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ORIGINAL ARTICLE

PP/Jute Fiber Composites: Effect of Biological Route of Surface Treatment and Content of Jute on Composites

Rudresh Naik[1] · **Sai Gowtham Allu**[1] · **D. Purnima**[1]

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Abstract

Jute as a fiber has many applications. It is also used in polymers as reinforcement due to its good tensile properties. However, when it is used in polymer matrices, there is a lack of adhesion between the polymer and jute fiber. Surface treatment of fiber using chemicals has been found to improve the properties. However, the use of chemicals causes environmental pollution, when these chemicals are discharged into the environment. In this paper, an attempt has been made to study the effect of the biological route to surface treat the jute fiber. The effect of surface treatment on the morphology of jute was examined. A comparative study was on the crystalline, thermal, and tensile fracture morphology of the composites to understand the effect of the incorporation of untreated and treated jute fibers in polypropylene (PP).

Keywords PP · Jute · Biological · Agricultural residue · Surface treatment · Crystalline

Introduction

Natural fiber–reinforced composites are popular as an environment-friendly alternative to the existing synthetic fiber composites. The incorporation of natural fibers in a polymer matrix increases the stiffness and strength of composites [1]. A lot of attention has been received by PP/jute composites. Jute is an abundant fiber known for its properties like non-toxicity, degradability, low cost, and renewability. It is used in a wide range of applications from manufacturing ropes to as fillers in composites. Jute is made up of cellulose (45–73%), lignin (12–26%), and other components with 8° microfibrillar angles [2–4]. The mechanical properties include tensile strength in the range of 393–800 MPa, elongation at break % of 1.5–1.8, and density of 1.3 g cm^{-3} [2–4].

PP is a polyolefin used in various applications. It is recyclable, thermally stable, flexible, cheaper, and resistant to absorb moisture and chemicals [2, 5, 6]. PP is also known for low processing temperature and low cost. To improve its application in engineering applications, composites of PP were prepared with various synthetic fibers like glass fiber

✉ D. Purnima
dpurnima@hyderabad.bits-pilani.ac.in

[1] Department of Chemical Engineering, Birla Institute of Technology and Science, Jawaharnagar, Shamirpet Mandal, Hyderabad, Telangana 500078, India

and carbon fibers [7, 8]. However, these fibers have been found to create environmental concerns in the form of safety hazards for the people handling them and high energy requirements for processing, and also when disposed in the environment, it is found that these fibers being non-degradable cause a lot of environmental challenges. So, taking these environmental concerns into consideration, there is a need to focus on using natural fibers which have many advantages such as lower energy requirements and causing less wear and tear to the equipment used.

Among the natural fibers, jute has a special advantage such as easy availability and low cost along with its advantage of being biodegradable [1]. Jute has been widely used as filler in PP [5, 9]. One of the problems faced when a filler or polymer is used in a polymer is interfacial adhesion [5, 6, 10]. The hydrophilic nature of jute makes it incompatible with hydrophobic resin. The poor compatibility is due to the high polarity and hydrophilicity of jute fibers [11–14]. The interfacial adhesion can be improved by the use of coupling agents and surface treatment [4, 9, 14, 15].

Surface modification involves breaking of atomic bonds between fibers to enable functionalization. Various treatments for surface modification are alkaline treatment, silane treatment, acetylation [4, 13], maleic and succinic anhydride grafting [14], bacterial treatment [12, 17], and enzyme treatment [5, 11, 16, 18]. Chemical treatments were found to be efficient, but the large use of chemicals and their disposal after treatment pose a major problem to the environment [5, 11, 12]. The physical processes consume a lot of energy and can deteriorate the mechanical properties of fibers [19, 20]. The high energy demands and environmental pollution drawbacks with physical and chemical treatments, respectively, have turned the world towards eco-friendly treatments.

In recent years, research has been focused on exploring environment-friendly approaches to treat the fibers for various applications. Kalia et al. [14] reviewed the various methods for the treatment of fibers and reported that the enzymatic approach for the treatment of fibers is a green route for surface treatment as it can reduce the use of chemicals and therefore reduce the amount of the chemicals disposed into the environment. Enzyme treatment is found to be specific and eco-friendly and has mild operational conditions [16, 18, 21]. Surface modification by enzymatic and bacterial treatment on jute gave good results with respect to better surface modification and improvement in interfacial adhesion with the polymer [4, 5]. The treatment also removes pectin, lignin, and hemicellulose components [16]. Agrawal et al. [5] used different concentrations of pectinase to find the optimum concentration to be used to modify jute in the manufacture of PP/jute composite. Karaduman et al. [16] studied the effect of different enzymes on jute fiber in fabric form and the effect of laccase-treated jute in polyester/jute composite. Laccase-treated flax fiber in epoxy was found to improve the interfacial shear strength thereby proving that the fiber matrix bond improved on treatment as observed by Brodowsky et al. [22]. As the fiber properties vary with its components, the type of enzyme also acts specifically. So, there is a need to identify various enzymes suitable for the modification of various fibers and study the effect on various properties of the composites.

In this paper, jute fiber was treated with pectinase, and untreated and treated jute samples were used in PP/jute composites. The concentration of enzymes for treatment of jute was kept constant which was optimized in our previous study [5]. The weight % of jute was varied as 5%, 10%, and 15% in the case of both untreated and treated PP/jute composites. SEM, X-ray diffraction (XRD), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA) were used to characterize untreated and treated PP/jute composites. This study aims to understand the effect of a green enzymatic route of surface treatment which is environment friendly and sustainable. In this paper, an attempt has been made to

study the effect of incorporating treated fiber by comparing the properties of PP/jute composite before and after treatment to understand the fiber-polymer matrix adhesion by studying the thermal and crystalline morphology of injection-molded samples and the morphology of tensile-fractured samples.

Experimental

Jute Fiber

Treatment of Jute Fiber

The process was the same as mentioned in a previous paper [5]. In brief, jute fiber is chopped into small pieces. Pectinase enzyme is dissolved in water for 48 h while the pH of the solution is adjusted to 5.6% of pectinase and was used. The fiber is vacuum dried after it was removed, and the enzymes were deactivated. Samples were dried and stored in polythene bags for further analysis.

Tensile Specimen Preparation

Dog bone specimens of the sample were prepared as per ASTM D638 for PP/jute composites with untreated and treated fibers as per the procedure mentioned in our previous paper [5]. The jute content was varied from 0 to 15 wt% in both untreated and treated PP/jute composites.

Characterization Techniques

Field Emission Scanning Electron Microscopy The topographical morphology of the fibers and tensile fractured composites was studied using SEM. The sample images were captured using FEI Apreo LoVac. The tensile-fractured samples were gold-coated using the sputter Leica EM UC7 ultramicrotome, and the images were captured.

XRD The crystallinity of jute/PP composite is found using XRD. The samples were taken from the tensile specimen and analyzed by the Rigaku Ultima IV X-ray diffractometer, to find the crystallinity index at an angle range of 2θ of 13–25° with a monochromatic CuKα radiation source at 40 kV and 30 mA with a step size of 0.02 and a scan rate of 1° min^{-1}. The % crystallinity is calculated by finding the area under the crystalline curve and the area under the amorphous part of the XRD pattern as per the procedure given by Gupta and Purwar [23].

TGA TGA was used to study the thermal stability of the sample taken from the tensile specimen using the Shimadzu DTG-60 apparatus. The weighed amount of 4 to 5 mg of the sample was heated from 30 to 800 °C with the heating rate of 20 °C min^{-1} under a nitrogen atmosphere with a flow rate of 100 mL min^{-1}.

DSC DSC was used to study the thermal behavior of the PP in the presence of untreated and treated jute samples which was taken from the tensile specimen, using the Shimadzu

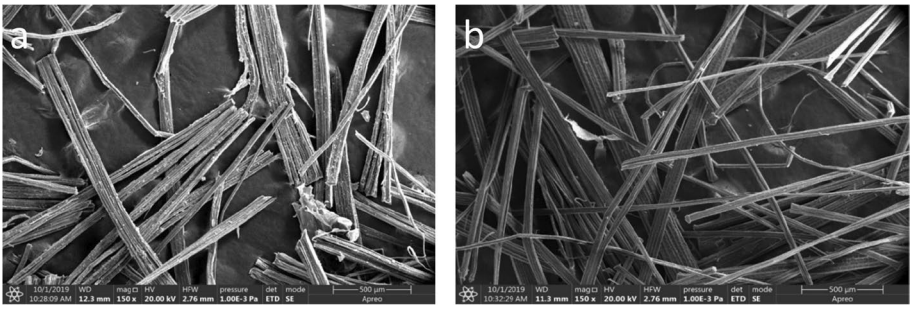

Fig. 1 Jute fibers **a** before and **b** after enzyme treatment

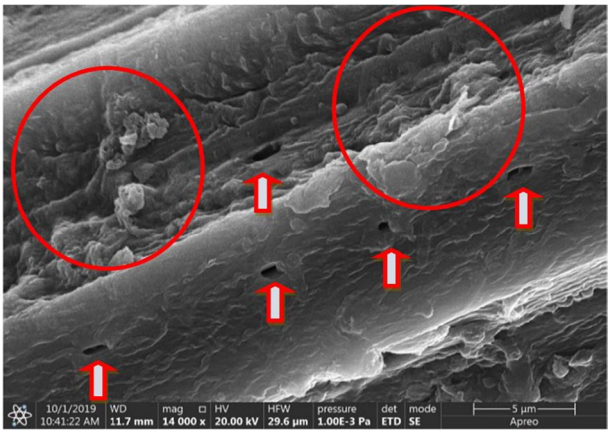

Fig. 2 Pores, after surface treatment, and rough surface of jute fibers. Pores are denoted by arrows. Circles show the rough surface which is due to the effect of pectinase on the composite

DSC-60 apparatus. The sample of 4 to 5 mg was heated from 30 to 300 °C at a heating rate of 10 °C min^{-1} and then cooled to 30 °C at a cooling rate of 10 °C min^{-1} and reheated to 300 °C at 10 °C min^{-1} under a nitrogen atmosphere with a flow rate of 100 mL min^{-1}.

Results and Discussions

Scanning Electron Microscopy

Figure 1a and b show the images of untreated and treated jute samples, respectively. The treated fibers in Fig. 1b are seen to be clean and more dispersed. The clean fibers are probably due to the enzymatic removal of lignin. It is seen in our previous work [5] that with 6% pectinase treatment, the lignin content of jute reduced from 23 to 13%, and here, the same fiber was used to make the composite. In our earlier work done to study the effect of variation of pectinase concentration on the treatment of fiber to optimize this for treatment of fibers and use it for making composites, it was found that Fourier transform infrared spectroscopy shows an OH stretch in the range of 3400 to 3500 cm^{-1} which corresponds to lignin [5] in the case of untreated jute fiber. However, the intensity of this peak reduced in the case of treated jute fiber. As shown in Fig. 2, jute fibers show that there is an increase

in the number of pores, after surface treatment, and rough surface. The number of pores improves the fiber/matrix adhesion. The rougher surface improves gripping, leading to an improvement in the strength of adhesion [24]. Enzymes remove impurities, pectin, and lignin creating a rougher surface, facilitating resin bonding onto fibers, and successfully improving fiber-matrix bonding covered [14]. In ramie fibers hydrolyzed with *Streptomyces albaduncus*, the treated fibers turned to be soft and bright which is due to the removal of gum materials from fibers [12]. Chemical treatment increased the crystallinity of fibers which is due to the removal of cementing materials [25]. Alkali treatment enhanced the surface roughness by removing oils and wax that covers the external surface of the cell wall [26]. The alkali-treated hemp fibers show a surface which is free of residues compared to the untreated hemp fibers [27]. The enzyme treatment in jute fibers in this study is able to produce desired roughness and pores suitable for better mechanical gripping resulting in better interfacial adhesion with polymer.

The morphology of tensile-fractured specimen of untreated and treated jute samples containing PP composites is given in Fig. 3a to f. In Fig. 3a, it can be seen that the matrix surface is brittle and that fiber is not covered with the matrix. In Fig. 3b, the PP composites with 5 wt% treated jute fibers are seen to be covered with polymer; however, there are some voids in the specimen. In Fig. 3c, it can be seen that tensile-fractured samples of 10 wt% untreated sample show a lot of void spaces around the fiber, indicating poor fiber matrix adhesion and also matrix fibrillation is taking place. In Fig. 3d, it can be seen that the fiber is covered with polymer; however, there is fiber aggregation in the case of 10 wt% jute fiber–treated composite. In Fig. 3e which shows the morphology of untreated 15 wt% PP/jute composite, the presence of matrix fibrillation and big voids can be observed, indicating poor matrix fiber adhesion. However, in the case of 15 wt% treated fiber as shown in Fig. 3f, the fiber is covered with polymer; however, fiber aggregation is taking place which could be due the improper mixing.

XRD

Figure 4a and b shows the XRD diffraction pattern of PP/jute composites with untreated and treated jute fibers. It can be seen from the figures that peaks for PP in both untreated and treated PP composites were found to be at 14.19(110), 17.02(040), 19.0(130), and 21.8(111) in accordance with data reported in the literature [28]. The standard peaks of PP have been retained, and the alpha crystalline nature of PP has been preserved. The variation of crystallinity of PP with untreated and treated fibers is shown in Fig. 4c. It can be seen from the graph that the % crystallinity is slightly higher in the case of 5 wt% untreated PP/jute composite and decreases with jute content in the case of untreated PP/jute composites. As seen, it is slightly lower in the case of untreated composite and decreases with an increase in untreated jute content. As seen from DSC data of S_i and peak temperature, it can be observed that both increase with the addition of 5 wt% jute in the case of untreated PP jute composite, resulting in higher overall crystallization. However, for the content above 5 wt% in the case of treated jute composite, the rate of nucleation is increasing, but the rate of crystallization is low which may have resulted in lower overall crystallization. However, in the case of treated fiber composites, the value of crystallinity is higher, which may be due to the better interfacial bonding between the fiber and polymer as can be seen in Figs. 3b, d, and f.

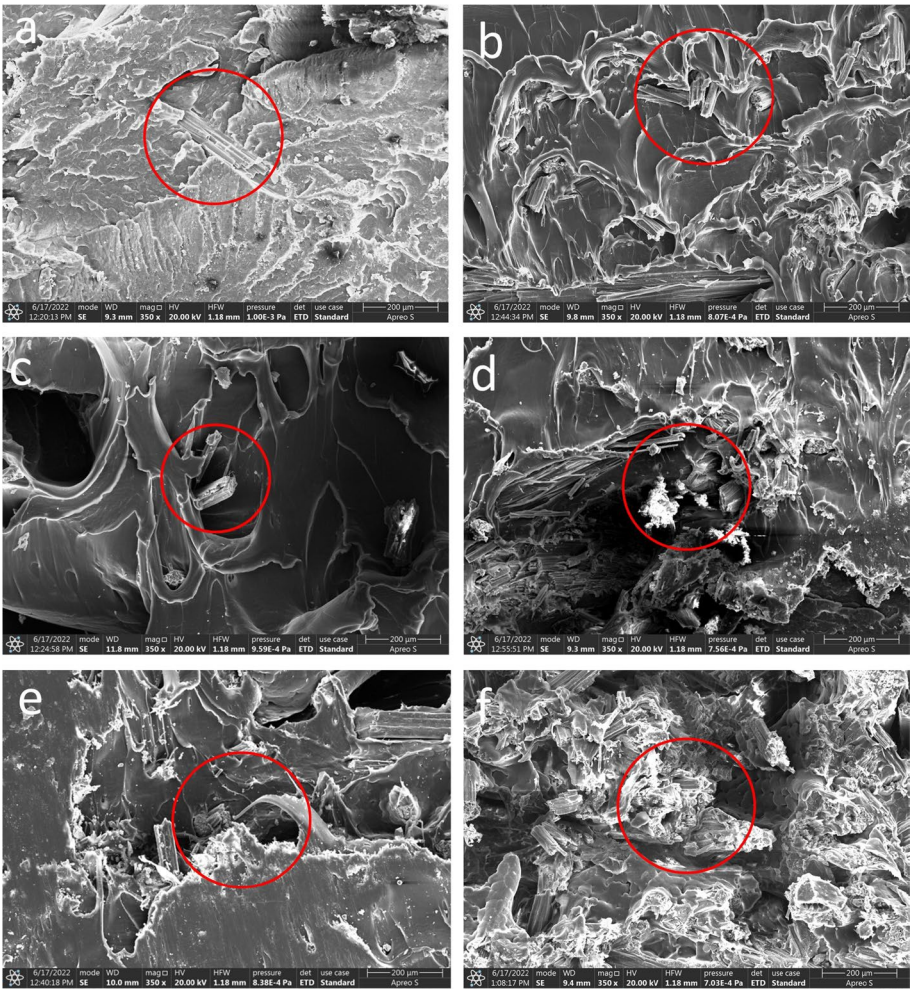

Fig. 3 Jute contents of 5 wt% in **a** untreated and **b** treated PP/jute composites, 10 wt% in **c** untreated and **d** treated PP/jute composites, and 15 wt% in **e** untreated and **f** treated PP/jute composites

DSC

The variation of the crystallization behavior can be seen in the case of untreated and treated PP/jute composites from Fig. 5a and b, respectively. The parameters such as onset temperature of crystallization (T_{onset}), peak crystallization temperature (T_{peak}), rate of nucleation (S_i), and crystallite size distribution (Δw) can be found from crystallization curves as reported in the literature [23, 29].

The variation of these parameters with the content of jute is presented in Fig. 6a–d, respectively. From Fig. 6a, it can be seen that T_{onset} for 5 wt% untreated jute composite is the same as that for PP. However, as the jute content increases, T_{onset} decreases in the case of untreated composites. For treated composites, T_{onset} is lower than that for untreated composites at all concentrations of jute. However, it increases with the content

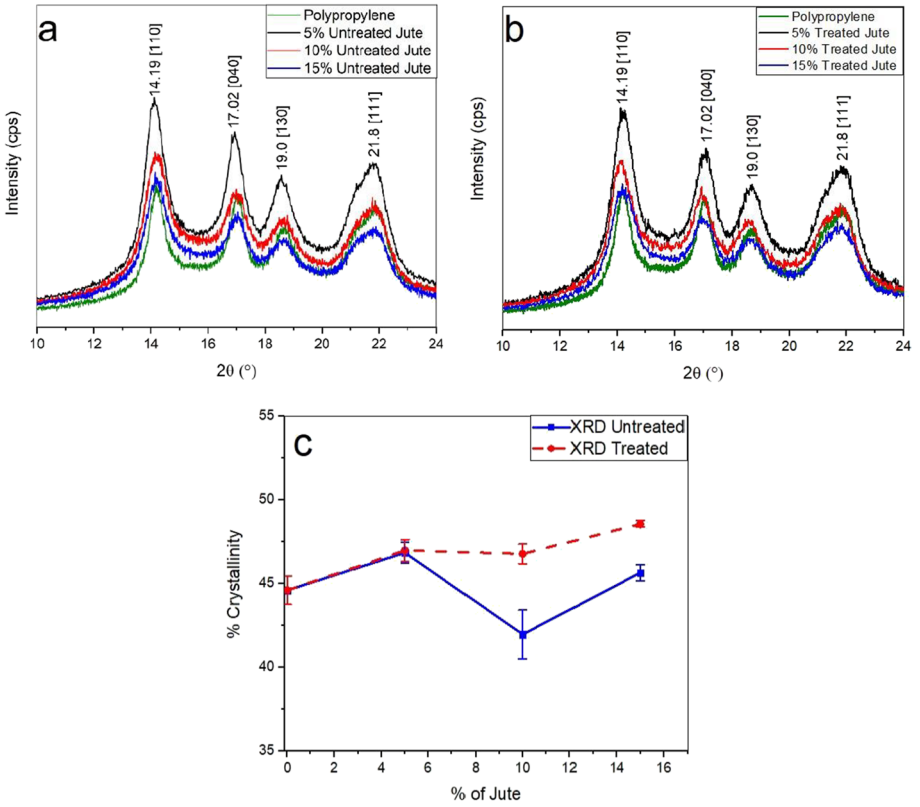

Fig. 4 Variation in XRD with jute content in the PP/jute composite. **a** Untreated jute fiber. **b** Treated jute fiber. **c** Variation in crystallinity with jute content in untreated and treated PP/jute composites

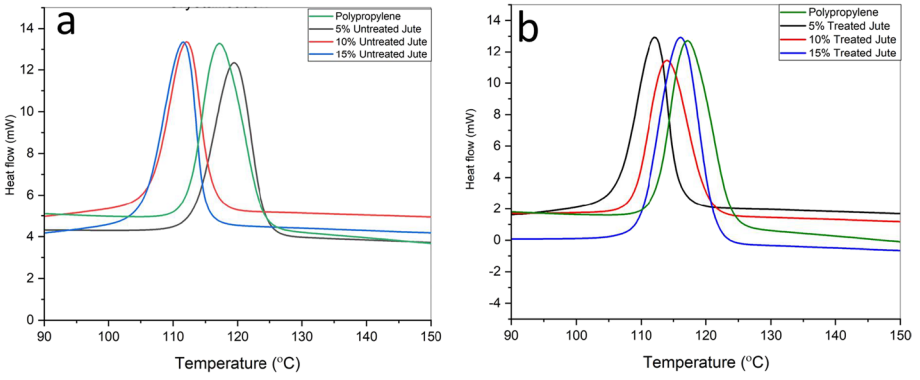

Fig. 5 Heat flow vs temperature (°C) in **a** untreated and **b** treated PP/jute composites

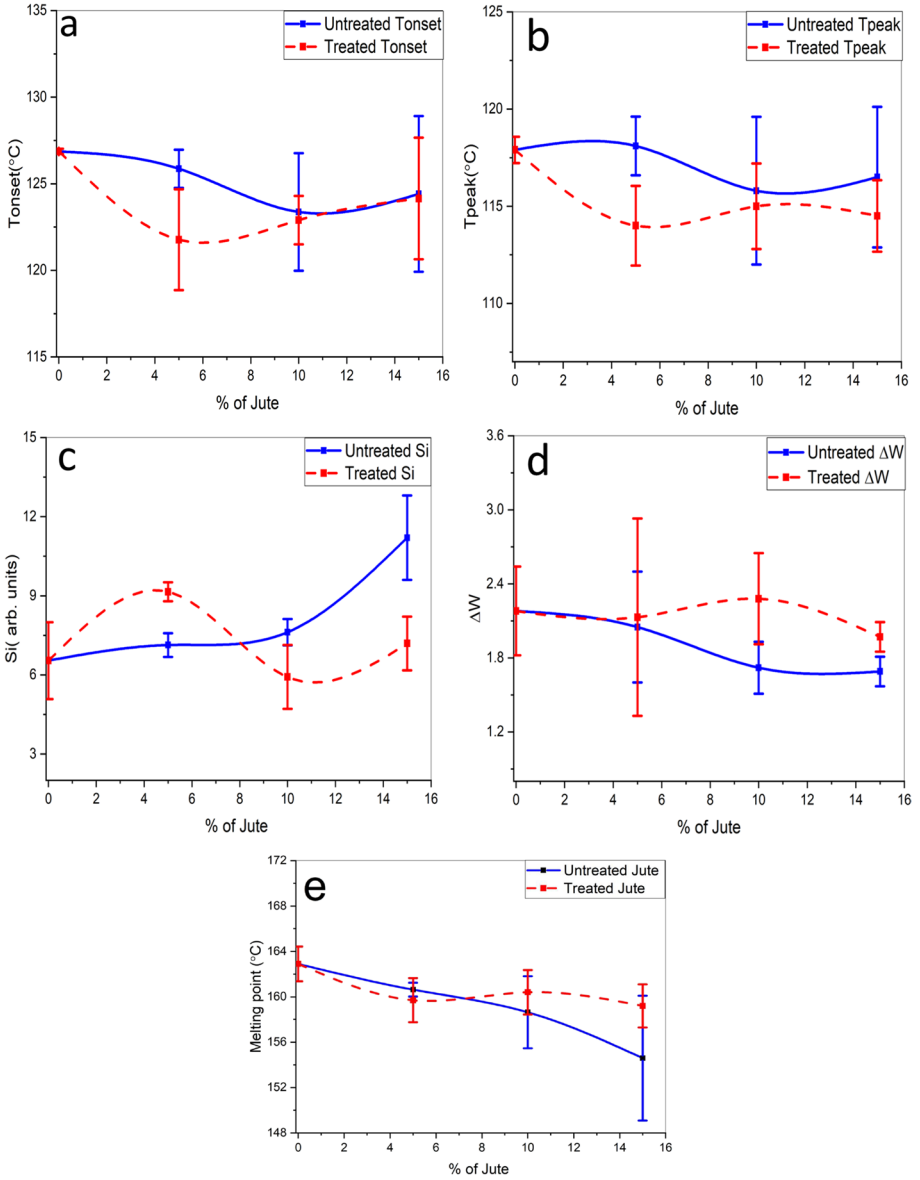

Fig. 6 **a** T_{onset} and **b** T_{peak} variations with jute content in untreated and treated PP/jute composites. **c** S_i and **d** Δw variations with jute content in untreated and treated PP/jute composites. **e** Variation in the melting point of jute content in the PP/jute composite

of jute in this case. A similar trend of T_{peak} as that of T_{onset} is observed in untreated and treated PP/jute composites which is shown in Fig. 6b.

S_i increases with an increase in the content of jute in untreated composites, and in the case of treated composites, it increases at 5 wt% jute content and decreases as the

composition of jute increases as shown in Fig. 6c. Δw decreases with increases in jute content for untreated composites, and an opposite trend is shown for treated composites.

The increase in the rate of nucleation which is supported by the narrow crystal distribution as shown by Δw means that the rate of nucleation and crystallite distribution show an opposite trend which can be seen from Fig. 6c and d as reported in the literature [23, 29]. T_{peak} depends on the overall crystallization process. It decreases in the case of untreated PP/jute and increases in the case of treated composites as the fiber content increases, indicating an increase in the overall crystallization with an increase in fiber content, and values at higher content are closer to those of untreated composites at the same fiber content but lower than those of PP.

The variation in melting point is shown in Fig. 6e. The melting point of the untreated composites decreases with an increase in jute content. In the case of treated composites, it can be seen that values are only lowered by around 4 °C with respect to PP and almost remain the same at all concentrations of jute. However, in the case of untreated jute composite, the value decreases with an increase in jute content at all concentrations.

TGA

TGA was carried out to understand thermal stability and degradation mechanism [2, 30]. TGA scans of untreated and treated samples are shown in Fig. 7a and b, respectively, and it is seen that all the composites (untreated and treated) showed a similar type of single-step degradation [4, 11]. With an increase in jute content in the composite, the thermal stability decreases as seen from DTG curves in Fig. 8a. Upon enzymatic treatment, the thermal stability of the composite further decreased as seen from DTG curves in Fig. 8b. The thermal stability decreases which is due to the removal of the lignin which has been reported in our earlier work [5]. It has also been reported in the literature that lignin removal decreases the thermal stability. Some treatments result in a decrease in thermal stability as seen in ramie fiber, and chemical treatment reduced the thermal stability [12].

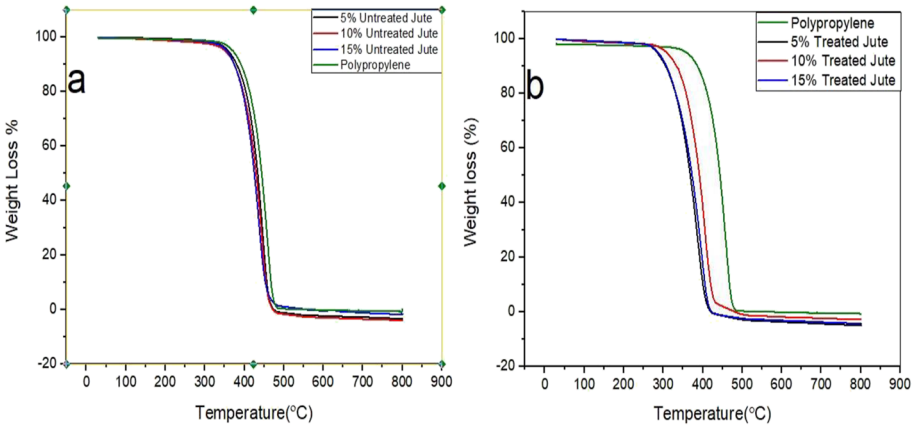

Fig. 7 TGA graph with a variation in jute content in **a** untreated and **b** treated PP/jute composites

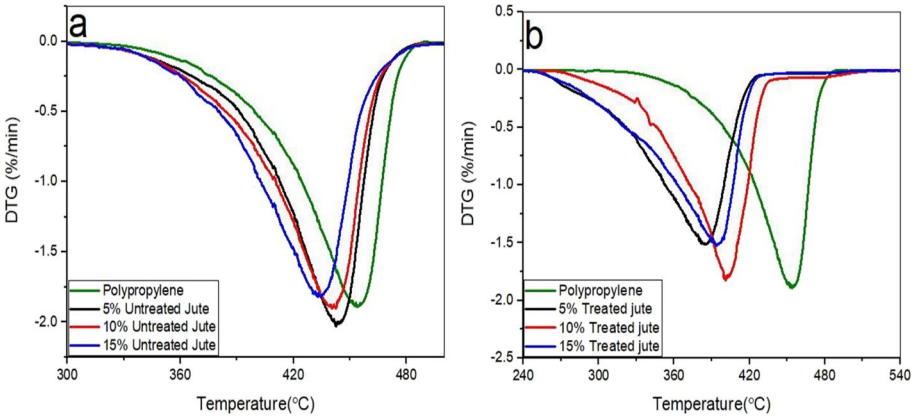

Fig. 8 DTG graph with variations in jute content in **a** untreated and **b** treated samples

Conclusion

The effect of the enzymatically treated fiber on morphology, thermal, and crystallization properties was studied by comparing the properties of the untreated fiber–based PP composites with treated fiber–based composites. The following observations were found:

1) On comparing the untreated and treated fibers, it was found that the treated fiber had pores and rough surface which are desired for better interfacial adhesion between fiber and PP matrix as it causes improved mechanical gripping between fiber and matrix. This indicated that enzyme pectinase was effective in modifying the surface of the fiber.
2) The tensile fracture morphology of untreated fiber–based composites showed more voids and poor adhesion between matrix and fiber. The treated fiber composites showed voids too, but the fiber was well covered by polymer, indicating better interfacial adhesion; however, there was an aggregation of the fiber, indicating the need for a better mixing technique.
3) The crystallization process indicated that the rate of nucleation increased with an increase in jute content for untreated PP composites. However, in treated composites, only at lower concentrations the rate of nucleation was more. However, the overall rate of crystallization though lower than PP increased with an increase in jute content for treated PP/jute composites while untreated composites have a decreasing trend.
4) The % crystallinity of treated composites was higher than that of untreated composites, indicating better interfacial adhesion.
5) The thermal stability of untreated composites was higher than that of treated composites.

The enzyme treatment favored the interfacial adhesion and resulted in an improvement of % crystallinity which will lead to better mechanical properties. So, the enzymatic treatment can further give better results with the inclusion of a mixing technique such as extrusion prior to injection molding and lead to a green sustainable method of surface modification.

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Author Contribution D. Purnima: ideation, analysis of the results, and editing of the manuscript.
Rudresh Naik: execution of the experimental work.
Sai Gowtham: analysis of the results and editing of the manuscript.

Data Availability The data sets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing Interests The authors declare no competing interests.

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