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Effect of CuO nanoparticle on mechanical and thermal properties of palm oil based alkyd/epoxy resin blend

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Abstract

In the present work, palm oil based alkyd resin was prepared with and without the presence of CuO nanoparticles. The curing of the prepared alkyd resin with different ratio of epoxy resin in the presence of poly(amido amine) as hardener was investigated. The mechanical properties, such as tensile and flexural strength of the cured blends were measured. The interaction between alkyd, epoxy and CuO nanoparticle were investigated by using Fourier transform infrared spectroscopy (FTIR). It was found that, crosslinking between alkyd and epoxy is possible through the formation of interpenetrating polymer network (IPN). The thermal stability and wettability of the blend was also investigated. It was found that, the CuO incorporated nanocomposite presented greater thermal stability and hydrophobicity than neat composite.

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1. Introduction

In recent years, intensive research on the development of polymers from renewable resources has been triggered due to the environmental concerns¹. Vegetable oils due to their low cost, availability and biodegradability are attractive starting materials for the production of alkyd, epoxy, polyurethane resins²⁻⁶. Oil derived alkyd resin is mainly used as binder in paints formulation, surface-coating adhesive and making composite materials⁷⁻⁹. However,

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the vegetable oil based alkyd resin are suffers in terms of practical applications due to some drawbacks such as longer curing time, low thermal and mechanical stability. To overcome this drawbacks, vegetable oil based alkyd resin can be blended with epoxy, amino and ketone functionalised resins to form the composite¹⁰.

Nanoparticle is promising nowadays due to its ultrafine size and high surface area. The inclusion of nanoparticle (<100 nm) in polymer can enhance the thermal, mechanical, electrical properties¹⁰⁻¹². The commonly used nanoparticles for incorporation with polymer are zinc oxide (ZnO)¹³, titanium dioxide (TiO₂)¹⁴, silver (Ag)¹², copper oxide (CuO)¹⁵ etc. Among them, CuO attracted more attention due to its high catalytic activity and low cost^{16,17}. The incorporation of nanoparticle into the polymer matrix usually performed through mixing after the formation of polymer^{12,13,15,18}. However, homogeneous distribution of nanoparticle in polymer matrix is difficult to achieve via mechanically stirring¹². In present work, CuO nanoparticle was prepared in glycerol medium which later used as one of the reagents during alkyd resin preparation. Our attempt was to create homogeneous dispersion of CuO nanoparticle throughout the polymer matrix. The effect of CuO nanoparticle, blending with different ratios of alkyd/epoxy on mechanical, thermal and wettability properties were investigated.

Nomenclature

CuO	Copper oxide	g	Gram
ZnO	Zinc Oxide	mL	Mililiter
TiO ₂	Titanium dioxide	w/w	Weight/weight
Ag	Silver	h	Hour
FTIR	Fourier transform infrared spectroscopy	cm ⁻¹	Centimeter ⁻¹
IPN	Interpenetrating polymer network	s	Second
MG	Monoglycerides	kN	Kilo-newton
NaOH	Sodium hydroxide	min	Minute
AV	Acid value	mm	Milimeter
TGA	Thermogravimetric analysis	mm/min	Milimeter/minute
UTM	Universal testing machine	MPa	Megapascal
°C/min	Degree Celsius/minute	°C	Degree Celsius
mg/L	Miligram/liter		

2. Experimental

2.1. Materials

Copper (II) chloride salt (CuCl₂•2H₂O), hydrazine monohydrate (64%), ethanol (99.9%), anhydrous methanol, sodium hydroxide, glycerol, copper (II) oxide (97%), copper (I) oxide (99.99%) and phthalic anhydride were obtained from Sigma-Aldrich, Malaysia and used without further purification. Epoxy resin (Epoxy equivalent weight: 170-180 g/eq) and hardener poly(amido amine) of commercial grade (Team Pacific Sdn. Bhd, Malaysia) were used as received. Refined palm oil was provided by Malaysian Palm Oil Board (MPOB), Malaysia.

2.2. Preparation of copper nanoparticles

The copper sol was synthesized by sol-gel method reported elsewhere^{16,19}. In brief, the required amount of CuCl₂•2H₂O was dissolved in 44 mL glycerol followed by 6 mL of hydrazine solution injected into the mixture drop-wise and the concentration of copper precursor in the sol was 200 mg/L. Reaction was conducted in a two-necked round bottom flask (100 mL) with magnetic stirrers at room temperature. Prior to this, the hydrazine solution of 7.062 mM was prepared by diluting 64% hydrazine monohydrate in ethanol. The solution was stirred continuously for 10 h at room temperature.

2.3. Preparation of alkyd resin

Alkyd resin was synthesized by alcoholysis of palm oil with glycerol followed by polyesterification. During alcoholysis, 100g of palm oil was heated at 240 °C and 22.2 g of glycerol (molar ratio of glycerol/oil = 2:1) was incorporated into the system under constant stirring. Thereafter, 0.3 g of NaOH was added and the reaction was conducted under nitrogen purging. Monoglycerides (MG) formation was determined via methanol solubility test²⁰: reaction mixture (1 mL) was withdrawn from reactor and cooled at room temperature prior to 2 mL of anhydrous methanol addition. Complete solubility of reaction mixture in methanol confirmed the 'end point' of alcoholysis and the reaction mixture was allowed to cool down at 140 °C. Thereafter, fine phthalic anhydride (38.7 g) was added into the reaction mixture to initiate the polyesterification process. The reaction temperature was maintained at 240 °C under nitrogen atmosphere. The samples were withdrawn at different time intervals to determine the acid value (AV) accordingly by ASTM D 1639-90²¹. The reaction mixture was quenched to room temperature when the AV reached ~ 10. For the preparation of alkyd resin catalysed by CuO-NaOH system, pure glycerol was substituted by the as-prepared copper sol in glycerol at concentrations, 200 mg/L.

2.4. Preparation of alkyd/epoxy blend

Alkyd resin and epoxy resin were blended in different wt. ratios of alkyd to epoxy 50:50, 40:60, 30:70, 20:80 and 10:90. In each case, the poly(amido amine) was added maintaining the ratio 2:1 (w/w) with respect to epoxy resin. The mixtures were vigorously mixed under continuous mechanical stirring for 1 h. The uncured mixture was placed on a Teflon coated mold with a uniform thickness (5 mm) and left at room temperature for 24 h and thereafter the sample was transferred to oven at 110 °C and kept for 3 h for curing.

2.5. Fourier transform infrared spectroscopy (FTIR)

The structural information of the sample was attained by FTIR Spectrometer, Nicolet, Impact 410, USA equipped with an attenuated total reflectance (ATR) device in the wave number range 600–4000 cm^{-1} and resolution 4 cm^{-1} . The FTIR spectra were taken in a transmittance mode.

2.6. Thermogravimetric analysis (TGA)

Thermogravimetric analysis of the blend samples were carried out by using Shimadzu TA-50WSI TGA instrument, thermal analyser under the nitrogen flow rate of 30 mL/min at the heating rate of 10 °C/min from 40 to 600 °C.

2.7. Contact angle measurement

The hydrophobic behaviour of the blend was evaluated by contact angle measurements using a Drop Shape Analysis System (model DSA10MK2, Kruss GmbH, Germany) with a high speed CCD camera for image capture. Deionized water was used for the evaluation of the hydrophobic properties of the sample. Angle measurements were done in triplicate. Water was taken up in a syringe and drops (15 μL) were allowed to fall onto the substrate, and the left and right contact angles were measured for 10 s at 1 s intervals². The average contact angle was calculated by using the three values of left and right contact angles.

2.8. Mechanical properties measurement

Mechanical properties of blend samples such as tensile strength, flexural strength, were examined using a Shimadzu universal testing machine (UTM, Model: AG-1). Tensile testing was conducted according to ASTM 638-08; UTM was fitted with a 5 kN load cell and operated at a cross-head speed of 1 mm/min by keeping 65 mm gage length. Flexural test was conducted according to ASTM D790-97; UTM was fitted with a 1 kN load cell. The support was set at 20 mm and the crosshead speed was fixed at 10 mm/min.

3. Results and discussion

3.1. Mechanical properties

All The composite material was prepared by blending the as-prepared alkyd resin with commercial epoxy resin at different ratio. All the samples were cured at 110 °C for 3 h. The tensile and flexural strength of composite materials with and without CuO nanoparticle were measured and presented in Table I. It can be seen that, the tensile and flexural strength decreased with the increase of the alkyd resin content. Around 331% increase in tensile strength was observed when the alkyd to epoxy ratio was changed from 1:1 to 1:9. The flexural strength was also increased by 443% with the increase in epoxy content. The results indicated the presence of chain entanglement in alkyd-epoxy matrix, due to the formation of interpenetrating polymer network (IPN)²².

The tensile strength of sample (A/E 50:50) with and without CuO nanoparticles was 13 and 20 MPa, respectively showed 50% increment. However, tensile strength of the samples (A/E 10:90) with and without CuO nanoparticle was found to be 56 and 55 MPa, respectively showing almost no change. The unchanged values of tensile strength might be due to the presence of very low concentration of CuO nanoparticle in the matrix.

The increase in tensile and flexural strength of the alkyd/epoxy blend due to the incorporation of CuO nanoparticle might be due to the formation of nanocomposites^{10,23}. The increment in mechanical strength is rational, because CuO nanoparticles can act as plasticizer, which contribute to load sharing during the testing. Moreover, it can also increase the thermal conductivity of the matrix resulting to a higher curing rate. Although with the increase in alkyd content, the tensile and flexural strength decreased drastically, the loss was recovered by the incorporation of CuO nanoparticle into the blend matrix. The nanocomposite (A/E 30:70) exhibit about 18% and 41% improvement in tensile and flexural strength, respectively in comparison to neat blend (A/E 30:70).

Table 1. Tensile and flexural strength of blend

Alkyd/Epoxy	Tensile strength (MPa)		Flexural strength (MPa)	
	neat	CuO nanoparticle	neat	CuO nanoparticle
50:50	13	20	21	28
40:60	29	37	48	58
30:70	40	47	58	82
20:80	45	49	91	105
10:90	56	55	114	123

3.2. FTIR

FTIR analysis of the alkyd, epoxy, alkyd/epoxy blend at a ratio of 30:70 without CuO nanoparticle and with CuO nanoparticle are shown in Fig. 1. In the FTIR spectrum of alkyd (Fig. 1, curve i), a broad band was observed at 3540-3200 cm⁻¹ representing the O-H stretching vibration^{21,24-28}. The spectrum also showed a strong absorption in the region of 3030-2840 cm⁻¹ due to C-H stretching^{29,30}. The absorption bands at 1740-1725 cm⁻¹ was attributed to C=O and that at 1240-1050 cm⁻¹ for C-O-C stretching vibration of ester group³¹. Absorption at 1465-1445 cm⁻¹ was assigned to C-H bending. In addition, absorption peak at 743 cm⁻¹ was assigned for the presence of C-H bending of aromatic ring contributed by phthalate units³⁰.

The spectrum of epoxy is different compare to alkyd. From the spectrum of epoxy (Fig. 1, curve ii), it also showed a strong absorption in the region of 3030-2840 cm⁻¹ due to C-H stretching. The adsorption peak at 1609 cm⁻¹ and 1508 cm⁻¹ were assigned to the C=C stretching of aromatic ring^{32,33} and C-C stretching of aromatic³³, respectively. The absorption peaks at 1240-1030 cm⁻¹ were attributed to the C-O-C stretching of ether group³³. The absorption at 910 cm⁻¹ and 829 cm⁻¹ are attributed to stretching of C-O and C-O-C of oxirane group, respectively^{32,33}. The peak at 761 cm⁻¹ is due to rocking of CH₂³³.

The FTIR spectra of alkyd/epoxy blend (Fig. 1, curve iii and iv) indicated the formation of amide bond between ester of alkyd and amine hardener, where the peak of C=O was shifted from 1730 to 1740 cm⁻¹³⁴. Apart from that, the peak intensity of C-O and C-O-C of oxirane group were reduced, especially peak at 910 cm⁻¹ was diminished.

This indicates the formation of network between oxirane groups with amine hardener. The peak of O-H groups became broader compared to its origin, which might be due to the formation of hydrogen bonding between hydroxyl-carbonyl (Scheme 1). Furthermore, the stretching frequencies of the blend spectra without CuO (curve iii) and with CuO nanoparticle (curve iv) were almost the same. However, slight shifts of C-O-C of oxirane group was noticed, from 829 to 824 cm^{-1} , suggesting the interaction between polymer matrix and inorganic material (CuO)³⁵.

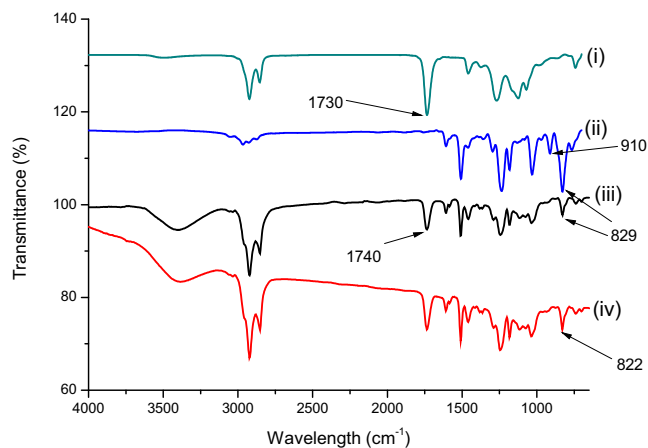


Fig. 1. FTIR spectra of alkyd (i), epoxy (ii), alkyd/epoxy blend (A/E 30:70) without CuO (iii) and with CuO (iv).

3.3. TGA

The thermal behavior of the resins was analyzed using TGA at a constant heating rate of 10 °C/min in the temperature range of 40 to 600 °C. The studies were conducted in inert (under N_2) atmosphere to eliminate the diffusion of oxidant gas through the polymeric blends with different composition³⁶. In Fig. 2a, a 5-10% weight loss was noticed in the temperature range of 100 to 150 °C due to the loss of adsorbed solvent molecules². The significant weight loss from 250 to 550 °C resulted from the degradation/decomposition of aliphatic moieties of epoxy and aromatic moieties of alkyd^{1,10}. The TGA curves showed that the blend resins were degraded in a single step pattern leaving 10-17.5% residue above 580 °C. As seen from Fig. 2a, A/E 10:90 is more thermally stable than the other blends, indicates that epoxy resin improved the thermal stability of the blends. The good thermal stability of the resins might be due to some cross-linking generated between free hydroxyl groups present in alkyd or epoxy via hydrogen bonding³².

The effect of CuO nanoparticle on thermal stability of the blend sample (A/E 30:70) is presented in Fig. 2b. The incorporation of CuO nanoparticle improved the thermal stability of blend sample. The initial degradation temperature (T_i) for A/E 30:70 blend was 280 °C, where the blend incorporated with CuO nanoparticle shifted to 300 °C which might be due to the heat shielding effect of nanoparticle¹⁰. Linear coefficient of thermal expansion of polymers are much higher than metal oxides and due to this fact, most of the heat absorbed from surrounding by the epoxy/alkyd blend is used in increasing interatomic distance rather than increasing the temperature of the polymer. This leads to the chain scission leading to lower thermal stability. On the other hand, the presence and homogeneous distribution of CuO nanoparticle in the nanocomposite might hinder the diffusion of volatile products through the composite resulting in higher thermal stability. Similar findings were also obtained for nanocomposites reinforced with nano Al_2O_3 , TiO_2 and NiO ^{10,37,38}. Mbhele, et al.³⁹ investigated the polymer decomposition mechanism, where the decomposition of polymers started with the free radical formation at the weak bonds and/or chains ends, followed by radical transfer to adjacent chains through inter chain reactions. In that context, the reduction of polymer chain mobility should suppress the chain transfer reaction. The homogeneous dispersion and strong

interfacial interaction between the CuO and the polymer matrix reduces the polymer chain mobility in the nanocomposites and consequently the degradation process is delayed³⁹.

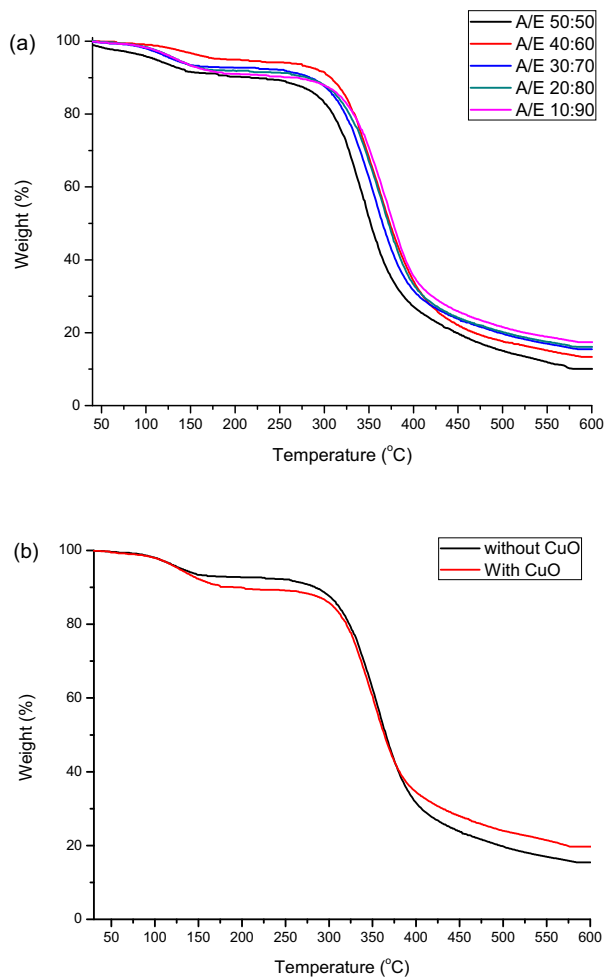


Fig. 2. TGA thermograph of alkyd/epoxy blends resin (a) and effect of CuO nanoparticle on A/E 30:70 blend (b).

3.4. Surface wettability test

The CCD camera images of water droplets on the surface of the alkyd/epoxy blends with and without CuO nanoparticle are shown in Fig. 3a–d. It can be seen from Fig. 3 that with the increase in epoxy composition, the contact angle value increased (from 55° to 85.5°) indicating the increase in the hydrophobicity of the blend. The result was expected, as the epoxy provided the hydrophobicity in the blend⁴⁰. The contact angle value of blend samples (A/E 30:70) with and without CuO nanoparticle were 85° and 75°, respectively. The contact angle increases 10° for sample incorporated by CuO nanoparticle, representing the enhancement of hydrophobicity of the sample. The hydrophobic nature of the sample could expand its applicability as outdoor material.

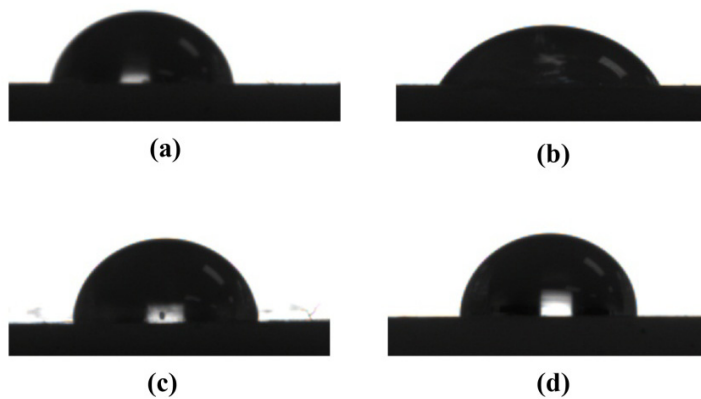
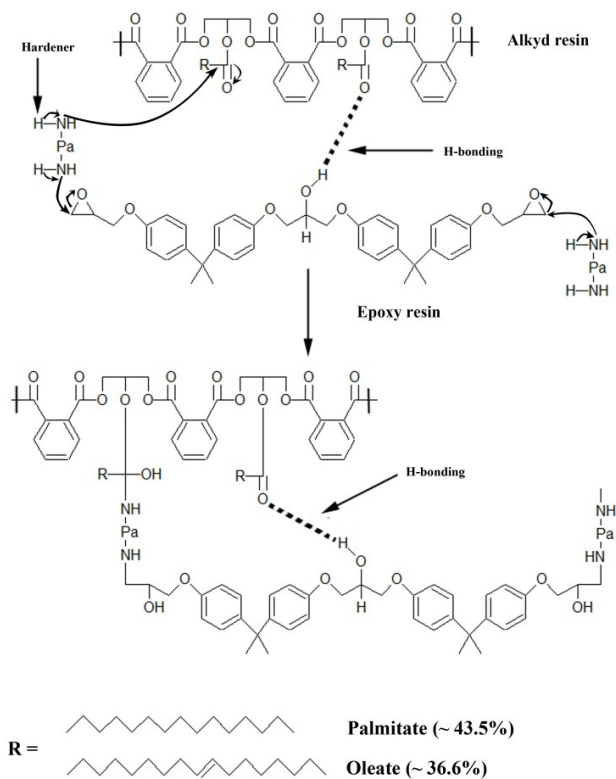


Fig. 3. Contact angle images of A/E blend without CuO nanoparticle 30:70 (a), A/E blend with CuO nanoparticle 50:50 (b), 30:70 (c) and 10:90 (d).



Scheme 1. Schematic diagram of the phenomenon occurring during the crosslinking of the blend.

4. Conclusion

In the present work, composite comprising of different ratio of alkyd/epoxy with and without CuO nanoparticle was prepared and effect of CuO nanoparticle on its thermal and mechanical properties was investigated. The tensile and flexural strength of blend samples decreased with the increase in alkyd resin content. The loss was recovered by incorporation of CuO nanoparticle, where nanocomposite (A/E 30:70) exhibited about 18% and 41% improvement in tensile and flexural strength, respectively when compared to neat blend (A/E 30:70). The formation of network between oxirane groups with amine hardener and interaction between polymer matrix and CuO nanoparticles were revealed via FTIR. Besides that, the CuO incorporated nanocomposite presented greater thermal stability than neat composite. The present work revealed that, the CuO nanoparticles prepared in the glycerol phase facilitated their homogeneous distribution in the resin phase and significantly influenced the thermal and mechanical properties of the nanocomposite.

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