EFFECT OF LAYERING PATTERN ON THE DYNAMIC MECHANICAL PROPERTIES AND THERMAL DEGRADATION OF OIL PALM-JUTE FIBERS REINFORCED EPOXY HYBRID COMPOSITE

M. Jawaid, and H. P. S. Abdul Khalil*

Dynamic mechanical and thermal analysis of oil palm empty fruit bunches (EFB)/jute fiber reinforced epoxy hybrid composites were carried out. The effect of layering pattern on dynamic mechanical properties (storage modulus (*E'*), loss modulus (*E"*), and tan *δ*) was investigated as a function of temperature. The storage modulus (*E'*) was found to be decreased with temperature in all cases, and hybrid composites had almost the same values of *E'* at glass transition temperature (*Tg*). The tan *δ* peak height was minimum for jute composites and maximum for epoxy matrix. Layering pattern affected the dynamic mechanical properties of hybrid composites. Cole-Cole analysis was carried out to understand the phase behaviour of the composite samples. Thermogravimetric analysis (TGA) results indicated an increase in thermal stability of pure EFB composite with the incorporation of jute fibers. The overall results showed that hybridization with jute fibers enhanced the dynamic mechanical and thermal properties.

Keywords: *Hybrid composites; Oil palm fibers; Jute fibers; Epoxy resin; Dynamic mechanical analysis; Thermal analysis*

Contact information: School of Industrial Technology, Universiti Sains Malaysia, 11800 Penang, Malaysia
*Email: *akhalilhps@gmail.com*

INTRODUCTION

Natural fibers possess high specific strength and modulus, low density, low cost, and easy recyclability that make them an attractive alternative to glass fibers. The scope for using jute fiber in place of traditional glass fibers has been gaining attention because of their specific modulus, lower cost, renewable nature, market appeal, and nonabrasive nature. Natural fiber composites made up of jute fiber are particularly attractive in automotive applications because of reduced vehicle cost and weight (Alves et al. 2010). Previous studies have shown that oil palm empty fruit bunches (EFB) as well as jute fibers can be used as effective reinforcement in epoxy matrix (Gassan and Bledzki 1999; Gassan and Gutowski 2000; Mir et al. 2010; Zuhri et al. 2010)

In the case of polymer composites, hybrid composites are systems in which one kind of reinforcing material is incorporated in a mixture of different matrices (blends) (Karger-Kocsis 2000), or two or more reinforcing and filling materials are present in a single matrix (Fu et al. 2002; Pukánszky 1995) or both approaches are combined. The incorporation of two or more natural fibers into a single matrix has led to development of hybrid composites. The behaviours of hybrid composites can be regarded as a weighted sum of the individual components in which there is a more favourable balance among the inherent advantages and disadvantages. While using hybrid composites that contain two or more types of fiber, the advantages of one type of fiber could compensate for aspects that are lacking in the other. As a consequence, a balance in cost and performance could be achieved through proper material design (Thwe and Liao 2003). The prospective benefits of reinforcing different types of natural fiber in single matrix has also been studied by other researchers (Idicula et al. 2005b; Uma Devi et al. 2010)

Dynamic Mechanical Analysis (DMA) is one of the most powerful tools to study the behaviour of polymer composite materials, and it allows for a quick and easy measurement of material properties (Swaminathan and Shivakumar 2009). The dynamic mechanical properties of polymer composites depend on the fiber content, filler, compatibilizer, fiber orientation, and mode of testing (Jacob et al. 2006). It is clear from previous study that DMA gives more information about composite material compared to other tests of composites. Researchers have investigated the dynamic mechanical properties of randomly oriented intimately mixed short banana/sisal hybrid fiber reinforced polyester composites (Idicula et al. 2005a). The effect of layering pattern on storage modulus (E'), damping behaviour (tan δ), and loss modulus (E'') of bilayer (banana/sisal), trilayer (banana/sisal/banana and sisal/banana/sisal), and intimate mix composites was studied by keeping the relative volume fraction of banana and sisal at 1:1 and the total fiber loading to a 0.40 volume fraction as a function of temperature and frequency (Idicula et al. 2005b). The effects of various solutions on DMA properties of pultruded kenaf fiber reinforced unsaturated polyester composites was studied, and it was observed that the largest effects followed immersion in pH 8.9 solution (Mazuki et al. 2011). Thermal properties of the natural fiber composites were improved by hybridization with glass fibers (Nayak and Mohanty 2010). Researchers investigating thermal properties of jute/bagasse hybrid composites observed that thermal stability of hybrid composites increased by increasing residual char left at 600^{o}C (Saw and Datta 2009).

In the present work, oil palm EFB fiber is hybridized with small quantities (20%) of stronger jute fiber and reinforced epoxy matrix to develop cost-effective high-performance hybrid composites. Oil palm EFB/jute fiber reinforced epoxy hybrid composites, having a weight ratio of oil palm EFB and jute of 4:1 were prepared at a total fiber loading of 40% by weight. Dynamic mechanical analysis and thermal properties of oil palm EFB/jute fiber reinforced epoxy hybrid composite were studied. The effect of layering patterns such as trilayer composite (EFB/jute/EFB and jute/EFB/jute) were investigated and compared to control samples, pure jute composite, pure EFB composite, and epoxy composite with respect to storage modulus, loss modulus, and damping factor. Thermal properties were studied by TGA to investigate the thermal stability of the hybrid composites.

EXPERIMENTAL

Materials

Oil palm EFB fiber mat was supplied by Ecofiber Technology Sdn. Bhd., Malaysia. Jute fiber mat was procured from Indarsen Shamlal Pvt. Ltd. (Jute House Since

1948), Kolkata, India. The physical and mechanical properties of oil palm EFB and jute fiber are given in Table 1 (Ahmed 2003; Bledzki and Gassan 1999; Fu et al. 2001; Munikenche Gowda et al. 1999; Shinoj et al. 2011; Sreekala et al. 2002). The epoxy A331 (diglycidyl ether of Bisphenol A) and epoxy hardener A062 (reactive polyamide (RP) were used in this study. Both the epoxy resin and commercial curing agent were obtained from Zarm Scientific & Supplies Sdn. Bhd., Malaysia. Benzyl alcohol, used as diluent, and silicone oil, used as releasing agent, were supplied by Aldrich Company.

Table 1. Physical and Mechanical Properties of Oil Palm EFB and Jute Fiber (Sreekala et al. 2002; Fu et al. 2001; Shinoj et al. 2011; Bledzki and Gassan 1999; Ahmed et al. 2003; Munikenche Gowda et al.1999)

Properties	Oil Palm EFB fiber	Jute fiber
Density(g/cm^3)	0.7-1.55	1.3
Tensile Strength (MPa)	50-400	393-773
Young's modulus(GPa)	1-9	26.5
Elongation at break (%)	8-18	1.5-1.8
Cellulose content (%)	49.6	58-63
Hemicellulose content (%)	18%	12%
Lignin content (%)	21.2	12-14
Diameter(μm)	150-500	20-200
Microfibrillar angle(°)	46	8.1
Lumen width (μm)	6.90	3.40

Preparation of Composites

The chopped fiber mat of oil palm empty fruit bunches (EFB) fiber and jute fiber were used in preparation of hybrid composites. In order to make the epoxy matrix, epoxy resin and polyamide were mixed in the ratio 100:60 parts by weight, respectively. Later on, 10 wt% benzyl alcohol was added with the epoxy resin, and the mixture was stirred by mechanical stirrer for 15 minutes. The mould cavity was coated with a thin layer of silicone oil solution, which acts as a good releasing agent. EFB fiber mat and jute fiber mat were stacked together with the layer of jute fiber mat sandwiched between the layer of EFB fiber mat and vice versa in mould. It was then impregnated with epoxy matrix in the mould with dimensions of 304 mm x 203 mm. Hybrid composites with different sequence of fiber mat arrangement were prepared by varying the weight ratio of the two fibers. All composites have same thickness. A neat epoxy matrix (unfilled) sample was prepared, and epoxy matrix samples with oil palm EFB fiber and jute fiber mat were also prepared.

Dynamic Mechanical Analysis (DMA)

DMA was used to determine mechanical properties and glass transition temperatures of pure composites, epoxy, and hybrid composites. The curves of the storage and loss modulus and the tangent as functions of temperature were obtained. The temperature of the maximum in the loss tangent peak was taken as the glass transition temperature. A dynamic mechanical analyzer (Mettler Toledo, Model 861) was used for the evaluation of storage modulus, loss modulus, and mechanical damping (tan δ). Three point bending mode was used. The heating rate used was 5^{o}C/min and frequency was 1 Hz under amplitude control. Liquid nitrogen was used as cooling agent, and the temperature range was from -150^{o}C to 500^{o}C. The amplitude was set within the range 7 to 10 mm, depending on the thickness of the samples. The samples had a thickness of 4 to 5 mm, width of 9 to 10 mm, and length of 50 to 60 mm.

Thermogravimetric Analysis (TGA)

A thermal gravimetric analyzer (TGA), model 2050, (TA Instruments, New Castle, DE) was used to investigate the thermal stability of the composites. The samples (about 3 mg) were heated from 30 to 900^{o}C under nitrogen environment at a heating rate of 20^{o}C/min.

RESULTS AND DISCUSSION

Dynamic Mechanical Analysis (DMA)

Effect of layering pattern on storage modulus (E')

Storage modulus is a measure of how stiff or flimsy a sample is. Figure 1 shows the effect of layering patterns on the DMA curves of storage modulus verses temperature of epoxy matrix, pure jute composite, pure EFB composite, and the hybrid composites at a frequency of 1 Hz. On investigating the variation of storage modulus with temperature of trilayer composite (EFB fiber as the skin and jute fiber as the core and vice versa) in Fig. 1, storage modulus was found to be decreased with temperature in all cases. At low temperature, the *E'* value of pure epoxy and composites were very close; this follows from the fact that at lower temperature fibers do not contribute much to imparting stiffness to the material. As temperature increases, the components become more mobile, lose their mobility, and lose their close packing arrangement. As a result, in the rubbery region there is no significant change in modulus. It is seen that pure epoxy shows a minimum and pure jute composite shows maximum storage modulus value. Neat resin (pure epoxy) comprising only the rubbery phase gives the material more flexible character, resulting in a low degree of stiffness of material and consequently low storage modulus (Joseph et al. 2010). In the case of the pure epoxy sample, there is a sharp fall in *E'* on passing through the glass transition temperature (T_g), due to the increased molecular mobility of the polymer chains above T_g. The drop in storage modulus in the glass transition region is much less for natural fiber reinforced composites than for the pure epoxy (Hameed et al. 2007). It is readily apparent that the addition of jute fiber increases the storage modulus of the epoxy matrix due to high stiff behaviour of the jute fiber. Besides, this may because the interfacial bonding between jute fiber and polymer matrix in composite is enhanced. In case of fiber reinforced polymer composite, the drop in modulus was dramatically reduced while passing through T_g compared to pure epoxy, which shows the greater reinforcing effect of oil palm EFB and jute fiber on the modulus above T_g than below it. These effects can be attributed to the combination of the hydrodynamic effects of fibers embedded in the viscoelastic medium and to the mechanical restrain introduced by fibers at high concentrations, which reduce the mobility and deformability of the matrix (Marcovich et al. 1998). The dynamic modulus curves of composites showed a higher *E'* value than the pure epoxy above T_g region in the rubbery plateau. Jute/EFB/jute represent a trilayer composite having jute as skin and EFB as core material and EFB/Jute/EFB represent trilayer hybrid composites having EFB as skin and jute as core material. EFB/jute/EFB and jute/EFB/jute composites had almost the same values of *E'* at T_g. But above T_g, the *E'* value was slightly greater in jute/EFB/jute. The effectiveness of fillers on the moduli of composites can be represented by a coefficient C given in the following equation (Chua 1986),

$$C = \frac{\left(\frac{E'_g}{E'_r}\right)comp}{\left(\frac{E'_g}{E'_r}\right)resin} \tag{1}$$

where E'_g is the storage modulus value in the glassy region, and E'_r is the storage modulus value in the rubbery region.

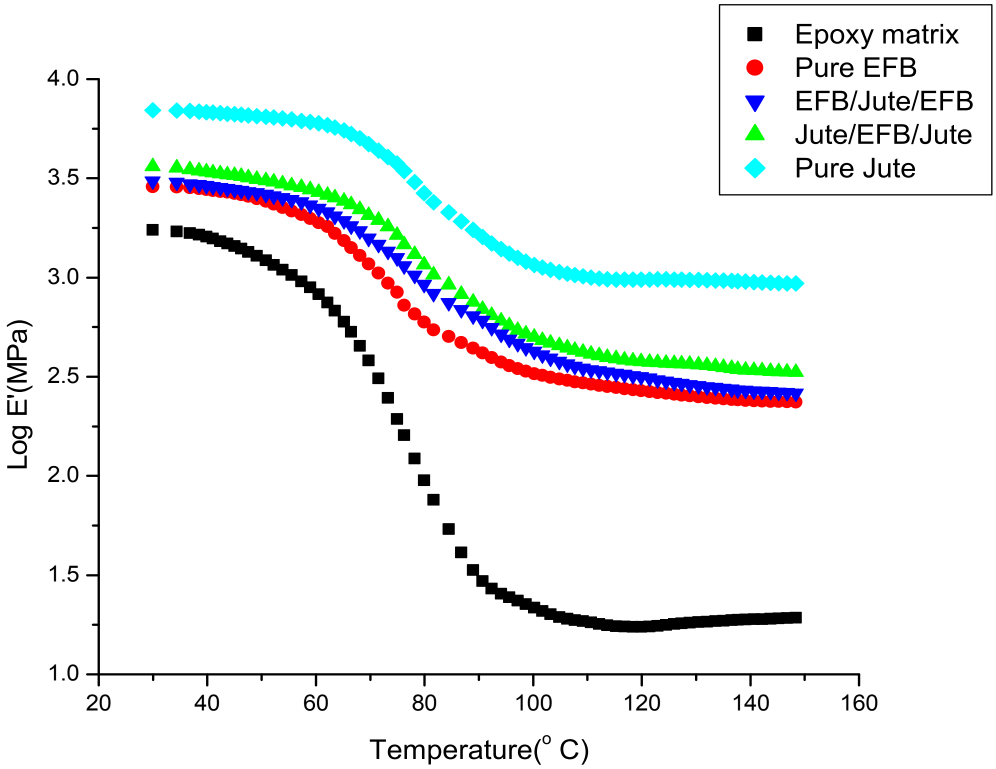

Fig. 1. Effect of layering pattern with temperature on storage modulus of epoxy, pure EFB, pure jute, and hybrid composites

The measured *E'* values at 66 and 104°C (for epoxy resin) were employed as E'_g and E'_r, respectively. The values of *C* obtained for different composites are given in Table 2. The value of *C* is maximum for pure EFB composite, minimum for pure jute composite, and the hybrid composites show values of *C* between pure EFB and pure jute composites.

A high value of *C* indicates less effectiveness of filler. The high stiffness of hybrid composites (EFB/jute/EFB and jute/EFB/jute) is in agreement with their tensile properties reported in our previous research (Jawaid et al. 2011). Above T_g, the *E'* value of jute/EFB/jute was higher than EFB/jute/EFB and pure EFB composites. Since oil palm EFB fiber has lower tensile properties compared to jute fiber, the stiffness of composite will decrease when oil palm EFB is used as the skin material.

Table 2. Peak Height, Coefficient(*C*), Tan$\delta_{\max}$ (*Tg*) and $E''_{\max}$ (T_g) of Epoxy, Pure EFB, Pure Jute, and Hybrid composites

Composite	Coefficient	Peak height of Tanδ curve	Tg from Tanδ_{max} (^{0}C)	T_g from $E''_{\max}$ (^{0}C)
Epoxy matrix	-	0.36	81.65	71.31
Pure EFB	0.553	0.29	85.37	73.82
Pure Jute	0.488	0.24	80.10	76.44
EFB/Jute/EFB	0.526	0.29	80.77	74.76
Jute/EFB/Jute	0.522	0.28	83.33	75.45

Effect of Layering Pattern on Loss Modulus (E")

Loss modulus (*E"*) is a measure of the energy dissipated as heat/cycle under deformation or is a measure of viscous response of materials. Figure 2 shows the loss modulus curve of neat epoxy as well as different composites at a frequency of 1 Hz. From Fig. 2 it is clear that reinforcement of fiber in epoxy matrix causes broadening of loss modulus peak, which can be attributed to the inhibition of relaxation process within composites (Woo et al. 1991). The E''_{max} (T_g) of different composites can be seen in Table 2. The highest T_g was observed for pure jute composite and the minimum for pure epoxy. The maximum heat dissipation occurred at the temperature where *E"* was maximum, indicating the T_g of the system (Sepe 1998). The loss modulus value in transition region was much higher for composites compared to pure epoxy, which may be due to the increase in internal friction that enhances the dissipation of energy (Hameed et al. 2007). Above T_g the *E"* value of jute/EFB/jute was higher than that of EFB/jute/EFB hybrid composites. The effect of reinforcing fibers was prominent above the T_g, and the highest T_g was observed for the pure jute composite, followed by jute/EFB/jute, EFB/jute/EFB, and pure EFB composites.

Effect of Layering Pattern on the Damping Factor (tan *δ*)

Trends of change in the damping factor of the composites and pure epoxy with temperature are shown in Fig. 3. In composites, damping is influenced by the incorporation of fibers. It was observed that as temperature increased, damping factor went through a maximum in a transition region and then decrease in a rubbery region. These phenomena are associated with the movement of small groups and molecules within the polymer structure, all of which was initially frozen (Joseph et al. 2010). Therefore, the higher the tan δ peak value, the greater is the degree of molecular mobility (Sepe 1998). The pure epoxy had the highest tan δ value, indicating a large degree of molecular mobility. Incorporation of fibers in epoxy resin substantially lowered the viscoelastic damping factor because fibers carry a greater extent of stress and allow only a small part of it to strain the interface.

The peak height values are given in Table 2. A lower tan δ peak was obtained for pure EFB, hybrid composites and pure jute composite. In EFB/jute/EFB, the peak height was slightly higher than jute/EFB/jute but closer to pure EFB. Thus, the incorporation of small amount of jute fiber to oil palm EFB/epoxy composite enhances the damping characteristics of the hybrid composites.

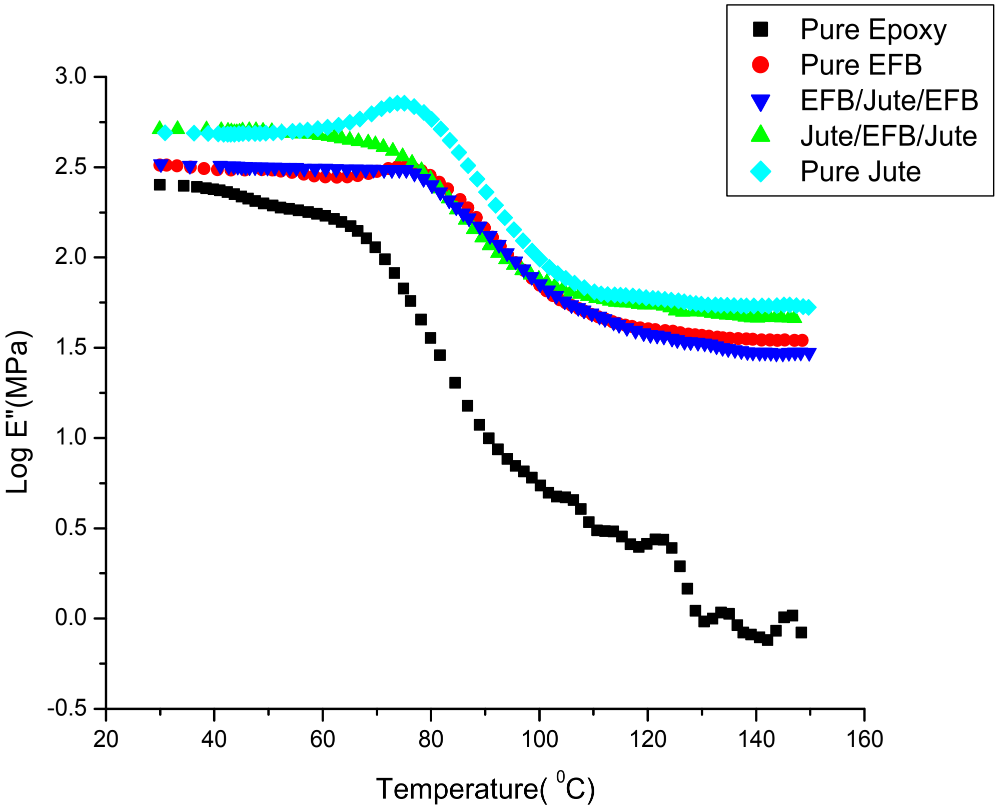

Fig. 2. Effect of layering pattern with temperature on loss modulus values of the hybrid composites at a frequency of 1 Hz

The damping effect of jute fiber/glass hybrid composites has been reported (Ghosh 1997); it was found that jute fiber contributes to the lowering in the damping factor of the composites. The lowering of peak height also indicates good interfacial adhesion. In case of composites, it is said that the damping peak height is related to the internal energy dissipation of the fiber/matrix interphase (Dong and Gauvin 1993). The positive shift in T_g value shows the effectiveness of the fiber as reinforcing agent. Hybrid composites showed higher T_g values compared to pure EFB and pure epoxy.

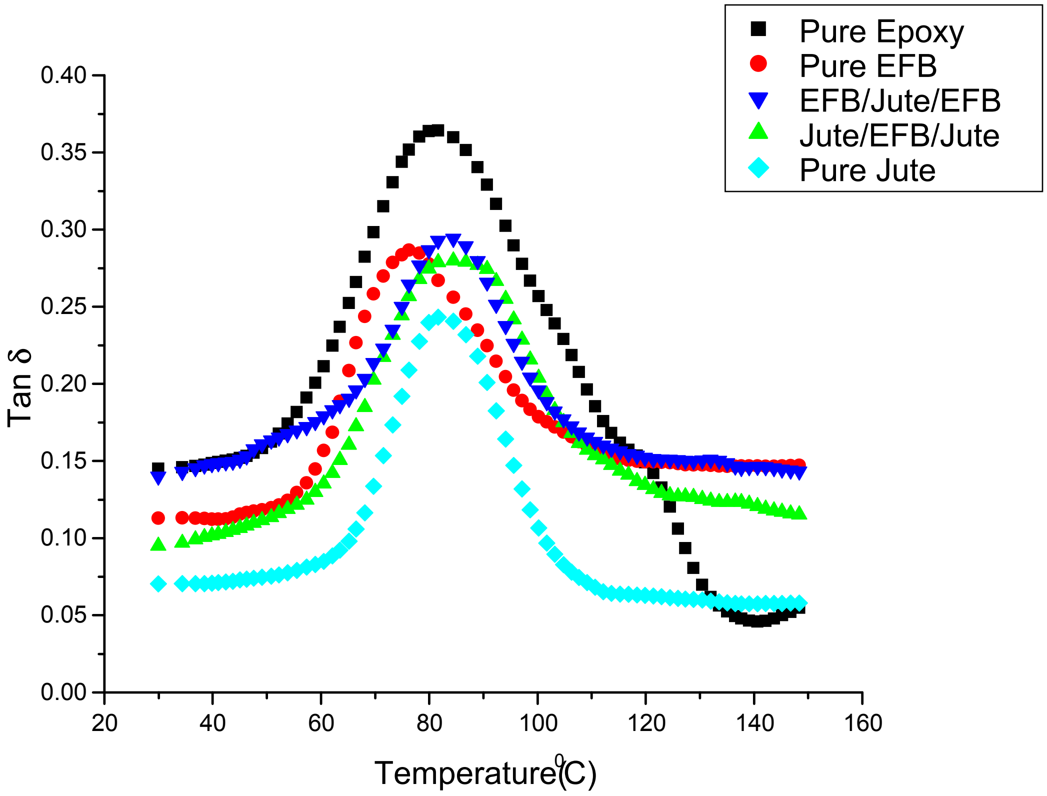

Fig. 3. Effect of layering pattern with temperature on tan delta value of the hybrid composites at a frequency of 1 Hz

Cole-Cole Plots

Linear viscoelastic mechanical properties of polymers or composites, especially in the vicinity of the glass transition, including the dynamic compliance and dynamic viscosity functions, can be demonstrated by a Cole-Cole plot. A Cole-Cole plot is a particular treatment of dielectric relaxation data obtained by plotting *E''* against *E'* at one frequency (Aklonis 1983). It is used to examine structural changes taking place in cross-linked polymers after addition of fibers to polymeric matrices (Harris et al. 1993). The dynamic mechanical properties of composites or polymeric material, when measured as a function of temperature and frequency, are represented on the Cole-Cole complex plane $E = f(E')$.

Figure 4 shows a Cole-Cole plot in which the loss modulus (*E''*) was plotted against storage modulus (*E')* for hybrid and pure composites. All the curves of the Cole-Cole diagram of different layering patterns shown in Fig. 4 have an imperfect semicircular shape. The nature of the Cole-Cole plot is reported to be indicative of the homogeneity of the system (Uma Devi et al. 2010). A semicircular curve indicates that a polymeric system is homogeneous (Ferry 1980). By contrast, an imperfect semicircular shape indicates that there is heterogeneity among pure EFB, pure jute, and hybrid composites. However, it was observed that the layering pattern of fibers, surface characteristics of fiber, and type of fibers affected the shape of the Cole-Cole plot and thereby influence dynamic mechanical properties.

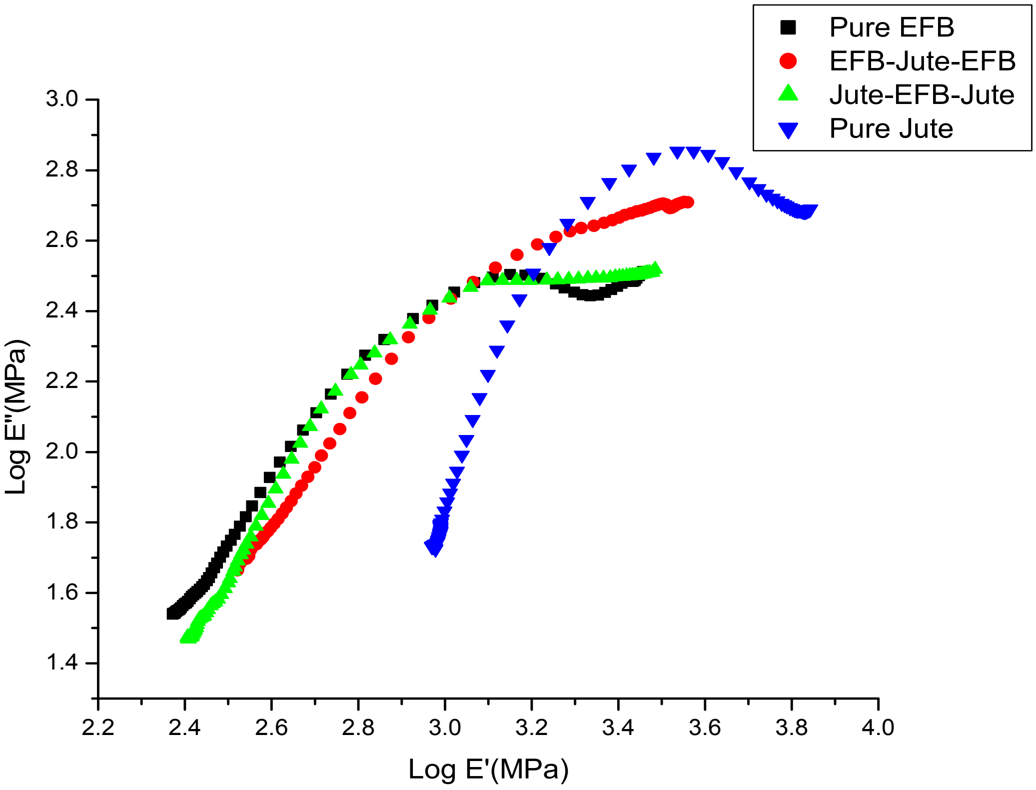

Fig. 4. Cole-Cole plots of the oil palm EFB/jute hybrid composite with different layering pattern

Thermogravimetric Analysis (TGA)

TGA curves of epoxy resin, pure jute, pure EFB, and hybrid composites are shown in Fig. 5. Thermal treatment of pure EFB composite and EFB based hybrid composite gave an initial weight loss below 100°C due to loss of moisture. The weight loss observed with epoxy polymer at this region might be due to the low molecular weight compounds. This initial weight loss was followed by complex secondary reactions and formation of volatile products, which arose from random chain scission and intermolecular transfer involving tertiary hydrogen abstractions from the polymer (Abdul Khalil et al. 2010; Grassie et al. 1986). From Fig. 5, it is obvious that no degradation occurred until 200°C. For the samples, the onset being above 200°C, the maximum rate of weight loss was observed in the range of 250 to 450°C. The amounts of residue left are tabulated in Table 3.

From the thermograms it is apparent that all the samples underwent a two-step degradation processes. The first stage thermal degradation process occurred in the temperature range of 256 to 343 ^{o}C and could be due to degradation of hemicellulose and α-cellulose (Saw and Datta 2009). The second stage degradation occurred in the temperature range of 418 to 499^{o}C and may be due to the complete decomposition of fiber and matrix. The significant thermal behaviour of the hybrid or pure composites were determined from the initial degradation temperature (T_{IDT}), which is taken as the temperature at which degradation started, and the residual weight (%), denoted as the char. The initial degradation temperature for hybrid composites shifted to a higher temperature, well over 268-271°C, compared with the pure EFB composite (260°C), indicating the higher thermal stabilities of the hybrid composites. Final degradation (T_{FDT}) occurred at 418°C for epoxy composite and between 441 and 443 °C for hybrid composite due to complex reactions. Degradation occuring in composites above 400°C may be due to decomposition of aromatic rings. Further heating leads to saturation of rings, rupture of lignin C-C bonds, release of methane, CO, and CO_2, and structural rearrangements (Rials and Glasser 1984). The lignocellulosic materials decompose thermochemically between 150 and 500°C: hemicelluloses, mainly between 150 and 350°C, cellulose between 275 and 350°C, and lignin between 250 and 500°C (Kim et al. 2004).

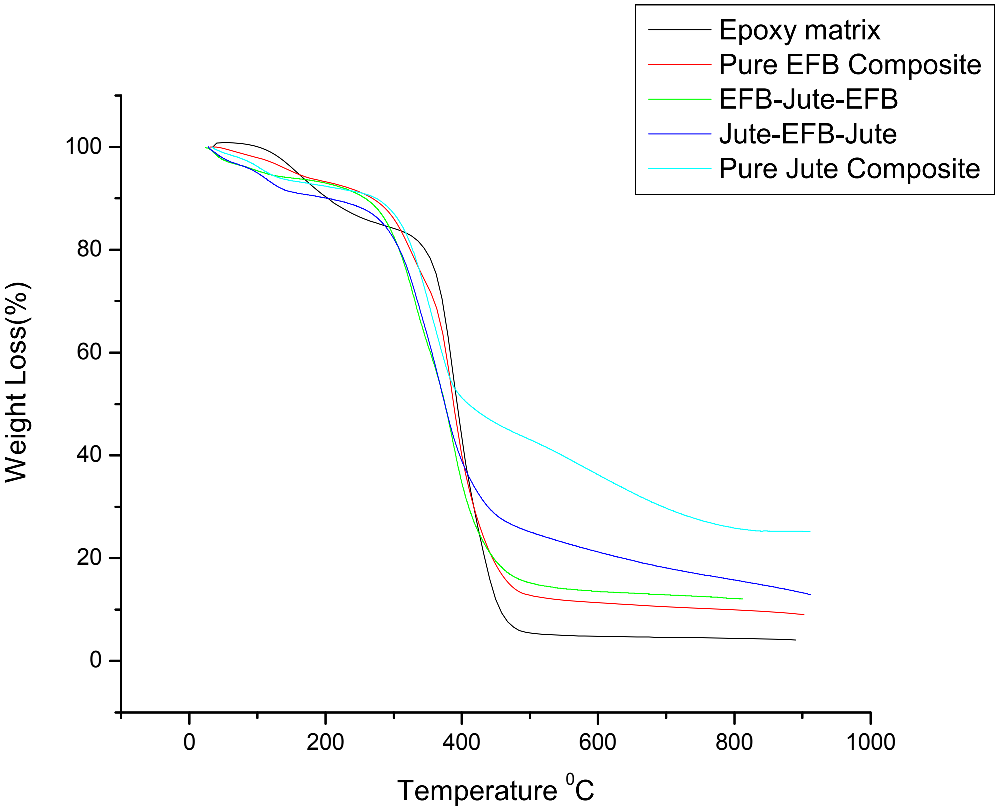

Fig. 5. Thermal Gravimetric Analysis (TGA) graph of Hybrid, epoxy, pure EFB and pure jute composites

All the composites had rapid degradation within a narrow temperature range between 260 and 480°C. The hybrid composites recorded 60 to 70% weight loss compared with pure EFB composite, which recorded well over 76% weight loss within a similar temperature range of 260 to 480°C. Epoxy matrix recorded 80% weight loss in the same temperature range. It was observed that hybrid composite gave better heat resistance than pure EFB and epoxy composites at temperatures between 30°C and 500°C. Further heating to 900°C led to an average residual weight of 9 to 25% for all the composites. With the hybridization of oil palm EFB fibers with jute fibers, the final decomposition temperature and ash content of the hybrid composite shifted slightly towards higher temperature range than that of epoxy matrix and other composites. This result indicates that the incorporation of jute/ oil palm EFB fibers into the epoxy matrix improved thermal stability of the pure EFB composite. The thermal stability of jute fiber is enhanced in epoxy matrix due to better jute/epoxy interaction. In case of hybrid composites, thermal stability slightly increased as a result of the high thermal stability of jute fiber, which acts as barriers to prevent the degradation of oil palm EFB fibers.

Table 3. Thermal Properties of Epoxy, Pure EFB, Pure Jute and Hybrid composites

Composites	**Degradation Temperature(^{0}C)**		**Char Residue (%)**
	T_{IDT*}	**T_{FDT*}**	
Epoxy	256	418	3.95
Pure EFB	260	433	9.04
EFB/Jute/EFB	268	443	12.1
Jute/EFB/Jute	271	441	12.92
Pure Jute	288	499	25.2

* IDT= Initial decomposition temperature * FDT= Final decomposition temperature

CONCLUSIONS

The results presented here showed that hybridization of oil palm EFB fiber with jute fiber is an effective way to improve dynamic mechanical and thermal properties of pure EFB composites. Dynamic mechanical behaviour of pure EFB composite and oil palm EFB/jute fibers hybrid composites have been studied with special reference to the layering pattern of fiber mat. The effect of temperature on storage modulus, loss modulus, and damping factor was studied by keeping the relative weight fraction of oil palm EFB and jute to 4:1.

The storage modulus results showed that pure epoxy had the lowest, and pure jute composite the highest storage modulus value. It is readily apparent that the hybrid composite showed high storage modulus due to addition of jute fiber. Jute fiber increased the storage modulus of the pure EFB composite due to the stiff nature of the jute fiber. The trilayer composite (jute/EFB/jute), in which jute was used as skin and oil palm EFB as core material showed better stiffness compared to EFB/jute/EFB. The loss modulus values of pure EFB composite increased with jute fiber loading. It was found that incorporation of small amount of jute fiber to oil palm EFB/epoxy composite enhanced the damping characteristics of the hybrid composites.

Cole-Cole plots showed an imperfect semicircular shape, indicating heterogeneity of the system as well as good fiber/matrix adhesion. The thermal stability of hybrid composite was higher than that of pure EFB and epoxy matrix, as was clearly demonstrated from the residue content obtained at the end of the degradation process.

The thermal stability of epoxy matrix was enhanced after the incorporation of jute or oil palm fibres due to formation of a crosslinked polymer network in the composites. In case of hybrid composites, thermal stability slightly increased as a result of reinforcement of the more thermally stable jute fiber, which can acts as barriers to prevent the degradation of oil palm EFB fibers. We anticipated that these studies will contribute to the optimized use of oil palm EFB fibers and their utilization in development of unique cost-effective advanced composites possessing appropriate stiffness, damping behavior, and thermal stability.

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