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Synthesis and PVP Coating Effects on Cadmium Ferrites for Antibacterial Applications

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Abstract

This work focuses on the effect of coating with polyvinylpyrrolidone on the structure and morphology of synthesized powdered cadmium ferrites through the microwave-assisted combustion method. Antibacterial activities test of Cd-ferrite before and after coating with PVP for Escherichia coli and Staphylococcus aureus were also studied. Structure and impurity phases characterizations, surface morphology with chemical composition analysis, as well as lattice vibrations determined by XRD analysis, field emission scanning electron microscopy with energy dispersive X-ray spectroscopy, and foruier transform, exhibited spinel structure with crystallite size decreasing with coating with polyvinylpyrrolidone from 21.23nm to 19.71nm. The images recorded by field emission- scanning electron microscopy showed that polyvinylpyrrolidone plays a very important role in preventing Cd-ferrite agglomeration. The presence of Fe-O bonds from Cd-ferrite was observed in the FT-IR spectra. Antibacterial activity was found to be enhanced with the coating of cd-ferrite.

Keywords: Cd-ferrite, coating, (poly (vinyl pyrrolidone)), microwave-assisted combustion**,** antibacterial activity

تصنيع ودراسة تاثير الطلاء على التركيب والتشكلية لفرايت الكاديميوم للتطبيقات المضادة للبكتريا

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الخلاصة

تم دراسة تأثير الطلاء بمادة (البولي (فينيل بيروليدون، PVP)) على البنية والتشكل لمسحوق فرايت الكادميوم المركب من خلال طريق الاحتراق بمساعدة الميكروويف. كما تمت دراسة النشاط المضاد لبكتيريا الإشريكية القولونية والمكورات العنقودية الذهبية لـ Cd–ferrite قبل وبعد التغطية بـ PVP. تشير خصائص التركيبية ونقاوة الطور وتشكلية السطح مع تحليل العناصر الكيميائية بالإضافة إلى اهتزازات الشبيكة التي تم تحديدها من خلال تحليلات XRD، والمجهر الإلكتروني الماسح للانبعاث مع التحليل الطيفي للأشعة السينية المشتتة من الطاقة (EDX) وتحويل فوريير، إلى تشكل بنية خلية وحدة الإسبنيل المكعب مع انخفاض متوسط حجم الجسيمات بوجود PVP من 21.23 نانومتر إلى 19.71 نانومتر. بينما تُظهر صور FE–SEM أن

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PVP يلعب دورًا حرجا في منع تكتل فرايت الكادميوم. وقد لوحظ وجود روابط Fe–O من فرايت الكادميوم
في أطياف FT–IR. تم العثور على نشاط مضاد لبكتريا يتم تعزيزه من خلال وضع غطاء لفرايت الكادميوم

1. Introduction

The widespread usage of nanotechnology in business, agriculture, and biological sciences during the twenty-first century has earned it the moniker "golden age" of research. Nanomaterials have been gathering great attention due to their wide applications, including ‘‘Nanomedicine’’ as well as their properties in different fields that lead to unique characteristics depending on their overall size, shape, composition and distribution. In addition to metal and metal oxide nanoparticles, ferrite nanoparticles are attracting interest because of their superparamagnetic characteristics and high surface area to volume ratio completely distinct from those of their bulk counterparts [1,2]. Ferrites are magnetic oxides. According to their crystal structure, ferrites can be classified as hexagonal, spinel and garner[3]. Recently, spinel ferrite nanoparticles have been researched for their potential as photocatalysts in wastewater treatment and antibacterial activities, potentially reducing the presence of bacterial microbes that are found in water and flood water, such as Staphylococcus aureus and Escherichia coli, which are responsible for many skin diseases [4]. Among different types of ferrites, spinel ferrites of the composition MFe_2O_4 [M can be Mn, Ni, Zn, Co, etc.) are widely used for emerging biomedical applications because of their biocompatibility, chemical stability, and reasonable cost compared to other magnetic nanoparticles[5,6]. Cadmium ferrite has a typical spinel structure, with cadmium ions occupying the tetrahedral sites[7]. Cadmium is often considered a toxic heavy metal. It has recently received much attention as a result of its numerous applications in various fields. Over the past century, several exposures to cadmium have been observed; cadmium is found in the environment as a result of numerous human activities. In humans, cadmium can cause a range of harmful effects, including testicular damage, renal and hepatic dysfunction, osteomalacia, pulmonary oedema and damage to the adrenals and hemopoietic system. Occupational or environmental cadmium exposure has been related to breast, lung, pancreas, prostate, urinary bladder, and nasopharynx cancers [8]. The literature on cadmium nanoparticles' clinical and biomedical applications is extensive. They also have a wide range of antibacterial activity against various pathogens[9]. Several studies have been published using concepts regarding the interaction of nanostructured materials and microorganisms, studying the possible effects of this contact[10,11]. A protein in the bacterial cell wall attracts cations, which causes the creation of insoluble metal proteinate and consequent bacterial death. This mechanism is responsible for the antibacterial activity of spinel-type ferrites [12]. Antibacterial agents can be classified as: organic and inorganic according to their chemical composition. However, the inorganic antibacterial agents have received more recognition in the antibacterial product market because the organic antibacterial agents have disadvantages, such as low heat resistance, short life expectancy and high decomposability, causing limitations in their applications [4]. On the other hand the shape, size, and surface charge of the nanoparticles, among other physico-chemical properties, may impact their biological activity and internalization, such as antibacterial potential. Smooth-surfaced nanoparticles, as an example, have a higher risk of interacting with a bacterial cell wall. Additionally, compared to larger spherical nanoparticles, smaller spherical nanoparticles have higher antibacterial activity as a result. Synthesized nanoparticles need some modification to modify their antibacterial activity before implementation for biomedical applications[13,14,4]. Polyvinylpyrrolidone can be employed as a surface coating agent to preventing the aggregation of NPs and control the shape and average particle size[15]. Polyvinylpyrrolidone is an inert, and water-soluble polymer. It has been gaining popularity as a result of its widespread use in the synthesis of nanostructures. Because of its unique colloidal and complexing properties, it has been widely used in medicine and pharmaceuticals as a defensive colloid, viscosity enhancer, solubility promoter, granulating/tabletting agent, and film-forming or coating material[16]. The present investigation focuses on the effect of PVP as a coating on the structure and morphological of Cd-ferrite and to examine their antibacterial activity.

2. Materials and Methods

2.1. Synthesis of Cd-Ferrite

Cd-ferrite was synthesized through a microwave-assisted combustion route. It is an environment-friendly route [17]. An aqueous solution of cadmium ($Cd(NO_3)_2 \cdot 6H_2O$) and ferric ($Fe(NO_3)_3 \cdot 9H_2O$) nitrate salts as starting precursors with a molar ratio 1:2 was prepared by dissolving them in a suitable quantity of deionized water. The precursor of the materials was allowed to age for 20 minutes under constant stirring at room temperature using a magnetic stirrer. An amount of glycine ($C_2H_5NO_2$) was added to the metal solution under continuous stirring for approximately 20 min. The final solution was transferred into microwave oven with an 800-watt microwave power to evaporate, and release gases for the solution until the mixture became solid. The product was washed in de-ionized water several times, dried at 80 ^{o}C for two hours and then the obtained product was ground to get a fine powder of Cd-ferrite

2.2. Coating Cd- ferrite with polyvinylpyrrolidone (PVP)

In this step, aqueous solution of polyvinylpyrrolidone was prepared by dissolving 3g of polyvinylpyrrolidone in 100 ml of deionized water and then stirred at 40^{o}C. After this, 1 gram of Cd-ferrite that had been prepared, as previously mentioned, was added to the above transparent solution with continuous stirring at the same temperature until it dried. Finally, a scraper blade was used to obtain a polymer- coated Cd- ferrite powder**.**

2.3. Antibacterial Test

The bacterial activity, using the well diffusion method, of the synthesized Cd-ferrite powder and the PVP-coated Cd-ferrite was tested on gram-negative bacteria Escherichia coli and Staphylococcus aureus supplied from Kofa University laboratories. 38 mg of Mueller-Hinton agar was dissolved in one liter of distilled water. To fully dissolve Muelle-Hinton agar, heat was applied in a stirrer at 100°C and brought to a boil for one minute, then put into an autoclave at 121°C for (15 min.). To achieve uniform depth, the cooled agar was poured into sterile Petri dishes (90ml) with a level, horizontal surface and allowed to solidify. A sterile cotton swab with the reference bacterial strain was streaked across the agar surface of each plate. The plates were allowed to stand for 30 min then were incubated for 24 hours at 37°C and inhibition zone was observed after this period.

3. Results and Discussion

3.1. X- Ray Diffraction Spectroscopy

X-ray diffractometer(Shimadzu diffractometer model XRD 6000) using CuK lines was used to analyze the structure and impurity phases of both synthesised Cd-ferrite powder and PVP-coated Cd-ferrite and to determine the nanoparticles parameters, such as crystallite size and crystalline phase, which influence their properties and biological activity [6]. The XRD patterns are exhibited in Figure1.

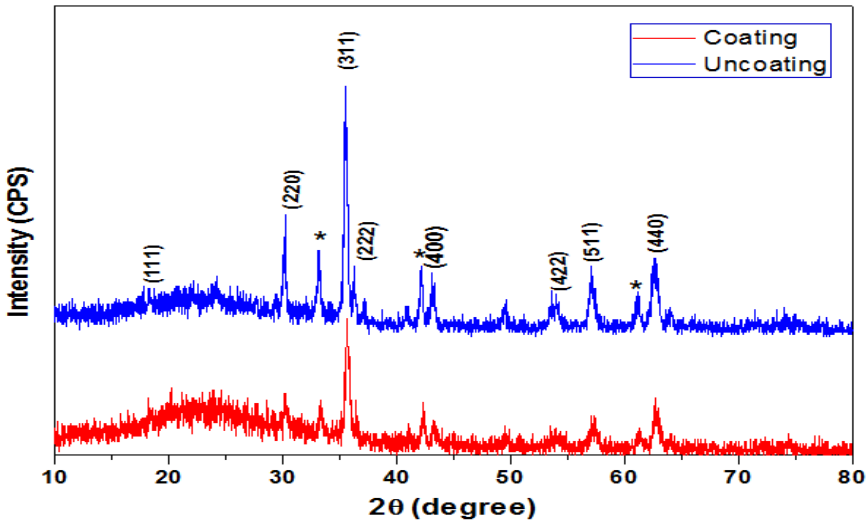

Figure 1: X-ray diffraction patterns of Cd-ferrite sample before and after coating

X-ray diffraction pattern confirmed the formation of the Cd-ferrite powder and PVP-coated Cd-ferrite, which have a phase cubic spinel crystal structure. The diffraction peaks indexed as (220), (311), (222), (400), (422), (511), and (440) well matched the standard pattern of Cd-ferrite (JCPDS file No: 00-005-0674). Impurity peaks (e.g. CdO) are marked with an (*). It was noted that the positions of the peaks of both XRDs were the same, which means that no significant structural variations occurred after coating the Cd-ferrite surface with PVP; the diffraction peaks intensity for PVP-coated Cd-ferrite were less than those for the Cd-ferrite powder. This confirms that the PVP significantly improved the degree of crystallinity of these products.

Crystallite size (D) for the two ferrites was determined from the XRD data using Debye-Scherer equation [18]. The calculated values are summarized in Table 1. Lattice constants (a) of the respective ferrites were also obtained by Equation 1 [19] and are also listed in the Table 1.

$$a = \frac{n\lambda}{2sin\theta}\sqrt{(h^2 + k^2 + l^2)}\ \text{Å} \qquad \ldots\ldots 1$$

where λ represents the wavelength of the X-ray used (λ = 1.540Å), θ_{hkl} is the diffraction angle, (hkl) are Miller indices and n = 1 is the order of reflection for cubic structure.

Table 1: Structure parameters of Cd-nanoferrite and coating Cd-nanoferrite with PVP.

samples	2θ (deg.)	d (Å)	FWHM (deg.)	a (Å)	D (nm)
Uncoating	35.5679	2.5220	0.3723	8.3645	21.23
Coating	35.7305	2.5109	0.4000	8.3277	19.71

It is evident from Table 1 that the crystallite size decreased for the coated Cd-ferrite. The ionic properties in the polymer that binds the molecules together could be the cause of this decrease [20]. The crystallite size (D) and lattice constant (a) of the NPs are directly related, where increasing crystallite size led to a increase or decrease in value of lattice constant [3]. In this paper, the value of the lattice constant decreased from 8.3645 Å to 8.3277 Å as values of crystallite size decreased from 21.23nm to 19.71nm.

3.2. Morphological Analysis

The FE-SEM images of Cd-ferrite sample before and after coating with polyvinylpyrrolidone are shown in Figure 2.

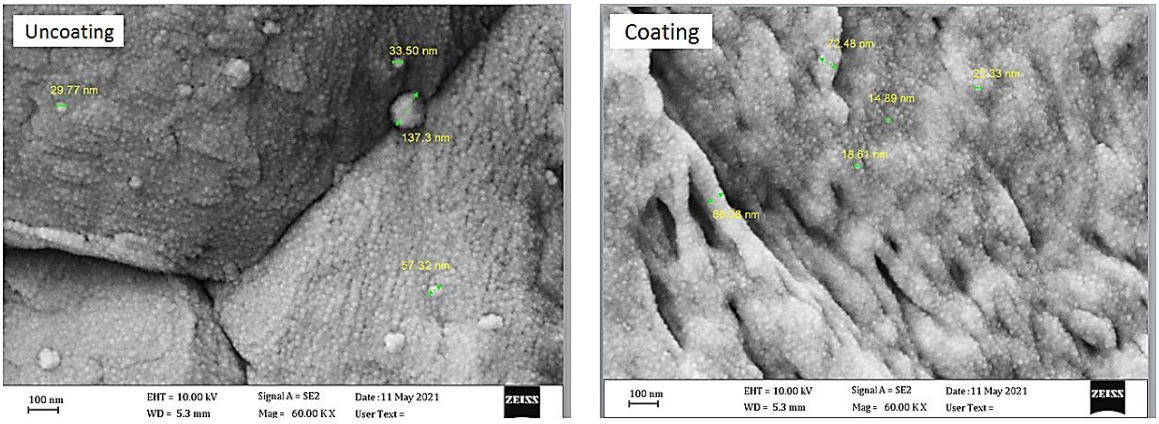

Figure 2: FE-SEM images for Cd-ferrite sample before and after coating

FE-SEM images shows very strong agglomeration in the Cd-ferrite sample. The agglomeration is because of the Van der Waals force between the particles[10]. Due to the high surface energy and high specific surface area of small size nanoparticles, there is a strong adhesion between particles that resulted agglomeration, for the Cd-ferrite without PVP, as observed from FE-SEM images. Although some agglomeration is still seen for the Cd-ferrite with PVP because of the inter-particle magnetic interaction, the PVP lowered the agglomeration. This is consistent with previous work that was reported by Yuan et al. for other ferrites [21]. FE-SEM images confirm the successful PVP coating, which was immobilized on the Cd-ferrite surface.

3.3. *Vibrational spectroscopy*

FTIR spectroscopy results of the as-synthesis samples are presented in Figure 3.

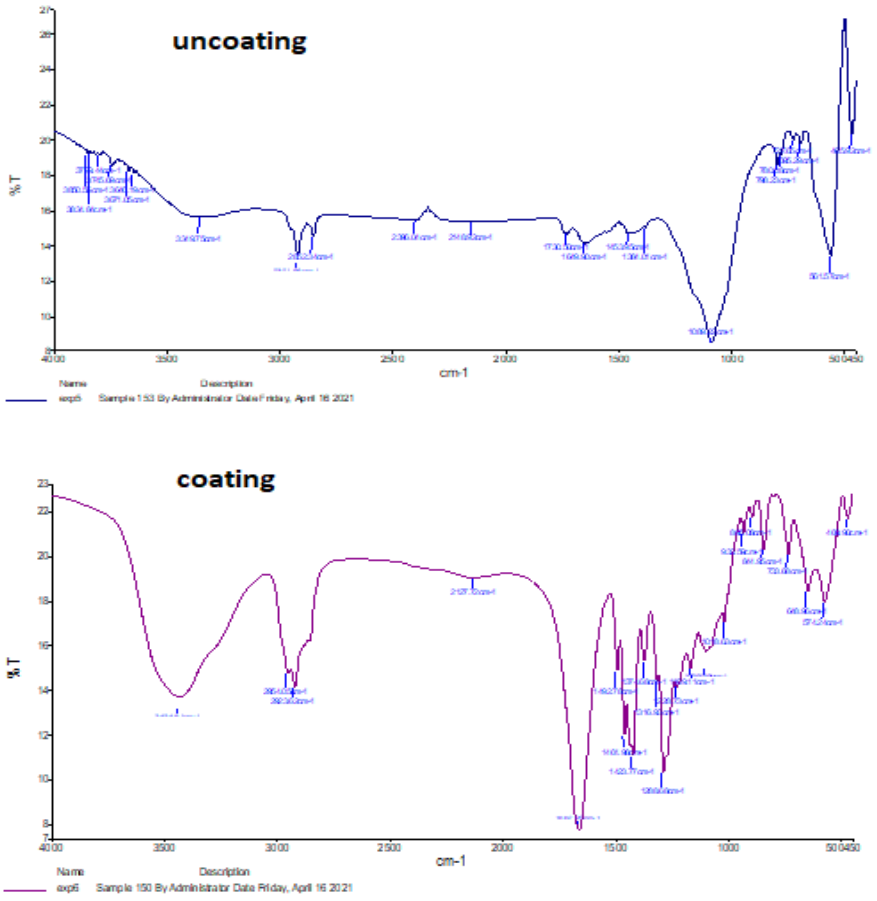

Figure 3: The FTIR spectrum of Cd-ferrite before and after coating with PVP.

It can be seen that the vibrations of the $M_{tetrahedral}$ resonance with O and lower absorption bonds are responsible for the peaks at (561.57 and 465.92) cm^{-1}, respectively. The peaks at 1089.22 cm^{-1} for synthesized Cd-ferrite and 1098.98 cm^{-1} after coating Cd- ferrite with PVP are attributed to the C-C stretching, respectively. The group (C=O) absorbs energy at 1649.90 cm^{-1} for synthesized Cd-ferrite and at 1662.95 cm^{-1} after coating Cd-ferrite with PVP. The broadest peaks observed at 3349.75 cm^{-1} for synthesized Cd-ferrite and at 3434.61 cm^{-1} after coating Cd-ferrite with PVP are due to an O-H group. It is confirmed that the PVP compound is present in Cd-ferrite by the presence of the functional groups C=O, C-H, C-C, C-N and O-H.

3.4 . Antibacterial activities of Cd-ferrite before and after coating with PVP

The antibacterial action of ferrite nanopowders may be possible by numerous mechanisms. It has been suggested that ferrite powder bind to the microorganisms membranes, prolonging the lag phase of the growth cycle and increasing the generation time of the organisms so that it takes each organism more time to complete cell division [12]. The first effect arise from the specific surface area of nanopowders, which allows them to strongly interact with the surface structures of the bacteria. The second reason is that the Cd-ferrite nanoparticles are small in size, they can be uptaken by bacteria rapidly[4]**.** Figure 4(a-d) illustrate disc diffusion test for Escherichia coli and Staphylococcus aureus and the inhibition zones formed by the ferrites.

According to the present work the antibacterial activity of Cd-ferrite increased with coating with PVP. For both Escherichia coli and Staphylococcus aureus, the inhibitory zone value at the maximum inhibitory concentration (1mg/ml) of the ferrite nanopowders without PVP was observed to be less than ferrite nanopowders with coating for both Escherichia coli Staphylococcus aureus. Staphylococcus aureus bacteria had the largest inhabitation zone for both ferrite nanopowders with and without coating with pvp.

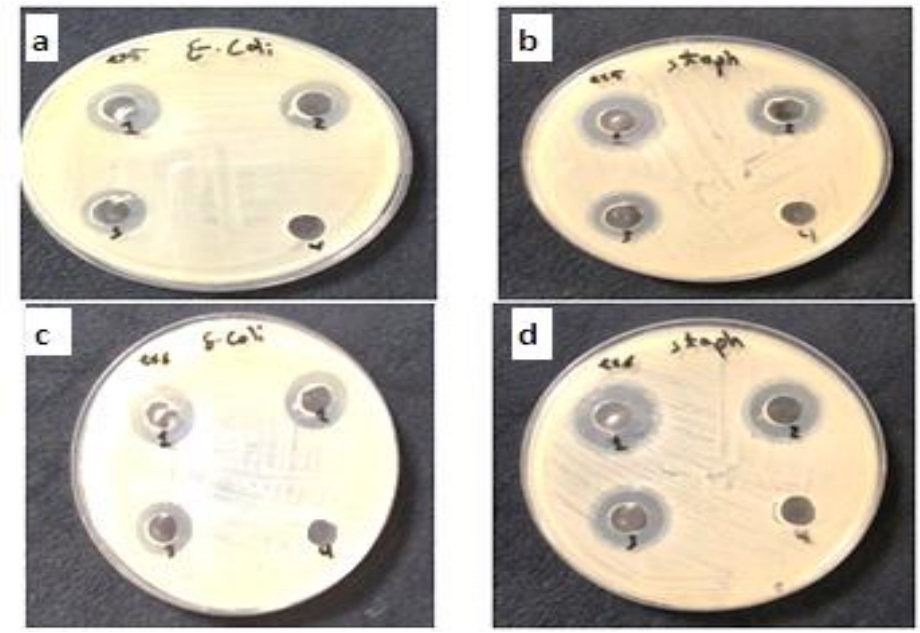

Figure 4(a-d): Photographs showing the disc diffusion test for Escherichia coli and Staphylococcus aureus and inhibition zones formed by the ferrites.

Table 2. Inhibitory zone formed by Cd-ferrite and PVP-coated Cd-ferrite against E. colibacteria and *Staphylococcus aureus*

Cd-ferrite samples	Inhibitory concentration mg/ml	Inhibitory Zone mm	
		Escherichia coli (E. coli)	*Staphylococcus aureus*
Uncoating	1	17	18
	0.5	15	16
	0.25	13	15
	0.125	0	0
Coating	1	17	19
	0.5	16	17
	0.25	15	16
	0.125	0	0

4. Conclusion

In summary, Cd-ferrites were successfully synthesized through the microwave-assisted combustion method. It was proved that this method can produce nanoparticles. According to the XRD analysis, the crystallite size of the ferrite decreased after PVP mediation. The XRD analysis of the coating samples indicates no significant structural variations except that the diffraction peaks intensity for PVP-coated Cd-ferrite are less compared to the uncoated ferrite. Depending on the FE-SEM images, the average particle size of the coated sample decreased. Fourier transforms infrared spectroscopy analysis has shown that the presence of PVP on the surface of the Cd-ferrite with a red shift as a result of the cationic distribution on the lattice sites. In the present study, the antibacterial activity of Cd-ferrite nanoparticles increases with coating by PVP. Cd-ferrite nanoparticles with and without coating with PVP are effective as antibacterial agents, which makes them useful in a wide range of applications, such as biomedical devices, food packaging and processing, water and waste treatment, and the textile industry.

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