Preparation, Characterization and Antibacterial activity of polyaniline-CdS nanocomposites

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Abstract. Polyaniline (PANI) semiconductor polymer and cadmium sulfide nanoparticles (CdS NPs) was prepared by chemical oxidation method and sol-gel method respectively. (10-40) wt% content of CdS NPs was added polyaniline conductive polymer powder to prepared PANi-CdS nanocomposites. The Fourier transform infrared analysis FT-IR spectra of the prepared samples revealed that the effective groups and peak locations referred to the confirming chemical bond are well-matched with the reported literature. The XRD results shows a very wide peak of PANi polymer which indicate to some crystallinity of PANi structure. Whereas the diffraction pattern of CdS NPs shows high crystallinity and polycrystalline nature of hexagonal wurtzite phase. The diffraction pattern of PANi-CdS nanocomposites shows lower peaks intensity than of CdS NPs and improved with the increase of CdS NPs content. The antibacterial activity of PANi and PANi-CdS nanocomposites was estimated by agar well diffusion method against four types of pathogenic bacteria Proteus mirabilis, Staphylococcus aureus, Escherichia coli and Klebsiella pneumonia. It was found that there is a strong antibacterial activity and the greatest effect was for the prepared PANi-CdS NPs 40 wt% and the highest activity was found against Staph. aureus bacteria as well as with the increase of nanoparticles content.

1. Introduction
The nanoscale of inorganic-polymer hybrid materials has attracted considerable attention due to its use in different fields of potential applications such as field-effect transistors and optoelectronic devices [1-3]. The optical, electrical, and dielectric properties of the polymer can be controlled and modified by inorganic nanoscale fillers by exhibited a high surface to volume ratio. New composites materials that have integral behaviors of polymer and inorganic materials are the main goal for the synthesis of these hybrid materials. The most polymers attractive studied is polyaniline due to its relative simplicity and ease in preparation and its good environmental stability and tunable conductivity [4,5]. Various literature has been demonstrated for the synthesis of PANi-nanofillers using CdS, ZnO, Pd, and Au for synthesis nanocomposites [6,7]. The conductive polymers have a lot of attention due to the wide range of their applications. Among the many conductive polymers, Polyaniline (PANI) has been widely studied for its supreme electrical conductivity, low permeability control its conductivity, and the simplicity of chemical oxidation and reduction[8,9]. The mixing of polymers with nanoparticles in their various shapes spherical, rods, tubes, fibers, wires, and other forms where shape and size plays an important role in determining the ability to obtain a homogeneous mix of polymer and nanomaterial with advanced characteristic characteristics compared to mixing materials with microsatellite [10, 11]. Each year in the United States, at least 2 million people become infected with bacteria that are resistant to antibiotics and at least 23,000 people die as a result [12]. Poorer countries with weaker healthcare systems are more affected [13]. In present work preparation, characterization and...
antibacterial activity of PANi and PANi-CdS nanocomposites at (10-40) wt% of CdS NPs content were investigated and this work combines microbiology and nanotechnology, perform probable advances in the formulation of a new kind of antibacterial. Other paragraphs are indented.

2. Experimental details

2.1. Preparation of Polyaniline (PANI) polymer
Polyaniline conductive polymer was prepared by polymerizing the aniline in acidic medium by chemical oxidative polymerization method. Where, 5 ml of aniline was dissolved in 100 ml of 1.0 M hydrochloric acid HCl under continuous stirring for 30 minutes at room temperature. 15 g of ammonium peroxidisulphate (APS) was dissolved in 50 mL of 1.0 M hydrochloric acid HCl under continuous stirring for 30 minutes at room temperature, then it is placed inside a separating funnel. The solution of alanine/hydrochloric acid is placed in a three-neck beaker at ≈ 0 °C by using ice. The three-neck beaker connected to a thermometer to measure the temperature of the chemical reaction, condenser, and neck connected to a separating funnel, which contains the oxidizing agent (APS) (see Figure 3-1). The three-neck beaker is placed on a magnetic stirrer, and the oxidizing agent (APS) is added dropwise from the separating funnel and the temperature is fixed at ≈ 0 oC by placing pieces of ice. After the oxidizing agent has been completely dropped, the ice was removed. The solution is kept under constant stirring for 24 hours at room temperature. Then a dark green precipitate solution was obtained and this precipitate is polyaniline. The precipitate is filtered and washed using a Buchner a period of 24, after which the polyaniline Emeraldine salt PANI-ES is obtained in the form of an agglomerate powder. The prepared PANi.ES was reduced and treated it by ammonia hydroxide (NH4OH) for 15 min to get undoped polyaniline E.B (emeraldine base form). The polyaniline E.B was washed with methanol to remove the oligomers and the residual was dried in oven at 60 oC for 24 h and ground in pestle mortar to get an polyaniline E.B in powder form.

2.2. Preparation of (CdS) nanoparticles
CdS nanoparticles powder was synthesized by a sol–gel process, Cadmium acetate (CH3COO)2H2O and thiourea (CH4N2S ) were used as a source of Cd and S respectively. 3 g of Cadmium acetate and 2 g of thiourea (CH4N2S) was added together into beaker 40 ml of methanol and mixture was stirred continuously forcibly at 60 oC for 1 h. A gelatinous substance will appear. Then the heating was stopped with continues stirring until get a yellow powder. The powder is then dried at 300 °C for 30 minutes until we get monocrystalline CdS.

2.3. Synthesis of PANi–CdS nanocomposite powder
PANi–CdS nanocomposites powder were prepared by mixing of CdS nanoparticles in a weight percentage (10–40) wt% with PANi (EB) matrix.

2.4. Antibacterial Activity of PANi–CdS nanocomposites
The antimicrobial activity of CdS nanoparticles were evaluate against four different types of clinical bacteria Proteus mirabilis, Staphylococcus aureus, Escherichia coli and Klebsiella pneumonia using agar well diffusion method. Standardized suspension of each tested bacteria (1.5x108 cfu/mL) by Dense Check standard (0.5) was swabbed separately onto Mueller Hinton agar plates using sterile cotton swabs. The agar was punched with sterilized corn borer 9mm and 100μl from each the PANi-CdS nanocomposites were added into the four holes for different CdS NPs content and incubated for 24 hours at 37°C, after incubating, the inhibition zones were measured in millimeter diameter.

2.5. Characterization techniques
Crystal structure studies were carried out using a DX-2700 x-ray diffractometer (XRD). The XRD patterns were recorded in the2 θ range of 10–80 using CuKa radiation λ =1.5406 Å. Fourier Transform Infrared (FTIR) spectroscopy of PANi, CdS NPs and PANi–CdS nanocomposites were studied in the
frequency range of 400–4000 cm-1 using Shimadzu FTIR-8400S. The morphological study of the prepared samples was carried out using Field Emission Scanning Electron Microscopy (FESEM) FEL, Quanta450, Czech operating at 20 kV.

3. Results and discussion

3.1 Fourier transform infrared analysis

Figure (1 a, b and c) display the PANi (EB) CdS NPs, and PANi-CdS nanocomposites FT-IR spectra at active groups sites the peak locations related to the corresponding chemical bonds which compared to previous and reported studies. PANi (EB), FT-IR spectra shows that transmittance peaks was observed at wavenumbers of (1560, 1457,1287 and 818.75) cm-1 C-H, 1156.20 cm-1 (N=Q=N modes, Q=NHB modes and B-NH-B), 1303.45 cm-1 (C-N benzenoid mode stretch), 1374 cm-1 (C-N aromatic rings stretching), 1494.5 cm-1 (C = C stretching mode), 1494.1 cm-1 of (C = C stretching mode of benzoid rings), 1374 cm-1 (C-N) [14, 15] (CH2 stretching). Whereas at the wave number 818.75 cm-1 refers to C = N imino quinone; the C-N (stretching motions in main aromatic amine) equate to 1225.47 cm-1 [16, 17]. A 3614 cm-1 transmitting band that is allocated to the O-H water vibration band [18]. The peaks of 3837, 3738, 2929, 1165 and 829 cm-1 refer to the stretching motions of N=quinoid=N. The peaks seen at 3420, 2365 and 683 cm-1 are methanol characteristics [19, 20]. The FT-IR spectrum of CdS NPs is illustrated in Fig. (1b). The band at 3827cm-1 is attributed to OH extending water molecular vibrations. At 2066.68 and 1632.40 cm-1, the bending vibrations of water molecules is C-C stretching, CdS NPs display two asymmetric stretches and C–H related, 3310.85 and 3123.55 cm-1 [21]. At 773.92 cm-1, medium to heavy bands are allocated to stretch CdS [22]. Figure (1-c) display a decrease in FTIR PANI-CdS nanocomposite (40 wt%) strength of polymer band due to amine and imine groups. This may be attributed to the existence of numerous polymer oxidation states. These shifts often occur in aromatic peaks that extend in C–H mode of vibration, consistent with the values usually observed for PANi (EB), where the frequency of C–H absorption has changed by approximately 49cm-1 from its well-reported value of approximately 3,000 cm-1 and is found at 3029.73 cm-1, an additional shift in PANi (EB) from 2980.01 cm-1. The FT-IR values of PANi (EB), CdS and (PANI-CdS) indicates that almost all absorption values for CdS have changed to higher and lower values. This demonstrates that nano-sized CdS has influenced the composite absorption phenomenon in the IR region.
3.2. Structure analysis

Figures (2 a,b,c) indicates the XRD patterns of Polyaniline powder and cadmium sulfide (CdS) nanoparticles and PANi-CdS nanocomposites. PANi XRD pattern (fig.1a) reveals a very wide peak at 2θ=25.57° which correlates to (110) PANi plane indicates to some crystallinity and related to the repetition of benzenoid and quinoid rings in PANi chains [23, 24]. The wide peak of PANi XRD pattern indicates that it has certain crystallinity, which can be due to the repetition of PANi benzenoid and quinoid rings in PANi chains [25]. Figure (2b) illustrate the x-ray diffraction pattern of CdS NPs which was polycrystalline structure nature, and identified by the presence of diffraction peaks at diffraction angles 2θ of 25.021°, 26.489°, 28.046°, 43.8°, 47.8° and 51.9° that were belonging to the (100), (002), (101), (110), (103) and (112) planes. All peaks of diffraction can be indexed to the hexagonal structure CdS wurtzite phase according to the standard (JCPDS No. 77-2306). The XRD patterns of PANi–CdS (10–40) wt% nanocomposites shows the characteristic of crystalline CdS peaks for the hexagonal structure, wurtzite phase as shown in figure (2 c). This means that CdS crystal is not modified by the existence of PANi. The diffraction peaks of CdS nanoparticle and PANi–CdS composites (10–40) wt% in XRD patterns were hexagonally wurzite CdS structured, which was compatible with the CdS standard values for CdS in JCPDS card. The diffraction peak for the preferential orientation (002) of CdS nanoparticles were observed to move to lower 2θ values (left shift) in PANi–CdS 10 wt% nanocomposite due to the high disorder and low crystallinity of the PANi structure. Then the diffraction angles were right shifted to high 2θ with the increase of CdS nanoparticles content, where the diffraction intensity was increased and the crystallinity of the nanocomposies was enhanced by reduced the lattice defects with the increase of high crystallinity of CdS nanoparticles. Also the lattice constants for the preferential orientations of (002) plane, were found to be a = b = 4.106 Å and c = 6.724 Å for CdS NPs and increased to b = 4.165 Å, c = 6.750Å for PANi-CdS10 wt% nanocomposite by the high lattice distortion and a large number of defects caused by the disorder atom arrangement lead to the largest lattice constant as shown in Tables (1). For (PANi–CdS) (20–40) wt% nanocomposites, a slightly decreases of the lattice constants was observed to be a= 4.1204 Å and b= 6.7314 Å for PANi-CdS 40 wt% nanocomposite. The low defect density made the atoms orderly arranged in the crystal that reduces lattice constant, indicated by the right-shift of the peaks. Furthermore, with the increase of CdS NPs content the defects in the samples is slightly decreased [26] Table (1). These results were in good agreement with literature [27, 28]. The existence of such an interaction can also be examined by the crystal aspect ratio described as the ratio of crystallite size in the (002) plane. The average crystallite sizes Dav of CdS and PANi–CdS (10–40) wt% nanocomposites for the preferential orientation along (002) plane are determined by the Scherrer equation [29]:

$$D_{av} = \frac{k\lambda}{\beta \cos \theta}$$  \hspace{1cm} (1)
Where \( k \) is the shape factor and \( \lambda \) is the x-ray wavelength and \( \beta \) is the beaks full width at half maximum (FWHM). The crystallite sizes of CdS for (002) plane was 14.266 nm and increases in general in the PANi-CdS Nanocomposites to be 22.624 and 23.962 for (10 and 20) wt% of CdS NPs content in PANi-CdS Nanocomposites then it slightly decreases to 20.598 nm for 40 wt% of CdS NPs content. The increase and then decrease in the crystallite sizes of CdS in PANi–CdS (10–40 wt%) nanocomposites shows that the crystallinity of CdS is disturbed by the adsorption of PANi molecular chains on the surface of CdS nanoparticles. These results indicate that the interaction between the CdS NPs and PANi molecular chains due to the adsorption of PANi molecular chains on the surface of the CdS [28, 22].
Figure 2: X-ray diffraction patterns of (a) PANi, (b) CdS NPs and (c) PANi–CdS nanocomposites.

Table 1. The Crystallite Size, Dislocation Density, Fine Strain, PANi EB Strain, and (PANi-CdS) (10-40%) Nanocomposites for (002) Plane.

| Material          | Crystallite Size (nm) | $\delta \times 10^{15}$ m$^2$ | Micro-Strain $\times 10^{-3}$ |
|-------------------|-----------------------|--------------------------------|-------------------------------|
| CdS               | 14.266                | 4.913                          | 2.538                         |
| PANi              |                       |                                |                               |
| PANi -CdS (10 wt%)| 22.624                | 1.954                          | 1.60                          |
| PANi -CdS (20 wt%)| 23.962                | 1.742                          | 1.511                         |
| PANi -CdS (30 wt%)| 20.458                | 2.398                          | 1.770                         |
| PANi -CdS (40 wt%)| 20.598                | 2.357                          | 1.758                         |

3.3. Morphological Analysis

The field emission scanning electron microscope (FESEM) images of PANi and PANi-CdS nanocomposites powder, revealed a fibrous structure with a wide number of pores and fiber holes in the polyaniline. Where it is found that all prepared samples were well uniform and compatible in general and the key color of the samples is the polyaniline green color [30, 31]. It has been noticed that PANi can keep the (CdS) particles from clumping to some degree. The above (PANi- CdS) overlapping is what allows it possible for electrons to transition from PANI to CdS. The micrographs of (PANi–CdS) (10–40) wt% nanocomposites revealed that the composites has mixture of agglomerated and non-agglomerated particles.
and evenly CdS NPs are dispersed inside the PANi-matrix (Fig. 3 a, b, c, and d). It was siderites that the CdS particles were trapped in the nanostructure. These results were in good agreement with the literature [32].

![Figure 3. FESEM image of a. PANi and PANi–CdS (10-40)wt% nanocomposites b, c, d and e respectively.](image)

3.4 Antibacterial activity of a. PANi and PANi–CdS nanocomposites

The antibacterial activity of PANi (EB) and PANi-CdS nanocomposites with different content of CdS NPs (10-40) wt% were estimated using the agar well diffusion method against four bacteria types of bacterial pathogens of Proteus mirabilis, Staphylococcus aureus, Escherichia coli and Klebsiella pneumonia. The activity of (PANi-CdS wt%) nanocomposites against pathogenic bacteria resulted in clear inhibition zone. Through figures (4) (a,b,c and d) it is proved that (PANi-CdS) nanocomposites were able to inhibit growth of bacterial pathogens and varied for different CdS content in PANi matrix. Table (2) shows the inhibition zones diameter for PANiEB and PANi-CdS (10-40)wt% nanocomposites as antibacteria. It was developed to inhibit the growth of pathogenic bacteria, as it was observed that with present and an increase in the CdS NPs content in the PANi matrix would lead to an increased inhibition of different types of
bacteria. The activity of PANiEB as antibacterial against P. mirabilis, exhibited inhibition zone diameter about (19 mm) and its increased with the present and increase of CdS NPs content in PANi matrix, the largest inhibition diameter is at 40 wt% of CdS NPs, where its diameter reaches (29 mm). This means that the nanoparticles with an increase are improved the inhibition and killing the bacteria. Also, the activity of PANi and PANi-CdS nanocomposites against Staphylococcus aureus bacteria were evaluated. The inhibition zone diameter was (29 mm) for PANi sample and the inhibition zones were increased with the increasing of CdS NPs content in PANi matrix to be 37 mm at 40 wt% of CdS NPs content. The antibacterial activity of PANi and PANi-CdS nanocomposites against E.coli bacteria was evaluated and the inhibition zone diameter was (22) mm for PANi and the inhibition zone diameter was increased with present of CdS NPs content in the PANi matrix. The high activity was found at high CdS content 40 wt% which about (28 mm). The same behavior of antibacterial activity was observed for PANi and PANi-CdS nanocomposites Klebsiella pneumoniae, where the inhibition diameter is (21 mm) for PANi sample and it increased with present of CdS NPs content in the PANi matrix. The inhibition zone diameter at 40% CdS NPs content is about (26 mm). All results of inhibition zones diameters were illustrated in Table (2). From the Table it can be notice that the prepared polymer PANi and PANi-CdS nanocomposites exhibits a strong activity against different types of bacteria where all zones of inhibition are larger than 16 mm. The strong antibacterial activity when the zone of inhibition is larger than 16 mm and the moderate activity the zone of inhibition lies in the range of 10–16 mm, while for weak activity it is reported to be less than 10 mm. These results are consistent with the literature. The antibacterial mechanisms can be attributed the particles of PANiEB and (PANi-CdS wt%) can anchor to the bacterial cell wall and infiltrate it and lead to damage to the cell membrane and cellular content leakage, also can penetrate inside to microbial cells, and they can interact with cellular structures and biomolecules such as proteins, enzymes, lipids, and DNA. Also, PANi-CdS nanocomposites get attached to the cell membrane and shrink the respiration of the bacterial cell which eventually causes the death of the cell as depicted [33, 34]. It can be interpreting the activity of CdS nanoparticle, maybe the composition of the cell wall differential between the Gram-positive and Gram-negative bacteria plays a part in the forming of the region. In Gram-positive bacteria such as (Staphylococcus), the cell wall forms of a deep layer of a membrane composed of linear polysaccharide chains, while Gram-negative bacteria such as (P. mirabilis, K. pneumonia and E. Coli) have a thin layer of membrane that shows that nanoparticles have a wide surface area sufficient for interactions that improve the antibacterial activity [34, 23, 26].
Figure 4. The Estimated Inhibition Zones of PANi and PANi-CdS Nanocomposites (10-40) wt% Against (a) *P. Mirabilis*, (b) *Staph. Aureus*, (c) *E. coli* and (d) *K. Pneumoniae* Bacteria.

Table 2. Inhibition Zone of PANi and (PANi-CdS wt%) Nanostructures as Antibacterial Agents in Millimeter.

| Kind of used Bacteria | Concentration of PANi and (PANi-CdS NPs wt %) /inhibition zone (mm) |
|-----------------------|---------------------------------------------------------------------|
|                        | PANi (EB)               | PANi-CdS 10 wt% | PANi-CdS 20 wt% | PANi-CdS 30 wt% | PANi-CdS 40 wt% |
| *P. mirabilis*         | 19                     | 23             | 0               | 25             | 28             |
| *Staph. aureus*        | 29                     | 31             | 33              | 36             | 37             |
| *E. coli*              | 22                     | 23             | 24              | 26             | 28             |
| *K. pneumoniae*        | 21                     | 23             | 23              | 25             | 26             |

4. Conclusions
It has been shown that the films PANi–CdS nocomposites were synthesized by spin coating technique. A change in the value of lattice parameter of CdS in the PANi–CdS composite was observed which also indicated the presence of interaction between CdS particles and PANi matrix. From FTIR spectra it is revealed that the characteristic absorption peaks of PANi(EB) shifted by significant amount into PANi–CdS nanocomposite, which indicate the different interfacial interactions between the CdS NPs and the PANi matrix. The crystal structure results of PANi (EB) shows some crystallinity whereas the diffraction pattern of CdS NPs exhibits polycrystalline structure nature hexagonal wurzite phase. Also the XRD pattern of PANi–CdS (10–40) wt% nanocomposites shows the characteristic of crystalline CdS peaks.
Morphological study revealed the prepared CdS nanoparticles their uniform dispersion in PANi matrix. It was found that there is a strong antibacterial activity and the greatest effect was for the prepared PANi-CdS NPs 40 wt% and the highest activity was found against (Staph. aureus) bacteria as well as with the increase of nanoparticles content. This work combines microbiology and nanotechnology, perform probable advances in the formulation of a new kind of antibacterial.

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