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# EVALUATING MECHANICAL AND THERMAL PROPERTIES OF CONSTRUCTION EPOXY/TIRE PYROLYTIC POWDER COMPOSITE

## OCENJEVANJE MEHANSKIH IN TOPLOTNIH LASTNOSTI GRADBENEGA EPOKSIDNEGA KOMPOZITA Z OJAČITVIJO IZ PIROLITNEGA PRAHU ZA PNEVMATIKE

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In line with global sustainability objectives, the incorporation of manufacturing byproducts into various materials to develop composite materials not only aims to enhance performance and reduce the costs of raw materials, but also promotes environmental responsibility in manufacturing processes. This investigation used different weight fractions (0, 1, 2, 3 and 5) % of micro-sized tire pyrolytic powder (TPP) for reinforcing a particular epoxy resin designed for construction purposes. Using the hand lay-up technique, composite specimens were fabricated and examined to evaluate the influence of TPP as a reinforcing phase on the thermal and mechanical characteristics of the epoxy. Particle size analysis, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and Fourier transform infrared spectroscopy (FTIR) were performed to characterize TPP. Furthermore, tests such as Shore D hardness, tensile strength, three-point bending strength, impact strength, and thermal conductivity were conducted to assess enhancements in the composite's performance. The test results show that the mean particle size of TPP was 1.27 μm, with a narrow size variation of 0.005 μm. EDX and FTIR tests show that TPP consists of a wide variety of metal oxides added to carbon black. The findings also demonstrate significant improvements in tensile and flexural strength, by roughly 87.5 % and 72.2 %, respectively, compared with non-reinforced specimens, with the optimal performance at 3 *w*/% TPP. Beyond this TPP concentration, the strength began to decline. The hardness, impact strength and thermal conductivity were found to increase markedly at 5 *w*/% TPP, by approximately (14.8, 350 and 86.5) %, respectively,.

Keywords: pyrolysis process, end-of-life tires, composite materials, sustainability, bending strength

Z vključevanjem stranskih produktov v različne kompozitne materiale si raziskovalci prizadevajo razviti inovativne rešitve, ki ne le izboljšajo učinkovitost in zmanjšajo stroške surovin, temveč tudi spodbujajo okoljsko odgovornost v proizvodnih procesih ter zasledujejo trajnostne cilje. Avtorji v tem članku opisujejo raziskavo ojačitve posebne epoksi smole, zasnovane za gradbeništvo, z različnimi masnimi deleži (0 %, 1 %, 2 %, 3 % in 5 %) pirolitnega prahu za pnevmatike (TPP). Vzorce kompozitov so izdelali z uporabo tehnike ročnega polaganja, da bi preučili vpliv TPP kot ojačitvene faze na toplotne in mehanske lastnosti epoksi smole. Za karakterizacijo uporabljenih surovin so avtorji članka izvedli analizo velikosti delcev, pregled z vrstičnim elektronskim mikroskopom (SEM), spektroskopijo energije dispergiranih rentgenskih žarkov (EDX) in Fourierjevo transformacijsko infrardečo spektroskopijo (FTIR). Poleg tega so, da bi ocenili izboljšanje učinkovitosti izdelanih kompozitov, izvedli še teste trdote po Shore-D, natezne trdnosti, tritočkovne upogibne trdnosti, odpornosti na udarce in toplotne prevodnosti. Analize raziskav so pokazale znatno izboljšanje trdote, natezne in upogibne trdnosti za približno 14,8 %, 87,5 % oziroma 72,2 %, pri čemer je optimalna zmogljivost dosežena pri 3 mas. % TPP. Nad to koncentracijo so se mehanske lastnosti začele slabšati. Poleg tega se je pri 5 mas. % TPP opazno povečala trdota za 14,8%, udarna trdnost za približno 350 % in toplotna prevodnost za 86,5 %.

Ključne besede: piroliza, izrabljene pnevmatike, kompozitni materiali, trajnost, upogibna trdnost.

## 1 INTRODUCTION

Yearly, about three billion tires are fabricated, while a similar number of exhausted end-of-life tires (ELTs) are discarded. Countries such as USA, Japan, and the European Union together generate about five million tons of ELTs. The accumulation of this trash material, caused by rapid industrial growth and an increased number of drivers, keeps filling the landfills and other places where it is kept. The issue causes serious environmental, economic, and public health challenges. Landfills, which are the most common way to get rid of waste tires, are dangerous areas because it is difficult to control fires in them, and diseases spread through pests such as mosquitoes and rodents.[1] Tires are made up of many chemicals and additives, and their physical structure is very strong. They are mostly made of rubber, steel fibers, polymer fibers, carbon black, and a lot of other chemicals, both organic and inorganic. These include plasticizers, anti-aging compounds, sulfur, and zinc oxide.[2] The exact formula differs based on the tire's designated use. Natural rubber, originating from latex (cis-1,4-polyisoprene) is a polymer synthesized via the polymerization of

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monomers such as butadiene, styrene, propylene, isobutene, and neoprene, facilitated by a catalyst. Tires possess a rigid physical composition and a complex chemical structure due to the multitude of substances and additives incorporated to enhance their performance. They mostly comprise rubber, carbon black, steel, and polymer fibers.[3] A broad range of organic and inorganic additives, including plasticizers, anti-aging agents, sulfur, and zinc oxide, are utilized in the composition of tires.[4–6] Cross-linking of rubber chains through irreversible vulcanizing reactions improves elasticity and mechanical strength, converting rubber into a thermosetting polymer that cannot be remolded by simple heating and is non-degradable under natural weathering conditions.[7] Recycling ELTs is a common method of sustainable treatment. This process involves grinding the tires into small particles, which can then be reused as flooring for sports fields and playgrounds;[8] furthermore, many experimental studies are dedicated to evaluating ground ELTs as a filler material in various civil engineering sectors, including concrete, asphalt, sand, and soil.[9,10] Moreover, the possibility of reusing steel wires from tires to reinforce concrete has been examined in several research studies,[11,12] while the use of ground ELT powder as the filler material in thermoplastics and thermosets has also been the subject of extensive experimental research.[13]

Although using waste materials as fillers to reduce raw material costs may lead to a decrease in certain mechanical properties, this compromise could be acceptable for applications that do not require high mechanical strength. Furthermore, the inclusion of filler may enhance other characteristics, such as thermal insulation and sound absorption performance.[14] Converting ELTs into an alternative fuel through the pyrolysis process could help reduce fossil fuel consumption and conserve natural resources. This can be achieved when whole ELTs are burned, or when they are first shredded or ground into fine particles, depending on the combustion unit type,[1] where the energy produced can be utilized in various industries, including cement production, paper mills, and power plants.[13] Pyrolysis refers to the thermochemical process that breaks down long organic molecules into smaller, low molecular weight products. This process takes place in an inert environment, either at normal or lower pressure, to prevent the chemicals from changing or oxidizing. It requires very high temperatures, usually between 400 °C and 1200 °C.[15] The pyrolysis technology for ELTs is a crucial method that effectively decomposes it into valuable products, including pyro-oil, pyro-gas, and carbon black powder, after removing steel wires. This process also addresses pollution concerns, as illustrated in **Figure 1**.

Various reactor types have been developed and employed for the ELT pyrolysis process, including fixed bed, auger, rotary furnace, stirred tank, fluidized bed, and conical spouted bed reactors. Researchers have thoroughly examined the benefits and limitations of each reactor type, alongside the influence of diverse pyrolysis process parameters such as ELT particle size, processing temperature, heating rate, pressure, and catalyst application on the properties of the resultant ELT pyrolysis products (oil, gases, and residual solid powder).[8,16] However, although burning ELTs can produce significant energy due to the high calorific value of tires, this method is not recommended. The combustion process releases toxic byproducts that can cause serious environmental pollution and health problems if not managed properly.[16] Tire pyrolytic powder (TPP) is a carbon-rich substance containing over 85 % carbon, derived largely from carbon black, which improves tire performance and influ-

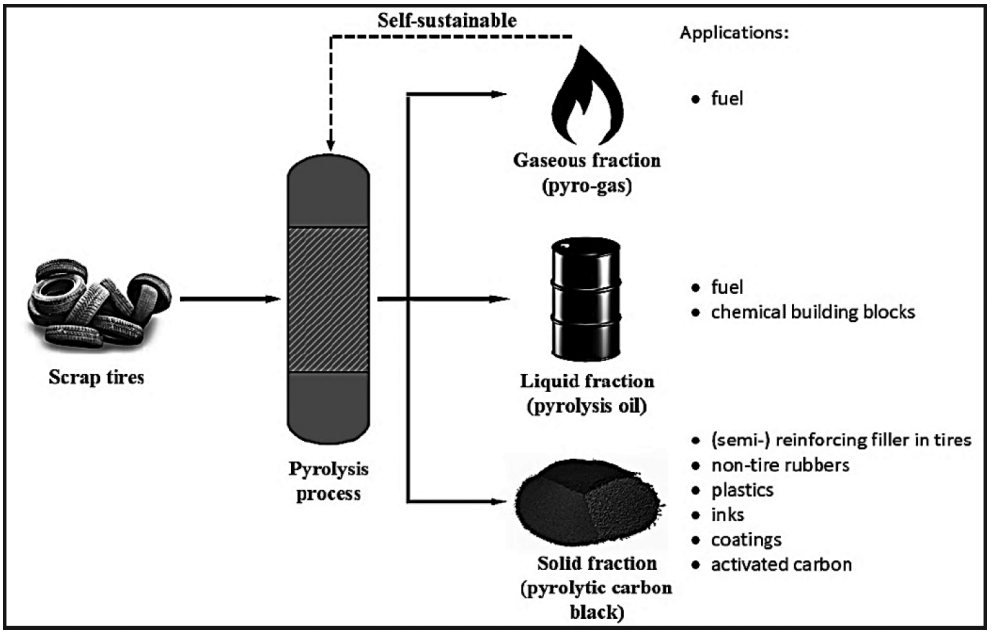



**Figure 1:** Tire pyrolysis process diagram[16]

ences the rubber pyrolysis process. Prior research delineated surface functional groups, such as C-O/C-O-C bonds, phenols, alcohols, and carboxylic acid groups, utilizing FTIR, XRD, and HHV analyses.[8,18] Extensive studies explored the use of TPP across various industrial sectors. It was proposed as a pigment for preparing printing ink[19,20] and as an additive for enhancing the performance of bitumen asphalt pavement, following appropriate thermal and chemical post-treatments.[21,22] Furthermore, TPP was suggested as an environmentally friendly alternative to traditional black carbon for reinforcing rubber,[23,24] as well as for applications in wastewater treatment, catalyst support materials, and the fabrication of supercapacitors and batteries.[8,25] However, high concentrations of inorganic components and residual carbonaceous materials can weaken mechanical properties and reduce the effectiveness of TPP as a reinforcing agent.[24] Many previous works focused on using end-of-life tire products as filler materials. Jamal I. Abdulhameed et al. investigated the incorporation of silane-treated crumb rubber from waste tires into epoxy resin.[13] Their results show that the use of a coupling agent can mitigate the deterioration of mechanical properties when using crumb rubber as a filler. P. Caputo, et al., discussed using TPP as an additive to improve the performance of bitumen.[22] N Cardona, et al., presented a review of potential recovering of pyrolytic carbon black from waste tires and its use as a reinforcing filler in rubber products.[24]

Epoxy resin is a highly versatile thermosetting polymer widely used across various industries, such as paint production, automotive industry, aerospace, and structural adhesives. Different epoxy resins are primarily low molecular weight liquids that contain two or more epoxide functional groups capable of interacting with different curing agents. During the curing process, the molecular weight, chain length, and branching increase, resulting in a three-dimensional network structure. Once fully cured, epoxy resins demonstrate exceptional strength, modulus, thermal stability, minimal shrinkage, chemical resistance, and dimensional stability. However, they can also be quite brittle due to high crosslink density and moisture absorption.[26]

The enhancement of mechanical and thermal performance of epoxy resin through blending it with other polymers, the incorporation of specific reinforcing materials, or the addition of rubber particles, was extensively investigated over the past decade. On the other hand, limited research efforts were dedicated to examining the effects of incorporating TPP in an epoxy matrix. In this context, the utilization of TPP, which contains a wide range of constituents – including also a micro-sized rubber phase, originating from an uncompleted pyrolysis process, in addition to carbon black and various oxides – may represent an advance step toward enhancing the mechanical and thermal properties of epoxy resins. Moreover, employing such byproducts contributes to mitigating the environmental impact associated with the accumulation of these waste materials. The present study investigates the effect of TPP on an epoxy matrix, determining the optimum loading on the basis of a series of standard mechanical and thermal tests. The obtained results provide useful insights about this type of filler used as a reinforcing substance for improving polymer matrices.

## 2 EXPERIMENTAL PART

### *2.1 Materials used*

Tire pyrolysis particles (TPP), a byproduct supplied by Abrage-Alkut Company, Iraq, were employed as a filler or reinforcement in an epoxy polymer matrix. Particle size distribution analyses, FTIR, and SEM were performed to evaluate the average particle size, chemical bond types, surface morphology, and elemental composition of TPP. The results section will address these findings. For the matrix, phase Sikadur-52 LP was utilized, a specialized epoxy resin manufactured by Sika AG in Switzerland for construction applications. It is a two-component, solvent-free, low-viscosity injectable liquid formulated from high-performance epoxy resins. This resin is employed for filling, sealing voids, and repairing cracks in structures including bridges, columns, beams, floors, walls, and various civil engineering applications. The principal physical and mechanical properties, as indicated by the manufacturer, are presented in **Table 1**.

**Table 1:** Main physical and mechanical properties of Sikadur-52 LP

| Properties | Density | Compressive strength | Tensile strength | Viscosity at 30 °C |
|---|---|---|---|---|
| Value/unit | 1.06 kg/L | >70 N/mm$^2$ | ~ 27 N/mm$^2$ | 150 MPa |

### *2.2 Manufacturing and testing the samples*

Epoxy specimens were created by combining two components, A and B resins, in a 2:1 ratio. The mixture was stirred with a wooden tool for three minutes before being transferred into a rubber mold with cavities that satisfy the specifications for tensile strength, three-point

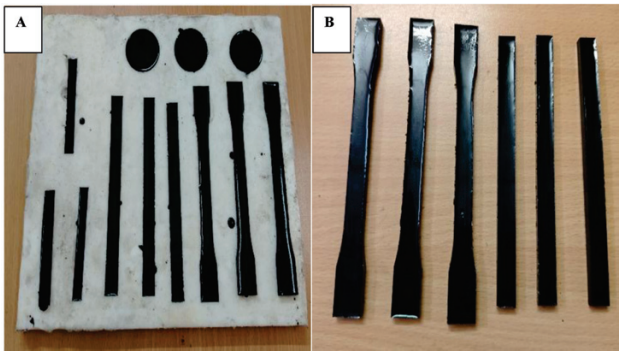
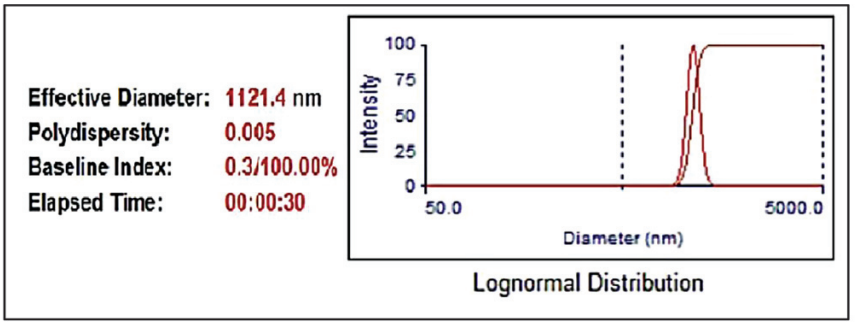



**Figure 2:** A) rubbery mold, B) composite specimens for tensile and bending strength tests




| Run | Eff. Diam. (nm) | Half Width (nm) | Polydispersity | Baseline Index |
|---|---|---|---|---|
| 1 | 1446.8 | 102.3 | 0.005 | 8.6/100.00% |
| 2 | 987.2 | 156.1 | 0.025 | 7.3/100.00% |
| 3 | 1386.5 | 98.0 | 0.005 | 1.5/100.00% |
| Mean | 1273.5 | 118.8 | 0.012 | 5.8/100.00% |
| Std. Error | 144.2 | 18.7 | 0.007 | 2.2/ 0.00 |
| Combined | 1121.4 | 79.3 | 0.005 | 0.3/100.00% |

**Figure 3:** Particles size analysis details of TPP

bending strength, impact strength, and thermal conductivity tests, as shown in **Figure 2**. Composite samples were generated through incorporating TPP into the resin mixture before stirring. The experimental part of the research utilized composites with various weight percentages of TPP (0, 1, 2, 3, and 5 %).

A particle size analysis was performed utilizing the principle of dynamic light scattering. Fourier transform infrared spectroscopy (FTIR) was conducted in accordance with ASTM E1252 over a range of 3000–400 $cm^{-1}$, employing a TENSOR-27 instrument from Bruker Optics. Scanning electron microscopy (SEM) imaging was conducted as well. The tests aimed to ascertain the principal characteristics of TPP, including effective particle size, types of chemical bonding, and elemental composition.

Shore D hardness (ASTM D2240), determined as the mean hardness at five locations, was employed to evaluate the hardness of composite specimens. Tensile strength and three-point bending strength tests were conducted using a computerized universal testing machine with a crosshead speed of 5 mm/min, according to ASTM D638 and ASTM D790 standards, respectively. The tests were conducted on three specimens in each case. Izod impact tests (ISO 179) were conducted to assess the average impact strength by dividing the recorded fracture force by the cross-sectional area of the specimens, with the reported values representing the average of three specimens. Thermal conductivity assessments were conducted utilizing a hot disk apparatus.

## 3 RESULTS

### *3.1 Characterizing TPP*

The particle size analysis of TPP revealed a predominantly fine distribution. The results indicated that most

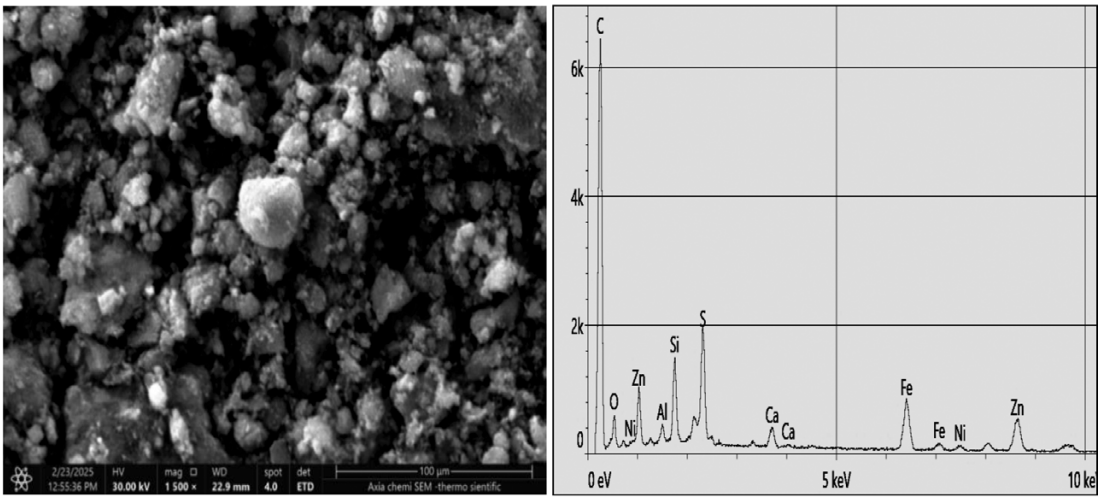



**Figure 4:** SEM image, and EDX analysis of TPP

particles were within the micrometers range, therefore guaranteeing good performance as filler materials in polymer matrix resin. The average particle size was 1.121 μm. Additional information regarding the results may be found in **Figure 3**.

SEM images provide critical details regarding the powder morphology and particle size analysis results. EDX analysis coupled with SEM provides valuable information on the chemical composition and element weight fraction of the powder. **Figure 4** presents a SEM image and chemical composition of TPP.

The above figure demonstrates significant size variation of the particles, ranging from several microns to tens of microns, having irregular shapes and rough surfaces. This morphology indicates partial pyrolysis or incomplete combustion, which is the expected form of pyrolyzed tire particles. However, high surface roughness of TPP can help improve mechanical bonding between particles and the matrix. The SEM image also shows agglomeration of numerous particles, perhaps a result of van der Waals interactions or residual carbonaceous substances. The TPP chemical composition and element weight fractions are summarized in **Table 2**.

**Table 2:** TPP chemical composition according to EDX

| Element | Atomic % | Atomic % error | Weight % | Weight % error |
|---|---|---|---|---|
| C | 85.1 | 0.5 | 69.5 | 0.4 |
| O | 8.5 | 0.3 | 9.3 | 0.4 |
| Si | 1.4 | 0.0 | 2.8 | 0.0 |
| S | 1.7 | 0.0 | 3.6 | 0.1 |
| Ca | 0.4 | 0.0 | 1.1 | 0.0 |
| Fe | 1.4 | 0.0 | 5.2 | 0.1 |
| Zn | 1.3 | 0.0 | 5.9 | 0.2 |
| Ta | 0.2 | 0.0 | 2.7 | 0.3 |

The Fourier-transform infrared spectroscopy (FTIR) test provided valuable insights into the molecular structure and active groups in the samples. It is an essential analytical technique used to identify chemical bonds and understand the properties of various compounds. **Figure 5** shows the FTIR test results for TPP.

The figure shows primary peaks at 2895 $cm^{-1}$ and 2836 which fall within the 3000-2800 $cm^{-1}$ range associated with C-H aliphatic groups. This suggests the presence of hydrocarbon products, including C-H, $CH_2$ and $CH_3$.[27] Wavelengths ranging from 2400 $cm^{-1}$ to 2250 $cm^{-1}$ are generally associated with gaseous $CO_2$ produced during pyrolysis.[28] The range of 1600-1580 $cm^{-1}$ indicates the C=C aromatic stretch, suggesting the presence of carbon black. [4] The peak at 1360 $cm^{-1}$ may correspond to carboxylate (carboxylic acid salt), while the peak at 1001 $cm^{-1}$ is found within the 1000-1100 $cm^{-1}$ range, indicating aliphatic bending bonds (C-C).[29] Peaks at (865, 792 and 706) $cm^{-1}$ are associated with the out-of-plane bending of aromatic C-H bonds in mono- or di-substituted rings.[30]

### *3.12Results and discussion of mechanical and thermal properties of composites*

The hardness test refers to the ability of materials to resist penetration or scratching. This property is critical for the durability and performance of composites in various applications. A high hardness value of a composite indicates high wear resistance, making the material more durable under severe service conditions. **Figure 6** shows the average Shore hardness values and standard error bars for both epoxy and epoxy/TPP composites.

The data demonstrates that the hardness increases with increasing TPP weight fraction, reaching its maximum at 5 *w*/% of TPP, being about 14.8 % higher than the epoxy hardness. This enhancement is attributable to the high hardness of TPP, which contains a high content of fly ash[1], composed of various high-hardness metal ox-

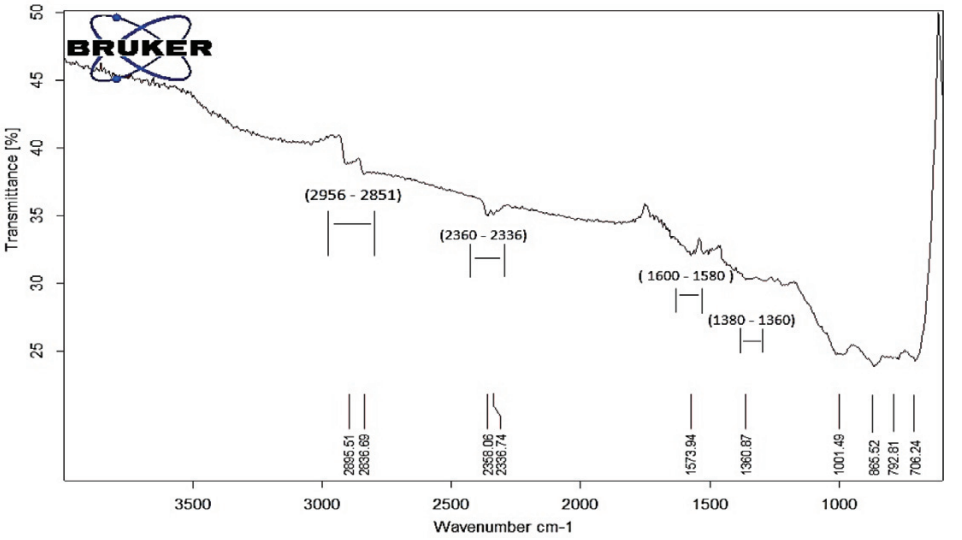



**Figure 5:** FTIR of TPP

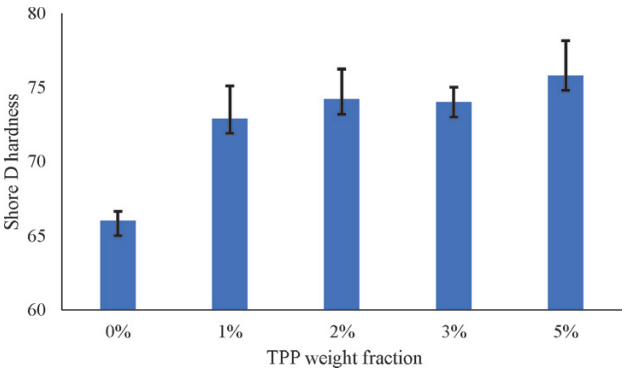



**Figure 6:** Hardness with the standard error of the composite

ides, including $Al_2O_3$, $SiO_2$, and ZnO, as previously shown in **Table 2**.

The incorporation of these hard particles into the matrix restricts the mobility of resin chains under applied stresses during testing and contributes to load bearing through interfacial bonding between the two phases, thereby enhancing the matrix strength.[31,32] Tensile and bending strength tests are the cornerstones in evaluating mechanical properties of materials. Ultimate (or maximum) tensile strength is the maximum stress that a material can withstand before failure, while bending strength refers to its ability to resist deformation under loads. The average maximum tensile strength and bending strength, along with standard error bars, for epoxy and epoxy/TPP composite specimens are presented in **Figures 7** and **8**.

It is clear that both the tensile and flexural strengths of epoxy increase with the weight fraction of TPP, reaching the maximum strength in the specimens with 3 % TPP by weight. This signifies an improvement of approximately 87.5 % in the tensile strength and 72.2 % in the flexural strength compared to pure epoxy resin. The reinforcing particles in the resin matrix impede the crack growth; the oxide particles reduce the void percentage within the matrix, thus enhancing its resistance to crack propagation. Oxide particles hinder the mobility of resin chains when subjected to applied loads.[32,33] When the TPP weight fraction exceeds its critical value, the strength of the composite begins to decline. This reduction in strength is due to an uneven distribution, or agglomeration, of TPP particles within the matrix, causing an increase in voids, consequently reducing the mechanical strength.[33,34]

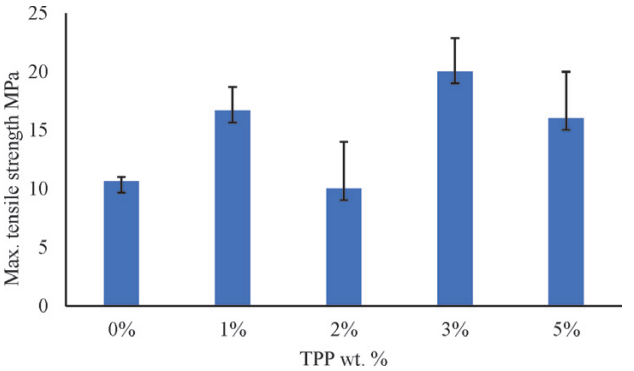



**Figure 7:** Average maximum tensile strength with standard error bars for composite specimens

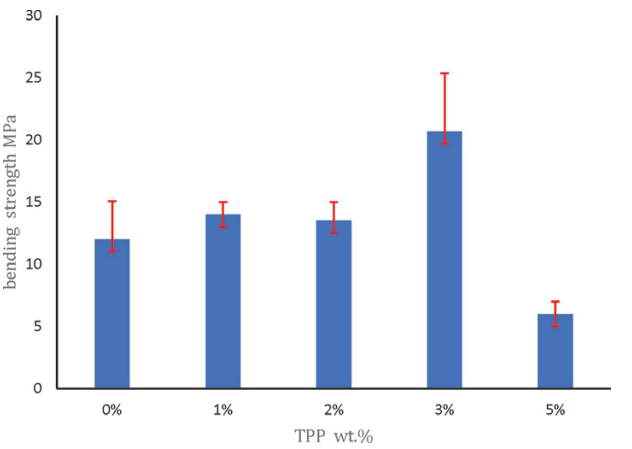



**Figure 8:** Bending strength with standard error bars in composite specimens

Impact strength is a critical parameter in assessing the durability and reliability of materials, particularly in applications where they may be subjected to sudden forces or stress. The results for the impact strength of epoxy and epoxy/TPP composite specimens are shown in **Figure 9** below.

The impact strength test is utilized to evaluate a material's toughness when subjected to impact energy before fracture. Although epoxy resin reveals relatively high strength, it has low toughness and impact strength due to its high cross-linked chain structure. However, the incorporation of rubber particles into epoxy can modify the plastic/shear deformation of the structure under external stresses, so this method can enhance fracture energy dissipation.[35] In this context, rubber particles located at the tips of cracks can deform to absorb crack energy, consequently increasing the energy required for crack propagation.[36] The figure shows a notable increase in the impact strength with the increasing concentration of TPP, primarily consisting of incompletely decom-

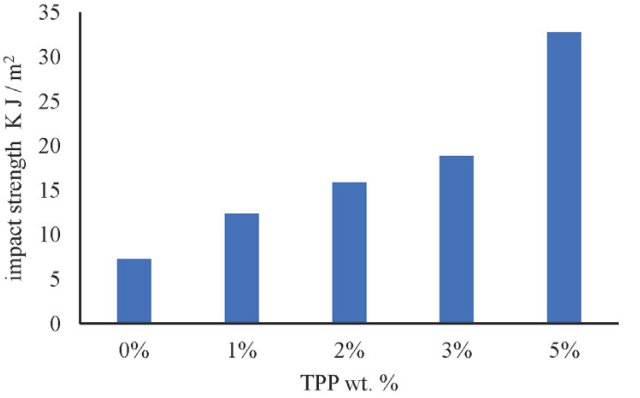



**Figure 9:** Impact strength of composite specimens

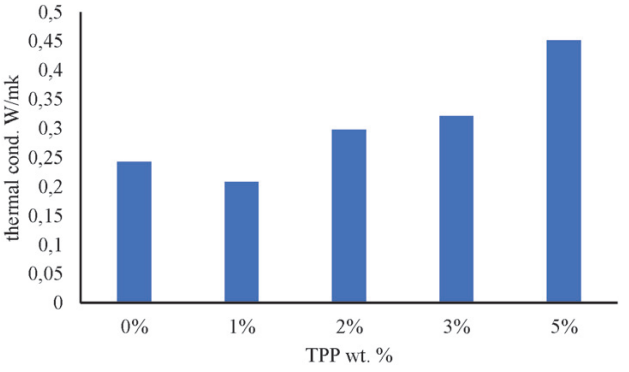



**Figure 10:** Thermal conductivity in composite specimens

posed rubber particles formed during the pyrolysis process.

To understand how materials work at different temperatures, it is necessary to determine their thermal conductivity. This information can have a big effect on how things are built and what materials are used for everything from electronics to construction. **Figure 10** below shows the results of the thermal conductivity tests made on epoxy and the epoxy/TPP composite. It shows that as the amount of TPP in the epoxy matrix increases, the thermal conductivity is significantly improved.[37] The biggest improvement takes place at 5 % TPP by weight, where its thermal conductivity is about 86.5 % higher than that of pure epoxy. This effect takes place due to a large amount of carbon black, which is either added to tires to improve their performance or produced through pyrolysis, along with partially graphitized carbon ash. These materials have much better thermal conductivity than epoxy, providing interconnected thermal pathways that enhance heat transfer efficiency.[38,39]

## 4 CONCLUSIONS

Based on the experimental results, the following conclusions can be drawn:

- The tire pyrolysis powder (TPP), a byproduct, can be utilized as a filler and reinforcing substance, enhancing the mechanical and thermal properties of epoxy resin.
- The maximum mechanical strength is attained with a 3 *w*/% TPP addition; however, a weight fraction above this amount leads to reduced reinforcement efficacy.
- The results show that at 3 *w*/% TPP, the tensile and flexural strengths can be improved by about 87.5 % and 72.2 %, respectively, while at 5 *w*/% TPP, the hardness, impact strength and thermal conductivity were also markedly increased by approximately (14.8, 350 and 86.5) %, respectively.
- The epoxy/TPP composite displays enhanced toughness and greater ductility relative to pure epoxy resin.
- However, using the TPP/epoxy composite as the core filler of a sandwich-structured composite panel instead of conventional polyurethane foam could support sustainable aims, such as waste management, while also offering the potential to improve certain mechanical properties. Thus, more experimental research is needed to explore these opportunities.

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