

Synthesis and Characterization of the PP Fibers Coated by ZnO Nanoparticles: Investigation of Antibacterial Properties

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Abstract: This research is focused on the synthesis of zinc oxide nanoparticles by means of the sol-gel conventional method, followed by the formulation of a zinc oxide-polypropylene (ZnO:PP) masterbatch using a twin-screw extruder. The next step involved the fabrication of antibacterial fibers through electrospinning. The resulting Nano powders, masterbatch, and fibers were subjected to a series of characterization techniques, including X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDXS), and Fourier-transform infrared spectroscopy (FTIR).

Thermogravimetric analysis (TGA) indicated a three-stage weight loss process, and differential scanning calorimetry (DSC) showed a primary endothermic peak centered at about 317°C and a pronounced exothermic peak at approximately 455°C. XRD confirmed the hexagonal wurtzite structure of the ZnO nanoparticles and the presence of the alpha (α) crystal form in polypropylene. In the FTIR spectrum of ZnO:PP, the absorption bands showing PP and ZnO were evident. FESEM images revealed that the ZnO and masterbatch samples had a uniform size and shape, predominantly in the nanometer range, with an elongated spherical morphology. The presence of peaks for Zn and O elements, indicative of ZnO, and the C element characteristic of PP, further confirms the fabrication of ZnO:PP composite. The antibacterial properties of polypropylene fibers containing varying concentrations of ZnO, including 1.2, 2.4, and 5 wt.%, were tested against *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*). The results showed that the samples with 1.2 and 2.4 wt% ZnO reach higher antibacterial activity against *E. coli* and *S. aureus* compared to other samples.

Keywords: Polypropylene (PP), Zinc oxide (ZnO), Composite, Antibacterial properties.

1. INTRODUCTION

Polymers possess various favorable properties; these include being lightweight, easy to manufacture, low-cost and high mechanical strength. For these reasons, they are highly favorable materials in many industries and daily applications [1-3]. Among the various polymers available, polypropylene (PP) stands out as one of the most frequently used thermoplastic materials. This organic polymer is characterized by its lack of color, odor, and toxicity. It boasts an optimal temperature during processing, excellent insulation against electricity, extraordinary tensile strength, and negligible absorption of moisture [4-7].

Given its versatile attributes, polypropylene finds application across a wide array of industries, including machinery, automotive, electronics, textiles, packaging, agriculture, forestry, fishing, and the food sector [6]. Its relatively low melting point (160 to 175°C) makes it especially acceptable for fibers and nonwoven fabric production [4]. More specifically, polypropylene is instrumental in materials using spun bond and melt-blown

fabrics, especially in sanitary and medical uses [8]. Polymer materials suffer from degradation, a widespread problem that can produce modifications in their chemical, physical and mechanical properties. Several factors, including exposure to sunlight, elevated temperatures, various chemicals, mechanical stress, moisture absorption, and biological agents, can cause the degradation of polymer materials. Ultraviolet (UV) radiation is a dominant factor in the photodegradation of polymers used in outdoor environments, leading to short working life and imposing limitations on polypropylene (PP) fibres [9-10]. Additionally, polypropylene and its copolymers are often utilized in applications such as food packaging and water transfer systems where antibacterial properties are required to mitigate bacterial contamination [11]. Research continues to assess the mechanisms of photo-oxidative degradation and enhance the antibacterial properties of PP within the scope of stability. As some solutions, the incorporation of inorganic nanoparticles into organic polymer matrices is being researched. Using organic-inorganic nanocomposites has

proved to be an effective means to improve conventional polymers' performance. Improved properties include physical and mechanical properties, thermal resistance, flame resistance, reduced vapor transmittance, and resistance to chemicals [12-15]. These enhancements can be achieved through the inclusion of negligible amounts of nanofillers, which is their bonus over their counterparts having micro sizes. To upgrade the resistance to UV radiation and diminish the impact of the photo-degradation phenomenon in PP, UV absorbers can be employed. These substances are typically categorized into two major groups, consisting of organic and inorganic [15, 16]. Inorganic antibacterial particles having nano to micro sizes possess various benefits in comparison with their organic counterparts, including extraordinary thermal stability, excellent chemical resistance, notable safety, and greater effectiveness. The properties of the prepared composite materials are significantly tied to several parameters related to inorganic additives, which are their dimensions and shapes, the type of those materials used in the structure of the composite, their surface properties, and the quality of their distribution within the matrix of the polymer. The small size, in other words, the large surface area of nanofillers can provide polymers with strong interactions, which give rise to considerable improvements in all their properties [17].

A growing fascination has recently emerged with the use of inorganic materials, particularly metals and their oxides, due to their remarkable durability in challenging processing environments. Some notable candidates among metal oxides are titanium dioxide (TiO_2), zinc oxide (ZnO), magnesium oxide (MgO), and calcium oxide (CaO), which are highly well-known for their robustness in harsh circumstances and also their safety for humans and other creatures [18].

ZnO nanoparticles, as one of the main kinds of metal oxides, are extensively used in various applications from the fabrication of functional devices to the preparation of catalysis, pigments, and cosmetics. In addition, because of their intrinsic oxygen deficiencies, ZnO nanostructures play a critical role in breaking down the complicated organic materials during their exposure to UV light, a process defined as photocatalysis [18]. Numerous studies have demonstrated that ZnO and its similar nanoparticles can be efficiently incorporated into the backbone of various polymer matrices,

such as PP, polyamide (PA), and polyethylene terephthalate (PET) [19]. Despite its numerous benefits, incorporating ZnO into PP can present several challenges. PP is inherently hydrophobic, which makes it difficult to disperse hydrophilic fillers homogeneously and, subsequently, to achieve a uniform mixture within the polymer [20].

ZnO can be obtained by means of different techniques, which enable tailoring its particle size and shape. Among the methods used for the fabrication of ZnO , flame spray pyrolysis, chemical synthesis, and solvothermal are the most effective. One of the significant challenges with ZnO particles is their tendency to agglomerate, which arises from their large surface energy. This issue can become worse when ZnO makes contact with organic solvents or polymers. The agglomeration of ZnO particles can make the fabrication of ZnO /polymer composites complicated, where the evenly spread of the nanoparticles throughout the composites is essential. To surmount this issue, researchers have inaugurated finding new routes to coat the surface of the ZnO particles with organic materials. Surface modification offers several advantages, including the control of particle size and the prevention of agglomeration. ZnO nanoparticles are highly sought after for their ability to block UV radiation, making them suitable for a variety of applications. The unique properties of ZnO have made it ideal to be adopted as a filler in numerous fields, which is what our study aims to explore [20, 21].

In this study, we aim to explore the antibacterial properties of the fabricated PP: ZnO composites and also investigate the effect of different amounts of ZnO on the antibacterial properties of the prepared composites. These PP: ZnO composites are obtained by means of the melt mixing method with a twin-screw extruder, with the aid of which we can ensure a uniform blend of the materials. To structurally probe the ZnO nanoparticles and the resulting fibers, field emission scanning electron microscopy (FE-SEM) is used, which enables us to observe their shapes meticulously and also their arrangements. We also assess the thermal behaviors of the composites using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). To probe the elemental composition of the produced samples, energy dispersive X-ray spectroscopy (EDXS) can be a powerful technique. X-ray diffraction (XRD) is another structural analysis to detect the

crystalline forms of the composites. We also investigate the functional groups that exist in the materials through Fourier transform infrared spectroscopy (FTIR). Ultimately, to evaluate the antibacterial activity of the composites, we conducted tests against *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*) bacteria.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

The chemicals adopted for conducting this study were purchased from Merck. They were of laboratory grade and used without further purification.

2.2. Preparation of ZnO Nanoparticles

To prepare ZnO, zinc acetate (30 g) was dissolved in deionized water (100 ml) under incessant stirring. Another solution was obtained through dissolving citric acid (30 g) in deionized water (100 ml), which was stirred for 30 minutes to ascertain the complete dissolution of citric acid. Afterwards, two prepared solutions were mixed in a molar ratio of 1:4. Then, ethylene glycol (39 g) was added to the mixture above. The resulting solution was put in an oil bath to be heated for one hour at a temperature of 90°C. After heating, the solution was transferred to an oven for another heating, which was carried out at 200°C for three hours. At the end, the solution was calcined at 700°C for two hours to produce a white ZnO nanopowder.

2.3. Preparation of ZnO:PP Masterbatch

To fabricate the ZnO:PP, the synthesized ZnO nanoparticles in the previous stage were mixed with PP at a weight ratio of 10:90. A binder was also added at a concentration of 1% by weight. This mixture was processed using a twin-screw extruder at 220°C for one hour to obtain the masterbatch of ZnO:PP.

2.4. Fabrication of Antibacterial Fiber

Various composites of ZnO:PP were synthesized through mixing PP granules with different concentrations of ZnO, including 1.2, 2.4 and 5% by weight (Table 1).

Table 1. Different composites were synthesised for the investigation of antibacterial properties

No.	Composite
1	PP (Control)
2	PP + 1.2 wt% ZnO
3	PP + 2.4 wt% ZnO
4	PP + 5 wt% ZnO

This mixture was then fed into an electrospinning apparatus to make antibacterial spunbond fibers.

3. RESULTS AND DISCUSSION

3.1. FESEM and EDX Results

The morphology, size, and distribution of particles within the polymer matrix of zinc oxide (ZnO), masterbatch, and spunbond fabric samples were analyzed using FESEM analysis. The FESEM images of the ZnO and masterbatch samples displayed in Figure 1(a) and 1(b) reveal uniformity in particle size and shape. Most particles are in the nanometer range, exhibiting an elongated, spherical morphology. The average particle size varied from 80 to 100 nm, which facilitates a homogeneous dispersion throughout the fibers [22].

Figure 1(c) presents the spunbond fabric containing 5 weight percent ZnO, showcasing a flat and smooth surface with an average fiber diameter of 20 μm . The FESEM images indicate that the PP fibers enclose part of the ZnO nanoparticles, while others are found on the surface, a detail that will be further supported by the Energy Dispersive Spectroscopy (EDS) analysis presented in the following section.

To verify the chemical composition of the PP fibers, EDS was employed. As shown in Figure 2(a), the EDS analysis of the ZnO nanoparticles confirmed the presence of both zinc (Zn) and oxygen (O). Figure 2(b) illustrates the EDS results for the masterbatch, showing the detection of carbon (C), oxygen (O), and zinc (Zn), which confirms the presence of zinc oxide on the surface of the PP fibers. Similar elemental compositions were identified in the spunbond fabric containing 5% ZnO, as presented in Figure 2(c).

3.2. XRD Spectroscopy

XRD patterns of the fabricated samples were captured to confirm the incorporation of ZnO nanoparticles within the matrix of the fiber. PP typically possesses three specified crystalline forms, consisting of α , β , and γ phases, among which the β phase exhibits enhanced thermal stability owing to its hydrogen bonds with high strength and also crystalline arrangement with more durability [23, 24]. In accordance with Figure 3(a) showing the XRD pattern of ZnO, major peaks at 2θ of 32°, 34°, 36°, 47°, 56°, 63°, and 68° are easily found. These peaks are attributed to the crystallographic planes (100), (002), (101), (102), (110), (103), and (112),

respectively. This pattern aligns with the hexagonal wurtzite structure characteristic of zinc oxide, as indicated by the JCPDS reference number 36–1451 [25–27].

Figure 3(b) displays the XRD results for polypropylene, which revealed five distinct diffraction peaks at angles of 14.0°, 16.78°, 18.4°, 21.58°, and 25.28°. These peaks relate to the crystal planes (110), (040), (130), (111), and (041), respectively, indicative of the diffraction

characteristics typical of the α crystalline form of polypropylene [28, 29]. Interestingly, the addition of ZnO nanoparticles to the polypropylene fibers resulted in a noticeable reduction in the intensity of the diffraction peaks associated with the polypropylene structure (Figure 3(c)); nevertheless, these peaks remained distinguishable. This suggests that the integration of ZnO nanoparticles into the polypropylene matrix was successfully achieved.

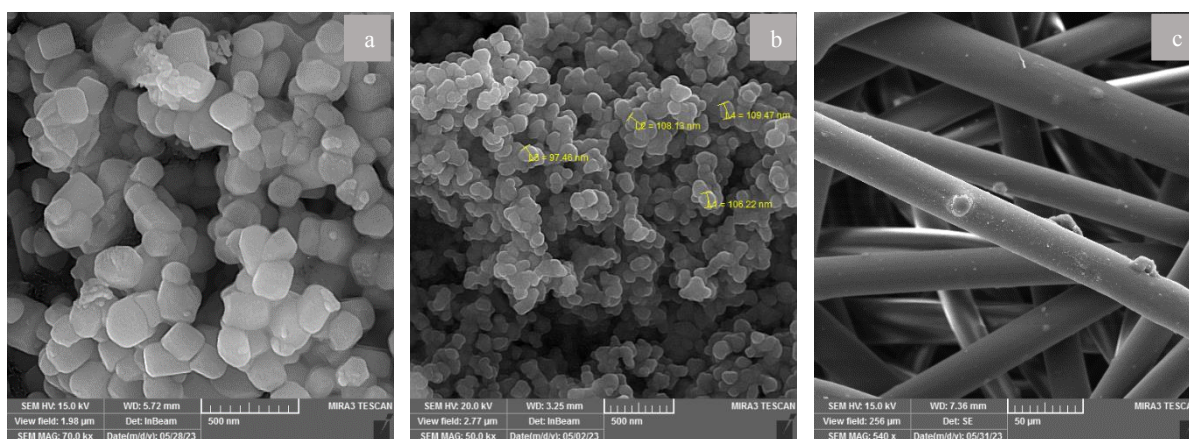


Fig. 1. FESEM Images of a) ZnO Nanoparticles, b) ZnO-Loaded Masterbatch, and c) Spunbound Fabric

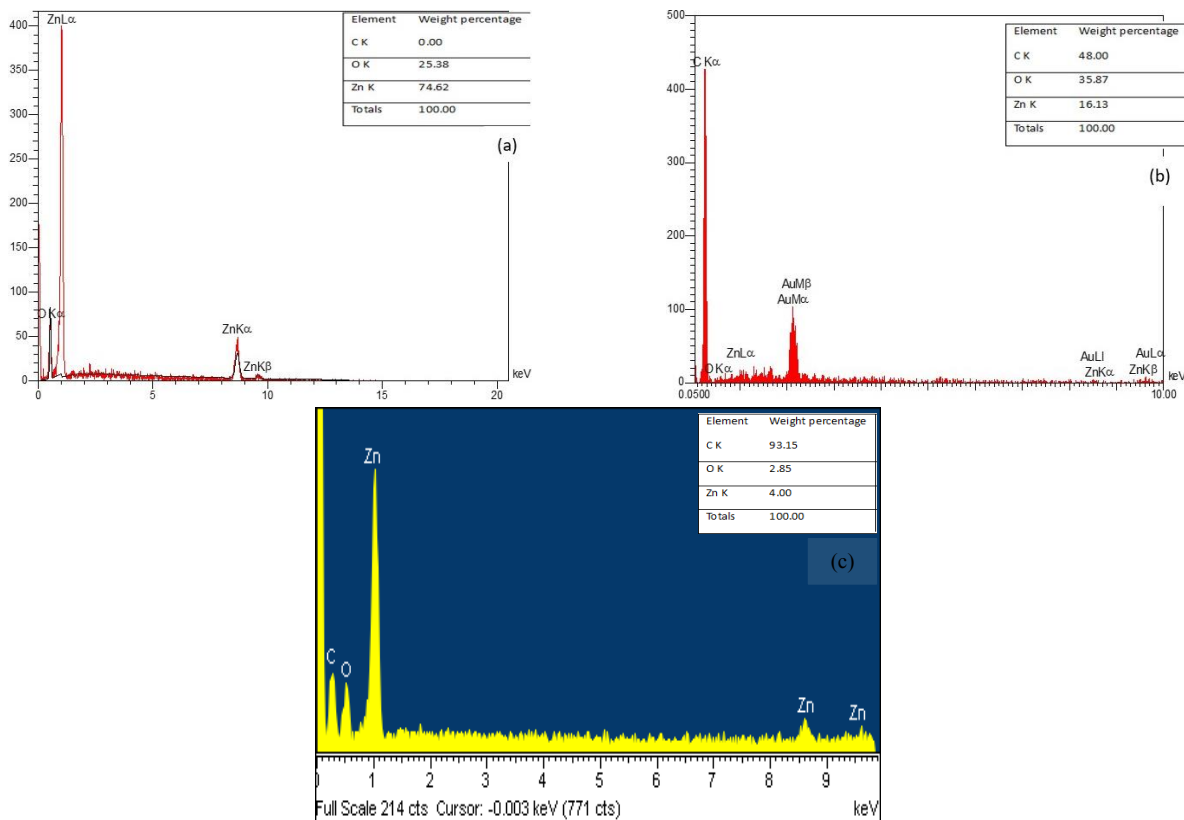


Fig. 2. EDS Elemental Analysis of a) ZnO Nanoparticles, b) ZnO-Loaded Masterbatch, and c) Spunbound Fabric, Revealing the Elemental Composition of Each Sample

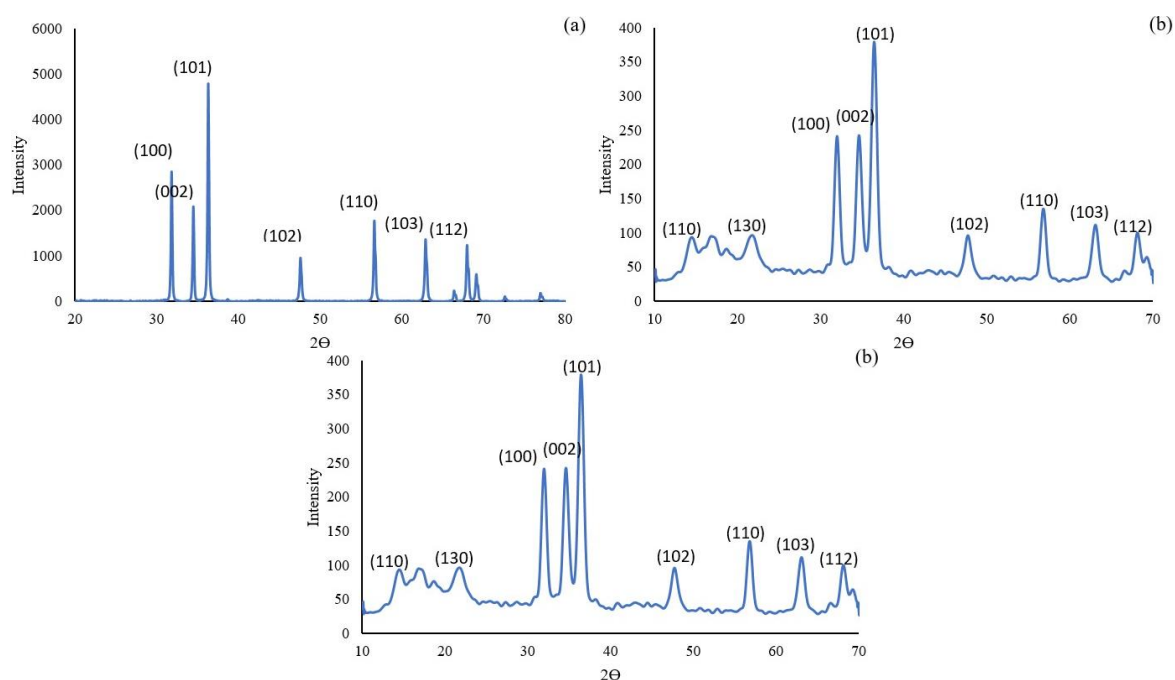


Fig. 3. XRD Spectra of a) Zinc Oxide (ZnO) Nanoparticles, b) Pristine Fabric, and c) ZnO-Loaded Masterbatch Material

3.3. FTIR Result

As mentioned earlier, the photodegradation of PP fibers results from their exposure to sunlight or UV rays. To this end, FTIR is an advantageous tool for the evaluation of the degradation mechanisms in PP. The photodegradation of PP starts from the generation of alkyl radicals, which stem from the interactions between UV radiation and the polymer. This reaction can bring about the formation of hydroperoxide, whose breakdown produces alkoxy groups [30, 31]. These alkoxy groups can either eliminate a hydrogen atom from the polymer or undergo β -cleavage, a reaction by which varied carbonyl-containing compounds such as ketones, esters, and acids are generated [30, 31]. FTIR spectroscopy is required for tracing the aforementioned chemical transformations, as it can identify different functional groups in a sample [32].

The FTIR spectra of ZnO, ZnO/PP masterbatch, and the spunbond fabric including 5 wt% ZnO are shown in Figures 4a-c. Accordingly, in the ZnO spectrum, a peak is observed at a wavenumber of 3450 cm^{-1} , which is attributed to the stretching vibrations of hydroxyl groups in water molecules [33, 34]. Besides, a peak is found at 461 cm^{-1} , which is attributed to the Zn-O bond [35]. For the masterbatch sample, the peak centered at approximately 2960.2 cm^{-1} corresponds to the

CH_3 group in polypropylene, reflecting both asymmetric and symmetric stretching vibrations. The peaks at 2921.6 cm^{-1} and 2838.7 cm^{-1} are associated with the CH_2 group in polypropylene. The pronounced peak at 1463.7 cm^{-1} is linked to the bending vibration of the CH_2 groups within the polypropylene structure, while the peak at 1377.9 cm^{-1} corresponds to the deformation vibration of the CH_3 groups. Furthermore, the absorption peak identified at 840 cm^{-1} is related to the stretching vibration of C- CH_3 bonds. Absorption peaks at 972 , 997 , and 1165 cm^{-1} are associated with the rocking vibrations of the CH_3 groups [36-39].

After the incorporation of ZnO with PP during the extrusion process, the peaks indicative of ZnO were reduced in intensity, potentially due to the high temperatures involved or an inadequate amount of ZnO used. Moreover, the FTIR spectra for all spun PP fibers demonstrated that there were no significant changes to the PP structure or its crystallinity characteristics [40], which is in good agreement with the results obtained from XRD analysis, where no changes were observed in the structure of PP as a result of the introduction of ZnO into this polymer.

3.4. TG-DSC Analyses

Figure 5 portrays the TGA curve of the prepared ZnO, which was taken using a heating rate of $10^\circ\text{C}/\text{min}$.

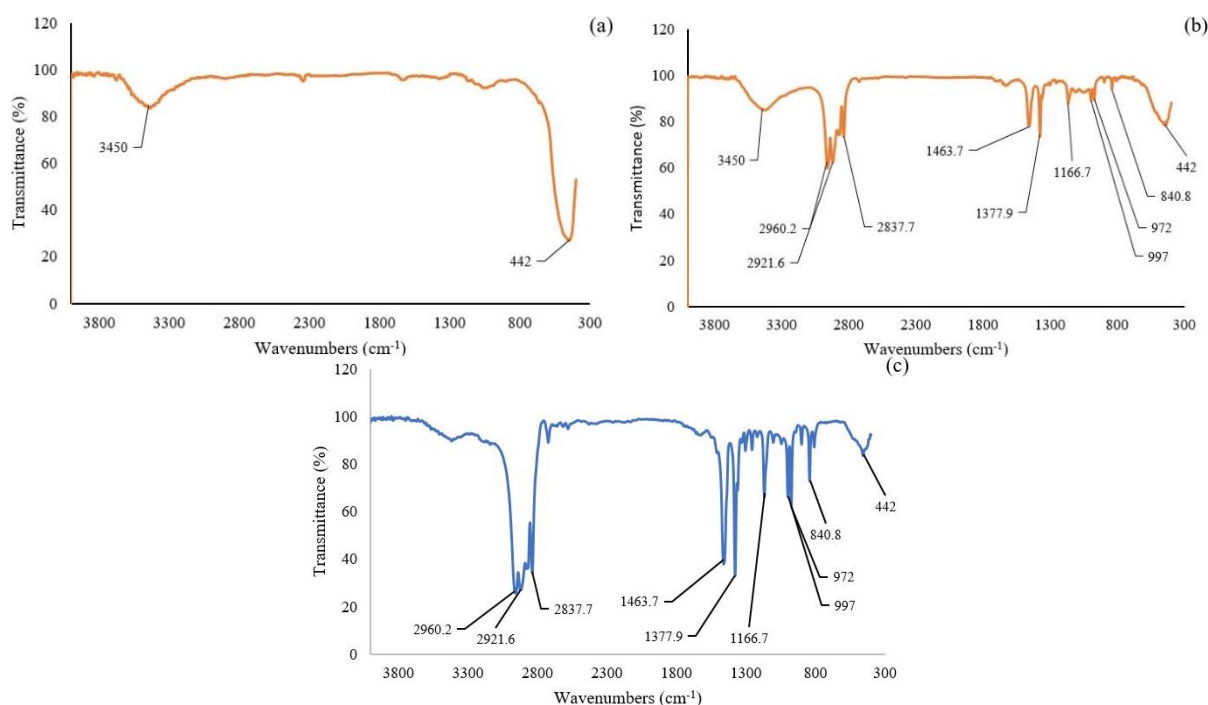


Fig. 4. FT-IR spectra of a) zinc oxide (ZnO), b) masterbatch, and c) spunbond fabric

TGA was employed to assess the thermal stability of ZnO by applying heat to this material, leading to the breakdown of molecular bonds [41, 42]. The profile displayed in the figure reveals a precise sequence of weight loss, marked by three specific temperature points around 100°C, 280°C, and 470°C, after which the weight stabilizes. The initial weight loss, about 10% of the total mass, is primarily because of the release of adsorbed water molecules from the surface of ZnO nanoparticles. The subsequent phases of weight loss—24% and 30%—are likely linked to the breakdown of chemically bound water, along with the evaporation of alcohols, organic compounds, and hydroxide groups in the structure. These successive changes in thermal behavior of ZnO nanostructures show its stability during heating [42-44].

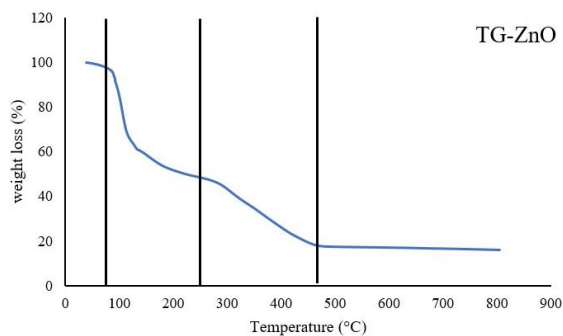


Fig. 5. Thermal Analysis Profile of ZnO Nanoparticles

The results obtained from DSC analysis of ZnO are illustrated in Figure 6. Accordingly, an exothermic peak is detected at 317.21°C, which can be assigned to the transformation of the material to zinc hydroxide. Moreover, an endothermic peak is found at 455.17°C, which is possibly pertinent to the formation of ZnO nanostructures and the destruction of any extant organic materials in the sample. These peaks are indications for substantial chemical transformations, which take place during thermal assessment, showing the complex thermal properties of the material [45, 46].

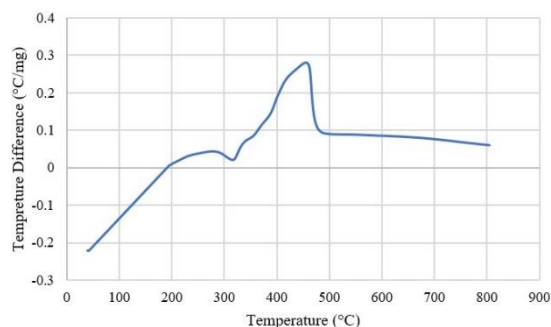


Fig. 6. DSC curve taken for the ZnO nanostructures

3.5. Antibacterial Test Results

Polymers are frequently used to produce packaging materials that adhere to strict hygiene standards. Consequently, the development of polymer composites showing antibacterial activities has

newly attracted significant attention in both scientific and industrial sectors [41, 42]. ZnO nanostructures are well-known as antimicrobial agents owing to their efficacious antibacterial activities, non-toxicity, biocompatibility, and affordability [43-45]. Currently, three primary mechanisms have been identified regarding the antibacterial activities of ZnO, one of which is the release of the antimicrobial ions. Another mechanism is the interaction between the nanoparticles and microorganisms, which compromises the integrity of bacterial cells. [46-48]. And the last is the generation of reactive oxygen species induced by light exposure, which enhances the antibacterial activities of ZnO inside the composites [46-48]. In this section, the antibacterial properties of the produced composite fibers were evaluated. Figures 7 and 8 illustrate the color changes observed in samples containing 1.2% and 2.4% ZnO, highlighting notable differences in both bacterial colonies. In contrast, the sample with 5% ZnO exhibited a color similar to that of the control samples. The results indicate that samples containing 1.2% and 2.4% ZnO displayed enhanced resistance to bacterial adhesion. Our findings are well in agreement with previous studies [49-51]. For instance, and in accordance with the research done by Zhang and colleagues

[49], polylactic acid (PLA) film containing 3% ZnO considerably reduced the number of *S. aureus* and *E. coli* bacteria by roughly 60% and 58%, respectively. In another similar study, Silvestre and his team [50] discovered that PP composites including 5% ZnO showed an 85.3% reduction in the number of *E. coli* bacteria only after 24 hours. In addition, the work carried out by Zhang [51] demonstrated that PP fibers possessing 3% ZnO manifest extraordinary antibacterial activities whose inhibition rates exceed 99% against both bacterial strains [51]. The antibacterial activities of ZnO nanostructures can somewhat originate from their ability to create reactive oxygen species during their exposure to light [52]. These reactive oxygen species can destroy bacteria. The positive effects of these composite materials possibly stem from the presence of ZnO nanoparticles. Generation of hydrogen peroxide (H_2O_2) on the surface of ZnO nanostructures plays a pivotal role in their antibacterial activities [53].

4. CONCLUSIONS

The twin-screw extruder was adopted to prepare PP masterbatches infused with varied contents of nano-sized ZnO particles. These masterbatches were then processed using melt spinning to generate ultra-fine fibers showing antibacterial activities.

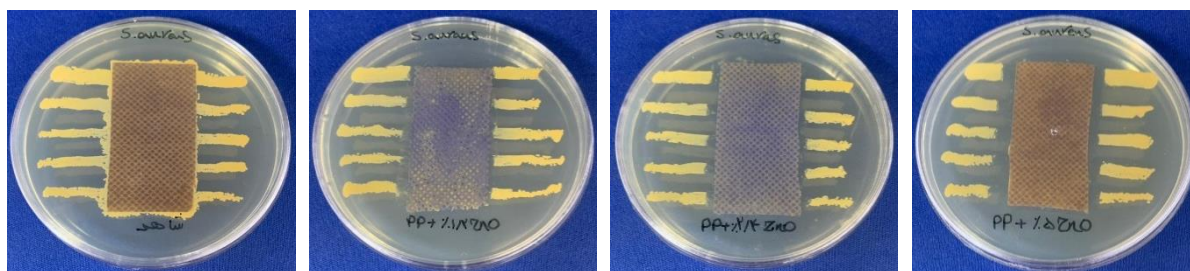


Fig. 7. The Results Obtained from the Measurements of In Vitro Antibacterial Activity of the Prepared Samples Against *S. aureus* bacteria for Spun PP Fibers with Various Concentrations of ZnO: a) Control Sample Free of ZnO, b) 1.2 wt.% ZnO, c) 2.4 wt.% ZnO, and d) 5 wt.% ZnO

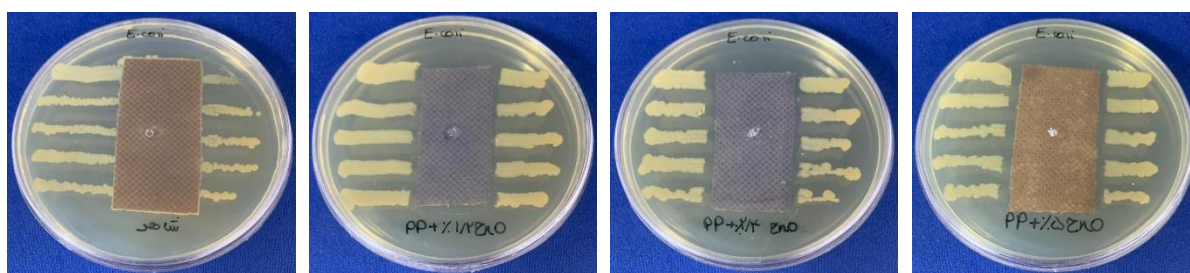


Fig. 8. The Results Recorded from the Assessment of In Vitro Antibacterial Activity of the Prepared Samples Against *E. coli* bacteria for Spun PP Fibers Containing Various Concentrations of Zinc Oxide (ZnO): a) Control Sample with 0 wt.% ZnO, b) 1.2 wt.% ZnO, c) 2.4 wt.% ZnO, and d) 5 wt.% ZnO

TGA curves exhibited that ZnO nanoparticles possess high thermal stability. Moreover, several transformations were observed during the DSC test of ZnO, which indicated its complicated thermal stability. XRD examinations pointed out that the ZnO nanostructures have a hexagonal wurtzite structure, whereas the PP polymer demonstrated an α crystalline form. The images obtained from SEM microscopy revealed that ZnO nanoparticles were uniformly distributed inside the matrix of the PP polymer. FTIR, along with XRD analyses, showed that the introduction of ZnO into the PP polymer has negligible changes on its structure. The antibacterial activities of the produced PP fibers with various amounts of ZnO were investigated. In this regard, the fibers with 1.2 wt% and 2.4 wt% of nano-sized ZnO particles exhibited higher antibacterial activity against both *S. aureus* and *E. coli* bacteria compared to other samples.

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