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The Influence Of Zinc Peroxide On The Properties Of Polyethylene Nanocomposite Materials

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Enhancing of the properties of low density polyethylene (LDPE) by the addition of inorganic fillerin addition to a novel method to synthesis zinc peroxide (ZnO_2) nanoparticles are the main objectives of this study.

Inorganic nanocomposites were prepared by cast solutionof LDPE with different amounts of ZnO_2 nanoparticles: 0%, 1%, 1.5%, 3%, and 5% by weight. Thermal, chemical, morphological, and tensile properties were investigated, respectively, using differential scanning calorimetry (DSC), Fourier-transform infrared spectroscopy (FTIR), X-Ray Diffraction (XRD), scanning electron microscopy (SEM), and a universal testing machine. The characteristics of the antibiotics were also evaluated using an antibiotic sensitivity test.

ZnO_2 nanoparticles improve melting and cooling points as well as the crystallization degree. These results are consistent with the assumption that inorganic additives act as nucleating agents. Additionally, the SEM images demonstrated good filler dispersion within the polyethylene matrix.Tensile testing showed that the incorporation of ZnO_2improves the elastic modulus, tensile strength and fracture strength while elongation at breakwas decreases. The antibacterial properties study showed poor activity of LDPE/ZnO_2nanocomposites against all types of aerobic and anaerobic of bacteria studied.

Highly pure crystalline cubic-ZnO_2 nanoparticles grown in a near- spherical shape were obtained with a novel method with average size of 82 nm based on SEM and XRD analysis and a decomposition temperature about 238^oC found by DSC.

Keywords: *nanocomposites, low density polyethylene, nucleating agents, zinc peroxide,antibiotic*

1. Introduction

One of the most useful polymers is polyethylene (PE), which can be employed in flexible, semirigid, and rigid forms. Interms ofworldwide plastic production, it is ahead of polyolefin. This is as a result of its lower price, wider availability, excellent mechanical properties characteristics, and variety of properties. However, a number of limitations prevent it from being used widely in its pure form: particularly low rigidity, low fire resistance, low heat resistance, and low strength. Inorganic fillers are added to the matrix to overcome these issues and provide material with better characteristics [1]. Fillers may have an impact on the polymer's dimensional stability, crystallinity, mechanical, and thermal properties [2].

One of the most families of nanoparticles that have been found and investigated are metal oxides such as ZnO, MgO and ZnO_2, which have special properties such being antibacterial [3], antifungal [4] wound healing [5], and low toxicity [6]. One of the fillers that has drawn scientists' attention for a number of years is zinc peroxide (ZnO_2). It is primarily employed in various industrial applications as an accelerator for the vulcanization of rubber [7]

and the processing of plastic [8]. It can be utilized in pyrotechnic and explosive compositions as an oxidizing agent [9], Zinc peroxide nanoparticles have recently been demonstrated in numerous studies to exhibit antibacterial capabilities against various microorganisms [10].

Recently, several studies on the synthesis of zinc peroxide nanoparticles such as chemical method [11], sol-gel [12], co-precipitation [4], surfactant free [10] and hydrothermal method [13] were developed.However, all of these studies prepared and synthesized of crystal cubic zinc peroxide with nano size used various way to get the zinc peroxide nanoparticles with variation in nanosized range. Numerous factors, including reaction temperature, reactant concentration, reaction duration, and capping agent use, all affect the size of different synthesis methods [14].

Numerous other studies have concentrated on composite and nanocomposites of zinc oxide reinforcement due to the biocompatibility and nontoxicity to human cells [15] with different polymer matrix such as polypropylene [16], polyethylene (PP) [17], polyvinyl chloride (PVC) [18], poly (ethylene terephthalate) (PET) [19] with different methodologies to combine reinforcement within polymer to study the mechanical, thermal, morphological, and the antibacterial properties.

There is limited information of ZnO_2 NPs' antibacterial properties in polymer composites materials. In a related investigation, nanosized ZnOpowders were used to create composites of polyolefin matrices, and the effectiveness of the composites' antibacterial properties against Staphylococcus aureus and Escherichia coli was evaluated. The composite exhibits very strong polyolefin/ZnO composites activity against tested microorganisms [20]. The impact of ZnO nanoparticles on polypropylene (PP) was observed in another study, and antibacterial testing showed that nanocomposites had superior antibacterial properties to neat PP [16]. Additionally, the antibacterial activity of high-density polyethylene (HDPE) composites containing ZnO nanoparticles was examined. HDPE films doped with modified ZnO nanoparticles had good antibacterial activity, particularly against Staphylococcus aureus [21].In addition, the antibacterial properties of the nanocomposite were examined in relation to the ZnO concentration in the PP matrix, and antimicrobial analysis revealed that PP/ZnO nanocomposite films also showed better bacterial growth inhibition against E. coli than against Lactobacillus [22].

The goal of this research was to examine how the structure and properties of polyethylene composite materials are impacted by dispersed ZnO_2 nanoparticles in different compositions. Morphological features, thermal properties, mechanical properties, and antibacterial effects were examined. To the best of our knowledge, this work is the first investigation using LDPE and $ZnO_2$to prepare composites.

2. Experimental

2.1 Chemicals and materials

Low density polyethylene (Ipethene 670) with a melt flow index of 7.5 g/10 min (190C, 2.16 kg, according to ISO 1133) was purchased from Carmel Olefins Ltd. and was used as the raw material. Zinc peroxide (50-60%) with a decomposition temperature of 212°C and a density of 1.57g/ml, Xylene (98.5%,), Zinc acetate dihydrate (99-102 %), Hydrogen peroxide (30%) were purchased from Aldrich Sigma and were all used as received.

The American Type Culture Collection (ATCC) supplied the MRSA Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa, gram-positive streptococcus, and gram-negative bacilli.

2.2 Methods

2.2.aSynthesis ofZnO_2nanoparticles

A reflux reaction process was used to synthesis zinc peroxide nanoparticles. 5 mL of 30% hydrogen peroxide (H_2O_2) aqueous solution was diluted with 50 mL of deionized water, and the mixture was magnetically stirred for 5 min. After that, 1 g of zinc acetate dihydrate (99.9%) was dissolved in the H_2O_2 solution, and stirring continued for an additional 5 min [23].The mixture then refluxed for 24 hours at 150°C in a rounded bottom flask in a heat mental device, producing a white precipitate. After centrifuging this precipitate at 4900 rpm for 10 minutes, it was repeatedly washed with distilled water. To create dry, fine powder, the precipitate was baked at 60^0C.

2.2.bComposite preparation

Different compositions of LDPE/ZnO_2 and LDPE/nano ZnO_2 composites were prepared by solution cast technique [24] with ZnO_2 concentration of (1 %, 3 % and 5%) for composite and (0.5%, 1%, 1.5%, 3% and 5 %) for nanocomposite as shown in Table 1.

Table 1:Composition of materials used

Sample code	LDPE (wt/wt)%	ZnO_2 (wt/wt)%	ZnO_2 NPs (wt/wt)%
PE	100.0	0.0	--
PEC1	99.0	1.0	--
PEC3	97.0	3.0	--
PEC5	95.0	5.0	--
PEN0.5	99.5	--	0.5
PEN1	99.0	--	1.0
PEN1.5	98.5	--	1.5
PEN3	97.0	--	3.0
PEN5	95.0	--	5.0

In a 500 ml round-bottom flask, LDPE pellets and xylene solvent were added. For around two hours, the mixture was refluxed at 100°C. Once the reflux process was finished, a hot, transparent, viscous solution was obtained. Zinc peroxide powder was then added to the solution and continuously stirred for an hour at the same temperature to disperse it.Immediately after heating, the combined solution was cast with magnetic stirring at room temperature to immobilize the zinc peroxide fillers. The solidified organic phase system was then evaporated over the course of 48 hours at room temperature in the fume hood to evaporated xylene.

In a hot press operating at 140°C for five minutes under 30 MPa, the mixed material was compression molded into thin films with a thickness of 1.60 mm, which were then removed and allowed to cool to room temperature. Both LDPE and zinc peroxide were dried in a vacuum oven for 4 hours at 80°C before extrusion and/or compression molding to prevent hydrolytic degradation during processing.

2.3Characterization

The thermal properties of PE/ZnO_2 nanocomposites were examined using differential scanning calorimetry (Perkin Elmer Pyris 1 DSC outfitted with Intercooler-IIP) in the temperature range of 25–140°C at a heating rate of 10°C/min under a nitrogen flux of 50 ml/min. Both the crystallization temperature (T_c) and the melting temperature (T_m) were determined. The degree of crystallinity (X_c) of the films was calculated from the enthalpies of the relevant thermal transitions using the following formula:

$$X_c = \frac{\Delta H_m}{(1-\emptyset)\Delta H_f^0} * 100\%$$

where ϕis the weight proportion of additives in the composites, ΔH_m is the enthalpy of final melting, and ΔH_f^o = 288.0 J/g is the heat of melting of a fully crystalline LDPE [25].

The morphology and dispersion of ZnO_2 particles were investigated using a HRSEM (Sirion 200, FEI, USA) scanning electron microscope (SEM). The composites' surfaces were coated with gold/palladium (Au-Pd) using a vacuum sputter chamber prior to scanning.

The FTIR Spectrometer Tensor II from Bruker was used to study the binding vibrations of ZnO_2 nanoparticles. The range of wave numbers used to determine the nanoparticle's spectra was 500 cm^{-1} to 4000 cm^{-1}. Typically, a powder sample weighing around 100 mg was used.

According to ASTM D638, a universal testing machine (Instron 4502, England) at a crosshead speed of 10 mm/min was used to measure the tensile strength and modulus of the composites.The tensile properties of the rectangular press-molded tensile specimens with dimensions of 60 mm length, 10 mm width, and 0.15 mm thickness were investigated.

According to ISO 22196: 2007 [26], the antibacterial activity of LDPE/ZnO_2 composites was evaluated. Samples were placed on the bacteria growth medium during the disc-diffusion test against various microorganisms. The plate was then placed in an oven and left for 24 hours at 37°C [27].The diameter of the plate was then measured using a precise ruler, and the rate of growth surrounding the samples, which is known as the zone of inhibition, was determined.

3. Results and discussion

3.1 Production of ZnO_2 nanoparticles

The XRD pattern of the produced ZnO_2 nanoparticles is shown in **figure 1**. Crystallites of cubic-ZnO_2 with space group Pa3- (no. 205) made up the diffraction peaks of ZnO_2 nanoparticles. Strong reflections with indices of 111, 200, 220, and 311 are visible in the XRD pattern at 2Θ = 31.0°, 36.5°, 53.0°, and 63.0° [10]. No impurity peaks were found, indicating a clean single-phase structure that matched the typical cubic ZnO_2 crystal structure, indicating the creation of high-quality ZnO_2 nanoparticles.

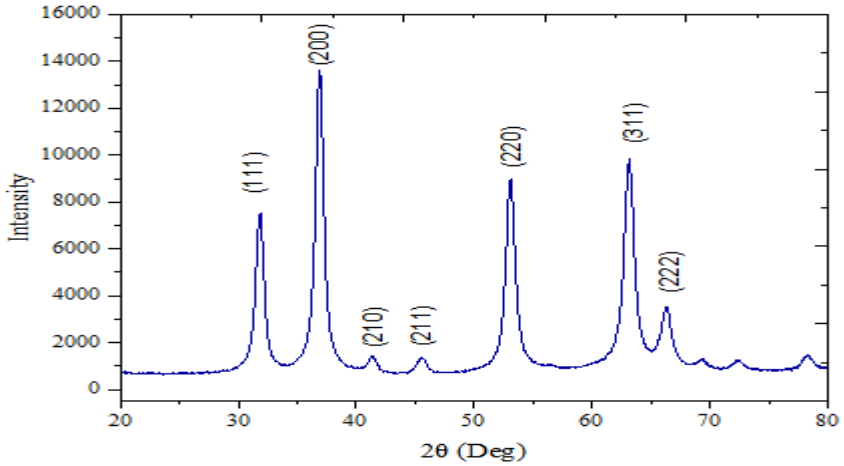

Figure (1): XRD pattern for zinc peroxide nanoparticles

For synthetic zinc peroxide nanoparticles, **figure 2** shows the FTIR spectrum. The picture displays two peaks, one at 1584 cm^{-1} and the other at 3404 cm^{-1}. Both of these can be attributable to the distinctive hydroxyl group absorption (O-H), which corresponds to stretches of the water molecule [13].

The vibrational modes of the O-O bands corresponding to the peroxide (O_2) ions in ZnO_2 nanoparticles might be attributed to the band at 1421 cm^{-1}. Around 650 cm^{-1}, a second distinct absorption band of synthetic ZnO_2 is seen. This band corresponds to Zn-O vibration [10].The absence of any stretching mode absorption bands at 2924 and 2853 cm^{-1} for the C-H bonds or -COO groups of zinc acetate in the FT-IR spectra suggests the purity synthetic zinc peroxide nanoparticles from the reactants [28].

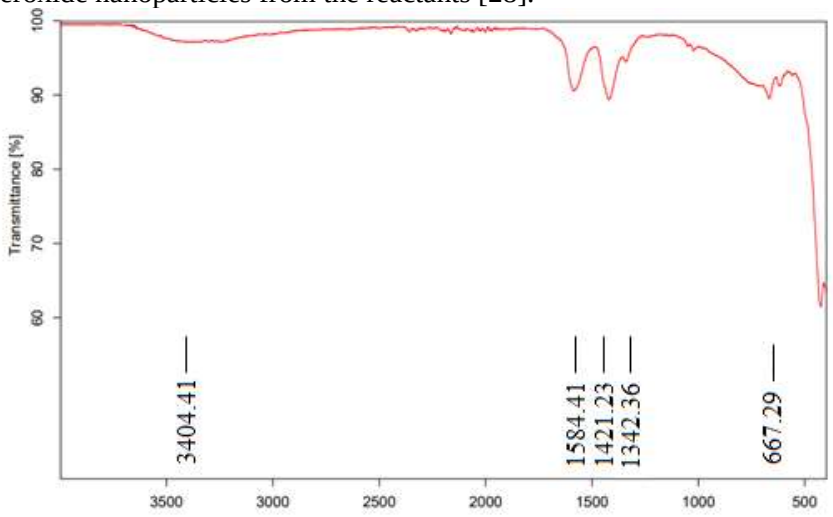

Figure (2): The infrared spectrum of the zinc peroxide nanoparticles.

The scanning electron microscope (SEM) was used to determine the morphology and size of the zinc peroxide nanoparticles as shown in **figure 3**. It is shown that the nanoparticles have grown in a near- spherical shape nanoparticles with a range of nanometer diameter (80 nm) which demonstrates the good quality of the ZnO_2-NPs. The SEM image reveals that the observed particles are smaller crystals and they are well dispersed in the powder form with small number of nanoaggregates.

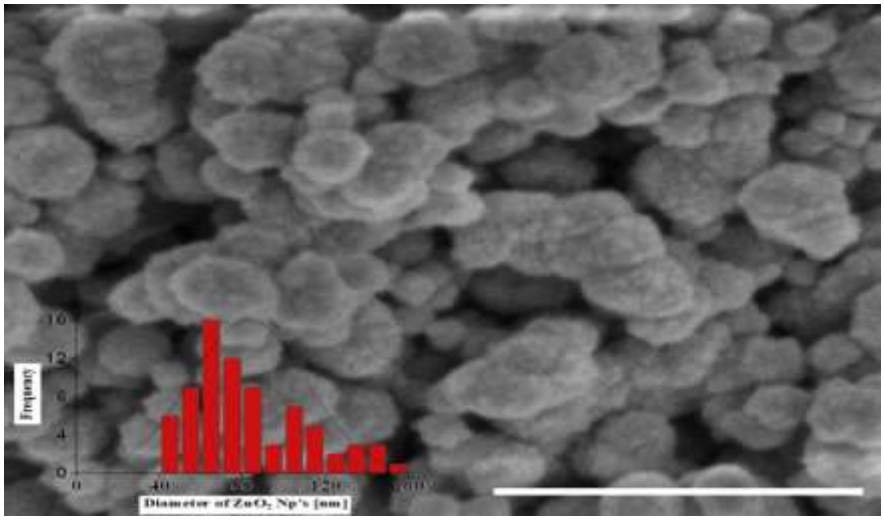

Figure (3): Scanning electron microscope images of zinc peroxide nanoparticles (scale bar 500 nm).

3.2 Dispersion of ZnO_2 particles

Figure 4 shows the micrographs of surfaces of the composite samples. Images taken on the virgin LDPE(**Fig. 4a**) appear smooth and clear surface area, meanwhile composite sample show some white spots like cotton in the surface of composite (**Fig. 4d**) indicate to zinc peroxide particles. Similar phenomena can be referred to the literature [29]. As such, ZnO_2 particles were dispersed and embedded in the matrices of the composite.

ZnO_2 particles were found to have good dispersion; they had a uniform distribution throughout the entire polymer matrix and weren't locally agglomerated. In general, better dispersibility was found in the composites with a lower amount of ZnO_2 particles.

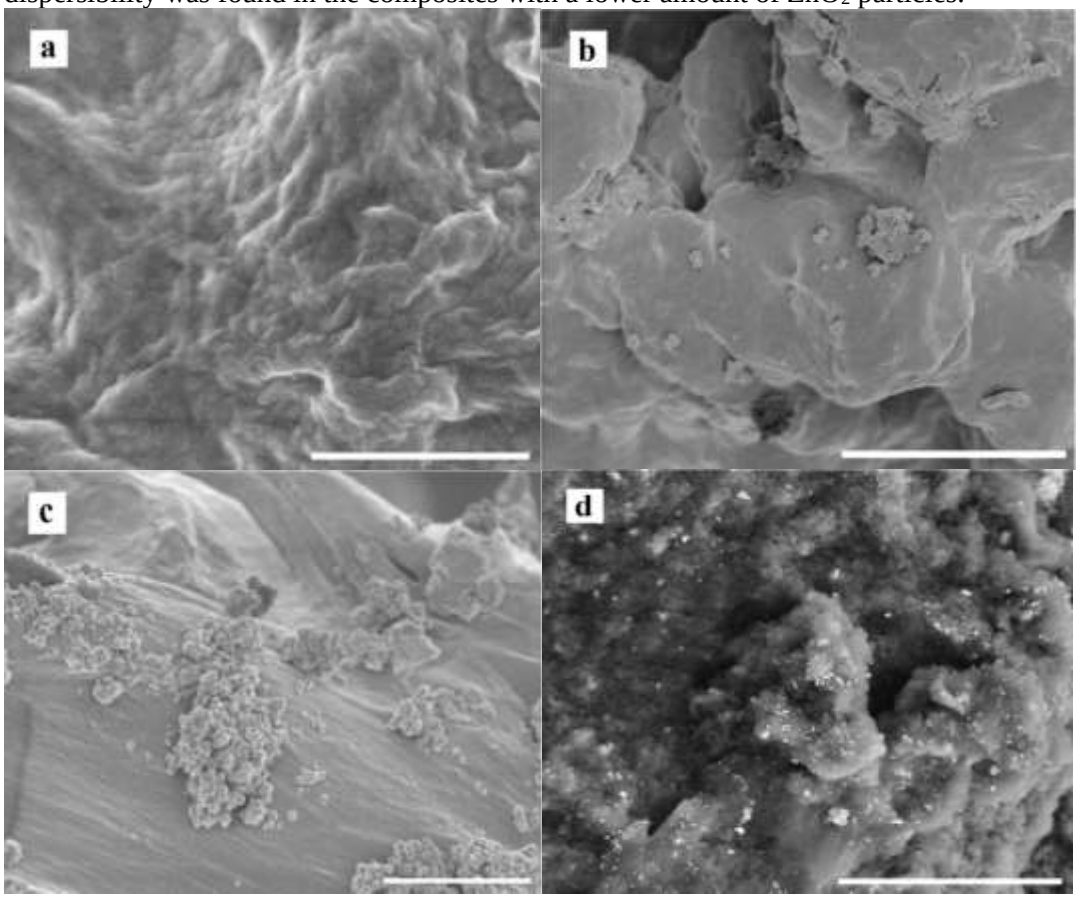

Figure4: SEM micrograph of (a) 0%PE (b) 1%PE (c) 3%PE (d) 5%PE.

3.3 Thermal properties

Table 2, **figure 5, and figure 6** present the DSC results for LDPE/ZnO_2 composites and nanocomposites. It is possible to see that there is a slight change in melting temperatures and the addition of the ZnO_2 presents an increase in the crystallinity degree especially in LDPE/ZnO_2nanocomposites as seen in **table 2**. This behavior can be related to the intercalated/exfoliated state observed in the SEM image (Figure 4).The highest increase was in the PEN3 sample. In the literature, some authors [30] reported that the crystallinity of PE increases with an addition of inorganic fillers, while others observed no changes [31]. The third group of authors says that the addition of inorganic fillers decreases the crystallinity of LDPE [32].

We noticed that the reinforced of LDPE with ZnO_2 particles increased the crystallinity of polyethylene. This was due to the ability of these particles to act as nucleating agents for crystallization on PE, especially when a small amount of inorganic filler was used (max 5%). Moreover, the small particles were well distributed, so the overall influence can be considered to enhance the nucleation effect and increase the crystallinity of the matrix.

This phenomenon can be declared also by T_c values where the initial crystallization temperature of all composite samples is higher than that of pure PE. This is because the addition of ZnO_2 particles especially nanoparticles to PE matrix increases their crystallization

temperature, inducing earlier PE crystallization and enhancing its crystallization abilitywhere similar results have also been reported in the study of Wang et al [33].

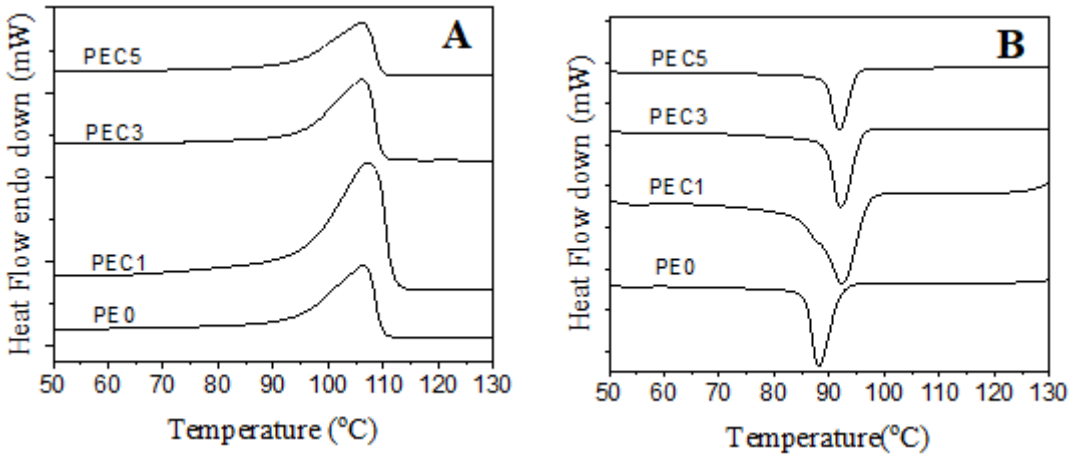

Figure5: First heating and cooling scans of LDPE/ZnO_2 composites

Table 2: DSC characterization of composite samples, calculated from the first heating scans.

Material	T_m (°C)	T_c (°C)	X_c (%)
PE	106	88	20.2
PEC1	107	92	20.2
PEC3	106	92	23.3
PEC5	106	92	19.2
PEN0.5	107	91	21.6
PEN1	106	92	21.8
PEN1.5	106	92	23.2
PEN3	106	92	23.3
PEN5	106	92	20.6

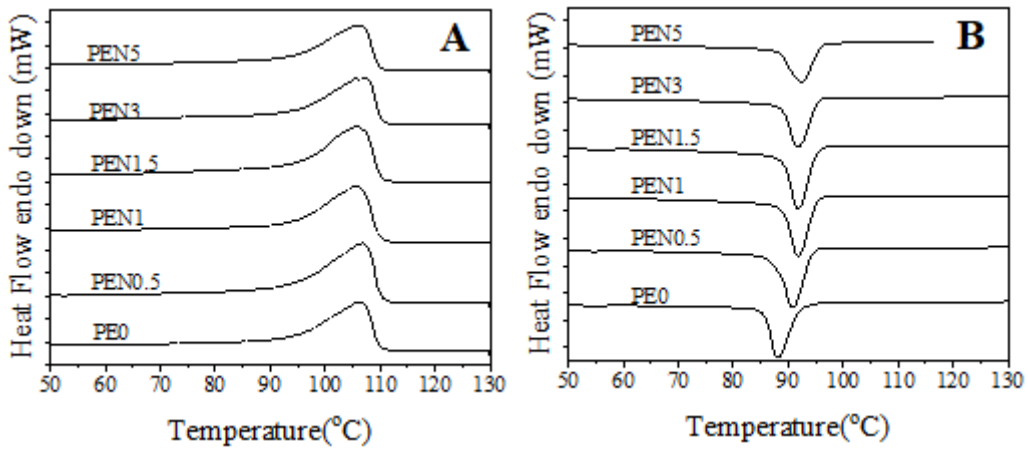

Figure6: First heating and cooling scans of LDPE/ZnO_2nanocomposites

The nanoparticles may act as nucleating agents and accelerate the formation of crystals, decrease the dimensions of spherulites, and increase crystallinity to improve the nanocomposite's thermal properties, as shown by **figure 7b**, which showed that for the nanocomposite, the degree of crystallinity increased roughly as zinc peroxide nanoparticle concentration increased.Many investigations had previously suggested that nanofiller may act as a nucleating agent [20].

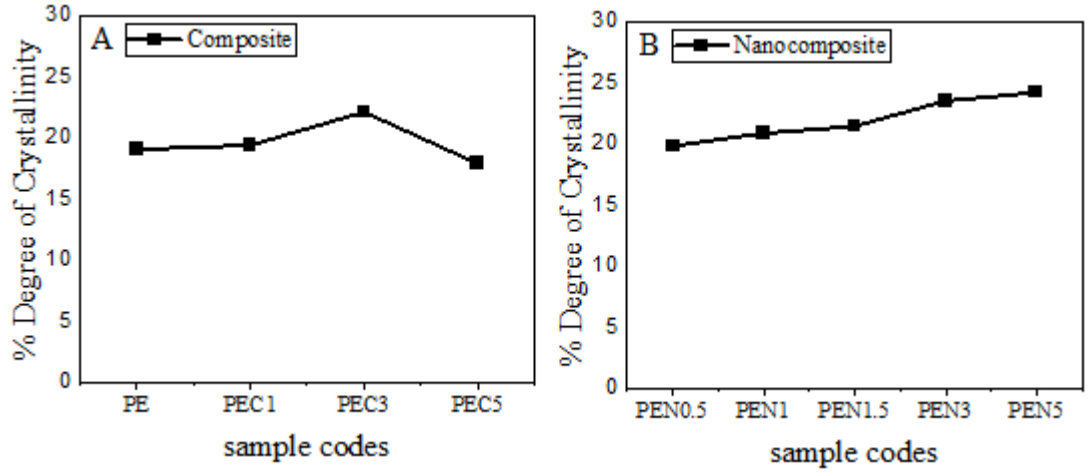

Figure 7: % Degree of crystallinity trend for: (a) Composite, (b) Nanocomposite.

3.4 Tensile properties

All PE/ZnO_2composites, in general, present an enhancement in tensile properties, as can be seen in **Table 3**. It is apparent that, in the presence ofinorganic additive, Young's modulus (E) of PE composites was slightly greater than that of 0%PE throughout all the content ranges within an acceptable standard deviation, which is the typical characteristic for inorganic filler composites [34].

Table 3. Tensile properties of PE/ZnO_2 composites

Sample code	Tensile strength (MPa)	E modulus (MPa)	Fracture strength (MPa)	% Elongation at fracture
PE	4.94 ± 0.38	103.0 ± 5.0	3.19 ±0.64	39.0 ±3.0
PEC1	4.76 ± 0.80	110.0 ± 9.0	3.74 ±0.25	36.0 ±4.0
PEC3	4.53 ± 0.46	115.0 ±12.0	3.81 ±0.40	33.0 ±2.0

PEC5	4.35 ± 0.56	117.0 ± 10.0	3.39 ±0.21	29.0 ±2.0
PEN0.5	4.98 ± 0.79	109.0 ± 6.0	4.28 ±0.33	48.0 ±3.0
PEN1	5.01 ±0.71	112.0 ± 7.0	4.41 ±0.23	42.0 ±3.0
PEN1.5	5.16 ±0.93	114.0 ± 6.0	4.40 ±0.36	42.0 ±2.0
PEN3	5.22 ±0.28	121.0 ±8.0	4.73 ±0.32	41.0 ±2.0
PEN5	5.28 ±0.73	124.0 ±10.0	4.90 ±0.54	42.0 ±2.0

The addition of 1%ZnO_2NPs increasedYoung's modulus by 1.5% compared with the 0%PE sample, while the addition of 3%ZnO_2 increased Young's modulus by 4%. The presence of 5% ZnO_2 resulted in a 5% increase in the maximum E for LDPE nanocomposites. This may be caused by both the stiffness of ZnO_2 nanoparticles and a rise in LDPE crystallinity brought on by ZnO_2, as seen in **Table 2**.

Also, it was found that the tensile strength values remained the same with the incorporation of micro inorganic additives while slightly increase with the addition of nanofillers as expected. This behavior can be interpreted as a mechanism where stress is transferred from the matrix to the dispersed rigid phase, which is associated with the intercalated/exfoliated state seen in the SEM image (**Figure 4**) [35].

The results revealed that the fracture strength of nanocomposites increases as the concentration of zinc peroxide increases, this could be attributed to the fact that the zinc peroxide nanoparticles impede the formation of porosity within nanocomposite and enhanced fracture properties[36].

It is obvious from the data that elongation decreased steadily with zinc peroxide concentration increased specially for composite samples. This is attributed to the brittle nature imparted by the zinc peroxide filler in the composites and nanocomposite. The improvement in elongation at break is better in the nanocomposite than in the composite. Due to their higher ductility, nanocomposite materials are more likely to deform under tensile loads rather than breaking, as opposed to brittle materials that will fracture before much deformation occurs [37].

3.5 Antibacterial properties

The antimicrobial activity of different concentrations of composites were investigated by disc diffusion method against aerobic bacteria such as Staphylococcus aureus (MRSA), Escherichia coli (E. coli) and Pseudomonas aeruginosa. Also, the same test was done against anerobic bacteria such as anerobic gram-positive streptococcus and anaerobic gram-negative bacilli.

The results, as shown in **figure 8**, indicate that all nanocomposites had poor antibacterial activity against all species of bacteria examined.

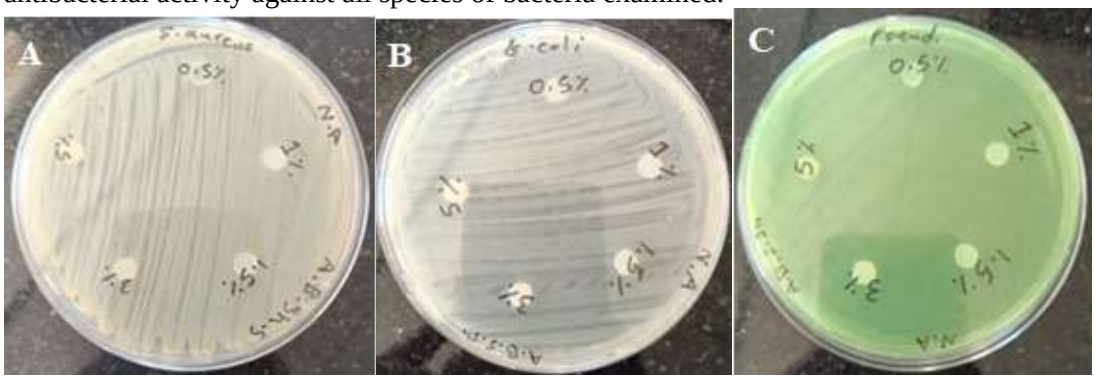

Figure 8: Anti-microbial activity of PE/ZnO_2nanocomposite of different concentrationsagainst A: Staphylococcus aureus, B: Escherichia coli and C: pseudomonas aeruginosa.

In LDPE/ZnO_2, the ion release is the main mechanism behind their bacterial activity but due to the particle's incorporation into the polymer and its low concentration on the surface as confirmed by the SEM analysis so the polymer interacts more with bacteria compared antibacterial agent [38].

4. Conclusion

Thermal, mechanical, tensile, and antibiotic properties of ZnO_2/LDPEnanocomposites were investigated. SEM revealed a good dispersion of inorganic filler inthe polyolefin matrices.

The thermal results of nanocomposite showed there are slight variations between the melting temperature of different concentration of filler while imparts significant improvements in crystallization temperature and the degree of crystallinity which means thatthe crystallization rate of nanocomposite have become fasterdue the filler act as nucleating agent**.**

The addition of ZnO_2 to the LDPE caused an increase in theYoung's modulus and tensile strength of the nanocomposites considering the values obtained by the pure polymer. This trend canbe attributed to the higher crystallinity of the PE matrix and to the adhesion between filler and matrix. ZnO_2/PEnanocomposites exhibited a poor antibacterial activity and demonstrated no effect against all types of bacteria used especially at low concentrations.

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