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Electrical conductivity of the composite thin films of polyvinylpyrrolidone impregnated with nickel oxide nanoparticles.

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ABSTRACT

In this study, we prepared composite thin films of polyvinylpyrrolidone polymer by using 0.02-0.2% weight percent nickel oxide nanoparticles (NiONPs). hydrothermal method utilized to produce NiO NPs. By using XRD and FTIR analysis, the crystal structure was thoroughly examined. Surface morphology was well studied by scanning field emission (SEM) and atomic force microscopy(AFM). The optical properties of the composite thin film of PVP-NiO NPs have been characterized. the charge type of the thin film composite and electric properties determined by Hall effect measurement. XRD analysis reveals that NiO NPs have a cubic phase structure. The main chemical bonds of PVP pure have been determined, where Ni-O bonds appear at 408 cm-1 when NiO NPs are added to the polymer matrix. Composite thin film has a lower energy gap than PVP pure. The hall effect measurement shows Composite thin film is p-type and has $2*1^{-4}$ Sm at 0.2 %wt of NiO NPs. As a result, a composite thin film of PVP-NiO NPs can be utilized as a semi-conductor in photo sensor and solar cell applications.

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1.Introduction

The traditional concept of polymer is insulating materials elimination when polyacetylene polymer discovered in1970s, and Conductive polymers (CPs) entered a new era. Conductive polymers often possess the -conjugated system with alternate single and double bonds, which gives rise to their intrinsic electrical/electronic, electrochemical, and optical capabilities. The conjugation duration, crystallinity, and intra-chain and inter-chain interactions all have an impact on these physical characteristics. Compared to their counterpart, which is inorganic. CPs have benefits such low density, chemical variety, flexibility, adaptable conductivity, easily controllable form, and morphogenesis [1].

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Consequently, they might be used in large-area optoelectronic devices [2], absorption of microwaves materials, different kinds of sensors ,storage of energy engineering, anticorrosive coating, physiological field, etc. The well studied CPs also include polyacetylene, polyaniline, polypyrrole, polythiophene, poly(3,4-ethylenedioxythiophene), and poly(p-phenylene vinylene). [4].

Conductive polymers' conductivity is influenced by the kind, concentration of the dopants , quantity, and doping time of their dopants. Dopants can be divided into large polymeric species and tiny cations/anions depending on their molecular size. Because they are tightly attached to the polymer chain, large dopants can be hard to leach from it. Additionally, they affect the physical characteristics, density, and surface topography of CPs. Small dopants, on the other hand, quickly and easily dedope to produce CPs with low stability. The concentration of the dopant and the doping time have a significant impact on the conductivity of CPs, which increases as the doping level increases[5,6]. The conductivity is enhanced by an increase in dopant concentration. Due to the conductive polymers' organized, dense structure, the dopant ions slowly diffuse into them in the meantime. After a lengthy doping period, perhaps even several hours, the conductivity grew progressively saturated. Additionally, the process of doping and dedoping can be reversed [7,8].

The various types of doping techniques include photodoping, chemical doping, which includes vapor-phase doping and solution doping, non-redox doping, electrochemical doping, and charge-injection doping. Chemical and electrochemical doping are most frequently utilized because they are convenient and inexpensive [9,10].

2.Material and Method

2.1 polyvinylpyrrolidone (PVP) polymer

PVP is an amorphous polymer has chemical structure(C6H9N)n that is non-toxic, flexible, and has a numbetur of intriguing characteristics. It has strong environmental stability, is easily processed, is water soluble and biodegradable, is fairly electrically conductive, and has a wealth of physics in charge transfer mechanisms.known as polyvidone or povidone, is created from the monomer N-vinylpyrrolidone [11].

PVP readily absorbs up to 40% of its weight in water while in its dry form, when it is a thin, flaky powder. Because it quickly forms films in solution and has excellent wetting properties, it functions well as a coating or coating additive. PVP is a nonionic, linear, and amphiphilic polymer. a variety of electrolytes and resins are compatible. Esters, ethers, ketones, and hydrocarbons are insoluble, whereas water and polar solvents are soluble.ineffective for thermoplastics processing Oxygen-permeable, strong, glossy, clear, and films that adhere to a range of substrates Hygroscopic properties of adhesion and cohesion Cross-linkable biologically inert[12].

The only factor limiting the concentration of PVP polymer is viscosity. PVP polymer dissolves in cold water. A number of organic solvents, including alcohols, certain chlorinated compounds like chloroform, and methylene chloride, are also readily soluble in the PVP K-30 polymer. ethylene and isopropanol esters [14].

PVP polymer films are transparent, glossy, and durable when they haven't been altered or dried out. The visual appearance of films made using different solvent systems, like water, ethanol, chloroform, or ethylene dichloride, is unaffected.

2.2 Nickel Oxide Nano particales properties

Nickel Oxide Nano particales, also known as transition metal oxide, It is a metal-deficient p-type semiconductor with an ambient-temperature band gap of between 3.6 and 4.0 eV.

NiO is a green nano-material exhibition high conductivity, optimal switching speed, , and well-defined and steady redox kinetic. As catalysts, NiO films have been used. A substance made of different n-type semiconductors can be used to make batteries, supercapacitors, electrochemical capacitors, gas sensors, humidity sensors, memory devices, high-energy density devices, antibacterial materials, and more. It is ideal for energy conversion and storage devices and is favored as anode materials due to its high theoretical specific capacitance. In addition to their improved functionality and nonvolatile memory capabilities, NiO NPs also offer appealing optical, electrochemical, and magnetic properties.[15,16]

2.3 Nanostructure NiO synthesized by hydrothermal approach

the hydrothermal technique is an intriguing alternative synthetic method. Due to its low treatment temperature and simplicity of controlling particle size, Metal oxide and various other chemicals have been successfully synthesized at the nanoscale using this method. The hydrothermal method enables shaping and sizing of particles by varying reaction temperature, time, and precursor concentration.[17]

for synthesize the nickel oxide nanoparticles, liquids of NiO nitrade as Ni(NO3)2.6H2O and hexamethylenetetramine (HMT) (C6H12N4) was mixtured in 80 ml water under stirring [all material purchased from Sigma-Ald].

The concentration (weight in grams) of Ni nitrate and (HMT) is dictated by the molarity (M) of the solution, which is 5%, citing [18]

$M\% = \frac{W}{M.W} * \frac{1000}{V}$..........................(1)

The chemical reaction to obtain NiO NPs are [15,19]:

(CH2)6N4+ 6H2O→6HCHO+ 4NH3 .... (2)

NH3+ H2O→ NH^{+4} + OH^{-} ....(3)

Ni^{+2} + 4NH3→ $Ni(NH3)4^{+}$ ....(4)

Na+ is drawn to the OH surrounding the nanocrystal, forming a virtual capping layer that prevents the nanocrystal from developing [20].

Ni2+ + 2OH– ←→ Ni(OH)2 (5)

Ni(OH)2 + 2OH– ←→ [Ni(OH)4] 2– (6)

[Ni(OH)4] 2– ←→ NiO2 2– + 2H2O (7)

NiO2 2– + H2O ←→ NiO + 2OH– (8)

Pure crystals of NiO were obtained. One or more of the experimental growth parameters that have a substantial impact on the morphology and aspect ratio are the initial solution pH, precursor concentration, and growth temperature[21].

2.4 Hall Effect measurements

Hall measurements are frequently used to determine whether a semiconductor is n- or p-type and to assess the carrier concentration and mobility during the first characterisation of semiconductors.

Hall Effect's advantage is due to its competence to precisely measure the properties of a specimen of any arbitrary and irregular shape. In practically two dimensions, the sample (with low thickness), and solid (no holes), and "The Van der Pauw Method" [21] has four electrodes with the placement of 1cm around the perimeter of the specimen, and employs a linear four-point probe. In the existence of a z-directional magnetic field (B), a steady current (I) travels along the x-axis from left to right.

at first Electrons are exposed to the Lorentz force ,and They move in the direction of the negative y-axis as a result of a transverse voltage brought on by an excess surface electrical charge on the sample's side. According to Figure, this transverse voltage is referred to as the Hall voltage (V_H).

that forms the Hall field through the specimen thickness (t), as follow[22]:

$$R_H = \frac{V_H}{I}\frac{t}{B} \quad ....9$$

We can estimate the carrier's from the Hall coefficient equation. The semiconductor concentration and the carrier type,the sighn of R_H if negative or positive which determined n– or p-type, the semiconductor[23]:

$$R_H = \frac{1}{pq} \quad ....10.\text{for p-type}$$

$$R_H = \frac{-1}{pq} \quad ....11.\text{for n-type}$$

So, (q) is represented electron charge.

$$\sigma_n = q_n \mu_n \quad ....12.\text{for n-type}$$

$$\sigma_n = q_n \mu_n \quad ....13 \text{ p- type}$$

In the relationship between the Hall coefficient and conductivity (σ), the Hall mobility (μ_H) can also be obtained[24]

$$\mu_H = |R_H| \quad ....14$$

Mobility may be calculated by specified (σ) and (RH).Or can be performed to calculate the films' resistance (ρ)[25]:

$$\rho = R \cdot W \cdot t / L \quad ....15$$

W is the electrode's width, L is the distance between the electrodes, and t is the thin film's thickness, where R is the resistance.. The conductivity (σ) of the film based on the relationship , it could be specified [26]:

$$\sigma = 1/\rho \quad ....16$$

2.5 Preparations of Composite thin films

A thin film was formed by casting 1 gm of PVP solvent in 50 ml of water (2% PVP by weight) for 10 minutes at 60 °C. The concentrations of NiO NPs added to the solution were (0.01, 0.02, 0.03, 0.04, 05, 06, 0.07, 0.08, 0.09, and 0.1 gm). The solutions have to be cast on a glass Petri dish to form a thin film once the solution polymer has dried. According to Fig. 1, a thin film with a thickness of 1 µ is formed over the course of two days using the coating thickness meter CM8829S.

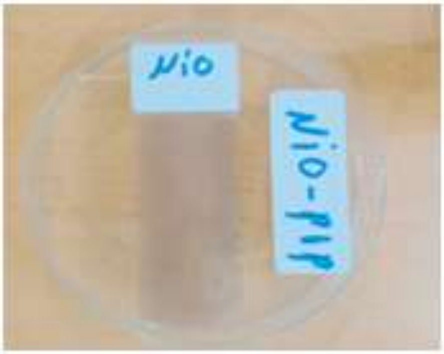


Figure **(1)**: PVP-NiO thin film composite

3.Results and Discussions

3.1 XRD spectrum analysis

Figure 2 shows the PVP-NiO nanopowder that was analyzed using an X-ray diffraction instrument. The results of the NiO NPs data, showed the presence of diffraction peaks at the angles (2θ): 37.20°, 43.20°, 62.87°, 75.20°, and 79.38°, which correspond to the Miller indices (111), (200), (220), (311), and(222) ,respectively.These Miller indices suggest that the cubic phase of NiO structure is the crystal form of NiO NPs. The diffraction peak data are consistent with the Standard (JCPDS No. 10-0325). Thus, the NiO nano-particles were cubic structure. The XRD Pattern spectra of NiO NPs matched
with[27].

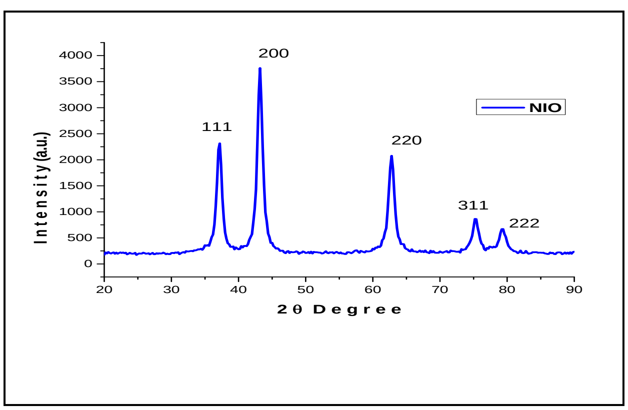


Figure (2) : XRD pattern of NiO NPs

From X-ray diffraction, the mean crystallite size can be calculated using Scherrer's formula, d = 0.9λ /β cos θ, where d is the crystallite size, is the diffraction angle, is the full width at half maximum, and is the X-ray wavelength [28]. The particles' crystalline average size (D) is 25 nm.

3.2 FTIR of PVP-NiO NPS Composite Thin Films

The FTIR spectrum was determined for the pure PVP and composite thin film of PVP-NiO nanostructure, which is displayed in Fig. 3.

The main active groups characteristic of the pure PVP polymer are shown clearly in Figure 3(a) and are as follows: [O-H] stretching, [C-H] asymmetric stretching, and [C=O] stretching vibration In the FTIR spectrum, C-N stretching, pyrrolidone ring, and CH2 bending vibration are located at and the two peaks, .Corresponding wave number at 3434 cm -1, at 2955 cm -1, at 1661 cm -1, 1424 cm -1, 895 cm−1, 1291, and 1018 respectively.[29,30]

As for PVP doped with NiO NPs 0.2 wt%, there is a difference in the locations of the backbone of PVP active groups due to the interaction with NiO NPs, where a bond Ni-O appears in the 408 cm-1, and this is identical to the research [31] where there was a shift to the right and intensity increased compared with the main active group of PVP pure due to doping processing as shown in figure 3(b)

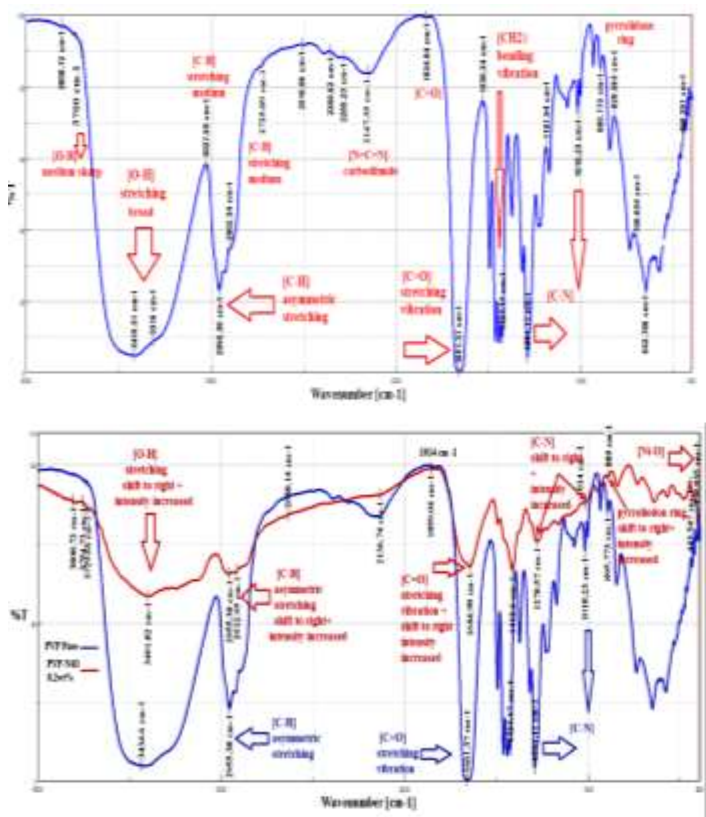
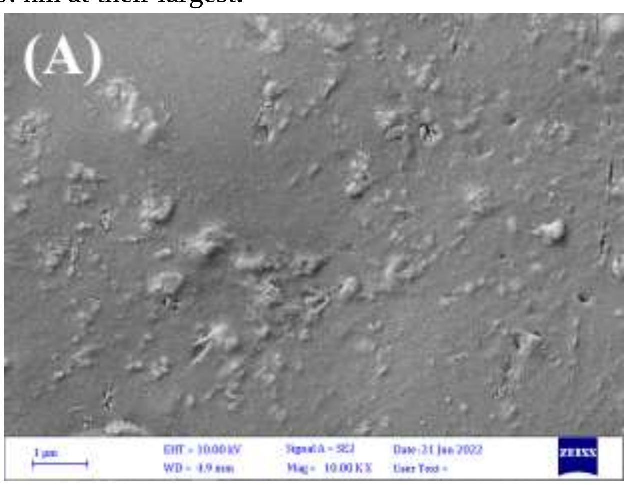


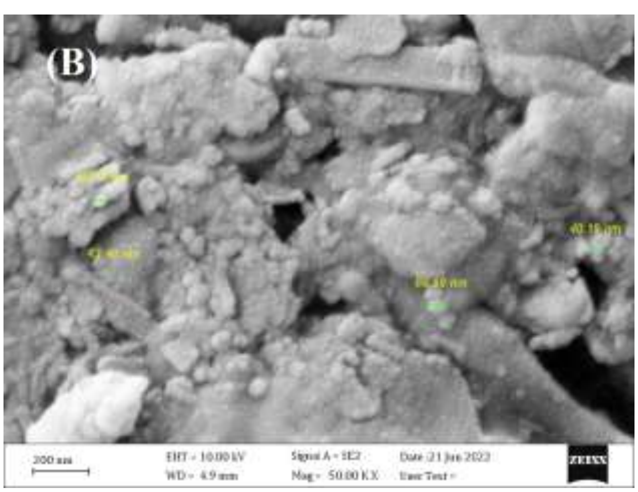
Figure 3(a) shows the curve of the infrared spectrum of a pure PVP in blue line, (b) elucidate overlapping between a PVP pure (blue line) and PVP- NiO NPs composite thin films (red line) .

3.3FESEM Images of PVP-NiO nanostructure composite Thin Film

NiO NPs features are depicted Fig. 6 with high-density image of scanning electron microscopic (SEM), synthesized via a hydrothermal approach , Figures 4 (a,b) demonstrate magnification of 1µm and 200 nm respectively , that the product is primarily made up of nanoparticles produced with various dimensions of an agglomerated formulation cluster of the nanoparticles. The minimum size of produced nanoparticles is 40.19 nm as shown in Fig 4.(b)

Histogram of The FE-SEM represented in Images (c), according to calculation of Image J software program has been selected 100 particles as a minimum indicates that the particles have a standard deviation of 10.9 nm and an average diameter of 30 nm. NiO particles ranged in size from 11.8 nm at their smallest to 80. nm at their largest.





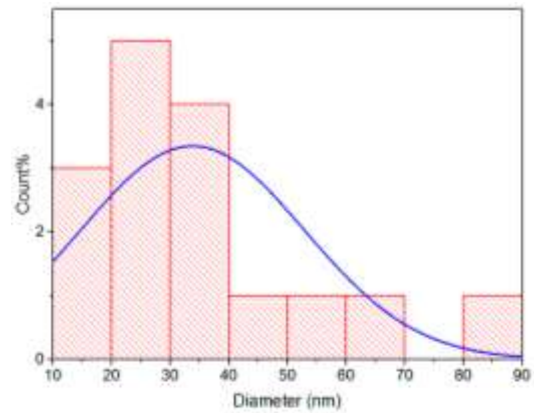


Figure 4. FESEM images of NiO nanoparticales (a) FESEM images of NiO nanoparticales with 1µm scale (b) NiO nanoparticales with,200 nm scale exhibit particles size (c)Histogram of The FE-SEM of NiO nanoparticales with size distributions

3.4 AFM

The surface topography and morphology of a PVP-NiO NPs composite thin film were analyzed by atomic force microscopy (AFM) for the sample with nano- and micron-scale dimensions shown in Figure (5) in two dimensions and in three dimensions, respectively

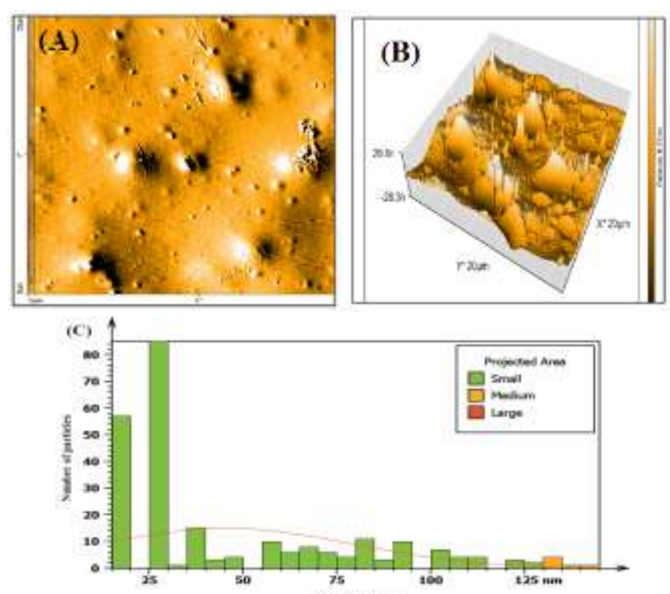


Figure. 5 Section line analysis, (A) topography 2D, and (B)morphology 3D AFM images and (C)particle size distribution of PVP-NiO Composite thin film

It is known that the morphology of the film greatly depends on the formation of nanoporous structures within a thin composite film. The image shown in Figure (5) indicates that after impregnation of NiO nanoparticles into the composite thin film, the surface becomes much more homogeneous, and the dimensions of most of the particles were smaller than (1.0 µm), which could be due to aggregation of the NiO nanoparticles, which is a very common phenomenon in nanomaterials synthesis. Mean root square height and mean diameter are significant parameters associated with surface roughness with values at the nanoscale, as seen in figure 7 (c), which is consistent with the findings of (SEM IMAGE), which depict particles with nano-scale sizes. This confirms that the casting process is a successful method for the synthesis of a thin film without affecting the nano-size of the NiO particles that are inserted in the PVP matrix.

3.5 Ultraviolet-visible (UV-Vis) spectroscopy analysis

Figure (6) The curve depicted the relation between (hv) and $(\alpha h\nu)^2$, where the band gap energy of the thin film is represented by the intercept between the lowest point in the curve and the x-axis, which is (4.8 eV) for PVP pure, and the result coincided with Pan, Mingming, et al.(2020)[20] and Vani, G. Naga Sudha, et al ,2013.[33]

Figure 6 (b) shows that the band gap energy for a thin film of PVP doped with wt%0.2 NO NPs is 4.6 eV smaller than that of PVP pure. This finding is in agreement with Mohammed, M. I., et al. (2022) [34]. band gap of NiO is 3.6 eV while NiO NPs in range (3.6 - 4 eV) [35].

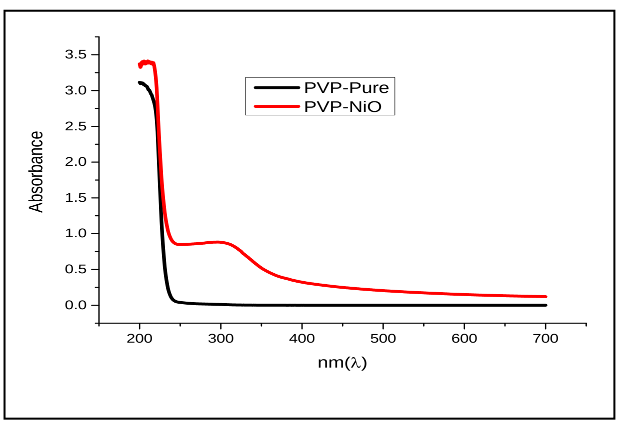


Figure 6(a) UV-Spectra analysis (Absorption spectra versus the wavelength) for PVP Pure thin film (black line) and PVP- NiO Composite thin film (red line) depicted their Absorption versus Of their Wave length.

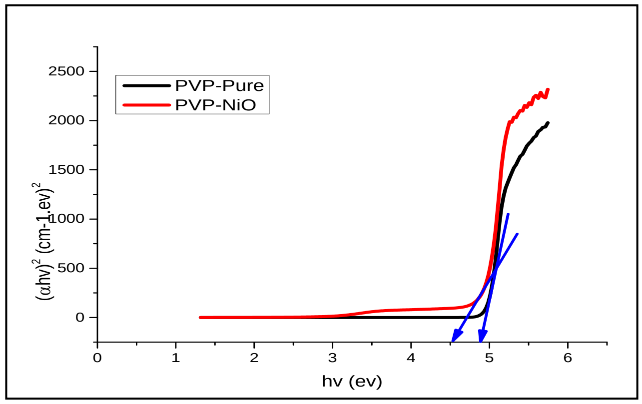


Figure 6 : (b) Energy band gap of PVP Pure thin film (black line) ,(B) Energy band gap of PVP-NiO Composite thin (red line)

3.6 Hall Effect results

In Fig. 9, the thin film's electrical conductivity PVP-NiO NPs composite thin films has been evaluated and plotted as a function of NiO NPs doping concentration at various concentrations in the order of: (0.01, 02, 03, 04, 05, 06, 07.08.09, 0.1) gm, The conductivity increases with concentration, reaching a maximum of (1.091*10-4) Sm at a concentration of 0.1 gm. Table 1 lists the conductivity, average Hall coefficient, charge type, and mobility of NiO NPs at various concentrations. The P-type doping is in the thin film. The electric conductivity of pure PVP is in the range of 10-6 and 10-7 [36].

The doping procedure with NiO NPs increases electrical conductivity by increasing NiO NP concentrations, producing a semiconductor made of PVP-NiO NP composite thin film. The highest value of conductivity was obtained when it was concentrated NiO NPs, which is 0.1 gm.

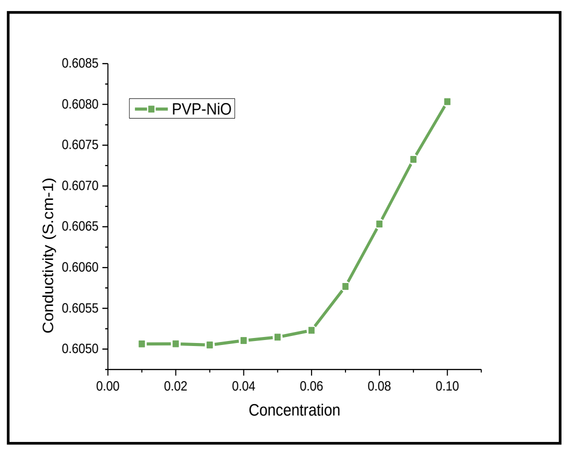


Figure (7). Relationship between conductivity and concentration of PVP- NiO NPs thin film

TABLE .1 measurements of hall effect of PVP-NiO Composite thin films

Concentration (gm)	Conductivity $1/\Omega$.cm (Sm)	Avarage Hall Coefficient (m^2/c)	Mobility (cm^2/v_s)
0.01	0.0 2234*10^{-5}	-1.91434*10+6	1.4731*10^{+2}
0.02	0. 04185*10^{-5}	-1.97033*10+6	1.7457 *10^{+2}
0.03	0. 06153*10^{-}5	-2.01137*10+6	1.8753*10^{+2}
0.04	0. 1431*10^{-}4	-2.10741*10+5	2.073610^{+2}
0.05	0. 1810*10^{-}4	-2.21328*10+5	2.1214*10^{+2}
0.06	0. 2075*10^{-}4	-2.3020*10+5	2.1872*10^{+2}
0.07	0. 4047*10^{-}4	-2.41271*10+5	2.2327*10^{+2}
0.08	0. 6016*10^{-}4	-2.50552*10+5	2.2836*10^{+2}
0.09	0. 827*10^{-}4	-2.60783*10+5	2.3463*10^{+2}
0.1	0. 2080*10^{-}3	-2.76164*10+4	2.432*10^{+2}

Conclusion

The hydrothermal method was used to produce nickel oxide nanoparticles, where the scanning electron microscope(SEM) examination image showed the nanoscale size of nickel oxide and the particle size avarage was calculated and was 30 nanometers, while the X-ray diffraction examination determined the Miller indices, which showed that nickel has a Cubic Phase structure and that the crystalline average size of particles is equal to 25 nanometers, while the atomic force microscope(AFM) image showed the distribution of nanoparticles on the surface of the composite thin film randomly with the appearance of semi-spherical shapes indicating the presence of nickel oxide nanoparticles, we conclude that the polymer was doped with nanoparticles based on the results of the nanoscale size of nickel oxide particles . The energy band gap of a pure polymer is larger compared to With the doped polymer, an increase in the concentration of nickel oxide nanoparticles led to a decrease in the energy gap. The electrical conductivity of the polymer doped with different concentrations increases with increasing concentration, and the best value is at the weight ratio (0.2%). This result shows the effect of adding NiO NPs on both the optical and electrical properties of the polyvinylpyrrolidone polymer.

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الموصلية الكهربائية للأفلام الرقيقة المركبة من البولي فينيل بيروليدون مشوبة بجسيمات أكسيد النيكل النانوية

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الخلاصة:

تم تحضير اغشية من بوليمر بولي فاينيل بيرليدون مشوبة بنسب وزنية مختلفة من جسيمات اوكسيد النيكل النانوي(0.2-0.02%) التي انتجت باستخدام الطريقة الحرارية المائية ذات الدرجات الحرارية المنخفضة. باستخدام تحليل حيود الاشعة السينية و تحليل فورييه للاشعة تحت الحمراء تم فحص التركيب البلوري للجسيمات النانوية. تم التحقق من مورفولجية الاغشية المشوبة باستخدام فحص المجهر الالكتروني الماسح و مجهر القوة الذرية، تم تمييز الخصائص البصرية للفيلم الرقيق المركب .اظهر نمط حيود الاشعة السينية ان اوكسيد النيكل له تركيب بلوري متعدد مكعب.اجريت قياسات تحويل فورييه للطيف بالأشعة تحت الحمراء لبوليمر بولي فاينيل بيرليدون المشوب باكاسيد المعدن النانوية ، حيث اظهر اختلاف واضح عن البوليمرالنقي حيث ظهرت رابطة Ni-O عند الطول الموجي 408سم -. لتأكيد تأثير إضافة NiO على الترابط الكيميائي لأفلام بولي فاينيل بيرليدون تم استخدام قياسات معامل هول لحساب الخصائص الكهربائية بما في ذلك التوصيلية حركية ناقلات الشحنة ونوعها .اظهرت نتائج قياسات معامل هول بان افضل توصيلية تم الحصول عليها عند النسبة الوزنية (0. 2% لاوكسيد النيكل النانوي تساوي ($2*10^{-4}$ سيمنز) حيث تم تحضير غشاء شبة موصل يمكن استخدامة في المتحسسات وتطبيقات الخلايا الشمسية.

الكلمات المفتاحية: بوليمر بولي فاينيل بيرليدون، جسيمات نانوية من أوكسيد النيكل، التوليف الحراري المائي، التوصيل الكهربائي، فيلم رقيق مركب، بوليمر موصل.