

## Electrochemical deposition of gelatin particles on electrode ITO thin films

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In this paper, an electrochemical study of Gelatin was carried out on the glass semiconductor ITO electrode in different media: acidic medium (sulfuric acid) and basic medium (sodium hydroxide solution), using chronoamperometric and cyclic voltammetry methods in order to obtain an electrochemical response of a substance Gelatine. In order to study the structural and optical properties of the film, we used several techniques, including the DRX technology and the Scanning Electron Microscopy (SEM).

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### 1. Introduction

A single-strand molecule known as gelatin is produced when collagen is partially hydrolyzed [1]. There are two forms of gelatin: type A gelatin is produced by an acidic pretreatment, while type B gelatin is produced by an alkaline pretreatment. Gelatin has special qualities that allow it to create thermo - reversible gels with melting points close to body temperature [2]. The production and quality of gelatin rely on the parameters of extraction (pretreatment, pH, temperature, and time), which may change the functional characteristics of the gelatin [3-5] and the collagen hydrolysis relies on the acid concentration utilized [6]. Gelatin thin films have been employed as coverings for safety. Such coatings are applied according on the kind of coating (organic or inorganic), the operating circumstances, and the necessary service life of the material while working. In order to create a high-efficiency solar cell, a thin gelatin coating is consequently necessary. For this, physical and chemical deposition methods are becoming more popular. They encompass a broad range of techniques such sputtering [7] chemical bath deposition [8] chemical vapor deposition [9] [10] or confined space sublimation [11] which calls for a low vacuum and high temperature. Electrodeposition technique presents a simple non complicated and non expensive method compared to some of the above techniques[12]. In comparison to traditional

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approaches, this strategy has a number of benefits, including cheap cost, extensive surface area coverage, low-temperature growth, and direct control over layer thickness, shape, and composition.

Coating methods frequently involve electrodeposition (ED) of polymers. At least two significant benefits of this approach include the ability to apply coatings to surfaces with complicated geometries and the production of coatings with consistent thickness. A large electrical equivalent weight of a coating is another characteristic of polymer electrodeposition [13], (As opposed to the electrodeposition of metal alloys). Colloid particles of sub micrometer and micrometer sizes have been extensively researched in electrodeposition (ED). [14] The production of film solar cells is ideally suited for the electrodeposition of such films on conductive substrates. In this study, we compared the gelatin films electrodeposited onto In-doped SnO<sub>2</sub> (ITO) coated glass substrates using cyclic voltammetry (CV) and a three-electrode cell. The films' structure and content.

In this study we, we demonstrated the possibility of electrodeposition of gelatin polymer- films on ITO electrode in different mediums and studying the characterizations.

## **2. Experimental section**

### **2.1. Apparatus**

Electrochemical experiments were performed with a CHI 660C electrochemical workstation (Shanghai Chenhua, China). Gelatine modified ITO were observed by field-emission scanning electron microscope (SEM, Philips XL 30 FEG) and X-ray diffractometer (GBC-EMMA, Australia) for determine. A three-electrode system was employed for electrochemical with gel/ITO as working electrode, an Ag/AgCl (3MKCl) electrode and a platinum wire as reference electrode and counter electrode, respectively. All potentials were referred to the Ag/AgCl (3 M KCl) electrode.

### **2.2. Preparation of the gelatin /ITO electrode**

To perform the experimental setup, we use a three-electrode electrolytic cell. We carried out a preliminary study of the two solutions by measuring the equilibrium voltage of the ITO working electrode by adding a sample of volume ( $V=12$  ml), then studied in the cell in order to draw the voltage curve in fonction of time (OPC) for 4 minute at room temperature, the electrochemical measurements have been carried out with a Volta lab PGZ301 system (Radiometer Analytical)

The solution contains mass of gelatin  $m = 6 \times 10^{-3}$ g, and sulfuric acid as electrolyte ( $V = 12$  ml,  $C = 0.00990$  M). Electrodeposition of gelatin onto ITO was conducted in the electrolyte consisting of  $0.009$  M H<sub>2</sub>SO<sub>4</sub> solution, ITO as working electrode, a Ag/AgCl electrode as reference electrode, and a platinum wire as counter electrode. The ITO/gel electrode was first polarized at constant potential of  $1$  V for different time, and then taken out from the deposition solution.

### **2.3. Thin film characterizations**

Various structural parameters of the gelatin developed on ITO coated glass substrates were investigated by X-ray diffractometer (GBC-EMMA, Australia) at  $2\Theta$  position (from  $20^\circ$  to  $90^\circ$ ) with CuK $\alpha$  X-ray of wavelength  $\lambda = 1.54056$  Å.

SEM characterizations were carried out by FESEM (JEOL JSM-7600F, Japan) at  $5$  kV to  $7$  kV accelerating voltage.

### 3. Results and discussions

#### 3.1. Electrochemical study of gelatin on the electrode ITO

The value of the equilibrium potential in an open circuit case is shown for a solution of  $H_2SO_4$ /Gelatin that contains a Gelatin solute, a solvent and a  $H_2SO_4$  co-electrolyte equal to:

$$E = 358 \text{ mV/ECS.}$$

We study the changes of electric current in terms of the Gelatin potential to follow the electrochemical behavior of a substance, applied with the voltametric cyclic technique during a certain period of electrolysis in different media (acidic - basic) using the Voltabal device with the selection of the scanning field and speed at room temperature and the results obtained are recorded in the curves.

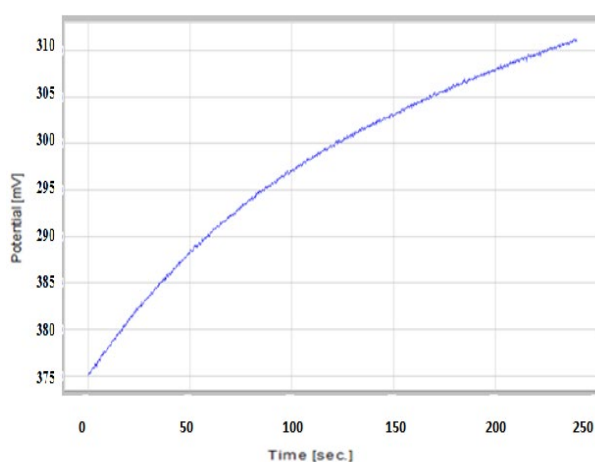


Fig. 1. Curve of the electrolyte solution( $H_2SO_4$ ) for  $t = 250s$ .

The value of the equilibrium potential ECS is shown in an open circuit of the electrolyte containing the solvent and electrolyte  $H_2SO_4$  equal to /ECS.  $E = 340\text{mv}$ .

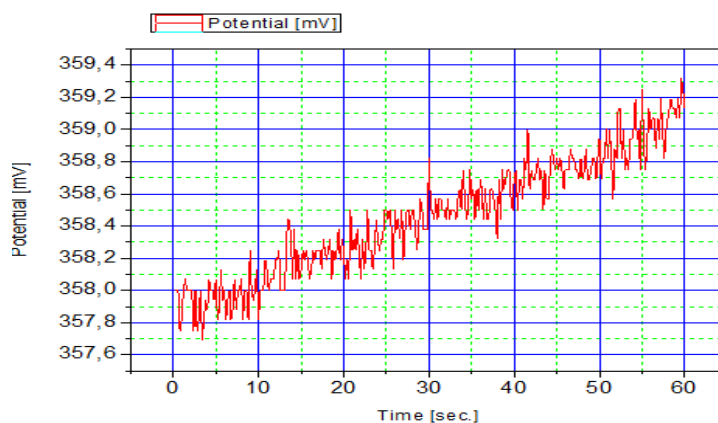


Fig. 2. Curve of  $H_2SO_4$ /Gelatin solution for  $t=60s$ .

Table 1. The values of the equilibrium potential ECS in an open circuit in acid medium.

Medium	Open circuit potentiel $E_{eq}(mV)$
H <sub>2</sub> SO <sub>4</sub>	340
H <sub>2</sub> SO <sub>4</sub> /gel	358.8

### 3.2. Cyclic voltammetric method

#### 3.2.1. Acid medium

At the beginning of the study the H<sub>2</sub>SO<sub>4</sub> without gelatin in the rang - 650 mv to 600 mv as shown in Figure 3, after that we carried out the study of the H<sub>2</sub>SO<sub>4</sub>/ Gelatin solution in the range 500 mv to -500 mv as shown in Figure 4, no redox protrusion was observed. Accordingly, the range 500 mv was expanded to - 700 mv to show in Figure 5, two reactions of reduction  $E = -450$  mv and oxidation  $E = 0$  mv, where an increase in current is noticed before the peak of the reduction and then it decreases and the surface of the electrode changes due to the interactions that take place.

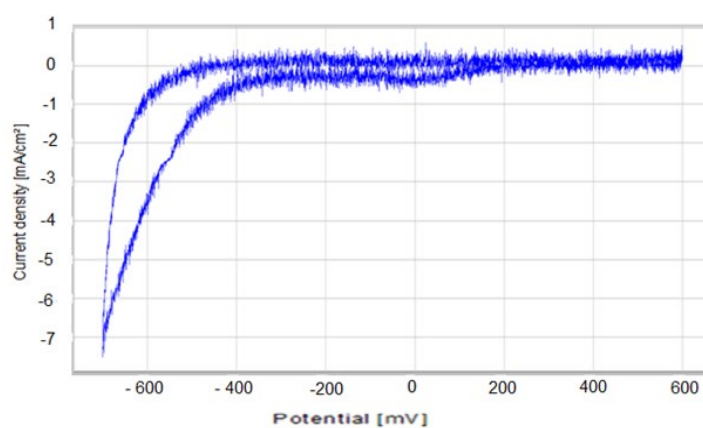


Fig. 3. The Cyclic voltammetric curve of study the solution 1M of H<sub>2</sub>SO<sub>4</sub> on the ITO electrode at a range of -700mv to 600 mv at a scanning speed of  $V = 30$  mv/s.

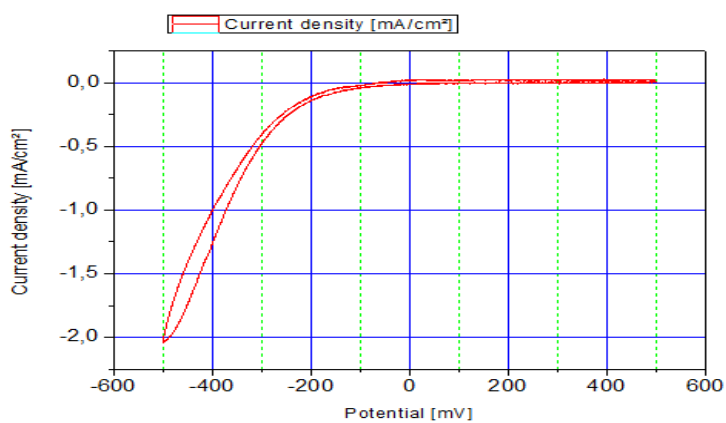


Fig. 4. The Cyclic voltammetric curve to study the  $H_2SO_4$ /Gelatin solution on the ITO electrode at a range of -500mv to 500 mv at a scanning speed of  $V = 30\text{mv/s}$ .

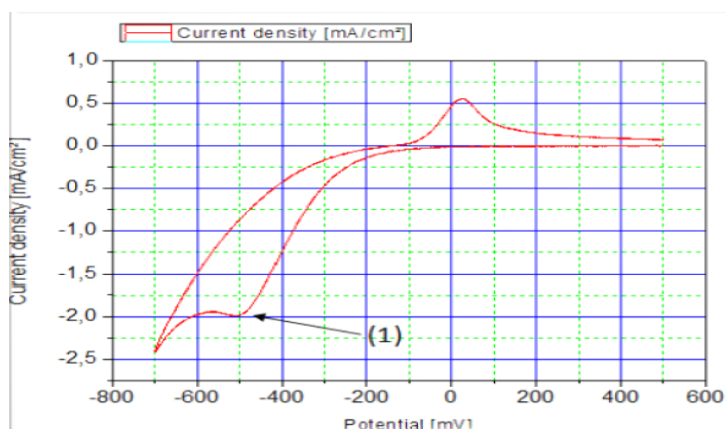


Fig. 5. Cyclic voltammetric curve of  $H_2SO_4$ /Gelatin solution on ITO electrode at range 500 mv to - 700 mv at scan speed  $V=30\text{mv/s}$ .

After giving an electrochemical response to gelatin in the acid medium at a scanning speed  $V=30\text{mv/s}$ , we changed the scanning speed in order to observe their effect on the reduction and oxidation peaks. The results obtained through Figure 6.

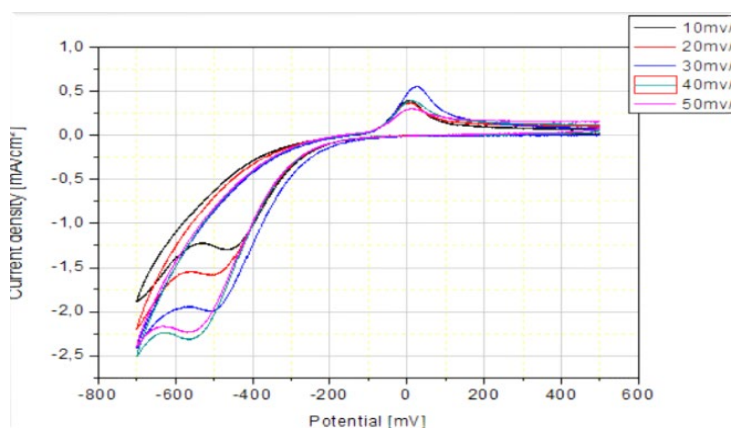


Fig. 6. The cyclic voltammetric curve for the electrochemical study of the  $H_2SO_4$ /Gelatin solution on the ITO electrode at the range from 500 mv to 700 mv at scanning speed mv/s [10, 20, 30, 40, 50] mV.

We note that the increase in scanning speed has a direct effect on the reduction and oxidation peaks. In Figure 7, the current intensity changes in terms to the root of scanning speed.

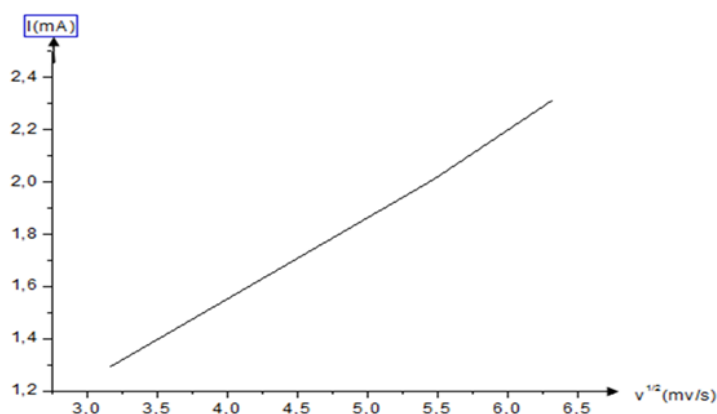


Fig. 7. The trend of changing the current intensity in terms of the root speed.

After tracking the electrochemical behavior of Gelatin in an acidic medium, the results showed an electrochemical response. Therefore, we discussed conducting this study in a basic medium in order to compare the results obtained in both mediums.

### 3.2.2 Acid medium

We prepared a NaOH/Gelatin solution containing a dissolved mass of Gelatin ( $m=6 \times 10^{-3}$ g) and NaOH ( $V=12$  ml,  $C=0.01$ M) and the auxiliary electrolyte NaOH, then measure the value of the equilibrium potential of the prepared solution with the working electrode ITO by drawing the voltage curve in terms of time Figure 9.

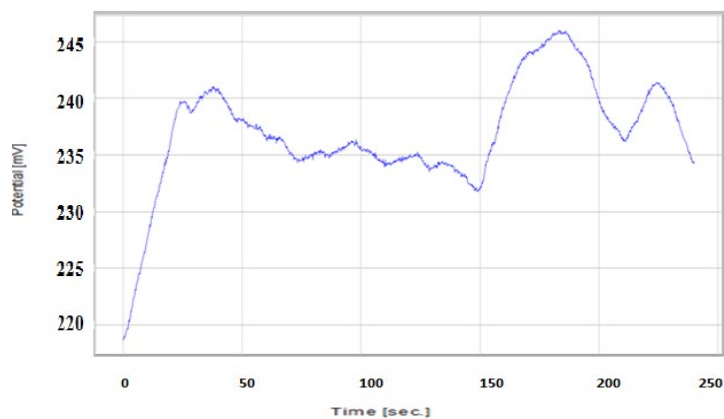


Fig. 8. Curve of the electrolyte solution (NaOH) for  $t=60s$ .

The value of the equilibrium potential in the case of an open circuit of the NaOH / Gelatin solution is shown as ECS ,  $E= 222$  mv.

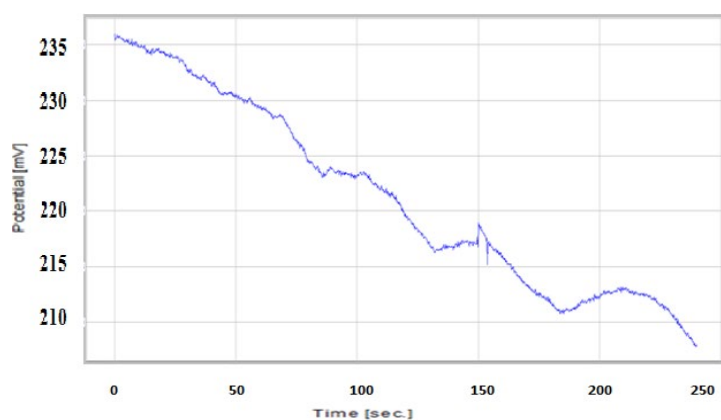


Fig. 9. The curve of the NaOH / Gelatin solution for a period of  $t = 250s$ .

Table 2. The values of the equilibrium potential ECS in an open circuit in basic medium.

Medium	Open circuit potentiel $E_{eq}(mV)$
NaOH	240
NaOH/gel	222

After finding the value of the equilibrium voltage, the electrochemical activity field is determined at a scanning speed of  $v = 30$  mV/s at the same room temperature, at the range from -1200 mV to -300 mV in Figure 10.

It is noted that the cathodic and anode peaks do not appear, so it was reduced to the range from 0 mV to -1500 mV in Figure 10, where an electrochemical response was observed, and then 50 loops were recorded in Figure 11, for the previous solution, with the field reduced again at -300 mV to 1500 mV.

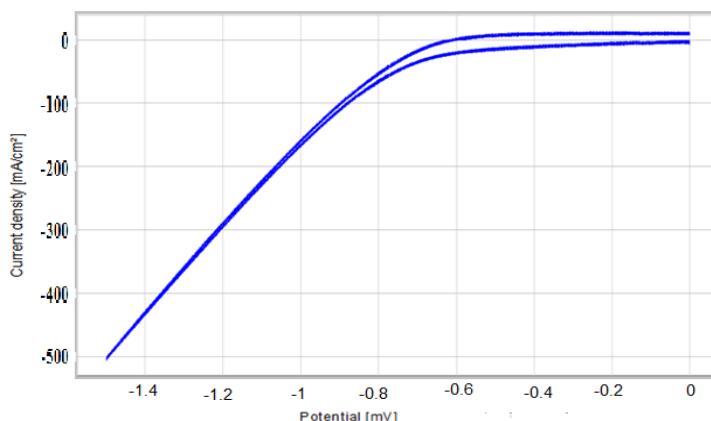


Fig. 10. The Cyclic voltammetric curve of study the NaOH, 1M solution on the ITO electrode at a range of -500mV to 500mV at a scanning speed of  $V = 30$  mV/s.

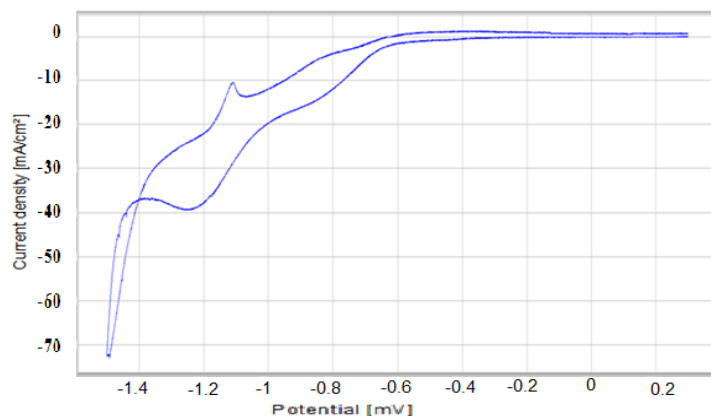


Fig. 11. Cyclic voltammetric curve of NaOH/Gelatin solution on ITO electrode at range 300 mV to -1500 mV at scan speed  $V=30$  mV/s.



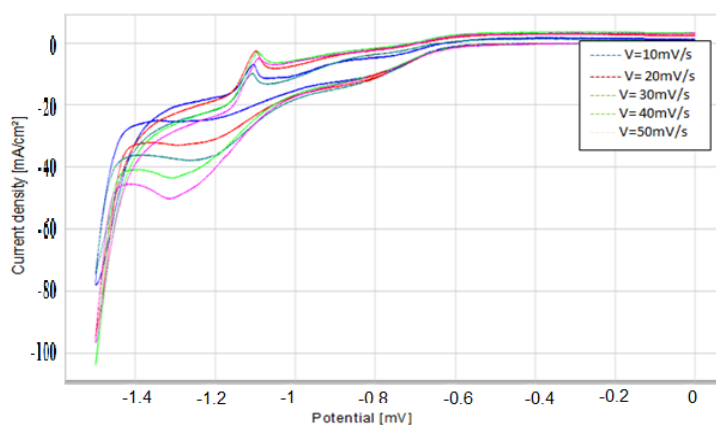


Fig. 12. The Cyclic voltammetric curve for the electrochemical study of the NaOH/Gelatine solution on the ITO electrode at the range from 500 mv to 700 mv at scanning speed mv/s [10, 20, 30, 40, 50]V.

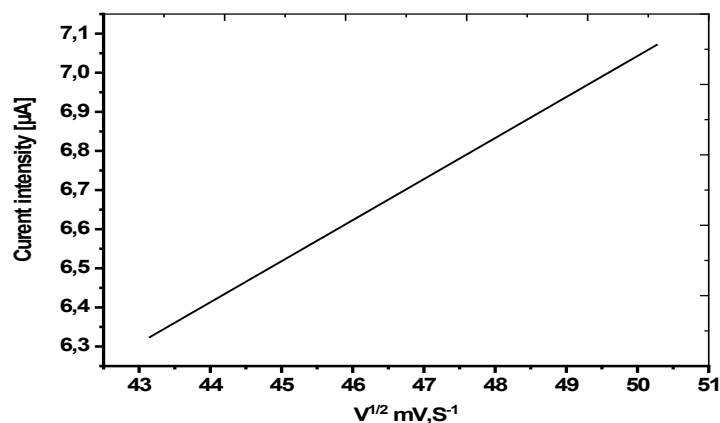


Fig. 13. The trend of changing the current intensity in terms of the root speed.

In Figure 12 an increase in the cathode peak was observed, then it gradually decreased, and this is explained by the deposition of Gelatin until it covered the entire surface of the electrode, which reduces the response of the modified electrode and the conductivity decrease.

Following the results obtained during the electrochemical study of an acidic medium and a basic medium through the previously shown curves.

### 3.3. Properties studies

The analysis was carried out using X-rays using the PROTO (AXRD) device on the modified electrodes prepared with different electrolytes.

The ITO electrode and the modified ITO/gelatin electrode were studied by X-ray machine. The study of the structural properties of thin layers depends on the analysis using X-rays, as this study allows to provide a picture of the structure and quality of the studied material, by measuring the intensity in terms of the diffraction angle ( $2\theta$ ).

### 3.2.1. XRD result

Figure 14 represents a spectrum of gelatin between 10 to 80 degrees. Several peaks at  $2\theta$  are observed at 41, 62 and 75 which correspond to the diffraction levels of (200), (220), (102) and (311) respectively of the gelatin phase according to the JCPDS card [16]. The results confirm that the prepared product has clearly a visible structure particles. Moreover, it appear as fine white particles. It is crystallized and can be used effectively to prepare our gelatin layers.

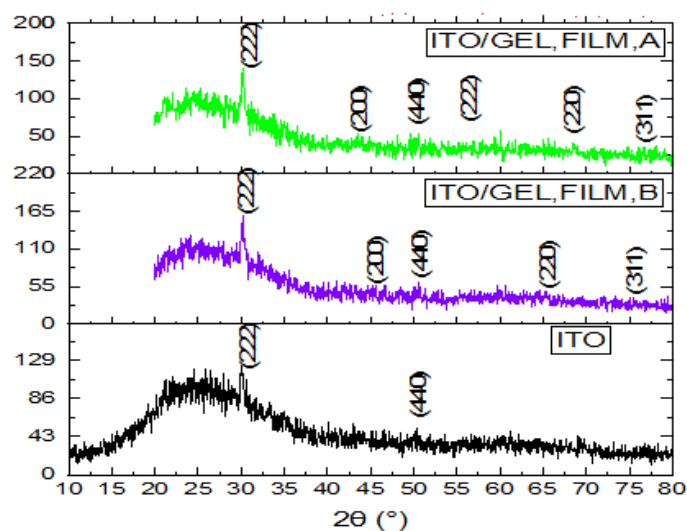


Fig. 14. X-Ray Results ITO, ITO/Gelatin Acidic and basic medium.

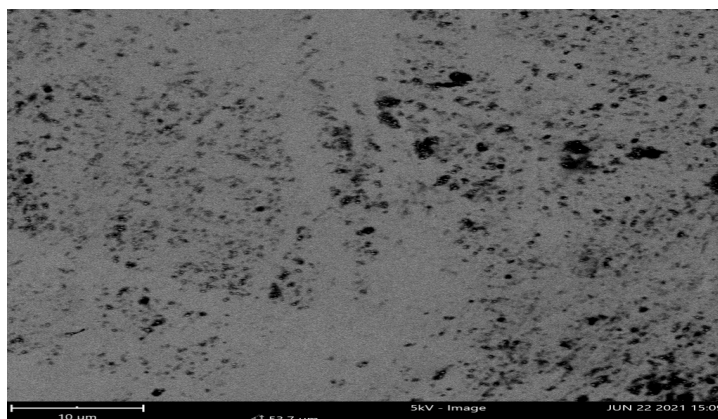
Table 3.  $hkl$ ,  $2\theta$ ,  $d_{hkl}$ , lattice parameters, and grain size of the deposited gelatin layers.

medium	(hkl)	$2\theta$ (°)	Calculated $d(\text{Å})$	Grain size (nm) XRD	$D_{\text{moy}}$
ITO/gelatin Acid medium	200	41.191	3.0368	17.68837754	31.65740932
	220	62.904	1.476	34.63948708	
	311	75.71	1.25524	42.64436334	
ITO/gelatin basic medium	200	41.13217	1.17102	17.68497163	31.54503661
	220	62.875	1.47689	34.63948708	
	311	74.538	1.27205	42.31065113	

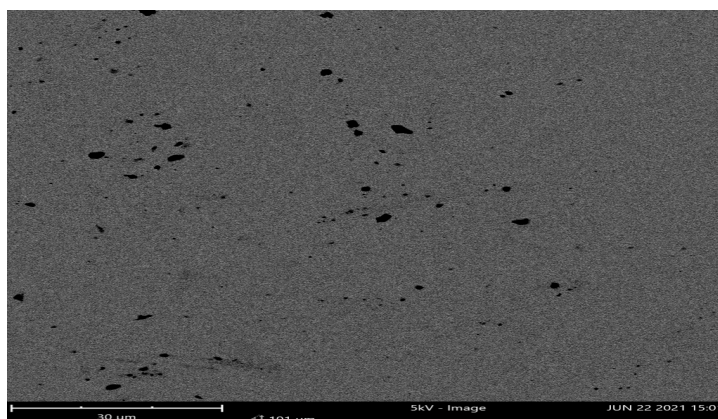
### 3.2.2. SEM result

Morphological examinations of gelatin deposits on ITO were analyzed by the SEM to confirm the electrochemical results, and obtain structural information on the influence of gelatin on the nucleation process.

The formation of crystallites is observed and regular. The free water in the gelatin microspheres was frozen, which caused the polymer chains to gather and condense. Gelatin as an additive modifies the nucleation mode, thus identifying instantaneous nucleation and enabling smaller grains. Therefore, gelatin has a grain refining effect as well as leveling properties on electrodeposits of alloy.



*Fig. 15. SEM image of gelatin deposited in basic medium.*



*Fig. 16. SEM image of gelatin deposited in acid medium.*

#### **4. Conclusion**

In this work, gelatin films were electrochemically deposited on ITO by CV and CA techniques from an aqueous solution gelatin– electrolyte.

An electrochemical study of solutions containing a mass of gelatin in different media was carried out on the ITO carrier glass electrode, using the cyclic voltammetry and chronoamperometric method at certain experimental conditions.

Despite the different media, the gelatin material gave an electrochemical response in the acidic medium, the base medium and the mixture.

When the scanning speed is increased, the results showed through the toroidal voltammetric curve a direct proportion between the intensity of the current and the speed and this is due to the occurrence of a rapid interaction.

When determining the crystalline properties, the X-ray diffraction scheme was studied on the ITO conductor glass electrode, and several peaks at 2 degrees are 41, 62 and 75 that correspond to the diffraction levels (200), (220), (102) and (311) respectively from the phase Gelatin according to the

JCPDS label. The results confirm that the prepared product has a crystalline structure and can be used effectively to prepare gelatin layers.

After determining the optimum experimental properties from the field of study and the electrochemical method for preparing the solutions, we hope in the future to work on expanding research on this type of electrochemical studies and their biological and technological applications to form a more accurate idea about the development of their use.

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### References

- [1] P. I. Rose, H. F. Mark, N. M. Bikales, C. G. Overberger, G. Menges, & J. I. Kroschwitz (Eds.), Encyclopedia of polymer science and engineering. New York: Wiley-Interscience. (1999).
- [2] M. H. Norziah, A. Al-Hassan, A. B. Khairulnizam, M. N. Mordi, & M. Norita, Food Hydrocolloids , 23(6), 1610-1616, (2009); <https://doi.org/10.1016/j.foodhyd.2008.12.004>
- [3] M. Alshafiee, M. K. Aljammal, D. Markl, A. Ward, K. Walton, L. Blunt, International Journal of Pharmaceutics, 559, 245-254, (2019); <https://doi.org/10.1016/j.ijpharm.2019.01.038>
- [4] S. R. Derkach, N. G. Voron'ko, Yu A. Kuchina, Carbohydrate polymers. vol. 197, p. 66-74. (2018); <https://doi.org/10.1016/j.carbpol.2018.05.063>
- [5] K. Ilona, K. Krzysztof, P. Barbara, Food Chemistry, vol. 86, no 2, p. 203-209, (2004); <https://doi.org/10.1016/j.foodchem.2003.08.036>
- [6] L. Wang, M. A. E. Auty, A. Rau, J. F. Kerry, & J. P. Kerry, Journal of Food Engineering. 90(1), 11-19. (2009); <https://doi.org/10.1016/j.jfoodeng.2008.04.030>
- [7] P. K. Ghosh, U. N. Maiti and K. K. Chattopadhyay, Materials Letters, vol. 60, no 23, p. 2881-2885, (2006); <https://doi.org/10.1016/j.matlet.2006.02.007>
- [8] H. Zhang, X. Ma, J. Xu and D. Yang, Journal of Crystal Growth, vol. 263, no 1-4, p. 372-376, (2004); <https://doi.org/10.1016/j.jcrysgro.2003.11.090>
- [9] H. Uda, H. Yonezawa, Y. Ohtsubo, M. Kosaka and H. Sonomura, Sol. Energy Mater, vol. 75, no 1-2, p. 219-226, (2003); [https://doi.org/10.1016/S0927-0248\(02\)00163-0](https://doi.org/10.1016/S0927-0248(02)00163-0)
- [10] S. H. Yoon, S. S. Lee, K. W. Seo and I. W. Shim, Bulletin of the Korean Chemical Society, vol. 27, no 12, p. 2071-2073, (2006); <https://doi.org/10.5012/bkcs.2006.27.12.2071>
- [11] Z. Hu, Y. Yu and H. Hu, Mater. Lett, vol. 64, no 7, p. 863-865, (2010); <https://doi.org/10.1016/j.matlet.2010.01.044>
- [12] A. Kamarchou, S. Atia, A. Zobeidi, H. Chettah, S. Benhamidad, D. Abdic, F. Rahal, A. Berchic, N. Chaabi, Chalcogenide Letters, p. 447 - 456, July(2022); <https://doi.org/10.15251/CL.2022.197.447>
- [13] N. M. Bikales, Proceedings of a Symposium held by the American Chemical Society, Division of Organic Coatings and Plastics Chemistry, Polymer Science and Technology, Vol 2 , Water Soluble

Polymers , p. 213-225., (1973); <https://doi.org/10.1007/978-1-4613-4583-1>

[14] I. Zhitomirsky, Colloid Interface Sci, 9, 279-317. (2002); [https://doi.org/10.1016/S0001-8686\(01\)00068-9](https://doi.org/10.1016/S0001-8686(01)00068-9)

[15] A. Makhlof, Handbook of smart coatings for materials protection, London, Woodhead Publishing Elsevier, No. 64, (2014).

[16] M. Taguchi, A. Suzuki, T. Oku, N. Ueoka, S. Minami, and M. Okita, Chemical Physics Letters.737.136822, (2019); <https://doi.org/10.1016/j.cplett.2019.136822>

[17] A.K .Mohammed, Studying the effect of Annealing on the Structural and Optical Properties of (Zn<sub>1-x</sub>FexO) Thin Films Prepared by Chemical Spray Pyrolysis Method, University of Diyala in Partial Fulfillment of the Degree of M.Sc. in Physics, (2006).