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Utilization of Agrowaste-derived Nanoparticles as Reinforcement in Microfilled Epoxy Composites

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The substantial release of oil palm ash into ground water has been a serious concern to the environmentalist due to the enormous generation of oil palm ash waste from oil palm incineration. The effective utilization of this agrowaste is yet to be fully exploited. In this context, herein we, investigated the potential of oil palm ash nanofiller as an effective reinforcement in epoxy-based composites. Transmission electron microscopy (TEM) revealed that the prepared oil palm ash nanoparticles had circular morphology with particle size in the range of 20 to 25 nm. Xray diffraction patterns of the prepared oil palm ash nanoparticles revealed the crystalline nature of the oil palm ash nanoparticles. Tensile strength and tensile modulus of the epoxy composites were substantially improved to 64, 67, 70, and 75 MPa and 1.01, 1.05, 1.16, and 1.18 MPa at oil palm ash nanofiller loading of 1%, 2%, 3%, and 4%, respectively. The impact strength of nanocomposite was enhanced from 2.7015 ± 0.13 kJ/m² to 3.98 ± 0.17kJ/m² at 3% of oil palm ash nanofiller loading. The optimum values of mechanical properties were attained at 4% filler loading, after which further loading resulted in the decrement of mechanical properties of epoxy nanocomposite. Thermal stability of the epoxy nanocomposite was enhanced substantially to 435 °C by the incorporation of oil palm ash nanofillers. This study proved that nano-sized oil palm ash could be an efficient reinforcement in polymer composite.

Keywords: Oil palm ash; Epoxy resin; Nanocomposite; Thermal stability, Impact strength

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INTRODUCTION

Oil palm ash (OPA) is an abundant agricultural solid waste in major oil palm producing countries including Indonesia and Malaysia. This bio-agricultural waste has been produced after the combustion of oil palm fiber, shell, EFB, and mesocarp as boiler fuel to produce steam for palm oil mill consumption (Suraya *et al.* 2018). Although there are some studies on the utilization of OPA such as a cement replacement material (Thomas 2018) as an adsorbent for the removal of zinc from aqueous solution (Mondal and George 2015), and flue gas desulphurization (Thomas *et al.* 2017), most of the ash is still disposed of in landfills that require a lot of land area and have the potential to cause environmental problems. Raw OPA consists of spherical particles with a median size of 183.0 μ m, while medium and small particles ground palm ash were individually noted having crushed shape

structures with a median size of 15.9 and 7.4 μ m (Khankhaje *et al.* 2018). In most cases, the chemical elements of oil palm ash are found to be silicon dioxide, aluminium oxide, iron oxide, calcium oxide, magnesium oxide, sodium oxide, potassium oxide, and sulfur trioxide *etc.*, depending upon the geographical conditions, fertilizers used, climatic variation, soil chemistry, *etc.* (Foo and Hameed 2009). In Malaysia alone, oil palm ash production is around 4 million tonnes/year (Som *et al.* 2013), which results in the substantial accumulation of oil palm ash waste, thereby leading to environmental pollution. Moreover, the disposal of these oil palm ash waste is very expensive. Approximately \$5/tonnes are generated per year by developing countries, and the corresponding number is \$50/tonnes in developed countries. In this context, it is very important to find a solution to use these agro-wastes for developing value-added materials (Ooi *et al.* 2014).

In order to reduce the cost of production, over 50 million tons of fillers valued at about 25 billion euros are consumed globally for industrial applications, such as rubber, paint, paper, adhesives, etc. (Clark 2014). The main factors which influence the mechanical properties of the natural fillers based composite are the composition of the materials, the type of raw fibres, and their physicochemical properties (Fogorasi and Barbu 2017). Nanotechnology has become a rapidly emerging area with potential applications ranging from daily-used products to advanced applications. A nanocomposite with superior mechanical properties should have a good adhesion between the filler and matrix, a high modulus, low stress, minimal filler setting, low moisture absorption, fast cure, and should be highly durable (Parandoush and Lin 2017). In order to achieve the above mentioned properties, the parameters such as particle size distribution, percentage filler loading, filler dispersion in the matrix and the shape of the fillers are of utmost importance because of their effect on the properties of filled composites (Sri Aprilia et al. 2018). Replacing the use of the synthetic fillers in the composite manufacturing has been a serious challenge to reduce the ever-increasing cost of synthetic fillers. In this context, it is essential to study and investigate the reinforcing potential of agro-waste nanoparticles. So far, there are limited reports on the use of the unexploited nano-sized OPA as a nano-filler in epoxy resin composites. Moreover, this study also investigates the effect of OPA nanofiller with various filler loading on the functional properties of the composite. It is proposed that the demonstrated work will be an effective platform to design a nanostructured material as reinforcement for fabrication of polymer composites.

EXPERIMENTAL

Materials

The OPA used for this study was obtained from a palm oil mill incinerator at Ulu Keratong, Segamat, Johor, Malaysia. The epoxy resin used (D.E.R 331) was a clear epoxy resin of bisphenol A (DGEBA) supplied by GT Instruments Sdn. Bhd., Kuala Lumpur, Malaysia, while both the epicholorohydrin and the curing agent A 062 (a reactive polyamide epoxy hardener) were procured from Zarm Scientific and Supplies Sdn., Meleka, Malaysia

Methods

Preparation of OPA nanoparticles

Oil palm ash's major precursors include EFB, nut shells, and waste fibers. The OPA was first oven-dried at 105 °C for 8 h to remove water/moisture and ease processing. It was then sieved *via* a 250- μ m mesh size sieve to remove some impurities such as sand, stones, and other accompanying filths. Then, the OPA was sieved sequentially *via* 150- μ m, 75- μ m, and 50- μ m mesh size sieves for microfiller composites preparation. Fine OPA powder was further ground using a grinder/refiner (Janke & Kunkel GmbH & Co., KG, Zhejiang, China) followed by high-energy ball milling (ASTMD 88; Cast Iron; Associated Scientific & Engineering Works Sadar Bazaar, New Delhi, India) for 30 h (Swami *et al.* 2009) at 170 rev·min⁻¹. The ball mill was loaded with a ball-to-powder weight ratio of 10:1 in a stainless-steel chamber using stainless steel balls of diameters 19 mm, 12.7 mm, and 9.5 mm. The dried OPA was allowed to cool in a desiccator containing silica gel to prevent moisture absorption.

Preparation of nanostructured OPA-filled epoxy composites

The OPA-filled epoxy composite was prepared according to the procedure reported by Abdul Khalil et al. (2014) with some modifications. The initial compounding of the epoxy matrix with the microsized OPA was completed with an initial mixing of the epoxy resin with 30% microsized OPA from the 50-µm mesh size sieve in a stainless-steel mould of 160 mm (L) \times 160 mm (W) \times 3 mm (thickness) dimensions with the aid of a mechanical stirrer (Esel International Manufacturer and Exporter, Shandong, China). This composite blend was reinforced with 0%, 1%, 2%, 3%, 4%, and 5% loading of nanostructured OPA (based on wt% of resin) at room temperature for 30 min using a mechanical stirrer. This was followed by the addition of polyamide hardener at the ratio of 3:5 by weight of polyamide to epoxy while the stirring of the blend continued for 10 min. The compounded material mixture was poured into a flat surface mould and degassed in a vacuum chamber for 15 min to remove the bubbles. With the aid of the compression moulding technique, the compounded material was then casted into a mould, and the nanocomposite materials were left to cure at 105 °C for 1 h in a hot press (Gotech Hot Press-Gt-7014; Taichung Industry Park, Taichung City, Taiwan) at a pressure of 200 psi as per the supplier specifications. The specimen samples used for the determination of mechanical properties were prepared according to the American Society for Testing and Materials (ASTM) standards described in the following sections.

Characterization

Transmission electron microscope (TEM)

A Philips PW1050 (FEI Company, Washington DC, USA) TEM was employed to investigate the dimensions and morphology of the OPA nanofillers. The OPA nanofillers were mixed with water and a drop of this solution was dropped onto a carbon-coated copper grid and dried for 10 min.

X-ray studies

An X-ray diffractometer (WR14 1XZ; Rigaku SmartLab SE, Tokyo, Japan) was employed for obtaining X-ray diffraction (XRD) patterns of the OPA nanofillers at an accelerating voltage of 40 kV with a current of 25 mA. The OPA nanofiller was scanned in the range of 0° to 90° (2 θ). The particle size and crystallinity of the OPA nanofillers were obtained from the XRD profiles of OPA nanofiller. The average crystallite size of the OPA nanofiller was obtained using the Debye-Scherrer formula,

$$D = (k \cdot \lambda) / (\beta \cdot \operatorname{Cos}(\theta)) \tag{1}$$

where k is a constant, λ is the wavelength (cm⁻¹) of the radiation at the full width half maximum (FWHM) radiance of the XRD peak obtained at 2θ (°), β is the full width at half maximum intensity of the peak (in radians), and D is the crystallite size (nanometres).

Tensile test

The tensile strength, tensile modulus, and elongation at break of nanostructured OPA-filled epoxy composites were measured using an Instron 5582 universal testing machine (New Delhi, India). This test was conducted according to the ASTM D3039 (2000) standard specifications. The rectangular samples of dimensions 120 mm \times 15 mm \times 3 mm were cut with a circular saw. The gauge length was set at 60 mm and the testing speed used was 5 mm/min. In each case, five replicates were completed.

Flexural test

A flexural analysis was completed at room temperature through the three-point bending test according to the ASTM D790 (2003) standard protocol using an Instron universal testing machine (Instron Model 5582; Gujarat, India) Rectangular samples of dimensions 160 mm (L) \times 20 mm (W) \times 3 mm (thickness) were cut with a circular saw. Bending was conducted using a load cell of 5 kN with a crosshead speed of 2 mm/min loading. The test was replicated five times.

Impact test

The elastic capability of the reinforced composite for shock absorption prior to any permanent damage was demonstrated *via* the impact test. The Izod notched impact test was conducted according to ASTM D256 (2003) using an impact pendulum tester (Model: CS-1370; Zwick Roell Group, Selangor, Malaysia). The impact test was conducted on each of the representative samples of the composite at different levels of nanoparticle loading with dimensions of 70 mm (L) \times 15 mm (W) \times 5 mm (thickness). The samples were gripped in a vertical position and were struck using a pendulum at the centre of the samples. Each sample was tested with five replicates and the average value of the results was recorded.

Morphological study of the nanocomposite

Field emission scanning electron microscopy (FESEM) (EVO MA-10; Carl Zeiss, London, UK) was used to investigate the morphology of the OPA nanocomposite. The deposited nanocomposite samples were detected using backscattered secondary electrons (BSE) with a 15 keV electron beam at $500 \times$ magnification. The tested samples were gold coated with a Fisons Polaron sputter coater (Polaron SC500; Fisons, London, UK) prior to the examination.

Thermogravimetric analysis (TGA)

The TGA was performed to evaluate the thermal stability of OPA nanoparticles at different OPA nanofiber loadings. The weight losses of the composite samples were measured as a function of temperature. The thermal analysis was completed under nitrogen atmosphere using a thermogravimetric analyzer (Perkin–Elmer, Shelton, USA) at a flow rate of 20 mL/min. A total of 10 mg each of dried composite sample was transferred into

the sample holder and heated at 10 °C/min under nitrogen flux from 30 °C to 760 °C using a Perkin Elmer YGA7 instrument. The thermogram recorded the percentage of weight loss against the temperature and the amount recorded.

RESULTS AND DISCUSSION

Morphological and Dimensional Characterization of the OPA Nanofillers

To ascertain the morphological and dimensional values of the applied OPA particles, the TEM was used to monitor the nanosized scale of the OPA particles. From the TEM image in Fig. 1a, it was clearly evident that the OPA nanoparticle were circular in shape, and the particle size was in the range of 20 to 25 nm. Figure 1b shows the particle size frequency distributions of the prepared OPA nanoparticles and from this, particle size of OPA nanoparticle was evaluated. The XRD analysis of the OPA nanofillers was performed to investigate the crystal nature and evaluate the particle size using Scherrer's equation.



Fig. 1. TEM image of the OPA nano-filler (a), particle size frequency distributions of the OPA nanofiller (b) and XRD diffractogram of OPA nano-filler (c)

The XRD profile of the OPA nanofiller showed the crystalline nature of the OPA nanofiller. At 22.6°, a high intensity peak was observed (Fig. 1b), which was due to the presence of silicates in the OPA nanofillers, whereas the low intensity peaks were assigned to other metal oxides that were present in the OPA nanofillers (Abdul Khalil *et al.* 2013). From the XRD analysis, the calculated particle size of the OPA nanofiller was 26 nm, which was in agreement with the particle size obtained from the TEM image. It is well known that nanoparticles in the range of 20 to 25 nm can be an efficient reinforcement for biocomposites (Fortunati *et al.* 2016).

Effect of OPA Nanofiller Loading on the Mechanical Properties

Figure 2 presents the effects of increasing the nanosized OPA filler content on the tensile strength and elongation at break of the nanocomposite. As shown in Fig. 2, the tensile strength and tensile elongation were enhanced with respect to the increase in the OPA nanofiller loading up to 4%.





Fig. 2. Effect of OPA nano filler content on (a) tensile strength, (b) tensile modulus, and (c) elongation at break the of nano composites

Tensile strength and the tensile modulus of the micro-sized OPA particles with pure epoxy materials without the nano particle loading was 45.8 MPa and 0.78 MPa, respectively. There was a gradual increase in the mechanical properties by the addition of nano sized OPA filler at 1%, 2%, and 3% up till 4% nano filler loading (64, 67, 70, and 75 MPa) and (1.01, 1.05, 1.16, and 1.18 MPa) for both the tensile strength and tensile modulus respectively (Fig. 2) after which further OPA nano particle loading resulted in a decrease in the mechanical properties. This result is in agreement with the observation reported by Abdul Khalil *et al.* (2013). The increase in both the tensile strength and tensile modulus could be attributed to the good dispersion of OPA nano-fillers in the epoxy matrix which facilitated the effective stress transfer from the epoxy matrix to the OPA nano-fillers. When bonding between matrix and filler is strong enough, there is improvement in the interfacial shear strength and the tensile strength of a particulate composite (Rizal *et al.* 2018).

The nano-filler loading further than 4% OPA resulted in the decrement of both the tensile strength and the tensile modulus. This could be due to the agglomeration of OPA nano-fillers at the higher percentage of loading, as shown in the SEM image (Fig. 5). This agglomeration could have resulted from the weak filler-matrix interaction and weak interfacial shear bond. This observation was comparable with the result reported by Bhat and Abdul Khalil (2011), which stated that there was a high tendency for OPA nanoparticle to agglomerate in humid conditions due to low surface interaction of the filler with the unsaturated polyester matrix. Sutopo and co-workers (Sutopo et al. 2017) found that at higher loading, OPA nanoparticles could agglomerate without proper dispersion, which resulted in the decrement of mechanical properties of OPA reinforced unsaturated polyester matrix. Consequently, at this stage there was bound to be poor stress transfer from the clustered fillers to the matrix and vice versa. From these results, it can be concluded that at higher percentage OPA nanofiller loading, the mechanical properties were decreased due to the agglomeration of OPA nano-fillers at higher loading of OPA nano-fillers. The effect of OPA nano particle loading on the elongation at break of the nano-composite is also shown in Fig. 2. The result shows that the elongation at break values of the composite were reduced with increasing OPA nanoparticle loading.

Basically the elasticity response of a composite is its accessibility to the elasticity of its matrix (Riaño *et al.* 2016). Since the OPA particles are inelastic nano-particles, they tend to reduce elasticity property of the polymer matrix as the OPA filler loading increases. Hence, a high value of elongation at break is which indicate highly elastic material is observed in the nanocomposite with no nanofiller loading. This result is in agreement to the result reported by Gourier and co-workers (2017). The cited work indicated that the reduction of elongation at break of mica/epoxy composites was due to the increased rigidity of the composite as a result of the higher loading of OPA into unsaturated polyester. Therefore, the nanocomposite with the highest OPA nano-filler loading showed the least elongation at break.

Flexural Properties of the Nanocomposite

The results of the tests of flexural strength and flexural modulus of the OPA nanocomposite system as a function of filler loadings are presented in Fig. 3. The results showed an increasing trend for the flexural properties as the OPA filler loading increased. The flexural strength and the flexural modulus of the nanocomposites increased as the OPA filler content of the nanocomposites increased up to 4%, after which further loading recorded a drop in the flexural properties. The enhancement in the flexural strength and flexural modulus of the composite up to 4% of OPA filler loading was due to the good dispersion of OPA nanofillers in the epoxy matrix. Gradual increase in the flexural properties with increased nanofiller loading were due to the good dispersion of the nanoparticles in the epoxy matrix, which was confirmed from the FESEM images to be shown later. The flexural modulus, which indicates the relative stiffness of a material, was also attributed to the ability of the material to maintain its original dimensions before being ruptured by the flexural load.



Fig. 3. Effect of OPA nanofiller content on the flexural strength and flexural modulus of the nanocomposites

From the results, both the flexural strength and the flexural modulus of the OPAfilled epoxy nanocomposites were 135 MPa and 7.036 GPa, respectively, at 4% OPA nanofiller loading.

Impact Properties

The impact strengths of the nanocomposites are presented in Fig. 4. The impact strength of the composite without the OPA nanofiller loading was $2.7015 \pm 0.13 \text{ kJ/m}^2$. The impact strength of the composite enhanced with the OPA nanofiller loading reached an optimum value of $3.98 \pm 0.17 \text{ kJ/m}^2$ at 3% OPA nanofiller loading, while further addition of the OPA nanofiller resulted in a decrease in the impact strength to $3.19 \pm 0.15 \text{ kJ/m}^2$. The results showed that the nanocomposite with 3% OPA nanoparticle filler loading had the highest impact strength. It has been reported that the impact properties are governed by the interfacial and interlaminar bonds between the fiber and the matrix because the matrix is not the only influence on the impact properties of polymer composites (Liu *et al.* 2015).



Fig. 4. Effect of OPA nanofiller content on the impact strength of nanocomposites

In addition, it was reported that, delamination of the particles serving as reinforcement within the polymer matrix enhances the impact strength properties in addition to interfacial strength with composites reinforced with OPA nano fillers (Lu *et al.* 2015). The same observation was reported by Chan and co-workers (2015) relative to the impact strength of oil palm empty fruit bunches hybrid epoxy composite, where higher strength of glass fibres had provided greater impact strength even with interface in a low polyester strength matrix. Hence, the impact strength of the composites was found to be increase with increase fiber particle quantity. This was attributable to fact that the long fibrous particles can absorb force without breaking, meaning that increased impact resistance was due to the formation of an indirect fracture path that retards crack propagation. Furthermore, increased polymer-fiber interaction increases interfacial tension and reduces chances of crack initiation at the interface.

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Morphological and Compatibility Analyses

The FESEM micrographs of the cross-sections of the surfaces of the nanocomposites at different levels of nanofiller loading are shown in Figs. 5a through 5f. Differences in the surface morphology of the biocomposites and the dispersion of the OPA nanoparticles in the epoxy polymer matrix were observed. Figure 5a shows surface fracture as a result of low surface interaction between the OPA nanoparticles and the epoxy matrix, which gave rise to low mechanical properties. This was attributable to the pattern of cracks, which resulted in a low resistance against crack propagation during tensile or impact fracturing and thus lowered the mechanical strength. Figures 5b through 5f show the FESEM micrographs of the fractured surfaces at 1%, 2%, 3%, 4%, and 5% nanoparticle-loaded nanocomposites, respectively. These FESEM images showed dissimilar patterns of cracks for all of the prepared nanocomposites.



Fig. 5. FESEM micrograph of OPA composites: (a) Neat + microsized OPA filled epoxy, (b) 1% OPA nanofiller loading, (c) 2% OPA nanofiller loading, (d) 3% OPA nanofiller loading, (e) 4% OPA nanofiller loading, and (f) 5% OPA nanofiller loading

From the images, it was deduced that the roughness and irregularly fractured surfaces of the nanocomposites showed that they were relatively less brittle in nature. Moreover, the SEM images of 1%, 2%, 3%, and 4% OPA reinforced epoxy composites displayed a good dispersion of OPA nanoparticles in the epoxy matrix when compared to the 5% OPA reinforced epoxy composite, as shown in Fig. 5f. From Fig. 5f, the agglomeration of OPA nanoparticles in the epoxy matrix is clearly apparent.

Thermal Properties

The thermal stability of the OPA nanocomposites was studied through monitoring of the thermal decomposition pattern using TGA with the decomposing curves shown in Fig. 6. The heating temperatures for the TGA ranged from 0 to 800 °C.



Fig. 6. TGA of neat epoxy resin and all epoxy nanocomposites with different levels of OPA nanofiller loading

Two stages of mass loss steps were exhibited by the TGA graph, which included the early stage mass loss ascribed to the gradual loss of moisture or water at approximately 100 °C. This value indicated the temperature at which 5% of the initial mass loss was observed. A second stage of mass loss occurred from approximately 320 to 450 °C, which was attributed to the decomposition of the polymeric material (Han *et al.* 2018). The TGA results gave the effect of the level of filler loading on the thermal stability of the nanocomposites (Fig. 6). Table 1 results show that increasing the OPA filler content of the nanocomposites had a considerable effect on the thermal stability. The results showed that the maximum thermal stability of the composites was 435 °C, with the composite at 5% nanofiller being the most stable (Table 1).

OPA Nanofiller Loading (%)	Degradation Temperature (°C)		DTG Peak Temperature (°C)	Char
	T _i	T _f	T _{max}	Residue (%)
0%/Epoxy	322	385	352	8.75
1%	351	402	381	9.67
2%	353	419	382	10.21
3%	354	428	383	11.88
4%	357	430	384	12.13
5%	358	435	385	13.25

Table 1. Thermal Properties of Macro/Nanostructured OPA-filled Epoxy

 Composites

This enhancement in the thermal stability of all composites was attributed to the presence of silica in the OPA nanofillers that has resistance to high temperature (Abdul Khalil *et al.* 2013). Moreover, OPA nanofiller itself contains many inorganic elements that can withstand high temperatures (Abdul Khalil *et al.* 2013). The char residue was obtained at temperatures above 500 °C, and it ranged between 8.75% and 13.2%. The char residue was obtained at temperatures above 500 °C and it ranged from 8.75% to 17.83%. This was attributable to the silica component of the oil palm ash (Suriapparao and Vinu 2015).

CONCLUSIONS

- 1. The OPA nanoparticles were successfully prepared via the ball-milling technique.
- 2. The TEM images revealed that OPA nanoparticles had circular morphology and a particle size in the range of 20 to 25 nm. From the XRD analysis, the calculated particle size of the OPA nanofiller was 26 nm, which was in agreement with the particle size obtained from the TEM images.
- 3. Tensile strength and tensile modulus of the epoxy composites were substantially improved to 64, 67, 70, and 75 MPa and 1.01, 1.05, 1.16, and 1.18 MPa at OPA nanofiller loading of 1%, 2%, 3%, and 4%, respectively.
- 4. The impact strength of the nanocomposites increased from $2.7015 \pm 0.13 \text{ kJ/m}^2$ to $3.98 \pm 0.17 \text{ kJ/m}^2$ at 3% OPA nanofiller loading.
- 5. The flexural strength and the flexural modulus of the OPA-filled epoxy nanocomposites were 135 MPa and 7.04 GPa, respectively, at 4% OPA nanofiller loading.
- 6. The epoxy composite reinforced with 5% OPA showed high thermal stability than other fabricated composites. The thermal stability was enhanced from 385 to 435 °C with char content of 13.2%.

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