ORIGINAL PAPER

The influence of metal nanoparticles on the performance properties of a polymer film material based on polyethylene

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ABSTRACT

This study investigates the effects of incorporating copper (Cu), zinc oxide (ZnO), and zirconium dioxide (ZrO_2) nanoparticles at concentrations of 2.2, 4.3, and 8.7 wt.% on the tensile strength, water vapor transmission rate, and antimicrobial properties of polyethylene-based films (46 g base polymer). Polyethylene nanocomposites were prepared via melt extrusion. Results demonstrate that nanoparticle types and concentrations significantly influence composite performance through distinct mechanisms. Cu nanoparticles exhibited the most balanced enhancement, with optimal elongation and barrier performance at intermediate concentrations. With 4.3wt.% loading, ZnO nanoparticles improved tensile strength the most, while ZrO_2 nanoparticles demonstrated clearly mechanical reinforcement at lower loadings and performance degradation at higher concentrations due to particle agglomeration. Bacterial activities showed significant results at the highest loading concentration for all the nanoparticles. However, ZrO_2 achieved 94% reduction against both B. subtilis and E. coli at 2.2 wt.% loading surpassing Cu and ZnO. The incorporation of nanoparticles led to a reduction in water vapor transmission rates, with the best barrier properties noted at 4.3 wt.% for the Cu and ZnO types, excluding ZrO_2. This comprehensive analysis provides critical insights for designing multifunctional polyethylene nanocomposite films for packaging and biomedical applications.

Keywords: copper nanoparticles; zinc oxide nanoparticles; zirconium dioxide nanoparticles; nanocomposite; mechanical properties; barrier and antimicrobial activity.

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INTRODUCTION

Polyethylene (PE) remains the most widely used polymer globally, finding extensive application in packaging, agricultural, medical devices, and consumer products due to its ease of synthesis, chemical resistance, and cost-effectiveness [1,2]. Nonetheless, conventional PE films face inherent limitations in mechanical strength, barrier properties against gases and moisture, and lack of antimicrobial functionality, which restrict their performance in demanding applications, particularly as active edible packaging, electronics, and biomedical materials.

The emergence of polymer nanotechnology has opened new avenues for addressing these limitations through the integration of nanoparticles either in the form of metal or metal oxide [3,4]. The main

advantage of the nanoparticles is the high surface-to-volume ratio, which enables significant property modifications at relatively low filler loadings. Among the various nanofillers investigated, copper (Cu), zinc oxide (ZnO), and zirconium dioxide (ZrO_2) nanoparticles have attracted particular attention for their distinct functional characteristics.

Copper nanoparticles are renowned for their potent antimicrobial properties, specifically against fungi, viruses, and Gram-positive and Gram-negative bacteria [5]. The antimicrobial nature involves the release of Cu^{2+} ions that disrupt bacterial cell membranes, generate reactive oxygen species (ROS), and interfere with essential enzymatic functions [6-8]. Studies on LDPE/Cu nanocomposites have demonstrated that Cu-NPs can simultaneously improve mechanical properties and barrier performance while providing effective antimicrobial protection [9–11]. However, the concentration-dependent effects of Cu nanoparticles on polymer properties require careful optimization, as excessive loading can lead to agglomeration and performance degradation [12,13].

Zinc oxide nanoparticles offer a compelling combination of biocompatibility, UV-blocking capability, and antimicrobial activity [14,15]. ZnO material is safe and recognized by the FDA, making it particularly attractive for food contact applications [16-19]. The incorporation of ZnO nanoparticles in LDPE with different concentration levels has been examined in literature. Particularly, ZnO nanoparticles with 1%, 3%, and 5% extruded with LPDE and used in packaging of strawberries. It showed maximum antimicrobial activity with 5% concentration of ZnO nanoaprticles. The study of ZnO nanoparticles can enhance the mechanical properties of polyethylene composites at low concentrations, with optimal performance typically observed below 5 wt.% loading, as reported [20]. The antimicrobial mechanism of ZnO involves the generation of ROS and release of Zn^{2+} ions, which exhibit selective toxicity toward microbial cells while maintaining biocompatibility with mammalian cells [21].

Zirconium dioxide nanoparticles are distinguished by their exceptional chemical inertness, thermal stability, and radiation resistance [22]. ZrO_2 has been explored as a reinforcing filler for polyethylene matrices, with studies investigating its effects on mechanical properties and barrier performance [22]. Research on LDPE/ZrO_2 nanocomposites has revealed that nanoparticle incorporation affects tensile characteristics and water vapor transmission rates, though excessive concentrations may lead to diminished performance due to agglomeration.

Despite the individual promise of these nanoparticles, a comprehensive comparative analysis of their effects on polyethylene properties under identical processing conditions and concentration ranges has been lacking. The present study addresses this gap by systematically investigating the influence of Cu, ZnO, and ZrO_2 nanoparticles at three concentrations (2.2, 4.3, and 8.7 wt.%) on the tensile strength, WVTR, and antimicrobial activities of polyethylene films prepared from 46 g of polymer base.

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Experimental

Materials

Low-density polyethylene (LDPE) with a white granule colour, GOST 16336-2013, density 0.921g/cm^3 was obtained from manufacturer Tomskneftekhim LLC, Tomsk, Russia. Copper nanoparticles (average particle size 50 nm, density (1- 4 g/cm^3), purity> 99.5%), zinc oxide nanoparticles (average particle size 20-40 nm, purity >99.0%, density 4-8g/cm^3), and zirconium dioxide nanoparticles (purity 99.9%, Particle size 80-100nm and density 5.68g.cm^3) where all the three nano particles were purchased from mega group of Russia. Nanoparticles are represented by NPs throughout the whole text.

Preparation of nanocomposite films

Polyethylene nanocomposite films were prepared via a melt extrusion process through a single-screw extrusion elastomer machine (model: ISD551M21B, screw diameter (20 mm), length-to-diameter ratio (25-36), motor power (1.5-3 kW), Moscow, Russia). Melt compounding was used to extrude the material and various fillers. Prior to processing, 46 g of LDPE pellets and the requisite quantities of nanoparticles were maintained in a controlled environment. It has four zones for temperature setup, such as feed (both LDPE and Nanoparticles mixed and entered), compression, metering, and die zone. Each zone of the extruder was calibrated at 100,120, 150, and 145 ℃, respectively. The nanoparticle loadings were calculated as weight percentages relative to the total composite mass: 2.2 wt.%, 4.3 wt.%, and 8.7 wt.%, corresponding to nanoparticle masses of approximately 1.03 g, 2.07 g, and 4.38 g per 46 g polymer, respectively. According to current literature, nanoparticle concentrations of less than 10% are more effective at altering the characteristics of traditional materials [20].

The single screw rotation of the extruder was adjusted at medium rpm and 23 Nm of torque. The die zone of the extruder was set with a melting pressure of 5 kg/cm^2. After all the parameters were acquired, the LDPE with Cu, ZnO, and ZrO_2 NPs were added to the feed section separately. 46 g of LDPE pellets incorporated 2.2wt.%, 4.3wt.%, and 8.7wt.% of nanoparticles, and control without nanoparticles were examined. LDPE-0, LDPE/NPs-1, LDPE/NPs-2, and LDPE/NPs-3 represented 0, 2.2, 4.3, and 8.7% concentration of the NPs, respectively. The pellet of the LDPE and powder of the three NPs were thawed in each section of the barrel zones through the shearing operation. The continuous screw strand at the die zone was cooled through the water channel. The composite synthesized was followed by cooling to room temperature under pressure. The produced films were wrapped with aluminum foil to minimize contamination and loss of the antimicrobial agent. Neat LDPE films were prepared under identical conditions for comparison.

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Characterization methods

Mechanical properties

Mechanical properties were evaluated according to ASTM D638 using a universal Labthink, China Auto tensile tester machine (model: C610H). Five replicated slices were calculated to get the average tensile strength, elongation, and young's modulus of the polyethylene nanocomposite film materials.

Barrier properties

Water vapor transmission rate (WVTR) and Water vapor permeability (WVP) were determined through a water vapor test system (Labthink, Model: C360M, Serial number: B1360V2R1-000-024001-E03XP, Jinan - 250031, Shandong, China). The standard GB-1037 and wet methodology were operated at a temperature of 38℃, 10% RH, and a wind speed of 0.5 m/s. The results were recorded after 24 h.

Antimicrobial activity

Two bacterial strains were selected for antimicrobial properties analysis. Bacillus subtilis and Escherichia coli, which are Gram-positive and Gram-negative microorganisms, respectively. The shake flask method was used to evaluate the antibacterial nature of the synthesized nanocomposite film materials.

For the shake flask method, film specimens (5 cm × 5 cm) were immersed in nutrient broth containing approximately 10^5 CFU/mL of test bacteria and incubated at 37°C with shaking for 24 hours. Bacterial viability was determined by serial dilution and plate counting. Percent reduction was calculated relative to control samples without nanoparticles.

A colony-forming unit (CFU) refers to a collection of cells capable of forming a colony. It's important to understand that a CFU can consist of a single cell or a group of cells that are clumped together.

Percentage reduction calculation

$$\text{Percent reduction} = \frac{A - B}{A} \times 100 \quad (1)$$

where:

A is the viable count of the control (before treatment) and B is the viable count of the supplements (after treatment).

Morphological characterization

Nanoparticle dispersion in the polyethylene nanocomposite film materials was examined using a digital microscope (Bresser GmbH, LCD Microscope V5.0, Gutenbergstr, 2-46414 Rhede, Germany). A digital microscope is a device that uses a camera sensor to turn an enlarged image of an item into a digital signal, which is then shown on a laptop. The microscope specifications are three lenses (4X,

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10X, and 40X) and 30- 1015X optical magnification, with an LCD touch screen and 5 MP camera. Only the 10× lens was used to scan the surface of the nanocomposite samples.

OriginLab software (OriginLab Corporation, America) was used to conduct the statistical analysis in order to identify the important variations in the characteristics of polyethylene nanocomposite film materials. This software enabled a direct visual comparison of the means for correctness in analysis within each data set and provided access to more precise scientific figures and graphs that aid in publication in recurring journals.

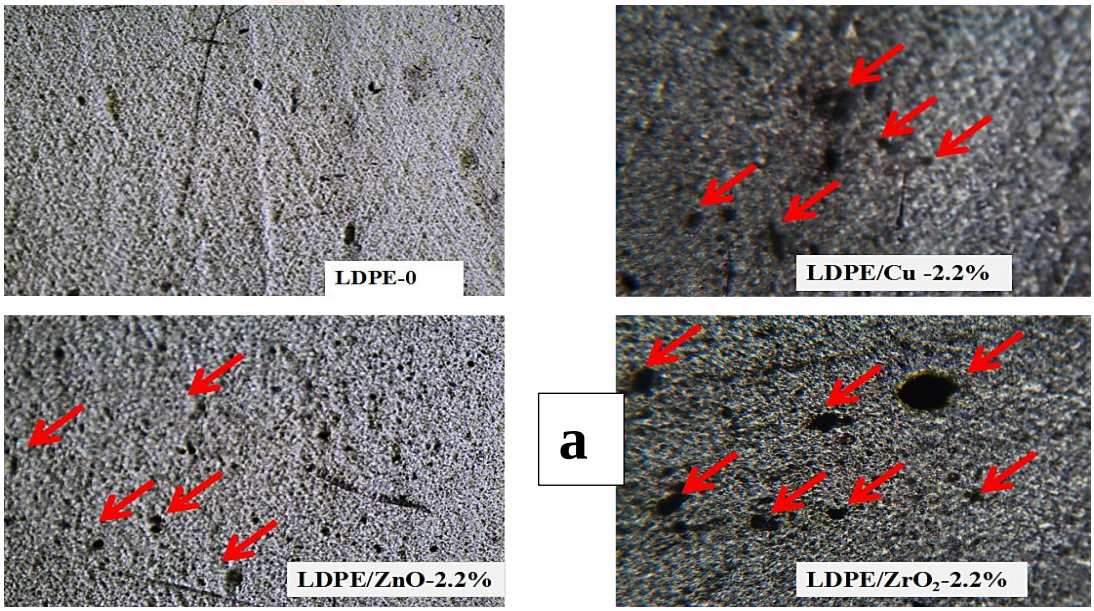
RESULTS AND DISCUSSION

Morphological analysis

The morphological samples are presented in Figure1. Digital microscope images of the polyethylene nanocomposites revealed distinct dispersion characteristics for each nanoparticle type. Cu nanoparticles at 2.2 and 4.3 wt.% loadings exhibited relatively uniform dispersion throughout the polymer matrix, with individual nanoparticles and small agglomerates observed. However, at 8.7 wt.% Cu loading, significant agglomeration was evident with particle clusters [3,23]. This observation is consistent with the high surface energy of metal nanoparticles, which drives aggregation at elevated concentrations [3].

ZnO nanoparticles demonstrated good dispersion quality across the concentration range studied, with only minor agglomeration observed at 8.7 wt.% loading. The relatively better dispersion of ZnO compared to Cu nanoparticles may be attributed to surface oxide layers that reduce interparticle interactions and improve compatibility with the polyethylene matrix [21,24].

ZrO_2 nanoparticles exhibited the most pronounced agglomeration among the three nanoparticle types, particularly at 4.3 and 8.7 wt.% loading, where large particle clusters were frequently observed [22]. This agglomeration behavior is consistent with the reported tendency of ZrO_2 nanoparticles to form strong interparticle bonds due to their high surface energy and lack of surface functionalization [25,26].



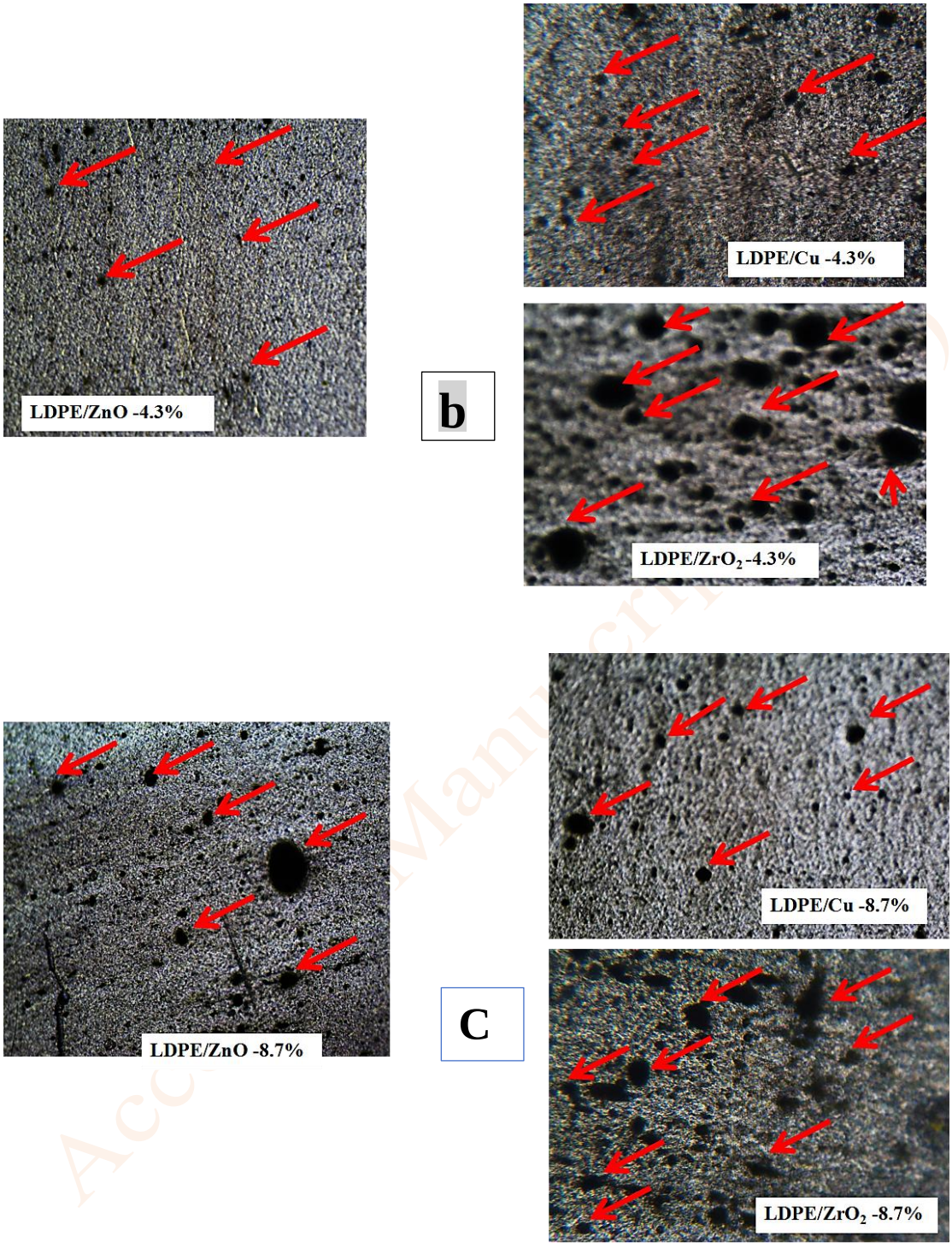


Figure 1. Morphological analysis of neat LDPE and all nanocomposites with 2.2wt.%(a),4.3 wt.%(b), and 8.7wt.%(c) loading.

Mechanical properties

Tensile strength

The incorporation of nanoparticles significantly influenced the tensile strength of polyethylene films, with the effect being highly dependent on both nanoparticle type and concentration as shown in Figure

2. At 2.2wt.% concentration of Cu naoparticles, the tensile strength recorded to 8.07MPa. It showed 26% decrement compared to the pure PE. On the other hand, Cu nanoparticles exhibited good pronounced reinforcing effect, with tensile strength increasing from 10.84 MPa for pure LDPE(LDPE-0) to 12.62 MPa at 4.3 wt.% Cu loading, representing a 16.4% improvement. This enhancement can be attributed to the uniform dispersion of Cu nanoparticles, which act as stress concentrators and promote energy dissipation through interfacial interactions and plastic deformation mechanisms [3]. The observed improvement is consistent with previous studies on LDPE/Cu nanocomposites [3,23].

ZnO nanoparticles showed highest improvement, with tensile strength reaching 14.35 MPa at 4.3 wt.% loading (32.4% improvement) compared with Cu and ZrO_2 nanoparticles at the same loading. The reinforcing effect of ZnO is attributed to good dispersion and favorable polymer-nanoparticle interactions that accelerate stress transfer from the polymer matrix to the rigid nanoparticles [1,21]. However, at 8.7 wt.% ZnO loading, tensile strength decreased to 10.5 MPa, slightly above the neat polymer value, indicating that excessive ZnO content leads to agglomeration and reduced stress transfer efficiency.

ZrO_2 nanoparticles demonstrated the weakest reinforcing effect among the three nanoparticles, with tensile strength increasing only to 12.3 MPa at 2.2 wt.% loading (13.5% improvement). At 4.3 and 8.7 wt.% ZrO_2 loading, tensile strength fell below the neat polymer value (9.25 and 6.0 MPa), representing a 25 and 45% reduction, respectively. This performance degradation is consistent with the observed agglomeration of ZrO_2 nanoparticles at high concentrations, which creates stress concentration points and reduces the effective polymer-nanoparticle interfacial area [22].

The concentration-dependent behaviour observed for all nanoparticle types is consistent with the established understanding that nanoparticle reinforcement follows a threshold effect: at low to moderate concentrations, nanoparticles enhance mechanical properties through efficient stress transfer and energy dissipation, while at high concentrations, agglomeration leads to performance degradation [23]. The optimal loading for mechanical reinforcement was determined to be 4.3 wt.% for Cu and ZnO nanoparticle types, though the magnitude of reinforcement varied significantly with nanoparticle composition.

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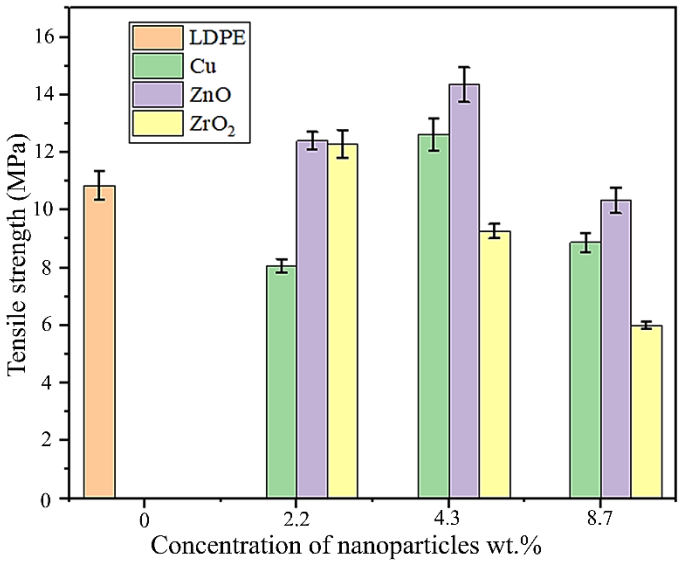


Figure 2. Tensile strength versus concentration of nanoparticles.

Elongation at break

Figure 3 elaborated on the elongation at break. Cu nanoparticles caused a significant increment, with elongation increasing to 22% for neat LDPE at 4.3 wt.% Cu loading. At 2.2 wt.% Cu loading, elongation was equal to neat LDPE. The reduction in elongation at 8.7wt.% Cu loading relative to 4.3wt.% (22.9% reduction) indicates that high Cu content substantially increases the brittleness of the polyethylene matrix [3].

ZnO nanoparticles had a moderate increment effect on elongation, increasing to 536% at 4.3 wt.% loading (8.9% improvement) and further to 511% at 8.7 wt.% loading (3.9% improvement). This relatively modest increment suggests that ZnO nanoparticles maintain some increase in ductility in the nanocomposite films, which may be attributed to their surface characteristics and interactions with the polymer matrix [27].

Interestingly, ZrO_2 nanoparticles showed the highest reduction in elongation at break, with values of 482% at 2.2 wt.%, 465% at 4.3 wt.%, and 367% at 8.7 wt.% loading. The highest reduction in elongation at 8.7 wt.% ZrO_2 loading (367%) compared to neat LDPE (492%) suggests that low concentrations of ZrO_2 may promote localized deformation mechanisms that enhance extensibility before failure [25]. However, the significant reduction at 8.7 wt.% loading (367%) indicates that excessive ZrO_2 content compromises ductility.

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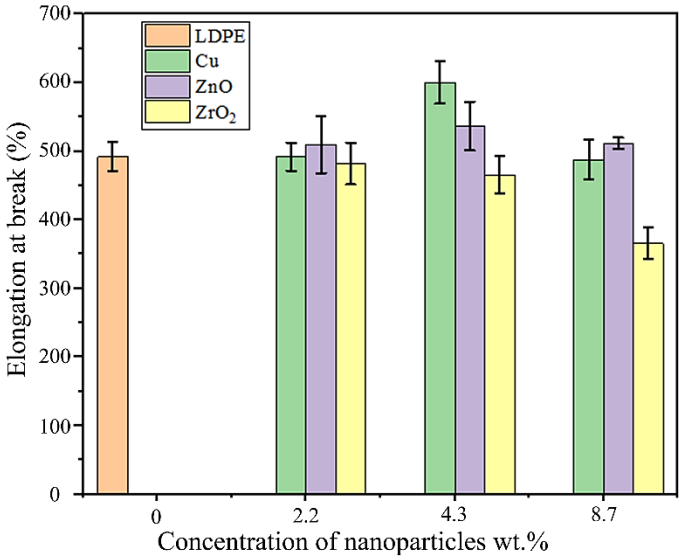


Figure 3. Elongation at break versus concentration of nanoparticles.

Young's Modulus

The Young's modulus value versus the concentration of the nanoparticles is presented in Figure 4 below. Young's modulus increased with nanoparticle incorporation for all nanoparticle types with small loading, reflecting the stiffening effect of rigid inorganic fillers. Cu nanoparticles demonstrated the most significant stiffening, with modulus increasing from 105.52 MPa for neat LDPE to 150.49 MPa at 2.2 wt.% Cu loading (42.6% increase). The stiffening effect is attributed to the high inherent stiffness of Cu nanoparticles and their effective stress transfer capacity when well-dispersed [3]. However, the Young's modulus with 4.3wt.% loading, shows significant decrement (28% reduction).

ZnO nanoparticles showed high stiffening, with modulus reaching 155.49 MPa at 4.3 wt.% loading (47.4% increase) and 122.38MPa at 2.2wt.% loading (16% increase), but low stiffening was recorded with 8.7wt.% loading (21% decrease).

ZrO_2 nanoparticles exhibited the smallest modulus enhancement, reaching 116 MPa at 2.2 wt.% loading (10% increase). With 4.3 wt.% loading, the modulus obtained smallest to 92MPa. The lower stiffening effect of ZrO_2 is consistent with its tendency to form agglomerates, which reduces the effective filler aspect ratio and stress transfer efficiency.

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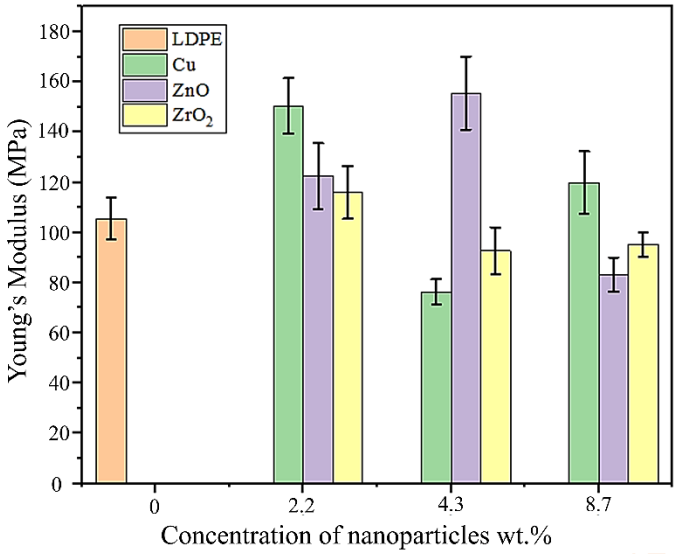


Figure 4. Young's Modulus versus concentration of nanoparticles.

Barrier properties

Water vapor transmission rate and water vapor permeability

All nanoparticle types reduced the WVTR and WVP of polyethylene films, indicating improved water vapor barrier properties. Cu nanoparticles were most effective, reducing WVTR from 29.42(g/ (m^2.24h)) for neat LDPE to 4.45(g/ (m^2.24h)) at 8.7 wt.% loading (84% reduction). Besides, the value of WVP with 2.2wt.% loading recorded to 0.9 (g*cm/ (cm^2.s. Pa))×10^{-14}(71% reduction). The barrier enhancement is attributed to the creation of tortuous diffusion pathways for water molecules around impermeable nanoparticles and the immobilization of polymer chains near nanoparticle surfaces, which restricts segmental mobility and water vapor permeation [3].

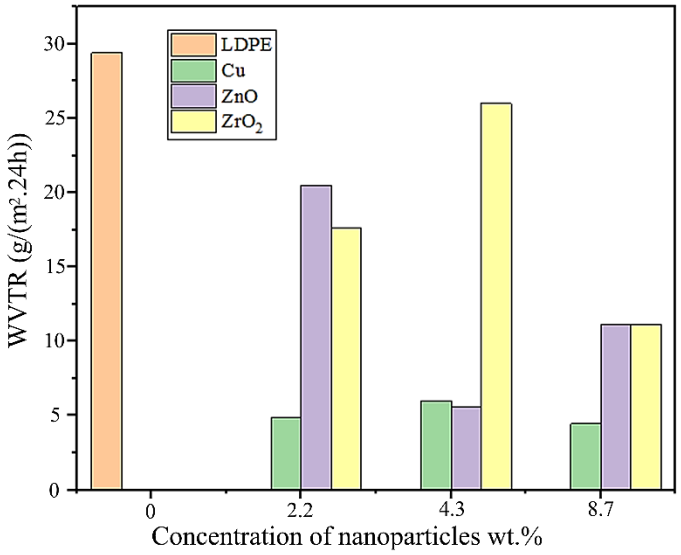
ZnO nanoparticles showed moderate WVTR reduction, reaching 20.7 (g/(m^2 ·24 h)) at 2.2 wt.% loading (29.6% reduction). The improvement is consistent with the well-established barrier enhancement effects of ZnO nanoparticles in polymer matrices [1]. ZnO nanoparticles exhibited the highest WVTR reduction, reaching 5.6(g/ (m^2.24h)) at 4.3 wt.% loading (81% reduction).

ZrO_2 nanoparticles led to a slight reduction in WVTR and WVP, recording 26 (g/ (m^2.24h)) and 2.8 (g.cm/ (cm^2.s.Pa))×10^{-14} with 4.3wt.% loading, respectively. With 8.7wt.% loading, the ZrO_2 nanoparticles showed a significant reduction of the WVTR and WVP. The relatively lower barrier enhancement of ZrO_2 may be attributed to its agglomeration tendency, which reduces the effective nanoparticle surface area available for barrier improvement [22].

Remarkably, ZrO_2 nanoparticle types compared to Cu and ZnO showed increased WVR at all concentration loading, indicating that nanoparticle and PE have weak interaction that compromises barrier performance. This phenomenon is consistent with the formation of nanoparticle agglomerates that create defects and micro voids in the polymer matrix, providing preferential pathways for water vapor permeation [16].

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(a)



(b)

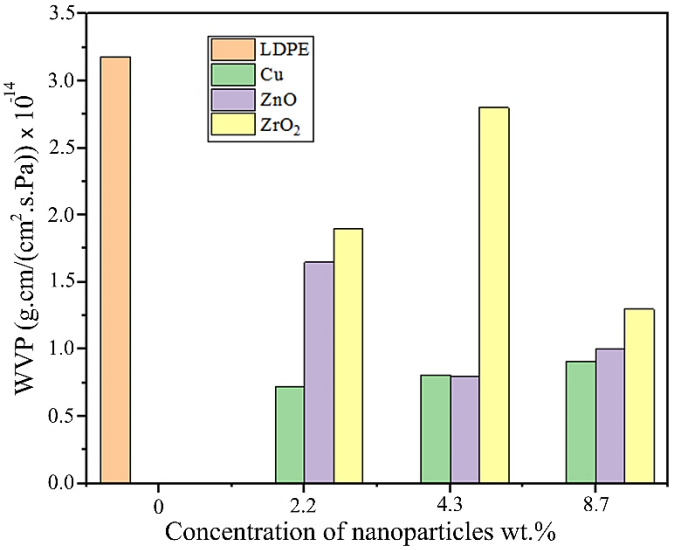


Figure 5. Barrier properties of WVTR(a) and WVP(b) versus concentration of nanoparticles.

Antimicrobial properties

The antibacterial efficacy of the polyethylene nanocomposite samples is displayed in Table 1, and the percent reduction of the microorganism is determined by equation 1.

Table 1. Antimicrobial activity of polyethylene nanocomposites.

ample	B. Subtilis colonies	B. Subtilis Reduction (%)	E. coli colonies	E. coli Reduction (%)
Neat LDPE	>300	0	>300	0
LDPE/Cu 2.2%	83	71	7	97.7
LDPE/Cu 4.3%	18	94	6	98
LDPE/Cu 8.7%	1	99.7	2	99.3
LDPE/ZnO 2.2%	55	81	50	83.3
LDPE/ZnO 4.3%	8	97	3	99
LDPE/ZnO 8.7%	4	98.7	5	98.3
LDPE/ZrO_2 2.2%	17	94	20	93.3
LDPE/ZrO_2 4.3%	14	95	15	95
LDPE/ZrO_2 8.7%	3	99.3	2	99.3

With the exception of B. subtilis at 2.2wt.% loading, Cu nanoparticles demonstrated strong antimicrobial activity, reducing both bacterial cells by more than 93.9% at all concentrations. At 2.2 wt.% Cu loading, bacterial reductions of 71% for B.subtilis, and 97.7% for E. coli were achieved, demonstrating significant antimicrobial efficacy even at low concentrations but less than ZnO and ZrO_2. At 4.3 wt.% Cu loading, bacterial reductions exceeded to 94% for B.subtilis and 98% for E. coli, representing nearly complete elimination of viable bacteria. The antimicrobial activity of Cu-containing composites remained high at 8.7wt.% loading, achieving essentially complete bacterial elimination (greater than 99.3% in both of them) [1,3].

The antimicrobial mechanism of Cu nanoparticles involves multiple pathways which obstruct the cell membranes by discharge the of Cu^{2+} ions and interfere with essential enzymatic functions, and direct physical interaction with bacterial cells leading to membrane disruption [3]. The concentration-dependent antimicrobial activity reflects the increased availability of Cu^{2+} ions and ROS at higher nanoparticle loadings.

ZnO nanoparticles demonstrated moderate antimicrobial activity, with bacterial reductions recorded for 81% B.subtilis and 83.3% (E. coli) at 2.2wt.% loading. At 4.3 wt.% loading, the bacterial reduction was higher in E. coli (99%) than B.subtilis (97%). At 8.7 wt.%, significant bacterial reductions recorded in B. subtilis and E.coli, were recorded 98.7% and 98.3%, respectively. The antibacterial activity of ZnO is attributed to the generation of ROS (particularly hydrogen peroxide, hydroxyl radicals, and superoxide anions) on the nanoparticle surface, which cause oxidative stress and cellular damage

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[28,29]. Additionally, the release of Zn^{2+} ions contributes to antimicrobial activity, though Zn^{2+} is less potent than Cu^{2+} [30,31].

ZrO_2 nanoparticles showed the strongest antibacterial effectiveness at low loading with B.subtilis bacterial reductions. Interestingly, the ZrO_2 nanoparticles showed approximately the same reductions in antimicrobial activity in both microorganisms at all loading levels. With 8.7wt.% loading, the highest reduction of antibacterial activity of the B.subtilis and E. coli were obtained approximately 99%. The antimicrobial mechanism of ZrO_2 is less well-understood but may involve the generation of ROS through photocatalytic activity when irradiated [32–34].

In summary, the highest reduction in antimicrobial activity of the nanocomposites was exhibited in all nanoparticles with the highest concentration. A little higher recorded at Cu and ZrO_2 than ZnO at high concentration. The higher antimicrobial activity of Cu compared to ZnO is consistent with the established antimicrobial potency of Cu ions relative to Zn ions[3,35,36].

CONCLUSIONS

This comprehensive study investigated the influence of Cu, ZnO, and ZrO_2 nanoparticles at concentrations of 2.2, 4.3, and 8.7 wt.% on the tensile, barrier, and antimicrobial activities of polyethylene nanocomposite samples. The sample containing ZnO nanoparticles exhibited a more uniform distribution and less agglomeration at a loading of all concentrations compared with the samples containing Cu and ZrO_2 nanoparticles. The Cu and ZnO nanoparticle types showed that the mechanical properties of the low-density polyethylene materials at the medium loading (4.3 wt.%) improved in tensile strength of 16.4 and 32.4% as well as elongation at break 22 and 8.9%, respectively. Cu nanoparticles demonstrated the most balanced and highest overall performance enhancement in barrier properties. The antimicrobial efficacy of the three nanoparticles was most pronounced at the highest concentration against both gram-negative and gram-positive bacterial strains. Cu nanoparticles are recommended for applications requiring high antimicrobial efficacy. ZnO nanoparticles provided moderate improvements across all properties except in tensile strength, with the advantage of FDA GRAS status and biocompatibility. ZnO is recommended for food contact and biomedical applications where biocompatibility is critical. ZrO_2 nanoparticles exhibited the lowest overall performance, but high antimicrobial activity and a tendency to agglomerate at high concentrations. Future work should explore the effects of nanoparticle surface functionalization, processing conditions, and long-term stability on the performance of these nanocomposites.

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