

RESEARCH ARTICLE

Polymer
COMPOSITES

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Tailored Ni-Zn-Cu ferrite/EPDM nanocomposites: Synthesis, characterization, and multifaceted properties for mechanical and electrical applications

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Funding information

Researcher's Supporting Project Number, Grant/Award Number: RSP2024R43; King Saud University, Riyadh, Saudi Arabia; National Research Foundation of Korea

Abstract

In this study, we prepared Ni-Zn-Cu Fe₂O₄ ferrite/ethylene propylene diene monomer (EPDM) rubber nanocomposites with enhanced mechanical and electrical properties. First, Ni-Zn-Cu Fe₂O₄ ferrite nanoparticles were synthesized with a tailored composition of NiZn_(0.3)Cu_(0.2)Fe₂O₄₍₂₎. Subsequently, these nanoparticles were incorporated into the EPDM rubber matrix through a two-roll mixing mill. Scanning electron microscopy and x-rays diffraction techniques were used to explore their morphology and structure. Fourier-transform infrared spectroscopy was used to identify metal-oxygen bonds in the nanoparticles and their incorporation in the nanocomposites. Similarly, curing properties, including optimum cure time, scorch time, and torque profiles, were evaluated using a moving die rheometer. While, mechanical properties of these composites, such as tensile strength and Young's modulus, were determined with a universal testing machine. Tensile strength improved from 0.11 MPa to 0.22 MPa (100% increase), whereas, the Young's modulus increased from 0.03 MPa to 0.052 MPa (86% increase). Additionally, we investigated the dielectric properties of these nanocomposites, encompassing permittivity, resistivity, and dielectric constant, using a dielectric properties analyzer. Our findings revealed a significant reduction in dielectric resistivity of nanocomposites (from 33.2 GΩ to 23.38 GΩ) upon the introduction of Ni-Zn-Cu Fe₂O₄ ferrite nanoparticles, indicating the formation of a continuous nanofiller network within the EPDM matrix. Interestingly, as the nanofiller loading increased, both dielectric permittivity and dielectric constant exhibited a gradual rise. The permittivity value increased from 2.31 to 2.39 (Fm⁻¹), while the dielectric constant increased from 0.0091 to 0.0179.

Highlights

- Synthesized Ni-Zn-Cu Fe₂O₄; tuned composition NiZn_(0.3)Cu_(0.2)Fe₂O₄₍₂₎.
- Integrated ferrite into EPDM; optimized mixing for enhanced properties.

Nazia Parveen and Asma Anam Hanif contributed equally to this work.

(NRF) grant funded by the Korean Government (MSIT), Grant/Award Number: RS-2023-00251670; Technology Development Program of MSS, Grant/Award Number: RS-2023-00251434

- Employed SEM, XRD, and FTIR; revealed morphology, structure, and bonding.
- Evaluated curing using MDR; unveiled optimal time and torque profiles.
- Explored dielectric properties; demonstrated reduced resistivity, heightened permittivity.

KEYWORDS

electrical properties, ferrite/EPDM rubber, mechanical properties, nanocomposites, nanofiller network

1 | INTRODUCTION

In the realm of advanced materials, the synergy of diverse constituents often yields remarkable outcomes, opening new horizons in technology and engineering. Among these multifaceted materials, nanocomposites, formed by incorporating nanoscale fillers within a polymer matrix, stand out as versatile platforms for tailoring a spectrum of properties.^{1,2} The synthesis of nanocomposites, a well-established discipline, provides a strong ground for enhancing the mechanical and electrical properties of materials. Such improvements hold the promise of transformative applications across diverse domains, such as electronics, telecommunications, automotive, and aerospace.^{3,4}

Ethylene propylene diene monomer (EPDM) stands as a pivotal player in the field of unsaturated polyolefin rubber, holding significant commercial importance.^{5,6} Renowned for its significant resistance to various environmental factors, including water, ozone, brake fluid, radiation, and adverse weather conditions, EPDM is highly regarded for its versatility and resilience.^{7,8} This rubber not only demonstrates chemical resistance but also exhibits good electrical properties when vulcanized, making it a preferred choice for a wide range of applications.⁹

One important advancement in the EPDM domain involves a gel-free process wherein diene vinyl norbornene is integrated into the EPDM backbone, resulting in a terpolymer with significantly improved properties.^{10–12} This polymer exhibited extensive utility in cable and wire applications, contributing to improved performance and durability. Beyond this, EPDM finds its footing in various sectors such as automotive manufacturing, where it is employed in body seals, mounts, roofing materials, weather-stripping, hoses, ducts, tubing, and tire production.¹³ Additionally, EPDM molded rubber has proven invaluable in the development of dampeners and bumpers, effectively mitigating vibrations in vehicles.¹⁴ This comprehensive versatility and resilience of EPDM mark its paramount role in various industries, making it a focal point of interest in the field of materials science and

engineering. In recent years, the widespread use of EPDM rubber, especially in high-performance products, has surged.¹⁵ However, the current electrical and mechanical properties of EPDM fall short of meeting growing demands.¹⁶ Recognizing this limitation, it becomes imperative to enhance the electrical and mechanical characteristics of EPDM rubber.^{9,13} Various studies have highlighted the production of rubber nanocomposites achieved through the incorporation of various materials, including clays,¹¹ carbon nanotubes,^{17,18} and graphene.¹⁹ These investigations have primarily focused on enhancing the mechanical, thermal, electrical properties, and morphological characteristics of EPDM nanocomposites. Besides these nanofillers, the incorporation of nanoparticles into rubber matrices has sparked a surge in the development of polymeric nanocomposites, driven by their desirable properties. Such nanocomposites possess a distinctive set of characteristics, including high tensile strength, enhanced flame retardancy, reduced gas permeability, and heightened thermal stability.^{20–22} In specific, metal oxide-based nanoparticles have emerged as particularly noteworthy due to their extensive applications. Several metal oxide nanoparticles, including CaO, ZnO, TiO₂, CeO₂, Fe₂O₃, and Fe₂O₄ have found utility in a wide array of fields, including sensors, catalysis, environmental remediation, and optoelectronic materials.^{23–26} Numerous synthesis methods have been developed for producing these nanoparticles, including thermal decomposition method,²⁷ solution combustion method,²⁸ microwave processing,²⁹ sonochemical method,³⁰ coprecipitation method,³¹ and sol-gel method.³² Among these metal oxide-based nanoparticles, ferrites represent a class of materials characterized by their remarkable properties of high permeability and moderate resistivity. These unique properties render them highly valuable in a diverse range of electronic devices, particularly those designed to harness higher frequencies, withstand elevated temperatures, maintain cost-efficiency, or exhibit enhanced corrosion resistance. Comprising iron oxide along with one or more additional metals (MFe₂O₄, where M being Mn, Zn, Cu, Co, Ni, etc.) in precise chemical compositions, ferrites have emerged as

pivotal components in diverse applications.^{33,34} Specifically, Ni-Zn ferrites are well known for their high electrical resistivity, substantial relative magnetic permeability, minimal magnetic coercivity, low hysteresis, and eddy current losses even at a frequency as high as 10 kHz. Furthermore, Ni-Zn ferrites possess impressive mechanical hardness, robust chemical stability, and a remarkable resistance to corrosion, exhibiting their potential as filler for preparing various polymer nanocomposites.^{35,36}

Bezerra et al.³⁷ prepared nanocomposites of PA-6 by incorporating NiFe₂O₄ ferrite using a combination of melting and compression molding techniques, targeting a filler loading of 60%. Nevertheless, it was only at the 10% ferrite concentration that a satisfactory dispersion was achieved. Luna et al.³⁸ reported synthesis of PA6/EPDM-MA/Ni_{0.5}Zn_{0.5}Fe₂O₄ nanocomposites with addition of 7 parts per hundred rubbers (phr) of Ni_{0.5}Zn_{0.5}Fe₂O₄. In their work, significant improvement in thermal, mechanical (increased from 40.6 MPa to 44.2 MPa), and magnetic properties of final nanocomposites was achieved. Furthermore, in Co-CaFe₂O₄/EPDM rubber nanocomposites, the incorporation of ferrite has demonstrated a significant impact on the structural, morphological, magnetic, thermal, mechanical (tensile strength was improved from 2.03 MPa to 8.50 MPa), and rheological properties of EPDM rubber.³⁹ Likewise, Mn-Zn ferrites have been incorporated into the EPDM matrix, resulting in the nanocomposites with high tensile strength (improved from 5.8 MPa to 7.2 MPa) and excellent dielectric properties (dielectric constant was increased from 2.4 to 8.1), intended for use as a microwave absorber material.⁴⁰ In another related study, Barium ferrite/EPDM rubber composites have been synthesized and employed as radar absorbing material (RAM) due to their enhanced permittivity and permeability (improved from 2 to 18 GHz) in comparison to pure EPDM rubber.⁴¹

In various studies, it has been reported that doping of Cu into Ni-Zn Fe₂O₄ ferrite resulted in improved structural, morphological, magnetic, and electric properties.^{42,43} To the best of our knowledge, Cu doped Ni-Zn Fe₂O₄ ferrites have not been incorporated before in EPDM rubber for improving its mechanical and electrical properties.

In the current study, we have prepared Ni-Zn-Cu Fe₂O₄ ferrite/EPDM rubber nanocomposites, driven by the ambition to propel the boundaries of material performance. Here, we have used the sol-gel method to synthesize Ni-Zn-Cu Fe₂O₄ ferrite nanoparticles with a tailored composition. These nanoparticles, with a desired composition of NiZn_(0.3)Cu_(0.2)Fe₂O₄₍₂₎, were subsequently integrated into the EPDM rubber matrix using an internal mixing method. The morphological and structural characteristics of the prepared ferrite/EPDM nanocomposites

was examined using scanning electron microscopy (SEM) and XRD analysis. Similarly, FT-IR spectroscopy was used to evaluate the presence of various stretching vibrations of various metal-oxygen bonds. To assess the curing characteristics such as optimum cure time (tc₉₀), scorch time (ts₂), maximum and minimum torque, moving die rheometer (MDR) was employed. Mechanical properties of the prepared polymer nanocomposites, such as tensile strength and Young's modulus, were deduced using universal testing machine (UTM). Similarly, dielectric permittivity, resistivity, and dielectric constant of these nanocomposites were determined from dielectric properties analyzer.

The prepared nanocomposites exhibited enhanced mechanical strength, and the introduction of Ni-Zn-Cu Fe₂O₄ ferrite nanoparticles led to a reduction in dielectric resistivity, confirming the formation of a continuous nanofiller network within the EPDM matrix. Additionally, an increase in nanofiller loading was observed to correspond with a gradual rise in dielectric permittivity and dielectric constant. These findings collectively indicate that the prepared Ni-Zn-Cu Fe₂O₄ ferrite/EPDM nanocomposites hold significant potential as advanced materials suitable for various mechanical and electrical applications.

2 | MATERIALS AND METHODS

2.1 | Materials

All the chemicals like Nickel Nitrate (Ni(NO₃)₂·6H₂O) (99.5%), Zinc Nitrate (Zn(NO₃)₂·6H₂O) (99.5%), Copper Nitrate (Cu(NO₃)₂·3H₂O) (99.5%), ferric Nitrate (Fe(NO₃)₃·9H₂O) (99.5%), citric acid (C₆H₈O₇), sodium hydroxide (NaOH), ethylene propylene diene monomer rubber (EPDM Rubber-8550), zinc oxide (ZnO), stearic acid, sulfur, tetramethyl thiuram disulfide (TMTD), and 2-methyl-CBS-oxazaborolidine (Corey-Bakshi-Shibata catalyst, CBS) were purchased from Sigma-Aldrich and were used without any further purification.

2.2 | Synthesis of NiZn_(0.3)Cu_(0.2)Fe₂O₄₍₂₎ (NiZCF) nanoparticles

The synthesis of nanoparticles with a targeted composition of NiZn_(0.3)Cu_(0.2)Fe₂O₄₍₂₎, referred to as NiZCF nanoparticles, was achieved via sol-gel method following a reported procedure.⁴⁴ In this procedure, precise stoichiometric quantities (1:0.3:0.2:2) of nickel nitrate, zinc nitrate, copper nitrate, and ferric nitrate were sequentially dissolved in 100 mL of distilled water under

vigorous stirring. Subsequently, citric acid (5% solution in water) was added into this mixture under continuous stirring to achieve a homogeneous blend. The pH of the resultant solution was maintained within the range of 6 to 7 by the addition of a NaOH solution. Subsequently, the mixture was heated at 100°C to facilitate the evaporation of water.

Upon gel formation, the material was dried by heating in an oven at 80°C for a duration of approximately 9 hours. Afterwards, the dried material was carefully ground to obtain a loose powder. Compact nanoferrites were then obtained through a sintering process conducted at 1000°C for a period of 3 hours, followed by a grinding step.

2.3 | Preparation of NiZCF/EPDM rubber nanocomposites

The incorporation of ferrite nanofiller into rubber matrix was achieved using two-roll mixing mill operating at a speed of 60 revolutions per minute (r/min) and maintained at a temperature of 50°C. Initially, the elastomer matrix was softened and masticated through slightly heated rolls. Subsequently, homogenized composites were formulated by introducing NiZCF using a two-roll mixing mill at various parts per hundred rubber (phr) values of 1, 3, 5, and 8, giving a total four composite samples. Addition of nanofiller at a phr value of higher than 8 resulted in a complete phase separation, thus it was not possible to prepare samples with a phr higher than 8. This can be attributed to a decline in filler-rubber interaction as higher filler loadings result in agglomeration. In such cases, the filler–filler interaction begins to exceed the filler-rubber interaction, which can negatively impact the overall properties. A complete reaction recipe is given in Table 1.

Curing parameters were determined based on rheometer data acquired using a MDR. The curing curve was generated by identifying the point at which torque reached its maximum value, defining the optimal cure time (tc_{90}). The resulting nanocomposites were subjected to vulcanization or compression molding, yielding test samples measuring 120 mm × 120 mm with a thickness of 1 mm. This molding process was executed using a hydraulic compression press at a temperature of 150 ± 1°C and a pressure of 20 kN for tc_{90} determination. All procedures adhered to ASTM standards.⁴⁰

2.4 | Characterization

The structure and crystallinity of NiZCF ferrite particles and NiZCF/EPDM rubber nanocomposites were studied

TABLE 1 Ingredients for the synthesis of NiZCF/EPDM rubber nanocomposite.

Serial #	Ingredients	Parts per hundred rubber (Phr)
1.	EPDM Rubber-8550	100
2.	Zinc oxide	1
3.	Stearic acid	1
4.	Sulfur	2
5.	TMTD	1
6.	CBS	1
7.	NiZCF	0, 1, 3, 5, 8

using Miniflex Benchtop powder x-ray diffractometer (Rigaku, Japan) with Cu Ka irradiation ($\lambda = 1.5418\text{Å}$, 200 kV, 50 mA) in the range of $2\theta = 10^\circ\text{--}80^\circ$ (ASTM standard D5187-91R02⁴⁵). The average crystallite size, with a precision error of ±0.5 nm, was determined by applying the Scherrer equation to the x-ray diffraction (XRD) diffractogram.^{46,47} The dispersion quality of the filler(s) and their interactions at the interfaces within the polymer matrix were meticulously examined through field emission scanning electron microscopy (FESEM) using a Hitachi S-4800 instrument from Tokyo, Japan. To facilitate the analysis, the fracture samples (cross-sections) were affixed to standard specimen stubs using double-sided adhesive tape. Prior to scanning, a thin layer of gold was deposited onto the mounted samples using a BIO-RAD SEM coating system. This gold coating served to prevent electrostatic charging, as uncoated samples may lead to diminished resolution during the investigative process (No specific ASTM standard, but standard imaging procedures were followed). For structural and compositional characterization of the prepared materials, Fourier-transform infrared spectroscopy (FT-IR) was employed using a Bruker Alpha Platinum ATR instrument. The analysis was conducted within the wavelength range of 4000–400 cm^{-1} , utilizing standard KBr beam splitters (No specific ASTM standard, but standard FT-IR techniques were followed). The curing characteristics of the nanocomposites were determined using a Moving Die Rheometer (MDR-3000) at a temperature of 150°C. A cure curve was generated by plotting torque against time, specifically when the torque reached its maximum value⁴⁸ (No specific ASTM standard, but standard MDR testing protocols were followed). The mechanical properties such as tensile strength and Young's modulus were determined by universal testing machine (UTM) according to BS ISO 37: 2018 ASTM standard.⁴⁹ The dielectric properties like dielectric permittivity, dielectric resistivity, and dielectric constant were studied using dielectric

properties analyzer I and II. Both Dielectric permittivity and dielectric constant were measured using E4980A Precision LCR meter (Agilent Technologies, Inc.) at 50 Hz frequency in accordance with ASTM standard D150-19.

3 | RESULTS AND DISCUSSION

3.1 | Fourier transform infrared spectroscopy analysis

In Figure 1A,B, the FT-IR spectra of NiZCF nanoparticles and NiZCF/EPDM rubber nanocomposite (5phr) are furnished, spanning the wavelength range from 4000 to 500 cm^{-1} . The structural characteristics of the ferrite nanoparticles are confirmed by the presence of two prominent absorption bands at 880 and 660 cm^{-1} , as shown in Figure 1A. These bands can be attributed to the stretching vibrations of Fe—O bonds located at both

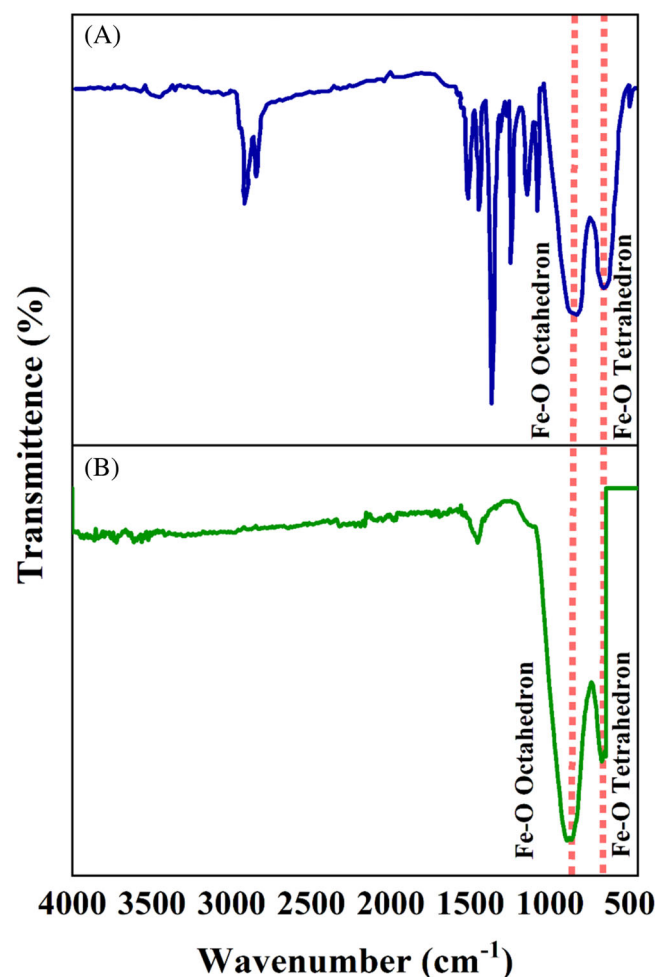


FIGURE 1 Fourier transform infrared (FT-IR) spectrum of NiZCF and NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposites at 5 phr.

the octahedral and tetrahedral sites within the ferrite structure.⁵⁰

A noteworthy observation consistent with earlier studies, is the impact of charge imbalance when Zn^{2+} ions are replaced by Cu^{2+} ions. This phenomenon results in the migration of some Fe^{3+} ions from tetrahedral sites to octahedral sites, resulting in an increased stretching vibration of $\text{Fe}^{3+}\text{—O}^{2-}$ bonds. This migration process has been extensively discussed and elucidated in the literature.^{50,51} This structural modification is an important aspect contributing to the distinctive FT-IR spectra of the $\text{NiZn}_{0.3}\text{Cu}_{0.2}\text{Fe}_2\text{O}_4$ nanoparticles.

The FT-IR spectrum of NiZCF/EPDM rubber nanocomposites (Figure 1B) displays absorption bands at 880 and 660 cm^{-1} , confirming the successful incorporation of NiZCF ferrite nanofiller into the EPDM rubber matrix. The absorption bands at 2922 and 2848 cm^{-1} correspond to asymmetric and symmetric stretching vibrations of the C—H bond, while the bands at 1450 and 1370 cm^{-1} indicate C—H bending vibrations.^{38,52}

3.2 | X-rays diffraction analysis

Figure 2 presents the XRD diffraction patterns of both NiZCF nanoparticles and their nanocomposite with EPDM rubber at 5 phr. Notably, diffraction peaks for both NiZCF and NiZCF/EPDM rubber nanocomposites correspond to cubic spinel structure. These peaks were successfully compared and indexed using the JCPDS card number 10-0235.^{50,53-56}

For NiZCF nanoparticles, diffraction peaks are observed at 2θ values of 32.8, 37, 42.1, 43.2, 60.1, and

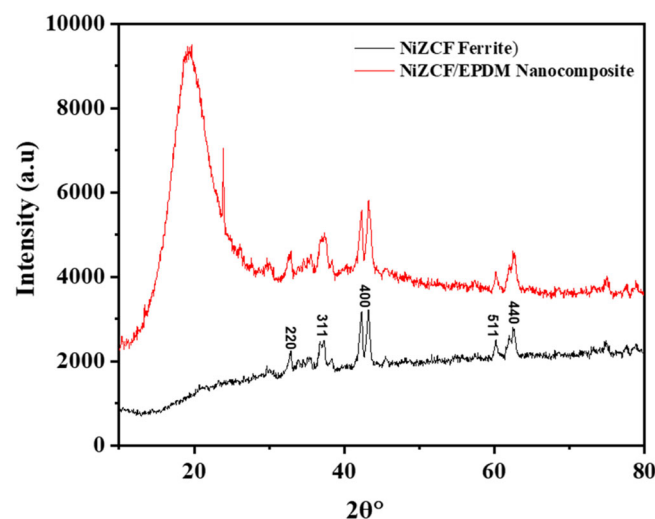


FIGURE 2 X-rays diffraction (XRD) diffractogram of NiZCF nanoparticles and NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposite (5 phr).

62.5°, corresponding to crystal planes (220), (311), (400), (511), and (440) respectively.³⁸ All these peaks are present exactly at same positions in the XRD diffractogram of nanocomposite. In addition to these peaks, XRD diffractogram exhibits a broad peak of high intensity at a small angle ($2\theta = 20^\circ$). Additionally, low-intensity peaks are observed, which can be attributed to the polymer ingredients. The average crystallite size (D) was found to be 8.10 nm, it was calculated using Scherer's formula.⁴⁴ Remarkably, the collected data indicates that the pure NiZCF nanoparticles did not undergo any structural transformation during the composite formation. This is evident from the consistent peak positions in the diffractogram of composites and virtually unchanged particle size compared to pure NiZCF nanoparticles. Furthermore, the NiZCF nanoparticles and NiZCF nanoparticles/EPDM rubber nanocomposite exhibit nearly identical lattice values, signifying that the structural integrity remains unaltered. This successful preparation of the nanocomposites is corroborated by previous publications in the field.⁴⁶

3.3 | Scanning electron microscopy analysis

In Figure 3, SEM micrographs of NiZCF nanoparticles are exhibited, revealing their morphology as roughly conglomerated and spherical with an average particle size of 102 nm. While, Figures 4 and 5 illustrates SEM images of the nanocomposites (3 phr and 5 phr, respectively), depicting a distinct two-phase structure. Here, NiZCF

nanoparticles are homogeneously dispersed throughout the EPDM rubber matrix. This distribution highlights the effective adhesion between the two phases, a phenomenon known to significantly influence the properties of the resulting nanocomposites, as elucidated by Newnham.⁵⁷

The SEM analysis reveals a rough morphology for the EPDM rubber matrix in the provided micrographs.⁵⁸ This texture results from the favorable physical interactions between the rubber matrix and the nanoferrites, as obvious from Figures 4 and 5 (3 phr and 5 phr, respectively).

Increased mechanical and electrical properties observed at 3 and 5 phr filler loadings can be attributed to the uniform dispersion of the filler within the EPDM matrix. SEM analysis revealed a well-dispersed morphology of the nanofiller at these concentrations, indicating strong interactions between the filler and the polymer matrix. This uniform dispersion promotes efficient stress transfer between the filler and the matrix, resulting in enhanced mechanical properties such as tensile strength and Young's modulus. Additionally, the increased filler-matrix interface area facilitates improved electrical conductivity and dielectric properties. However, at 8 phr filler loading, a decline in both mechanical and electrical properties was observed. This deterioration can be attributed to phase separation phenomena, where excessive filler loading leads to agglomeration and poor dispersion within the matrix. This highlights the importance of maintaining an optimal filler loading to achieve the desired balance between dispersion and reinforcement in nanocomposite materials.

Similarly, Figure 4D displays the EDX spectrum of NiZCF/EPDM rubber nanocomposites (3 phr), confirming

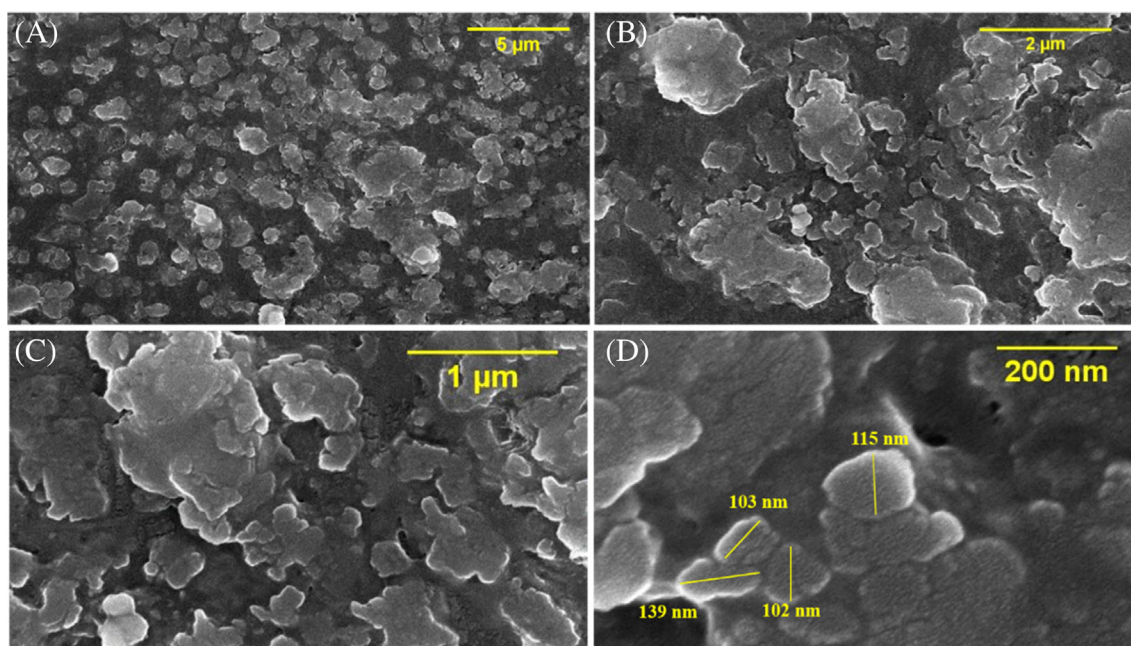


FIGURE 3 Scanning electron microscopy (SEM) micrographs of NiZCF at different magnifications (200 nm–5 μ m).

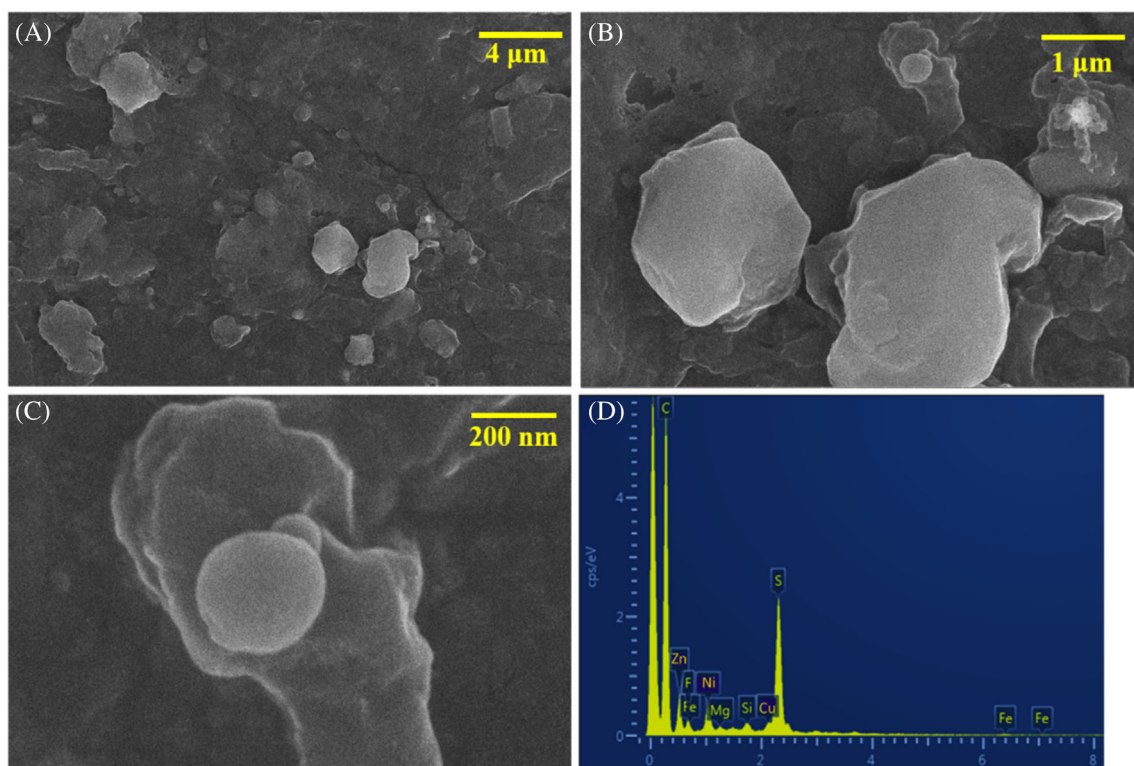


FIGURE 4 Scanning electron microscopy (SEM) micrographs of NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposite (3 phr) at different magnifications (200 nm–4 μm).

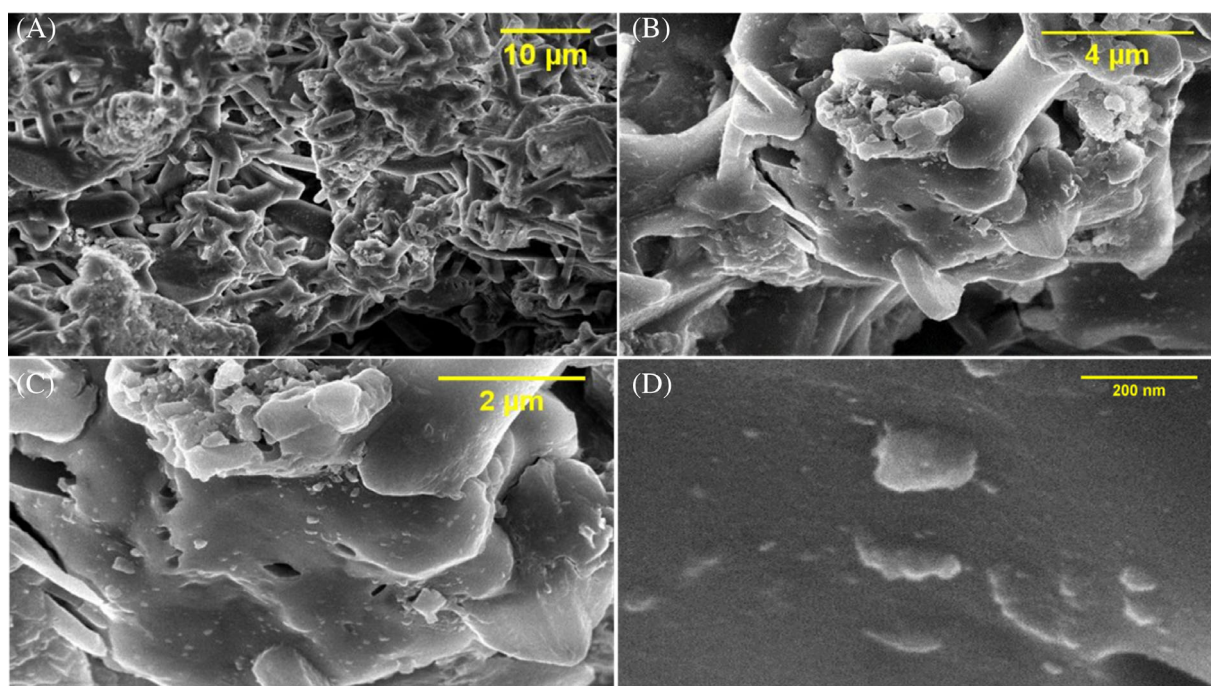


FIGURE 5 Scanning electron microscopy (SEM) micrographs of NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposite (5 phr) at different magnifications (200 nm–10 μm).

the presence of Ni, Zn, Cu, and Fe elements in the prepared nanocomposites.

These SEM results offer important insights into the microstructure of both NiZCF and NiZCF/EPDM

rubber nanocomposites, providing a visual understanding of their structural characteristics and the effective dispersion of nanoparticles within the polymer matrix.

3.4 | Curing characteristics

Table 2 presents the rheometer properties of both pure EPDM rubber and the NiZCF/EPDM rubber nanocomposite. It reveals important curing characteristics, including the optimum cure time (tc_{90}), scorch time (ts_2), maximum torque (M_H), minimum torque (M_L), and torque change (ΔM) of EPDM rubber, which are influenced by the incorporation of ferrite nanofillers, as illustrated in Figure 6A,B. Table 2 depicts that the M_L , M_H , and ΔM of the composite exhibit higher values in comparison to pure EPDM rubber from 0 to 5 phr. This can be attributed to the increased surface area of the composites, resulting from enhanced rubber-ferrite interactions. However, at 8 phr, a decline in all these values was observed, which can be correlated with decrease filler-matrix interactions at high loading. These findings align with similar observations in Ca—Co ferrite/EPDM nanocomposite reported elsewhere.⁵⁹ Figure 6B illustrates a notable decrease in both scorch time (ts_2) and cure time (tc_{90}) compared to pure EPDM, indicating accelerated sulfur curing in the presence of loaded ferrite nanoparticles. This acceleration can be attributed to several factors. First, the high surface area of the NiZCF nanofiller facilitates increased interaction with the sulfur curing agent (TMTD), promoting more efficient crosslinking within

the rubber matrix. Additionally, the presence of transition metal ions on the surface of the nanoparticles may catalyze sulfur vulcanization reactions, further enhancing the curing process. Moreover, the nanofiller's dispersion within the EPDM matrix creates a heterogeneous environment that facilitates nucleation and propagation of crosslinks, leading to faster curing kinetics. Overall, these mechanisms collectively contribute to the observed changes in curing characteristics.⁴⁸

These results highlight the impact of ferrite nanoparticles on the curing properties of the nanocomposite, showing the potential for improved material performance due to enhanced interactions between the rubber and ferrite components.

3.5 | Mechanical properties

The assessment of mechanical properties, elucidated through stress-strain curves, is important in characterizing the quality and characteristics of rubber composites. Among these properties, tensile strength and Young's modulus stand out as key parameters, shedding light on the behavior of the prepared composites. As depicted in Figure 7, an increase in filler loading within the EPDM rubber matrix correlates with a corresponding rise in

TABLE 2 Rheometric characteristics with respect to phr of filler loading in NiZCF/EPDM rubber nanocomposites.

	0 phr (Pure EPDM)	1 phr	3 phr	5 phr	8 phr
M_L (Nm)	2.22	1.83	2.35	2.56	2.44
M_H (Nm)	12.85	12.92	12.96	12.88	12.40
ΔM (Nm)	10.63	11.09	10.61	10.32	9.96
tc_{90} (min)	29	7.19	9.19	6.76	11.72
ts_2 (min)	24.08	6.81	6.60	3.71	3.50

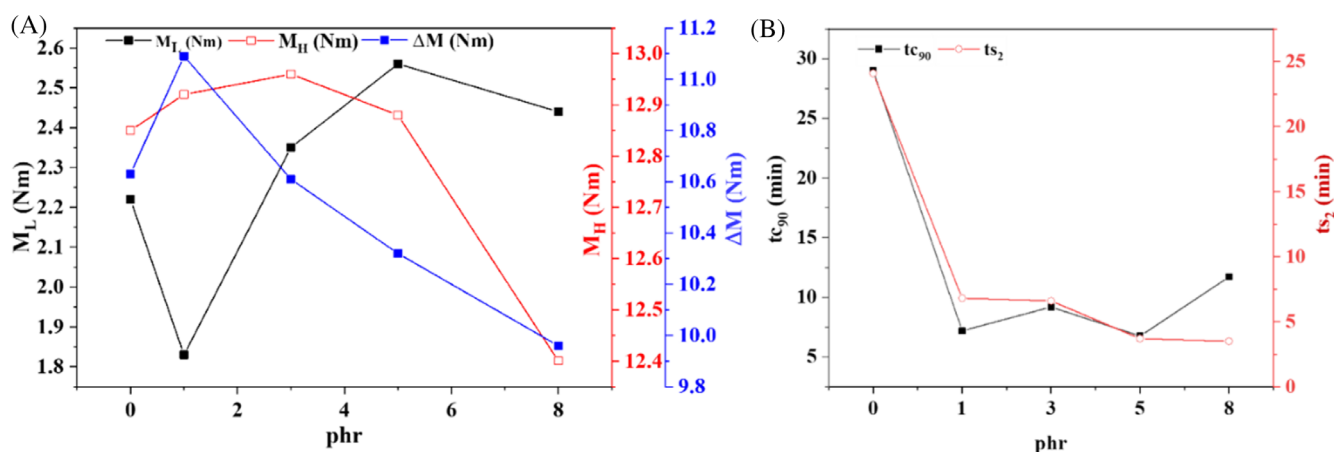


FIGURE 6 (A) phr versus maximum torque (M_H), minimum torque (M_L), and torque change (ΔM) (B) phr versus scorch time (ts_2) and cure time (tc_{90}).

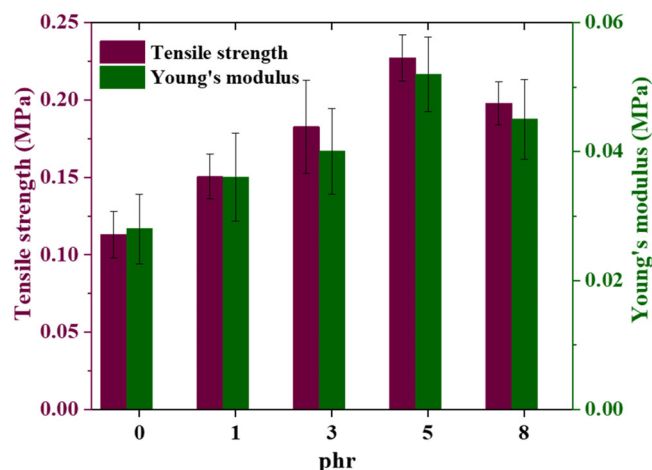


FIGURE 7 Tensile strength and Young's modulus of synthesized NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposites as a function of increasing phr of ferrites.

both tensile strength and Young's modulus. This phenomenon can be attributed to the reinforcement effect imparted by the NiZCF nanofillers. Consequently, the mechanical properties experience enhancement due to the significant interactions between the rubber matrix and the filler, indicating a uniform and homogeneous dispersion of nanofiller in the matrix, as depicted by SEM micrographs. Comparable outcomes have been reported in the context of Mn-Zn ferrite nanocomposites.^{39,60} Notably, within the filler loading range of 0 to 5 phr, tensile strength and Young's modulus showed significant improvement, increasing from 0.11 MPa to 0.22 MPa and 0.03 MPa to 0.05 MPa, respectively. This corresponds to a percentage increase of 100% for tensile strength and 86% for Young's modulus. However, a decline was observed in both tensile strength and Young's modulus at 8 phr, which can be attributed to the agglomeration of ferrite particles at higher loading levels. The accelerated sulfur curing process, indicated by decreased scorch time (t_{s2}) and cure time (t_{c90}) compared to pure EPDM (Figure 6A,B), contributes to the observed mechanical enhancements. The high surface area of the NiZCF nanofillers facilitates enhanced interaction with the sulfur curing agent, promoting more efficient crosslinking within the rubber matrix, resulting in improved mechanical properties. These findings highlight the significant role of filler loading in modulating the mechanical properties of the nanocomposites and provide valuable insights into their behavior under different loading conditions.

3.6 | Dielectric properties

The dielectric properties of the prepared nanocomposite were measured in term of dielectric resistivity, dielectric

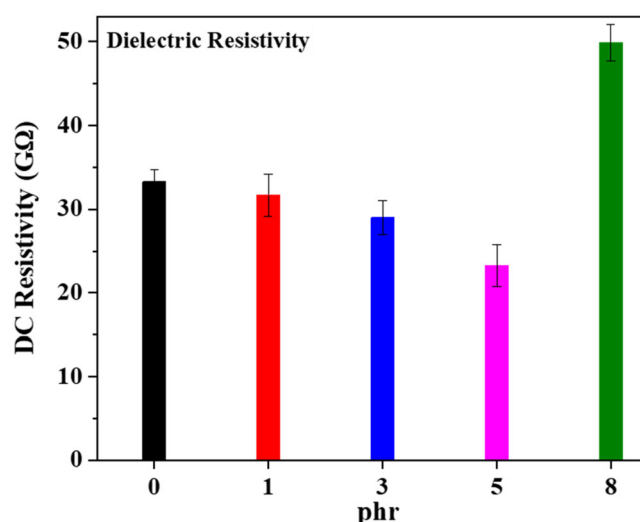


FIGURE 8 Dielectric resistivity of NiZCF/ethylene propylene diene monomer (EPDM) nanocomposites as a function of increasing phr of ferrite loading.

permittivity, and dielectric constant. That all are discuss below:

3.6.1 | Dielectric resistivity

As shown in Figure 8, incorporating conductive ferrite into an insulating EPDM rubber nanocomposites facilitates the formation of a continuous network, ultimately reducing the resistivity of the material. An interesting phenomenon occurs after the addition of a certain amount of filler: resistivity experiences a sudden and significant decrease. This marks the point at which further filler addition does not produce substantial changes in resistivity, a phenomenon known as the percolation limit.

Prior to reaching this limit, the NiZCF nanoparticles are dispersed in an isolated fashion within the polymer matrix. It is well established in previous reports, once the percolation limit is reached, the resistivity stabilizes and remains constant. This stability arises from the formation of a continuous network of ferrite particles through an aggregation process.⁶¹ However, in our study, an unexpected phenomenon occurs at 8 phr. The dielectric resistivity increases sharply at this point. This abrupt increase can be correlated with the agglomeration of NiZCF nanoparticles at 8 phr. In the specific case of the NiZCF/EPDM rubber nanocomposite, the incorporation of ferrite initially leads to a reduction in the resistivity of EPDM rubber. Remarkably, the percolation limit for this composite is determined to be 5 phr, where the uniform dispersion of filler in the rubber matrix was achieved which is also clear from the SEM results (Figures 4 and 5).

These observations highlight the electrical behavior of the nanocomposite, its transition from dispersed particles to a continuous conductive network, and the unexpected resistivity change at 8 phr due to ferrite nanoparticle agglomeration. These findings align well with previous research in the field of nanocomposites, substantiating the concept of the percolation limit.⁴⁰

3.6.2 | Dielectric permittivity

Figure 9 exhibits the variation in permittivity as a function of filler loading. As obvious from the Figure 9, as the filler loading increases from 0 to 5 phr, dielectric permittivity gradually rises. This phenomenon can be attributed to the presence of various polarization mechanisms, including molecular, ionic, electronic, and interfacial mechanisms, within the prepared composite material.

At lower frequencies, interfacial and dipolar polarization mechanisms dominate, while atomic and ionic polarization mechanisms become more pronounced at higher frequencies.^{62,63} In the case of NiZCF/EPDM rubber nanocomposite, ferrite nanoparticles contain ions such as Ni^{+2} , Zn^{+2} , Cu^{+2} , Fe^{+2} , and Fe^{+3} which are surrounded by O^{-2} ions. When an external electric field is applied, these electric dipoles align themselves with the field, leading to dipolar polarization. As ferrite nanoparticles loading increases in the EPDM rubber matrix from 0 to 5 phr, more electric dipoles become available, enhancing the dipolar polarization and consequently the dielectric permittivity of the composite material.⁶⁴

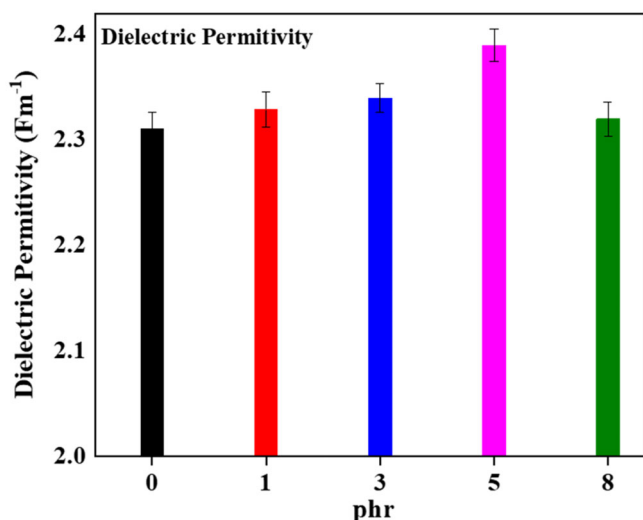


FIGURE 9 Dielectric permittivity of NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposites as a function of increasing phr of ferrite loading.

Figure 9 shows that the permittivity value increases from 2.31 to 2.39 (Fm^{-1}) with the incorporation of ferrites into the composite. This observation aligns with similar findings reported for ferrite/EPDM rubber nanocomposites.^{65,66} Furthermore, At the interfaces between the ferrite nanoparticles and the EPDM rubber matrix, interfacial polarization occurs. This polarization mechanism involves the movement of charges at the interface in response to the applied electric field. The hopping of electrons within and across boundaries also contributes to interfacial polarization, further influencing the permittivity of the composite materials.³⁹ However, at 8 phr, the value of dielectric permittivity decreased, which again can be correlated with the agglomeration of NiZCF nanoparticles at 8 phr.

3.6.3 | Dielectric constant

The dielectric constant serves as a valuable parameter for studying the relaxation behavior of the nanocomposites. Figure 10 provides insights into the variation in the dielectric constant of NiZCF/EPDM rubber nanocomposites as a function of increased filler loading fractions, ranging from 0 to 8 phr in the rubber matrix. Basically, the relaxation behavior of polymer nanocomposites is intricately linked to the interaction and distribution of the filler within the rubber matrix.⁶⁷ Similar observations have been reported for other nanocomposites, such as nanographite/EPDM rubber nanocomposites⁶⁸ and CaO/EPDM rubber composites.⁶² The graph in Figure 10 reveals a clear trend: as the ferrite loading increases from 0 to 5 phr, the dielectric

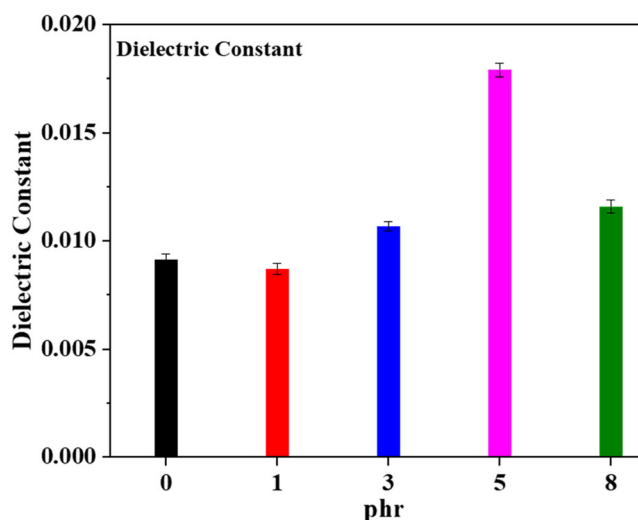


FIGURE 10 Dielectric constant of NiZCF/ethylene propylene diene monomer (EPDM) rubber nanocomposites as a function of increasing phr of ferrite loading.

constant value also rises, indicative of improved physico-chemical and hydrodynamic interactions between the filler and the rubber matrix.⁶⁹ However, at 8 phr, a decrease in the dielectric constant is observed, which can be correlated with the agglomeration of ferrite nanoparticles at 8 phr, a factor that is consistent with the observed effect on electrical resistivity in the same material. This deviation from the previously observed trend at higher filler loading shows the complexity of filler dispersion within the nanocomposite and its direct influence on electrical properties.^{70,71} These results demonstrate the significance of filler loading in modulating the dielectric constant and, by extension, the relaxation behavior of NiZCF/EPDM rubber nanocomposites.

Remarkably, both the mechanical and electrical properties exhibited a similar trend: their values steadily increased from 0 to 5 phr of filler loading and then declined at 8 phr. This consistent behavior suggests a notable correlation between filler loading and the observed properties.

4 | CONCLUSIONS

In this study, we successfully prepared Ni-Zn-Cu Fe₂O₄ ferrite/EPDM rubber nanocomposites, aiming to enhance their mechanical and electrical properties. The tailored composition of NiZn_(0.3)Cu_(0.2)Fe₂O₄ (2) ferrite nanoparticles was synthesized via the sol-gel method and uniformly incorporated into the EPDM rubber matrix using a two-roll mixing mill. Through comprehensive characterization, we gained valuable insights into the morphology and structure of the resulting ferrite/EPDM nanocomposites. SEM and XRD analysis unveiled their physical attributes, while FTIR spectroscopy identified specific metal-oxygen bonds within the nanocomposites. To evaluate their suitability for practical use, we examined crucial curing properties, including optimum cure time (tc₉₀), scorch time (ts₂), and torque profiles, utilizing a MDR. The values of tc₉₀ and ts₂ presented a gradual decrease with increase in filler loading from 0 to 5 phr. Similarly, torque profiles increased from 0 to 5 phr. The mechanical performance of the nanocomposites, evaluated in terms of tensile strength and Young's modulus, demonstrated increases of 100% and 86%, respectively, compared to EPDM rubber without the addition of a filler. Furthermore, the dielectric properties of these nanocomposites, including permittivity, dielectric constant, and resistivity were thoroughly investigated using a dielectric properties analyzer. The values of dielectric permittivity and dielectric constant were increased from 2.31 to 2.39 (Fm⁻¹) and from 0.0091 to 0.0179, respectively. Meanwhile, resistivity was decreased from 33.2 GΩ to

23.38 GΩ upon the introduction of Ni-Zn-Cu Fe₂O₄ ferrite nanoparticles, confirming the establishment of a continuous nanofiller network within the EPDM matrix.

In conclusion, these findings unveil the significant potential of the prepared Ni-Zn-Cu Fe₂O₄ ferrite/EPDM nanocomposites. These materials exhibit enhanced mechanical strength, reduced dielectric resistivity, and tunable dielectric properties, rendering them promising candidates for a wide range of mechanical and electrical applications. Our study contributes to the ongoing advancements in ferrite/EPDM rubber nanocomposites, offering new possibilities for innovation in materials science and technology.

AUTHOR CONTRIBUTIONS

The research project was a collaborative effort led by Asma Anam Hanif, Nazia Parveen, Shahid Nazir, and Zulfiqar Ali. Asma Anam Hanif and Toheed Akhter conceptualized the study, with methodology design primarily by them. Validation was conducted by Toheed Akhter, Shahid Nazir, and Zulfiqar Ali. Asma Anam Hanif handled formal analysis, while investigative efforts were shared between Asma Anam Hanif and Toheed Akhter. Resource allocation was overseen by Shahid Nazir, Zulfiqar Ali, Asif Mahmood, and Waheed Al-Masry. The original draft of the manuscript was prepared by Toheed Akhter, Sadaf Ul Hassan, Aamal Rehman, and Nazia Parveen, with subsequent review and editing undertaken by Muhammad Maqbool, Aamal Rehman, Nazia Parveen, Shumaila Razzaque, Taewook Kim, Soo-Kyung Han, and Chan Ho Park. Supervision throughout the project was provided by Toheed Akhter, Shahid Nazir, and Zulfiqar Ali, with all authors consenting to the final published version of the article.

ACKNOWLEDGMENTS

The authors would like to acknowledge the Researcher's Supporting Project Number (RSP2024R43), King Saud University, Riyadh, Saudi Arabia. This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korean Government (MSIT) (no. RS-2023-00251670) and Technology Development Program of MSS (RS-2023-00251434).

DATA AVAILABILITY STATEMENT

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

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How to cite this article: Parveen N, Hanif AA, Hassan Su, et al. Tailored Ni-Zn-Cu ferrite/EPDM nanocomposites: Synthesis, characterization, and multifaceted properties for mechanical and electrical applications. *Polym Compos.* 2024;45(12): 10720-10733. doi:[10.1002/pc.28503](https://doi.org/10.1002/pc.28503)