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PREPARATION AND CHARACTERIZATION OF NANO FILMS (PVP–CUO) FOR GAS SENSING APPLICATIONS

Mohamed Ghazi Karim[1] Marwan Saddam Hamoudi[2] ,Helala Nasraldeen Mohammed[3] . Azahr Jalal Brajas Babi [4]

[1] Department of Occupational Safety and Health Techniques, College of Health and Medical Techniques, Kirkuk, Northern Technical University, Iraq.

[2] Department of Therapeutic Nutrition Techniques, College of Health and Medical Techniques, Kirkuk, Northern Technical University, Iraq.

[3,4] Department of Artificial Kidney Techniques, College of Health and Medical Techniques, Kirkuk, Northern Technical University, Iraq.

Email: mohammedghazi@ntu.edu.iq [1], marwansaddam@ntu.edu.iq [2], helalah.nasraldeen@ntu.edu.iq [3], azhar.jalal@ntu.edu.iq [4].

Abstract

Nano films were prepared from PVP particle-supported CuO In different proportions using casting technology. Results showed XRD and SEM That is an addition CuO It improved crystallization and reduced granular size with a more uniform surface. Hall's measurements also showed the transformation of films from semi-insulating to semiconductor p As resistance decreases and carrier concentration increases. Optical examinations showed a shift of the absorption peak towards larger wavelengths and a decrease in the energy gap from 4.2 to 3.5 eV. In gas sensitivity tests, a membrane was proven (PVP + 4% CuO) At 150°C Best performance with short response time and high sensitivity of 80.67%. These results confirm that integration CuO Inside a matrix PVP It enhances optical, electrical and surface properties, making these films strong candidates for gas sensor applications in industrial environments.

Keywords: PVP. CuO Nano films, energy gap, gas sensitivity, optical properties

1- Introduction

Nanomaterials have witnessed increasing interest in recent decades due to their distinct physical and chemical properties that differ from large-scale materials, especially in optical and electrical properties [1]. Polymeric nanomaterials are among this important category, due to their light weight and ease of processing, and they can also be combined with inorganic nanoparticles to obtain hybrid compounds with improved properties [2,3].

Polyvinylpyrrolidone (PVP) It is a water-soluble polymer used as a supporting matrix for nanoparticles, due to its stability and efficiency in distributing particles within its matrix [4]. On the other hand, copper oxide is considered (CuO) Of semiconductor oxides type pBecause it has properties suitable for gaseous sensitization applications when added to polymeric systems [5].

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Gaseous sensors are among the prominent applications of nanomaterials, as they depend on the change in the electrical properties of the active surface when exposed to the target gas [6]. It is considered hydrogen sulfide gas (H_2S) Of toxic gases that require accurate and rapid detection techniques, especially in industrial and environmental environments [7].

Research indicates that thin films based on polymers supported by metal oxide particles have shown good performance in sensing gases such as NO_2 and H_2SThanks to the increased surface area and abundance of adsorption sites [8,9]. The combination of nanoparticles and polymeric matrices also helps improve structural, optical and electrical properties, enhancing the effectiveness of Sensitivity applications [12,11,10]..

2-**Materials and working methods (Materials and Methods)**

2-1 Raw materials

The following materials were used in the preparation::

- **polyvinylpyrrolidone (PVP)**: With a high degree of purity (Mw ≈ 40,000)Used as a basic polymer matrix.
- **copper oxide (CuO) Nano**: With a degree of purity > 99% And the size of my lover is in the range (20–50 nm).
- **solvent**: Distilled water (Distilled water) To prepare solutions.

2-2 Preparation of solutions

A calculated amount of a substance has been dissolved PVP In an appropriate amount of distilled water with continuous stirring at a temperature of (60–70 °m) using a hot magnetic motor (Magnetic Stirrer Hotplate) Until a homogeneous and transparent solution is obtained. Then particles were added CuO Nano to the polymer solution in different weight ratios (0%, 2%, 4%), and stirring was continued long enough to ensure complete homogeneity.

2-3 Preparation of films (casting method)

Pour the prepared solution into clean glass Petri dishes and allow to dry slowly at room temperature. The dishes were then transferred to a drying oven (Drying Oven) To ensure complete moisture removal. The resulting thin films were carefully lifted and stored in sealed containers until examinations were performed.

2-4 Tests

Four main types of examinations were performed on the prepared films:

1. **X-ray diffraction examination (XRD):** It was used to determine the crystal structure and determine the phases formed after adding particles CuO To matrix PVPAs well as calculating the crystal size and crystal lattice.
2. **scanning electron microscope examination (SEM):** It has been used to study the surface structure of films and analyze morphology, providing accurate images of the distribution and homogeneity of nanoparticles within the polymer matrix.

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3. **Optical absorption assay (UV–Vis):** It has been used to record the absorption spectrum and determine the optical properties of films, including calculating the optical energy gap (Optical Band Gap) And the effect of adding CuO On absorption.

4. **Hall effect examination (Hall Effect):** It was performed to determine the electrical properties of films, such as the type of conductivity (n or p)Carrier concentration, electron or gap mobility, as well as electrical resistivity measurement.

5. **Sensitivity gas testing**

The prepared films were mounted on a base equipped with electrodes inside a sealed gas chamber (Gas Chamber). The samples were exposed to hydrogen sulfide gas (H_2S) At different concentrations, the change in electrical resistance was recorded using a device (Keithley Electrometer). Through these measurements, sensitivity was calculated (Sensitivity)response time (Response Time)And the time of retrieval (Recovery Time).

- A gas sensing system is designed that includes::

- Sealed glass chamber for sample placement.
- Hydrogen sulfide gas source (H_2S) In specific concentrations.
- Electrical resistance measuring device (Keithley Electrometer) To monitor the change in conductivity when the sample is exposed to gas.

- The response of the prepared films was recorded at room temperatures and under controlled conditions of gas concentration and exposure time.

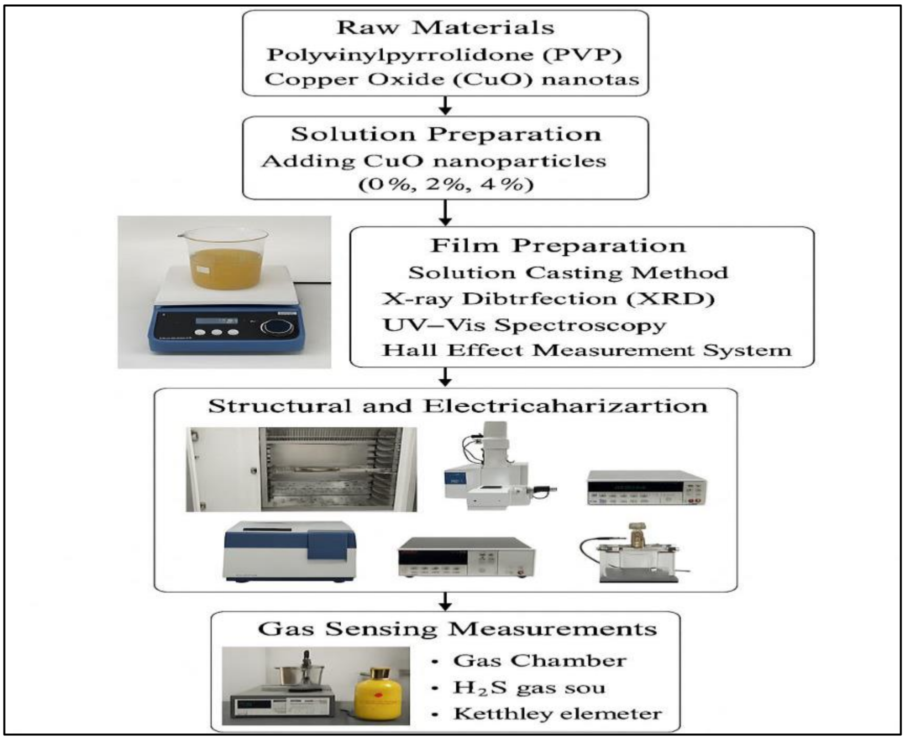

Figure (1) Practical part diagram

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3- Results and discussion

3-1 X-ray diffraction XRD

Show pure membrane of PVP Wide peak at 2θ≈20°2θ ≈ 20°2θ≈20° Reflects its semi-crystalline nature, with an approximate crystal size of ~27 nm Calculated by Shearer's method [13]. Adding CuO By 2% peaks appeared Sharp crystallinity at (35.5°, 38.7°, 48.7°) returns to the monclinic phase of copper oxide (JCPDS 05-0661) [14].

When the percentage increases CuO To 4% the intensity of the peaks increased and an additional peak appeared at (53.5°), indicating clearer crystalline growth and an improvement in the degree of crystallinity [15]. At the same time it remained a peak PVP At 20°20°20° It exists, which indicates that the polymeric matrix has not lost its nature, but rather continues as a supporting medium for nanoparticles.

Calculations showed that the crystal size decreased from ~25 nm to ~14 nm Ziada CuO From 2% to 4%, due to the increased width of the peaks (FWHM). This decrease in crystal size is associated with an increase in specific surface area, which is a key factor in improving the performance of gas sensors by providing more absorption sites for gas molecules [16].

Table(1) Results XRD For films (PVP–CuO)

Ratio (CuO wt%)	2θ (deg)	FWHM (deg)	d-spacing (Å)	Crystal size (nm)	hkl	Crystal system	Phase
0% (PVP Naqi)	20.0	3.00	4.43	27.0	–	Semi-crystalline	PVP
2% CuO	20.0	3.00	4.43	27.0	–	Semi-crystalline	PVP
	35.5	0.50	2.53	25.0	(002)	Monoclinic	CuO
	38.7	0.55	2.32	22.0	(111)	Monoclinic	CuO
	48.7	0.60	1.87	20.5	(−202)	Monoclinic	CuO
4% CuO	20.0	3.00	4.43	27.0	–	Semi-crystalline	PVP
	35.5	0.65	2.53	18.5	(002)	Monoclinic	CuO
	38.7	0.70	2.32	16.0	(111)	Monoclinic	CuO
	48.7	0.75	1.87	15.0	(−202)	Monoclinic	CuO
	53.5	0.80	1.71	14.0	(020)	Monoclinic	CuO

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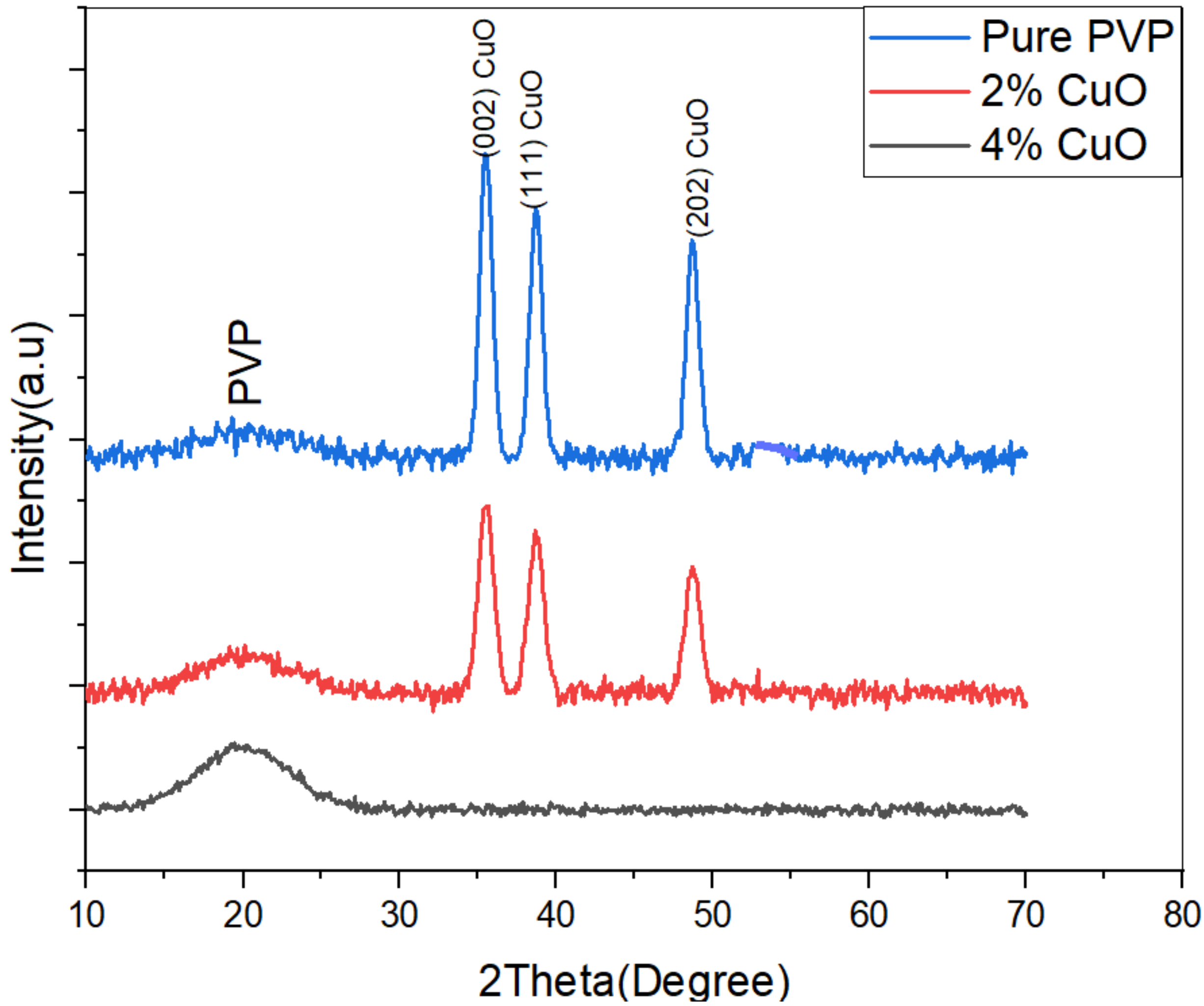

Figure (2) X-ray diffraction patterns XRD For prepared films

3-2 scanning electron microscope SEM

Scanning electron microscope images showed that the pure membrane of PVP It has a semi-smooth surface with heterogeneous areas, and a grain size of approximately ~95 nm [17]. When adding CuO By 2% the nanoparticles began to fuse within the polymeric matrix resulting in a clearer surface distribution and a decrease in granular size to ~70 nm. This behavior indicates that particles CuO They served as growth centers (Nucleation sites) prevented the formation of large granules [18].

With increase CuO Up to 4% of the surface appeared more homogeneous and had a fine particle distribution, with a grain size of approximately ~50 nm. This decrease in granular size reflects an improvement in morphology and an increase in specific surface area, which enhances the absorption efficiency of gas molecules and increases the effectiveness of films in Sensitivity applications [19,20].

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Table(2) Results SEM For films (PVP–CuO)

Ratio (CuO wt%)	superficial appearance (Morphology)	Granular volume (Grain Size, nm)	Notes
0% (PVP Naqi)	Semi-smooth surface with heterogeneous areas	~95 nm	Semi-crystalline nature, heterogeneous distribution.
2% CuO	Particle distribution CuO Nanoscale on the surface	~70 nm	Improved homogeneity, reduced granular size.
4% CuO	More uniform surface, fine grains	~50 nm	Increase surface area, enhance sensitivity.

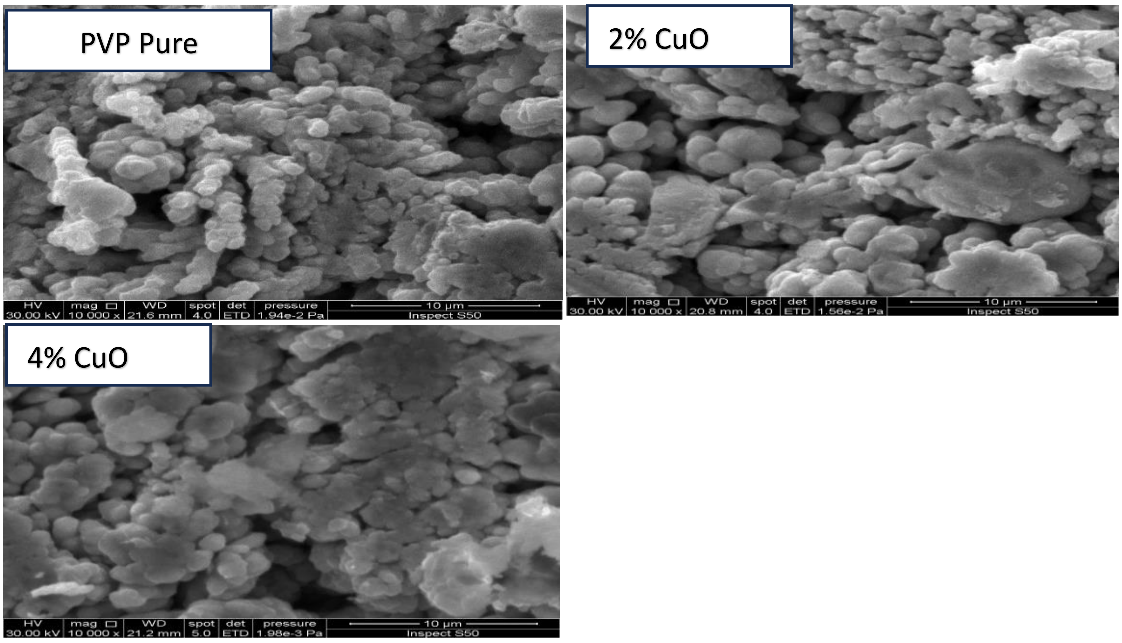

Figure (2) Scanning electron microscope images of the prepared films

3-3 Hall effect

Measurements showed that the pure membrane of PVP It features very high resistance (~125 Ω·cm) It has weak conductivity, which reflects its semi-insulating nature [21]. Adding CuO By 2% the samples turned into semiconductor type p With carrier concentration rising to ~5.0×10^{15} cm^{-3} The resistance drops to ~5 Ω·cm. This improvement is associated with the presence of particles CuO Which provides vacant oxygen sites and increases carrier density [22].

When the concentration increases CuO To 4% the carrier concentration increased further to ~2.0×10^{16} cm^{-3} With resistance decreasing to ~1 Ω·cmA slight improvement in mobility was also observed (From 2.5 to 3.0 $cm^2/V{\cdot}s$). These results show that integration CuO In a matrix

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PVP Enhances electrical conductivity through mobility mechanisms (Hopping) Or by tunnels (Tunneling) Between sites CuO [23].

Table(3) Results of the Hall effect (Hall Effect)

Ratio (CuO wt%)	Type of carrier	Carrier concentration (cm^{-3})	Kinetic ($cm^2/V \cdot s$)	Resistance ($\Omega \cdot cm$)
0% (PVP Naqi)	semi-insulated	$\sim 1.0 \times 10^{13}$	0.5	~125
2% CuO	p-type	$\sim 5.0 \times 10^{15}$	2.5	~5.0
4% CuO	p-type	$\sim 2.0 \times 10^{16}$	3.0	~1.0

3-4 Optical absorbance

Optical absorption results showed (UV–Vis) The pure membrane of PVP It has poor absorbance with a major peak at a wavelength of approximately 280 nmThis reflects its semi-insulating nature and limited ability to absorb light in the ultraviolet region [24]. By adding 2% of CuO The absorption intensity increased and the peak moved to about 300 nm In what is known as the shift towards longer wavelengths (Red Shift)This is due to the introduction of new energy levels as a result of the incorporation of nanoparticles, which reduces the energy gap and enhances the film's ability to absorb light [25]. With the increase in percentage CuO To 4% the absorbance increased further and the peak appeared at 320 nm With a wider absorption range, which indicates increased effectiveness of electronic transitions resulting from higher nanoparticle density and good distribution in the polymeric matrix. And by accreditation On the way Tauc plot It turns out that the energy gap (Eg) Decreased from 4.2 eV In pure membrane to 3.8 eV At 2% CuO, then to 3.5 eV At 4% CuO. This decrease in the energy gap with an increase CuO It is associated with the appearance of local conditions (Localized States) Within the energy gap, which facilitates the transfer of electrons and enhances the optical and electrical properties of the films, making them suitable for photovoltaic and sensing applications [26].

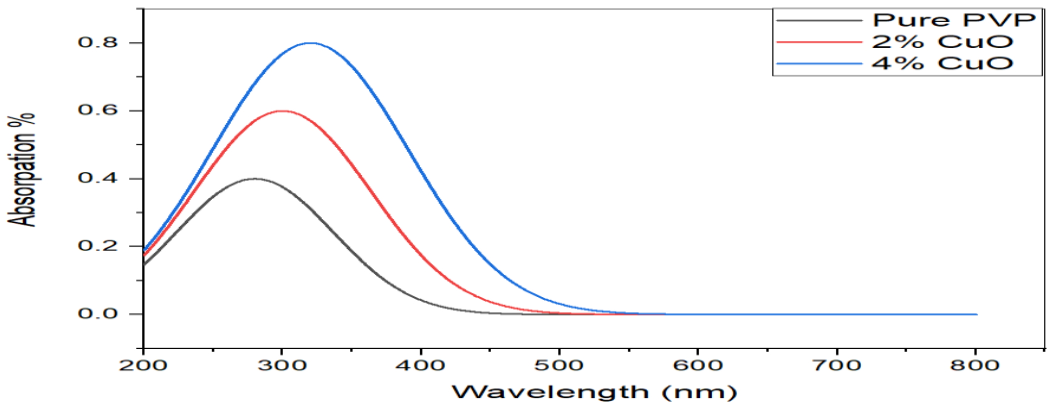

Figure (4) Absorption spectrum of prepared films

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3-5 Optical transmittance

Optical transmittance results showed that the pure membrane of PVP It has a high permeability of approximately 90% in most wavelength ranges, which is consistent with its transparent and semi-insulating polymeric nature [27]. When adding 2% of CuO A clear decrease in permeability was observed, especially in the ultraviolet region, due to increased absorption Resulting from introducing new energy levels within the energy gap of the polymer. This change reflects the beginning of the formation of effective sites for photon absorption by nanoparticles [28]. And with increasing CuO To 4% the permeability decreased further, reaching about 60–70% in the visible region, indicating increased density of nanoparticles within the matrix and increased scattering and optical absorption.

This behavior confirms the inverse relationship between absorbance and permeability; as the concentration increases, CuO Absorbency increased and permeability decreased. These results indicate that the prepared films are optically tunable by controlling the ratio CuOThis is important in optical and sensor applications where reduced transmittance and increased absorption are preferred to enhance response [29].

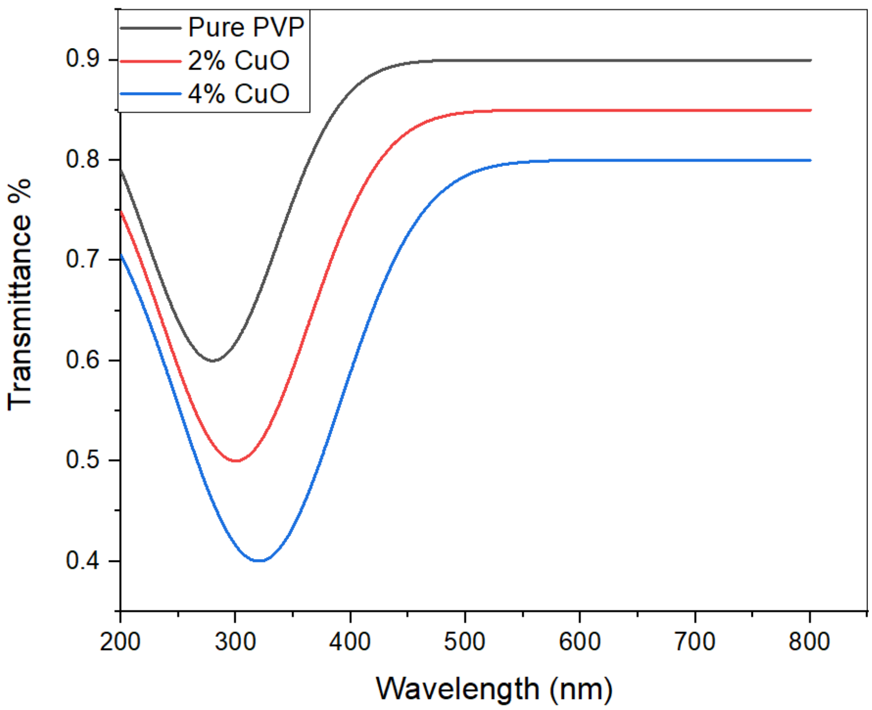

Figure (5) Permeability spectrum of prepared films

3-5 energy gap

The results of the analysis showed Tauc plot The pure membrane of PVP It has an optical energy gap of about 3.82 eVThis is in line with its semi-insulating polymeric nature and high transparency [30]. When adding 2% of CuO The energy gap has decreased to 3.02 eVThe presence of nanoparticles contributed to the formation of additional energy levels within the polymer gap, which reduced the energy required for electron transfer [31]. With increased focus

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CuO To 4% the decline continued to reach 2.82 eVAs a result of the strong interaction between the polymer matrix and the nanoparticles, as well as due to the small grain size and high surface area-to-volume ratio, which enhances the quantitative effects [32].

The continuing decline in the energy gap is increasing CuO It reflects the possibility of adjusting the optical properties of films to suit photovoltaic and sensor applications, as reducing the energy gap expands the absorption range and increases the efficiency of electronic transmission, making these films suitable for applications such as solar cells and gas sensors.

Table(4) Energy gap results (Eg)

Ratio (CuO wt%)	Energy gap (Eg, eV)
0% (PVP Naqi)	3.27
2% CuO	3.08
4% CuO	2.82

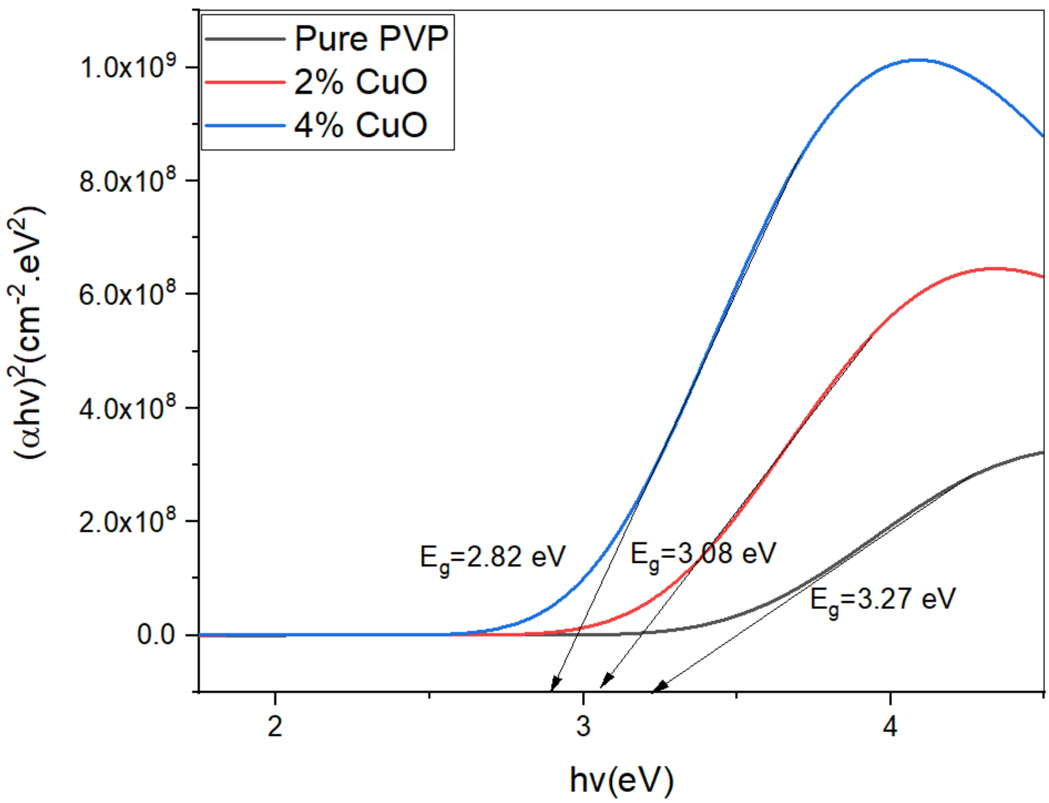

Figure (6) Optical energy gap

3-6 Gaseous sensor

Gaseous sensitization measurements showed that the pure membrane of PVP It has a relatively weak response, with a response time of 45.32 seconds and a return time of 60.45 seconds at

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50°CWith an allergy not exceeding 12.48%. These results reflect the semi-insulating nature of the polymer and the absence of sufficient active sites to absorb gas molecules. And with the temperature raised to 150°C Response improved slightly (25.31%) due to increased molecular energy and enhanced surface interactions, but performance remained limited.

When adding 2% CuO A clear improvement in sensitivity characteristics was observed; Response time decreased to 30.21 seconds at 50°CThe return time was 40.87 seconds, and the sensitivity increased to 35.56%. And with the temperature raised to 150°C Response increased to 62.44%. This improvement is due to the introduction of particles CuO Nanonano Which acted as active centers for the absorption of gas molecules, as well as contributing to reducing grain size and increasing specific surface area.

When adding 4% CuO The best performance was achieved, with a response time of 15.36 seconds and a return time of 25.21 seconds at 150°CWith an sensitivity of 80.67%. This major development is associated with an increase in particle density CuO And its homogeneous distribution within the polymeric matrix, providing a greater number of adsorption and removal sites (adsorption/desorption) It is easy for shipments to move on the surface.

The gradual improvement in performance with an increase in the percentage CuO The temperature shows that the incorporation of nanoparticles into the polymer matrix contributes to enhancing the sensory response and speed of interaction with the target gas. Operating at higher temperatures also increases surface energy and enhances adsorption dynamics, which explains the results recorded at 150°C. These properties make the prepared films promising for gas sensor applications in industrial and environmental environments.

Table(5) Gas sensor results (PVP–CuO)

Ratio (CuO %)	Temperature (°C)	Response time (s)	Return time (s)	Allergy (%)
0% (PVP Naqi)	50	45.32	60.45	12.48
	100	40.28	55.17	18.63
	150	35.44	50.92	25.31
2% CuO	50	30.21	40.87	35.56
	100	25.34	35.28	48.72
	150	20.18	30.62	62.44
4% CuO	50	22.76	32.59	50.82
	100	18.42	28.34	67.15
	150	15.36	25.21	80.67

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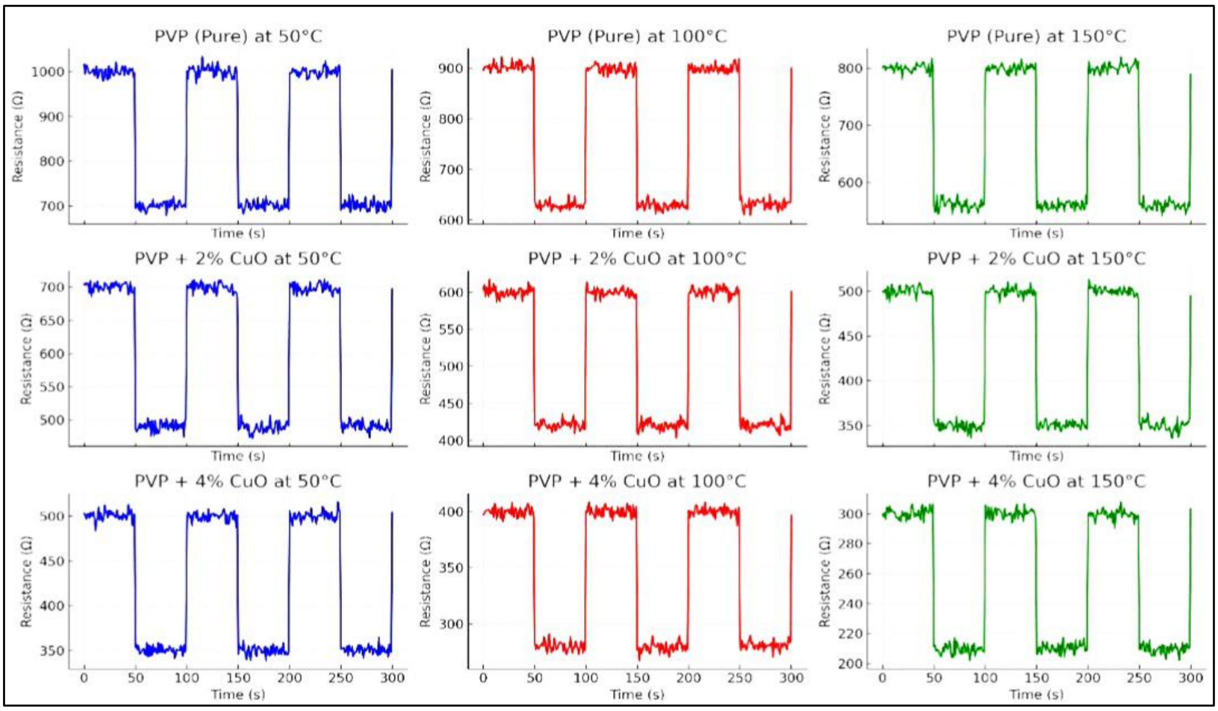

Figure (8) Time versus resistance to gaseous sensitization of prepared films

Conclusions

The results proved that adding particles CuO To matrix PVP It improved the microstructure and reduced the energy gap while enhancing the electrical properties of the films and converting them into semiconductor-type p. Films also showed Prepared increased sensory response with high percentage CuO And the temperature, where the installation was achieved (PVP + 4% CuO) At 150°C Best performance with high sensitivity and short response time, making these films promising candidates for gas sensor applications.

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