

RESEARCH PAPER

## Preparation and Study the Effect of Adding Zinc Oxide/ $\text{Fe}_2\text{O}_3$ Nanoparticles on the Properties of the Mixture Polymers (PVA+PVP)

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### ABSTRACT

The study deals with the preparation of multicomponent composite materials using two polymers (PVA and PVP) reinforced with nanoparticles of zinc oxide and iron oxide. Several samples (HB1-HB4) were prepared to test the effect of different components on the structural properties of these compounds, X-ray analysis (XRD), scanning electron microscopy (SEM) and also Elemental spectroscopy (EDS) that showed the presence of clear crystalline structures for metal oxides, good distribution of particles within the polymeric matrix. The possibility of using these materials in multiple applications such as photocatalysis, antibacterials, electronic devices, and material delivery systems.

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### INTRODUCTION

Polymers are one of the most important materials, that have received significant attention from scientists, chemists, and physicists worldwide due to their applications in the fields of technology and scientific research, such as solar energy conversion, coatings, adhesives, lithography, light-emitting diodes, sensors, laser development, and many other applications. Selected additives with specific properties are added to these materials to improve their properties [1,2]. Polymer materials are high-molecular-weight compounds, manufactured through a series of chemical reactions [3]. Traditionally, most polymers were used only to produce inexpensive products, typically designed to perform specific basic functions. However, rapid advances in technology have necessitated the replacement of

some materials used in the industrial sector with performance-enhancing alternatives. As a result, polymer development has witnessed significant and rapid growth. In the current era, scientists are actively seeking to develop cost-effective, adaptable, and versatile polymers. [4].

Polymers have numerous applications in various fields, including food, where Polymer materials are used in food industry. First, we consume a variety of foods, such as vegetables, meat, and staple foods, which are primarily composed of polymers classified by molecular weight. Second, plastic takeaway food containers and bags are popular in the food industry due to their light weight, non-toxicity, and low cost, making them ideal for everyday diets. [5]. In the early days of relatively simple technology, humans began using these polymer materials to improve their lives, such as

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by making woolen clothing for heating. Nature consists of many similar polymer materials, such as proteins, starch, wool, silk, and others, which are the basic materials that make up living organisms [6]. Polymers have been present in our lives since the dawn of time. They are the foundation of life (its basic building blocks), as animals, plants, and all living organisms are made of polymers. However, it wasn't until the mid-20th century that we truly understood their nature. This understanding was evident with the development of plastics, man-made materials that represent the ultimate tribute to human creativity and intelligence. The use of polymeric materials has permeated every aspect of our lives. It's difficult to imagine today's world, with all its convenience and ease, without man-made polymeric materials. [7]. A polymer is a large molecule of high molecular weight obtained through the chemical reaction of many small molecules of low molecular weight of one or more types. The process of manufacturing polymers is called polymerization [8]. There are many types of polymers that different in composition and properties, the most important is the polyvinyl alcohol (PVA) which is a thermoplastic polymer having a very high dielectric strength, good charge storage capacity, excellent electrical, optical, and mechanical properties, high tensile strength, and excellent film formation. Both polyvinyl alcohol (PVA) and polyvinyl alcohol (PVP) are environmentally friendly and biodegradable polymers, and thanks to their polar groups, they are easily soluble in water and suitable organic solvents [9,10,11].

Zinc oxide (ZnO) is a white powder that is an essential element in many industrial manufacturing processes and used as an additive to many materials and products. However, most zinc oxides are prepared industrially [12]. Over time up to this day, zinc oxide has received significant attention in various fields and became a valuable material because of its distinctive properties, such as UV absorption, electrical conductivity, transparency, piezoelectricity, luminescence, easy to manufacturing, biocompatibility, and radiation hardness, etc. with these properties, zinc oxide has emerged and played a significant role in several fields, such as its role in developing growth techniques for the fabrication of high-quality single crystals and epitaxial layers, enabling the realization of zinc oxide-based electronic and optical devices. Furthermore, zinc oxide is

environmentally friendly [13,14]. Its properties include a wide-bandgap semiconductor with high optical transparency and luminosity in the visible and near-ultraviolet spectrum. Therefore, it is commonly used in light-emitting diodes and solar cells. Zinc nanoparticles have a high exciton binding energy of approximately (60) MeV. This means that excitonic transition in the case of zinc oxide nanoparticles is possible at room temperature [15]. Several methods are used to manufacture ZnO nanoparticles, and the preparation process is followed by controlled drying and freezing processes such as deposition, spray pyrolysis, microemulsion, hydrothermal, and sol-gel methods [16]. Zinc oxide has three main crystal structures: hexagonal, cubic, zinc alloy, and rock salt. The zinc alloy structure is mostly stabilized by growth on cubic structures, while the rock salt structure is a high-pressure metastable phase (at ~10 GPa) that cannot be superimposed [17,32].

Iron oxide is one of the most important minerals, its nanoparticles have attracted great attention due to their outstanding properties, including their excellent thermodynamic properties, strong ecotoxicity, resistance to biotoxicity and corrosion, high stability, strong redox potential, and adsorption capabilities [18]. Iron oxide is a transition metal oxide that crystallizes in various stoichiometric and crystalline structures, including wustite (FeO), hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), and magnetite (Fe<sub>3</sub>O<sub>4</sub>) [19]. Iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>) is a dark red, ferromagnetic mineral that is easily polluted by acids. Fe<sub>2</sub>O<sub>3</sub> has multiple crystal structure forms, such as alpha ( $\alpha$ ), beta ( $\beta$ ), gamma ( $\gamma$ ), and epsilon. These different crystal phases indicate an octahedral coordination geometry, with iron (Fe) at the center bonded by six oxygen bonds. Iron oxide is a suitable compound for the general study of magnetic and structural polymorphisms and phase transitions of nanoparticles [20,21]. Hematite is the most stable iron oxide and an environmentally friendly semiconductor used in red pigments [22,23], catalysts [24], electrodes [25], gas sensors [26,27], magnetic materials [28], and photocatalysis [29]. The most common and widespread mineral forms are the hexagonal " $\alpha$ " corundum structure and the cubic " $\gamma$ " spinel structure, which occur in nature as the minerals hematite and maghemite. Other metallic shapes have been synthesized, namely rhombic, bicubic and epsilon structures, as well

as nanoparticles of all shapes [30]. The properties of nanopowders depend largely on their phase, microstructure, and surface properties. The importance of iron oxide cannot be ignored due to its necessity for accurate measurement of electricity and magnetism. Researchers have used various methods to prepare homogeneous iron oxide nanoparticles, such as sol-gel processes [31].

## MATERIALS AND METHODS

### *Preparation of Materials*

Polyvinyl alcohol (PVA), Polyvinyl pyrrolidone (PVP), Sodium hydroxide (NaOH), Zinc (Zn) and Iron (Fe).

### *Preparation of Polyvinyl alcohol (PVA), Poly vinyl pyrrolidone (PVP)*

We weight (10) grams of PVA and put it in a beaker containing (100) ml of distilled water, then we placed it on the magnetic stirrer, a small magnet was placed inside the mixture (Magnetic Bar), which helps to stir the solution continuously to obtain a homogeneous solution at a temperature of (50°) degrees Celsius. The process continues for minutes until the powder dissolves completely in the water and turn into gel substance we obtain a homogeneous mixture, which is called (PVA). We prepare PVP in the same way, then mix (3) grams of PVA and ( 7) grams PVP, we get (10) grams of (PVA/PVP), gave it a symbol (HB3).

### *Preparation of sodium hydroxide (NaOH)*

(5) grams of sodium (Na) powder were dissolved in (50) ml of distilled water. The mixture placed in a glass tube (Beaker), then we placed it on the magnetic stirrer, a small magnet was placed inside the mixture (Magnetic Bar), which helps to stir the solution continuously to obtain a homogeneous solution at a temperature of (50°) degrees Celsius. The process continues for minutes until the sodium dissolves completely in the water and we obtain a homogeneous mixture, which is Sodium hydroxide (NaOH).

### *Preparation of zinc oxide (ZnO)*

We weight (4) grams of zinc and put it in a glass beaker containing (50) ml of distilled water and put the mixture on the magnetic stirrer, which contains a magnet inside it that helps to stir the mixture continuously to obtain a homogeneous mixture. After the zinc is completely dissolved in the water, we get zinc hydrexide After that,

we add to the zinc hydroxide drops of sodium hydroxide that we prepared previously. The drops were added continuously until its color changes from transparent to milky white and we measure its acidity (PH) and it is about (9). Then we will stop adding drops of sodium hydroxide.

Put zinc hydroxide in a tube. We must make sure that the tube is not completely filled with zinc hydroxide to ensure effectiveness. Then we put it in the centrifuge for (10) minutes. It is a device used to separate the components of the mixture by rotating the sample at high speed. The device separates the substance from the solvent, as the substance collects at the bottom while the liquid remains at the top. Then we get rid of the liquid and throw it away, add distilled water to the remaining substance at the bottom of the tube, mix it well until the substance is homogeneous with the distilled water, and put it in the device again, that is, we repeat the process twice.

After separating the substance from the liquid, we get rid of the liquid, that we throw it away, so the substance remains in the tube. We pour it into a mold and place it in the convection oven at (100°) to dry After that, it becomes in the shape of a powder. We grind it with a manual mortar to obtain a fine powder. Then we put it in the oven again at (450°) degrees Celsius for one hour after completing the process, we obtain zinc oxide, gave it a symobl (HB1).

### *Preparation of Iron oxide (Fe<sub>2</sub>O<sub>3</sub>)*

We weight (4) grams of iron and put it in a glass beaker containing (50) ml of distilled water and put the mixture on the magnetic stirrer, which contains a magnet inside it that helps to stir the mixture continuously to obtain a homogeneous mixture. After the iron is completely dissolved in the water, we add drops of sodium hydroxide that we prepared previously to the mixture. The drops were added continuously until its changes to gel, we measure its acidity (PH) and it is about (9). Then we will stop adding drops of sodium hydroxide., We put the mixture in a tube. We must make sure that the tube is not completely filled with mixture to ensure effectiveness. Then we put it in the centrifuge for (10) minutes. It is a device used to separate the components of the mixture by rotating the sample at high speed. The device separates the substance from the solvent, as the substance collects at the bottom while the liquid remains at the top. Then we get rid of the liquid and

throw it away, add distilled water to the remaining substance at the bottom of the tube, mix it well until the substance is homogeneous with the distilled water, and put it in the device again, that is, we repeat the process twice, After separating the substance from the liquid, we get rid of the liquid, that we throw it away, so the substance remains in the tube. We pour it into a mold and place it in the convection oven at a temperature of (100°) to dry, After the material dries, it becomes in the shape of a powder. We grind it with a manual mortar to obtain a fine powder. Then we put it in the oven again at a temperature of (550°) degrees Celsius for two hours after completing the process, We obtain Iron oxide, we referred to it with the symbol (HB2).

#### Preparation of (PVA/ PVP and ZnO /Fe<sub>2</sub>O<sub>3</sub>)

We weight (10) grams of polymers (PVA and PVP) mixture and weight (0.3) grams of (ZnO) and (0.3) grams of (Fe<sub>2</sub>O<sub>3</sub>), Place the solution in a magnetic mixer at medium speed until the components are completely homogeneous. Continue mixing for (30) minutes to an hour, we gave it a symbol (HB4).

## RESULTS AND DISCUSSION

### XRD Analysis

XRD for four different samples labeled HB1 through HB4, shows in Fig. 1. The graph displays

diffraction intensity (on y-axis) versus (2θ) angle (on x-axis) from approximately (10° to 90°). Each sample shows a unique diffraction pattern:

1. HB1 (magenta/purple line at bottom): Shows the highest intensity peaks (up to 3600 a.u.) with sharp, well-defined diffraction peaks at around ((30°, 35°, 50°), and other angles. The sharp peaks indicate a highly crystalline material.
2. HB2 (cyan/light blue line): Displays medium intensity peaks with similar peak positions to HB1 but at lower intensities (up to ~280 a.u.).
3. HB3 (red line): Shows a distinctly different pattern with a broad peak around (20-25°) and a gradually decreasing intensity. This broad peak suggests an amorphous or poorly crystalline component.
4. HB4 (green line): Shows peaks at positions similar to (HB1) and (HB20) but with different relative intensities, reaching up to about (280 a.u.).

The vertical dotted green lines show alignment of peaks across different samples, indicating shared crystalline phases among the samples. The peak positions (marked with numbers like (19.65, 33.31, etc.) correspond to specific d-spacings and can be used to identify crystalline phases by comparing with standard diffraction databases, Without additional information about the samples' composition, it's difficult to definitively identify the crystalline phases, but this XRD

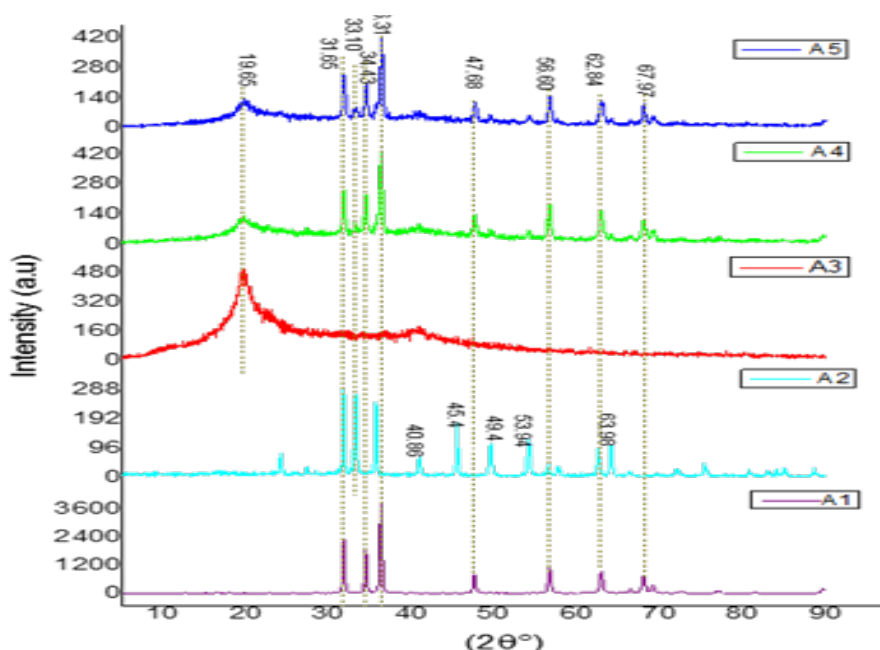


Fig. 1. XRD spectra of the as-prepared sample.

data shows that samples (HB1, HB2 and HB4), contain similar crystalline components, while (HB3) appears significantly more amorphous with different structural characteristics.

The sharp and distinct diffraction peaks in (HB1) in Fig. 1, with their high intensity and low background, indicate a crystalline Zinc Oxide (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 structure. The relative intensities and positions of these peaks serve as a fingerprint confirming the identity and phase purity of the Zinc Oxide (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 HB2 indicate a crystalline Fe<sub>2</sub>O<sub>3</sub> sample. The pattern matches the standard reference pattern (card 96-101 -1 2 41) (Fe<sub>2</sub>O<sub>3</sub>), 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<sub>2</sub>O<sub>3</sub> 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 HB3 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 phases present in the polymer blend. Hydrogen bond interactions between hydroxyl groups in (PVA) and 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).

#### *The Morphological Investigations*

The morphology surface of HB1 (Zinc), HB2 (Fe<sub>2</sub>O<sub>3</sub>), HB3 (PVA+PVP+ZINC) and HB4 (PVA+PVP+Zinc+Fe<sub>2</sub>O<sub>3</sub>), was studied at different magnifications (200 nm, 500 nm) as shown in Fig.. 2. The image (HB1) shows a scanning electron

microscope (SEM) view of what appears to be a microstructural sample, this is likely a high-magnification image of zinc oxide (ZnO) particles or structures. The image shows various rod-shaped and rounded particles clustered together. There's a mixture of elongated rod-like structures and smaller spherical particles, which is consistent with typical zinc oxide (ZnO) nanostructure morphologies. This level of magnification (100,000×) allows for detailed examination of the nano-scale morphology of the zinc oxide structures, which appear to have varied shapes including nanorods and nanoparticles in the size range of tens to hundreds of nanometers. Such morphological investigations are important for understanding how synthesis parameters affect the resulting ZnO structures, which can influence their properties for various applications like sensors, catalysts, or electronic components (Fig. 2, Fig. 3). The image (HB2) shows a scanning electron microscope (SEM) view of what appears to be particles that could be iron oxide (Fe<sub>2</sub>O<sub>3</sub>), though the image itself doesn't specifically identify the material as (Fe<sub>2</sub>O<sub>3</sub>).

The image reveals a highly detailed microstructural view with the following characteristics:

- The particles appear to have irregular, somewhat rounded morphologies with varied sizes
- There's a mix of elongated rod-like structures and more rounded particles
- The particles are tightly packed and show a relatively uniform distribution

For (Fe<sub>2</sub>O<sub>3</sub>) (iron (III) oxide) morphological investigation, this level of magnification would be appropriate to observe crystallite shapes, agglomeration patterns, and size distribution. If this is indeed Fe<sub>2</sub>O<sub>3</sub>, the particles shown could represent either hematite or maghemite phases, which are common forms of iron oxide with different crystal structures and properties. The sub-micron size of these particles (as indicated by the 200 nm scale bar) suggests they could be nanoscale iron oxide particles, which have applications in catalysis, magnetic materials, sensors, or biomedicine depending on their precise composition and properties.

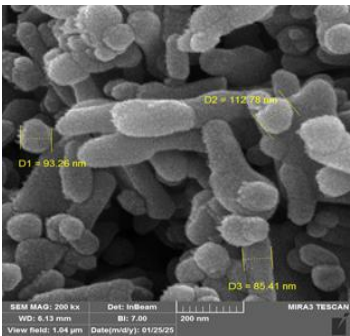
The image (HB3) shows a scanning electron microscope (SEM) micrograph of what appears to be a (PVA+PVP+ZnO) composite material. This is a high-magnification microscopy image taken at



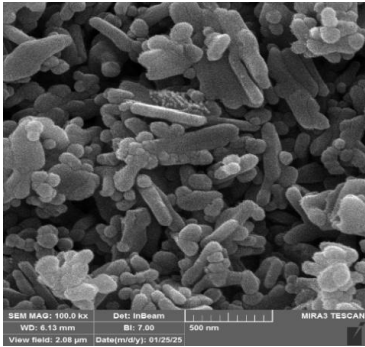
200kx magnification using an InBeam detector, as indicated by the parameters at the bottom of the

image. From the morphological features visible:

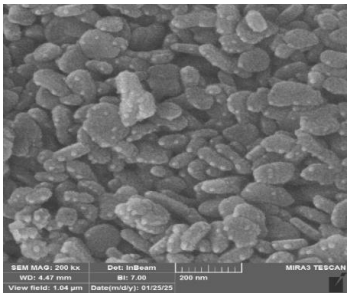
1. There's a distinct brighter particle in the



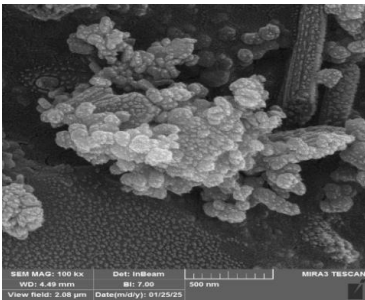
HB1-1



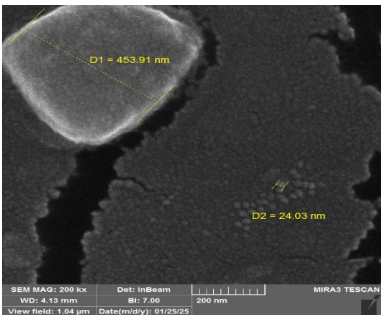
HB1-2



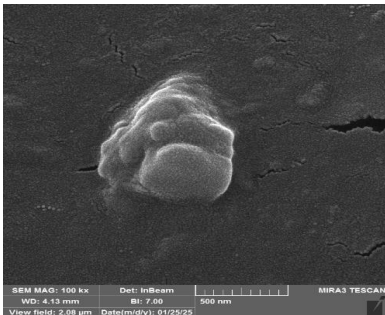
HB2-1



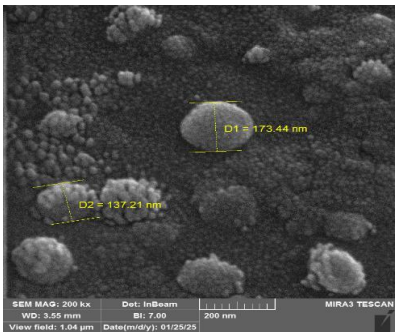
HB2-2



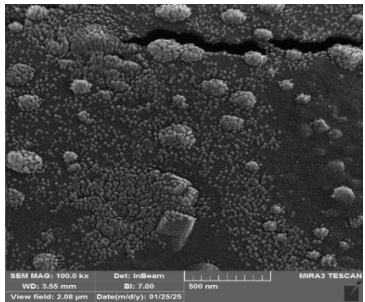
HB3-1



HB3-2



HB4-1



HB4-2

Fig. 2. SEM image of samples with three different magnifications.

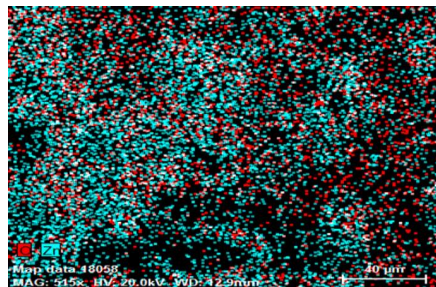
upper left with a somewhat rounded/faceted shape, which is likely a (ZnO) nanoparticle or agglomerate. ZnO typically appears brighter in (SEM) images due to its higher electron density.

2. The darker matrix surrounding it appears to be the polymer blend of PVA (polyvinyl alcohol) and PVP (polyvinylpyrrolidone), which typically

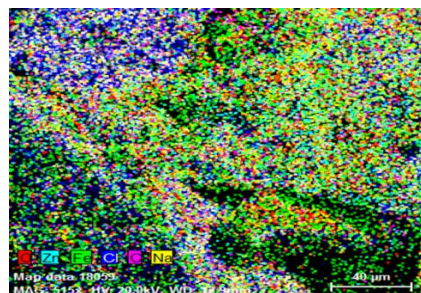
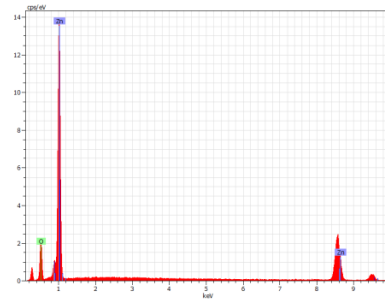
forms a continuous phase.

3. There's a clear interface or gap between the ZnO particle and the polymer matrix, suggesting possible incomplete integration or separation between phases.

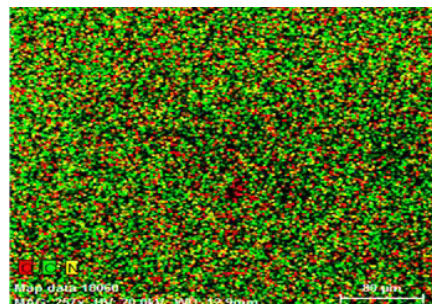
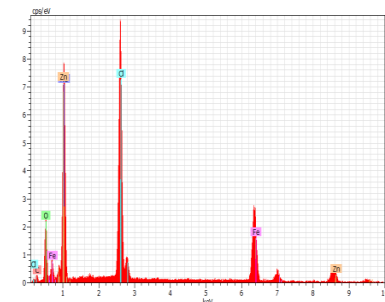
4. The polymer matrix shows some textured regions with small dot-like features, which



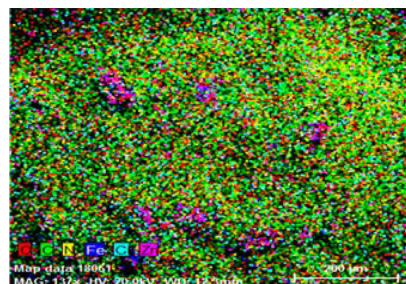
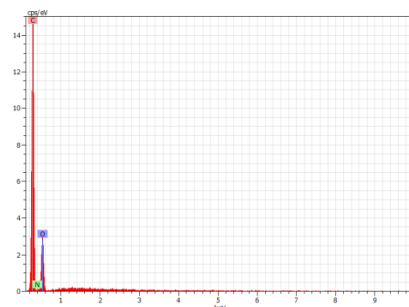
HB1



HB2



HB3



HB4

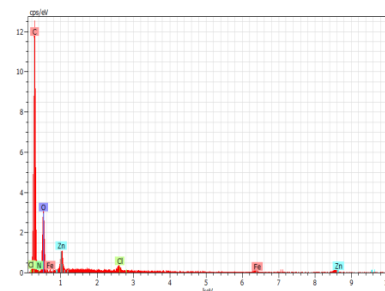


Fig. 3. EDS images of mapping with different continents.

could indicate smaller (ZnO) particles dispersed throughout the polymer or phase separation within the polymer blend itself.

This type of morphological investigation is typically done to study how well the Zinc oxide nanoparticles are dispersed within the (PVA+PVP) polymer matrix, which affects the composite's properties for applications like antimicrobial films, sensors, or optical materials.

The image (HB4) shows a scanning electron microscope (SEM) micrograph of what appears to be nanoparticles or nanostructures. Based on your question about "PVA+PVP+ZnO+Fe<sub>2</sub>O<sub>3</sub> morphological investigation," this is likely showing a composite material containing polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), zinc oxide (ZnO), and iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>).

From the SEM image, we can observe:

1. A prominent spherical particle in the center, approximately 200 nm in diameter based on the scale bar
2. Several irregularly shaped structures surrounding the central sphere, many with a clustered appearance
3. A textured background surface with fine granularity

This morphology is typical of polymer-metal oxide nanocomposites. The spherical particles likely represent metal oxide nanoparticles (ZnO or Fe<sub>2</sub>O<sub>3</sub>), while the surrounding matrix and irregular structures may show the polymer components (PVA and PVP) and their interactions with the metal oxides. In such composites, (PVA) and (PVP) often serve as stabilizing polymeric matrices, while (ZnO) and (Fe<sub>2</sub>O<sub>3</sub>) provide functional properties like magnetic behavior, photocatalytic activity, or antimicrobial effects. The varied morphology suggests a heterogeneous distribution of components, which can significantly affect the material's properties.

## CONCLUSION

1. The samples especially (HB1 and HB2) showed sharp peaks indicating good crystalline structure of (ZnO) and (Fe<sub>2</sub>O<sub>3</sub>).

2. HB3 showed a semi-crystalline structure, while HB4 showed a mixture of crystalline peaks indicating the interaction between the components.

3. Peaks at angles such as (36.31°) and (31.65°) confirmed the presence of zinc oxide (ZnO) crystals, and others such as (33.10° a).

4. HB1 (ZnO): Nanoparticles showed the form of columns and spheres.

5. HB2 (Fe<sub>2</sub>O<sub>3</sub>): showed particles of irregular shape and homogeneously distributed.

6. HB3 (PVA+PVP+ZnO): showed an irregular distribution of (ZnO) particles within the polymeric matrix.

7. HB4 (PVA+PVP+ZnO+Fe<sub>2</sub>O<sub>3</sub>): showed the presence of spherical nanoparticles distributed within the polymeric matrix. nd (40.86°) confirmed the presence of (Fe<sub>2</sub>O<sub>3</sub>).

It showed a distribution of zinc, iron and carbon elements, indicating the integrity of the components of the composite materials. The diverse combination of (ZnO) and (Fe<sub>2</sub>O<sub>3</sub>) with the polymeric matrix has certainly been successful. Having found distinctive structural and morphological properties that can make it for applications such as, Photocatalysis (photocatalysis), Antibacterial materials, electronic applications and Material delivery devices.

## CONFLICT OF INTEREST

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

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