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Development and Characterization of Polyethylene Terephthalate-rice Husk (PET-RH) Composites: Thermal Stability, Morphological Analysis, and Burning Rate Evaluation

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

A polyethylene terephthalate – Rice husk (PET-RH) composite was produced from waste plastic water bottle and rice husk. Burning rate test, scanning electron microscope (SEM), thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were employed to study

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the produced composite. The burning rate carried out on the composite revealed resistance to burning as result of the introduction of more PET material into the composite at varied proportions, it also revealed that to produce a more durable PET-RH composite, the ratio of PET to RH must be at optimum concentration to achieve a desired application. The SEM analysis revealed a well-structured morphology of the produced composite as there were less areas of fracture or unfilled fiber regions that could cause breakdown or fissure in the matrix of the composite. The TGA analysis of the produced composite revealed thermal stability of the PET-RH composite within the range 0 to 300 ℃ with a 20 % weight residue recorded in the temperature range 500 to 887 °C. The DTA analysis revealed and correlated with the results obtained from the TGA thermogram, whose initial drying was due to a 4 % loss in mass and a narrow endotherm which was visible at the 240 and 545 °C temperature range which corresponded to the pyrolytic stage visible in the TGA thermogram, this clearly suggested that the produced composite can serve in applications dependent on thermal resistant properties. Results so far obtained from this study suggested that the produced PET-RH composites exhibited good performance characteristics that could be likened to properties of conventional materials with the same matrix.

Keywords: PET; TGA; rice husk; composite; fibre; reinforcement.

1. INTRODUCTION

The exceptional strength and stiffness of fiber reinforced polymer composites (FRC) combined with their light weight have made them widely used in a variety of applications such as flexural strengthening of reinforced concrete (RC) beams owing to its observed high tensile strength, reduced density and installation ease, the use of polyethylene terephthalate (PET) fibres was also proposed to be incorporated to concrete so as to enhance their been ductile [1,2,3,4]. The use of FRC bars have graduated to being a popular alternative to conventional steel reinforcement. Various modifications of FRC, such as sheets, strips, bars, grids, and ropes are used as construction components, due to their resistance to corrosion and enhanced durability making them a potential option for use [5,6]. Fiber and matrix are the two components that make up FRC. Fibers can be derived from natural resources, such as plants, or they can be produced artificially. When compared to natural fiber reinforced composites (NFC), synthetic fiber reinforced composites (SFC) exhibit superior mechanical qualities [7]. However, the government and researchers are concerned about the limitations of synthetic fibers because of their non-biodegradability, which requires disposal, recycling, and environmental effect that causes significant contamination to the surrounding ecosystem [3]. Therefore, the quantity of synthetic fibers used in FRC must be reduced or replaced entirely with natural fibers, depending on the intended use and strength requirements. Natural fibers including curaua, jute, flax, banana, kenaf, sisal, and coconut husk are seen to be appealing replacements for synthetic fibers [3,7], such as in several consumer profit formulation schemes and the automobile and construction industries [8]. Natural fibers are advantageous for use as fillers or reinforcement in polymer composites because of their low density, sustainability, abundant supply, non-abrasive nature, and lack of toxicity [3,9].

Good dispersion of fibers in the matrix facilitates good bonding, which improves the mechanical properties of composites. The length of the fibers (long, short), as well as processing variables like temperature and pressure, generally affect the dispersion of the fibers [10].

The bonding within the fiber and matrix has a significant impact on the mechanical attributes of the composite materials. When there is strong interfacial bonding, enhanced polymer composites transfer the applied stress to the fibers [9]. Because the fiber is hydrophilic, and the resin is hydrophobic, plant-based fiber typically has poor bonding capabilities [9]. Mechanical interlocking, chemical bonding, electrostatic bonding, inter-diffusion linking, coupling agent, and chemical pre-treatment are some methods that could be used to address this issue [10].

Rice husks are among the typical agricultural residues, which are easily available in huge amounts [11,12]. The major hindrance in rice husks utilization for composite manufacture lies in the lack of direct interaction with most adhesive binders to form the anticipated interfacial bonds [13]. Rice is one of the most consumed crops worldwide, with an annual

production of 700 million tons according to the Food and Agriculture Organization of the United Nations database [13]. The by-products of rice harvest, namely rice straw and rice husk, pose environmental and economic challenges for farmers. Rice straw is often disposed of through uncontrolled burning, leading to air and wetland pollution [14]. Alternatively, rice waste is left to decompose in fields, resulting in the death of aquatic life in deeper water bodies [8]. RH has been effectively combined with diverse polymeric matrices such as Polyethylene (PE) Polypropylene (PP) Polyvinyl Chloride (PVC), and Polylactic acid (PLA) to create these composite materials [14].

PET (polyethylene terephthalate) is made up of polymerized repeating units of the ethylene terephthalate monomer (C10H8O4). It is a recyclable plastic with the number 1 as its identification code [15]. PET, which has a molecular weight of 192 gm/mole and contains 62.5 percent carbon, 33.3 percent oxygen, and 4.2 percent hydrogen, is utilized as synthetic fiber, polyester, plastic packaging, and soft drink containers all over the world [16]. The incineration of PET transforms into a rigid and complex material, posing significant environmental concerns. Furthermore, the incineration process releases toxic greenhouse gases, exacerbating environmental issues [17]. Agricultural waste-derived natural fibers are gaining significance within the polymer sector due to their numerous benefits, including their lightweight nature, cost-effectiveness, and eco-friendliness. Rice husk (RH) is a natural covering that envelops rice kernels during their development [3]. As a variant of natural fiber sourced from agricultural byproducts, RH holds potential as a filler in composite materials across different polymer matrices [14]. In this investigation, RH-PET composite was formulated, its burning rate, SEM morphology and TGA/DTA thermal analysis were conducted to ascertain for their heat resistance, thermal decomposition temperatures and effects of the filler (RH) in the PET matrix observable from the SEM micrograph.

2. MATERIALS AND METHODS

2.1 Materials

Waste plastic table water bottles were collected by hand within the vicinity of Gombe State University, rice husk was collected from a local rice mill in Gombe, Nigeria while, wood fibers were collected at a nearest carpentry workshop in Gombe metropolis. Beakers, measuring cylinder, top loader balance (HRB-S-3002 Model), mold, reagent bottles, distilled water, desiccator, BR Biochem hot plate (BIMS-005). Analytical graded 1,1,2,2-tetrachloroethane solution (Aladdin scientific) and phenol (Prasol Chemicals Pvt).

2.2 Methods

Waste plastic water bottles used are made from polyethene terephthalate (PET). The samples were washed thoroughly and then cut into small pieces to ease its dissolution in the solvent. The impurities such as fine sand grains accompanying the rice husk were removed by washing the rice husk thoroughly with distilled water and subsequently dried. The rice husk was then grounded and stored in clean glass containers for future use. Similar treatment as that of the rice husk was performed on the wood filling.

The solvent used, a mixture of phenol crystals and 1,1,2,2-tetrachloroethane was prepared by melting the crystals at 45°C which was followed by the addition of 1,1,2,2-tetrachloroethane in the ratio 3:2 w/w [16,18]. The cut waste PET plastics were dissolved by measuring 5, 10, and 15g and dissolving in a 100 ml of the dissolution solvent which was placed on a BR Biochem hot plate (BIMS-005) at 100 °C and stirred to form a viscous liquid [16], this was followed by the addition of a constant 5g of the rice husk in a 250 ml beaker to produce the PET: RH composites. The mixture was then transferred while hot into different labelled molds and allowed to cool [11,14].

2.2.1 Burning rate determination

The method enumerated by John et al. [16] and Balan et al. [19] were adopted with some modifications; the PET: RH samples were cut into 20 ×10 × 5 mm dimensions. This was followed by measuring the initial length of the cut pieces while been held with a crucible tong was ignited, a constant time of 30 seconds was taken as the flame propagation time (Fp), finally, the burnt zone length was measured and the difference between the initial and the burnt zone lengths was taken as the propagation distance (Dp) through the PET: RH composite. The burning rate was determined using equation 1:

$$Br = Dp/Fp \quad \text{-- Equation (1)}$$

Where:

Br is the burning rate in mm/s.
Dp is the propagation distance measured in mm.
Fp is the flame propagation time in seconds.

2.2.2 Morphological study of the PET: RH composite

The structural morphology of the composite was investigated using a JOEL JSM 5600 LV model Scanning Electron Microscope (SEM) with an accelerating voltage of 5-18 keV which was conducted at the National Research Institute for Chemical Technology, Zaria, Nigeria [18,20]. Carbon tape that was vacuum-compatible, conductive, and double-sided was used to mount the samples to holders. Images were taken using an electron beam voltage of 10 kV and in a low vacuum (50-60 Pa). To capture images, the secondary electron mode of the low vacuum detector (LVC) was used [21].

3. RESULTS AND DISCUSSION

3.1 Burning Rate Test

The capacity of a material to ignite and burn because of a fire or due to combustion is measured by a burning test. Fig. 1 illustrates how a percentage increase in the fiber content affects the burning rate at constant PET and Fig. 2 depicts how a rise in PET (%) affects the composite's burning rate while maintaining a constant amount of rice husk (fiber). It can be observed from Fig. 1 that the rate at which the produced composite burns in air is directly proportional to the amount of the RH added at constant PET amount. The burning rate increases as the composite ratios were increased. This can serve as a measure to ascertain and optimize the composition for a given application. When compared to Fig. 2, it was observed that keeping the ratio of RH constant and increasing PET led to an even higher burning rate for the composite, this can be attributed to the higher heating value of PET been 22.93 MJ/Kg [16], it has also been suggested that the presence of moisture could have caused the increase in the observed higher burning rate values obtained for PET: RH composite [8]. Overall, the increase in PET in the composite thus enhances durability and strength of the composite while been more resistant to burning due to higher PET composition as observed from Figs. 1 and 2 respectively.

3.2 Thermogravimetric Analysis of PET: RH Composite

Fig. 3 shows that the PET and RH underwent thermal degradation in three stages: drying, pyrolysis, and carbonization (decomposition to char) according to [22,23]. About 4 % of their initial weights were lost in the first stage as shown which could be attributed to initial moisture removal and possible volatile chemical species emissions, it was also observed that the PET-RH sample was thermally stable in the temperature range 0-300℃, which is considerably high enough resistant temperatures when compared to other literatures [20,22,23]. The second stage of pyrolysis revealed that the thermal degradation of the PET and RICE HUSK began at around 300 °C and spread over a wide temperature range between 300 to 490 °C, these temperature ranges are similar to findings by Kalu, [22] for Seamum indicum seed oil modified alkyd resin (SISOMAR) whose initial percentage weight loss and pyrolysis temperature range at 4 % were 300 to 500 °C respectively. This suggests that PET composite had higher initial degradation temperature than the RH and could offer better resistance to high temperature conditions unlike the SISOMAR. It was also observed that there were some multistep degradations evident in the PET curve which could be attributed to high heating rate or instability of the intermediate formed [20,22,23]. This also suggests that the SISOMAR underwent an extended period of pyrolysis due to the polymer matrix composition which must be fully overcome to initiate total decomposition to char. It has also been suggested that certain chemical species could have been eliminated in this stage and identified by employing a TG-MS couple to study their fragmentation processes and degradations [22,23]. It was also observed that the temperature at 50 % weight losses T50 and percentage residue was at < 320 °C for the PET: RH composite while, the % residue left after the destructive process was in the range 500 to 887 °C and < 20 % was recorded. Similar result was recorded for SISOMAR whose T50 is ≤ 440 °C and % residue is < 20 % at 500 °C [22]. It has been suggested by Kalu [22] and Yebra [24] that the temperature at 50 % weight loss is a common indicator for measuring the thermal stability of chemical species, which suggest that the PET and RH could meet certain temperature resistance requirements depending on the application it could be employed for.

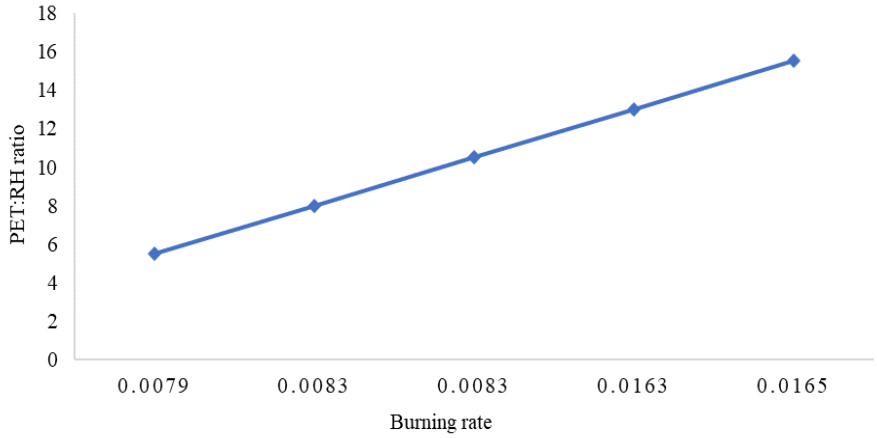

Fig. 1. Variation of PET: RH ratios with burning rate at constant PET

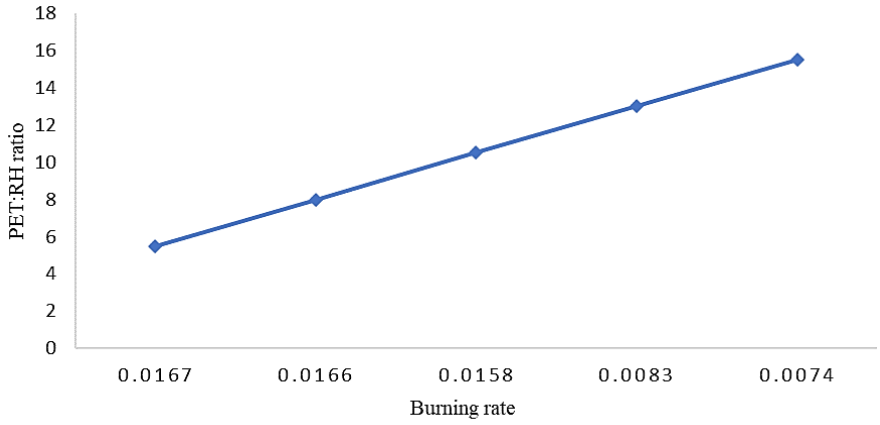

Fig. 2. Variation of PET: RH ratios with burning rate at constant RH

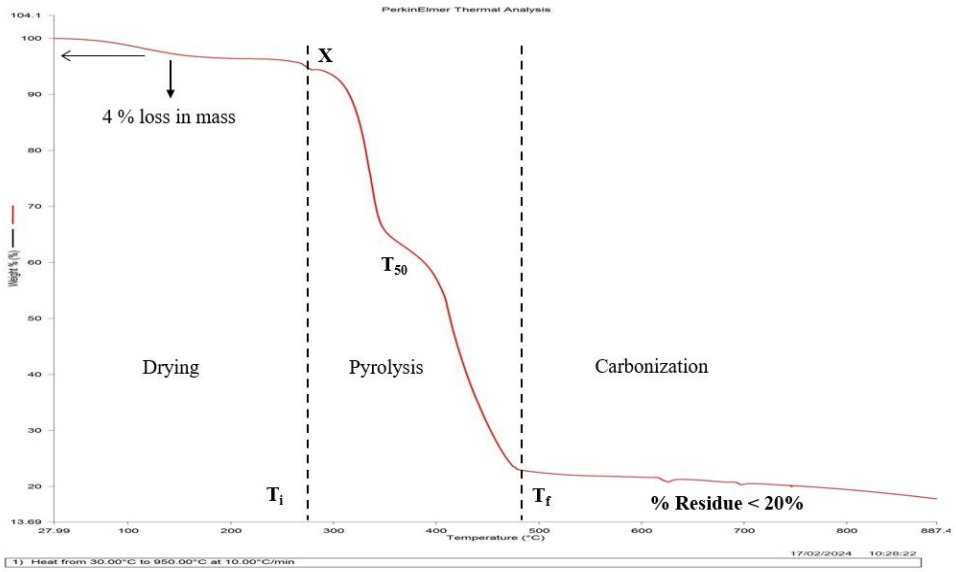

Fig. 3. Thermogravimetric curve of PET: RH composite (TGA)

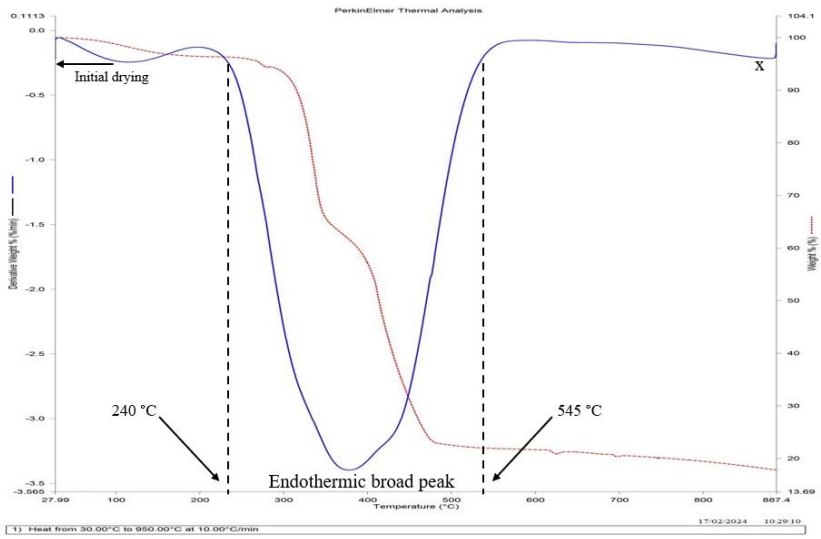

Fig. 4. Diffrential thermal graph of the PET: RH composite (DTG)

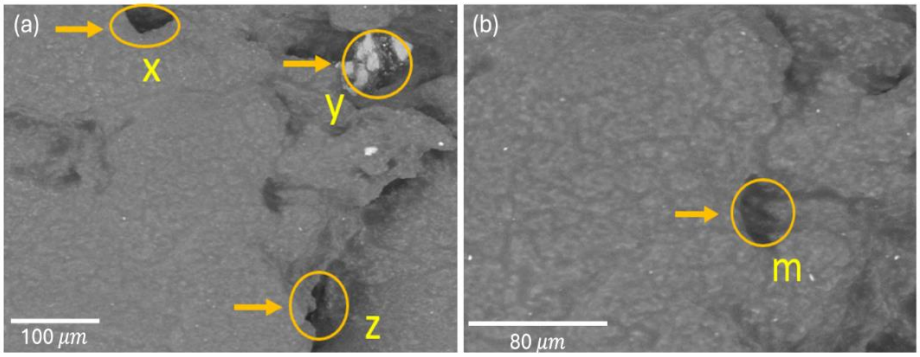

Fig. 5. Scanning electron micrograph showing the surface morphology of the PET-RH composite at: 500× (a) and 1000× (b) magnification

The DTA thermogram on the other hand for PET and RH exhibited a downward peak as seen from the negative values in the derivative weights, this also implies an increase in the weight loss which is attributed to the degradation, decomposition, evaporation or loss of volatile chemical species, since these processes are endothermic in nature, that accounts for the broad endothermic peak as seen in Fig. 4 which appeared as a downward deflection in the DTA curve in the range 240-545 °C. The first and slightly defined downward peak at 100 °C is as the result of the initial removal of moisture or volatile chemical species from the sample. These observed values were similar to the findings of Kalu et al. [22] whose SISOMAR endotherm were in the range 290 to 480 °C and Jose [23] whose HCN (Hydrogen cyanide) polymer had a very broad endotherm in the range 300 to 950 °C. Only slight differences were observed from the TGA and DTA thermogram of the PET and RH which could be attributed to differences in the techniques [22,23].

3.3 Morphological Study of the PET: RH Composite

Scanning electron microscopic analysis of the surface morphologies of the RH base and PET composite from this study has been displayed in Figs. 5a and b. Particularly, in terms of adhesion between the RH fiber and PET matrix, the surface morphology of the composite made visible some air spaces in the SEM micrograph captured at 500x and 1000x magnifications. This suggests good adhesive property across the produced composite as more filler particles were dominant [18,20]. On the other hand, less adhesion was observed between the RH fiber and PET matrix at regions x, y and z from the 500x magnification (Fig. 5a) and m region, from

the 1000x magnification (Fig. 5b). These substantial regions of weakly bonded materials may shatter brittlely [22,23].

4. CONCLUSION

This investigation on the production of a composite from waste plastic water bottles and rice husk for use as an egg tray and wood panel showed that the composited can withstand temperature and durability requirements for use in the egg tray and wood panel production, as reflected in the TGA/DTA, SEM and burning rate methods of characterization. However, only minor improvement could be made to fortify and prevent any sort of fissures or ruptures in the composite's matrix, as evident from the SEM images. The high temperature and stress requirements for polymers to overcome load can also be achieved based on the TGA/DTA analysis which showed an initial degradation temperature at more than 200 ℃ this would invariably serve as a good measure for thermal resistance of polymers or conventional materials that are employed for similar products, as to the making of wood panels. Also, evident, was the thermal decomposition to char of temperatures greater than 500 ℃. The DTA thermogram however, complemented these findings from the TGA analysis. It is however important that the utilization of a PET: RH composite must keep in mind its thermal properties to define most suitable applications it should be employed in.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Authors hereby declare that NO generative AI technologies) and text-to-image generators have been used during writing or editing of manuscripts.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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