

9-27-2021

Section: Chemistry

ADSORPTION OF COPPER IONS ON CHITOSAN-BASED FILMS FOR DETECTION OF H₂S GAS POLLUTANT

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Gamal, Ahmed; Ibrahim, Ahmed; and El-Zomrawy, Adham (2021) "ADSORPTION OF COPPER IONS ON CHITOSAN-BASED FILMS FOR DETECTION OF H₂S GAS POLLUTANT," *Al-Azhar Bulletin of Science*: Vol. 32: Iss. 1, Article 2.

DOI: <https://doi.org/10.21608/absb.2021.68993.1108>

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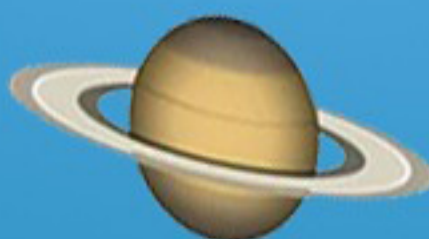
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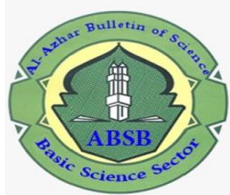
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Received: 25 Mar 2021; Revised: 13 Apr 2021; Accepted: 18 Apr 2021; Published: 27 Sep 2021

ABSTRACT

Sulfide in its form of free hydrogen sulfide is recognized to cause anxiety at its lower level of concentration, but at a higher level, it causes permanent brain damage, fainting, or even death through asphyxiation. In this study, chitosan films were fabricated, and the copper ions were loaded onto them by way of adsorption to be used in the detection of hydrogen sulfide (H₂S) gas electrochemically. The results presented an amazing variation in the electrical resistivity and conductivity of the chitosan and copper-loaded chitosan films against time in response to H₂S gas adsorption. Also, the results demonstrate a significant increase in the conductivity of the copper-loaded chitosan/H₂S film as compared with pure chitosan/H₂S. Furthermore, the films were examined after the H₂S adsorption process in many ways; resistor-capacitor circuit, electrochemical impedance spectroscopy, X-ray diffraction (XRD), energy-dispersive X-ray (EDX), as well as electron scanning microscopy (SEM). SEM images indicated the successful loading of copper ions onto the chitosan structure.

Keywords: Chitosan; Adsorption; Electrochemical sensitivity; Hydrogen Sulfide

1. INTRODUCTION

Recently, the use of “green materials” has proved to be a new trend in microelectronics for applications such as electronic devices, solid-state batteries, and sensors [1, 2]. Chitosan is appearing as a promising substitute for synthetic polymers in electrochemical devices because of its many peculiar properties and characteristics, such as its use in hosting ionic conductivity in batteries, dye-sensitized photovoltaics, supercapacitors, and fuel cells.

Moreover, many chitosan-based materials demonstrate electrical conductivity; it is very useful for chemical modifications to produce sensors and electrochemical devices, and also presents good interaction with different ions. All these properties allow the development of a wide variety of applications. Chitosan is a natural biodegradable, non-toxic, and biocompatible polymer. It is good for thin composite film fabrication owing to its superior film-forming properties and its capability to bind cationic and anionic forms of transition and noble metal ions [3-5].

The air surrounding us contains different species and amounts of harmful gases and atmospheric pollutants that come from medical or industrial operations. Therefore, it has become necessary to identify the presence of these gases since the environment consists of plants, animals, and humans, and maintaining their lives and safety is a top priority. Hydrogen sulfide (H₂S) is known as a harmful environmental pollutant. It is a product of decaying matter, colorless, corrosive, toxic, flammable, and explosive gas with the specific odor of rotten eggs. It exists in hot springs, volcanic gases, natural gas, crude petroleum, and groundwater. It is very toxic to plants and living organisms. When its concentration reaches 10 ppm in the air, it can affect human health [6, 7].

There are various technologies used for gas detection. Methylene blue colorimetric and iodometric titration tests have been the most popular means of measuring H₂S [8, 9]. Metal salts and metal oxide semiconductors have recently been used as materials in many gas detection methods [10-16]. The efficiency

characteristic of any detector is built on some properties such as selectivity, sensitivity, detection limit, recovery time, and response time [17].

There are simple electrochemical highly sensitive methods for in situ detection without the need for exogenous reagents [18]. In this work, we aim to detect hydrogen sulfide gas by designing a copper-loaded chitosan film. First, we loaded the copper metal ion onto the surface of the chitosan film by the adsorption method. After that, the copper-loaded film was investigated as a hydrogen sulfide gas detector electrochemically.

2. MATERIALS AND METHODS

2.1 Materials

Chitosan (Cs) with a deacetylation degree of 70-95% was purchased from Fluka. Copper sulfate pentahydrate was obtained from Nasr Company (Egypt). All other chemical reagents, such as glacial acetic acid, ferric sulfide, HCl, and glycerol, were of analytical reagent grade and used as purchased without further purification.

2.2. Characterization

Scanning electron microscopy (SEM) was employed on the dried samples to indicate the morphology of the films. This was done using SEM (FEI inspect 5 with EDX Unit, Holland) with an operating voltage of 20-30 KV, and the existence of elements was measured quantitatively by the EDX Unit. The dried samples were subjected to fracturing followed by sputtered gold thin coating before taking the SEM photos to provide electrical conductivity for the scanned samples. X-ray diffraction (XRD) was carried out to examine the crystalline phases of the films on a Shimadzu XD-1 diffractometer using a Cu K α radiation wavelength of 1.5406 Å at a beam voltage of 40 kV and a 40 mA beam current. The diffractograms were recorded in the 2 θ range of 4 – 89.9° with a 2 θ step size of 0.02 Å and a step time of 0.60 s. The Joint Committee on Powder Diffraction Society (JCPDS) database was used to index the peaks of XRD.

2.3. H₂S gas detection experiments

The pure chitosan-based films were connected to the electrical circuit as displayed in Fig. 1a and then exposed to H₂S gas for 1.0 h at a rate of 1.0 ml gas/36 seconds. The H₂S gas was produced in a closed system by adding hydrochloric acid to ferric sulfide. Then, the resistivity and conductivity of the film were followed. The films were prepared as follows: 0.5 gm of pure Cs was solubilized in 50 ml of acetic acid solution (1.0 w/v) at room temperature and under magnetic stirring for 3.0 h. The films were obtained by putting a 2.0 ml solution of pure chitosan on a slide of glass having an area of 6.25 cm². After that, the films were sprayed with glycerol then dried for 24 h. The copper-loaded films were prepared from pure Cs with copper ions. The dried films were moved to a beaker holding 10 mL of 0.05 mol/L CuSO₄.5H₂O aqueous solution and immersed for 2.0 h. The films were then dried at room temperature.

2.4. Resistor–capacitor circuit (R-C circuit) tests:

R-C circuit is an electric circuit possessed of a resistor (R) and a capacitor (C) driven by a current or voltage source. The simplest R-C circuit contains a charged capacitor and a resistor linked to each other in a single loop with no outer source of voltage. Once the circuit is closed, the capacitor starts to liberate its stored energy across the resistor. The voltage through the capacitor is time-dependent. The current across the resistor must be reversed in sign (but equal in magnitude) to the time derivative of the cumulative charge on the capacitor. In this electrical circuit, the resistor was changed by the samples under investigation (Fig. 1b).

2.5. Electrical impedance spectroscopy (EIS) tests:

This technique measures the system impedance over a variety of frequencies, and therefore the frequency response of the system, including the storage properties and energy dissipation, is revealed. The impedance spectra

were attained in the frequency variety of $10^4 - 0.2$ Hz with a 10 mV amplitude sine wave at OCP using a Gamry 3000 potentiostat. To achieve the quantitative information for the discussions of these experimental EIS results, a suitable model (Randles) for equivalent circuit quantification was used.

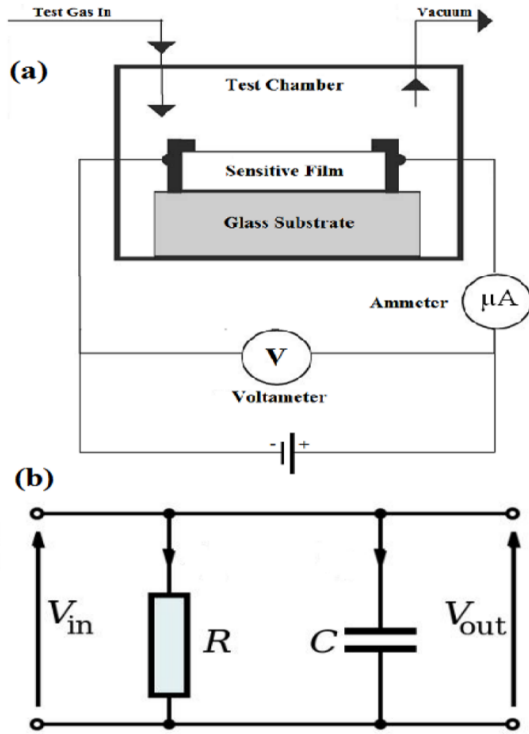


Fig. 1. Resistance and conductivity circuit for measuring adsorption of H₂S gas (a), R-C circuit (b)

3. RESULTS AND DISCUSSION

3.1. Electrochemical resistivity, conductivity, and gas sensitivity

The electrical resistivity of materials is the resistance of an electric current flow, with some materials resisting the current flow more than others. While a conductor's resistance provides the amount of opposition, it presents the electric current flow. The conductance of a conductor indicates the ease with which it allows the flow of the electric current. The conductivity is the efficiency with which a conductor passes a signal or electric current without resistive loss. Thus, a conductor or material that has a high conductivity will have a low resistivity, and vice versa. The conductivity and resistivity can be calculated from the following equations:

$$\rho = \frac{R \times A}{L} \tag{1}$$

$$\sigma = 1/\rho \tag{2}$$

Where R is the resistance in ohms (Ω), A is the area in square meters (m^2), L is the length in meters (m), the proportional constant ρ is the resistivity, and σ , is the conductivity.

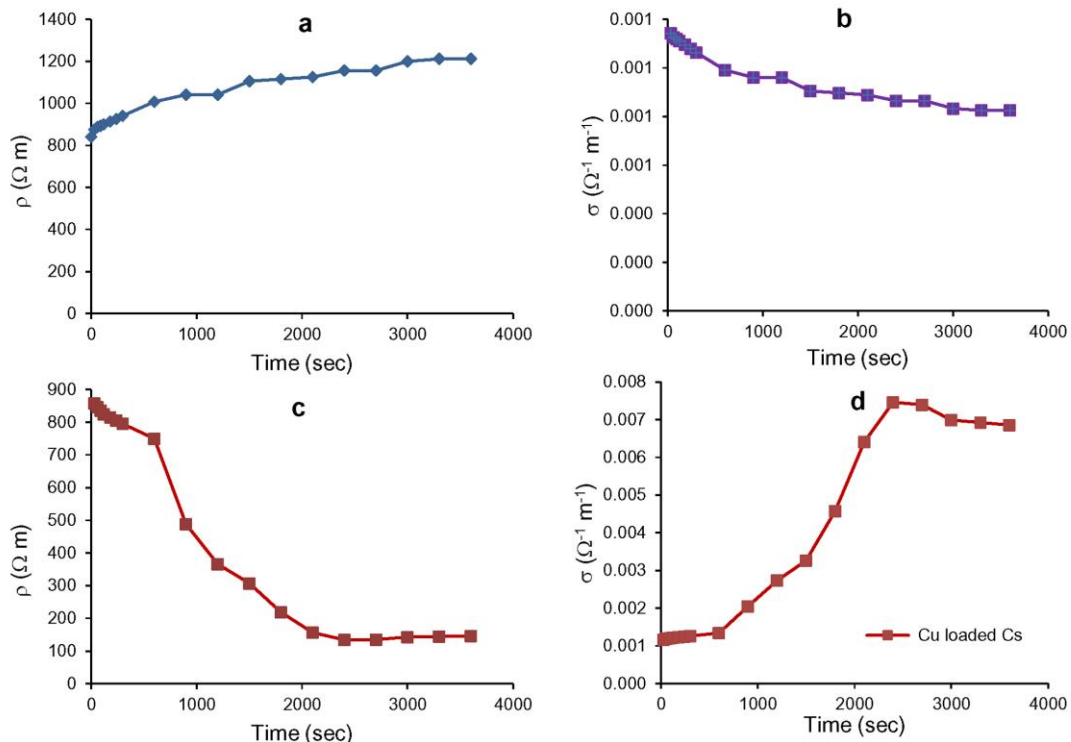


Fig. 2. Above (a) Resistivity and (b) conductivity of Cs and below (c) Resistivity and (d) conductivity of Cu-loaded Cs vs time after adsorption of H₂S gas.

3.1.1. Comparison of the electrochemical sensor of the pure chitosan films to the hydrogen sulfide and copper-loaded chitosan

The response of the obtained films (chitosan and Cu-loaded chitosan) to detecting the hydrogen sulfide gas was measured by electrical resistivity and conductivity measurements against time at 25 °C. The obtained results are displayed in Fig. 2. A slight increase in the resistivity of chitosan was observed on exposure to H₂S gas from 875 to 1211 Ωm after 3600 seconds, consequently a slight decrease in the electrical conductivity as displayed in Fig. 2. This behavior can be explained by the fact that the pure Cs films were acidified with hydrochloric acid and that acid has high conductivity, while hydrogen sulfide has lower conductivity. Therefore, when hydrogen sulfide gas is adsorbed on the acidified films, a mixture happens between H₂S and HCl, which leads to a decrease in conductivity and the resistivity increasing.

The determination of the electrochemical resistivity and conductivity of Cu-loaded chitosan films mainly results in the investigation of their sensitivity and selectivity to electrical responses when exposed to H₂S gas. A significant decline in the resistivity from 857 to 145 Ωm for Cu-loaded chitosan was observed, and a significant increase in electrical conductivity from above 0.001 to 0.0075 Ω⁻¹m⁻¹ for Cu-loaded chitosan, as presented in Fig. 2.

The large increase in the electrical conductivity is owing to the adsorption of gas onto the copper-loaded chitosan surface, and then a chemical reaction that occurs between hydrogen sulfide gas and copper ions on the surface of the films to form copper sulfide, which has a high conductivity. The increase in conductivity was followed by a slight decrease, which may be owing to the occurrence of physical adsorption of the hydrogen sulfide gas on the copper-loaded chitosan surface after consumption of the amount of copper ion loaded on the surface, which led to this decrease until reaching a state of stability which expresses the saturation of the films with the gas.

3.2. Resistor–capacitor circuit (R-C circuit) tests:

From the R-C circuit, it is possible to obtain the time constant (τ) of the circuit, which is the time required for the voltage on both sides of the capacitor to reach 0.37 of its maximum value, from the following relationships [19, 20]:

$$V_c = V_o e^{-\frac{t}{RC}} \quad (3)$$

$$\ln V_c = \ln V_o - \frac{t}{RC} \quad (4)$$

$$\tau = RC \quad (5)$$

Where V_o and V_c were initial and variable voltages, respectively, C is the capacity of the capacitor (220 μF, 50 V), R is the resistance of the sample and t is the time.

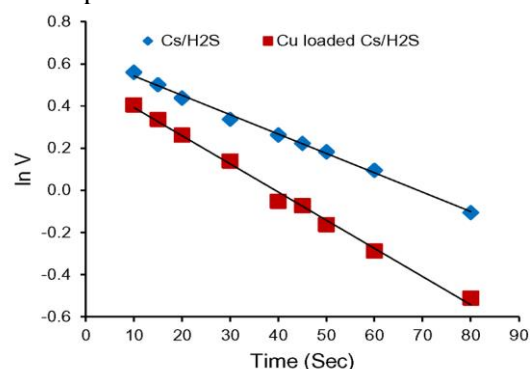


Fig. 3. R-C diagrams of Cs/H₂S and Cu-loaded Cs/H₂S.

The difference in potential between the two sides of the capacitor was measured at time intervals and the relationship between $\ln V_c$ with time was drawn to find the time constant (Fig. 3), and the results obtained are recorded in Table 1. By comparing the results listed in Table 1, it was noticed that the resistance values and the time constant vary between the films under study. Increasing the electrical conductivity of the films is due to a decline in the time constant and resistance values.

3.3. Electrochemical impedance spectroscopy (EIS) tests

Impedance is a measure of the ability of a circuit to counter the electrical current flow. The electric current loses some of its energy, whether in the form of stored energy as in capacitors or wasted energy doing work as in

resistors. Impedance with respect to alternating current is the resistance with respect to direct current.

The impedance value relies on the frequency because the inductance values of the inductor and the capacitance of the capacitor change with the change of the frequency. Unlike resistance, it is a constant value regardless of the frequency.

Table 1: R-C and EIS parameters of polymer samples

Sample	R-C test				EIS
	V_o		RC	R (k Ω)	R_{ct} (k Ω)
	V_o (Exp.)	(Calc.)			
Cu-loaded Cs/H ₂ S	2.0	1.69	74.8	340.0	173.1
Cs/H ₂ S		1.89	108.8	494.6	538.5

Impedance spreads the idea of resistance to alternating current (AC) circuits and owns both phase and magnitude, despite resistance, which has only magnitude. When a circuit is paid with direct current (DC), there is no difference between resistance and impedance; the latter can be assumed as impedance with zero phase angles.

The data was attained by electrochemical impedance spectroscopy (EIS) in the air for the Cs/H₂S, and Cu-loaded Cs/H₂S films. The data are expressed graphically in a Nyquist plot. Fig. 4 displays the Nyquist plots of H₂S adsorbed on the Cs film with and without loading of copper ions. The complex impedance appears as the sum of the real (Z_{real}) and imaginary (Z_{imag}) components. The broad semicircle is owing to a combination of resistive and capacitive elements of the films. The diameter of the semicircle for the Cu-loaded Cs/H₂S films is low at about 85 k Ω , whereas the corresponding value for pure Cs/H₂S film is about 280 k Ω , respectively. These data suggest the greater electrochemical activity of Cu-loaded Cs/H₂S film, and this is also evident from the values of the charge transfer resistance (R_{ct}) [21], which is shown in Table 1.

3.4. X-ray diffraction (XRD)

The nature of Cs, Cu-loaded Cs, and Cu-loaded Cs/H₂S films is determined using an X-ray diffraction pattern. As displayed in Fig. 5, the chitosan has low crystallinity with a

reflection peak at $2\theta = 23^\circ$ (22). The Cs film loaded with copper ions showed peaks at [$2\theta = 11.1^\circ, 20.2^\circ, \text{ and } 28.9^\circ$] which indicate the presence of Cu. The Cu-loaded Cs/H₂S film found peaks at [$2\theta = 11.3^\circ, 20.4^\circ, 67.5^\circ, \text{ and } 68.2^\circ$] which show the existence of Cu_xS [22].

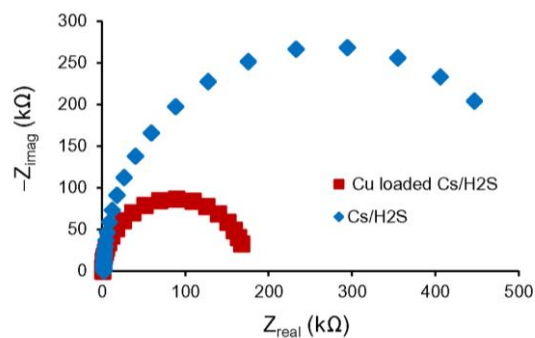


Fig. 4. Nyquist diagram of Cs/H₂S and Cu-loaded Cs/H₂S.

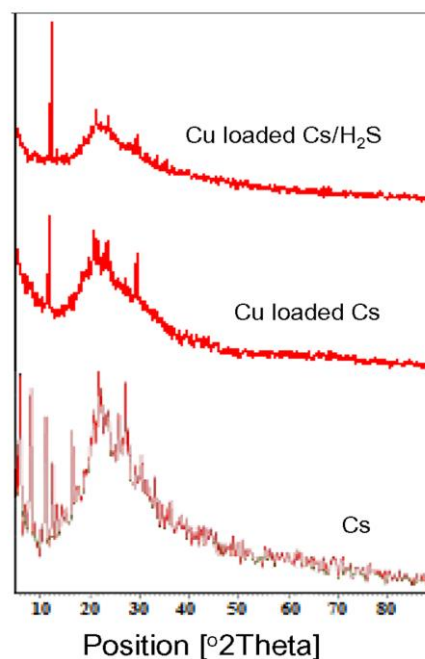


Fig. 5. XRD of the Cs, Cu-loaded Cs, and Cu-loaded Cs/H₂S films.

3.5. Energy dispersive X-ray (EDX)

To confirm the existence of the elements on the surface of the films, EDX was used to determine the presence of copper sulfide and copper ions in the pure chitosan films. The three elements of O, N, and C which were shown in each film were the component elements of chitosan as described in Figs. 6a, respectively. There was an absorption peak of sulfur and copper in the EDX film of the Cu-loaded Cs, suggesting the presence of Cu and sulfur in the film, which corresponds with the

slightly blue color of the Cu-loaded Cs film as described in Figs. 6b. The obtained sulfur is owing to the existence of sulfur in the copper sulfate. There was an absorption peak of Cu and S in the EDX film of the Cu-loaded Cs/H₂S, indicating the presence of Cu and S in the film, which corresponds with the slightly black color of the copper-loaded Cs/H₂S film as displayed in Fig. 6c.

3.6. Scanning Electron Microscope (SEM)

Figs. 7a-c show SEM photographs of pure Cs, Cu-loaded Cs, and Cu-loaded Cs/H₂S films, respectively. The SEM images of the Cs indicated the nonporous irregular-shaped particles on the polymeric backbone structure. The successful loading of copper ions onto the chitosan structure has also been confirmed by slight brightness grains. The SEM image of the Cu-loaded Cs/H₂S film (Fig. 7c) indicates the huge bright homogenous distribution.

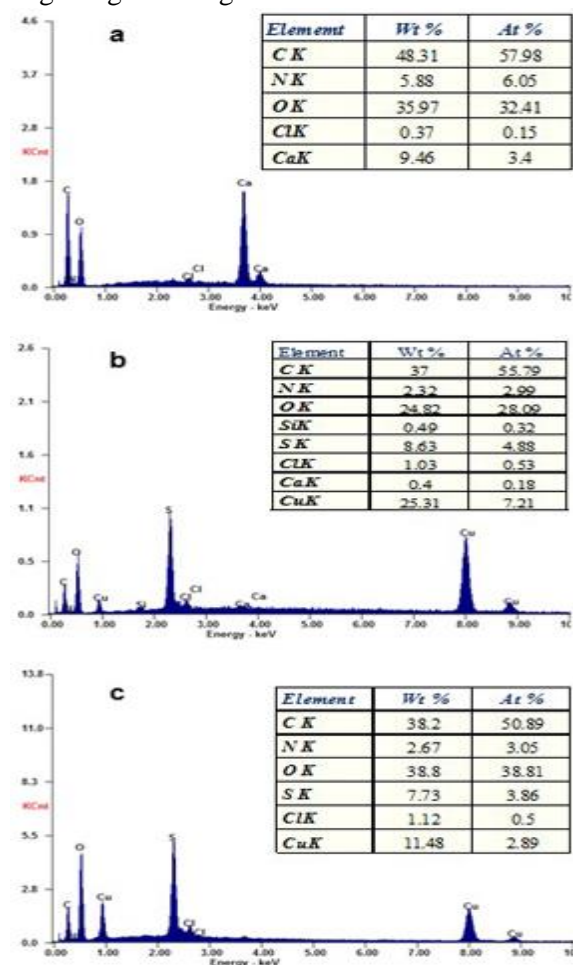


Fig. 6. EDX of (a) pure Cs, (b) Cu-loaded Cs, and (c) Cu-loaded Cs/H₂S.

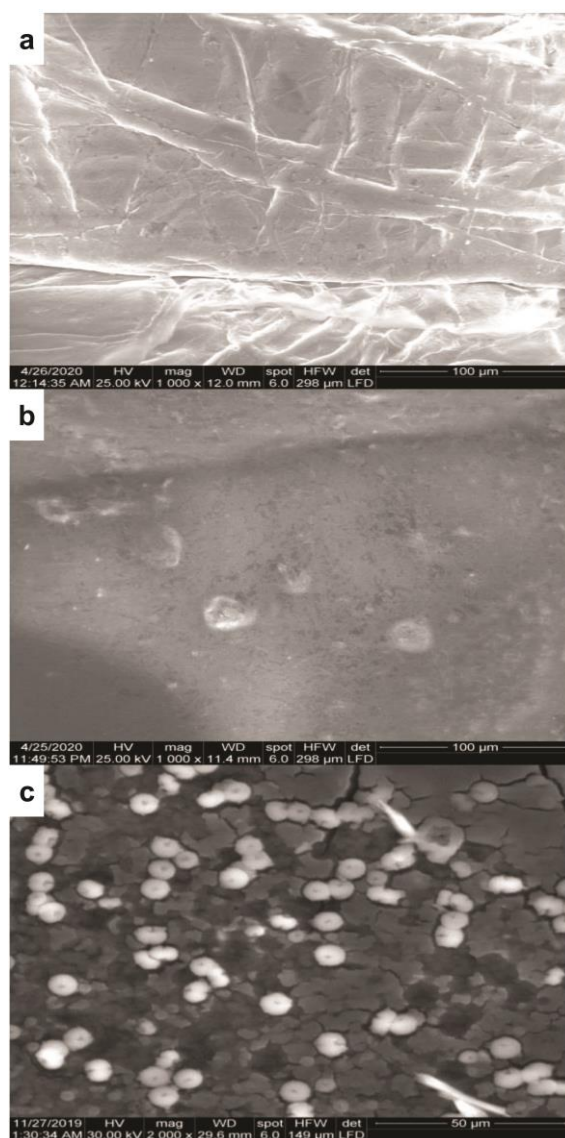


Fig. 7. SEM images of (a) pure Cs, (b) Cu loaded Cs, and (c) Cu loaded Cs/H₂S.

CONCLUSION

In the present work, films from Cs were loaded by Cu (II) and the loading process was successfully evidenced by SEM and EDX analyses. The ability of the Cu-loaded films to detect H₂S gas was investigated through electrochemical resistivity and conductivity measurements. It was found that the designed Cu-loaded pure chitosan films display high conductivity and consequently high response on exposure to H₂S gas. The R-C results confirmed that there is a significant change in the resistance values and the time constant between the films under study. The Impedance result, depicted as the Nyquist plot, confirmed the greater electrochemical activity of the Cu-loaded Cs/H₂S film. The existence of Cu_xS on

the chitosan film was indicated by XRD, SEM, and EDX. From the results, it can be concluded that the Cu-loaded chitosan is a promising material for sensing H₂S gas.

ACKNOWLEDGMENTS

We are grateful to the Faculty of Science, Al-Azhar University, Department of Chemistry, Cairo, Egypt for their continuous support during this work period.

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إمتصاص أيونات النحاس على الأغشية المستندة إلى الكيتوزان للكشف عن ملوث غاز كبريتيد الهيدروجين

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، القاهرة ، مصر

ملخص عربي

من المعروف أن الكبريتيد في شكله الحر لكبريتيد الهيدروجين يسبب القلق عند تركيزه المنخفض ولكن عند تركيزه الأعلى، فإنه يسبب تلفاً دائماً للدماغ أو الإغماء أو الموت من خلال الاختناق. في هذه الدراسة تم تصنيع أغشية الكيتوزان وتحميل أيونات النحاس عليها عن طريق الامتزاز لاستخدامها في الكشف عن غاز كبريتيد الهيدروجين بطريقة كهروكيميائية. قدمت النتائج اختلافاً مذهلاً في المقاومة الكهربائية والتوصيلية لأغشية الكيتوزان والكيتوزان المحملة بالنحاس مع مرور الوقت استجابةً لامتصاص الغاز. أظهرت النتائج أيضاً زيادة ملحوظة في موصالية غشاء الكيتوزان / كبريتيد الهيدروجين المحمل بالنحاس مقارنةً بالكيتوزان النقي / كبريتيد الهيدروجين. علاوة على ذلك، تم فحص الأفلام بعد عملية امتصاص الغاز بعدة طرق، دائرة المكثف و المقاومة، مطيافية المعاوقة الكهروكيميائية، الفحص المجهرى لمسح الإلكترون، حيود الأشعة السينية والأشعة السينية المشتتة للطاقة. أشارت صور إلى التحميل الناجح لأيونات النحاس على هيكل الكيتوزان.