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RESEARCH PAPER

Preparation and Study of the Properties of PVA/PVP Polymer Composite Materials Reinforced with Metal Oxides and MWCNTs in Medical Applications

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ABSTRACT

This study addressed the preparation of multicomponent samples composed of a double polymer matrix of polyvinyl alcohol (PVA) and polyvinyl pyrrolidone (PVP) reinforced with metal oxides (zinc oxide and iron oxide) and carbon fibers. Different slides (A1-A5) with various compositions were prepared to study the relative influence of the different components on the physical, chemical, and structural properties of the resulting composites. X-ray diffraction (XRD) analysis revealed the presence of distinct crystalline peaks at specific diffraction angles (31.65°, 33.10°, 36.31°, 47.68°, 56.60°, 62.84°, and 67.97°), confirming the crystalline structures of zinc sulfate. Furthermore, the probe peaks at 19.65° are attributed to the semicrystalline nature of the polymers used. The analysis also revealed the presence of iron oxide crystal phases at angles (33.10°, 40.86°, 49.4°, 53.94°, and 63.98°).SEM images at different magnifications (200 and 500)nm revealed a completely flat morphology, featuring spherical nanoparticles and cubic clusters within an outward-facing polymer matrix. This included a partial difference in the force distribution between the different bands, reflecting the effect of the ratio on the morphology. Energy distribution analysis (EDS) with elemental distribution mapping demonstrated the simultaneous presence of zinc, iron, carbon, and carbon in the composition and the relative distribution of the elements between the different bands. The results obtained indicate the potential for these samples to be used in diverse applications such as photocatalysis, antimicrobial materials, electronic applications, and specific analyte delivery systems.

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INTRODUCTION

The history of polymers is linked to the development of man, as he used them in the clothing industry and in the manufacture of dyes and glues, and they were later used to protect against water, such as asphalt used in painting boats [1]. The beginnings of laboratory studies of polymers date back to the beginning of the twentieth century, specifically to the pioneering studies of the scientist (Staudinger) (1920) in studying the long molecular chains involved in the composition of the polymer to be the cornerstone in building

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the science of polymers.[2] Polymer science they known as the science of large molecules that are built by repeating small chemical units called monomers, each of which represents the basic unit of polymer construction. These simple units they linked together by chemical bonds to form long molecular chains. Therefore, they usually have a high molecular weight compared to other compounds [3, 4]. The number of monomers that make up the polymer chain they called the degree of polymerization [3]. Polymer blends they defined as a combination of two or more types of polymers mixed physically. The blends consist of covalent bonds between them, and the resulting mixture has properties that are proportional to the ratio of the basic components in addition to having mechanical properties. [5] The process of mixing two polymers together to form a complex compound whose properties differ greatly from the properties of the individual polymers that make it up. Many mixtures are of lower quality than the two components that make them up, it were also many mixtures that have intermediate properties or that are superior to the properties of the components that make them up [6]. Blends exhibit a wide and variable behavior from brittle behavior of polymers to flexible behavior, thus showing a significant improvement in toughness and impact strength [7]. An example of this is mixing polybutadiene with polystyrene in small proportions to prevent the brittle polystyrene from gaining flexibility. [4] Among the most important reasons for adopting blends [8] are is the easiest and cheapest way to produce polymer materials with good specifications compared to other advanced methods, because it requires good mixing before casting. It is also possible to control the structure of the polymer. Compound materials are the materials resulting from the combination of two or more materials that do not interact chemically, but they combine physically to form a new material that has characteristics that differ from the properties of the materials that compose it from a practical standpoint, which is the low density and high durability of the manufactured product, as well as superficial properties such as corrosion resistance. Each type possesses a specificity in performance, properties, and application that distinguishes it from other types. Therefore, given that, the composite materials combine the properties of two or more materials; their properties are suitable for many building materials. Industrial applications, and thus occupy a prominent position among the various engineering materials, overcoming or reducing the disadvantages of each material, in addition to the unique ability to control its properties, whether through the type and proportions of the raw materials that make up it or through its engineering and manufacturing methods .[9] Polyvinyl Alcohol (PVA) It is an odorless, water-soluble synthetic polymer made from vinyl acetate monomer by subjecting it to polymerization and partial decomposition by dissolving it in water, and allowing the water to evaporate to form a transparent film with high plasticity and tear resistance. The solubility of the polymer depends on the degree of molecular decomposition and the ability of hydrogen bonding [10]. Research indicates that the combination of PVA and PVP leads to the formation of an interpenetrating polymer network (IPN), which enhances the mechanical properties of materials. The interpenetration of the IPN network reduces the mean distance between molecules and improves molecular mobility, contributing to better mechanical performance of the hydrogels formed [11]. PVP also acts as a stabilizing agent for nanoparticles, providing temporal stability to nanoparticles such as ZnO, making them suitable for environmental and nanotechnology applications[12].Studies have demonstrated that zinc oxide (ZnO) can significantly improve the mechanical properties of plastics. It has been found that the addition of ZnO to hydrogels accelerates the reaction between PVA and PVP, resulting in enhanced optical and mechanical effects [13]. Iron oxide is also considered prominent in enhancing material properties related to mechanical processing due to its strong bonds in hydrogels [14].Research has shown that PVP-supported ZnO nanoparticles have antibacterial properties, making them useful in environmental applications such as water disinfection [12]. These nanopatterns also contribute to improved electrical properties, with studies demonstrating the effect of zinc oxide on the electrical performance of PVP-integrated solar cells[15].On the other hand, research suggests that PVA and PVP are excellent examples of nanoconductive applications for various particles. Results have shown that PVP-enhanced hydrogels increase cross-linking efficiency by strengthening the network and increasing stability[16].Although hydrogels made from PVA and PVP offer numerous advantages, balancing the ratio of components is important, as increasing the PVP ratio can lead to poor mechanical properties due to multiple interactions between the different polymer chains[17].Research has shown that PVA polymer possesses important properties that make it suitable for medical applications. PVA is non-toxic and exhibits excellent biological properties such as biodegradability and bioadhesion, making it an ideal material for wound dressings and drug delivery applications [18, 19]. PVA-based hydrogels can be used in the development of chemically active wound dressings to reduce inflammation and improve wound healing [20]. PVP also offers distinct advantages, acting as a catalyst to help stabilize nanoparticles such as ZnO and Fe_2O_3, leading to the development of nanocomposites with antibacterial properties [21, 22]. Studies have shown that the combination of PVP and ZnO enhances antibacterial efficacy, as the nanoparticles destroy bacterial cells upon adhesion [23, 24]. This makes them ideal for use in the development of therapeutic coating materials and wound cleaning. Research also suggests that incorporating PVA and PVP with ZnO and iron oxide can improve the mechanical and chemical properties of new composites. For example, the effects of iron oxide on polymer formulations have been studied to provide enhanced structural properties [25, 26].

MATERIALS AND METHODS

Materials

Polyvinyl alcohol (PVA), Poly vinyl pyrrolidone (PVP), Sodium hydroxide (NaOH), Iron (Fe) and Zinc (Zn), Carbon fiber

Preparation Polyvinyl alcohol (PVA), Poly vinyl pyrrolidone (PVP)

Dissolve 10 grams of PVA powder in 50 ml of distilled water, stirring continuously using a magnetic stirrer, at 50 °C until the mixture becomes a gel-like consistency (this may take several minutes). After making sure that all PVA molecules have completely dissolved and there are no lumps, the PVP polymer was prepared in the same way. Then the two solutions they combined or mixed to gather and placed in a blender to homogenize them together and wait until the mixture becomes transparent. This sample is designated A3.

Preparation of Zinc Oxide (ZnO)

They mixed 4 grams of zinc powder with 50 ml of distilled water in a beaker and placed it on a magnetic stirrer. After ensuring the zinc was dissolved, they gradually added drops of the previously prepared sodium hydroxide solution and observed the transformation that occurred, forming a white precipitate of zinc hydroxide (Zn $(OH)_2$). They then calculated the pH of the solution, which is between 7 and 9, as zinc hydroxide (Zn $(OH)_2$) is formed because of the reaction of zinc with sodium hydroxide. To convert zinc hydroxide (Zn $(OH)_2$) to zinc oxide (ZnO), they placed the zinc hydroxide (Zn $(OH)_2$) in a tube inside a centrifuge for 10 minutes. They repeated the process twice, then placed the solid in a shower in an oven at 100°C and left it for an hour to dry and remove moisture. I then ground the dry material into a fine powder and put it back in the oven at 450 degrees for 2 hours until the zinc hydroxide was completely converted to zinc oxide (ZnO). This sample is designated A1.

Preparation of iron oxide (Fe_2O_3)

The process involved adding 4 grams of finely dispersed iron (Fe) powder to 50 mL of distilled water under continuous magnetic stirring. A previously made sodium hydroxide (NaOH) solution received drops while being intensely stirred until a uniform suspension. The reaction formed iron(II) hydroxide in a pale green precipitate through the process described by this reaction:

$$H_2(g) + Fe(OH)_2(s) \rightarrow H_2O\ (l) + 2NaOH(aq) + Fe(s)$$

The solution pH readings between 7 and 9 showed a basic condition, which promoted the formation of $Fe(OH)_2$ as the precipitate.

The precipitate was obtained by centrifuging at 3000 rpm for ten minutes, followed by two washes with distilled water. This method was repeated twice to completely remove the remaining sodium ions from the samples. After obtaining the iron(II) hydroxide, we transferred it to a heating apparatus for one-hour drying at 100 °C to evaporate moisture.

A mortar and pestle was used to grind the dried solid material for obtaining a homogeneous powder. The muffle furnace treated the powder at 450 °C during two hours for $Fe(OH)_2$ thermal decomposition into reddish-brown iron(III) oxide (Fe_2O_3).

$$3H_2O(g) + Fe_2O_3 \rightarrow 2Fe(OH)_3$$

Preparation of polymer (PVA, PVP) with ZnO and Fe_2o_3

Weigh 5 g of PVA and PVP polymers. Add distilled water to the polymers first, mixing well until the polymers are completely dissolved. After thoroughly preparing the polymers, weigh 0.3 g of zinc oxide and iron oxide. Add ZnO and Fe_2O_3 to the solution containing the polymers. Place the solution in a magnetic stirrer at medium speed until the ingredients are completely homogeneous. Continue mixing for 30 minutes to an hour. This sample is designated A4.

Preparation of polymer (PVA, PVP) with ZnO and Fe_2o_3 and carbon fibers

Weigh 5 g of PVA and PVP polymers. Add distilled water to the polymers first, mixing well until the polymers are completely dissolved. After thoroughly preparing the polymers, weigh 0.3 g of zinc oxide and iron oxide. Add ZnO and Fe_2O_3 to the solution containing the polymers. Then add the carbon fibers. Place the solution in a magnetic stirrer at medium speed until the ingredients are completely homogeneous. Continue mixing for 30 minutes to an hour. This sample is designated A5.

RESULTS AND DISCUSSION

XRD Analysis

The sharp and distinct diffraction peaks in A1in Fig. 1, with their high intensity and low background, indicate a crystalline ZnO sample with little amorphous content. The pattern matches the standard reference pattern for zinc oxide (card 96-230-0451), which has a hexagonal crystal

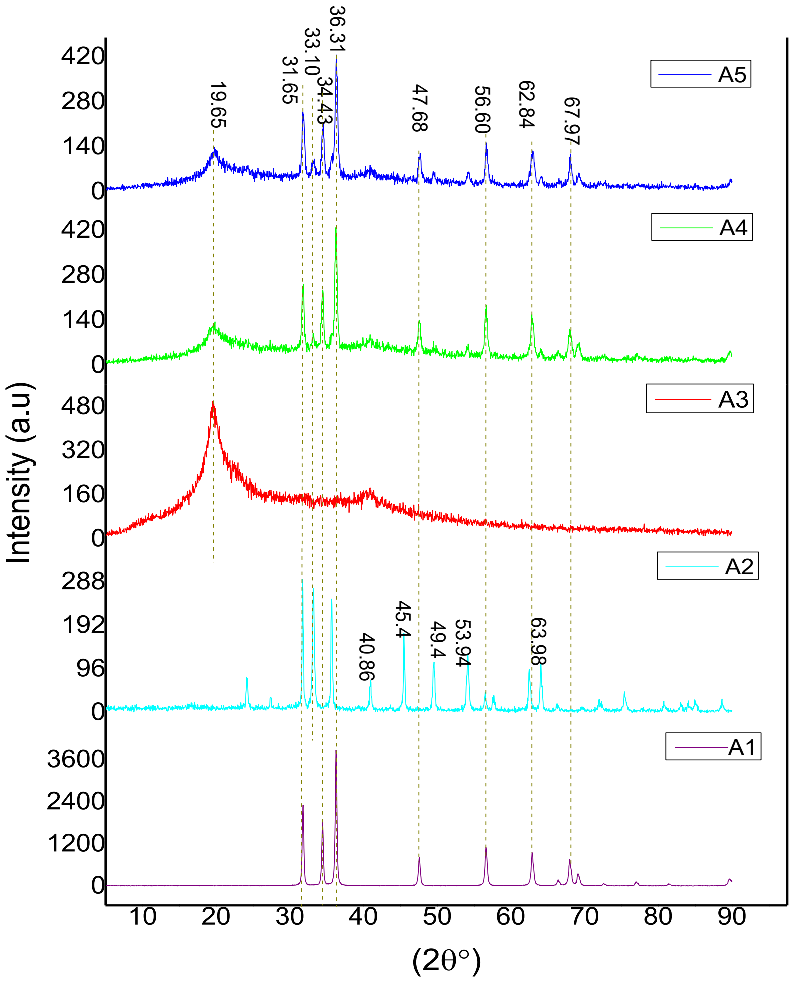
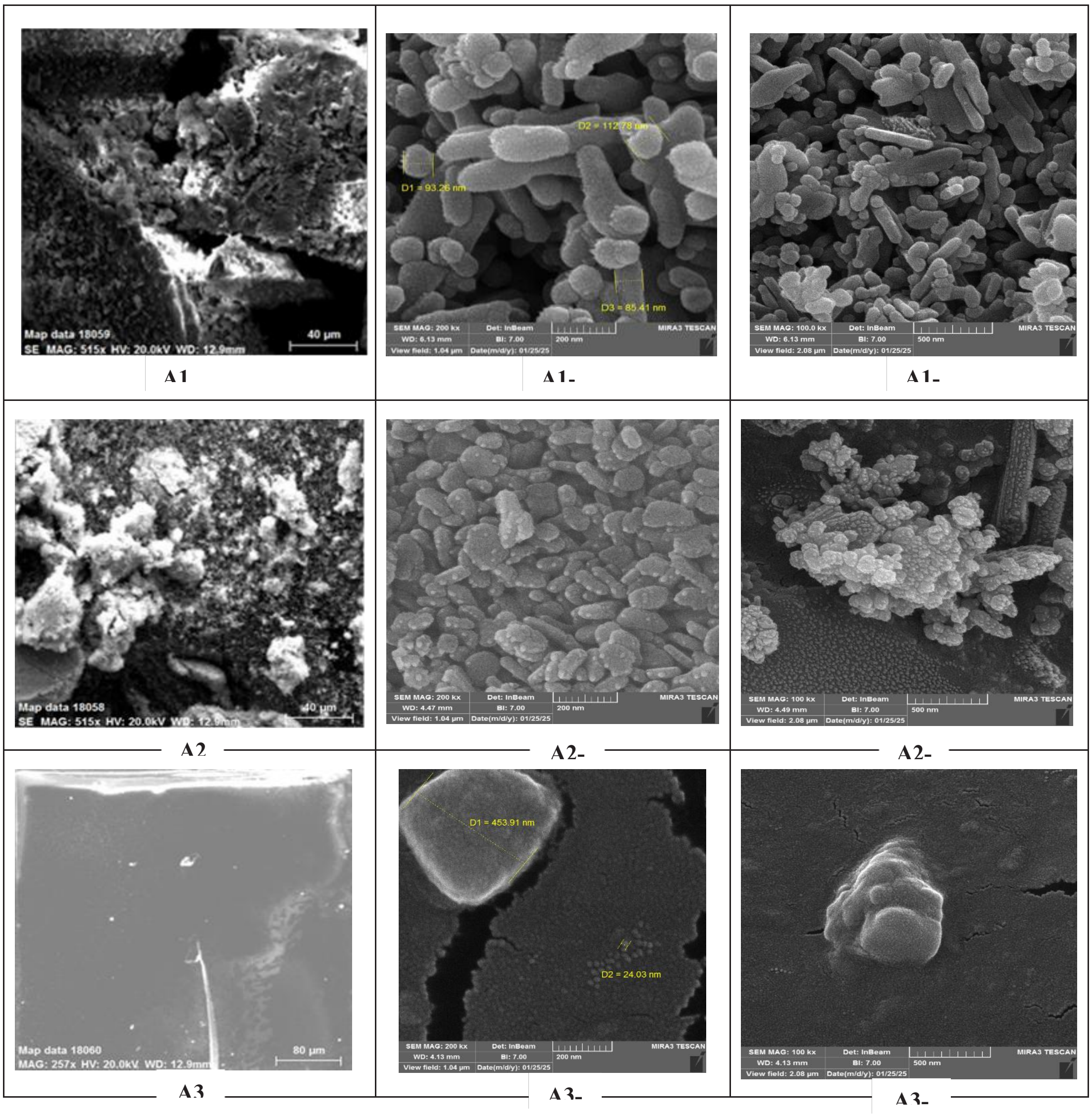


Fig. 1. XRD spectra of the as-prepared samples.

structure. The relative intensities and positions of these peaks serve as a fingerprint confirming the identity and phase purity of the ZnO sample. The extremely high intensity of the peaks, especially the 36.31° (101) reflection, indicates well-formed and well-crystallized crystals. The sharp and distinct diffraction peaks in A2 indicate a crystalline Fe_2O_3 sample. The pattern matches the standard reference pattern (card 96-101 -1 2 41) (Fe_2O_3), which has an orthorhombic crystal structure. The relative intensities and positions of these peaks are characteristic fingerprints that confirm the identity and phase purity of the Fe_2O_3 sample. The high intensity of the peaks, especially those at (211) 31.65° and (1-10) 33.10°, indicate well-formed crystals. The background pattern is relatively low, indicating a low content of amorphous material in the sample. Spectroscopic analysis of the A3 polymer samples reveals the interaction between the PVA and PVP polymers and the hydrogen bonding characteristics, as well as the relative intensities of specific molecular interactions. The sharp initial peak at 16.56°, followed by a secondary shoulder at 40.86° and then a gradual decline, is a feature of polymer spectroscopy, indicating the different crystalline


A1 A1- A1-

A2 A2- A2-

A3 A3- A3-

Fig. 2. SEM image of samples with three different magnification, 200 nm, 500 nm.

phases present in the polymer blend. Hydrogen bonding interactions between the hydroxyl groups in PVA and the carbonyl groups in PVP indicate varying degrees of molecular organization in the polymer blend. For sample A4, the main peaks are the high intensity peak at about 36.31° peak (101) and the highest peaks at 31.65° (100) and 34.43° (002). Additional peaks such as (110), (103), and (112). This design demonstrates the ultimate success of plastic composites with iron and zinc, as it demonstrates both the semi-crystalline nature of the polymers (region linked). Crystal structure of ZnO, Fe_2O_3. The diffraction pattern of sample A5 shows a broad region at about 20-30 units on the horizontal axis, typical of semi crystalline polymeric components. High-pressure peaks at about 35-40 units are seen corresponding to the crystalline planes of zinc oxide. Medium-intensity peaks of carbon at about 50-70 units are represented by the carbon fiber coating and oxides. The presence of the carbon-beneficial peaks indicates successful integration of the inorganic components (zinc oxide and carbon oxides) into the polymer. This type of carbon analysis is used to determine the composition of the final assembly and to understand the interactions between the organic (polymer) and inorganic (oxides and fibers) components, as well as to determine the crystalline properties of the final composite.

The Morphological Investigations

The surface morphology of A1, A2, A3, A4, and A5 was studied at different magnifications 200 nm, 500 nm as shown in Fig. 2. These images, as in the enlarged images A1 showed Iron oxide particles a hierarchical arrangement, with both large aggregates and small crystals appearing. There are visible voids and spaces between the particle clusters, indicating a porous microstructure. Zinc oxide typically exhibits a variety of morphologies depending on its synthesis method, including spherical particles, nanorods, rosette structures, and irregular cluster formations, the relatively rough surface texture and irregular shapes of these particles indicate this. Images A2 shows clusters of particles crystals with agglomerated bases and an unusual shape for clustering. The surface structure shows various chirality structures, consisting of large portions of agglomerated particles, small like cylindrical structures, and small spherical particles scattered in the background. The characteristic photoetching characteristics of iron oxides, which

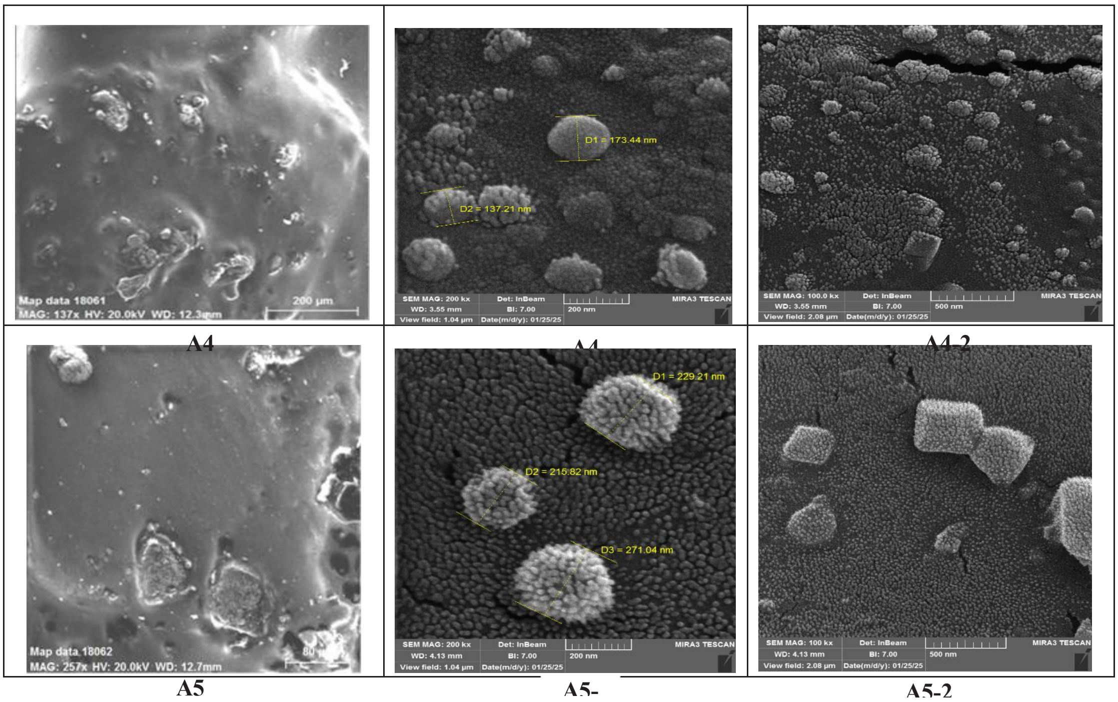


Fig. 3. EDS images of mapping with different continents.

often take on these agglomerated and branched shapes, are characteristic of these structures. The visible agglomerates are typical of nano- or micro-sized iron oxides formed either by chemical precipitation processes or by redox reactions. Images A3 shows a spheroidal structure, most prominent in the image, appearing as an aggregate composed of several closely packed spherical clusters. The back surface exhibits cracks and striations, typical of a polymeric film after drying or heat treatment. This image has distinctive properties, images A4 shows the background consisting of a regenerated nanostructure and a mixture of two polymers (PVA, PVP). The small grains, known for their high density, represent the electrochromic contrast of zinc and iron oxides. Larger, well-developed agglomerates (with several different features) are distributed on the surface. These represent the largest amount of zinc oxide and iron oxide particles, which exhibit

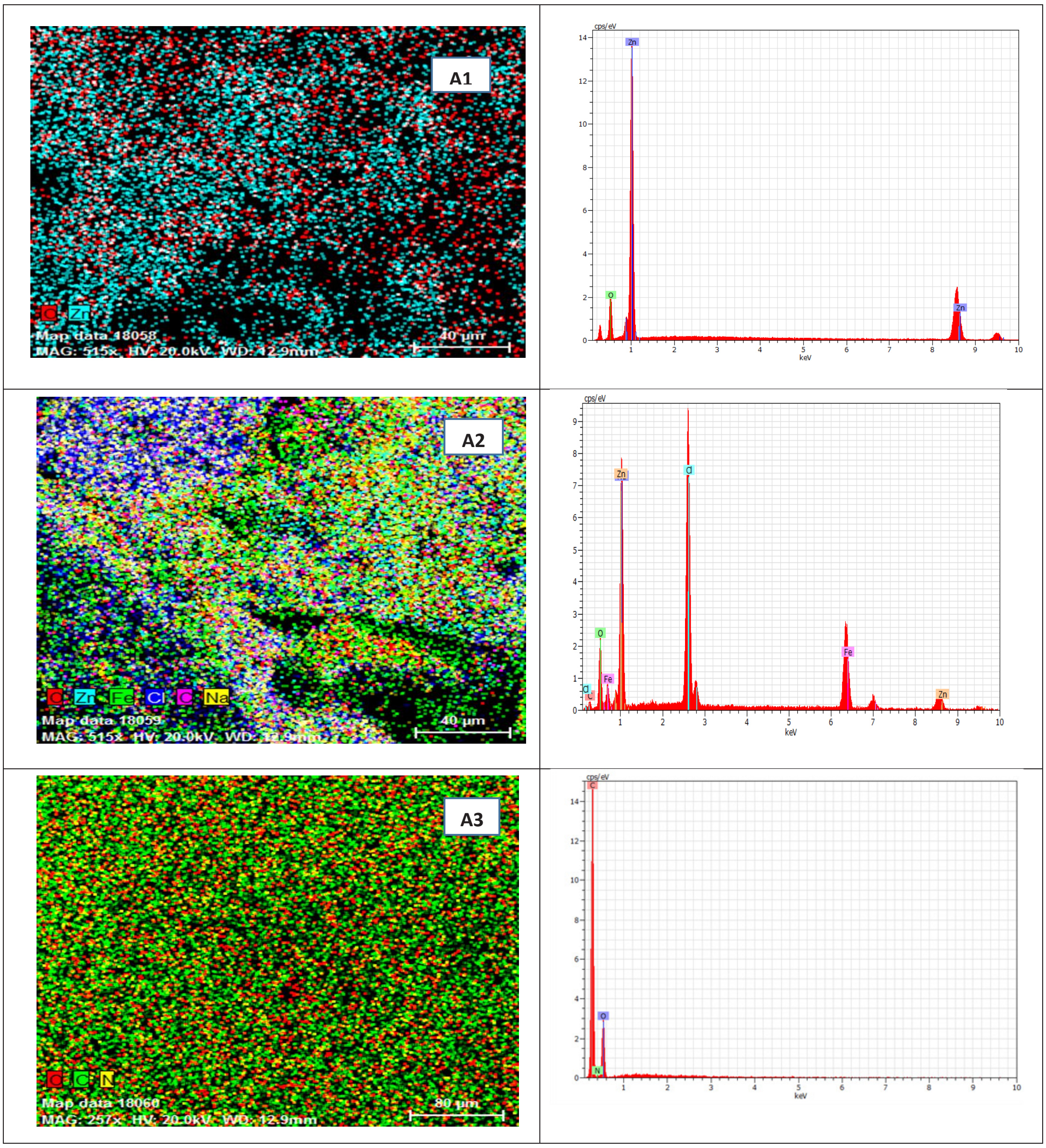


Fig 4. Antibacterial activity of A3, A4 andA5 at (0.5 M) against (a) Staphylococcus aureus, (b) E. coli.

agglomeration due to the van der Waals force and their magnetic properties. A core structure is observed in the central image, as well as surface cracks. These cracks are a result of internal stresses generated during the solvent and solidification of the polymer. This image demonstrates the successful incorporation of metal oxides into the polymer matrix. There are collective clusters in certain areas to form crystals that can be extracted from the matrix. Images A5 shows cubic or pseudo-cubic crystals: a clear transition to larger structures (about 1 µm in size). These distinctively shaped crystals represent zinc oxide crystals that grew rapidly during the preparation process. It consists of very small spherical nanoparticles partially dispersed in a regenerated form, which represents a stereoscopic background of anti-shock zinc oxide and iron oxide in the polymer matrix. Some advanced cracks can be observed in the surface, which are mostly caused by internal stresses during the polymerization and drying process. We note great success in synthesizing multi-component composites with a good relative distribution, the usual reproduction of oxidation under other conditions, the presence of interaction between the various components, and the success of the nanocomponents in the polymer matrix.

The EDS (Energy Dispersive X-ray Spectroscopy) image results, shown in images A1 to A5. The EDS images in Fig. 3 provide an elemental analysis of the distribution of various elements in polymer composites. This technique uses X-rays emitted by the sample to identify the elements present and their spatial distribution. In the case of PVP and PVA composites with Fe_2O_3, ZnO, and carbon fibers, the images show the distribution of Fe within the polymer matrix, reflecting the diffusion of iron oxide particles; the Zn distribution, which represents the distribution of zinc oxide particles; the C distribution, which shows the presence of carbon fibers as well as the structure of the polymer itself; and the O distribution, which is associated with metal oxides. In PVA, these composites combine the properties of polymers (such as flexibility and ease of molding) with the properties of additives. Iron oxides add magnetic properties; zinc oxide adds semiconductor, antibacterial, and UV-absorbing properties; and carbon fibers improve mechanical properties and thermal and electrical conductivity. The EDS images show the homogeneity of the distribution of these components, which is critical to the performance of the composite material.

Antibacterial Activity

Based on the inhibition zone study, Fig. 4 shows the antibacterial activity of three samples (A1, A2, and A3) calcined at 600°C at a single concentration (0.5) at pH 9 against Staphylococcus aureus and Escherichia coli. The antibacterial activity test results for calcining the three samples at 600°C showed resistance. The diameter of the inhibition zone was measured in millimeters. The disk diffusion method was used to conduct antibacterial testing on bacterial suspensions spread on a Mueller Hinton agar plate. The inoculated plates were incubated for 24 hours at the optimum temperature. One sample of each concentration of A3, A4, and A5 was placed on the agar surface to study their effect on the bacterial species used.

CONCLUSION

Examinations, including scanning electron microscope (SEM) images, simple X-ray diffraction (XRD) analysis, and energy distribution maps (EDS), revealed the crystal structure. Micro-X-ray diffraction evidence (A1-A5) revealed a clear contrast in the crystal structure. Sample A1 exhibited peaks only at specific angles (40.86°, 45.4°, 49.4°, 53.94°, and 63.98°), indicating a high degree of crystallinity, while sample A3 exhibited a broader peak at 19.65°, indicating the zonation of the semi-crystalline PVA and PVP polymers. The effect of carbon composition on morphology: Surface morphology varies greatly between different specimens, with the proportion of components (polymers, zinc and iron oxides, fibers) corresponding to a distinct size, shape, and morphology. This allows for the possibility of controlling the acoustic properties of the material by adjusting its apparent composition. The effect of carbon can be observed that the addition of carbon fibers increases the interconnectivity in the polymer matrix, as these fibers connect as bridges between different regions of the display material, resulting in its mechanical and electrical properties. Carbon fibers exhibit multi-scale component homogeneity ranging from the differential level (metal oxide particles) to the microscale level (polymer fibers and structure). This hierarchical system provides long-term, short-term properties. The influence of zinc oxide and iron oxide crystalline peaks at 31.65°, 33.10°, and

36.31° confirm the presence of zinc oxide crystals in the form of wurtzite, while the peaks at 34.43°, 62.84°, and 67.97° indicate the presence of iron oxide in the form of hematite. This feature gives the material its distinctive magnetic and optical properties. The data revealed the presence of chemical interactions between the hydroxyl groups in PVA and the carbonyl groups in PVP with surface atoms of metal oxides, which increases the bonding between the organic and inorganic components.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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