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INFLUENCE OF SURFACE MODIFICATION OF FIBRES ON THE PROPERTIES OF ENGINEERING THERMOPLASTIC ABS/KENAF FIBRE COMPOSITES

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ABSTRACT

This research is aimed at incorporating natural bast fibres, kenaf into engineering thermoplastic, Poly acrylonitrile-butadiene-styrene (ABS) at the processing temperature higher than 200°C without degradation. This was achieved by surface coating of the fibres with epoxy thermosetting resin before compounding with ABS in a twin screw extruder after analysing fibres thermogravimetrically and later determined the dynamic modulus and X-ray diffraction. It was observed that epoxy resin was able to protect the kenaf fibres from degrading even at higher temperature producing much less residue, increased the loss modulus and reduced the storage modulus and damping value, increased the composite crystallinity and was able to bind fibres together which in turns increased the interfacial bonding between fibres and thermoplastic. The belief that temperatures higher than 200 °C would be critical for natural fibres as degradation might start affecting the mechanical properties of the fibres above the aforementioned temperature do not hold again because this research has proved otherwise as it was established that surface coating of cellulosic fibres with epoxy resin has not only improved their ability to withstand much higher temperatures than expected but also improved the performance of the composites.

Key Words: Thermoplastic ABS, Epoxy resin, Kenaf fibres, Thermogravimentric analysis and Crystallinity.

INTRODUCTION

Epoxy is a copolymer; that is, it is formed from two different chemicals which can be referred to as the "resin" or "compound" and the "hardener" or "activator". The resin consists of monomers or short chain polymers with an epoxide group at either end. Fibre surface treatment before introducing the matrix material can modify the interphase region and alter the adhesion between the fibre and the matrix. It is generally believed that thermoset materials are generally stronger than thermoplastic materials, for the composites based on thermoset matrices such as epoxy, polyester, phenol formaldehyde, etc fall into this category. But with alkali treatment of natural fibres which is one of the most common treatments of the natural fibres (Ray, *et al*. 2001; Mishra, *et al*., 2001) improves the interfacial adhesion between the fibre and matrix (Ray, *et al*., 2002; Mwaikambo and Ansell, 2003). The adsorption of the epoxy resin on the jute fibre surface increases, which is a prerequisite condition for creating the interphases (George, *et al*., 1999). Meanwhile, in the materials group of natural fibre-reinforced thermosetting plastics, especially in the group of natural fibre-reinforced epoxides, there isn't much knowledge about the influence of suitable coupling agents on the parameters of composites in comparison with the natural fibre and reinforced thermoplastics. Some of the most extensive examinations are about the influence of silanes (γ-aminopropyltriethoxy-silane) on natural-fibre–epoxy composites as carried out by Bisanda and Ansell (1992) and Gassan, *et al*., (2001). The influence of chemical surface modification of kenaf fibre using hydrogen peroxide on the mechanical properties of biodegradable kenaf fibre/Poly(Lactic Acid) composites was studied by Nur Inani and co-researchers (2014) and found that improvement of interfacial adhesion between fibre surface and polymer matrix as well as the mechanical performance of the treated fibre over the untreated was reported. Moreover, fundamental study on the effect of alkaline treatment on natural fibres structures and behaviours was investigated by Bakri, *et al*., (2016), and also observed the improvement of interfacial adhesion. The same observations were the outcome of researchers, Tayfun, *et al*., (2016), in "influence of surface modifications of flax fibre on mechanical and flow properties of thermoplastic polyurethane based eco-composites". Improved fibre strength was the finding of Salim, *et al*., (2019) when kenaf fibres was treated with 6 % NaOH in 'Effect of fibre surface treatment on interfacial and mechanical properties of non- woven kenaf fibre reinforced acrylic based polyester composites'. Influence of layering pattern of modified kenaf fibre on thermomechanical properties of epoxy composites was studied by Mohammed, *et al*., (2019) where a chemical modification of kenaf fibre using propionic anhydride to enhance the compatibility with the epoxy matrix and they found that the thermal stability was much improved.

Engineering plastics are high-performance polymers used in engineering applications because of their outstanding balance of properties. On the other hand, kenaf, cellulosic fibre can be used as an alternative raw material to wood, in pulp and paper industries (Ashori and Raverty, 2007), and fibre reinforced plastics (FRP) is to replace synthetic fibres such as glass (Abdullah, *et al*., 2011); and also used as non-woven mats in the automotive industries, textiles and fibreboard (Ibraheem, *et al*., 2011). Previous studies have investigated the development of composite materials using natural fibres such as flax, bamboo, pineapple, silk, sisal, jute, kenaf, and ramie. Their studies examined moulding conditions, mechanical properties, and interfacial bonding (Ochi, 2008).

Ci *et al*. (2008) reported that structural polymer composites are engineered structures from strong load carrying reinforcements (e.g., carbon or glass fibres) and matrix polymer materials having considerably lower strength and density than the reinforcing phase. The combination of fibres and matrices often give rise to high strength materials with minimum weight. The main thermal properties of vegetable fibres for composite materials are thermal expansion and thermal degradation. In spite of other clear advantages, the thermal properties of vegetable fibres represent a clear drawback in comparison with synthetic fibres. Regarding thermal expansion, only few data are available in the scientific literature with values of the corresponding coefficient ranging in the order of 3x10^{-5}, while glass fibres are in the order of 5x10^{-6}. On the other hand, thermogravimetric and differential scanning calorimetric analysis demonstrate that noticeable mass loss in natural fibres start at about 200-220 °C; above this temperature, irreversible degradation of the fibres occurs (Wielage, *et al*., 1999). The relatively low degradation temperatures of vegetable reinforcements, in comparison with the process temperatures of polymeric matrices, represents the most important limit for their use in plastics with the conventional processing equipment (extruder, injection moulding, etc). However, Anuar and Zuraida (2011), studied the thermal properties of Injection moulded poly lactic acid / kenaf fibre (PLA/KF) biocomposite and found that the biocomposite remained amorphous with the fibre content and from the differential scanning calorimetry, the glass crystallization temperature and cold-crystallization enthalpy increased with fibre contents, while cold crystallization and melted temperature slightly decreased with fibre content. Crystallization kinetic studies were carried out by Chen and Porter (1994) on composites made of polyethylene and kenaf fibre, which is extracted from the bast of the plant *Hibiscus cannabinus*. The effect of surface treated kenaf fibres in this research is aimed, whether used independently or as composites with engineering thermoplastic, ABS.

MATERIALS AND METHODS

Materials

Poly acrylonitrile butadiene styrene (The engineering thermoplastic used as the matrix in this work was Polytac ABS which was an extrusion grade resin purchased from Chi Mei Company) pellets, 3 mm Kenaf fibres (KF), sodium hydroxide, acetic acid, Epoxy, curing agent (4,4-diaminodiphenyl sulfone, DDS) and chloroform. All materials were supplied by Science and Engineering Research Centre (SERC) of Engineering Campus, Universiti Sains Malaysia, Nibong Tebal, Malaysia.

Surface treatment with alkali and thermosetting resin

Kenaf fibre cut to 3 mm length produced from Kenaf Fibre Industries Sdn. Bhd. (KFI) Malaysia was supplied to SERC. Initially, the fibres were oven dried in the lab at 37 °C for 2 days prior to being immersed in 6 % NaOH for 3 hrs. They were then washed and neutralized with 100 % acetic acid and finally washed thoroughly with distilled water. The NaOH treated fibres were oven dried at 80 ^{o}C for 16hrs, and then coated with 30 % epoxy resin of very low viscosity that had been thinned with chloroform. The epoxy coated fibres were first oven dried between 30 – 40 ^{o}C for 16 hrs and then at 80 ^{o}C for 8 hrs to dry finally.

Thermogravimetric Analysis (TGA)

TGA of fibres was conducted in the Perkin-Elmer Pyris 6 TGA Analyzer (USA) from room temperature to 600 ^{o}C at a heating rate of 10 ^{o}C/ min under nitrogen atmosphere using 10 – 15 mg samples weight.

Compounding and Moulding of Composites

The mixing of curative (DDS) with epoxy coated kenaf fibres (EKF) was carried out prior to mixing with engineering thermoplastic, ABS in a bowl container before pouring into the hopper feeder of the twin screw extruder for compounding. The mixed materials were extruded in a twin screw extruder (PSM 30, SINO-ALLOY Machinery Inc., HIS-ChiH, TAIPEI HSIEN, Taiwan) with temperatures profile of 175 – 230 °C at 70 rpm, extrudate was immediately quenched in water (20 ^{o}C), pelletized and moulded at 210 ^{o}C temperature using 65 kg/cm^2 pressure after which it was dried for 3 hrs at 60 ^{o}C.

Dynamic Mechanical Analysis (DMA)

Dual cantilever dynamic properties of impact testing specimens were measured at a fixed frequency in the temperature range of 30–220 ^{o}C on DMA861^{e} (METTLER TOLEDO, Switzerland). The frequency used was 1 Hz at the heating rate of 5 ^{o}C/ min and 1N force amplitude with a displacement of 10 μm. The storage modulus (E′), loss modulus (E″) and tan δ of the samples were collected during the run.

X-Ray Diffraction (XRD)

XRD analysis of fibres and that of their composites were carried out with PANalytical X'pert Pro Mrd PW2040 XRD diffractometer (Netherlands) in a scan range from 5^{o} – 40^{o} and 2.0^{o}/min scanning rate. The X-Ray source was Cu-Kα radiation with a wavelength (λ) of 0.154 nm. The specimens of 15 x 8 x 1 mm prepared by cutting with a band saw were used in this test. Crystallinity, X_c (%) was calculated using the equation:

$$X_c = \frac{A_c}{A_a + A_c} x100$$

Where A_α = area of amorphous, A_c = area of crystalline.

Scanning Electron Microscopy (SEM)

Fractured surfaces from the un-notched Izod impact test samples of composites were observed under a field-emission scanning microscope (FESEM) (Zeiss LEO Supra 35VP, Germany). Prior to observation, the samples were sputter-coated with a thin layer of gold to avoid electrical charging during examination.

RESULTS AND DISCUSSION

Thermogravimetric Analysis of surface treated kenaf fibres

Surface treatment with NaOH was used to remove the waxes, hemi-cellulose and part of the lignin in the fibres, and the subsequent coating with epoxy helps in protecting the fibres. The effect of these treatments was studied with TGA and result presented in Fig. 1.

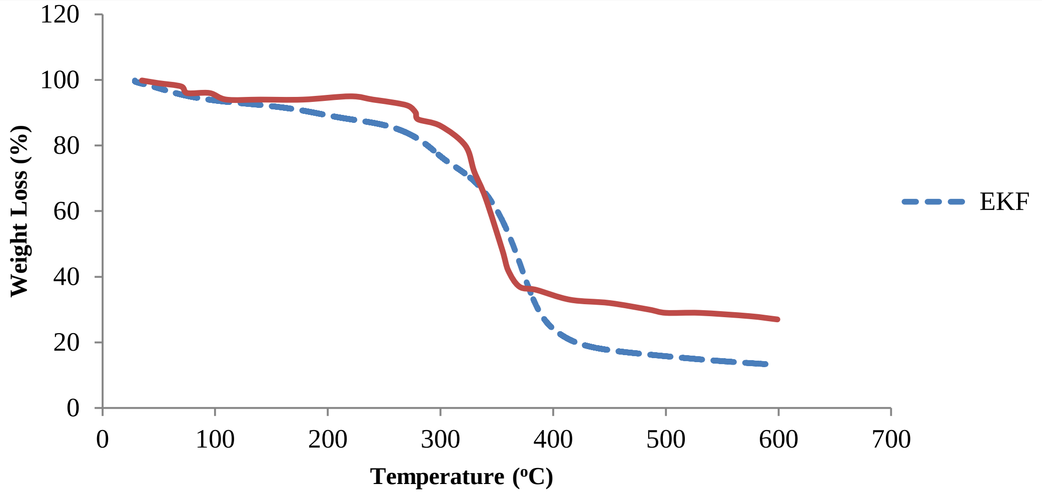


Fig. 1: The Thermogravimetric Analysis (TGA) of KF and EKF

Fig. 1 represents the TGA of epoxy coated kenaf fibres (EKF) and that of uncoated kenaf fibres (KF). KF has the tendency of withstanding thermal degradation above 200 °C as in contrasts to the observations of some researchers (19, 20) that cellulosic fibres degrade at temperature above 200 °C. KF showed a deviation from Bogoeva-Gaceva *et al*., (2007), and Rahman, *et al*., (2011), as the onset temperature degradation starts at about 280 °C, dropped drastically to 355 °C where the final degradation took place having as much residue as 32 %. This might be attributed to the treatment of KF with NaOH which removes the hemicelluloses and part of the lignin which are directly responsible for the thermal degradation during heating. However, loss of moisture was noted at temperature below 100 °C (between 75 and 95 °C) with a sharp fall at it onset. At 272 °C, KF had undergone thermal changes which might be attributed to degradation of low carbon sugar. On the hand, the EKF produced a smoother outline of TGA different from that of KF has no indication of moisture lost prompting the onset degradation to start earlier at 270 °C, degrading gradually and then finally at much more higher temperature, 399 °C far more than that of KF. This phenomenon might be attributed to encapsulation of epoxy resin that might be undergoing the process of drying on the fibre to improve the interfacial bonding. Instead of moisture loss, the moisture was entrapped by the epoxy and this gave room for earlier onset degradation but prolongs the final degradation. This is in line with the findings of Thitithanasarn, *et al*., (2011). The results are summarized in Table 1.

Table 1: TG analysis of Kenaf and Epoxy coated Kenaf Fibres

Samples	Onset Temperature (°C)	Peak Temperature (°C)	Final Degradation Temperature (°C)	Residue (%)
KF	280.79	355	355	32.35
EKF	270	380	399	13

KF= Kenaf fibre; EKF= Epoxy coated kenaf fibre

XRD of Epoxy coated (EKF) and Uncoated Kenaf (KF) Fibres

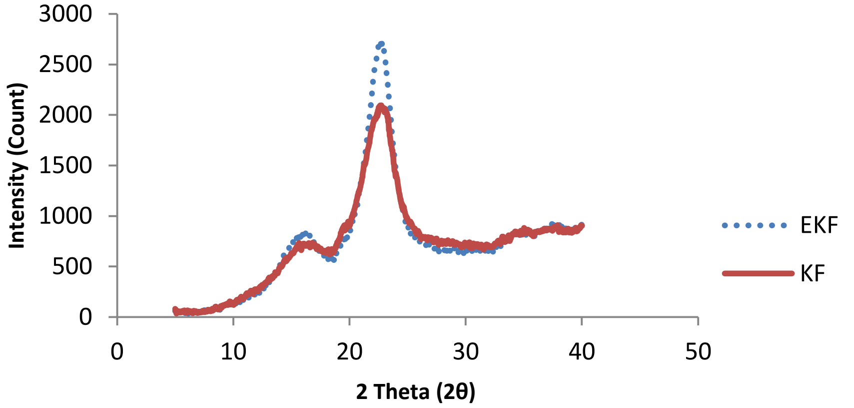


Fig. 2: XRD Partern of KF and EKF

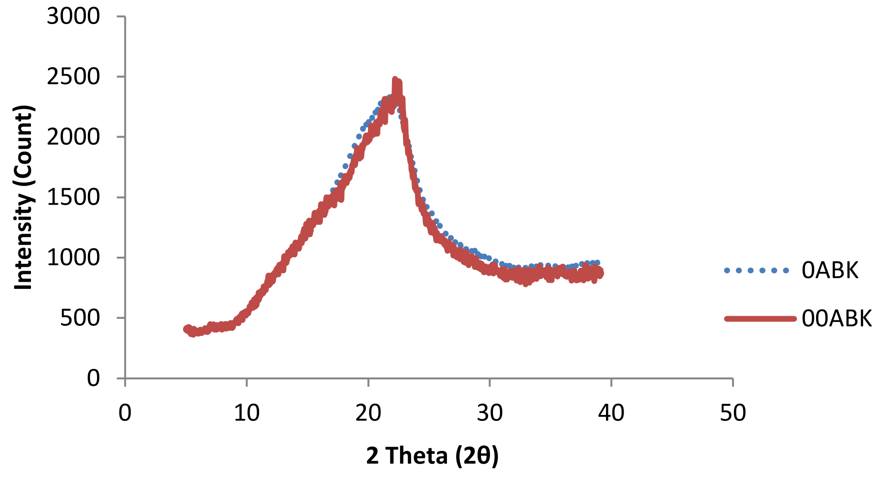


Fig. 3: XRD Pattern of 00ABK and 0ABK composites

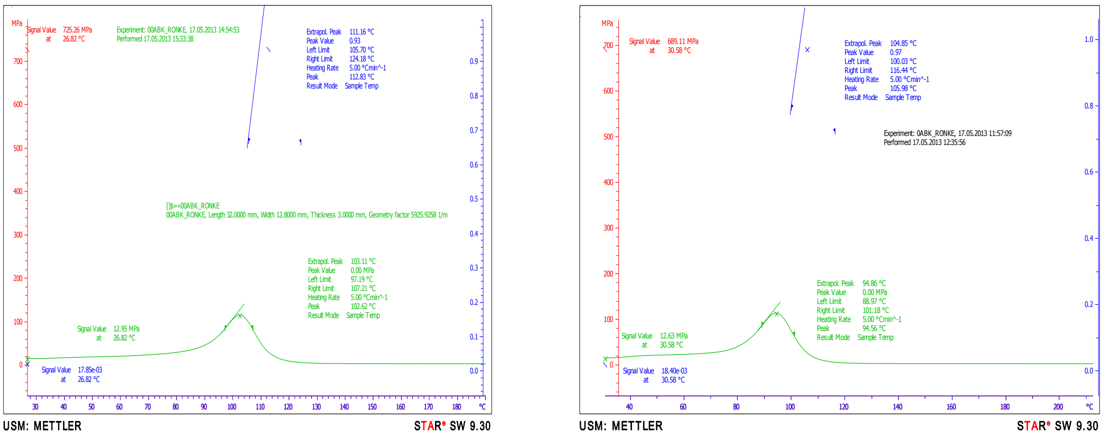
Table 2: Characteristic XRD peaks of Kenaf and Epoxy coated Kenaf Fibres

Samples	Peak (2θ)	Corresponding Intensity (Count)	X_c (%)
KF	17.2 and 23.3	700 and 2050	53.68
EKF	18.1 and 23	850 and 2680	57.45
00ABK	21	2500	50.74
0ABK	22.1	2350	55.56

00ABK= composite of ABS/KF (no epoxy no cure);
0ABK= composite of ABS/KF (with epoxy but no cure)

X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing. Fig. 2 shows the XRD pattern of KF and that of EKF while Table 2 is the summary of pattern for both the fibres and their composites. The pattern indicated two peaks for both fibres with that of EKF at much higher intensities which translated to its higher crystallinity. When ABS was compounded with the KF (uncoated kenaf fibres) to get the 00ABK (uncoated fibre/ABS composite), it can be seen that it does not only caused the disappearance of the first peak, but also reduced the peak by 2.3^o, increased the intensity to 2500 from 2050, which now decreased the crystallinity to 50.74 from 53.68% as shown in Table 2. Moreover, with the compounding of ABS with EKF (epoxy coated kenaf fibres), 0ABK (coated fibre/ABS composite), this follows the same pattern like 00ABK; there was a disappearance of the first peak, reduction in 2θ degree, decrease in intensity and crystallinity, as seen in Fig. 3 as summarized Table 2. The results of Figure 3 can be interpreted as little or insignificant effect of epoxy treated fibres when compounded with ABS due to the insignificant difference in their intensities, 00ABK and 0ABK.

DMA of Epoxy coated (0AKF) and Uncoated Kenaf (00AKF) Composites



(a) (b)

Fig. 4: The DMA of composites of (a) uncoated (00ABK) and (b) coated (0ABK) kenaf fibres showing the loss and storage moduli plus the tan δ.

Table 3:Temperature data for E″ and tan δ peaks of composites' DMA giving the storage and loss moduli

Samples	$(E'')_{max}$ (MPa) @60°C	$(E')_{max}$ (MPa) @60°C	$(E'')_{max}$ (°C)	$(\tan\delta)_{max}$ (°C)	Peak value of tan δ	ΔT(°C) = tan δ - E″
00ABK	20	680	102.62	112.83	0.93	10.21
0ABK	22	660	94.56	105.98	0.97	11.42

Dynamic mechanical thermal analysis is a sensitive technique that characterizes the mechanical response of a material by monitoring the dynamic property changes over a range of temperatures at a fixed frequency. DMA measures stiffness and damping. These are reported as modulus and tan delta. Application of a sinusoidal force enables the expression of the modulus as an in-phase component, the storage modulus, and an out of phase component, the loss modulus. Dynamic mechanical methods employ the techniques of monitoring the viscoelastic responses of materials when subjected to small oscillating strain. The storage modulus, either E′ or G′, is the measure of the sample's elastic behaviour. The ratio of the loss to the storage is the tan delta and is often called damping. It is a measure of the energy dissipation of a material.

Storage modulus

It is believed that the storage modulus (E′) provides valuable insight into the stiffness of materials as a function of temperature (Mandal and Alam, 2012). From Fig. 4, it can be observed that the storage modulus decreased with temperature for samples up to the minimum expected. This is in line with the findings of Akay, (1993), Mandal and Alam, (2012) and Han, *et al*., (2012); thus, indicating that the measurement of the maximum energy stored in the material decreased during a cycle of oscillation. Similarly, the stiffness behaviour and load bearing capability decreased with temperature increase. These curves are useful in assessing the molecular basis of the mechanical properties. Table 3 indicated that the storage modulus of KF composite is higher than that of EKF at 60 °C translating to KF being stiffer and less elastic than EKF.

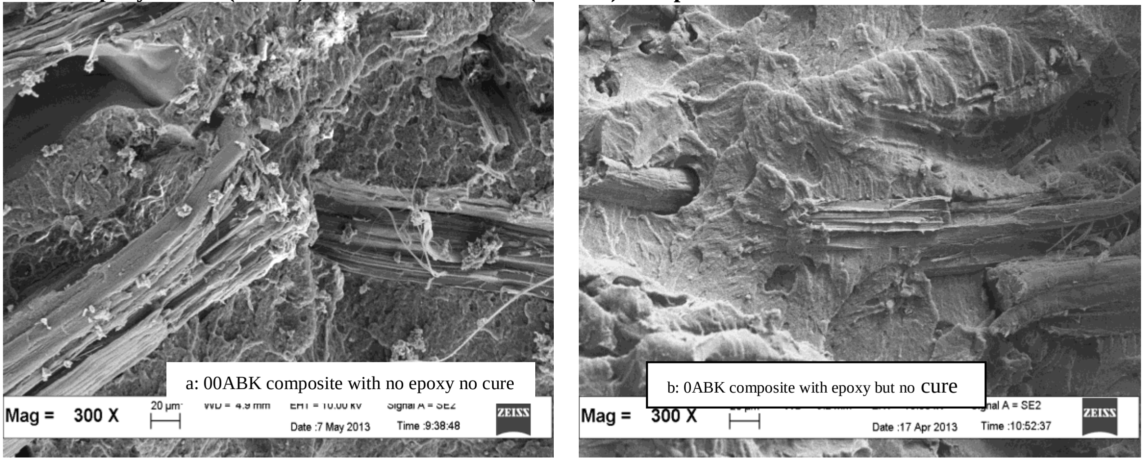
Loss modulus

The loss modulus (E″) is the amount of energy dissipated or lost as heat per cycle of sinusoidal deformation, when different systems are compared at the same strain amplitude. The amount of energy dissipated in KF is less at higher temperature than that of EKF at lower temperature, as presented in Table 3. The viscous response of the material might be the reason for this because loss modulus is most sensitive to the molecular motions.

Tan δ

Tan δ is a damping term that can be related to the impact resistance of a material. Since the damping peak occurs in the region of the glass transition where the material changes from a rigid to a more elastic state, it is associated with the movement of small groups and chains of molecules within the polymer structure. In a composite system, damping is affected by the incorporation of fibres which causes the KF composite to be more damped at lower peak than that of EKF composite indicating that EKF composite tends towards glass transition faster than that of KF.

SEM of Epoxy coated (0AKF) and Uncoated Kenaf (00AKF) Composites


a: 00ABK composite with no epoxy no cure

b: 0ABK composite with epoxy but no cure

Fig. 5: SEM micrographs of (a) 00ABK and (b) 0ABK

The SEM is used to study the dispersion, distribution, and the state of kenaf fibres in the polymer matrices. Figs 5a and 5b show the SEM micrographs of pure kenaf and epoxy treated kenaf fibres composites. Fig. 5a shows the 00ABK composite fibres without epoxy coating, and it can be clearly seen that there are holes, scattered, unwetted kenaf fibres, and significant fibres pull out; indicating a poor adhesion between the fibres and the matrix ABS. These observations are similar to that of Supri and Ismail, (2011), Han *et al*., (2012) and Nur Inani *et al.*, (2014). However, with the epoxy coated fibres, fewer holes, no scattering of fibres, good wetness of fibres and less pull out resulted, indicating good interactions of fibres and the matrix as depicted in Fig. 5b. The observation of nodular morphology confirms the evidence of formation of epoxy coating layer on the fibres, this was also observed by Jana and Prieto, (2002).

CONCLUSION

The properties of epoxy resin modified kenaf fibres and that of their composites with ABS thermoplastic were investigated and found that epoxy encapsulated fibres prevented the fibres from thermal degradation even at higher temperatures above 280 ^{o}C. This can be attributed to quality adhesion between coated fibres and matrix, that is, the interfacial bonding was improved as shown in the SEM where little or no pull out of fibres from the coated composite, and also increased in crystallinity and less stiffness of the composites resulted.

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