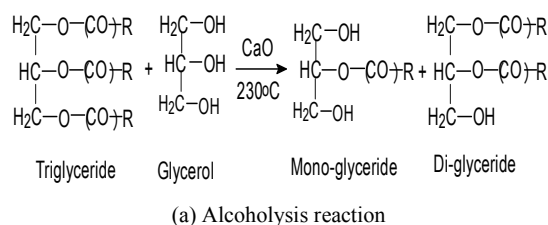


Fig. 1 Alcoholysis (a) and esterification reaction (b)

TABLE I
INGREDIENTS FOR THE PREPARATION OF RESIN

Resin type	Oil type	Oil (g)	Glycerol (g)	CaO (%)	PA (g)	MA (g)
Resin-A	Crude oil	100	20	0.05	37.5	12.5
Resin-B	Dehydrated oil (PTSA)	100	20	0.05	37.5	12.5
Resin-C	Dehydrated oil (SA)	100	20	0.05	37.5	12.5

D. Curing Process

The curing process of the liquid resins involves the formation of linkage among the polymeric chains through the unsaturation present on them. Likewise normal polymerization, initiation of the curing is required and for that an initiator must start the process. In this process, MEKP (0.04 g), cobalt naphthenate (0.02 g) and styrene (0.03 g) were mixed with 1 g of resin and agitated manually at 60°C for 10 min. After that, the resins were poured onto Petridis and put in oven for curing process. The samples were checked frequently to trace the dryness or tackiness by using the finger tip. Only trace free samples were considered as cured polymeric film for further characterization. The time for curing was recorded as drying or curing time of the resins.

E. Characterization

The physicochemical properties of oil and resins such as iodine value and saponification value were measured according to ASTM D5554 and ASTM D1962, respectively. The viscosity of the oil was measured by using BROOKFIELD (model- DV-IIIULTRA) programmable rheometer, USA. The measurement was taken at 20°C by using spindle no. 18. The density of the oil was determined by a gas pycnometer (MICROMETRICS, model-ACCUPYC II 1340) using helium gas at 25±2°. To examine the structural and functional groups, a Fourier transform of infrared spectrophotometer (Model- Thermo SCIENTIFIC, NICOLET AVATAR 370DTGS, USA) was used. The KBr method was employed for the analysis with scanning ranges from 400 to 4000 cm⁻¹. The proton nuclear magnetic resonance (¹H NMR) spectra were recorded on a Nuclear Magnetic Resonance 500 MHz (Model: FT-NMR Avance III, Bruker, Switzerland) dissolving the samples in deuterated chloroform.

To measure the drying time, the aluminum plate of 50 x 50 x 0.1 mm³ dimension was coated by immersing in resin at temperature 25± 2°C. The specimens were allowed to stand immersed in resin until free from bubbles and suspended for drying in dust free atmosphere. A piece of filter paper was used to check the tack free sample.

The gloss of the cured resins was measured by following ASTM D523 method using a 60° gloss meter (Model-DR60A, China). The pencil hardness of the cured resin was measured with the help of a pencil hardness tester (Model- B-3084) in scale of 6B to 6H of a standard set of pencils by dragging the pencil along the films by following ASTM D3363 method. A thermogravimetric analyzer, TA instrument (model- TA-Q500), was used to determine the thermal decomposition behavior of the polymeric films. Nearly 5 mg of the sample

was taken in a platinum pen for the testing. The heating rate was 20°C/min with the temperature range from 30 to 600°C.

For chemical resistivity, near about 0.5 g of each type of cured films was immersed in various chemicals, such as hydrochloric acid (5%), sodium hydroxide (5%), sodium chloride (5%) and distilled water for 24 h at room temperature. For the test, a definite amount of material of each type was immersed in the aforementioned solutions and measured the weight loss after the time period. A relative expression of the measurements such as excellent (not affected), fair (not affected but changes in color) and poor (less affected) were used to indicate the chemical resistivity of the film based on their weight losses.

The surface of the films was observed by a field-emission electron microscope (model-JEOL, JSM-7800F). The samples were coated with gold by a vacuum sputter coater prior to observation.

III. RESULTS AND DISCUSSION

A. Physicochemical Properties

The physicochemical properties of the oil are listed in Table II. The density and viscosity of the oil was found 0.8886 g/cm³ and 86.4 mPa.s at 25°C, respectively. The oil was non-drying in nature with low iodine value of 49 g I₂/100 g of oil. saponification value was found to be 209 mg KOH/g of oil sample. The fatty acid compositions of palm oil were found to be as follows: 43.5% palmitic acid, 36.6% oleic acid, 9.1% linolic acid, 4.3% stearic acid, and 1% myristic acids.

Various physicochemical properties such as iodine value, saponification value, density and viscosity of the resins are listed in Table II. The density of the resins was found to be in the range of 0.9045 to 0.9202 g/cm³, which is higher than the oil's density. The same trend of result was observed for the case of viscosity of the resins. The viscosity of the resin A, B and C was found 92.1, 97.2 and 99.6 mPa.S at 25°C. The density and viscosity found improved due to increasing of unsaturation in the polymeric chain of the alkyds. In addition, the crosslinking in the polymers are important determinate for the viscosity, which was found best in resin C compared to the others [12]. The unsaturation of the resins (Resin B and Resin C) was found to be increased compared to that of crude oil-based one, which is indicating the effectiveness of dehydration process. The saponification value was found to be in the ranges of 233 to 258 mg KOH/g of sample. However, the specific gravity of the resins was found to be changed little compared to that of oil. The formulated resins were found to be contained very low amount of moisture (0.1 to 0.15%). The non-volatile material content was found to be as high as 96.2 to 98.2%, which is indicating the solid portion of the resins and categorizing them as non-polluting materials [8].

TABLE II
PROPERTIES OF PALM OIL AND VARIOUS TYPES OF RESIN

Properties	Oil	Resin-A	Resin-B	Resin-C
Density (g/cm ³)	0.8886	0.9045	0.9134	0.9202
Viscosity (mPa.s) (Spindle-18, rpm-5 at 25°C)	86.4	92.1	97.2	95.6
Iodine value (g I ₂ / 100 g)	49	50.53	65.75	77.03
Saponification value (mg KOH/g)	209.21	233.32	245.43	258.33
Specific gravity	0.9630	0.9667	0.9601	0.9621
Colour	Light yellow	Light brown	Light brown	Light brown
Moisture content (%)	0.27%	0.10	0.11	0.15
Non-volatile material content (%)	-	97.2	98.2	96.2
Yield (wt%)	-	67	75	62
Drying time	-	11 hr 32 min	6hr 11 min	5hr 23 min
Gloss (60°)	-	65	75	78
Pencil hardness	-	2B	B	B

B. Structural Properties

In Fig. 2, the FTIR spectrum of palm oil is illustrated. The major peaks in against of their functional groups were identified through this observation. Peaks at around 720 and 1150 cm⁻¹ were probably due to the methylene rocking vibration and C-O-C stretching vibration of ester, respectively. The C-H bending vibration and C=O stretching vibration of triglyceride esters were confirmed by the peak at 1455 and 1750 cm⁻¹, respectively. The C=C stretching vibration for the unsaturated fatty acids can be depicted by the peak at 1650 cm⁻¹. The -CH stretching vibration for aliphatic compound was detected by the peaks rise at around 2855 and 2950 cm⁻¹. The -OH stretching vibrations were established by the small peaks in the region of 3200 to 3600 cm⁻¹.

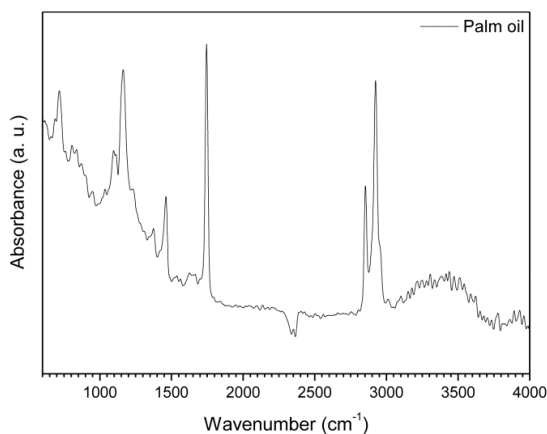


Fig. 2 FTIR spectra of palm oil

Fig. 3 demonstrates the ¹H NMR spectrum of palm oil. From the figure, the peaks at $\delta = 0.85\text{--}0.90$ ppm were probably for the proton of terminal methyl group. The protons of all the internal -CH₂ groups present in the fatty acids were confirmed by the peaks at $\delta = 1.60$ ppm. Peak at $\delta = 2.0$ and that at $\delta = 2.75$ ppm were probably for allylic and double allylic proton, accordingly. The α -proton of ester group was found to be at $\delta = 2.30$ ppm. The -CH₂- protons of glycerol

was found to be at $\delta = 4.1\text{--}4.3$ ppm and the protons for -CH of glycerol and unsaturated carbons were appeared at $\delta = 5.33\text{--}5.35$ ppm.

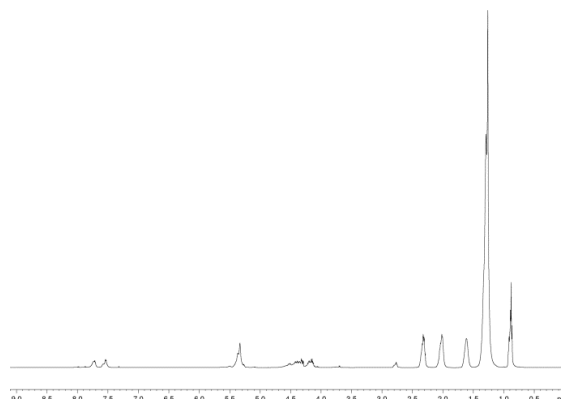


Fig. 3 ¹H NMR spectra of palm oil

The functional group and structure of the resins were determined by FTIR analysis (Fig. 4). From the figure, it can be seen that the absorbance peak at around 750 cm⁻¹ was due to aromatic -CH bending vibration, whereas the peak at 980 cm⁻¹ because of C-C stretching vibration. C-O-C stretching vibrations can be depicted by the peaks at around 1120 and 1250 cm⁻¹, respectively, for aliphatic and aromatic compound. For all the resins, the peaks at around 1740 cm⁻¹ for the carbonyl group (C=O) stretching vibration, which confirmed the changes around the peak obtained for the same for the case of oil, at around 1750 cm⁻¹. This change is probably due to the modification around the carbonyl group for the presence of aliphatic and aromatic anhydride compound in the backbone chain of the polymer. Next to carbonyl group, the asymmetric and symmetric vibration for -CH₂ can be ensured by the peak at around 2850 cm⁻¹, while that at 2920 cm⁻¹ helped to anticipate the stretching vibration for -CH moiety.

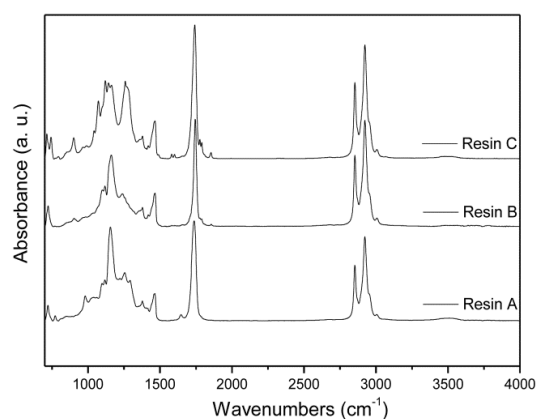


Fig. 4 FTIR spectra of different resins

The information for proton density of different moieties can be depicted in the Figs. 5-7 by ¹H NMR spectra of the resins A, B and C, respectively. The proton for terminal methyl

groups of fatty acids was confirmed by the peak at around δ 0.87. Peaks at around 1.25 to 1.30 were due to protons of all -CH₂ present in the chain. Peaks appeared at 4.2 to 4.4 were due to the methylene protons of glycerol molecule and that at 5.25 to 5.4 due to the unsaturated carbon. The peak appeared at 6.25 to 6.85 ppm was probably for the presence of -CH in glycerol molecule. The proton for the aromatic ring can be depicted by the peak at δ 6.8–7.5.

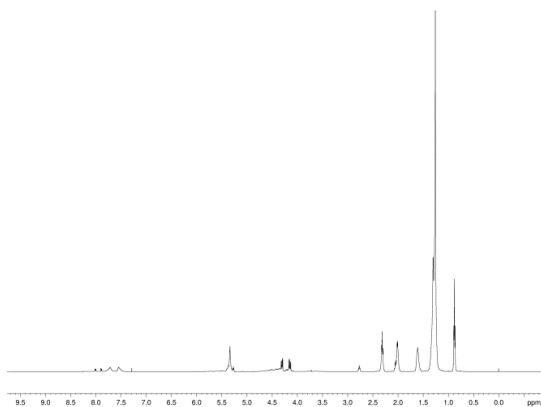


Fig. 5 ¹H NMR spectra of Resin A

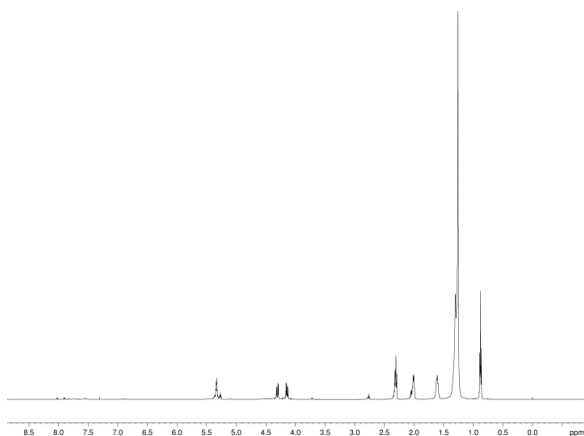


Fig. 6 ¹H NMR spectra of Resin B

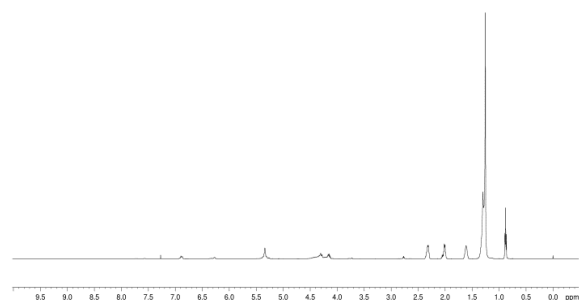


Fig. 7 ¹H NMR spectra of Resin C

C. Pencil Hardness, Drying Time, Gloss and Surface Morphology

The gloss at 60° was measured for all the films prepared from the resins. Acceptable gloss was showed by the resins of

values 65, 75 and 78 for the case of resin A, resin B and resin C, respectively (Table II). MWCNT based one showed the gloss value of 80. The drying time was found longer for the resin based on crude palm oil, but that of dehydrated-based oil showed shorter drying time. The drying time for resin A, resin B and resin C was found to be 11 h 32 min, 6 h 11 min and 5 h 23 min, respectively. Finally, the hardness of the polymeric film was measured by pencil hardness tester and found improved due to unsaturation increment and incorporation of MWCNTs. They passed for the assessment of hardness for the application of pencil category 2B, while PTSA and SA dehydration based resins passed B and MWCNT-based film passed HB type. Although the film hardness depends on the crosslinking density of the surface of the film, but presence of stable and rigid aromatic moiety in the backbone chain of the all the films showed higher hardness [13], [14]. Fig. 8 shows the surfaces of the films. Due to improved unsaturation the film produced from Resin B and Resin C showed smooth surface compared to resin A. On the other hand, MWCNT-based film showed the dispersion of nano particle on a smooth surface.

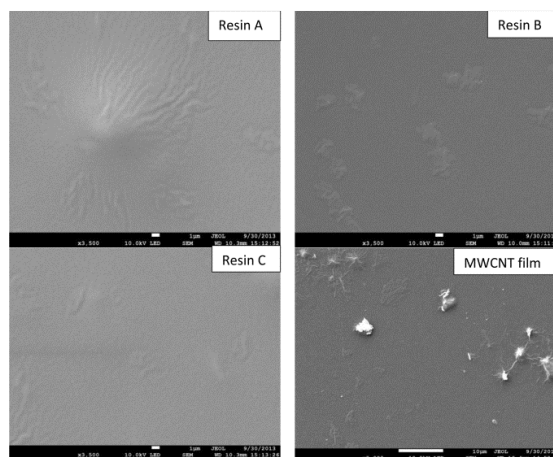


Fig. 8 FE-SEM images of different films

D. Thermal Properties

The thermal stability and degradation profile of various cured films are presented in Fig. 9. From the thermogravimetric analyses, it was found that nearly 1 to 2 wt% of moisture was removed during heating above 100°C for all the films. The removal of moisture and weight loss for that was confirmed by the isothermal heating of the polymers at 110°C for 2 h, which indicates a same amount of weight loss without any change in chemical structure as observed by FT-IR spectroscopy. It was found that all the types of films showed a higher thermal stability and the initiation of the degradation started near about at 306°C. It was found that resin C showed the highest thermal stability than the others. The onset degradation temperatures were found to be 306, 312 and 315°C for the case of Resin A, Resin B and Resin C, respectively. MWCNT based film showed the highest stability at onset 336°C. Thus the order of thermal stability was found as follows: MWCNT film > Resin C > Resin B > Resin A. The

higher thermal stability was probably due to the increase in cross linking density because of higher unsaturation present in the resin and presence of MWCNTs. The residues after 600°C for all the samples were found to be in the range of 5-7 wt%. The thermal stability of these resins was found to be high enough to be used for any type of high-temperature-sustained surface coating application.

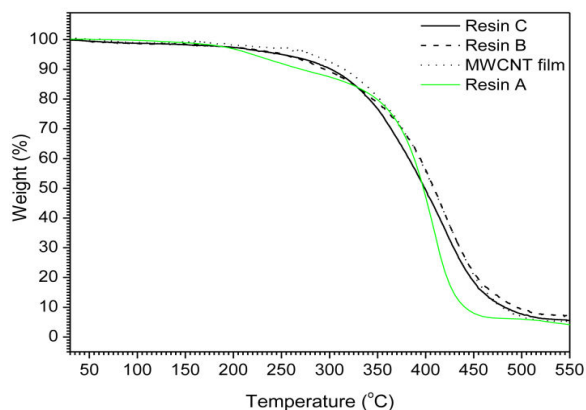


Fig. 9 Weight vs. temperature curves of different resins

E. Chemical Resistivity

The chemical resistivity performances of various cured films are presented in Table III. It was observed that all kinds of films were unaffected and highly resistant to distilled water, aqueous sodium chloride solution during the immersion period. Among the resins, resin C showed superior resistivity than the other two types of resins. For example, resin based on SA dehydration process showed excellent resistivity to NaOH solution and fairly resistivity to HCL solution, and this is thought to be happened due to the better crosslinking for additional unsaturation. On the other hand, MWCNT based film showed excellent property for all types of solution. The poor resistivity of Resin A is probably due to hydrolysable ester group present in it [15].

TABLE III
CHEMICAL RESISTIVITY OF VARIOUS TYPES OF RESINS

Solution	Resin-A	Resin-B	Resin-C	MWCNT film
Distilled water	Excellent	Excellent	Excellent	Excellent
5%HCl (aq.)	Fair	Fair	Fair	Excellent
5%NaOH (aq.)	Poor	Fair	Excellent	Excellent
5%NaCl (aq.)	Excellent	Excellent	Excellent	Excellent

IV. CONCLUSION

Two different types of dehydrating agents (sulphuric acid and para-toluenesulphonic acid) were used to improve the unsaturation in palm oil for the preparation of alkyd resin film. The increment of iodine value confirmed the enhancement of iodine value of the oil due to dehydration. Suphuric acid was found to be better compared to PTSA to improve the unsaturation in palm oil. Alkyd film was prepared by dispersing multi-walled carbon nano tubes at 0.5wt% for the improvement of film properties. MWCNTs based film showed

higher thermal stability with higher pencil hardness and chemical resistivity.

ACKNOWLEDGMENT

The authors would like to thank Universiti Malaysia Pahang for providing fund for this research project through research grant GRS 130357 and RDU 140332.

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