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*Research Article*

# Physical and Thermal Characterization of Alkali Treated Rice Husk Reinforced Polypropylene Composites

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Rice husk (RH) reinforced polypropylene- (PP-) based composites were prepared by compression molding. The RH was treated with sodium hydroxide solution (1 wt%); then composites were prepared using varying percentages of RH (5 to 20 wt%). The thermomechanical, spectroscopic, and morphological properties of the prepared composites (RH-PP) were investigated. The scanning electron microscopic (SEM) analysis showed better interfacial adhesion between alkali treated RH and the matrix PP. The Fourier Transform Infrared (FTIR) spectra confirmed the chemical bonding.The results revealed that physical properties as well as thermal stability of the composites improved significantly with the addition of alkali treated RH in PP matrix.

## 1. Introduction

Composite materials are manufactured by combining two dissimilar materials into a new material which may be better suited for a particular application than either of the original materials alone. Nowadays, modern technologies require materials with unusual combination of properties that cannot be met by the conventional materials. A relatively new class of composite material is fiber reinforced polymer manufactured from fiber and polymer matrix. It is efficient and economical for use in a variety of engineering applications. Thermoplastic or thermoset matrix material can be used in the fiber reinforced polymer-based composites. Fibers of various types can be used as reinforcing agent. Thermoplastic resins achieved much interest due to their economic and mechanical advantages, such as easy fabrication, intrinsic recyclability, unlimited shelf life, high toughness, and increased moisture resistance. Fibers provide stiffness and strength to the composites and improve their mechanical characteristics [1–4].

Every year billions of tons of agricultural crop residues are produced around the world. Among this large amount of residues, only a small fraction of the residues are utilized as household fuel or fertilizer and the rest is burned in the field. As a result, it causes air pollution. Utilization of the agricultural crop residues as reinforcement of polymer composites can help to protect the environmental problem [5, 6]. Application of natural fibers as reinforcing agent to composites gained much interests due to increased environmental awareness and consciousness throughout the world. Natural fibers are now considered as an alternative to synthetic fibers for use in various fields such as building and construction, packaging, automobile and railway coach interiors and storage devices, and various load bearing applications [7, 8]. Natural fiber reinforced composites are environmentally friendly, biodegradable, widely available, renewable, and cheap and have low density. The biodegradability of plant fibers contributes to a healthy ecosystem and their high performance satisfies industrial economy. Plant fibers are light in weight compared to glass, carbon, and aramid fibers. A wide variety natural fibers have been investigated for the reinforcement of the composites. These include jute straw, wood, rice husk, banana fiber, sisal, hemp, ramie, oil palm empty fruit bunch, barley, oats, rye, bamboo, grass, reeds, papyrus, kapok, coir, paper mulberry, pineapple leaf fiber hemp, flax, kenaf, and wheat [9–12].

Composite materials manufactured using unmodified plant fibers often exhibit unsatisfactory properties. To overcome this, surface treatments are required prior to composite fabrication. The properties of fiber reinforced polymer composites can be improved by both physical (corona treatment, cold plasma treatment) and chemical treatments (isocyanates, sodium hydroxide, maleic anhydride organosilanes, permanganate, and peroxides) [13, 14].

Yang et al. [15] prepared rice husk flour filled PP-based composites and measured mechanical and morphological properties. Rice husk content varied from 10 to 40 wt%. It was found that tensile strengths of the composites slightly decreased as the filler loading increased but tensile modulus improved with increasing filler loading. Notched and unnotched Izod impact strengths were lowered by the addition of rice husk flour. Similarly, Arjmandi et al. [16] reported that Young's modulus and flexural modulus increased, whereas yield strength and elongation at break decreased with the increasing in rice husk filler contents for PP-based composites. They investigated interfacial properties by SEM studies and reported some voids inside the composites and mentioned poor interaction between RH and PP. The reason behind the poor interaction was the polar nature of RH and nonpolar nature of the PP matrix. By increasing the RH content, a significant filler aggregation appeared, which contributed to the poor stress transfer from matrix to filler resulting in poor properties. On the other hand, Ndazi et al. [17] reported chemical and thermal stability of RH against alkali treatment with 2–8 w/v sodium hydroxide. They reported a significant decrease of lignin and hemicellulose from the RH but thermal stability decreased. In this investigation RH was selected because this is the by-product of rice hulling industry in Bangladesh and is not used yet. The RH was collected from local market, so from this investigation it will give a clear information about the quality of RH collected from Bangladesh. Moreover, RH was treated with sodium hydroxide solution and then neutralized by hydrochloric acid. No researches yet have been published on the effect of alkali treatment on the RH reinforced PP-based composites.

The aim of this investigation was to fabricate rice husk (*Oryza sativa*) reinforced PP-based composites by compression molding. The RH was treated with sodium hydroxide solution and then composites were prepared using varying percentages of alkali treated RH. Physical, thermal, spectroscopic, and morphological properties of the composites (RH-PP) were investigated and compared with the control composites to find out the effect of alkali treatment on RH and on the composites.

Table 1: Percentages of polypropylene and rice husk in composites of different compositions.

| Samples of composites | Composition (wt.%) | |
|---|---|---|
| | Polypropylene | Rice husk |
| $S_1$ | 100 | 0 |
| $S_2$ | 95 | 5 |
| $S_3$ | 90 | 10 |
| $S_4$ | 85 | 15 |
| $S_5$ | 80 | 20 |

## 2. Experimental Methods

*2.1. Materials and Chemicals.* Rice husk (local name: Aman rice husk; scientific name: *Oryza sativa*) was collected from local rice mill of Dhaka, Bangladesh. Polypropylene (PP) in the form of pellets was purchased from the Polyolefin Company Private Limited (Singapore). Sodium hydroxide and hydrochloric acid were purchased from BDH Limited (UK). The sodium hydroxide solution (1 wt%) was used for treating the rice husk to improve the bonding at the filler-matrix interface by removing the hemicelluloses, lignin, and other impurities present in rice husk [18, 19]. Hydrochloric acid (0.1 M solution) was used to neutralize the alkaline solution.

*2.2. Experimental Section.* Composites of different compositions (Table 1) were prepared following the standard procedure [20]. Rice husk was washed thoroughly to remove dust, soil, sand, and rice particles; then it was dried and sieved. After that, rice husk was treated by 1 wt% NaOH solution at 25°C for 72 h, maintaining a liquor ratio of 15 : 1. The reaction of sodium hydroxide with cellulose is as follows:

$$\text{Cell–OH} + \text{NaOH} \longrightarrow \text{Cell–O–Na} + \text{H}_2\text{O} + \cdots \qquad (1)$$

The solution was neutralized using 0.1 M HCl solution, and then rice husk was taken out from the solution and washed. Finally, the treated rice husk was dried in a vacuum oven at about 85°C for 24 h to remove moisture. Before the preparation of composites, rice husk was grinded using planetary ball mill (model PM 100, China) for 3 h at 300 rpm and then screened using vibratory sieving machine to get 45–70 mesh (equivalent sieve size 0.355 to 0.212 mm) size of rice husk flour. Then all components were simultaneously blended in a Haake PolyLab mixer at stir rate of 80 rpm and temperature 180°C for 5 min. Then the mixture was immediately pressed between two plates of a compression molding at 170°C and 5 kPa pressure for 8 min to prepare composite layer having size 15 cm length × 15 cm width × 3 mm thickness. The digital images of rice husk, alkali treated rice husk, and rice husk reinforced PP-based composites (RH-PP) are shown in Figure 1.

*2.3. Characterization.* Scanning electron microscopy (JEOL JSM-7600F, Japan) was used to observe the morphology of fracture surface of PP sheet and alkali treated husk reinforced composite. The FTIR spectroscopy of the PP and composite

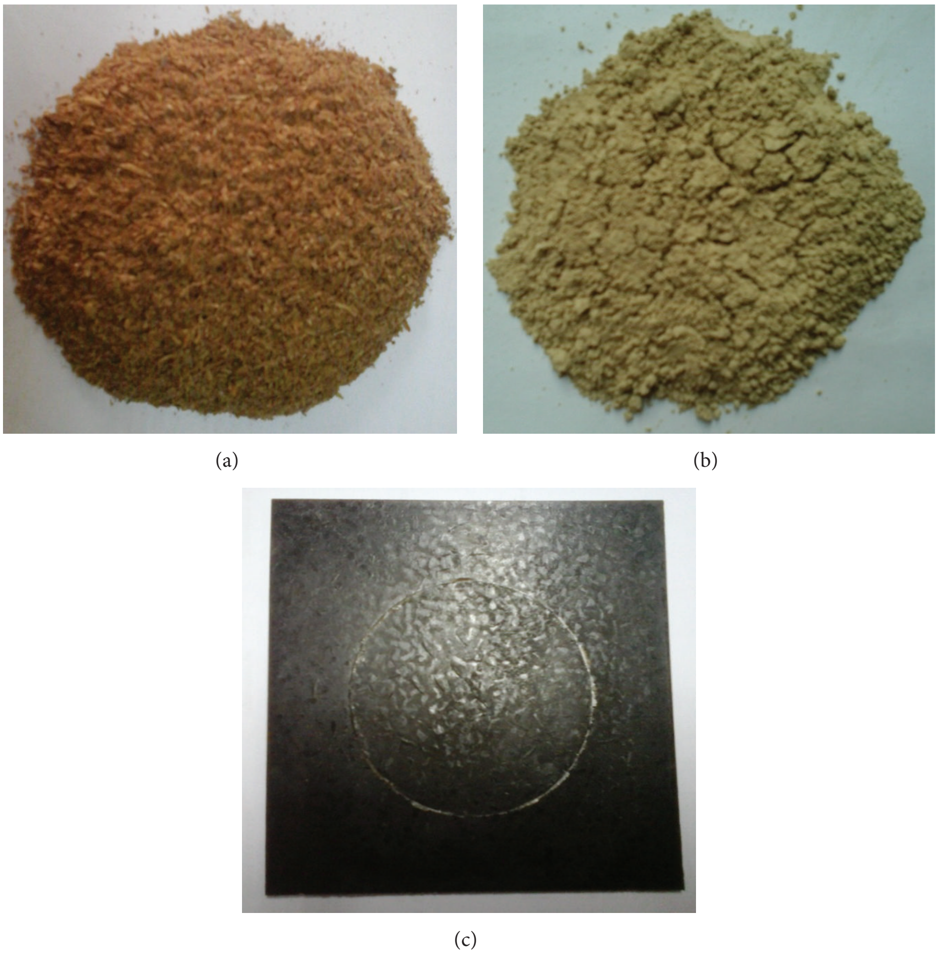


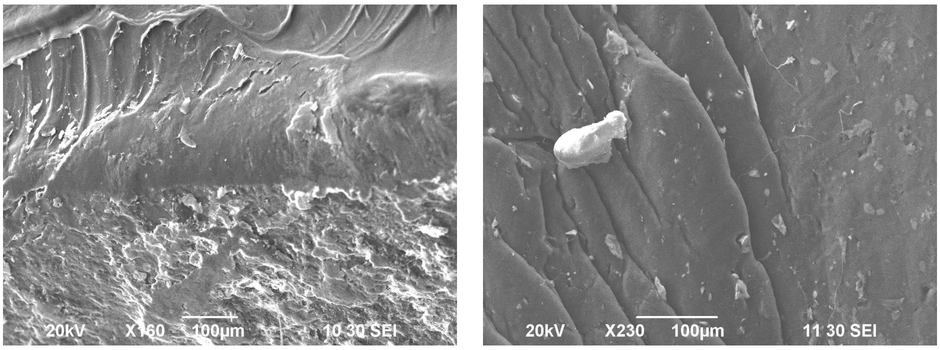
Figure 1: Digital images of (a) rice husk, (b) alkali treated grinded rick husk, and (c) rice husk reinforced (20 wt%) polypropylene-based composite.

was taken in the region between 500 and 4000 $\text{cm}^{-1}$ (with PerkinElmer 1650, USA) in order to understand the chemical structure of the samples. To measure water absorption properties, the specimens were prepared according to the ASTM method and then weighted by a digital balance to a precision of ±0.1%. Then the dried specimens were fully immersed into distilled water for 24 to 72 h at room temperature. Then samples were periodically taken out from water and wiped off with tissue paper. The samples were then reweighted. To measure bulk density, samples were prepared according to the ASTM C135 [1] and weighted on a balance to a precision of ±0.1%. Thermal stability of the samples was characterized by using a thermogravimetric analyzer TGA Q500 (TA Instruments co., USA). The samples were kept under nitrogen atmosphere using the temperature range of 25–600°C and heating rate of 10°C/min.

## 3. Results and Discussion

*3.1. Scanning Electron Microscopic (SEM) Analysis.* The SEM analysis was carried out to investigate the interfacial adhesion between rice husk and PP matrix of the composites. The fracture surfaces of neat PP sheet (a) and alkali treated rice husk (20 wt%) reinforced composite (b) are shown in Figure 2. It was found that the fracture surface of neat PP is rough which indicated the typical characteristic of elastic behavior of PP [21]. On the other hand, the interface of the alkali treated rice husk composite showed fiber pull-out characteristic and revealed the weak bonding between hydrophobic polymer matrix PP and the hydrophilic nature of rice husk filler. Few cracks are observed in the interface of the composite. Similar images are reported elsewhere in the literature [21, 22].

*3.2. Fourier Transform Infrared (FTIR) Spectroscopic Analysis.* The FTIR spectra of neat PP and rice husk reinforced PP-based composites are shown in Figures 3(a) and 3(b). The neat PP (Figure 3(a)) showed peaks ranging from 2850 to 3000 $\text{cm}^{-1}$ and 1454.33 $\text{cm}^{-1}$ corresponding to $CH_2$ or $CH_3$ vibrations. Bands in area 600–1300 $\text{cm}^{-1}$ were caused by the domains of regularity in PP matrix. The band located at 1029.99, 1149.57, and 1211.30 $\text{cm}^{-1}$ may be due to stretching of $CH{-}CH_2$ and vibration of the rocking of $-CH_3$ and $-CH_2$ and due to the isotactic band, respectively [1, 21–24].



(a) (b)

Figure 2: SEM images of the fracture surface of (a) neat PP sheet and (b) alkali treated rice husk (20 wt%) reinforced polypropylene-based composite.

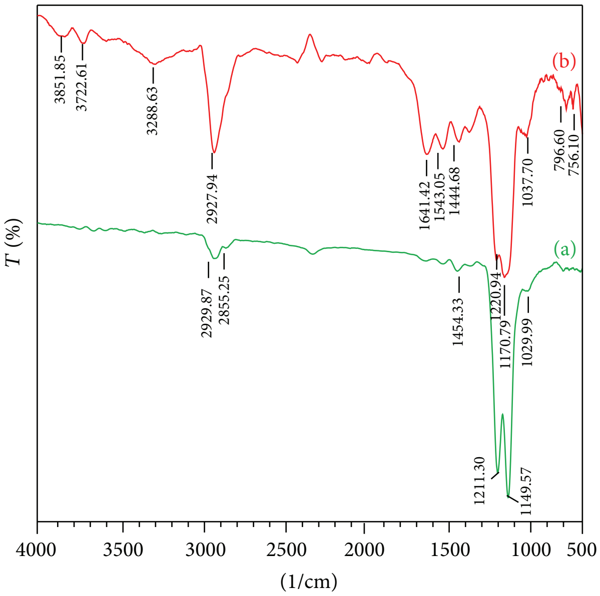


Figure 3: FTIR spectra of (a) neat polypropylene and (b) alkali treated rice husk (5 wt%) reinforced polypropylene-based composite.

Table 2: FTIR data for pure polypropylene and alkali treated 5 wt% RH-PP composite.

| Wavenumber, $cm^{-1}$ (PP) | Functional groups |
|---|---|
| 2929.87 (moderate) | $CH_2$ asymmetric stretching |
| 2855.25 (weak) | $CH_2$ symmetric stretching |
| 1454.33 (moderate) | $CH_3$ asymmetric stretching |
| 1211.30 (strong) | Isotactic band |
| 1149.57 (strong) | Vibration of the rocking of $-CH_3$ & $-CH_2$ |
| 1029.99 (weak) | Stretching of $CH-CH_2$ |
| **Wavenumber, $cm^{-1}$ (composite)** | **Functional groups** |
| 3200–3800 (moderate) | OH stretching of cellulose and water molecule |
| 2850–3000 (strong) | CH, $CH_2$, or $CH_3$ stretching |
| 1600–1700 (moderate) | C=C group |
| 1475–1600 (weak) | C=C stretching of aromatic rings from lignin |
| 1375–1450 (weak) | C–O–H bending of ester |
| 1000–1300 (strong) | C–O stretching |
| 690–900 (moderate) | C–H bending |

The FTIR spectrum of 5 wt% RH-PP composite (Figure 3(b)) shows the presence of the characteristic band of the C=C group in the region of 1600–1700 $cm^{-1}$ and C–O stretching in the region of 1000–1300 $cm^{-1}$ as the composite contains alkali treated rice husk as filler. Absence of carbonyl (C=O) peak (1740–1720 $cm^{-1}$) which represents hemicelluloses is the evidence that hemicelluloses are removed from rice husk surface during the alkali treatment. Peaks observed at 690–900 $cm^{-1}$ are due to C–H bending. Weak peaks for C–O–H bending due to presence of ester and C=C stretching due to presence of aromatic rings of lignin appear between 1375 and 1600 $cm^{-1}$ [25, 26]. Bands from 3200 to 3800 $cm^{-1}$ are due to the chemically absorbed water and also due to the stretching vibration of hydroxyl groups present in the composite containing cellulose [27, 28]. A strong absorbance peak around 2850–3000 $cm^{-1}$ is associated with the $-CH$, $-CH_2$, or $-CH_3$ stretching [29]. In Table 2, the FTIR data for PP and alkali treated RH-PP composite is presented.

### 3.3. *Water Absorption.*

The percentage of water absorption in the composites can be calculated by weight difference between the samples immersed in water and the dry samples using the following equation:

$$M_t = \left( \frac{[W_t - W_o]}{W_o} \right) \times 100, \qquad (2)$$

where $M_t$ is the moisture content in the specimen, $W_t$ is the weight of the specimen at the immersion time, and $W_o$ is the weight of the specimen before the water absorption test. Variation of water absorption with different weight percentages of rice husk reinforced PP matrix is shown in

Table 3: Variation of water absorption with different weight percentages of rice husk reinforced in polypropylene matrix.

| Samples | % of water absorption | | | |
|---|---|---|---|---|
| | 0 hr | 24 hr | 48 hr | 72 hr |
| PP | 0 | 0.102 | 0.184 | 0.195 |
| 5% rice husk PP | 0 | 0.213 | 0.245 | 0.287 |
| 10% rice husk PP | 0 | 0.256 | 0.293 | 0.336 |
| 15% rice husk PP | 0 | 0.297 | 0.365 | 0.406 |
| 20% rice husk PP | 0 | 0.323 | 0.382 | 0.433 |

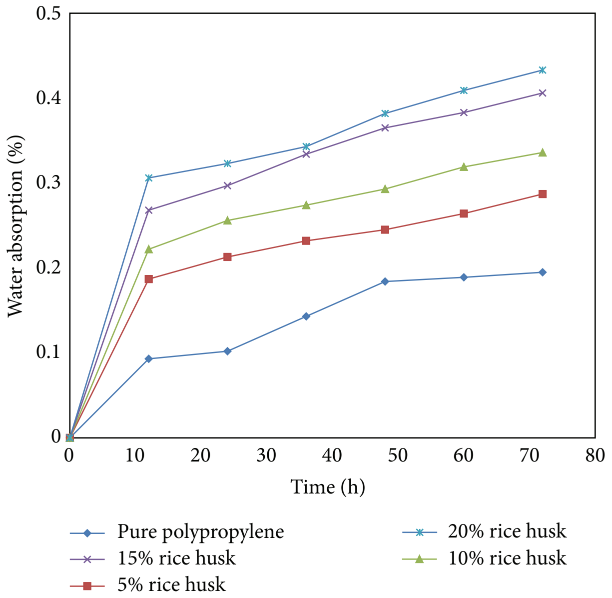


Figure 4: Percentage of water absorption of rice husk reinforced polypropylene-based composites.

Table 3. It is clear that the water absorption depends on rice husk content and immersion time. Figure 4 shows the effect of immersion time on water absorption of different percentage of treated RH-PP composites. Result shows that the water absorption increased with increasing rice husk content and immersion time [30]. The highest value of water absorption is observed for 20% treated RH-PP composite and water absorption increases with the increase of percentage of treated husk addition. The increasing water absorption is due to the hydrophilic nature of rice husk filler and the increased interfacial area between the fiber and the matrix. The amount of water uptake by polymer matrix is almost negligible as it is hydrophobic in nature [31]. The cellulosic effect and the micro void formation in the matrix may be responsible for the increase of water absorption with immersion time [32].

Table 4: Variation of density with different weight percentages of rice husk reinforced in polypropylene matrix.

| Samples | Bulk density, gm/cc |
|---|---|
| Neat PP | 0.750 |
| 5% rice husk PP | 0.954 |
| 10% rice husk PP | 0.929 |
| 15% rice husk PP | 0.883 |
| 20% rice husk PP | 0.857 |

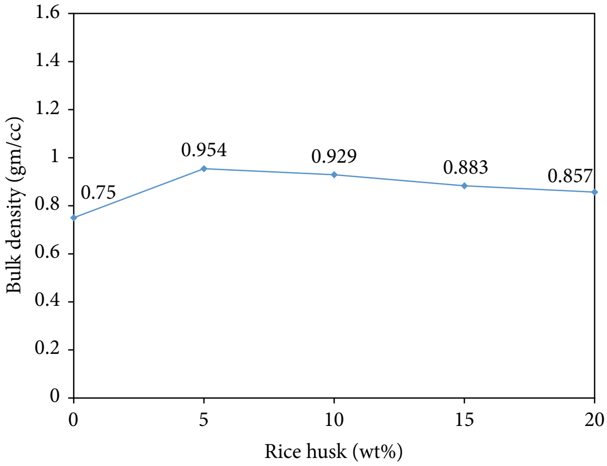


Figure 5: Bulk density of rice husk reinforced polypropylene-based composites.

*3.4. Bulk Density.* The mass density or density of a material is defined as its mass per unit volume. In some cases (e.g., in the United States, oil and gas industry), density is also defined as its weight per unit volume, although, this quantity is more properly called specific weight:

$$D = \frac{W_s}{V}, \tag{3}$$

where $D$ is the density, $W_s$ the weight, and $V$ the volume of the specimen.

Bulk densities of neat PP and RH-PP composites of different compositions are shown in Table 4. The effect of variation of weight percent of rice husk filler on the bulk density of rice husk reinforced PP composites is plotted in Figure 5. The bulk density of neat PP was found to be 0.75 g/cm$^3$. A monotonous increase of bulk density was observed with the increase of rice husk of the composite. The highest value of bulk density was observed for 5 wt% RH-PP composite. Higher amount of rice husk (20 wt%) created a lot of pores inside the matrix PP and thus decreased the density. Since the volume fraction of voids is increased with the addition of rice husk, the bulk density decreased [33, 34].

*3.5. Thermal Stability.* Figure 6 shows the thermograms of neat PP, composites at different compositions, and alkali treated rice husk filler. A sudden drop in the mass of the sample indicates the thermal degradation of the materials. It is evident from the figure that weight loss of neat PP and 5%, 10%, and 15% RH-PP composites occur at one-step degradation process. On the other hand, 20% RH-PP

Table 5: TGA data of neat polypropylene, RH-PP composites at different compositions, and alkali treated rice husk filler.

| Samples | Degradation temperature °C | | Final decomposition temperature °C |
|---|---|---|---|
| | 1st stage | 2nd stage | |
| Neat PP | 449.95 | — | 503.25 |
| 20% rice husk PP | 241.50 | — | 470.44 |
| 15% rice husk PP | 243.27 | — | 479.97 |
| 10% rice husk PP | 252.06 | — | 485.83 |
| 5% rice husk PP | 273.64 | 377.88 | 501.60 |
| Rice husk | 151.80 | 308.50 | 387.90 |

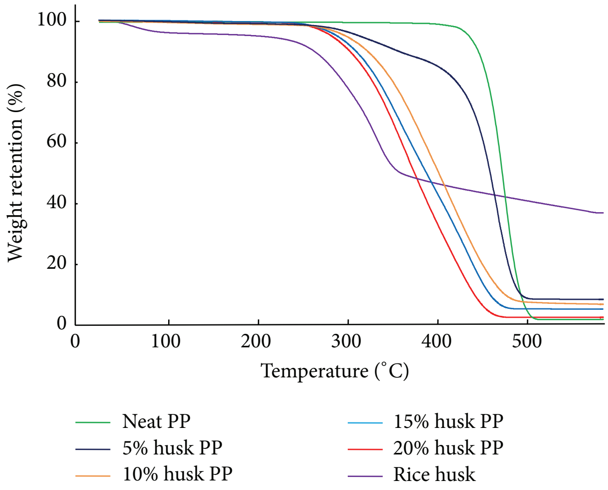


Figure 6: Thermograms of polypropylene and rice husk reinforced polypropylene-based composites.

composite and rice husk degraded in two steps. In case of neat PP, the weight loss started at 449.95°C and reached maximum value at 503.25°C [35]. The TGA data of neat PP, RH-PP composites at different compositions, and alkali treated rice husk filler is given in Table 5. It was observed that, with increase of rice husk content in the composites, there was a slight decrease of the onset degradation temperature. This indicates that thermal stability of the composites decreases with the increase of rice husk content. Therefore, increase in rice husk content in composite plays an important role in controlling its rate of thermal degradation [36].

## 4. Conclusions

The results of this research showed that rice (*Oryza sativa*) husk is a promising renewable resource to be applied for polymer composites as a filler. Alkali treated rice husk reinforced polypropylene (PP) matrix composites were successfully prepared by compression molding. The SEM images indicated that alkali treated rick husk had better interfacial bonding between filler and matrix than their untreated counter parts. Chemical composition of the composites was confirmed by the FTIR studies. Water absorption capacity increased with the increase of immersion time of the composite samples indicated the hydrophilic character of the composite due to the addition of rice husk. The bulk density of fabricated composites decreased with increase of rice husk addition. It was also observed that the thermal stability of composite was significantly improved due to incorporation of rice husk in the polypropylene.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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