

Characterization of new hybrid composites of PVA-Fe₂O₃-CdZnS

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This study examined the properties of polyvinyl alcohol (PVA) as a matrix composite, specifically focusing on the characterization techniques of UV-Visible spectroscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDX). The investigation centered around a novel hybrid structure composed of CdZnS nanoparticles implanted within a composite matrix of PVA and Fe₂O₃. The analysis of microstructure data provided evidence of the influence of CdZnS nanoparticles on the structural characteristics of PVA-Fe₂O₃. The composites that were synthesized exhibited significant absorption peaks at wavelengths of 233 nm and 234 nm for PVA-Fe₂O₃ and PVA-Fe₂O₃-CdZnS, respectively. A progressive shift towards higher wavelength regions of absorption was found in these composites. The X-ray diffraction (XRD) analysis revealed an average crystalline grain size of 38.417 nm for Fe₂O₃ and 27.267 nm for PVA-Fe₂O₃-CdZnS.

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1. Introduction

The PVA hydrogel is a well-known composite hydrogel with a lengthy research history. Its widespread use can be linked to a number of advantageous traits, including chemical stability, non-toxicity, biocompatibility, resistance to excessive water absorption, biological ageing, and simplicity of processing. Additionally, PVA-based hydrogels are relatively inexpensive to produce and are both chemically and thermally stable. As a result, they have undergone significant research to see

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whether they may be used as tissue engineering, medication delivery systems, articular cartilage, bandages, and other advanced materials like supercapacitors, sensors, and self-healing and shape-memory materials. PVA hydrogels have been used in a variety of nanotreatments for particular human diseases, often in conjunction with Fe₂O₃ and a number of other polymers [1].

Recent studies have demonstrated that PVA hydrogel can replace the artificial nucleus pulposus (ANP) in the management of cervical spondylosis. This alternate method aims to reduce pain and reestablish proper cervical motion [2].

Generally speaking, adding nanoparticles to the host polymer's mechanical, dimensional, and thermal stabilities are improved by the polymer matrix.. Previous research has demonstrated that attaching PVA to the nanoparticle surface raises the PVA cross-linking and melting temperatures, improving the PVA's thermal stability. The nanoparticles are likewise evenly distributed throughout the polymer matrix [3].

Fe₂O₃ nanocubes were synthesised using PVA interactions to produce well-dispersed crystallites. [4], α -Fe₂O₃/(PVA+PEG) were developed to study optical, and dielectric characterizations [5], PVA-Fe₂O₃-TiO₂ hybrid nanocomposite prepared for anticancer and antimicrobial activity, where, the PVA/Fe₂O₃ nanocomposites underwent a significant change in their optical band gap with the inclusion of nanoparticles. The optical band gap of the nanocomposites specifically increased along with the nanoparticle concentration [6]. The PVA-Fe₂O₃ nanocomposite films were made by the solution casting technique, and their properties were analyzed using morphological and spectroscopic methodologies. [7]. A composite matrix of Fe₂O₃-PVA including CuO nanoparticles is being studied for its structural, morphological, and optical characteristics [8], to assess the structure, morphology, and optical characteristics of a recently discovered hybrid structure, a composite composed of Fe₂O₃, Cu, and PVA was created [9], α -Fe₂O₃/(PVA+Chitosan) were fabricated to investigate magnetic properties [2].

This study investigates the preparation of novel a triple hybrid composite made from Fe₂O₃ and CdZnS nanoparticles to modify polyvinyl alcohol (PVA), the synthesis of the materials using the phase inversion method was followed by extensive characterization using Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM).

2. Materials and methods

2.1. Preparation of PVA-Fe₂O₃

In order to synthesize PVA-Fe₂O₃, a solution containing the Fe source was prepared by combining 50 ml of deionized water with 1 g of FeCl₃. A solution containing 1% polyvinyl alcohol (PVA) was employed as a complexing agent. A mixture was prepared by combining 10 ml of polyvinyl alcohol (PVA) solution with 10 ml of ferric chloride (FeCl₃) solution. Subsequently, a small quantity of 1 ml of 1 M sodium hydroxide (NaOH) was added to the resulting mixture. Subsequently, the solutions were subjected to a temperature of 70 oC and agitated for a duration of 60 minutes using a hotplate.

2.2. Preparation of CdZnS nanoparticles

CdZnS nanoparticles were produced by a multi-step method. The ion source solutions were first produced. Zinc nitride was appropriately dissolved in 100 ml of deionized water to produce a solution containing 0.25 M Zn ions. The water was added to the precursor and stirred continuously at room temperature. Clear and transparent solution were made. 0.25 M thiourea solution was prepared

in DI water , to complete the procedure, 10 ml of Zn ions solution were divided into two separate solutions, and 10 ml of thiourea were slowly added to each solution of Zn ions.. The magnetic stirrer was still working to mix the compounds. The pH of final solutions was adjusted to 12 by using ammonia solution. The temperature of reaction fixed at 75 °C and kept on hot plate for three hours. Yellow precipitate powder was formed at the bottom of reaction backer. The powder was collected by centrifuge. The final powder was purified by washing it with water , acetone and ethanol , two time washing with each solvent. The pure powder kept for one night in oven at 80 °C. The nanopowders were dissolved in ethanol for study the optical and structural properties.

2.3. Preparation of PVA-Fe₂O₃-CdZnS Nanocomposite

The physical mixing method was utilized to create a triple hybrid structure comprising PVA-Fe₂O₃-CdZnS. An evaluation was conducted using a 5% proportion of CdZnS NPs relative to the mass of FeCl₃.

2.4. FESEM /EDS Analysis

The FESEM/EDS microscope system examines an object's micro- and nanoscale features well as its chemical elemental makeup on its surface or interior using magnification up to 300,000 times.

2.5. XRD Analysis

The materials were investigated using an X-ray diffraction (XRD) diffractometer fitted with Cu-K alpha radiation, identification the samples done across the 2 range of 10 - 90° at a scanning rate of 2 s per step and a step size of 0.05° at 25°C.

2.6. UV-VIS Analysis

UV-VIS was utilized so that the optical properties of that were nanoparticles contained within a polymer matrix could be verified.

3. Results and discussion

In order to achieve new and improved qualities that cannot be attained by any one component alone, hybrid composites are materials that blend two or more different types of materials. A particular kind of hybrid composite called PVA-Fe₂O₃-CdZnS contains polyvinyl alcohol (PVA), iron oxide (Fe₂O₃), and cadmium zinc sulphide. (CdZnS). Due to biocompatibility, PVA, a water-soluble polymer, is frequently utilised in the manufacture of fibers, films, and other products. Known for its strong magnetic properties, Fe₂O₃ is a metal oxide that can be used in ferrofluids and magnetic storage systems, among other things. A semiconductor called CdZnS has unusual optical and electrical features. making it practical for uses like photodetectors and solar cells. The PVA-Fe₂O₃-CdZnS hybrid composite is able to demonstrate a variety of distinctive features, including increased mechanical strength, magnetic responsiveness, and photoconductivity. As a result, it can be used in a wide range of applications, such as solar energy harvesting equipment, magnetic drug delivery systems, and biological sensors. Overall, the synthesis of novel materials through the fusion of several components is an increasing trend, and the PVA-Fe₂O₃-CdZnS hybrid composite is an illustration of this.

Field emission scanning electron microscopy (FESEM) was used to confirm the morphology, FESEM images in figure 1a, which show the uniform and agglomeration distribution of Fe_2O_3 . figure 1b show the spherical shaped of CdZnS nanoparticles with some agglomeration, and in figure 1c there is almost no porosity.

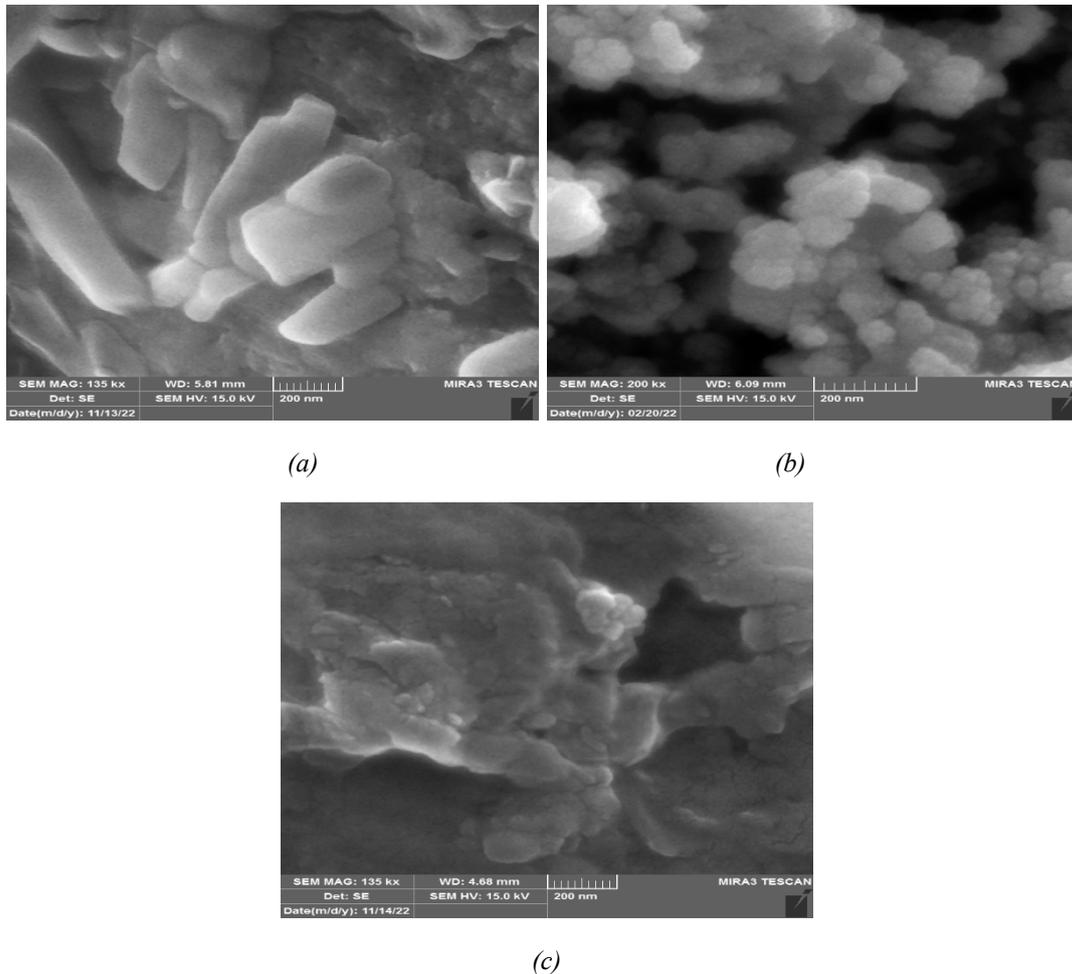


Fig. 1. FESEM images of (a) Fe_2O_3 , (b) CdZnS and (c) $\text{Fe}_2\text{O}_3\text{-CdZnS}$.

The energy dispersive spectroscopy (EDS) technique yielded quantitative results for C, O and Fe at concentrations 60.1 wt.%, 29.5 wt% and 10.4 wt% respectively in the Fe_2O_3 as shown in figure 2, the content of Cd, Zn and S at concentrations 60.6 wt.%, 31.3 wt% and 8.1 wt% respectively in the CdZnS particle as shown in figure 3, also it can be shown that concentration of C, O, Fe, Cd, Zn and S at concentrations 31.8 wt.%, 30.3 wt%, 22.5 wt%, 9.4 wt.%, 4.7 wt% and 1.3 wt% respectively found in $\text{Fe}_2\text{O}_3\text{-CdZnS}$ as shown in figure 4.

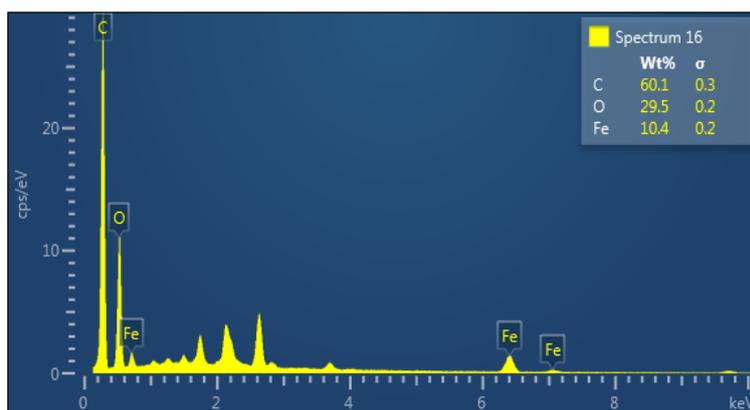


Fig. 2. EDS spectrum for distribution element of Fe_2O_3 .

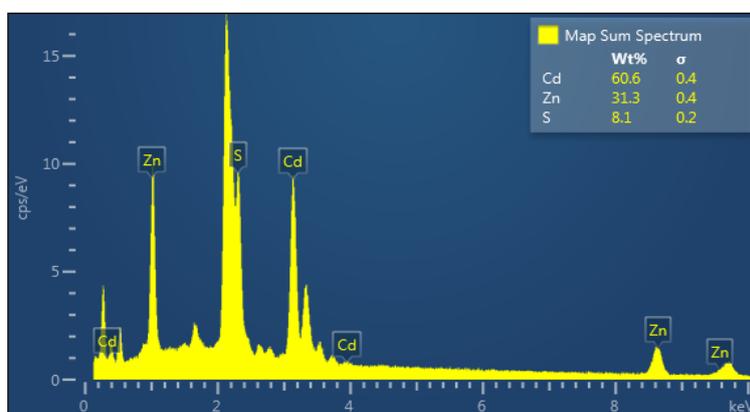


Fig. 3. EDS spectrum for distribution element of $CdZnS$.

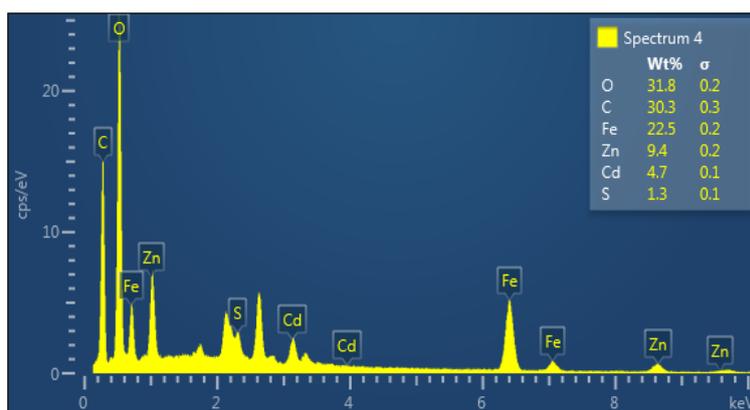
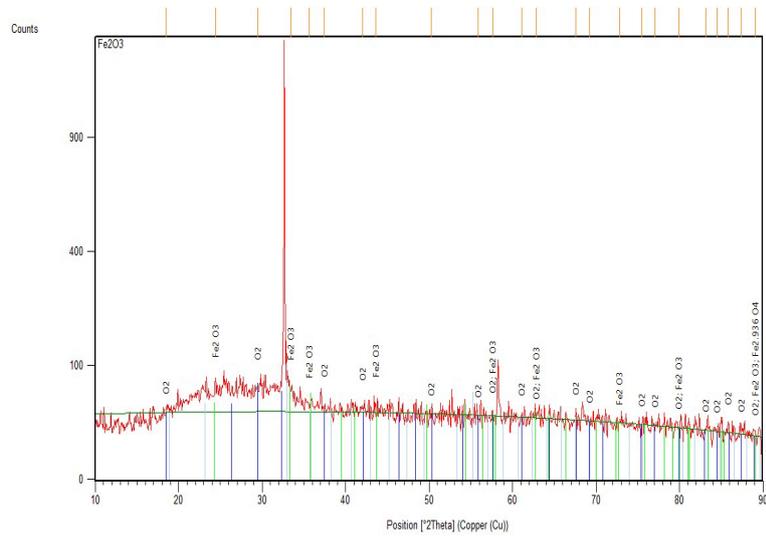
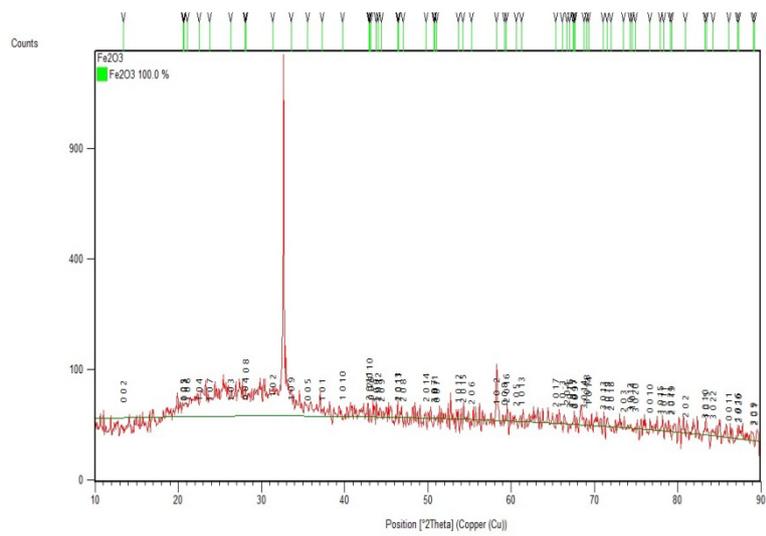


Fig. 4. EDS spectrum for distribution element of Fe_2O_3 - $CdZnS$.

Fig. 5 a , show the X-ray diffraction (XRD) reveals iron oxide II, peaks which revealed (Fe₂O₃) and oxygen (O₂) atom peaks. It was observed that in figure 6 a , the composite's overlay profile revealed Cadmium Sulfide (CdS) and Zinc Sulfide (ZnS) and Zinc (Zn) atom peaks.

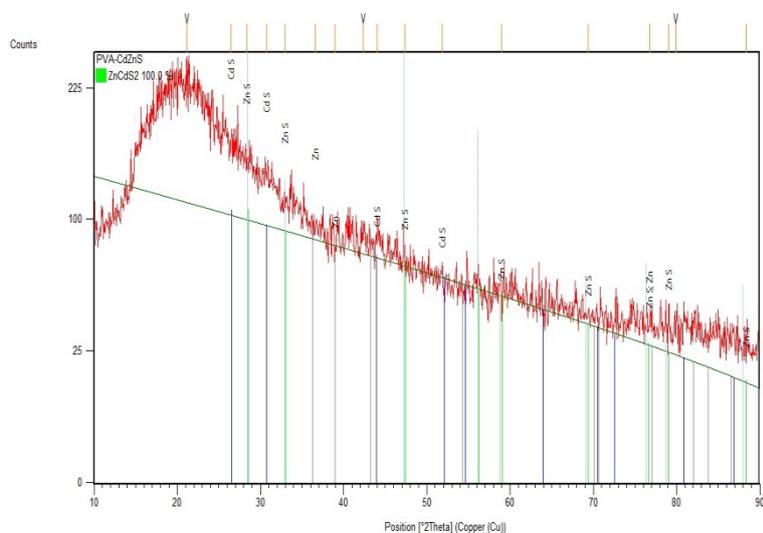


(a)

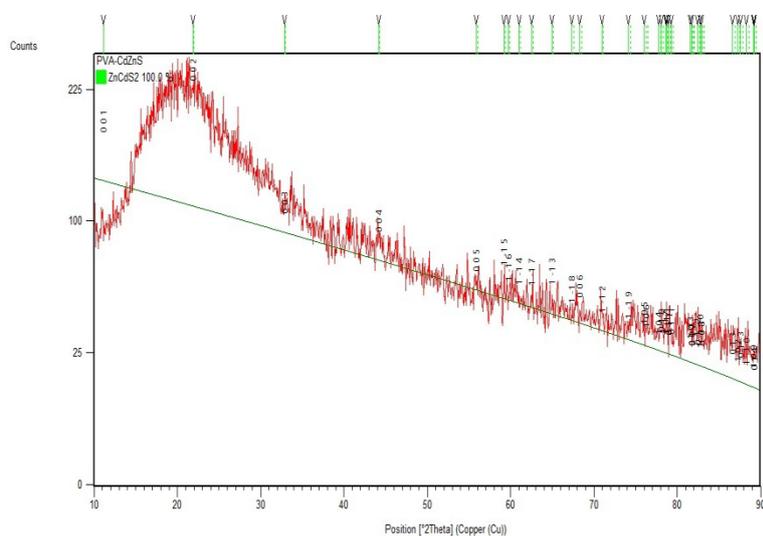


(b)

Fig. 5. X-ray diffraction of the Fe₂O₃ for (a) Chemical compositions, (b) Millers Indices.



(a)



(b)

Fig. 6. X-ray diffraction of the PVA-Fe₂O₃-CdZnS for (a) Chemical compositions, (b) Millers Indices.

It can be shown in Tables 1 and 2, that the average grains of crystalline is 38.417nm of Fe₂O₃ and, 27.267nm of PVA-Fe₂O₃-CdZnS respectively.

Table 1. Inter atomic distance, positions angles, and crystallite for Fe₂O₃.

No.	FWHM [°2Th]	Peak position [°2Th]	Crystallite size [nm]
1	0.295	14.085	27.9
2	0.148	32.682	59.1
3	0.394	52.885	23.0
4	0.148	58.308	65.0
5	0.295	68.508	33.5
6	0.492	83.503	22.0

Table 2. Inter atomic distance, positions angles, and crystallite for PVA-Fe₂O₃-CdZnS.

No.	FWHM [°2Th]	Peak position [°2Th]	Crystallite size [nm]
1	0.394	20.337	20.9
2	0.492	28.804	16.9
3	0.197	39.294	44.6
4	0.689	55.928	13.2
5	0.197	65.595	50
6	0.59	81.13	18

It is noted from infrared spectroscopy (IR) examinations in Figure 7, that the transmittance (T%) decreases with addition of CdZnS nanoparticles. The incorporation of CdZnS nanoparticles inside PVA-Fe₂O₃ matrix may act as scattering centers and leads to the observed decrease in T%.

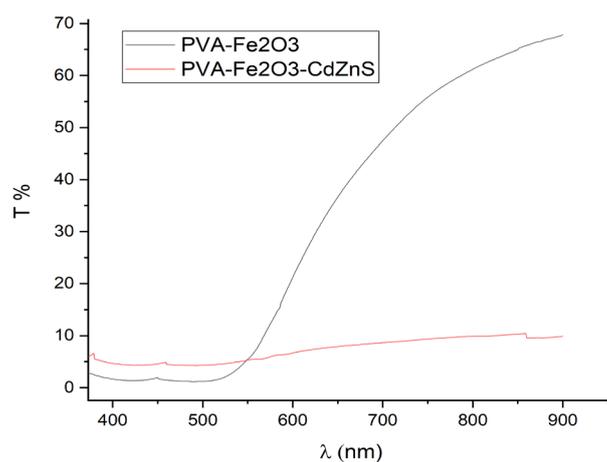


Fig. 7. The transmittance percentage (T%) of the PVA-Fe₂O₃ and PVA-Fe₂O₃-CdZnS

Figure 8, show UV-vis spectra of the PVA-Fe₂O₃ and PVA-Fe₂O₃-CdZnS samples exhibit maximum absorptions in the wavelength in 233nm and 234 nm for PVA-Fe₂O₃ and PVA-Fe₂O₃-CdZnS respectively. It can be noted that the absorption minimum of the materials gradually shift in the wavelength range 897-900 nm for PVA-Fe₂O₃, also that the absorption minimum of PVA-Fe₂O₃-CdZnS in the wavelength range 847-849 nm, The absorption minimum of the materials is observed to gradually shift towards the higher wavelength region of 847-900 nm, which is attributed to the quantum size effect.

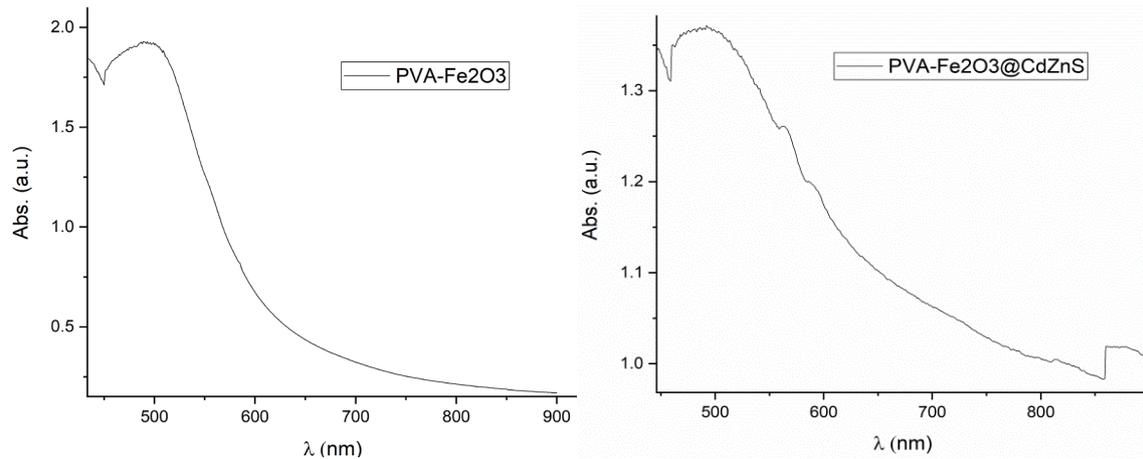


Fig. 8. Absorption spectra of PVA-Fe₂O₃ and PVA-Fe₂O₃-CdZnS.

4. Conclusions

Following conclusions were drawn from the experimental investigation's findings. X-ray diffraction and infrared spectroscopy of PVA-Fe₂O₃-CdZnS composites revealed homogeneous Fe₂O₃ particle dispersion in the PVA matrix at 5% of CdZnS nanoparticles. The preparation of PVA-Fe₂O₃-CdZnS composite was successfully dispersion in the structural, FESEM imaging indicated that both Fe₂O₃ and CdZnS materials are conglomerate to form the Fe₂O₃-CdZnS in the agglomerate particles.

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