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Mixing strategy and tensile strength characterization of WTR-PP composite

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ABSTRACT

As vehicles are on the rise globally, so is tire disposal when out of service. One potential recycling process is turning the waste tire into a useful composite by incorporating it with thermoplastic materials. In the present work, composite based on waste tire rubber (WTR) and polypropylene (PP) were developed using a single screw filament extruder for various concentrations of WTR (20, 40, 60, and 80% wt.). Physical and morphological characterization of WTR (425 µm) and WTR-PP composite was performed; furthermore, mechanical characterization of WTR-PP composites was also carried out under the tensile load. Morphological observation reveals that WTR-PP with low WTR content shows comparatively better distribution, maintaining the necessary strength and thermoplasticity of the composite compared to the higher WTR content in the composite. The tensile study shows that incorporating WTR with polypropylene decreased tensile strength but improved elongation at break. The macrofractography is carried out to study the failure origin and crack propagation in the WTR-PP composite.

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KEYWORDS
Waste tire rubber; polypropylene; composite; screw extruder; tensile test

Introduction

Tire rubber is a complicated blend of natural and synthetic rubbers, as well as a variety of other chemicals and construction materials. A modern car tire contains up to 25 components and over ten distinct rubber compounds. Tire rubber is usually a blend of styrene-butadiene rubber polymer, butadiene rubber, natural rubber and other additives such as zinc oxide, carbon black, etc.[1] As the tires are bulky and non-biodegradable, they consume valued space in landfills and the disposal of discarded tires has turned into a substantial environmental concern all over the world. Many abandoned tires wind up in open garbage disposal sites without being treated. The increasing number of waste disposal lands creates a severe danger to human health and a high risk of fire hazards. There are various techniques to recycle waste tire rubber. One of the methods to recycle waste tire rubber is to create composite blends of WTR with other waste materials like polypropylene with the help of an extruder machine.

WTR-PP composites are a mixture of rubber and thermoplastics. It is a high molecular weight material that surrounds rubber components forming a continuous matrix. These materials are typically low-cost common thermoplastics that can flow under certain conditions so that they can be shaped into different products. Thermoplastics can be an effective barrier to prevent moisture from reaching the composite since they shrink and swell but only absorb a small quantity of moisture. Compared to other comparable materials, polymer composite often has good durability, relatively high stiffness and strength, and low price. They may be utilized for various outdoor

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applications and are weather- and water-resistant. The recycled composite and WTR market has grown significantly in recent years, thanks mainly to the automation industries contributing to more waste tires and expanding construction which has a considerable potential to recycle waste tire rubber in many applications. High-density polyethylene (HDPE), polypropylene, and polyvinyl chloride are the three thermoplastics most frequently utilized for WTR composite.[2,3]

The extrusion process using a single screw extruder carried out in this work is one of the technologies to contribute toward sustainable waste tire recycling technologies, the most commonly used equipment for mixing and polymer processing. Extrusion is fundamental for processing composite materials. It is an essential part of the polymer processing industry, and the extrusion can be used for various complex engineering-shaped products, 3D printing, pipes, sheets, films, and other geometries. The extrusion process creates plastic products such as water bottles, automobile bumpers, and plastic plates. The polymeric material must be fed and processed using a barrel and screw system.[4,5]

Revelo et al.[6] studied WTR powder-based composite materials. They produced WTR-based tiles for various concentrations that can be used in multiple applications where impact loads are common such as floor tiles used in gymnasiums or pavement. Jeong et al.[7] investigated the mechanical properties of WTR particles containing mortar composites. Experiments were conducted on six various concentrations (0, 5, 10, 15, 20, and 25%) of samples for flexural and compressive strength. The highest compressive and flexural strengths were observed when 5% of the sand was replaced with WTR particles. Ayrilmis et al.[8] examined the successful utilization of WTR in particleboard manufacturing, where manufacturing parameters were water content and specific gravity. The finding of the work showed that the tire rubber enhanced the particleboard's water resistance due to its hydrophobic property. Xu et al.[9] studied the use of WTR powder as fillers for wood fiber composites and wood rubber composites. Morphological, Physical-mechanical properties of wood rubber composites panels were analyzed, and results showed that the use of WTR is feasible to modify the strength and viscoelastic properties of wood fiber composite furthermore, WTR addition can improve the hygroscopic stability and flexibility of panels. Garcia et al.[10] successfully developed a novel composite using WTR and rice husk using the sintering method. They analyzed the morphological and mechanical properties, such as modulus of elasticity and ultimate tensile strength for various concentrations. Ashori et al.[11] examined the utilization of WTR in hybrid plywood composite panels and studied Mechanical, physical, and acoustic properties. Overall findings showed that an increase in the WTR content is directly proportional to the improvement in physical properties and inversely proportional to the mechanical properties of the manufactured panel. Nguyen[12] recycled WTR with 3D printed composite, and experimental data showed that the overall damping properties of the 3D-printed WTR-based composite improved by about 260% when the rubber content increased from 0 to 50% wt.

This research aims to look into a way to recycle waste tire rubber (WTR) and make a novel composite material by mixing it with thermoplastic material where WTR is used as a filler material. Physical, mechanical, and thermal characterization is carried out to study the workability of WTR with thermoplastic to create new composite materials for various applications.

Experimental program

Materials

Materials used for this study were virgin polypropylene and a raw waste tire rubber material procured from a local waste tire rubber supplier as shavings of used car/truck tires containing unknown composition and non-uniform grain size. WTR powder (425 µm) was prepared by mechanical shredding, and grading was determined using sieve analysis ASTM C136 for fine and coarse aggregate.[13] Moisture content and density of WTR powder (425 µm) were evaluated using

Table 1. Characteristics properties of the materials.

Properties	Polypropylene	WTR
Color	White	Black
Density (@ 25 °C)	0.95 gm/cm^3	0.793 gm/cm^3
Melting point	170 °C	–
Water absorption (%)	0.03	0.955[14]
Thermal conductivity (W/mk)	0.2	0.32

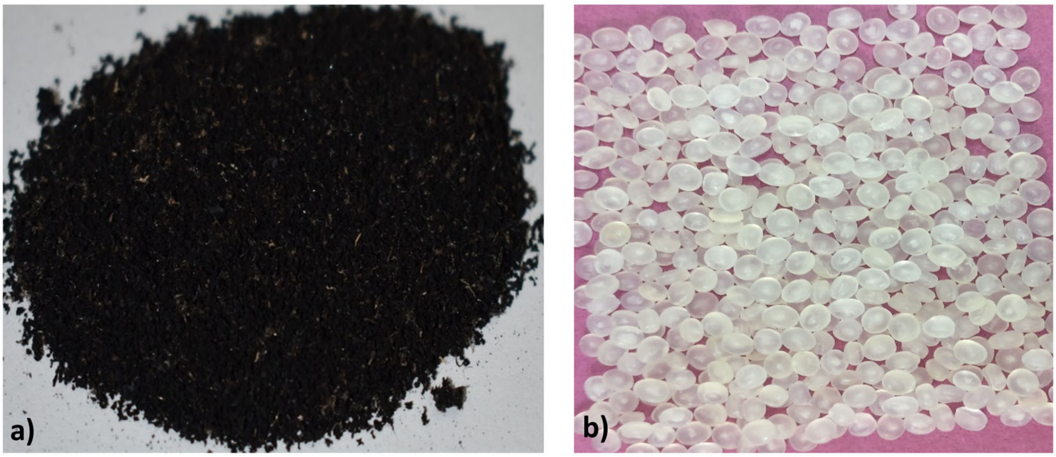

Figure 1. (a) WTR powder (grain size – 425 µm) and (b) Virgin polypropylene.

the dry oven method and density bottle experiment, and the properties of PP and WTR are reported in Table 1 (Figure 1).

Composite preparation

WTR and PP were melt blended for various WTR-PP ratios: 20/80, 40/60, 60/40, and 80/20 as mentioned in Table 2. Melt blending was performed using in-house fabricated single screw filament extruder with L/D ratio of 12 with 5 mm circular die to extract the extrudate. The extruder was operated at 50 rpm with a throughput rate of 2.467 kg/hr and a temperature profile of 170 °C (metering zone) as the melting point of PP is 170 °C. A homogenous and continuous extrudate free of air bubble was obtained however extrudates may have varied flow characteristics because of the various cooling rates in different areas of the extrudate due to shear heating induced by the molten material passing through a die or by the friction created by materials as they pass over one another, certain areas of the extrudate are hotter than others.

Voids and density deviation

The density of any composite is determined by the relative quantity of matrix and reinforcing components, and it is one of the most critical parameters influencing composite qualities. As density plays a significant role in composite materials, a water immersion test is used to determine the actual density of the developed composite. The theoretical density of the composite can be determined by the given mathematical expression.[15]

$$\rho_c V_c = \rho_f V_f + \rho_m V_m \tag{1}$$

where ρ_c, V_c, ρ_f, V_f, ρ_m, V_m are the density and volume fraction of the developed composites, filler (WTR), and matrix (PP), respectively.

Table 2. Sample codes of composites.

Sample code	WTR (% wt.)	PP (% wt.)	Actual density (gm/cm^3)	Theoretical density (gm/cm^3)	Voids (%)
WP1 (WTR20)	20	80	0.916	0.9332	1.843
WP2 (WTR40)	40	60	0.898	0.9219	2.592
WP3 (WTR60)	60	40	0.851	0.8825	3.569
WP4 (WTR80)	80	20	0.804	0.8482	5.116

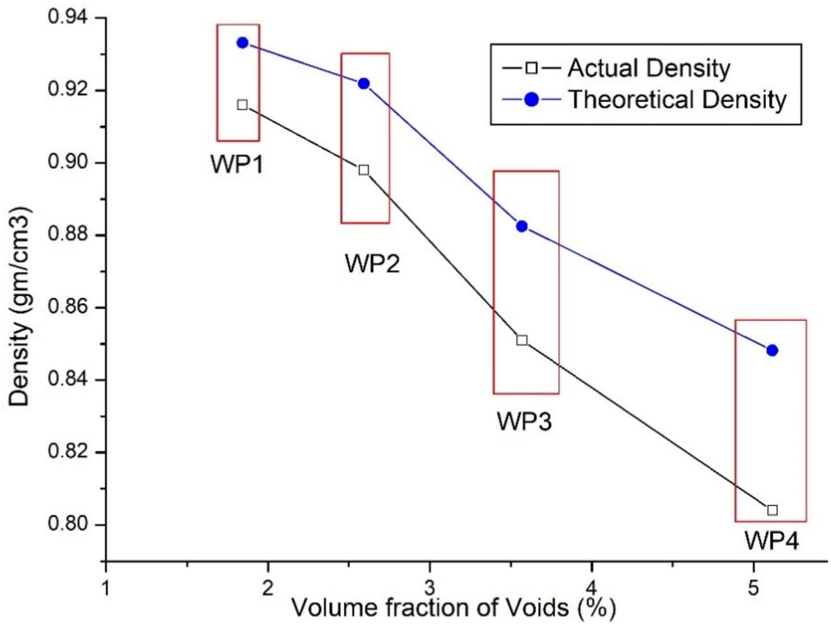

Figure 2. Density deviation and void fraction for composites of various sample.

Voids in the composites can be determined by the relation between actual density and theoretical density as given by the following relation.[16]

$$\text{Vvoid} = \frac{\rho_{th} - \rho_{act}}{\rho_{th}} \times 100 \quad (2)$$

where V_{void}, ρ_{th}, ρ_{act} is the voids volume, theoretical density, and actual density of the developed composites.

Void formation can occur for a variety of reasons, such as mechanical air entrapment during resin flow, temperature variations during the blending in a filament screw extruder, or insufficient pressure to fill the part/gaps.[17] Figure 2 shows the deviation in the actual and theoretical density of developed composite and void formation for each blended sample. As the amount of WTR increases, the voids content is also expected to increase and the same pattern is observed in the Figure 2.

Characterization of WTsR-PP composites

Samples of the WTR-PP composite were studied with a high magnification Model JSM JOEL scanning electron microscope (SEM) to examine the surface morphology. XRD patterns were acquired for each WTR-PP composite sample using a Bruker D8 Advance diffractometer. FTIR analysis was carried out using a PerkinElmer Spectrum two instrument, while TGA analysis was carried out on all the WTR-PP composites using a Shimadzu DTG-60 instrument.

Tensile tests were performed according to ASTM D 3916 on extruded WTR-PP composite rod of 5 mm diameter and a gauge length of 120 mm at room temperature for each composite sample for five repetitions on the universal testing machine (SET-T 5000 N) with 5000 N load cell at a crosshead speed of 5 mm/min. macrofractography analysis is performed to identify the failure origin.

Results and discussion

Morphology

The exceptional qualities of thermoplastic elastomers are essentially the result of their distinctive microstructure, which is made up of a continuous rigid plastic matrix with tiny elastic rubber particles spread throughout the matrix, allowing the mixture to be processed.[18] Understanding composite's mechanical performance requires an understanding of their morphology. Since brittle polymers that have been toughened by elastomers depend on a variety of elements, including the size, concentration, and distribution of the dispersed phases as well as the adhesion between the phases.[19]

SEM images were taken for WTR (425 μm) and WTR-PP samples to understand the surface morphology.

SEM image for WTR (425 μm) with various magnifications is shown in Figure 3(a). Magnification 500× reveals that the WTR is a rubbery composite of homogeneous components with uneven surfaces filled with holes and folds. Due to the presence of carbon black and other inorganic additions in the sample, which give it molar cohesion and elastic qualities, the surface morphologies of the WTR exhibit white portions with glowing edges that represent crystalline particles mixed uniformly with rubber. Whereas 2500× magnification indicates that the sample is

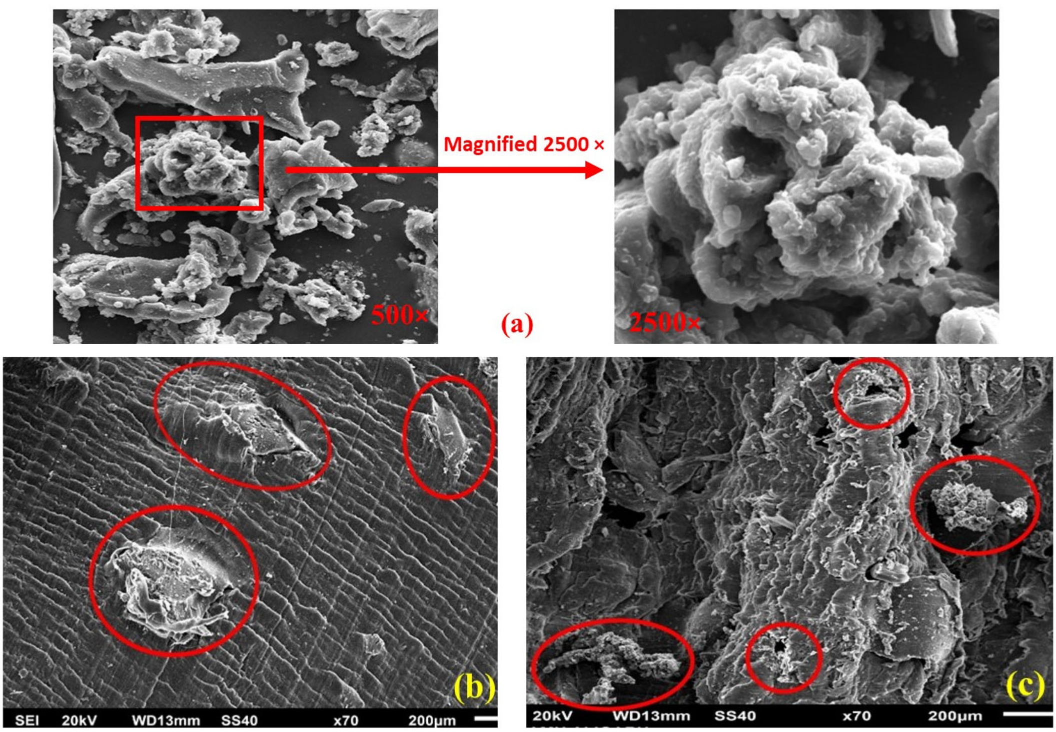

Figure 3. SEM of WTR (425 μm) and WTR-PP composites (a) WTR, (b) WP1 and (c) WP4.

in an elastic structure, with tiny particles dispersed in and between the folds in clusters, and is a carbon black nanocomposite.

Figure 3(b,c) show morphology for WP1 and WP4, respectively. As WP1 shows a continuous thermoplastic phase (darker domain) with finely dispersed and comparatively better distribution of WTR particles (bright domain), the continuity of PP (darker domain) provides the necessary strength and thermoplasticity. In comparison, the WP4 Figure 3(c) shows scattered WTR particles (bright domain) and comparatively less continuous thermoplastic phase (darker domain) indicating less strength for more WTR content composites. Similar dark domains for PP were reported in published literature.[20]

XRD analysis

XRD (Bruker D8 Advance diffractometer) was used to determine the crystallinity of the WTR sample. The atomic or molecular structure of materials can be examined using XRD. It is nondestructive and functions best with fully or partially crystalline materials. The XRD spectrum for the various composite is shown in Figure 4.

The most intense peak observed corresponds to Amorphous natural rubber where 2θ ranges from 13 to 13.8°.[21] Similar findings were reported by Johns et al.[22] few less intense peaks were observed, as shown in Figure 4. For synthetic rubber, 2θ ranges from 20 to 20.8°similarly 2θ for ZnO and Zns ranges from 34.78 to 35.4° and 41.8 to 42.6°, respectively.[21,23,24]

FTIR analysis

FTIR analysis (Fourier transform infrared) was carried out using a PerkinElmer Spectrum two instrument. FTIR spectroscopy categorizes chemical bonds in a molecule by producing an infrared absorption spectrum and provides qualitative and quantitative analysis for organic and inorganic samples. FTIR characterizations were performed from 300 to 4300 cm^{-1}, Figure 5 shows the FTIR spectra for pure WTR (425 µm) and blend composite. The intense absorption peaks visible around 1,036.14, 2918.5, and 3426.24 cm^{-1} in the samples represent the presence of the C-H bond corresponding to natural rubber and the C = O bond peak can be observed at around

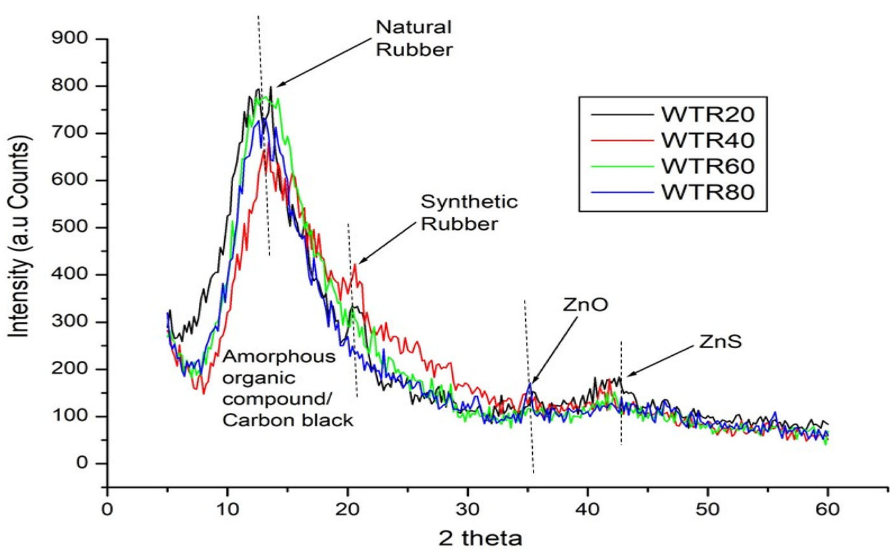

Figure 4. X-ray diffraction pattern of WTR-PP composites.

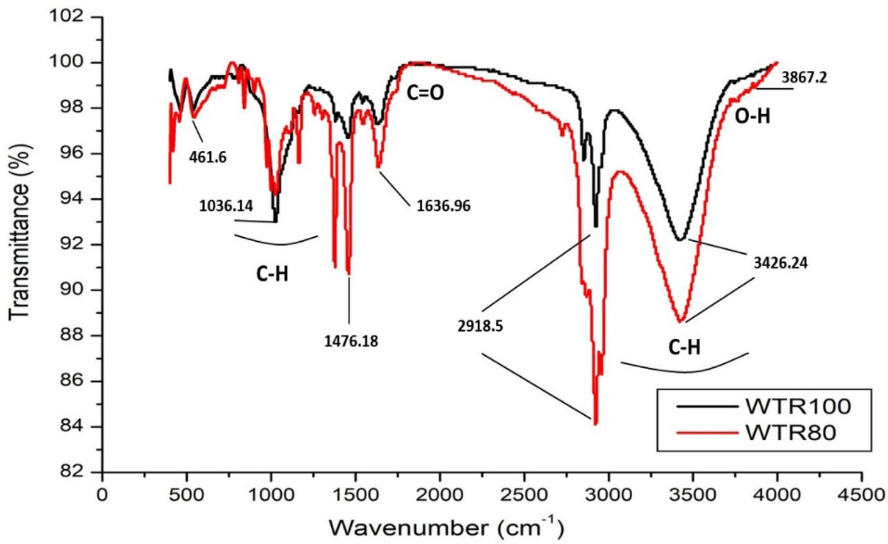

Figure 5. FTIR spectra for WTR (425 μm) and blend composite.

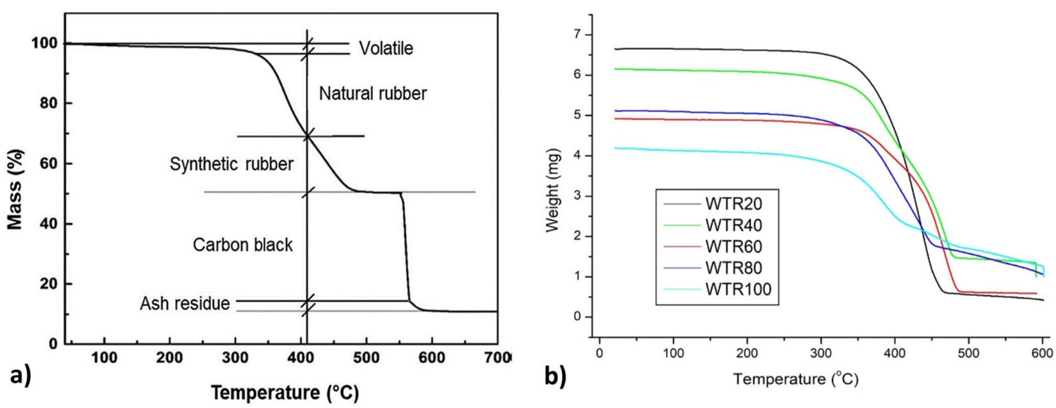

Figure 6. TGA profiles (a) Typical WTR and (b) WTR (425 μm) and WTR-PP composites.[28]

1900 cm^{-1} mainly corresponds to the aldehyde additives used during the manufacturing of tire. These findings are supported by Taleb et al.[25] and additionally, the presence of the hydroxyl function group (O–H) can be observed around 3600–3900 cm^{-1}, which is an essential part of tire manufacturing.[26,27]

TGA analysis

The thermal stability of the WTR (425 μm) and WTR-PP composites were studied using thermogravimetric analysis. All the samples had a mass of around 5 mg that was heated from 50 to 650 °C at a scanning rate of 20 °C/min under a nitrogen atmosphere. The TGA curve for the developed composites is presented in Figure 6 shows the first small weight loss (6–7%) around 300 °C corresponds to a loss of water and highly volatile materials, such as waxes, oils, low molecular weight plasticizers and antioxidants that were used for tire manufacture. The second thermal event occurred at around 400 °C, corresponding to the decomposition of natural rubber, whereas synthetic rubber decomposes at 450 °C. Carbon black and other higher boiling point

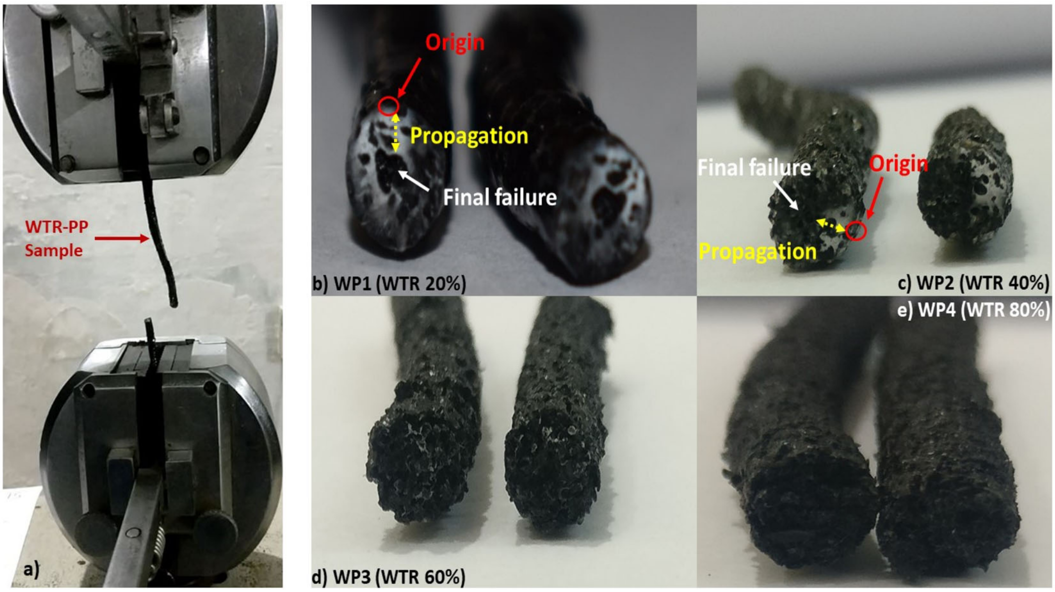

Figure 7. Tensile test: (a) universal testing machine, (b) macrofractograph of WP1, (c) macrofractograph of WP2, (d) macrofractograph of WP3, and (e) macrofractograph of WP4.

components decompose between 450 and 600 °C. Obtained thermograph for WTR-PP composite follows the typical TGA graph.

Tensile test

To compare the mechanical properties of the composite, a tensile test was performed on the WTR-PP composite of various concentrations according to ASTM D 3916 using the universal testing machine as shown in Figure 7(a). Tensile strength is given by the following expression.[29]

$$\text{Tensile strength, } S = 4\ P/\Pi D^2 \tag{3}$$

where

S = tensile strength in MPa,
P = maximum load in N
D = minimum diameter of rod in mm.

As shown in Figure 8(a), the Stress-strain graph shows non-linear behavior, indicating as the WTR percentage increases, the curve tends more toward ductile failure mode. Since the WTR material is soft and due to Polypropylene's hardness value, the curve observed a discrepancy. WTR-PP Composite with high PP/low WTR content has high-stress values but inconsistency is observed in elongation at break values for WP2, as shown in in Figure 8(b), which may be due to various reasons such as temperature variation in the extruded sample while blending in the metering section of single screw extruder or may be due to varied flow characteristics depicted by the extrudate sample as cooling rate of the sample is non-uniform in different areas of the sample.[30] Composite with higher WTR content exhibit slightly better elongation at break (%) but lower tensile strength. in Figure 8(c) shows the tensile strength graph where Sample WP1 with 20% WTR has a high tensile strength of 17.95 MPa, and for WP2, tensile strength was calculated as 9.51 MPa; tensile strength for WP3 and WP4 was 2.52 and 1.44 MPa, respectively. The tensile

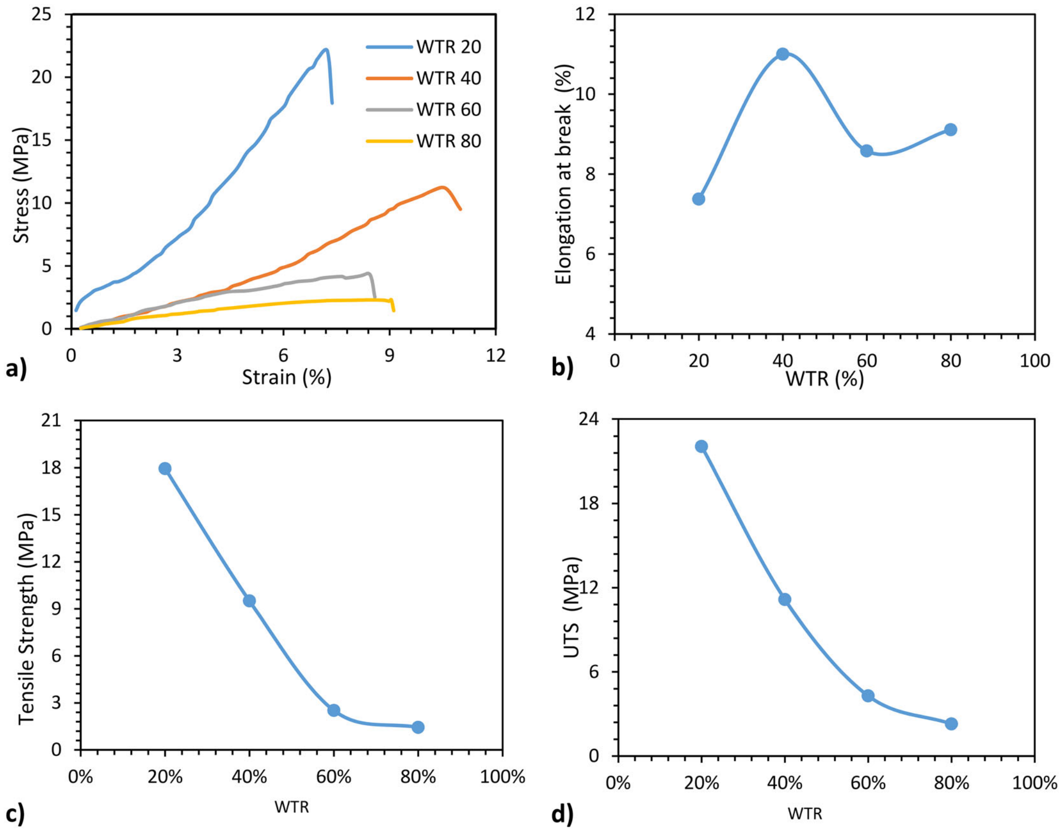

Figure 8. Tensile test (a) stress-strain curve, (b) elongation at break, (c) tensile strength and (d) UTS.

strength of Polypropylene is 34 MPa[31], upon comparing the tensile stress data of WTR-PP composites with virgin Polypropylene, it is evident from the graph that the presence of WTR content reduces the tensile strength of the sample. The more WTR less will be the tensile strength; furthermore, a similar trend is followed for ultimate tensile strength, as shown in in Figure 8(d).

Macrofractography

Fractured surfaces are shown in Figure 7 for macrofractographic analysis of the WTR-PP composites failed under tensile loads. Fracture surface of composites with low WTR content (WP1, WP2) showed three distinct regions, viz., origin (Red colour), crack propagation (yellow colour), and final failure (white colour). Significant variations in the fractographic features were noticed as WP1 and WP2 clearly showed origin, crack propagation, and failure furthermore propagation appeared to be continuous and uniform for WP1 and WP2 that helps in identifying the failure origin but as the WTR content in the sample is increased resulting in low stiffness of the sample, regions of fractures are not distinctive in WP3 and WP4 as seen in Figure 7(c,d). Similar findings on fractographic analysis and identification of origin, cracks and propagation for composite material are reported Kumar et al.[32]

Conclusion

In the present study, waste tire rubber and polypropylene were blended into WTR-PP composite using a single screw extruder at 170 °C (metering zone) operated at 50 rpm with a throughput rate of 2.467 kg/hr. The characterization of waste tire rubber powder (425 μm) and WTR-PP composites has been studied using TGA, SEM-EDS, XRD, and FTIR analysis to understand the vital aspect of the waste tire rubber powder from end-of-life tires and its applicability with thermoplastic materials. WTR-PP composites were created for various concentrations of 20, 40, 60, and 80% WTR and tensile tests were performed.

The morphological study of the WTR-PP composite shows that WP1 composite, which has lower WTR content in the PP matrix, shows the better distribution, maintaining the necessary strength and thermoplasticity of the composite as compared to the WP4. More WTR particles can successfully be used in the composite by varying processing conditions during the extrusion and the application of a compatibilizer that improves interfacial adhesion between the materials. The exploration of mechanical properties of the WTR-PP composite was done by tensile test. Based on the results obtained, it was observed that the incorporation of WTR with PP led to a significant decrease in tensile strength (up to 47%) for WP1 when compared with virgin polypropylene (34 MPa), and a similar trend was observed for WP2, WP3, and WP4. Elongation at break increased upon adding WTR with some inconsistency for WP2 corresponding to specimen fabrication error or material processing error.

This research study contributed with information about waste tire rubber and its workability with thermoplastic material to develop a novel WTR-PP composite as it is one of the most environmentally friendly ways to recycle waste tire rubber. More high-performance waste tire rubber-based composites could be effective recycling approach and a successful and ideal blend for a specific application would lead to commercialization as it can be used for many damping, acoustic and low-end thermal applications.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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