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## Matrix-Material Fabrication Technique and Thermogravimetric Analysis of Banana Fiber Reinforced Polypropylene Composites

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### ABSTRACT

From the environmental consideration, it would be very interesting to use natural fibers such as banana, jute or coir as reinforcement materials instead of artificial fibers or any kind of synthetic materials. Natural fibers have many advantages over synthetic ones. Polypropylene banana fiber composites (PPBC) are prepared using untreated and alkali-treated banana fibers at 10–25% by weight of the fiber loading. The thermal properties of polypropylene natural fiber composites are very important for technological uses. Thermogravimetric measurements show that the incorporation of banana fiber into PP enhances the thermal stability of composites containing treated fibers, in comparison with untreated fibers. A composite of biodegradable polypropylene (PP) reinforced with short banana natural fibers was prepared by melt blending followed by a hot press molding system. The thermal properties of matrix materials were studied using thermogravimetric analyzers TGA units. It is observed that the introduction of short banana fibers slightly improved the thermo oxidative stability of PP-banana composites. Physical and chemical changes occurred through dehydration, phase transition, molecular orientation, crystallinity disruption, oxidation and decomposition, and incorporation of several functional groups. Systematic investigations of the thermal behavior of polymers in gas, vacuum or inert atmosphere give the knowledge of how change takes place in polymers. To understand such changes thermogravimetric analysis (TGA) and thermal analysis (TG) were performed. It is observed reinforcement of short banana fiber leads to little improvement in the thermooxidative stability of PPBC. Due to the enhancement of thermo-mechanical properties, such composites may be used as building materials namely roof materials, selling materials and many other engineering applications.

**Keywords:** Polypropylene banana composites (PPBC); Natural fiber; Oxidative stability; Thermogravimetric analysis (TGA); Decomposition

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## 1. Introduction

There are many popular reinforcing fibers that are commonly used in the preparation of composites. In technological consideration, natural fiber composites are considered to be the most promising candidate. The term thermal analysis refers to experimental techniques used to study temperature dependent properties of materials<sup>[1-4]</sup>. Reinforcement of the natural fibers organic and inorganic polymers causes changes in their structural, physical, mechanical, and chemical properties. To study such changes in thermal properties thermogravimetric analysis (TGA) is conveniently used. To understand the behavior of treated and untreated PPBC, the mechanical properties are very significant ingredients to study. We have also studied tensile and flexural strength with different fiber loading % of PPBC. The results of the physical and mechanical properties of PPBC have already been studied by Islam et al.<sup>[5]</sup>. DTA techniques typically enable us to detect the thermal reactions and effects associated with chemical or physical changes in materials as they pass through a transition due to heating or cooling<sup>[6]</sup>. There are many methods have been proposed and employed for the estimation of kinetic parameters from dynamic thermogravimetric analysis (TGA)<sup>[7]</sup>.

In the present article, we reported banana-polypropylene composites prepared by using a very typical process developed by us. The thermal properties of polypropylene composites reinforced with untreated and alkali-treated banana fibers, PPBC are also studied in the present article. Very little scientific literature is available on the thermal stability of banana composites. Thermal analysis of untreated and chemically treated banana fibers was studied by Rana et al.<sup>[8]</sup> and the jute composite was studied by Mitra et al.<sup>[9]</sup>. The main influencing factors on the properties of composites are matrix structure, treatment or processing, and the use of fillers or reinforcements<sup>[10]</sup>. Furthermore, TG was performed for analysis and comparison purposes. The successful blending of PP and natural fibers has provided an easy way to improve the thermal properties of PPBC and create a fully biodegradable material.

The analytical experimental technique to investigate the temperature dependent behavior of a sample is popularly known to be thermal analysis<sup>[2]</sup>. There are so many polymers available today and they decomposed over distinct temperature ranges leaving behind certain proportions of volatiles and residues. TGA is a very efficient analytical technique to measure the weight loss of a test sample over a temperature range. TGA provides significant information about the stability and decomposition of polymer materials by studying volatile and residues. Significantly, the TGA trace can also be used to study the moisture content, volatile and plasticizer, and ashes, which of course extend for cross-linked polymers.

The thermal energy is supplied to the polymer in thermal analysis which results evolve of moisture content at low temperatures (80 °C to 110°C). The polymer species starts losing beyond this temperature range. The decomposition temperature is the range at which the polymer starts degrading and maintaining the process up to complete decomposition. The TGA curve traces relatively a simple sigmoidal path for many polymers. The TG curve mostly changed by the kinetic parameters: i) reaction order,  $n$ ; ii) rate constant,  $k$ ; iii) frequency factor,  $A$ ; and iv) activation energy,  $E_a$ . The elucidation mechanism of polymer degradation and thermal stability has a great impact on the values of these parameters. The TG curves may be complex if a polymer degrades by a multi-step mechanism. A number of methods have been proposed and employed for the estimation of kinetic parameters from dynamic thermogravimetric analysis (TGA) studies<sup>[6,7]</sup>. TGA offers a viable alternative to furnace aging. The materials are heated at various distinct rates including their decomposition region of temperature. Then the decomposition temperature is determined from the thermal curve.

## 2. Materials and Methods

### 2.1 Materials

The sample polypropylene (PP T101) used in the present observation has a specific gravity of 0.9. Banana fibers were collected from PP and PDC, BC-

SIR, Dhaka and Bangladesh. To remove the moisture the chopped fiber was annealed at 110 °C for 24 hours. The approximate dimensions of the chopped fiber were 2-3 mm in length and 2 mm in width (DIN IS03310/1, w = 2 mm, FRITSCH). The moisture from PP granules was removed following the same procedure of annealing. Characteristics of the raw materials (banana fiber) are presented below. We have used the commercially available PP; therefore one may consider supplied values for different properties by the manufacturer.

### Characteristics of raw materials

Banana fiber:

Physical properties:

Ultimate:

- |                         |               |
|-------------------------|---------------|
| 1) Length in mm         | : 80-280      |
| 2) Diameter/width in mm | : 0.011-0.034 |
| 3) Cell width (m)       |               |

Single fiber:

- |                              |             |
|------------------------------|-------------|
| 1) Density (apparent) gm/cc  | : 0.86-0.62 |
| 2) True density gm/cc        | : 1.33-1.31 |
| 3) Moisture regain at 65% RH | : 10-12     |
| 4) Porosity                  | : 35-53     |

Mechanical properties:

- |                           |        |
|---------------------------|--------|
| 1) Tensile strength in KP | : 3.53 |
| 2) Fiber twist in TPC     | : 5.75 |
| 3) Elongation %           | : 37   |

Chemical composition of banana fiber:

- |                              |           |
|------------------------------|-----------|
| 1) Cellulose                 | : 65.112% |
| 2) Hemicellulose             | : 17.325% |
| 3) Pectin & related compound | : 2.124%  |
| 4) Legnin                    | : 8.018%  |
| 5) Ash, wax                  | : 2.502%  |
| 6) Others                    | : 4.919%  |

Electrical properties:

- |   |                          |
|---|--------------------------|
| 1) Volume resistivity at 65% RH                   | : $6.5-7 \times 10^5$ cm |
| 2) Dielectric strength for 0.1 m length of fiber: | 5.0 KV                   |

### 2.2 Alkali treatment

To perform alkali treatment 20% NaOH was taken in a RB flask. Then the required amount of fibers was poured into this solution and kept for 15 minutes

at ambient temperature. The fibers were then moved to another container for cleaning and washing with distilled water. It was then neutralized by using a solution of low concentration sulfuric acid. The samples were washed again with distilled water following the method prescribed by Guha Roy et al. <sup>[10]</sup> and then air dried for 48 hours in an oven.

### 2.3 Composite fabrication

We have prepared a polymeric matrix by compression modeling using Paul-Otto-Weber hydraulic press as shown in **Figure 1**, which is explained in reference <sup>[2]</sup>. We have used a type die of inner diameter of 146 mm and an outer diameter is 158 mm having two discs of 7.5 on each side. During molding we have to maintain a temperature of 250 °C and 40 kN pressure. As the mold is closed under applied pressure the materials are squeezed between the two halves and brought into two desired shapes within the mold. Some materials flow out of the mold in the form of thin film which is known to be “flash”. The compacted mass becomes cured and hardened to shape due to heating. After cooling the mold is given a desired shape as requirement.

Thermal and other properties of the plastic and textile materials are to be molded, widely controlled by the actual temperature and pressure. Heat and pressure are introduced in a single stroke in polymer materials and fiber for compression modeling. This is achieved on a heated plate by hydraulic press. For drying, controlled and treated PP and banana fibers (chopped) were kept in an oven at 110 °C for the duration of one hour. For mixing fiber and PP were blended for two minutes with 400 rpm at room temperature. Then small quantity of releasing agent was added to the mixture and put into a die or mold. In Paul Otto Weber press machine 50 kN initial load was applied from the top of the mold having a dimension of (6 × 6 square inches). The temperature in the electrical heater was then set to 80 °C. It only took 25-30 minutes to reach a temperature of 180 °C. It took only 25-30 minutes to reach this temperature and the system was kept at 180 °C for 20 minutes. To achieve the required thickness and also to avoid any

void a final load of up to 100 kN was applied from the top of the sample. Then the heating system was stopped and the specimen was cooled by flowing water over the outer area of the heating plate. After cooling the specimen was separated from the mold. Following ASTM procedure D-2307 compact pellet of circular shape was prepared using cork-borer at high pressure.



Fig. 1: Weber Pressen Hydraulic Press

## 2.4 Thermogravimetric analysis (TGA)

TGA traces of untreated and treated PP fiber composite samples were recorded using a thermal analyzer controlled by a computer interfaced controller. TG/DTA 6300 system along with an EXSTAR 6000 controller (Seiko Instruments Inc., Japan) which uses a horizontal system balancing mechanism to collect data. The profile of the thermal stability of the sample composite was observed at temperature 30-600 °C with 20 °C·min<sup>-1</sup> heating rate.

## 2.5 Measurement of thermal properties

Nitrogen environment was maintained to perform TGA experiment. Samples of ≤ 30 mg were calcinated up to 600 charred at a heating rate of 20 °C·min<sup>-1</sup> while nitrogen was inserted at a rate of 100 mL/min.

The programmed controlled converter is used to keep the temperature of the furnace at the required state. As a reference material, we used alumina. In TGA a measuring range was set to ≤ 30 mg and, 1500 μV. During the process for first 25% of the weight loss sample was considered for the rest of the calculations subsequently.

## 3. Results and discussion

### 3.1 Thermal behavior

Figure 2a shows the TG curves for PP, banana fiber, and PP-B composites respectively in a static nitrogen environment at a heating rate of 20 °C·min<sup>-1</sup>. While Figure 2b shows the TG curves for both treated and untreated banana fibers. In Figure 2a top line is for PP, the bottom one is for banana fiber and mid line represents the composite respectively. It is revealed from the figure TG curve for composite lies between banana fiber and PP. The weight loss of the composites is due to the loss of moisture loss as it contains only 10% fiber. The major degradation occurs in a single step for fiber and PP although there is a small degradation observed for fiber at low temperature due to moisture loss, while it occurs in two steps for composite. In the case of composite the cause of initial loss is due to fiber and the second step is due to the degradation of PP. The  $T_{5\%}$  and  $T_{max}$  represent the temperature at which 5% and maximum weight loss occur respectively are presented in Table 1 for both untreated and treated fibers and composites. Figure 2a reveals that, for both treated and untreated fibers  $T_{5\%}$  and  $T_{max}$  increase steadily with the increase of PP content. Its underlying cause is that PP has a higher  $T_{5\%}$  and  $T_{max}$  than that of the fibers. Table 2 shows that the TGA data in the first step weight losses under nitrogen environment for untreated and treated fiber, take place at 250.12-300.76 °C with 13.87% weight loss and at 280.35-311.06 °C with a 12.92% weight loss respectively. On the other hand the first step wt. losses occur at 320.01-360.36 °C with 15.63% wt. loss and at 322.14-372.74 °C with 16.04% wt. loss for untreated and treated fiber-PP composites respectively. The second

stage or major wt. loss starts at 360.12-472.45 °C with 82.402% wt. loss and at 372.14-488.02 °C with 77.20% wt. loss for untreated and treated fiber-composites respectively. The weight percentages remaining at 500 °C are 0.65% and 1.30% for untreated and treated fibers and 2.01% and 3.21% for untreated and treated PP-B composites respectively. This must be the decomposition of PP. The  $T_{max}$  values of the PP-B composites seem to be higher than those of the PP-B composites in the untreated state. Therefore, introducing treated fibers into the pp matrix leads to improvement of the composite having alkali-treated fibers are higher than those containing fiber of untreated states. Strong NaOH has been successfully used for removing lignin, hemicelluloses, and other alkali-soluble compounds from the surface of the fiber. This increases the number of reactive hydroxyl groups on the surface of the fiber and remains available for chemical bonding [2].

Figure 3a and Figure 3b represent the DTG curves for the PP and PP-B composites for untreated and treated fibers respectively. The major weight loss peak occurs at 365.15-488.45 °C for the PP-B composite. The weight loss in the first region are mostly occurred due to the moisture or loosely bound untreated elements which may not closely related to any change in structure. We may interpret the second region as the loss of non-constitutional or hydrogen evolution, low molecular hydrocarbon and may also be due to few oxygen-containing compounds. The wide peak of DTG curve carries the significance of the maximum weight loss at  $T_{max}$  [2]. In all the cases, there is slightly more char formation in the non-compatibilized composite. The underlying cause of weight loss in this region occurred due to oxidative thermal breakdown of the composite materials and expulsion of higher molecular mass hydrocarbons and CO<sub>2</sub>, CO etc. like oxygen containing compounds.

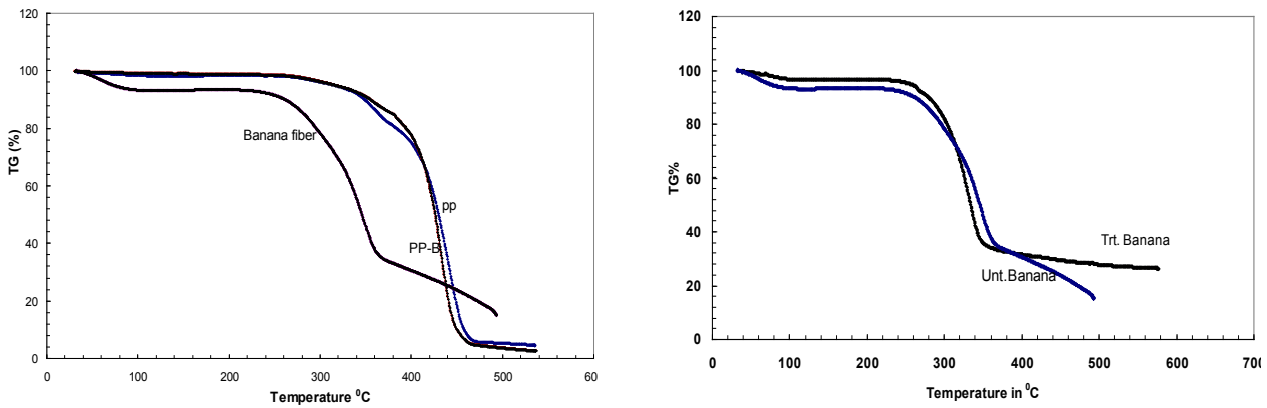


Figure 1. (a) TG profile of PP, banana fiber, and PPBC. (b) TG profile of treated and untreated banana fiber of PPBC.

Table 1. Thermal properties of banana fiber, PP and PP-B composites.

Specimen	$T_{max}^1$ (°C)	$T_{max}^2$ (°C)	$T_{5\%}$ (°C)
Untreated fiber	300.76	391.00	109.90
Treated fiber	311.06	420.50	253.91
PP-B (untreated)	360.96	472.45	318.04
PP	370.63	478.45	320.50
PP-B (treated)	372.14	488.02	338.9

**Table 2.** Weight loss of PP, untreated and Alkali treated banana fiber composites.

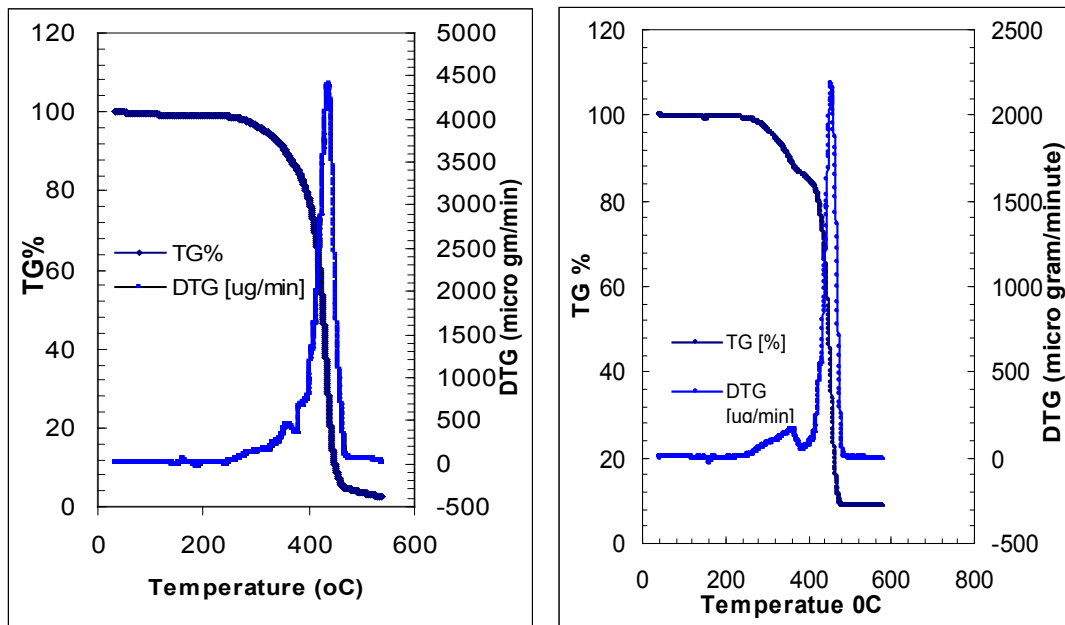
Weight % specimen	First stage		Second stage		Remaining at 500 °C
	Temperature (°C)	Weight loss (%)	Temperature (°C)	Weight loss (%)	
Untreated fiber	250.12-300.76	13.87	300.76-391.00	80.32	0.65
Treated fiber	280.35-311.06	12.92	311.06-420.50	81.26	1.30
PP	300.00-370.63	14.52	370.63-478.45	84.46	0.1
PP-B(untreated)	320.11-360.96	15.63	360.12-472.45	80.40	2.01
PP-B (treated)	322.14-372.14	16.04	372.10-488.00	77.21	3.21

It is thus seen that the oxidative thermal breakdown in the backbone chains of the PP composite occurs at higher temperatures. The DTG peak for PP-B composites and PP at around 450 °C may be due to the oxidative reaction, of corresponding TGA trace. The DTG thermo gram of treated PP-B composite in **Figure 3a and 3b** also show one small.

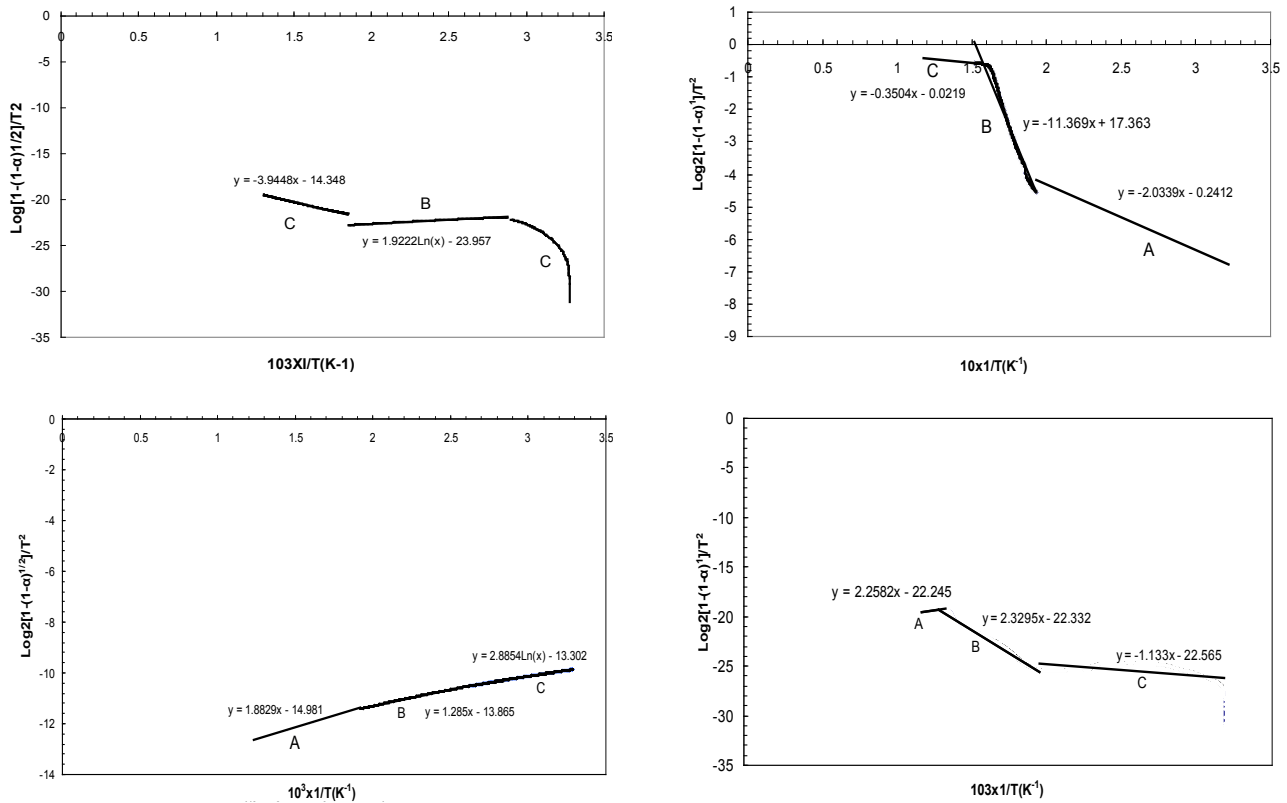
Transition/phase changes at  $(380 \pm 5)$  °C, and one big transition/phase changes with an exothermic peak at  $(460 \pm 5)$  °C. Thus, it is seen that the thermal stability of the composite remains stable up to the temperature of 500 °C. We have shown similar thermal behavior in our previous reports<sup>[11-13]</sup>.

To obtain the kinetic parameters from the TG

traces of fibers and PP-B composites differential method is used following the arguments of Sharp-Wentworth<sup>[2]</sup> considering the order of reaction 1/2 and 1. **Figure 4a-d** also represent the differential method for  $n = 1/2$  and  $n = 1$  as  $\frac{1}{T^2} \log 2 \left[ 1 - (1 - \alpha)^{\frac{1}{2}} \right]$  against  $\frac{1}{T}$  for both untreated and treated banana fiber and PP-B composites respectively. For  $n = 1/2$  this method is found less satisfactory, because the experimental points do not fit straight lines that well in all the different regions (A, B, C) of the curves rather than deviate to form a curved path in accordance with equation  $\frac{d\alpha/dT}{(1-\alpha)^n} = \frac{A}{\beta} \exp\left(\frac{-E_0}{RT}\right)$ .



**Figure 3.** TG% and DTG profile for untreated PPBC(a) and treated PPBC(b).



**Figure 4.** (a).  $\log_2 [1- (1- \alpha)^{1/2}] / T^2$  against  $10^3 \times 1/T$  graph for untreated banana fiber; (b).  $\log_2 [1- (1- \alpha)^{1/2}] / T^2$  against  $10^3 \times 1/T$  graph for treated banana fiber; (c).  $\log_2 [1- (1- \alpha)^{1/2}] / T^2$  against  $10^3 \times 1/T$  graph for untreated banana fiber; (d).  $\log_2 [1- (1- \alpha)^{1/2}] / T^2$  against  $10^3 \times 1/T$  graph for untreated banana fiber.

It is seen that for  $n = 1$ , the experimental points of TG traces for both fibers and composites have been found to fit straight lines in all the different regions (A, B, C). Thus, the reaction order of composites

may be considered as 1. The activation energies in different regions for all the methods were calculated and depicted for both the fibers and composites in **Table 3**.

**Table 3.** Temperature range, mass loss %, activation energies of fiber and PP-B composites.

Description of the materials	Temperature (K)	Mass loss %	Activation energy, $E_n$ (KJ/mole)		Remarks
			DM $n = 1/2$	DM $n = 1$	
Untreated banana fiber	305.25-523.12 Step C	$5.16 \pm 1$	0.9278	0.282	H <sub>2</sub> O and non constitutional elements evolved.
	523.12-573.76 Step B	$13.87 \pm 1$	0.036	0.0468	Low molecular mass hydro-carbons evolved. Expulsion of high.
	573.76-664.49 Step A	$80.32 \pm 1$	0.0754	0.107	molecular mass hydro-carbons and CO, CO <sub>2</sub> etc
Treated banana fiber	312.30-553.35 Step C	$4.52 \pm 1$	0.0177	0.0389	H <sub>2</sub> O and non constitutional elements evolved.
	553.35-584.06 Step B	$12.92 \pm 1$	0.2047	0.2175	Low molecular mass hydrocarbons evolved
	548.06-693.50 Step A	$81.26 \pm 1$	0.0316	0.7049	Expulsion of high molecular mass hydrocarbon CO, CO <sub>2</sub> etc.

Table 3 continued

Description of the materials	Temperature (K)	Mass loss %	Activation energy, En (KJ/mole)		Remarks
			DM n = 1/2	DM n = 1	
Untreated PP-B composite	303.22-593.11 Step C	2.21 ± 1	0.0532	0.0389	H <sub>2</sub> O and non constitutional elements evolved.
	593.11-633.96 Step B	15.63	0.2459	0.0114	Low molecular mass hydrocarbons evolved.
	633.96-745.45 Step A	80.40 ± 1	0.0554	0.0163	Expulsion of high molecular mass hydrocarbons and CO, CO <sub>2</sub> etc.
Treated PP-B composite	595.14-645.14 step C	3.42 ± 1	0.0196	0.0216	H <sub>2</sub> O and non constitutional elements evolved.
	645.14-761.02 step B	15.63 ± 1	0.1922	0.1786	Low molecular mass hydrocarbons evolved.
	310.69-595.14 Step A	77.21 ± 1	0.0435	0.0446	Expulsion of high molecular mass hydrocarbons and CO, CO <sub>2</sub> etc.

The activation energies for reaction in all the transformation modes are smaller for alkali-treated fiber and PP-fiber composites while it is compared to the untreated fiber and PP-fiber composites. This means that the alkali-treated PP-fiber composites emit more thermal energy at high-temperature regions. Thus, it can be inferred from the above discussion that PP-fiber composite stabilizes on alkali-treatment. This fact can be well understood from the relative increase in the intensity of the IR transmission band and thereby the structure stabilization of PP-fiber composites<sup>[14]</sup>.

## 4. Conclusions

Short banana fibers reinforced in PP composites were prepared by a melt mixing process which is followed by the hot press compression molding. The role of NaOH in PPBCs is very significant in improving the adhesion of banana fiber with polypropylene matrix<sup>[15,16]</sup>.

It is observed from the thermo gravimetric measurements that the addition of banana fibers into PP causes an increase in the decomposition and the thermal stability of composites in the case of treated fibers then seems to improve while it is compared to the untreated fiber. It is also seen that the value of  $T_{5\%}$  and  $T_{max}$  are higher for the composites containing treated fibers, than the composites containing untreated fiber. This phenomenon carries the informa-

tion that the PPB is more hydrophilic than PP.

We may conclude from the greater overall thermal stability of PPB treated composites over untreated PPBC. We can see from the reference<sup>[14]</sup> that the FTIR report suggests no structural changes due to the presence of lignin in the alkali treated banana fibers. Therefore the addition of such treated fibers into the pp matrix leads to the improvement of the thermal stability of the composites. So we may also conclude that the composites remain thermally stable up to the temperature 500 °C.

Weight loss takes place due to oxidative thermal breakdown of the composite structure and expulsion of higher molecular mass hydrocarbons, and the compounds containing oxygen like CO<sub>2</sub>, CO, etc. The oxidative thermal breakdown in the backbone chains of the PP composite is therefore found to occur at higher temperatures. Moreover the initial low tempts. weight loss occurs due to the removal of the solvent polymer matrix. The major weight loss occurs at higher tempt. Due to degradation and volatization of PP along with fiber present in the composite.

In TGA curve the weight loss of the composites is due to the loss of moisture loss as it contains only 10% fiber. The major degradation occurs in a single step for fiber and PP although there is a small degradation observed for fiber at low temperature due to moisture loss, while it occurs in two-step for composite. In the case of composite the cause of initial loss is due to fiber and the second step is due to the



degradation of PP.

The DTG peak near the temperature 500 °C may be due to the oxidative reaction, of the corresponding TGA curve. The weight percentages remaining at 500 °C are 0.65% and 1.25% for untreated and treated fibers and 0.06% and 1.25% for untreated and treated PP-B composites respectively. This must be the decomposition of PP. The  $T_{max}$  values of the PP-B composites seem to be higher than those of the PP-B composites in the untreated state. Therefore, introducing treated fibers into the pp matrix leads to improvement of the composite having alkali-treated fibers are higher than those containing fiber of untreated states.

The activation energies for reaction in all the transformation modes are greater than is being obtained for alkali-treated fiber and PP-fiber composites when they are compared with the untreated fiber and PP-fiber composites. The alkali-treated PP-fiber composites emit more thermal energy and stabilize at high-temperature regions.

PPBC also poses better thermal stability and excellent moisture properties. The environmentally friendly behavior of PPBC makes it very popular in various engineering applications like the automotive and construction industry. Decomposition and thermal stability rates are lower in untreated PPBC in comparison with treated PPBC.

## Author Contributions

Most of the experiential works are performed under the guidance of M. A. Gafur in BSIR Lab. Experiments and analysis of the data manuscript are carried out and prepared by Md. Nazrul Islam.

## Conflict of Interest

There is neither a conflict of interest nor any financial obligation from any funding agency work.

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