

Comprehensive Characterization of Waste Tire Rubber Powder

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Abstract Waste tire is a major environmental challenge because it takes years to degrade naturally and can release harmful pollutants into the environment, such as heavy metals, volatile organic compounds, and particulate matter. It can also contaminate soil and water, and pose a health hazard to humans and animals. Recycling waste tires is one of the most environmentally friendly ways to reduce its environmental impact. Recycled waste tire rubber (WTR) can be used to make a variety of products, such as rubber mats, asphalt, and composites. This study investigated the distinctive physical properties of waste tire rubber powder using the density bottle method and sieve analysis. The compositional, morphological, crystalline structure, functional groups, and thermal stability of WTR powder were also studied using SEM–EDS, XRD, FTIR, and TGA, respectively. Results showed that the grain size distribution was relatively uniform, with a gradual transition from fine to coarse. The moisture content of the powder was 0.964%. FTIR and DSC analyses showed that natural rubber and synthetic rubber were the main components of waste tire rubber powder. Compositional analysis showed the presence of iron (Fe), which corresponds to the steel belt used in tire manufacturing.

Keywords WTR · Waste tire rubber · Characterization · TGA · XRD · SEM–EDS

Abbreviations

ELT	End-of-life tires
SBR	Styrene-butadiene rubber
M_1	Weight of empty container
M_2	Weight of the container with moist sample
M_3	Weight of container with dried sample
W	Water content (%)
w_i	Total weight of the particle
G_s	Specific gravity
SDD-EDS	Silicon drift detectors- energy dispersive spectroscopy
TGA	Thermogravimetric analysis
XRD	X-Ray diffraction
FTIR	Fourier Transform infrared spectroscopy

Introduction

Tires are essential components used in various forms of transportation and machinery. They provide the necessary grip, traction, and stability for vehicles and equipment to operate effectively on different surfaces. Tires are typically made of a combination of natural and synthetic rubber, reinforced with materials like steel and textile fibres, and enhanced with chemical additives to optimize their performance and durability. Tires that have reached the end of their useful life and are no longer suitable for safe road use are called as waste tire rubber or ELT (end-of-life tires) [1]. These tires pose significant environmental challenges due to their sheer volume and slow decomposition rates therefore, proper management and recycling of waste tire rubber are crucial to mitigate their adverse impact on the environment [2, 3].

The tire industry has experienced substantial growth due to increased vehicle ownership and industrial development

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in many regions. The exact quantity of global tire waste is challenging to determine precisely due to various factors such as inadequate data, illegal dumping, and unregulated disposal. Some researchers estimate that around 1.5 billion tires are manufactured worldwide annually, with approximately 300 million produced in the United States [4]. As per the reports of The World Business Council for Sustainable Development (WBCSD), it estimates that over one billion tires are discarded annually. This figure includes tires from various sources such as passenger vehicles, trucks, motorcycles, bicycles, and industrial equipment [5–7]. The Central Pollution Control Board of India conducted a study on waste tire management in 2019 and Reports says that India generates about 1.3 million tons of End-of-life tires per annum. Only 450,000 tons is recycled by the formal sector. India's rubber tire waste generated accounts for about 6–7% of the worldwide [8].

Tires are complex products composed of various materials and chemicals with specific functions. Their composition varies depending on tire type, intended use, and manufacturer. Typically, tires consist of rubber polymers (natural or synthetic) for elasticity, flexibility, and durability. Carbon black is used as a reinforcing filler to strengthen the rubber and improve wear resistance. Tires also incorporate various chemical additives to improve performance, such as antioxidants, antiozonants, plasticizers, curatives, accelerators, and vulcanizing agents. Fabrics and textiles (polyester, nylon, or rayon) are often included in tire construction as plies to provide strength, stability, and reinforcement [9–11].

ELTs are not easily repaired hence considered ideal for recycling. By using a variety of characterization techniques, researchers can gain a comprehensive understanding of WTR powder properties that can then be used to develop new and innovative ways to recycle waste tire rubber into useful products. The most common techniques to characterize waste tire rubber powder include particle size analysis, density measurement, surface morphology analysis, chemical composition analysis, thermal stability analysis, and mechanical properties analysis. These techniques provide information about the physical and chemical properties of the rubber powder, which can be used to develop processing methods, design products, and predict performance.

Fernandez et al. [12] studied physicochemical characterization of discarded tire rubber powder. Thermal analysis of sample powder at temperatures from 0 to 650 °C showed two major weight loss events. The first event below 350 °C, shows 16% weight loss corresponding to the loss of highly volatile matter such as plasticizers, oils, waxes, and antioxidants. The second event around 350–500 °C shows 52% weight loss corresponding to the decomposition of medium volatile matters such as elastomers, curing agent, processing agents, etc. Carbon black and other substances with high boiling point start decomposing above 500 °C. XRD result

shows larger peaks corresponding to presence of ZnO in waste tire rubber powder. Euniza et al. [13] examined the chemical properties of waste rubber tire granules. Authors investigated the relation between temperature and the mineralogical compositions of rubber tire granules to study its viability to use as a substitute for aggregates in concrete mixes. SEM analysis was performed to study morphology of WTR granules. The SEM analysis revealed that the primary constituents of waste tire rubber granules are zinc (Zn), carbon (C) and magnesium (Mg). Chemical composition shows styrene-butadiene rubber (SBR) as major component of WTR granules. Bekhiti et al. [14] studied the properties of waste tire rubber (WTR) powder and reported that its density is 0.83 gm/cm³. Elemental analysis of the WTR powder revealed that it contains 54% rubber, 29% carbon black, 2% textile, 1% zinc oxide, 1% sulfur, and 13% additives where rubber is an intricate blend of elastomers, including polybutadiene, polyisoprene, and styrene-butadiene. Furthermore, moisture content of WTR powder is less than 3%. Fusser et al. [15] studied composition by weight in tire rubber and reported the presence of Natural rubber 40%, SBR 30%, Butadiene rubber 20%, Butyl and halogenated butyl rubber 10%. Findings shows that tire rubber contains high levels of extractable organic zinc (E-Zn). Kumata et al. [16] conducted a survey on the composition of tire tread, and their analysis showed that the bulk of tire tread (40–60%) is made up of various rubbers, including styrene-butadiene rubber (SBR), natural rubber copolymers, isoprene rubber, butadiene rubber, neoprene rubber, nitrile rubber, and polysulphide rubber. Warner et al. [17] studied tire material sampling and chemical analysis. Elemental findings of the investigation showed the presence of various materials such as barium, chromium, copper, nickel, lead, antimony, strontium, vanadium, zinc, etc. Fabrizio et al. [18] studied the direct molding of rubber granules and powders from tire recycling. The findings on density variation are reported in the table. The results show that the density of rubber plates made from waste tire rubber decreases as the granule and buffing size increases. This is because the buffing process breaks down the rubber granules into smaller pieces, which reduces the overall density of the material. Detailed density variation is reported in Table 1.

Table 1 Density of rubber plate [18]

Granules/buffing	Density (gm/cm ³)
Granulate 0–0.5 mm	1.18
Granulate 0.5–2 mm	1.10
Granulate 2–3.5 mm	1.08
Buffing 0–0.8 mm	1.07
Buffing 0.8–3 mm	1.05

The aim of this research is to comprehensively examine the fundamental properties of raw waste tire rubber powder through various characterization techniques to provide insight on WTR material properties and provide basis for workability and feasibility of recycling waste tire rubber powder into novel composites that can be used for low end thermal insulation and impact related application, ultimately contributing to sustainable materials development and waste tire recycling.

Experimental

Materials

The materials used for this study was raw waste tire rubber shavings from discarded tires, obtained from a local waste tire rubber trader in India. The composition and grain size of the rubber was unknown, and non-uniform as shown in Fig. 1a. The following sections illustrate the comprehensive qualitative and quantitative characterization of the WTR powder.

Methods

WTR powder sample was obtained after the careful sieving of raw waste tire rubber shavings, selecting the retained particulate material in sieves with meshes of 425 μm . the sample of waste tire rubber powder with a particle size of 425 μm underwent a series of essential assessments for its physical properties. These assessments included measuring the moisture content using the dry oven method and determining the specific gravity through the density bottle method. Furthermore, a comprehensive examination of the waste tire rubber powder (425 μm) was carried out which included a detailed morphological analysis using scanning electron microscopy with energy-dispersive X-ray

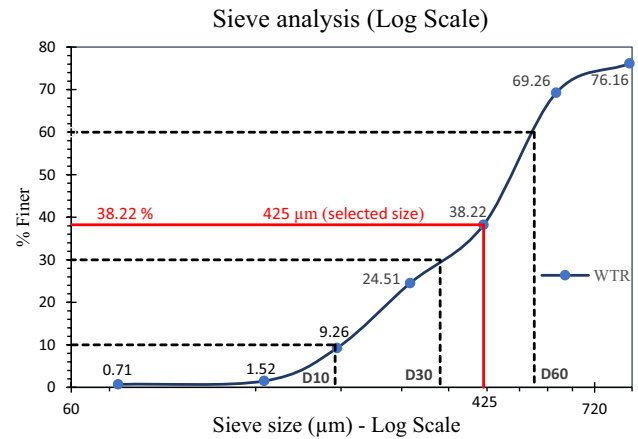


Fig. 2 Particle size distribution of WTR

spectroscopy (SEM–EDS), shedding light on its physical structure and elemental composition. Furthermore, the thermal stability of the WTR powder was carried out using thermogravimetric analysis in a nitrogen gas atmosphere. In addition, the crystalline nature of the waste tire rubber was explored using X-ray diffraction (XRD) tests. Moreover, Fourier Transform infrared (FTIR) characterization was carried out to examine the functional groups and covalent bonds present in the waste tire rubber powder.

Results and Discussion

Physical Properties

Sieve Analysis

The ASTM C136 standard for fine and coarse aggregate was followed in determining the particle size distribution of the waste tire rubber powder. [19]. Figure 2 shows the



Fig. 1 **a** Uneven raw Waste tire rubber **b** waste tire rubber powder (Grain size—425 μm)

grain size distribution of the waste tire rubber. Approximately 645 g of shredded waste tire rubber shavings were passed through a series of sieves with varying mesh sizes, ranging from 850 to 75 μm . Sample calculation for sieve analysis of WTR (425 μm) is presented in the Table 2.

Uniformity coefficient (Cu) is given by the following formulae:

$$\begin{aligned} \text{Cu} &= \frac{D_{60}}{D_{10}} \\ &= \frac{540.7}{210} \\ &= 2.5748 \end{aligned}$$

Coefficient of curvature (Cc) is given by the following formulae:

$$\begin{aligned} \text{Cc} &= \frac{D_{30}^2}{D_{60} \times D_{10}} \\ &= \frac{345.77^2}{540.7 \times 210} \\ &= 1.0529 \end{aligned}$$

D10, D30, and D60 signify the particle sizes below which 10%, 30%, and 60% of the particles in a sample are finer, respectively, aiding in understanding the sample's finer or coarser fractions. Additionally, the uniformity coefficient (Cu) indicates the range of particle sizes, while the coefficient of curvature (Cc) assesses the shape of the particle size distribution curve.

A uniformity coefficient (Cu) of 2.5748 indicates that the waste tire rubber granules have a wide range of grain sizes, with a significant proportion of fine (small grains) and coarse (large grains). The Coefficient of curvature (Cc) of 1.0529 indicates that the grain size distribution is relatively uniform, with a gradual transition from fine to coarse. A similar finding for waste tire granulated rubber has been reported in the literature aligning with the standard particle size distribution curve [20–22].

Table 2 Grain size distribution of raw WTR

Sieve size (μm)	Weight retained (gm)	Cumulative weight retained	Cumulative % weight retained	% Finer
850	153.8	153.8	23.8	76.16
600	44.5	198.3	30.74	69.26
425	200.2	398.5	61.78	38.22
300	88.4	486.9	75.49	24.51
212	98.4	585.3	90.74	9.26
150	49.9	635.2	98.48	1.52
75	5.2	640.4	99.29	0.71

The grain size distribution analysis of waste tire rubber is shown in Fig. 2 and data shows that the WTR granules have a non-uniform particle size distribution. Most of the granules (61.78%) have a particle size of 425 μm or less. WTR powder sample of 425 μm size was selected for further tests and experiments as shown in Fig. 1b. As 425 μm is a common particle size for WTR powder and there is existing research on its properties. The analysis results can be compared to published research to validate the findings.

Dry Oven Method It is a simple laboratory test, the most widely adopted method following the ASTM D2216, was used to determine the water content in material. WTR powder (425 μm) sample is taken for oven dry method, a clean container is weighed (M_1) and the moist sample is collected in the container and weighed accurately (M_2) and then container is placed in the oven maintained at temperature at 105 $^\circ\text{C}$ for 3 h, when container is cooled down at room temperature it is weighed again (M_3).

Where weight of container $M_1 = 36.2$ gm, weight of container with moist sample $M_2 = 55.993$ gm, weight of container after drying the sample $M_3 = 55.804$ gm

Water content is given by the following formula;

$$\begin{aligned} W &= \frac{M_2 - M_3}{M_3 - M_1} \times 100 \\ &= \frac{55.993 - 55.804}{55.804 - 36.2} \times 100 \\ &= 0.964\% \end{aligned}$$

Density Bottle Method The density and specific gravity of the solid were determined through the application of the density bottle method in accordance with ASTM D854. A density bottle is a small glass bottle with a fixed volume of liquid and a glass stopper. The glass stopper features a slender aperture. When the bottle is filled with liquid and sealed with the stopper, any surplus liquid ascends through the hole and flows out. As a result, the bottle contains the same liquid volume with each filling.

WTR (425 μm) sample is taken for the experiment; an empty density bottle is weighed (W_1), and then approx. 3 gm dried WTR sample is collected into a density bottle and weighed (W_2) and then distilled water is filled in the density bottle filled with dried material and weighed (W_3) and the density bottle filled with 100% distilled water is weighed (W_4).

Where weight of density bottle (W_1) = 29.858 gm, weight of density bottle with dried sample (W_2) = 32.783 gm, weight of density bottle with dried sample and distilled water (W_3) = 80.108 gm, weight of density bottle with 100% distilled water (W_4) = 80.862 gm, Specific gravity G_s is given by the following formula;

$$G_s = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)}$$

$$= \frac{32.783 - 29.858}{(32.783 - 29.858) - (80.108 - 80.862)}$$

$$= 0.795$$

Other calculated physical properties of WTR powder are reported in Table 3.

Morphological and Elemental Analysis

For the morphological and compositional study of the WTR material a CARL ZEISS EVO 50 scanning electron microscope configured with a Bruker SDD-EDS detector was used. SEM equipped with energy dispersive x-ray microanalysis can perform surface morphology and elemental analysis and provides the micro-texture details and compositional details of the sample. This procedure was based on ASTM E1508, "Standard guide for quantitative analysis by energy-dispersive spectroscopy".

SEM Analysis

SEM images were studied for WTR (425 μm) at various magnifications to understand the surface morphology, as shown Fig. 3. SEM images shows that the sample is a rubbery composite made up of components characterized by

uneven surfaces containing voids and folds, attributed to the inclusion of carbon black and other inorganic additives. These additives contribute to its molecular cohesion and elastic properties. Additionally, SEM images display bright areas with illuminated borders, signifying the presence of crystalline particles blended within the rubber. SEM image at a magnification of 1000 \times as shown in Fig. 3c, shows an elastic structure, with tiny particles clustered within and between the folds.

EDS Analysis

EDS analysis were carried out for WTR (425 μm) to study the microanalysis spectrum of the components as shown in Fig. 4. EDS images gives different peaks indicating the presence of elements such as C (carbon), Mg (magnesium), Al (aluminium), Si (silicon), K (potassium), Fe (iron), Zn (zinc), Mo (molybdenum), etc. The numerical result of the EDS quantitative microanalysis (spectrum 5) shows a higher percentage of Carbon (89%) present in the sample as shown in Fig. 4a, most likely due to an electron beam spilling on the rubber surface, generating X-rays from the carbon present in the WTR. Another EDS microanalysis (spectrum 11) shows the presence of Fe in the sample as shown in Fig. 4b, most likely due to the iron thread/steel belt used while manufacturing the tire. Similar data is reported by M.N. Siddiqui et al. [23].

Table 3 Characteristic property of WTR (425 μm)

Property	Experiment	Expression	Value
Moisture content	Oven dry method	$W = \frac{M_2 - M_3}{M_3 - M_1} \times 100$	0.964%
Specific gravity	Density bottle experiment	$G_s = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 - W_2)}$	0.795
Bulk unit weight	Density bottle experiment	$\gamma_t = \frac{\text{weight of solid}}{\text{Total volume}}$	0.303 gm/cc
Density	Density bottle experiment	$\rho = \frac{\text{mass}}{\text{volume}}$	0.795 gm/cc

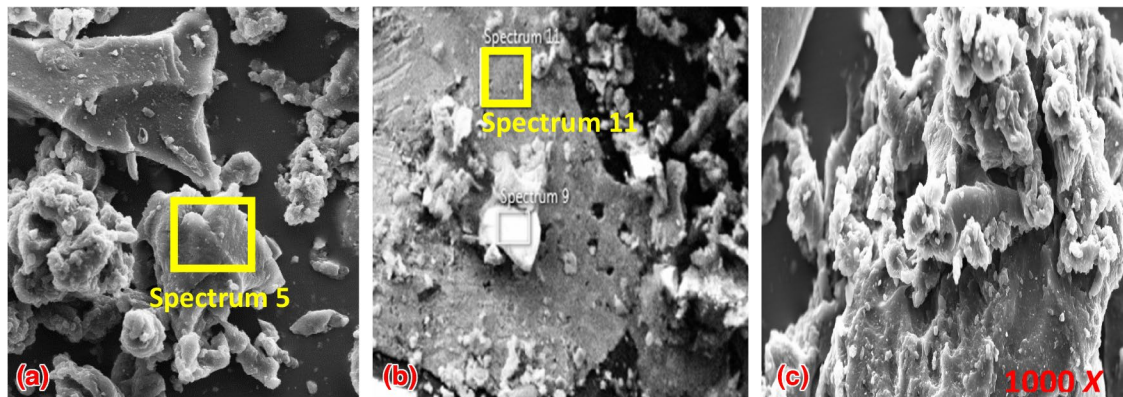
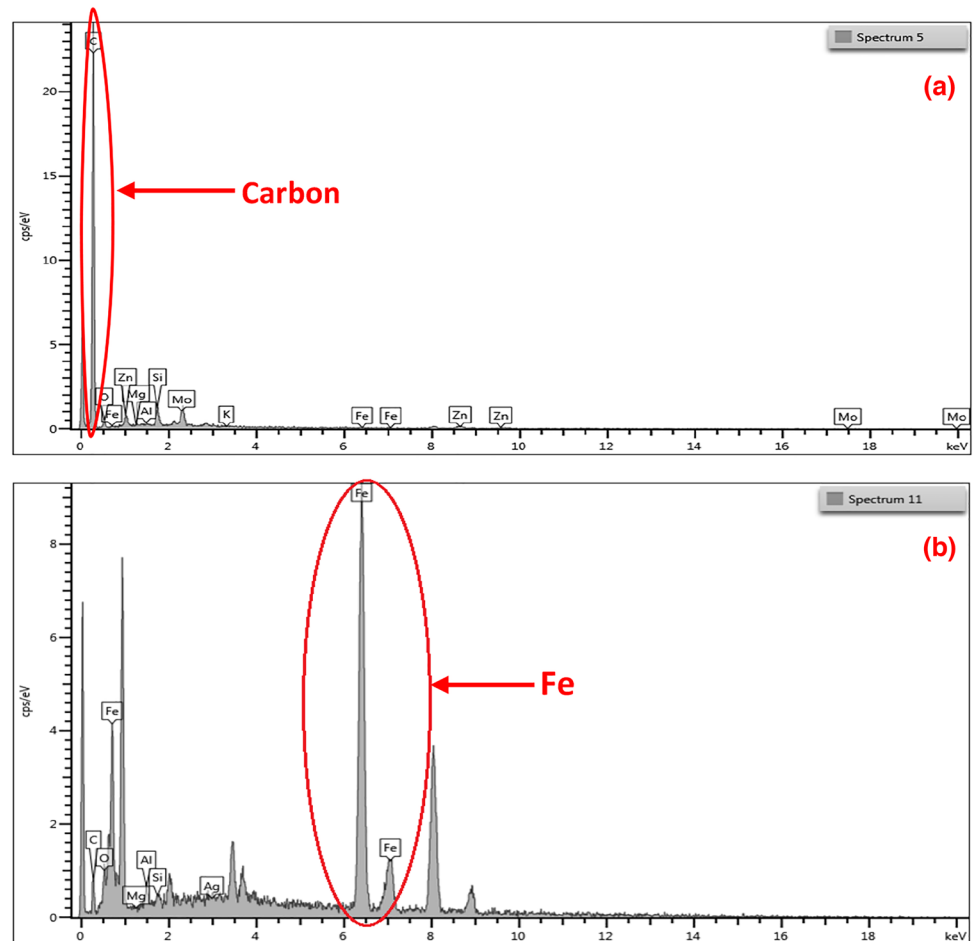


Fig. 3 SEM Images of WTR powder **a** Spectrum 5, **b** Spectrum 11 **c** Magnification of 1000 \times

Fig. 4 EDS analysis **a** Spectrum 5, **b** Spectrum 11

Although quantitative analysis of energy-dispersive spectroscopy (EDS) data is not very reliable, it can provide a qualitative indication of the composition of the sample. Therefore, the results of the EDS qualitative analysis for spectra 5 and 11 are reported in the Table 4.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a fundamental method for studying the thermal behaviour of materials. This method involves exposing the sample to a controlled temperature program while measuring the substance's mass as a temperature function. The resulting Δm versus T curve reveals information about thermal stability and composition of the initial sample and residue if any. WTR sample usually consists of a volatile substance, natural rubber, synthetic rubber, carbon black, and

ash residue, although the percentage of each component may vary from sample to sample.

The thermal stability of the waste tire rubber powder was examined according to ASTM D6370 standard ("Standard test method for rubber-compositional analysis by thermogravimetry") using thermogravimetric analysis. A 5 mg sample of WTR powder underwent a heating process, gradually increasing from 50 to 850 °C at a rate of 20 °C per minute under a nitrogen atmosphere. The TGA analysis graph, as depicted in Fig. 5a illustrates the weight loss patterns. The initial weight reduction (6.1%) was observed at approximately 300 °C, attributed to the evaporation of moisture and highly volatile substances. These substances include low molecular weight plasticizers, oils, waxes, and antioxidants commonly used in tire manufacturing. The second thermal transition occurred around 400 °C, corresponding to the decomposition of

Table 4 EDS qualitative microanalysis

Element	C	O	Mg	Al	Si	K	Fe	Zn	Mo	Ag
Weight (%)—spectrum 5	89.15	4.59	0.04	0.04	1.03	0.00	0.18	1.83	3.14	—
Weight (%)—spectrum 11	12.42	3.48	0.00	1.21	0.52	—	81.92	—	—	0.45

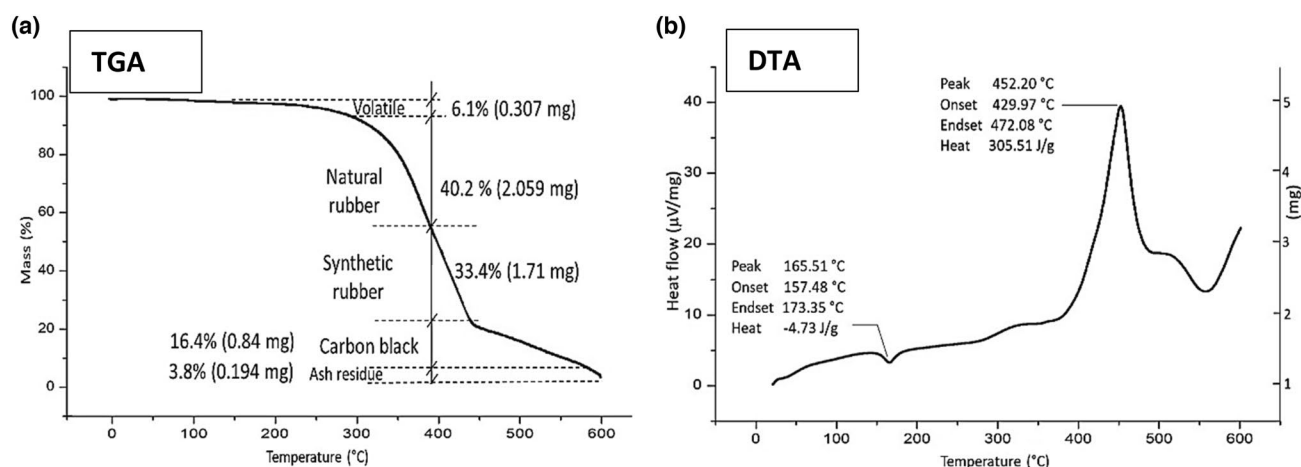


Fig. 5 TGA and DTA profile for WTR powder (425 µm)

natural rubber, while synthetic rubber decomposition took place at 450 °C. Subsequently, components with higher boiling points, such as carbon black, underwent decomposition in the temperature range between 450 and 600 °C as shown in Fig. 5a. Similar findings were reported by Ubaidullah et al. [24] and Ming-Yen [25].

The DTA curve in Figure Fig. 5b reveals specific temperature events. At 160 °C, there is an endothermic peak, which corresponds to the melting of crystalline domains of volatiles [26]. Additionally, an exothermic event observed at 450 °C corresponding to oxidation of synthetic rubber during thermal decomposition, a similar exothermic reaction was also reported by Kaiser et al. [27] which represents the synthetic rubber decomposition consistent with the polymer chains breaking during the process.

Crystallinity of Waste Tire Rubber Sample

XRD analysis, performed using a Bruker D8 Advance diffractometer according to ASTM D5357 was used to determine the crystallinity of the WTR sample. XRD is a non-destructive technique capable of revealing the atomic or molecular structure of materials. XRD is most effective when investigating materials with a fully or partially crystalline structure. The spectrum for WTR shows five crystalline peaks as shown in Fig. 6. The most intense peak observed at $2\theta = 13.03^\circ$ which corresponds to Amorphous natural rubber where $2\theta = 13.03^\circ$ [28], similar finding reported by Johns et al. [29]. A less intense peak was observed at $2\theta = 23.07^\circ$, which corresponds to synthetic rubber, two small peaks were observed at $2\theta = 35.02^\circ$ and $2\theta = 56.41^\circ$ which corresponds

Fig. 6 X-ray diffraction pattern of WTR powder (425 µm)

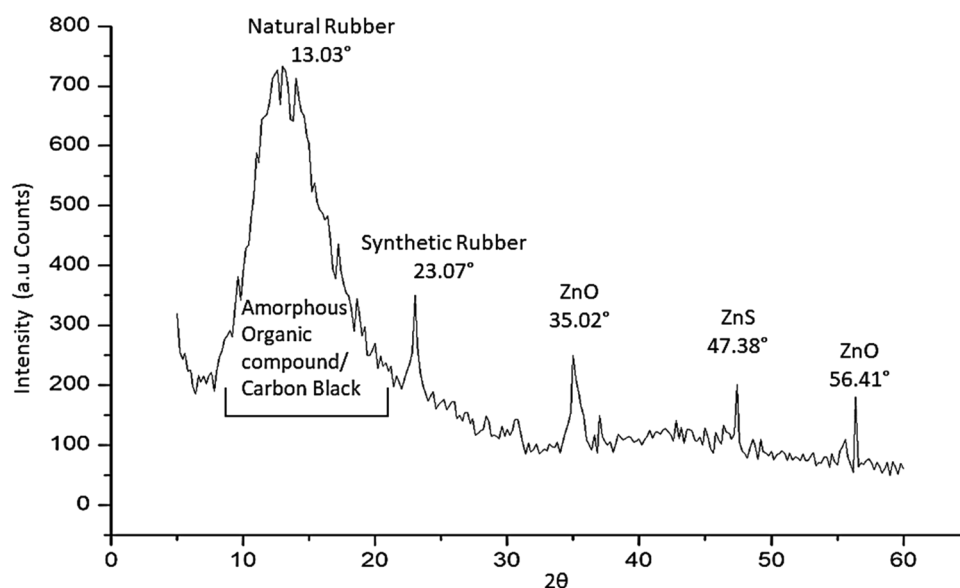
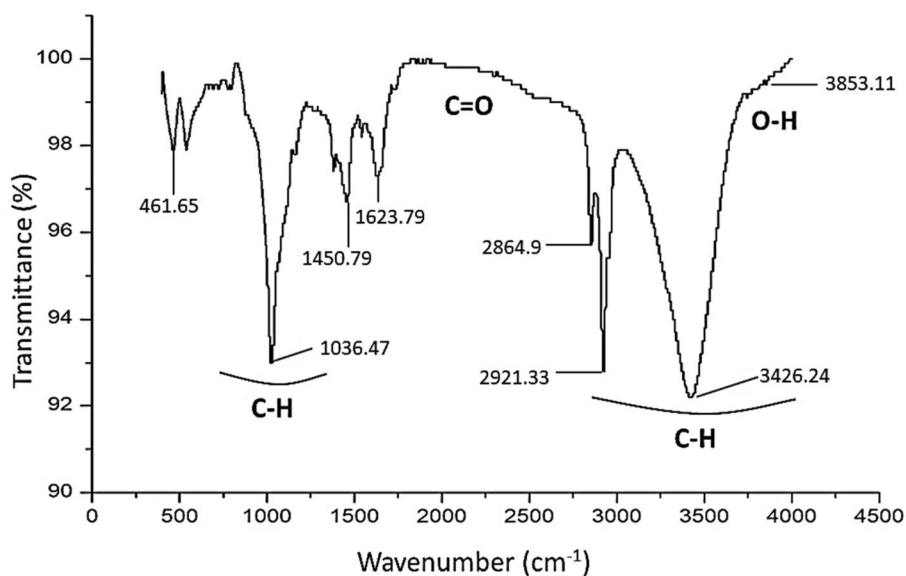


Fig. 7 FTIR spectra for waste tire rubber



to ZnO, and another small peak at $2\theta = 47.38^\circ$ corresponds to ZnS [28, 30, 31].

FTIR

Fourier transform infrared spectroscopy (FTIR) is an analytical tool employed to find functional groups and describe information about covalent bonds within molecules. FTIR accomplishes this by generating an infrared absorption spectrum, providing both quantitative and qualitative assessments of both organic and inorganic samples.

The FTIR spectra was recorded on a Perkin Elmer spectrometer in the range of $300\text{--}4300\text{ cm}^{-1}$ for waste tire rubber powder according to ASTM E168-06 standard. In Fig. 7, the FTIR spectra of WTR are depicted. Notably, there are intense absorption peaks observed at $1,036.47\text{ cm}^{-1}$, 2921.33 cm^{-1} and 3426.24 cm^{-1} in the sample. These peaks are indicative of the presence of the C–H bond, which corresponds primarily to natural rubber. Additionally, an absorption peak related to the C=O bond is noticeable at around 1900 cm^{-1} , largely attributed to the aldehyde-based additives used in tire manufacturing. These findings are supported the research findings of Taleb et al. [32] and Dubkov et al. [33]. Furthermore, the FTIR analysis reveals the presence of hydroxyl functional groups (O–H) within the range of $3600\text{--}3900\text{ cm}^{-1}$, which is an essential part in the tire manufacturing process [34, 35].

Conclusion

In the present study, the characterization of waste tire rubber powder ($425\text{ }\mu\text{m}$) has been studied using various techniques providing insights into its relevant physicochemical

properties. The results indicate that WTR powder exhibits low moisture content, measuring at only 0.96% that reveals the hydrophobic nature of WTR powder which can be useful in oil–water separation application. Furthermore, the analysis reveals that WTR powder is characterized by its low density, revealing ability to deform plastically and absorb energy. These attributes can be useful for developing lightweight and impact-resistant composites. SEM analysis confirms the rubbery and elastic nature of WTR powder made up of uniform components with uneven surfaces. The presence of carbon black and other inorganic additives in the sample gives it strength and elasticity that makes it useful for tensile applications. We concluded that these findings would be helpful in recycling of waste tire rubber and in development of waste tire rubber-based composite materials for variety of applications. Through effective waste tire rubber recycling, a more sustainable and eco-friendly approach for waste tire rubber recycling can be adopted.

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Declarations

Conflict of interest The authors state that they do not have any known financial, non-financial competing interests or personal associations that may have impacted the research presented in this paper.

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