

Thermal property determination of hybridized kenaf/PALF reinforced HDPE composite by thermogravimetric analysis

I. S. Aji · E. S. Zainudin · A. Khalina ·
S. M. Sapuan · M. D. Khairul

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Abstract This article presents the thermal degradation behavior of hybridized kenaf (bast)/pineapple leaf fiber (PALF) reinforced high density polyethylene (HDPE) composites by thermogravimetric and derivative thermogravimetric analyses (TG/DTG) with respect to the proportions of fiber in the composite, variation in fiber loading and fiber length. It was observed that the thermal decomposition of all the samples had taken place within the scheduled temperature range of 35–615 °C. For hybrid composites prepared at 40% fiber loading, the initial peak between 236.9 and 331 °C corresponds to a mass loss of between 23 and 26%, and expectedly, PALF composite and 1:1 hybrid composite have the highest mass lost at this point. Main decomposition temperature as revealed from DTG curves occurred around 467 °C for all except composite prepared with 0.75 and 2 mm fiber length. The mass

loss at this temperature was between 64.4 and 73.7%. However, at 464.87 °C, around 98% of neat HDPE had already degraded. Decomposition temperature of other composites was a little higher than the temperature at which HDPE concluded decomposition. Kenaf composite on its own showed initial thermal resistance, but above 240 °C, a sharp increase in decomposition occurred with temperature. Interestingly, hybridization took care of this. Kenaf and PALF composite have shown weaker thermal stability compared to neat HDPE at lower temperatures. The introduction of more fiber into the matrix at onset caused the thermal stability of the hybridized composite to decrease. This reduction in thermal stability of the hybrid with increase in fiber loading became obvious after the dehydration process. Decomposition of hybrid composite is directly proportional to increase in fiber loading. However, at 385 °C, where neat HDPE started decomposing, the percentage degradation of the hybrid showed inverse proportionality with increase in fiber loading. As observed, the size of the lignin and hemicelluloses shoulders in DTG curves deepen with increase in fiber loading, an indication of increased presence with increase in fiber loading.

I. S. Aji (✉) · S. M. Sapuan
Department of Mechanical and Manufacturing Engineering,
Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM
Serdang, Selangor, Malaysia
e-mail: suleimanaji@yahoo.com

E. S. Zainudin
Laboratory of Biocomposite Technology, Institute of Tropical
Forestry and Forest Products, Universiti Putra Malaysia, 43400
UPM Serdang, Selangor, Malaysia

A. Khalina
Department of Biological and Agricultural Engineering, Faculty
of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang,
Selangor, Malaysia

M. D. Khairul
Polymer and Radiation Division, Malaysian Nuclear Agency,
43000 Bangi-Kajang, Malaysia

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Introduction

It is now very clear that utilization of natural bioresources is critical to the sustenance of our dwindling environment. Combining these renewable resources with the sole aim of producing components that are environmentally benign cannot be over emphasized. Natural fibers have been combined in a variety of ways for many decades with great

success. Sustained effort had been put in place for decades to introduce high performance engineering materials from very interesting renewable resources. Natural fibers have been exploited for various purposes mainly as filler materials for automobile and construction materials. Reports shows that sisal, jute, oil palm empty fruit bunch (OPEFB), pineapple leaf, banana, bamboo, coir, kenaf fibers have all proved to be good reinforcement materials as composites in thermoplastic and thermosetting matrices [1–7], as previously reviewed [8]. The surge in interest for recycling and use of biodegradable materials such that thermo-mechanical properties and reduction of water sensitivities in composites are improved has led to the use of natural fibers as biodegradable fillers [9]. To characterize materials, it is important that its thermal properties be fully studied. This analysis can help us to study the properties of the raw materials used in the fabrication of their composite, which will then help us understand the behavior of the final product. Furthermore, to get the best balance of composite's properties, i.e., modulus, thermal stability, and energy damping ($T_g T_m$), there is the need to understand the matrix to be use, reinforcing elements and their interfaces [10].

High temperature processing of both natural and synthetic polymers no doubt influence the degradation of its mechanical properties [11, 12]. Earlier work [13] has shown that with hybridization, higher fiber loading can be obtained in composite formulation and because of the thermal stress involve during the manufacture of natural fiber composite prepared with thermoplastic material, knowledge of influential factors such as temperature is paramount. In most cases, thermogravimetric analysis (TG) and differential scanning calorimetry (DSC) is employed to study such factors. They can help determine the thermodynamic data and the kinetics of the system [12]. Through such means, Ramiah [14] reported that the high activation energies obtained from cellulose was as a result of its highly ordered structure. Reports [10, 15] from literature have shown that degradation of fibers does not occur until around 160 °C. Thermal stability gradually decreases, thus, enabling degradation at this temperature. Lots of work [16–19] has been done to investigate the thermal stability of natural fiber composites and results obtained have shown that fiber treatments/modification has greatly enhanced thermal stability of composites. George et al. [10] presented the thermal behavior of PALF reinforced with polyethylene (PE) by thermogravimetric and dynamic mechanical thermal analysis, effects of fiber loading and surface modification on the thermal properties were evaluated and the result showed that at high temperature, PALF degrades before the PE matrix. The degree of crystallinity and morphology play a significant part in influencing the macroscopic properties exhibited by polymers [20]. Elastic

modulus, tensile strength, drawing behavior, electrical and optical properties, and permeability to vapors are directly related to the film crystalline microstructure [21]. High density polyethylene (HDPE) is kind of amorphous, especially when used at high temperature, and since most polymers are processed at high temperature, thermal stability of natural fibers or fillers is one of the factors to be considered in developing natural fiber composites [22–24].

In hybridization, the properties to be obtained largely depends on the length of individual fibers, fiber loading and orientation, level of mixing, fiber to matrix bonding, and the arrangement of individual fibers in the composite. Limitation of hybrid strength is dependent on the failure strain of individual fibers, which is why maximum hybrid strength is obtained when the combining fibers are closely strain compatible [25]. In essence, the properties to be obtained in hybridizing two components can also be predicted by the rule of mixtures, i.e., [26]

$$P_H = P_1 V_1 + P_2 V_2,$$

where P_H is the property to be investigated, P_1 the corresponding property of the first system and P_2 the corresponding property of the second system. V_1 and V_2 are the relative hybrid volume fractions of the first and second system. However,

$$V_1 + V_2 = 1$$

A positive or negative hybrid effect means positive or negative deviation of certain mechanical properties from this rule. Hybrid effect has been used to describe the interesting synergistic improvement in the properties of composite housing two or more fibers [25].

The concept of “engineered natural fibers” is employed here with the intention of realizing superior strength bio-composite [27]. The concept explores the appropriate blending of bast(stem) and leaf fibers in order to achieve optimum balance in mechanical properties; correct intermingling of bast and leaf fiber is expected to provide a stiffness–toughness balance in the composite [26, 27]. Burgueno et al. [28] sequenced studies of natural biofiber composite, exposing how they can be utilize for structural application non exotically but by enhancing their performance through optimized hybridization. In this article, thermal decomposition behavior of kenaf/PALF reinforced HDPE composites is presented based on thermogravimetric and derivative thermogravimetric analyses with respect to proportions of fiber in the composites, variation in fiber length and fiber loading. It is expected that by combining kenaf and PALF, thermal stability of the composite will be improved through synergistic relationship between the fibers. Delay in the decomposition of the weaker fiber will also be achieved.

Materials and methods

Materials

Pineapple leaf (*Ananas comosus*) was bought from Perniagaan Benang Serat Nanas M&Z, with source from Johor pineapple plantation, Malaysia. It was manually decorticated from the variety “Josephine”. Kenaf (bast) (*Hibiscus cannabinus*) of variety V36, was purchased from the company, KEFI Malaysia Sdn. Bhd. and utilized in this research. These fibers were reinforced with injection-grade HDPE also purchased from KEFI Malaysia. Properties of HDPE used as provided by supplier are shown in Table 1, while Table 2 summarizes some chemical compositions of the two fibers used.

Method

Preparation of hybrid composites

Fibers of kenaf and PALF hybridized at a fiber loading of 40% and average fiber length of 0.25 mm were utilized for the first part of this experiment. The second part of the work that dwelled on effect of fiber loading utilized varying fiber loading of 10, 20, 30, 40, and 50% at constant fiber length of 0.25 mm, while the last part presents the effect of fiber length of 0.25, 0.5, 0.75, and 2 mm on the thermal stability of the composite at constant fiber loading of 50%. Kenaf and PALF at 1:1 fiber proportions were utilized for the section that dealt with effect of fiber loading

and fiber length on the hybridized composite. An average diameter of kenaf and PALF of 76 and 130 μm , respectively, were utilized in this work. The fibers were carefully and thoroughly mixed together before melt mixing them with the HDPE in a Thermo-Haake using roller blades and a mixing chamber with volumetric capacity of 69 cc that was preheated and set at 190 $^{\circ}\text{C}$ and 40 rpm processing speed in a total time of 25 min [30]. The HDPE was first charged at that temperature and allowed to stabilize (melt) before introducing the fibers. The mixed composite obtained from the internal mixer was cut into very small pellet for the TG/DTG experiment. To understand how the two hybridizing fibers contributed to thermal stability of the hybrid composite in the first part of the work, their proportions were varied and then equalized at 1:1 (Kenaf_PALF_40% + HDPE) as presented in Table 3. Composite of PALF and that of kenaf composite were also tested individually so that the merit of hybridization can be seen.

Thermal properties (thermogravimetric analysis, TG)

Kenaf fiber composite, PALF fiber composite, hybrid of kenaf/PALF, and neat HDPE were subjected to thermogravimetric analysis using Perkin-Elmer TGA-7 equipment. Samples of between 10 and 18.5 mg mass were scanned from 35 to 615 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ in nitrogen atmosphere and the corresponding percentage mass loss was recorded.

Table 1 Properties of HDPE used

Density/ g/cm^3	Melt mass flow rate	Dart drop impact	Tensile elongation	Forms	Melt temp./ $^{\circ}\text{C}$	Sec. Mod/ MPa
0.95	190 $^{\circ}\text{C}$ /2.16 kg 0.10 g/10 min	17 μm , Blown film 140 g	TD: Break 17 μm , Blown film 700%	Pellets	180–240	1% Sec, TD 17 μm , Blown film 1270

Table 2 Chemical composition of kenaf and PALF fibers [29]

	Extractive/%	Holocelluloses/%	α -Celluloses/%	Lignin/%	Ash/%
Kenaf Bast	5.5	86.8	55.0	14.7	5.4
PALF	5.5	80.5	73.4	10.5	2.0

Table 3 Proportions of fibers in the formulated hybrid

	Formulations/total fiber loading kept at 40%				
	K3P7/%	K4P6/%	K6P4/%	K7P3/%	KP1/%
Proportion of fibers in hybrid					
Kenaf	30	40	60	70	50
PALF	70	60	40	30	50

Fig. 1 TGA of various fiber ratios employed, kenaf composite, PALF composite compared to that of neat HDPE

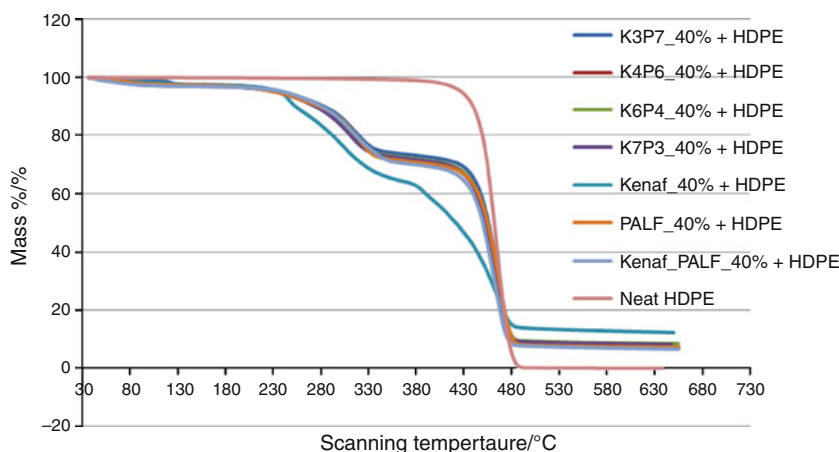
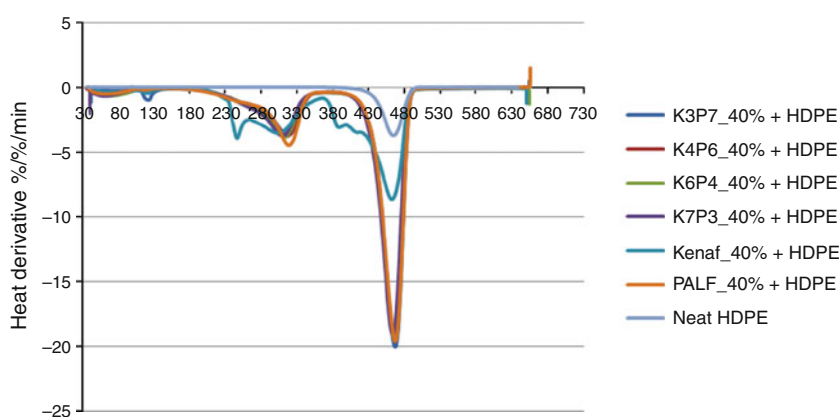


Fig. 2 DTG of various fiber ratios employed, kenaf composite, PALF composite compared to that of neat HDPE



Results and discussion

Variations in fiber ratios

Total overall fiber loading in this section of the work was kept at 40%. The abbreviated proportions are as described in Table 3. The TG and DTG curves of various fiber ratios employed, kenaf composite, PALF composite and that of neat HDPE are presented in Figs. 1 and 2, respectively. It was observed that the thermal degradation of all the samples has taken place within the scheduled temperature range of 35–615 °C. All the hybrid ratios employed, kenaf and PALF composites (Fig. 1), experienced dehydration around 35–130 °C and maximum percentage of cellulose was found to decompose at a temperature range of 462–467 °C. This decomposition temperature range was within the range of the hybrid composites.

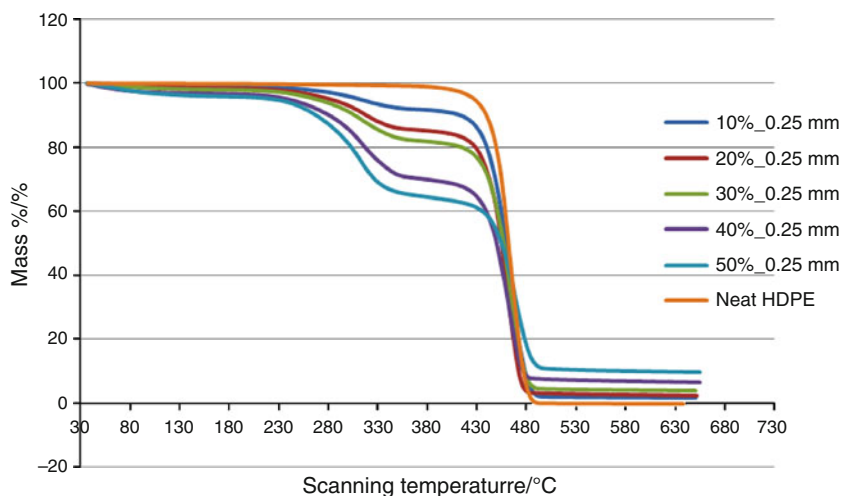
The decomposition of neat HDPE started at a temperature of 385 °C and 98.8% decomposition occurred at 464.9 °C (Fig. 2). This amount of decomposition is much greater than what was obtained in the composites under consideration. For hybrid composites prepared at 40% fiber loading (Fig. 1), the initial peak between 236.9 and 331 °C

corresponds to a mass loss of between 23 and 26%, and expectedly, PALF composite and 1:1 hybrid composite have the highest mass lost at this point. This was due to dehydration from cellulose unit and thermal cleavage of glycosidic linkage by transglycosylation and scission of C–O and C–C bonds, i.e., the thermal depolymerization of hemicellulose and the glycosidic linkages of cellulose [19]. Subsequent decomposition occurred between 362 and 513 °C with the main decomposition temperature as revealed from DTG curves around 467 °C (Fig. 2). The mass loss at this temperature was between 64.4 and 73.7% (kenaf composite having the lowest mass lost and K3P7 with the highest). This could have been because of aromatization, involving dehydration reactions [10, 31]. At 464.87 °C, HDPE (Fig. 2) fully decomposed. Decomposition temperature of other composites was a little higher than the temperature at which HDPE concluded decomposition. Kenaf composite on its own showed initial thermal resistance, but above 240 °C, a sharp increase in decomposition occurred. This could be as a result of higher percentage of lignin in kenaf (Table 2). Interestingly, on hybridization, this problem was taken care of as can be seen in Figs. 1 and 2 except at the maximum

Table 4 Degradation temperatures obtained from TG and DTG curves

Sample	$T_i/^\circ\text{C}$	$T_{\text{max}}/^\circ\text{C}$	$T_f/^\circ\text{C}$	Max. DTG/ $^\circ\text{C}$	Final mass left/%
Neat HDPE	386	–	503	464	0.06
Kenaf composite	179	363	514	463	13.6
PALF composite	160	384	502	467	7.7
K3P7	170	362	511	467	7.8
K4P6	150	358	509	465	8.1
K6P4	152	364	509	465	9.1
K7P3	168	386	497	464	9.1
KP	158	368	505	462	7.4

T_i , Initial degradation temp.; T_{max} , maximum degradation temp.; T_f , final degradation temp.

Fig. 3 TGA at various fiber loading compared to that of neat HDPE

decomposition temperature of the fibers. Hybrid ratio of 1:1 provided a better stability between 40 and 250 °C when compared with other hybridized composites. Generally, hybridization has enhanced the thermal stability of composite. Similar result was also shown by Jawaid and Khalil [32]. Table 4 summarizes the result obtained on the DTG data sheet to ease clarification.

Effect of fiber loading

The TG and DTG curves of hybridized kenaf/PALF reinforced HDPE at varying fiber loading is shown in Figs. 3 and 4. Dehydration due to the loss of water in fibers used still occurred in temperature range of 35–130 °C for all fiber loading. Figure 3 shows the change in thermal stability. This result indicates that kenaf and PALF fibers have weaker thermal stability compared with neat HDPE matrix at lower temperatures. The introduction of more fiber into the matrix at onset caused the thermal stability of the hybridized composite to decrease. This reduction in thermal stability of the hybrid with increase in fiber loading became obvious after the dehydration process. Lignocellulosic materials decompose thermochemically between

150 and 350 °C; cellulose between 275 and 350 °C, lignin at a very wide range of 250 to 500 °C [33]. Hydrogen bonding provides a source of stability in cellulose. This is because it allows thermal energy to be distributed over many bonds but this also creates less ordered regions with increase in temperature. As these less ordered region increases, the decreasing mobility of cellulose chains will strain and weaken the existing hydrogen bonding which will effectively decrease stability [34]. This is the reason why increasing the percentage of fiber in the composite decreased its thermal stability. Clearly, decomposition of the hybrid is directly proportional to increase in fiber loading [24], similar result was obtained by Wirawan et al. [35]. However, at 385 °C, where neat HDPE started decomposing, the percentage mass loss of the hybrid showed inverse proportionality with increase in fiber loading. In the current study, it was found that after the final thermal degradation, black carbonaceous residues were present; Mohanty et al. [31], reported the same observation. Table 2 shows the chemical composition of kenaf and PALF fibers. Derivative mass after final decomposition increased with increase in percentage of fiber in the hybrid.

Fig. 4 DTG at varying fiber loading compared to that of neat HDPE

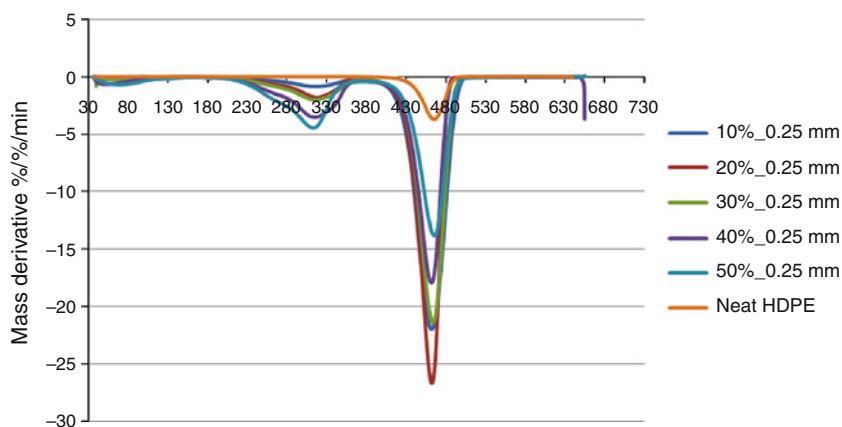
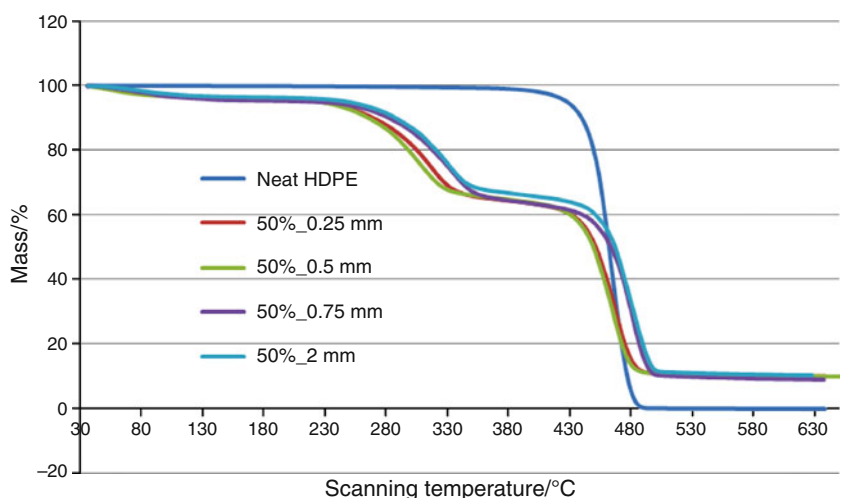


Table 5 Degradation temperatures obtained from TGA and DTG curves

Sample	$T_i/^\circ\text{C}$	$T_{\text{max}}/^\circ\text{C}$	$T_f/^\circ\text{C}$	Max. DTG/ $^\circ\text{C}$	Final mass left/%
Neat HDPE	386	–	503	464	0.06
10%_0.25 mm	236	373	509	462	1.8
20%_0.25 mm	189	362	492	462	3.1
30%_0.25 mm	206	371	505	463	4.6
40%_0.25 mm	158	368	505	462	7.4
50%_0.25 mm	176	373	514	466	10.6

Fig. 5 TG at various fiber lengths compared to that of neat HDPE



Here also, three mass loss stages were observed, which is in agreement with most lignocellulosic fiber studies. For all samples studied, the first mass loss observed is attributed to moisture desorption. Except for neat HDPE, all fiber loading DTG curves showed shoulder between 250 and 350 °C (Fig. 4) which generally corresponds with pectins and hemicelluloses content [36]. The size of the shoulders also corresponds to the amount of pectin and hemicelluloses in the composite and this deep with increase in fiber loading. Table 5 presents the degradation

temperatures obtained from TG and DTG curves with respect to fiber loading of the hybrid.

Effect of fiber length

As observed in Fig. 5, there is a shift in the thermal stability of the hybrid with increase in fiber length. The final decomposition temperature was also shifted to higher temperature, clearly away from that of neat HDPE. It is also observed that even the decomposition temperature of pectin

Fig. 6 DTG at various fiber lengths compared to that of neat HDPE

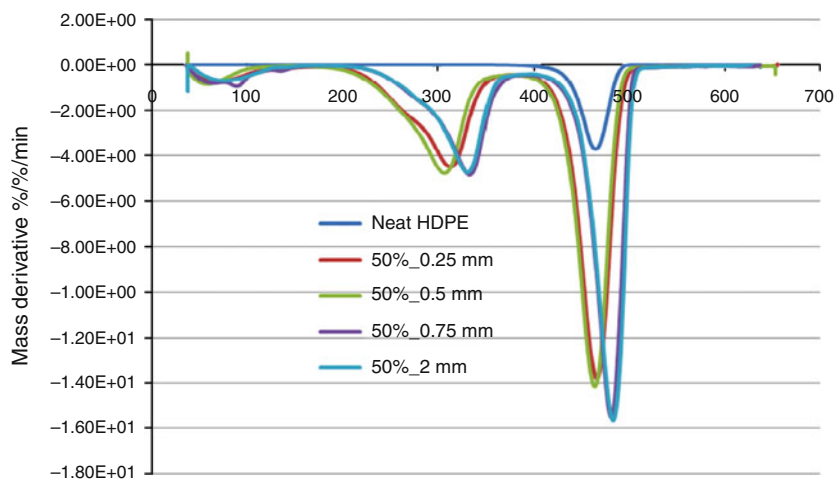


Table 6 Degradation temperatures obtained from TG and DTG curves

Sample	$T_i/^\circ\text{C}$	$T_{\max}/^\circ\text{C}$	$T_f/^\circ\text{C}$	Max. DTG/ $^\circ\text{C}$	Final mass left/%
Neat HDPE	386	–	503	464	0.06
50%_0.25mm	176	373	514	466	10.6
50%_0.5mm	183	362	503	463	11
50%_0.75mm	185	383	523	481	10
50%_2mm	195	387	528	483	11

and hemicelluloses shifted to higher temperature with increase in fiber length as depicted in DTG curve of Fig. 6. The trend observed for this work clearly shows that lower fiber content (Figs. 3, 4), in addition to longer fiber length (Figs. 5, 6), can induce better thermal stability of composites. This is because higher aspect ratio can provide greater interaction of the fibers with the matrix, increase covalent bonding and greater dispersion of matrix through the fibers, which will lead to better thermal stability of composite. However, fiber treatment has been shown to reduce the hydrophilic tendencies in natural fibers especially in the case of alkalinized fibers that can reduce hemicelluloses components such as xylans with an advantage of shift in thermal decomposition temperatures of composites to higher values. Summary of values obtained from the degradation data sheet is presented in Table 6 for ease of clarification.

Summary and conclusions

In this article, thermal decomposition behavior of Kenaf/PALF reinforced HDPE composites was presented based on thermogravimetric and derivative thermogravimetric analyses with respect to proportions of each fiber in the hybrid, fiber loading and variation in fiber length. It was observed that the thermal decomposition of all the samples had taken place within the scheduled temperature range of 35–615 °C. Main

decomposition temperature as revealed from DTG curves occurred around 467 °C for all except hybrid prepared with 0.75 and 2 mm fiber length. The mass loss at this temperature was between 64.4 and 73.7%. However, at 464.87 °C, around 98% of neat HDPE had already decomposed. Decomposition temperature of other composites was a little higher than the temperature at which HDPE concluded decomposition. Kenaf composite on its own showed initial thermal resistance, but above 240 °C, a sharp increase in decomposition occurred. Interestingly, this problem was taken care of by hybridization. Kenaf and PALF composite have shown weaker thermal stability compared to neat HDPE at lower temperatures. The introduction of more fiber into the matrix at onset caused the thermal stability of the hybridized composite to decrease. This reduction in thermal stability of the hybrid with increase in fiber loading became obvious after dehydration of the fibers. However, there is a clear shift in decomposition temperatures of the hybrid with increase in fiber length. Decomposition of hybrid composite is directly proportional to increase in fiber loading. Moreover, at 385 °C, where neat HDPE started decomposing, the percentage decomposition of the hybrid showed inverse proportionality with increase in fiber loading. As observed, the size of the pectin and hemicelluloses shoulders in DTG curves deepen with increase in fiber loading. Generally, hybridization has been shown to enhance thermal stability of composites at lower fiber loading and increased fiber length.

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