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A recyclability study of bagasse fiber reinforced polypropylene composites

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ABSTRACT

Natural fiber reinforced composites are gaining attention nowadays due to their low cost, biodegradable characteristics, lower density and comparable mechanical properties with synthetic fibers reinforced composites. As the recyclable properties of these composites are still not fully explored and this could be the reason that these are not much used in domestic and other applications to their full potential. In the current experimental investigation, the effects of mechanical recycling on the properties of bagasse fiber reinforced polypropylene composites have been studied. Tensile properties, crystallinity and aspect ratio exhibits an increase, while flexural properties decrease with each recycling step. No significant change in thermal behavior has been detected even after 5 cycles but a slight increase in the hardness has been observed. SEM images confirmed the change in aspect ratio due to crushing of fibers as well as fiber fracture became more dominant after each recycling step.

1. Introduction

Recycling is a key component of modern waste reduction technique and a third component in the “Reduce, Reuse and Recycle” waste hierarchy. Generally, recyclability and degradability are considered as opposite terms as degradable materials are unrecyclable due to the deterioration in the properties over a period of time. The opportunities for recycling offer sound economic benefits due to the high cost of the virgin materials. In common industrial practice, 20% of the process waste (after shredding) is mixed with 80% of the virgin thermoplastic polymer to produce the part, which also helps in reducing the polymeric waste. When material arrives in the waste stream, recycling is the process of using the recovered material to fabricate a new product [1]. In plastics, the waste generated during the fabrication of part in form of gate, flash and defective parts etc. are termed as “process waste”, while the wastage after serving the designed life of a product is termed as “aged waste”. The recyclability of plastic waste depends mainly on the aging time, as after a prolonged time, plastic losses its inherent properties i.e. mechanical properties, appearance, surface finish, flowability etc. Mechanical properties of pure polypropylene decrease with the number of cycles as when PP is exposed to high temperatures and shear, then due to oxygenation and other impurities i.e. peroxides and catalyst residues, resulting in a decrease in viscosity and a considerable decline in mechanical properties, which makes it more brittle and also exhibits staining [2].

Composites can be used in a wide variety of applications as a replacement of pure plastic in various engineering products, but they have not been properly recycled, due to their intrinsic nature of heterogeneity [3]. Glitches associated with recycling thermoplastics and their composites are much less compared with the reprocessing of thermosets. Therefore, the prevalent practice of thermoplastic composites in various applications is projected to have a more advantageous environmental impact. Present environmental concerns have generated awareness in the recycling of composite materials and plastics in particular. Until now, landfilling and burning have been the most common end-routes for FRP products and waste plastic material. However, with the environmental consciousness as well as government laws and legislation on waste management, the above-mentioned end-routes will be gradually unavailable. The use of natural fibers is often seen as the panacea for these glitches, such as environmental issues, end-of-life (ELV) vehicles, waste minimization etc. [4].

Much less energy (60–80%) is required in growing and harvesting of natural fibers as compared to the production of synthetic fibers (Glass fibers: 31.7 MJ/kg; Flax fibers: 6.65 MJ/kg, China reed

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fibers: 3.64 MJ/kg, Kenaf fibers: 15 MJ/kg) [5–7], but later, it also depends on the processing route followed to get them in their usable form. Due to growing environmental consciousness, natural fiber reinforced polymer composites are fetching dominance in usage in various engineering applications. Some other benefits of using natural fibers in composites are relatively lower cost, density, comparable specific properties, biodegradability and recyclable properties. These materials are durable, reliable, light-weight and are expressively better than traditional materials, therefore, empowering the demand for natural fiber reinforced composites in various industries [8,9]. As the largest application segment, use of natural fiber reinforced composite materials in the automotive industry is growing very swiftly (fabrication of body parts and interiors).

Recyclability of natural fiber reinforced thermoplastic composites can be categorized on the basis of the served life of the product (in context of aging behavior). The properties of the matrix, fibers and fiber-matrix interface changes with each recycling [10–12]. This is particularly true for the aspect ratio (L/D) of the fibers, which represents the ratio between the length (L) and the diameter (D) of the reinforcing fibers. Aspect ratio is the key factor in the reinforcement mechanisms of a composite as it defines the interfacial bonding as well as load transfer between fibers and matrix.

Few studies have been carried out on the recyclability of synthetic fiber reinforced thermoset and thermoplastic composites, but very few have been reported for natural fiber reinforced composites. A handful of investigations have also been carried out to study the effects of bagasse fiber reinforcement on mechanical properties of recycled polyethylene [13,14] and polyethylene terapthalate [15] and reported that tensile strength decreases with the increase of fiber volume fraction in the recycled composites. Bourmaud and Baley [10] experimentally investigated and reported that the tensile modulus of hemp and sisal reinforced polypropylene composites can be conserved with the number of reprocessing cycles. In another investigation by the same author [11], a reduction in the aspect ratio was reported with the number of cycles. A recyclability study of flax/PLLA composites [16] reported a higher reduction in mechanical properties for 30% fiber volume fraction, while the composites with 20% fiber volume fraction show better properties when compared to neat PLLA after six recycling steps. Also, a continuous reduction in aspect ratio as well as in molecular weight has been reported.

The feasibility of reprocessing of natural fiber reinforced polymer composites has not been explored appropriately till date, therefore recycled composites are not being used with full potential in industries. The objective of the present research is to study the recyclability of bagasse fiber (BF) reinforced polypropylene (PP) composites fabricated by extrusion-injection molding process, and to analyze the effects of recycling on hardness, mechanical behavior, surface roughness, thermal behavior and crystallinity. A total of 5 cycles has been performed to study the effects of recyclability on the mechanical behavior of the composites. Tensile and flexural tests have been conducted to track the mechanical behavior of recycled composite specimens and thermal behavior is investigated by TGA/DSC method. The XRD pattern has been used to analyze the effects of recycling on the crystallinity of the recycled composites.

2. Materials and method

2.1. Materials

Polypropylene (PROPEL-1110MAS) in granule form has been procured from Indian oil corporation Ltd. (IOCL) to be used as the matrix material. Basic properties of polypropylene have been shown in Table 1. High Melt Flow Index (MFI) indicates the low viscosity of material at elevated temperature, which may result in better interfacial bonding between fiber and the matrix in developed composites [17,18].

Raw bagasse was collected from M/s B. H. Sugar Mills Limited, Uttar Pradesh, which contained a lot of leaves residue, dust, pulp, pith, moisture and uncrushed sugarcane. Therefore, the raw bagasse was dried in sunlight for 2 days to remove foul gases and reducing the moisture content. The dried bagasse was first screened through a sieve (Mesh No: 20) to separate the uncrushed sugarcane as a residue in the sieving process. The sieved material was again screened through another sieve (Mesh No: 60) to separate the bagasse fibers from pith and dust. The fibers were then soaked in lukewarm water (Temp: 30–35 °C) for 3 h to separate pulp. As pulp is lighter, it floats on water surface and is easily removable. The collected fibers were then dried completely in a hot air oven at 80 °C for 6 h and mixed (20% by weight) with pre-dried (80 °C/2 Hrs.) polypropylene (PP) granules.

The diameters of obtained fibers were measured using an optical microscope and observed as 235–380 μm and length as 8–35 mm. The image of raw bagasse and extracted bagasse fibers are shown in Fig. 1.

2.2. Fabrication of composites

The weight fraction of bagasse fiber was selected as 20% for the experimental investigation. This mixture was fed into a single screw extruder (Make: Sai Extrumech, Model: SAI-25) at 170 °C temperature (temp profile: 170-170-170 °C nozzle) and 450 rpm (screw speed) to produce composite wire, which was then shredded into pellets of 5 mm length with help of pelletizer. 20% of the pellets were fed into injection molding machine (Model: ENDURA-60, Make: Electronica) to fabricate composite (BFPP) specimen at 180 °C injection temperature (temp profile: 155-165-175-180 °C nozzle), 50 bar injection pressure and 15 s cooling time. The specimens were fabricated according to the ASTM standards (ASTM D-3039 for tensile testing and ASTM D-7264 for flexural testing). The remaining pellets (80%) were extruded again in the form of composite wire at same process parameters and pelletized. A fraction of the pellets was again used to fabricate recycled composites specimen (RC1) by injection molding. The process is repeated for extrusion and injection molding to fabricate next 3 batches (RC2, RC3 and RC4 respectively) of recycled composite specimens.

2.3. Surface roughness analysis

Surface roughness of the fabricated composite was measured at room temperature using a surface roughness tester (Model: SURFTEST SJ-301, Make: Mitutoyo, Japan). It has been used to determine the surface finish with a 5 μm stylus radius and

Table 1
Properties of polypropylene (PROPEL-1110 MAS).

S.No.	Properties	Standard/Method	Unit	Values
1	Melt flow index	ASTM D 1238	g/min	11
2	Density	Archimedes' Method	g/cm^3	0.91
3	Tensile strength	ASTM D 3039	MPa	28.004
4	Tensile modulus	ASTM D 3039	MPa	687
5	Elongation @ max load	ASTM D 3039	%	10.15
6	Flexural strength	ASTM D 7264	MPa	50.766
7	Hardness	ASTM 2240	Shore D	72
8	Melting point	DTA	^0C	163

Fig. 1. (a) Nascent bagasse (b) Bagasse fiber.

0.25 mm/s speed over the center line of the specimens. The surface roughness of the part is an important aspect, if used for a surface contact application i.e. paint, wear.

2.4. Shore hardness test

The hardness of pure polypropylene, composites and recycled composites has been measured with Shore-D hardness tester (Accuracy: ±1) in accordance with ASTM standard D2240-15. Shore hardness is measure of resistance to indentation by spring-loaded indenter. The higher the number, the greater the resistance/hardness. The hardness is measured at different points on composite specimen over the center line, average value is calculated and rounded off to get Shore hardness number (whole number).

2.5. Aspect ratio analysis

Dissolution or burning of matrix material are the most common methods to extract fibers from the composites for further characterization, but these are not applicable for natural fibers due to their low degradation temperature and chemical reactivity [12,19]. Pieces of the fabricated specimen were compressed at 175 °C between two glass plates in order to fabricate a thin transparent film (Fig. 2 a). Subsequently, the images were taken (Fig. 2b and c) using an optical microscope (Make: Nikon, Model: SMZ-745T) at different magnifications. The average length and diameter were measured using image analysis software 'ImageJ' to get the aspect ratio of the fibers after each recycling process and to check its effects on mechanical properties of the recycled composites.

2.6. Mechanical testing

Tensile and flexural tests have been performed on universal testing machine (Model: INSTRON -5982, Make: Instron Inc., USA). Tensile testing has been performed at a crosshead speed of 2 mm/min with a gauge length of 50 mm. Flexural tests have been conducted at a crosshead speed of 2 mm/min with a span length of 60 mm.

2.7. Microstructure analysis

Microstructure analysis of the fractured specimen carried on scanning electron microscope (SEM) (Make: LEO, Model: 435 VP) along with a sputter coater (BAL-TEC-SCD-005). A thin film of gold was coated with help of sputter coater on the specimen to increase the conductivity of the specimen before micrographs were taken.

2.8. Thermal behavior

The literature highlighted that the crystallinity and degradation behavior relatively influences the thermal and mechanical property of polymer-based composites [20]. Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) have been performed on EXSTAR TG/DSC 6300. About 10 mg of the test sample was sealed in alumina pans, followed by heating from 35 °C to 500 °C at a heating rate of 10 °C/min under Nitrogen environment. The peak in the DSC curve corresponds to the melting temperature (T_m) and area under the peak is equal to the melting enthalpy (ΔH_f) of the specimen. The degree of crystallinity was calculated using equation (1) [21].

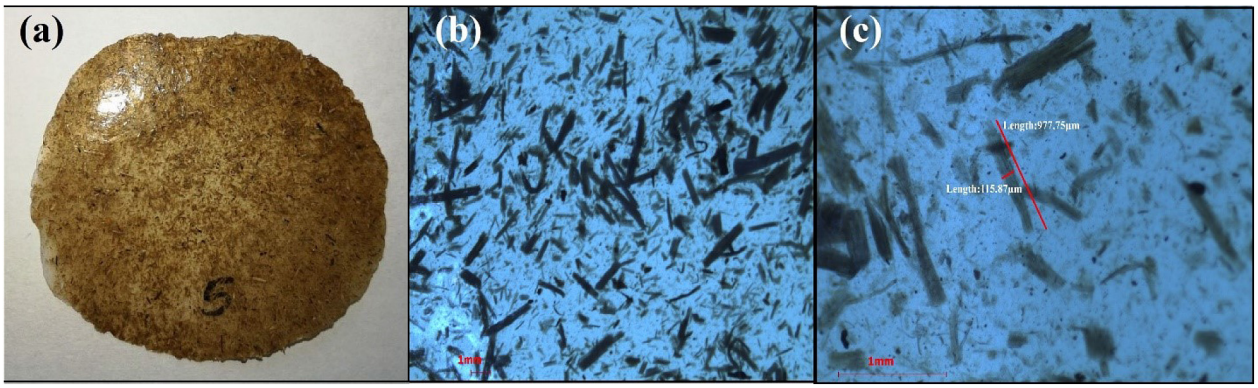

Fig. 2. (a) Thin film of composite (b) at 5× magnification (c) at 50× magnification.

$$X_C = \frac{\Delta H_f}{\Delta H_{100}} \text{x } 100 \quad (1)$$

Where X_C is the degree of crystallinity, ΔH_f is the enthalpy of fusion of the specimen and ΔH_{100} is the enthalpy of fusion for 100% crystalline polypropylene (209 J/g).

2.9. X-ray diffraction (XRD) and crystallinity analysis

XRD has been performed on Bruker (D8-Advance) to study the crystalline behavior of the material after each processing cycle with Cu as target material and crystallinity has been measured by using Ruland-Wonk method [22]. The mathematical relation used to calculate the crystallinity by XRD method is given by equation (2).

$$\%Crystallinity = \frac{Crystalline\ area \times 100}{Total\ area\ under\ the\ curve} \quad (2)$$

3. Results and discussion

3.1. Surface roughness and hardness analysis

The developed composite specimens have been tested initially for surface roughness and hardness and the obtained results are shown in Table 2. It was observed that average surface roughness (R_a) and root mean square roughness (R_q) increase continuously but the rate of increment decreases with each recycling, while the shore hardness increases continuously.

The increase in hardness is attributed to reprocessing of the matrix material that consists of heating, mixing and cooling. Multiple thermal processing also reduces the flow-ability of the matrix material [2].

3.2. Aspect ratio analysis

The change in diameter, length and resulting aspect ratio of fibers at different stages have been mentioned in Table 3. A reduction in fiber length and diameter was observed with each processing cycle, but the reduction in diameter (10.28%–30.71%) was observed higher than the reduction in the length (16.71%–26.62%). Therefore, an increase in L/D ratio was exhibited after each processing cycle.

Bagasse fiber has a structure like flute bundles, as shown in Fig. 3a, which are easy to tear in the longitudinal direction as compared to their cross section. The modifications in reinforcement geometry can be associated with shear rate generated during the extrusion as well as injection process, which depends on the rotational speed of the screw, pressure and barrel temperature [16]. Therefore, during extrusion and injection process, the fiber get crushed to some extent and length is reduced but at the same time, fiber bundles are also subdivided into thin fibers, resulting in the reduction of globally measured diameter of the reinforcement. The increase in L/D ratio of the reinforced fiber resulted in an increase of surface roughness of composites due to dispersion of fibers and higher inhomogeneous arrangement [23]. A higher aspect ratio signifies greater interaction area between the fiber and the matrix, resulting in better interfacial properties, which further results in the better tensile strength of the composite.

SEM images (Fig. 3) also endorse bundle division during the recycling process and can enlighten the significant change in fiber aspect ratio with each cycle. This can also be observed that fiber fracture became more dominant with each processing cycle.

Table 2
Surface roughness and hardness of composite specimens.

Material	R_a (μm)	R_q (μm)	Hardness (Shore D)
Pure PP	0.190 ± 0.008	1.570 ± 0.033	72
BFPP	0.753 ± 0.041	6.193 ± 0.128	73 ± 1
RC1	1.107 ± 0.067	8.06 ± 0.102	74 ± 1
RC2	1.277 ± 0.053	9.143 ± 0.254	74 ± 1
RC3	1.337 ± 0.074	10.067 ± 0.189	75 ± 1
RC4	1.361 ± 0.116	10.675 ± 0.153	75 ± 1

Table 3
Diameter, length and aspect ratio of fibers.

Material	Fiber dia. (μm)	Length (mm)	Aspect ratio
Bagasse Fiber	235–380	8–35	21.05–116.3
BFPP	185–243	1.81–4.32	10.3–22.6
RC1	153–218	1.44–3.17	8.7–18.3
RC2	106–167	1.16–2.64	9.7–19.2
RC3	75–124	0.96–1.93	10.8–24.7
RC4	56–105	0.58–1.72	11.5–26.4

3.3. Mechanical properties

Mechanical (tensile and flexural) strength and modulus properties obtained for fresh and recycled composites are shown in Fig. 4a and Fig. 4b, respectively. The tensile strength of all the specimen has been observed between 23 and 24 MPa with a standard deviation of 0.5–0.8%, while flexural strength lies between 50 and 53 MPa with a standard deviation of 0.7–0.9%. The obtained results show that tensile strength initially decreased by 3.3% when compared to BFPP, but after 2nd recycling (RC2), it shows an increasing trend, which lasts up to RC4, with an increase of 5.46% as compared to RC1. The similar trend can be seen for tensile modulus also with an initial decrease of 1.8% and then an increase of 7.17% as compared to RC1. An increase in the maximum load (1.47–2.17%) was discerned after 2nd cycle (RC1: 1425 N) under tensile loading condition, while insignificant reduction was detected in flexural load (146 N-142 N) with each processing cycle. A significant increase (9.83%–14.14%) in E_{max} (elongation at maximum load) was observed with each processing step, but D_{max} (deflection at maximum load) changed insignificantly (15.23%–15.95%).

The flexural strength exhibits a decreasing trend with a reduction of 6.48% up to RC4, while flexural modulus increases by 4.15% up to RC2 and then starts dropping, which is due to the variation in deflection observed. Initially, an insignificant reduction (up to RC2) in deflection was observed, followed by a minor increase, in flexural testing. It is observed that in all the specimen, the flexural properties are higher than those of pure polypropylene. Mechanical properties of a composite mainly depend on the fiber volume fraction, individual properties of fiber and matrix and interfacial bonding between these two. The study on recycling of pure polypropylene exhibited no significant change in tensile properties even after 12 cycles [24], therefore it can be said that the aspect ratio of fibers is the key factor in deciding the mechanical properties of the composites. Processing parameters and fiber weight fraction in fresh and recycled composites were same and the processing temperature was lower than the degradation temperature of natural fiber used, therefore the major factor affecting the mechanical properties of the composites is fiber-matrix interfacial bonding, which again depends on the aspect ratio of the fibers. The surface roughness (asperities) increased with each processing cycle, and increased hardness led to early crack generation during flexural loading (concentrated load over the line of contact), resulting in a reduction in flexural properties [25,26].

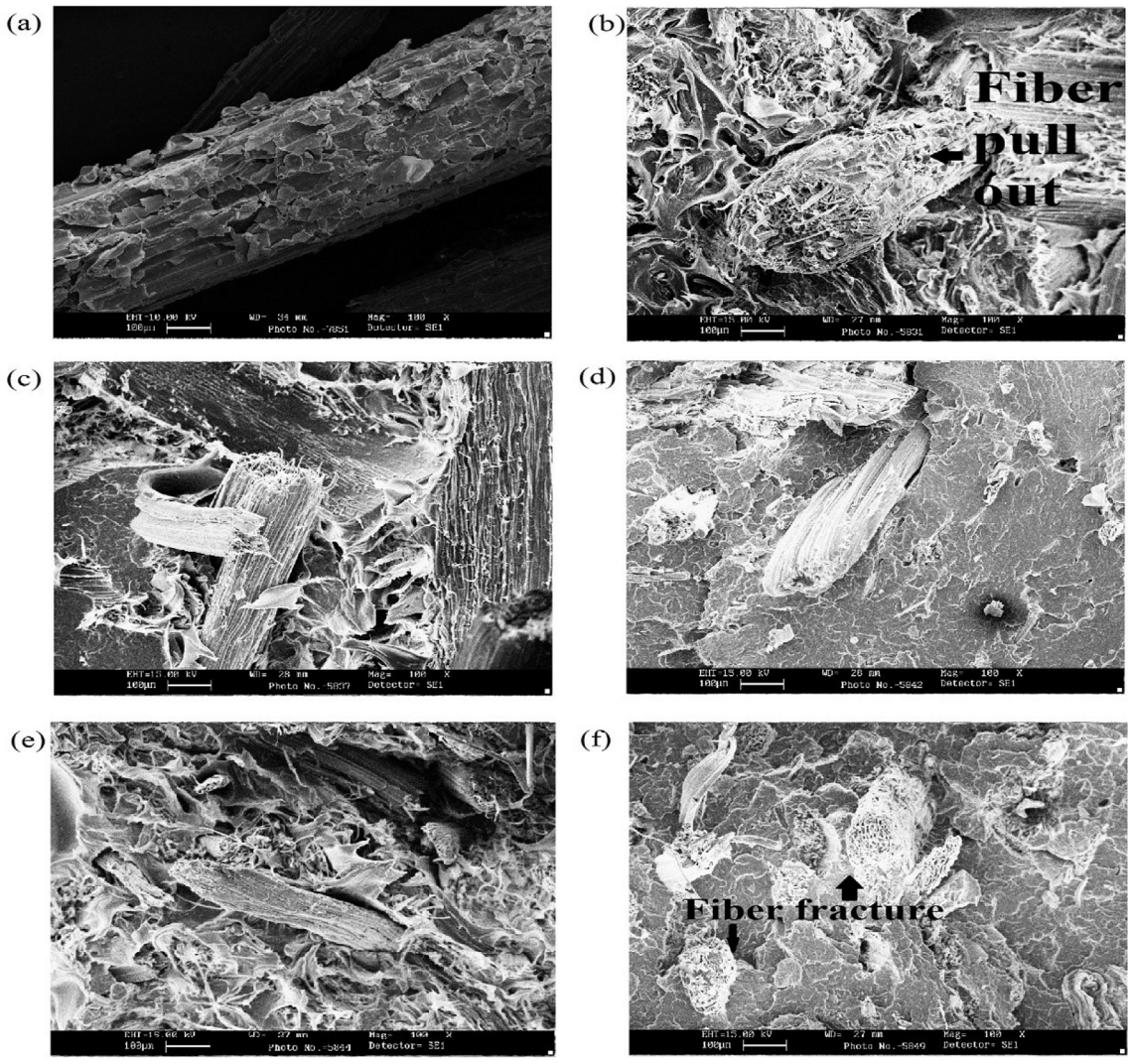

Fig. 3. SEM images of (a) Bagasse fiber (b) BFPP (c) RC1 (d) RC2 (e) RC3 (f) RC4.

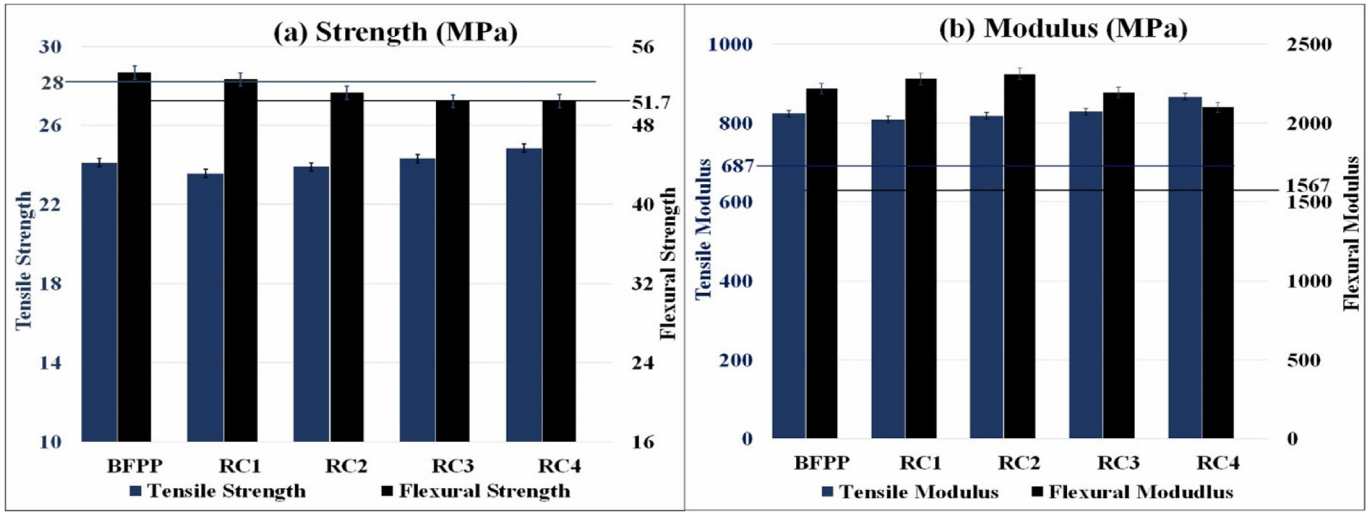

Fig. 4. Mechanical properties of fresh and recycled composites (a) Strength (b) Modulus.

3.4. Thermal behavior

The TGA curves obtained (Fig. 5a) for fresh and recycled composites show no significant change in the decomposition behavior of the material. A maximum mass reduction rate of 148.9 μg/°C, 141.9 μg/°C, 136.6 μg/°C, 136.9 μg/°C and 128.3 μg/°C was observed (between 350 °C and 360 °C) for BFPP, RC1, RC2, RC3 and RC4 specimens, respectively.

DSC curve (Fig. 5b) shows as an insignificant change in onset temperature (range between 153°C- 154 °C) as well as melting point of the composites (range between 163 °C and 165 °C). A huge reduction (~50%) in the enthalpy of fusion was observed, when bagasse fibers were reinforced in PP matrix, but thereafter, an apparent increase with each processing step was observed. Based on the enthalpy of fusion for the specimens, the crystallinity of the composites has been calculated and shown in Fig. 7. This has to be noted that the composite specimen comprises of 20% bagasse fibers by weight, which are not contributing in melting, so the enthalpy of fusion for the composite specimen can be taken 20% higher than observed one, but at the same time, this will show a similar (increasing) trend in crystalline properties.

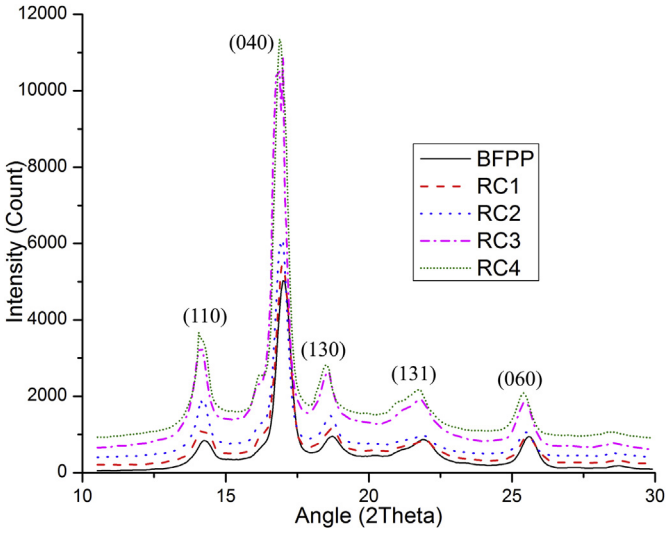

Fig. 6. XRD pattern for composite specimens.

3.5. X-ray diffraction and crystallinity analysis

Fig. 6 shows the X-ray diffraction profile and crystallographic planes for the surface of the processed composites. The penetration depth of X-rays are in the range of 10^2–10^5 nm [27], therefore solid samples were used for the characterization. The obtained curve has been analyzed on software (Origin Pro, Ver. 8.5) and area under the curve was evaluated for determining total area and crystalline area. Neither peak shift nor change in the crystallographic planes was observed but peak sharpening confirms an increase in crystallite size in the composite with each recycling step. Crystallinity by Ruland Vonk method (Eq. (2)) has been calculated and shown in Fig. 7. A slight increase was observed in the crystallinity (1.4–2.8%) with each cycle and showing a similar trend with the crystallinity calculated by DSC method. The incorporation of bagasse fiber obstructs the formation of long chains as well as discrete and inhomogeneous nucleation takes place due to the phase difference, between fiber and polymer, during consolidation, which further results in smaller crystallite and chain length.

As the interface, between fibers and matrix, is formed during incorporation stage, therefore it can be said that, further processing may result in the strengthening of the interface, which further results in better crystalline and tensile properties. This phenomenon can be confirmed with SEM images obtained for the fractured specimen, exhibiting less fiber pull out due to better interfacial bonding (Fig. 3). Crystallinity signifies the ordered region of molecular chains and these chains slide over each other when tensile load is applied. Therefore, higher crystallinity led to better elongation and load carrying capacity of the composite specimen. The increase in crystallinity can be explained by the higher mobility of the polymeric hydrocarbon chains, which is due to the reduced molecular weight of the chain as a consequence of the degradation [16,28]. This degradation allows the strained or entangled chains sections of the macromolecules to be released, and crystallization advances by the rearrangement of these unconstrained macromolecules sections [2].

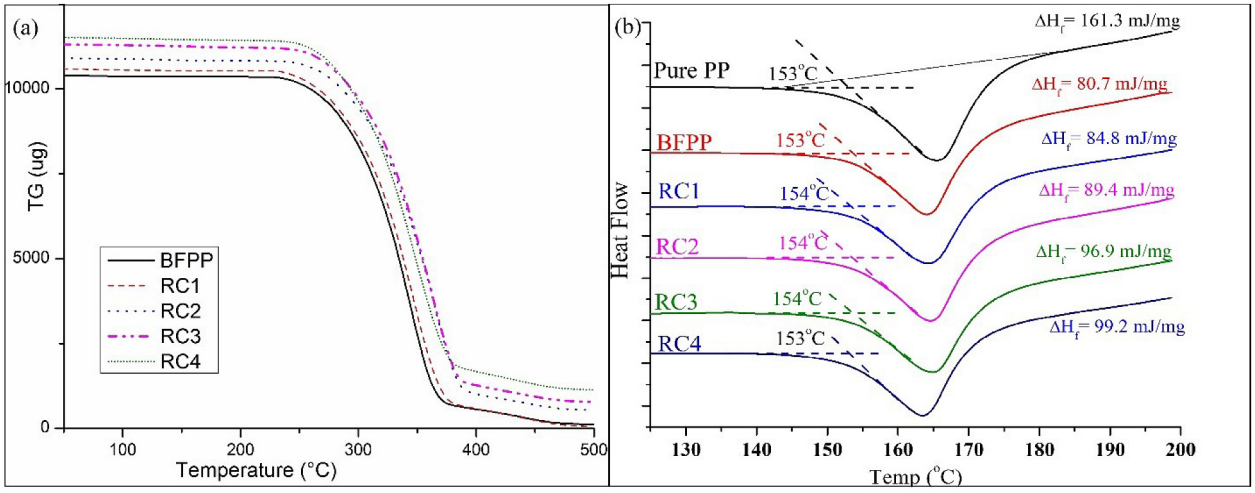

Fig. 5. (a) TGA Curve and (b) DSC curve for fresh and recycled composites.

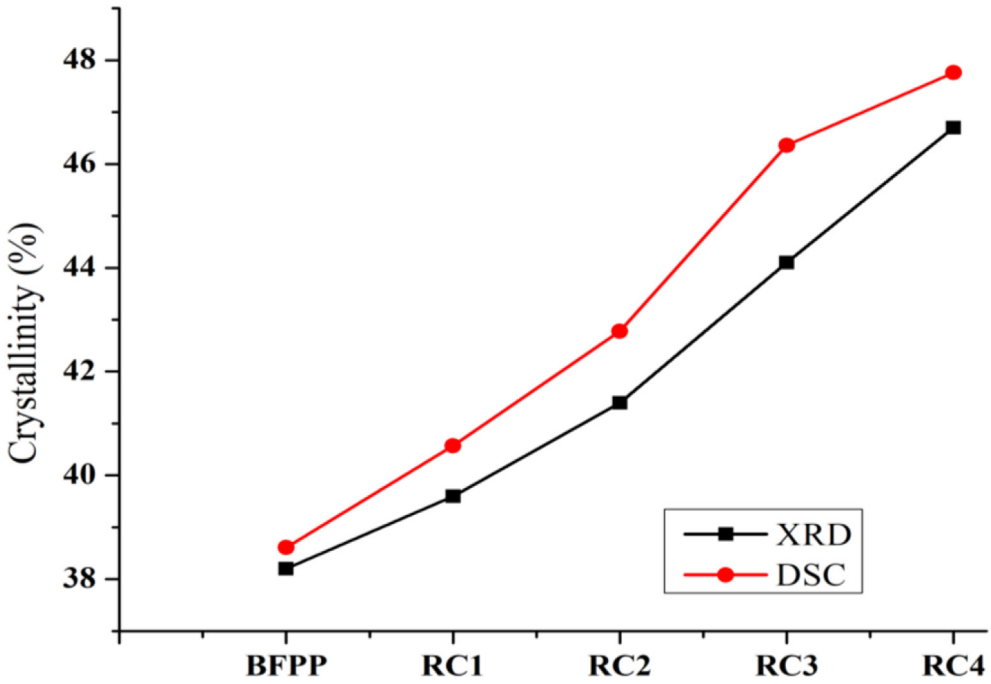

Fig. 7. Crystallinity of composite specimens by XRD and DSC methods.

4. Conclusions

Bagasse fiber reinforced polypropylene composites have been fabricated with extrusion injection molding process and the experimental investigation clearly exhibits the effects of recycling on their mechanical, aspect ratio, surface roughness and crystalline properties. Aspect ratio of fibers increases due to crushing of fiber during recycling, which further contributes to an increase in tensile strength of the composite. The increase in crystallinity led to higher elongation under tensile loads but increase in surface roughness and hardness led to reduction in flexural properties. The thermal decomposition behavior exhibits no significant change even after 5 processing cycles. The current research endeavor reveals that natural fiber based polymer composites preserve their inherent characteristics during recycling without addition of virgin material. Based on the experimental results, it can be concluded that recycled natural fiber reinforced composites have a huge potential to replace traditional plastic used in various applications.

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