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Developing Hybrid Bio-composites from Kenaf / coir natural fibers Reinforced Thermoset unsaturated polyester: Thermal properties.

[1]Sameer Adnan Ibraheem, [1]Khalina Abdan, [2]Shamsuddin A. Sulaiman, [3]Aidy Ali and [4]Dayang Laila Abang Abdul Majidf

[1]*Department of Biological and Agricultural Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor D. E. Malaysia*
[2]*Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor D. E. Malaysia*
[3]*Department of Mechanical Engineering, Faculty of Engineering, National Defence University of Malaysia, Sungai Besi, 57000 Kuala Lumpur, Malaysia*
[4]*Department of Aerospace Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor D. E. Malaysia*

Address For Correspondence:
Sameer Adnan Ibraheem, Department of Biological and Agricultural Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor D. E. Malaysia.

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ABSTRACT

One of the natural fibers that attracted the interest of researchers and manufacturers because of the advantages of high toughness, high strength and stiffness with fibers low density, and biodegradability is the Kenaf "Hibiscus cannabinus", kenaf have chemical composition present by 31 -57 % of Cellulose, 15-19 % Hemi-cellulose, 21.5-23 % Pectin and Lignin, and 2-5 % of Wax. The main aim of hybridization is to highlight the favourable properties of these involved materials and try to improve those properties, at the same time is to decrease the disadvantages of those materials by fabricating a hybrid composite from those individual materials. The purpose of this study is to develop Hybrid Bio-composites from Kenaf / Coir natural fibers Reinforced Thermoset unsaturated polyester using bulk molding compound manufacturing methods and to study the effect of hybridizing with Coir fibers on the thermal properties. Thermal analysis have been conducted on the premixed bulk materials using Differential Scanning Calorimetry (DSC), and Thermogravimetric analysis (TGA) to determine the effect of natural fibers loading on the composites. The DSC results showed two regions (glass transition and crystallization). The temperatures ranges of these regions were (114-116 C^0, 125-140C^0,) respectively. TGA results showed a good contribution of the fibers loading on the degradation temperatures of the composites and the range was (254-362 C^0) for 50% materials degradations. Thermal conductivity of 12mm fibers length (9) samples were tested using Laser Flash Analyzer (LFA) technique. The lowest results were 0.075 W/(m*K) obtained from 35% of mixed fibers loading with (45 weight % of Coir / 55 weight % Kenaf). The results also showed the increasing of Coir fibers loading in the composites reduced the thermal conductivity.

INTRODUCTION

The primary advantages of natural fibers

The main advantages of natural fibers over synthetic fibers can be illustrated intoeconomic benefits, light weight with high specific strength,renewable, and biodegradability (Mohanty *et al*. 2002). The physical and mechanicalproperties of the natural fibers can be different due to the differences of their chemical and physicalcomposition, such as the structure of the fibers, cellulose content and microfibrillar angle.Thus, the performance predictions of the natural fibers composites are also difficult because of these variations of fibers

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properties. On the other hand, Natural fibers thermal properties are unstable compared to most synthetic fibers, and consider on of the manufacturing limitations for processing and working temperatures above of 200°C (Ekhlas*et al*. 2009).

Natural fiber-reinforced polymers composites are now finding extensive uses in variousfields from household articles to automobiles, such as wood fiber reinforced polymers (Plackett 2002), walls, flooring, louvers, and indoor and outdoor furniture (Nickel *et al*. 2003).kenaf have chemical composition present by 31 - 57 % of Cellulose, 15-19 % Hemi-cellulose, 21.5-23 % Pectin and Lignin, and 2-5 % of Wax, while coir have chemical composition present by 32-43 % of Cellulose, 0.15-0.25 % Hemi-cellulose, 3-4 % Pectin, 40-45% Lignin. Many attempts have been recorded to study and improve the thermal performance of the natural fibers reinforced polymers composites such as on jute fibers (Sinha *et al*. 2009), sisal fibers (Orue *et al*., 2016), kenaf and hemp (Lee *et al*., 2009), (Aziz and Ansell, 2004), (Kabir *et al*., 2012) and (Xie *et al*., 2015), banana/sisal hybrid composites (Asaithambi *et al*., 2015).

The needs of the mass production technologyfor a complicated shapes in thermoset composites manufacturing techniques, where a high temperatures needs to complete the curing process of thermoset polymers in a short time, motivated the researcher to develop and manufacture hybrid bio-composites by hybridizing kenaf reinforced unsaturated polyester with Coir fibers using Bulk Molding Compounds (BMC) technique in order to meet these needs.

MATERIALS AND METHODOLOGY

Fiber preparation:

The kenaf and Coir raw fibres were collected with a lot of dirt, stalks and impurities mixed with the fibers, cleaning processes were conducted in which the raw fibres were handily cleaned and arranged into separated bundles of these fibers. These bundles of (kenaf and coir) were cut to (12mm) using cutting machine. This cutting machine was manufactured in a local company and the part of the machine is showed in Figure 1(A and B), using pneumaticactuator controller and magnetic sensors to control the sequences of the cutting process.

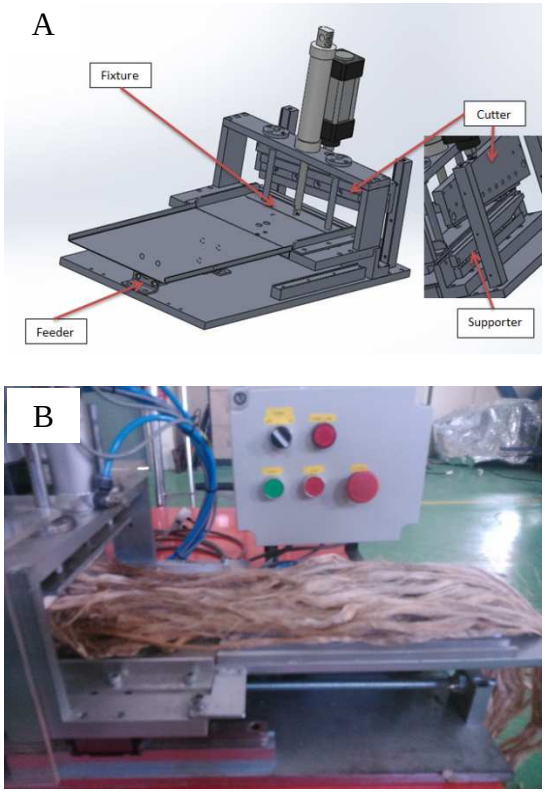

Fig. 1: Fibers Cutting Machine

After preparing the Kenaf and Coir fibers in (12mm) of length, the fibers were treated with 6% w\w concentration of silane (S. Sreenivasan *et al*., 2014). The kenaf and coir fibers were immersed separately in silane solutions for 24 hours, and subsequently washed with running water. The final process before manufacturing the samples is drying the kenaf and coir fibersat 85 °C for 24 hours using laboratory oven.

Matrix preparation:

The wet mixing compounds (resin, LP1 (Styrene monomers), PBQ (Para-Benzoquinone), Trig C (tert-Butyl peroxybenzoate), Trig 21-OP 50 (tert-Butyl peroxy-2-ethylhexanoate and SAk as releasing agent) have been supplied by local company (WAH MA CHEMICAL SDN. BHD), table 1 illustrates the name,weight percentage of these compounds, mixing the matrix components were carried out for 10 minutes using Shear Rotor Stator mixer.

For thermal conductivity samples fabrication, the mold was designed as a one open plate; plate thickness was (1mm). Plate was cut to form four Circle holes to give the circular shape to the molded samples, the diameter was 1.25 cm.

Table 1: Name and Weight Percentage of Matrix Compounds

Components	Weight %
Resin	66.3%
LPI	28.50%
PBQ	0.025%
Trig C	0.48%
Trig-21OP50	1,055%
SAK	3.64%

Mixing the hybrid bulk compounds and molding processs:

Treated two fibers (Kenaf, Coir) were mixed using industrial planetary mixerin three different weight percentage (85/15, 70/30 and 55/45 % of weight Kenaf/Coir), the mixing process were carried out for 15 minutes, while the total mass were fixed to be 90 gm for the mixed fibers for each mixing ratio.

The matrix were added after 15 minutes of fibers mixing process, mixed fibers weight were (15/75, 25/65, 35/55) to the matrix components weight (F/M) % and final step is adding the additives (5% of $Al(OH)_3$ and 5% of $CaCo_3$) measured by weight also, the additives were fixed as (5% of $Al(OH)_3$ and 5% of $CaCo_3$) measured by weight for all formulations. The adding process and mixing of matrix with mixed fibers and additives took 20 minutes. Table 2 illustratessamples names and weight ratio of fibers and matrix.

Using molding compression process, the samples were fabricated. All the mixed bulk compounds were preheated in the hot press machine for 1 minute and 30 seconds on 170 ^{0}C and then at the same temperature pressed inside the plate mold with 75 bar of pressure for 2 minutes then cool down with cooling plate at room temperature of the press machine for 4 minutes.

Differential Scanning Calorimetry (DSC):

The TA Instruments DSC Q20 Differential Scanning Calorimeter (DSC) module provides for calorimetric applications. Using two Aluminum pans once for the samples and the other is empty and considered as reference pan, the weight of the samples were 6-8 mg. This instrument allows the measurement of differential heat flow from a sample as it is heated to high temperatures. By calculating the differences of the heat flow to the temperatures and time between the reference pan and the sample pan the results will plot out. The test conducted starting with 30^{0}C to 300^{0}C with ramp of 10^{0}C/min for all samples, Tg., crystallization points and other endo or exotherm reactions can be point out from the graphs.

Table 2: Names and Weight Ratio of Samples Compounds

Samples names	kenaf%/ Coir %	Mixed fiber%/ matrix%	mass of matrix gm
K55C45U55	55/45	35/55	142
K55C45U65	55/45	25/65	234
K55C45U75	55/45	15/75	450
K70C30U55	70/30	35/55	142
K70C30U65	70/30	25/65	234
K70C30U75	70/30	15/75	450
K85C15U55	85/15	35/55	142
K85C15U65	85/15	25/65	234
K85C15U75	85/15	15/75	450

Thermo-Gravimetric Analysis (TGA):

The weight loss of samples to temperatures profile while heating up the samples was measured using TA Instruments Q500 TGAThermo-Gravimetric Analyser. Thermal decomposition temperatures can be measured using TGA which is considered as reflection of the thermal stability of the materials during thermal loading. Samples weights were between 5-8 mg, heated up from 30^{0}C to 1000^{0}C with ramp of 10^{0}C/min for all samples.

Thermal conductivity:

The NETZSCH LFA 447 NanoFlash isbased on the well-known flash method. In this method, the front side of aplane-parallel circularsample is heated by ashort light pulse. The resultingtemperature rise on the rear surface ismeasured using an infrared detector.By analysis of the resulting temperatureversus-timecurve, the thermal diffusivitycan be determined. In different four temperature (30, 80, 130, and 180)^{0}C the thermal conductivity was calculated for each sample.

RESULTS AND DISCUSSION

Differential Scanning Calorimetry (DSC):

Figure 2 shows the general trend of the DSC results for all the samples, from the graph one peak (exothermic) and two valleys (endothermic) can be shown for all samples. The peak represents the crystallization reactions temperature (Tcr), while the first valley represents the glass temperature (Tg) and the second valley represents the $Al(OH)_3$decomposition reactions and releasing the water molecules.

Table 3 shows the temperatures values of these peaks

Table 3: peaks values obtained from DSC

Samples names	Tg ^{0}C	(Tcr) ^{0}C	$Al(OH)_3$ Decomposition^{0}C
K55C45U55	116.6	135.2	223.5
K55C45U65	116	130	227
K55C45U75	114	125.5	259
K70C30U55	116.7	140	236
K70C30U65	114	132	238
K70C30U75	115	127	201
K85C15U55	116	134	185
K85C15U65	115	133	222
K85C15U75	115	133	231

The Tg values were found to be between 114 ^{0}C to 116.7 ^{0}C for all samples. The crystallization reactions temperatures where increased by increasing the fibers contents that's due to the appearance of these fibers within the crystals which is blocked heat and prevent the crystals from having the sufficient heat to complete the crystallization reactions and needed more heat and higher temperatures to do so.

$Al(OH)_3$decomposition reactions points shows differences in temperatures between 185 ^{0}C to 259 ^{0}C . These differences most probably due to the small amount of $Al(OH)_3$and the size of the samples were also small so the appearance of $Al(OH)_3$were different and the required heat and temperatures were also different.

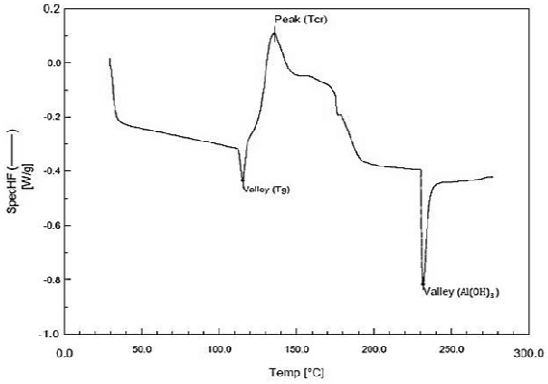

Fig. 2: DSC general trendsGraphs.

Thermo-Gravimetric Analysis (TGA):

As shown in figure 3and table 4 the 10%, 30% and 50% of degradations were happened with temperatures range of 259 - 290 ^{0}C, 328 -336 ^{0}C and 351-362 ^{0}C respectively.

Table 4: TGA temperatures results for (10%, 30% and 50%) of degradations

Samples names	10% at ^{0}C	30% at ^{0}C	50% at ^{0}C
K55C45U55	259	329	355
K55C45U65	260	328	354
K55C45U75	290	335	362
K70C30U55	271	333	357
K70C30U65	265	330	356

K70C30U75	279	334	359
K85C15U55	282	336	359
K85C15U65	264	329	351
K85C15U75	285	333	358

Up to 50% of degradation, most of the moistures, hemicelluloses and celluloses which is up 380 ^{0}C(Aziz and Ansell, 2004),(Buggy, 2006) and (Xie *et al*., 2015).However, decreasing the fiber ratio to the matrix from 25 fibers/ 65 matrix % of weight to 15 fibers/ 75 matrix % of weight showing higher temperatures for degradations thus due to the natural fibers degradability in lower temperatures than the thermosets matrix.However the effects of coir fibers were so obvious in the matrix ratio of 75% that most likely to the good absorption of coir fibers to the matrix which it's prevent the heat from degrade the fibers and also to the fewer amounts of celluloses compared to kenaf fibers.

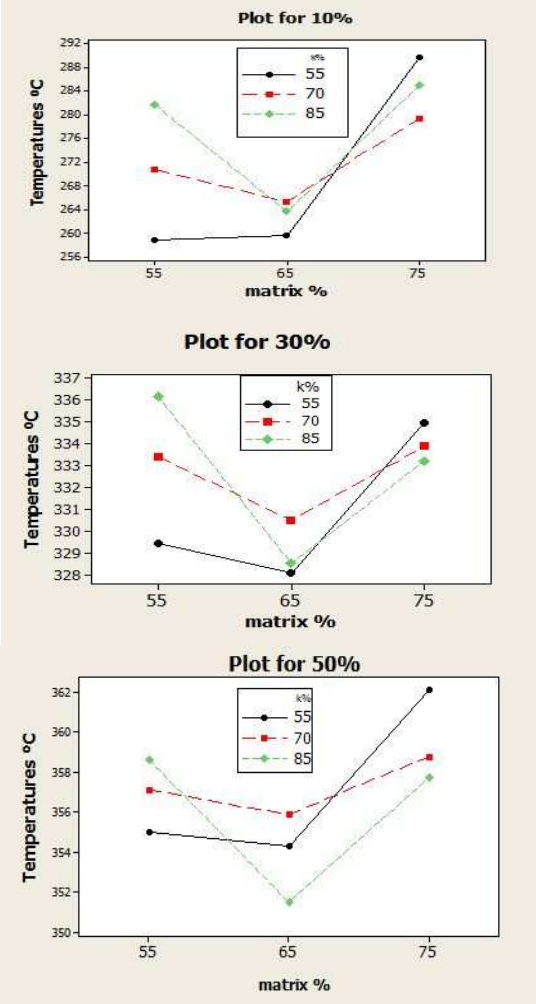

Fig. 3: TGA resultsTGA temperatures results for (10%, 30% and 50%) of degradations

Thermal conductivity:

Figure 4 and table 5 shows the results of conductivity for all samples and for four temperatures range of measurements (30 ^{0}C, 80 ^{0}C, 130 ^{0}C and 180 ^{0}C).

From figure 4 can be noticed that the thermal conductivity values were decreased by increasing the range of test temperatures from 30 ^{0}C to maximum 180 ^{0}C thus due to the moistures evaporation from the natural fibers and giving spaces to air to replace these moistures, knowing that the thermal conductivity of the air is lower than water.

Table 5: Thermal conductivity

Samples names	Thermal Conductivity W/(m*K)			
	30 C^0	80 C^0	130 C^0	180 C^0
K55C45U55	0.082	0.08	0.078	0.075
K55C45U65	0.255	0.246	0.228	0.215
K55C45U75	0.134	0.111	0.1	0.095
K70C30U55	0.135	0.125	0.119	0.114
K70C30U65	0.225	0.211	0.201	0.189
K70C30U75	0.165	0.142	0.136	0.13
K85C15U55	0.132	0.12	0.116	0.111
K85C15U65	0.171	0.154	0.148	0.14
K85C15U75	0.156	0.136	0.133	0.129

Different trends can be observed also for the thermal conductivity, the thermal conductivity for the samples that contain 25% fibers/ 65% matrix measured by weight were the highest values among all the samples , most probably the good adhesion between the matrix and the fibers with less moistures and air voids.In contrary the samples that contain 35% fibers/ 55% matrix measured by weightshow that the increasing in coir fibers ratio led to decrease the thermal conductivity, and thus due to the hollow and the big diameters of coir fibers compared to the kenaf fibers which made it more possible to capture the air within the hybrid composites.

Conclusions:

- An innovative hybrid bulk molding compound has been developed successfully by hybridizing short kenaf fibers (12 mm length) with short coir fibers (12 mm length) to reinforce modified unsaturated polyester.
- DSC results show that The Tg values were found to be between 114 ^{0}C to 116.7 ^{0}C for all samples and the crystallization reactions temperatures where increased by increasing the fibers contents.
- TGA results show the effects of coir fibers were so obvious in the matrix ratio of 75% measured by weight.
- The thermal conductivity for the samples that contain 25% fibers/ 65% matrix measured by weight were the highest values among all the samples, and the effect of the increasing coir fibers ratio were obviously reduced the thermal conductivity for the samples that contain 55% of matrix.

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