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Differential scanning calorimetry (DSC) analysis of abaca fibre (*Musa textile Nee*) reinforced high impact polystyrene (HIPS) composites

E.H. Agung[1,5,a], S.M. Sapuan[2,b], M.M. Hamdan[3,c], H.M.D.K. Zaman[4,d] and U. Mustofa[5,e]

[1,2]Department of Mechanical and Manufacturing Engineering, 43400 UPM Serdang, Selangor, Malaysia

[3]Faculty of Engineering, Universiti Pertahanan Nasional Malaysia, Kem Sungai Besi 57000, Kuala Lumpur, Malaysia

[4]Radiation Technology, Malaysia Nuclear Agency, Bangi, 43000 Kajang, Malaysia

[5]Faculty of Engineering, Universitas Malahayati, Kemiling, 35127 Bandar Lampung, Indonesia

[a]efriyo@yahoo.com, [b]sapuan@eng.upm.edu.my, [c]megat@upnm.edu.my, [d]khairul@Nuclear Malaysia.gov.my, [e]alfha_usman@yahoo.com

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Abstract. Differential scanning calorimetry (DSC) was used to study the thermal behaviour of abaca fibre reinforced high impact polystyrene (HIPS) composites. Thermal analysis is based upon the detection of changes in the heat content (enthalpy) and the glass transition temperature (*Tg*) of optimum condition of abaca fibre reinforced HIPS composites. In this research, glass transitions temperature (*Tg*) of neat HIPS occurred below the *Tg* of optimum condition of composites as the temperature of an amorphous state. The endothermic peak of composites was in to range 430-435^{0}C including neat HIPS and it observed that enthalpy of abaca fibre reinforced HIPS composites yielded below the neat HIPS 748.79 J/g.

Introduction

The combination of a plastic matrix and reinforcing fibres gives rise to composites having the best properties. Since the plastics are soft, flexible and lightweight in comparison to fibers, their combination provides a high strength-to-weight ratio to the resulting composite. The properties of composites depend on those of the individual components and on their interfacial compatibility.

Generally, some types of polymers have been used as matrices for natural fiber composites [1-3] including polyethylene (PE), polystyrene (PS) and polypropylene (PP). These polymers have a different affinity towards the fibre due to the difference in their chemical structures. Joseph, et al. [3] reported that sisal/LDPE (low density polypropylene) composites released a better reinforcing effect because of high matrix ductility and high strength/modulus ratio of sisal as compared to that of LDPE matrix. Nair, et aL [4] reported that the *Tg* values of polystyrene composites reinforced with short sisal fibres are lower than that of unreinforced PS and may be attributed to the presence of some residual solvents in the composites.

Thermal analysis is also used as an analytical method in understanding the structure property relationships and thermal stability of composite materials, such as the incompatibility between fibres and polymer matrices. The incompatibility of components affected deteriorating performance for thermal and mechanical properties. In the case of stress transfer from the matrix to the fibre depends on fibre and fibre-matrix interactions, cellulose fibres and lignin were an important term on the properties of the composites. Thermal analysis of cellulose fibres and the effects of crosslinking observed in glass transition and crystallinity states on the natural fibres composites as a function of cellulose fibres. The majority of natural fibres as a function of cellulose fibres and lignin, have low degradation temperatures (~200 ^{0}C), which make them inadequate for processing temperature above 200 ^{0}C [5]. To solve the processing of natural fibre composites, it is necessary to promote polymer modification with polar groups (such as maleic anhydride, stearic acid or glycidyl

methacrylate) to enhance the adhesion between matrix and the composite components. The coupling agent more often used for this application is a polyethylene copolymer grafted with maleic anhydride [6]. Araujo, et al. [7] reported that maleic anhydride grafted polyethylene, used as coupling agent, affected to the composite thermal stability.

In this paper, the investigation of thermal behaviour in optimum condition from abaca fibre reinforced HIPS composites at glass transition and crystallization processes are presented. Commercial HIPS were used for a comparator to the natural fibre composites. The DSC methods used for evaluation of the basic thermal parameters of optimum condition of abaca fibre reinforced HIPS composites.

Experimental

Materials. Abaca (*Musa textilis Nee*) fibres are produced by Ridaka Hand Craft, Pekalongan, Central Java, Indonesia. High impact polystyrene (HIPS), Idemitsu PS HT 50, density 1.04 g/cm^3, and melt index 4.0g/10 min obtained from Petrochemical (M) Sdn. Bhd, Malaysia. Maleic anhydride (MAH), (polystyrene-block-poly(ethylene-ran-butylene)-block-polystyrene-graft-maleic anhydride) is supplied by Sigma Aldrich Malaysia (M) Sdn. Bhd, Malaysia. Impact modifier, a styrene butadiene styrene (SBS) copolymer rubber (Cyclo resin) is supplied by PT. Wahana Makmur Kencana, Jakarta, Indonesia.

Formulation of the samples. All composites in this work were formulated with 40 wt% of abaca fibre. When the chemical additive, maelic anhydride (MAH) and impact modifier were used, the proportion of the MAH as a coupling agent was 1 wt% and 3 wt%, and then the proportion of the impact modifier was 4 wt% and 6 wt% (See Table 1).

Table1. Formulation of the composites in weight percentage (wt%)

SAMPLE	Proportion in wt%			
	HIPS	ABACA	MAH	IM
HIPS	100			
HIPS/ABACA/MAH/IM (A)	55	40	1	4
HIPS/ABACA/MAH/IM (B)	53	40	3	4
HIPS/ABACA/MAH/IM (C)	51	40	3	6

The formulation of abaca fibre reinforced HIPS composites (A, B, and C) were prepared by optimum condition technique (Box Behnken Design) as reported in the previous paper [8].

Composite processing. The abaca fibres were dried under the sun light between 27 and 30^{0}C for four days. The dry abaca fibres were cut into 2 – 3 mm by means of an electronic cutting machine. Based on the proportion of abaca fibre, maelic anhydride (MAH), and impact modifier were incorporated into the neat HIPS. The processing of abaca fibre reinforced HIPS composites were accomplished using a rolling machine as shown in Figure 2. The working temperature of the rolling machine was kept approximately 200^0 C and kept the speed in the slow rate. The process was continued until all the materials were well mixed and produced the sheets of abaca fibre reinforced HIPS composites with the thickness were an average of 1 mm.

Figure1. The production of abaca fibre reinforced HIPS composites by rolling machine

Differential Scanning Calorimetry (DSC). The characterization of a material requires the use of Differential Scanning Calorimetry analysis. DSC analysis obtained quantitative and qualitative data concerning the net heat changes as a thermal behavior. The samples used for the DSC analysis were cut from the sheet of composite in

order to have a weight from range 10-14 mg. A Mettler-Toledo DSC model 822 was used to determine the thermal behaviour. The temperature was programmed for heating from 25 °C to 500 °C with a heating rate of 10 °C min^{-1} under nitrogen atmosphere.

Results and discussion

In this research, DSC was used for determine the effect of different compositions of abaca fibre reinforced HIPS composites as in optimum condition. Typical dynamic DSC scans at 10^0C min^{-1} with the temperature that was programmed in the range of 25°C to 500°C, under nitrogen atmosphere. Figure 2(a) shows the DSC curve for neat HIPS. The *Tg* of neat HIPS was clearly observed at 103.83^{0}C and 105.43^{0}C representing onset and midpoint temperature respectively. It was in agreement with the previous studies; the glass transition temperature of HIPS reported was at 107.55^{0}C [9] and 90^{0}C [4] analyzed by DSC curve. *Tg* is manifested by a drastic change in the base line, indicating a change in the heat capacity of HIPS and there was no enthalpy is associated with such transition [10]. In the DSC scan, increasing temperature of neat HIPS implied increasing heat of polymer that demonstrated by an endothermic slope. It was shown that the endothermic peak of neat HIPS was on 430.6^{0}C with an enthalpy of caloric processes was on 748.79 J/g. That peak in agreement with previous study [11], where the endothermic peak of polystyrene (PS) scanned at 380^{0}C.

Figure 2 (a,b) shows the DSC scan of abaca fibre reinforced HIPS composites. The presence of wt% composition of constituents including maleic anhydride, impact modifier and abaca fibre showed significant effect on the thermal behavior of the abaca fibre reinforced HIPS composites. The heating thermograms of abaca fibre reinforced HIPS composites (A, B, and C) represent the one peak correlated with glass transition state and another peak in the endothermic due to crystallization state. The first peak of abaca fibre reinforced HIPS composites were on 89.42^{0}C (A), 89.84^{0}C (B), and 99.33^{0}C (C) respectively (see Table 2). This peak normally attributed to the release of absorbed moisture related to the humidity on the surface from the fibres. As the natural fibres are hydrophilic, a water desorption peak is observed around 100^{0}C [12]. In this study, the highest enthalpy of abaca fibre reinforced HIPS composites (B) at transition temperature was on 70.90 J/g. Refer to highest enthalpy, the enthalpy increased by 12.86 J/g by adding 2 wt% of maleic anhydride and decreased by 17.48 J/g by adding 2 wt% impact modifier.

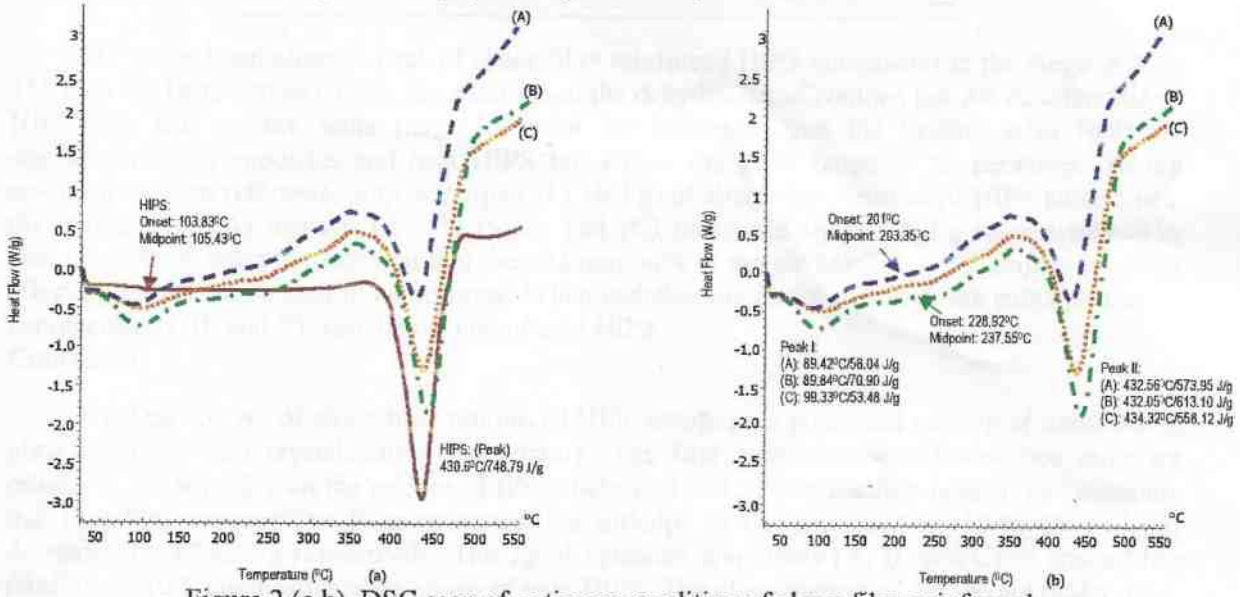

Figure 2 (a,b). DSC scan of optimum condition of abaca fibre reinforced HIPS composites (composition A, B, and C).

The compositions of abaca fibre reinforced HIPS composites (A, B, and C) may influence the energetic terms correlated with difference of *Tg*. The specimen with difference of composition was 1 wt% and 3 wt% of maleic anhydride respectively resulted in difference of *Tg*. The specimen with lower composition of Maleic anhydride (composites A compare to composites B) resulted the lower of *Tg* (see Table 2). The difference of *Tg* was ~ 34^{0}C, it summarized that maleic anhydride should play a dominant role in controlling the amorphous or glass transition state of abaca fibre reinforced HIPS composites. The composition (C) of abaca fibre reinforced HIPS composites shows the absence of *Tg*. The ability of the molecule to rotate freely/amorphous during heating yields *Tg*. Composition (C) of abaca fibre reinforced HIPS composites was less free and molecules undergo crosslinking. The value of *Tg* can provide very useful information regarding the end-use performance of a product [13]. Comparing with neat HIPS, *Tg* of abaca fibre reinforced HIPS composites (A and B) increased by 97.92^{0}C and 132.12^{0}C respectively.

Table 2. Results of DSC analysis for abaca fibre reinforced HIPS composites with those formulations (A, B and C)

Compositon of Sample	Glass Transition Tg (^{0}C)		Endotherm Temp (^{0}C)		Entalphy (J/g)	
	Onset Temp	Mid point Temp	Peak I	Peak II	ΔH Peak I	ΔH Peak II
Abaca reinforced HIPS composites (A), with composition: Abaca (40 wt%) Maelic Anhydride (1 wt%) Impact Modifier (4 wt%)	201.00	203.35	89.42	432.56	58.04	573.95
Abaca reinforced HIPS composites (B), with composition: Abaca (40 wt%) Maelic Anhydride (3 wt%) Impact Modifier (4 wt%)	228.92	237.55	89.84	432.05	70.90	613.10
Abaca reinforced HIPS composites (C), with composition: Abaca (40 wt%) Maelic Anhydride (3 wt%) Impact Modifier (6 wt%)	-	-	99.33	434.32	53.48	558.12
High Impact Polystyrene	103.83	105.43	-	430.6	-	748.79

The second endothermic peak of abaca fibre reinforced HIPS composites in the range of 430-435^{0}C in the DSC curves (figure 2b) determined the dehydration of composites. An endothermic of HIPS also was on the same range. It gives the indication that the heating rates (polymer degradation) of composites and neat HIPS fall within the same range of temperatures. Taking composition B as reference, with enthalpy (613.10 J/g) of abaca fibre reinforced HIPS composites, the enthalpy of (A) increased by 39.15 J/g and (C) decreased by 54.98 J/g respectively. The phenomenon of enthalpy reviewed that the addition wt% of maleic anhydride and impact modifier affecting the transition heat of composites. When endothermic peaks occurred, the enthalpies of all composites (A, B, and C) were below that of neat HIPS.

Conclusion

The DSC curves of abaca fibre reinforced HIPS composites give good reading of transition in glass transition and crystallization temperature. The first peak represented absorbed moisture release to the humidity on the surface of fibres below ~100^{0}C. From the first peak, it can conclude that by taking composition B as reference, the enthalpy of (A) increased by 12.86 J/g and (C) decreased by 17.42 J/g respectively. The *Tg* of optimum conditions (A, B, and C) of abaca fibre reinforced HIPS composites were above of neat HIPS. The phenomenon of the second peaks, peak temperature of endothermic occurred from range 430-435^{0}C including neat HIPS. The second enthalpy of abaca fibre reinforced HIPS composites yielded below the neat HIPS 748.79 J/g.

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