Preparation and Investigation of TiO_2, SnS_x, and SnO_2 Thin Film Properties for Use as UV Detectors

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ABSTRACT

Hydrothermal method was used to prepare TiO_2 films at temperatures (150,160,170,180)^oC with a holding time of 6h, and the SILAR method was used to prepare the SnS_x film. Through XRD assays, the shape of the TiO_2 compound is quaternary rutile in all samples and has a preferred growth direction towards (101), and we note that when the temperature increases, the intensity of the peaks decreases. XRD assays confirmed that the samples prepared by the SILAR method confirmed that the compound SnS_x consists of the two compounds SnS and SnS_2, as the compound SnS has a rhombic structure and the compound SnS_2 has a hexagonal shape.We note that all the manufactured FTO/TiO_2/Al, FTO/n-TiO_2/p-SnS_x/Al, FTO/n-TiO_2/p-SnO_2/Al optical reagents have a nonlinear Schottky behavior. Al-TiO_2 reagents at different temperatures have a high sensitivity of about 9900%, and the sensitivity of the detector decreases with the increase in the applied voltage, and it has a slow response, so we dope it with materials SnS_x, SnO_2. As for the TiO_2/SnS_x, TiO_2/SnO_2 reagents, we note that the highest sensitivity is 40400% for the FTO/n-TiO_2/p-SnS_x/Al detector with at a voltage of 0 V. Also, the sensitivity decreases with an increase in the applied voltage and its response is fast.

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1. Introduction

Nanotechnology is an active field of research with both new sciences and useful applications that have gradually established themselves in the past two decades. Nanotechnology is a term encompassing science, engineering and materials applications[1], which involves harnessing the unique physical, chemical, and biological properties of nanomaterials in fundamentally new and useful ways [2].This emerging technology It has many possible applications and therefore affects various technological fields including advanced materials, biotechnology, pharmacy, electronics, scientific instruments and industrial manufacturing processes. From a scientific point of view, nanotechnology can be defined as referring to materials and systems with structures and components that exhibit physical and chemical properties [3]. The use of UV detectors on a large scale in achieving the process of visible blindness, in the recent period, semiconductors with nanostructures such as nanosticks, nanowires, nanotubes, and nanostrands have attracted wide research interest due to their high surface-to-size ratio and surface conformation [4,5,6] N-structured conductors Nuclei such as titanium dioxide (TiO_2) are of much interest due to their photoelectric properties and remarkable catalytic activity [7]. The molecular formula for titanium dioxide is TiO_2. TiO_2 has very high photocatalytic activities and is non-toxic, chemically stable, biocompatible and strong oxidizing agent (large surface area), it is a cheap material and low production cost. Thermodynamically polymorphic structures. Titanium dioxide (TiO_2) is an important semiconductor material with a wide bandgap energy gap (3.02 eV and 3.20 eV for Rutile and Anatase respectively) and is an n-type semiconductor. On the other hand, tin sulfide is a semiconductor and SnSx (x = 1,2) compounds are non-toxic and low-cost, SnS_x materials have excellent chemical stability and reversibility due to their unique structure [8]. Tin sulfides have received great attention due to their structural diversity and containing Several binary compounds such as SnS, Sn_2S_3, Sn_3S_4, Sn_4S_5, and SnS_2 [9, 10]. Among these compounds, tin monosulfide (SnS) and tin disulfide (SnS_2) are the most important and have received a lot of attention for their intriguing properties and many applications [10]. The use of inexpensive methods for preparing high-quality nanocomposites has attracted the attention of researchers and the hydrothermal method is one of the Methods used for this purpose This method is a process for preparing many forms of nanomaterials such as thin films with nanostructures through a heterogeneous reaction in the presence of aqueous solvents under high temperatures and pressures [11]. One of the advantages of this method is that it is an easy way to manufacture many nanomaterials, as well as the growth parameters can be changed such as growth time and temperature [12, 13]. Another

method for depositing nano films is the SILAR method, reaction and adsorption of a layer of ions. This method is used to deposit thin films of metal ions, and it is a simple method It is fast and convenient for the deposition of films of semiconductors, oxides, chalcogens and polymers [14]. This method is an upgraded version of the Chemical Bath Deposition (CBD) method, and because it does not need to work at high temperatures, it does not need expensive technology equipment [15].

2. Experimental

2.1 Preparation of TiO_2 nanorods

At first we prepare Titanium(IV) butoxide by placing 5 ml of $TiCl_4$ in a three-necked flask and 10 ml of [$CH_3CHOHCH_2CH_3$]butanol in a separator funnel and then the Butanol was distilled at a drop rate every 2-3 seconds using a magnetic stirrer at $TiCl_4$ with stirring until the disappearance of fog In the three-necked flask, a yellow viscous liquid of Titanium (IV) butoxide appeared as in Figure 1. Then the FTO substrates were washed with water and detergent to get rid of suspended matter, the FTO bases were kept in diluted HCL for 5 minutes and then cleaned with Ultrasound apparatus using acetone, ethanol and propanol each for 10 min and then washed with distilled water respectively, TiO_2 nanorods are prepared by hydrothermal method FTO was immersed at an angle in a stainless steel autoclave containing a 50 ml Teflon vessel containing the solution Prepared by adding 15 ml of hydrochloric acid, 15 ml of distilled water and 0.5 ml of Titanium (IV) butoxide. The autoclave was placed at different temperatures, (150,160,170,180,190)°C for 6 hours in the oven, then the samples were cooled. and clean them with distilled water as shown in Fig. 2.

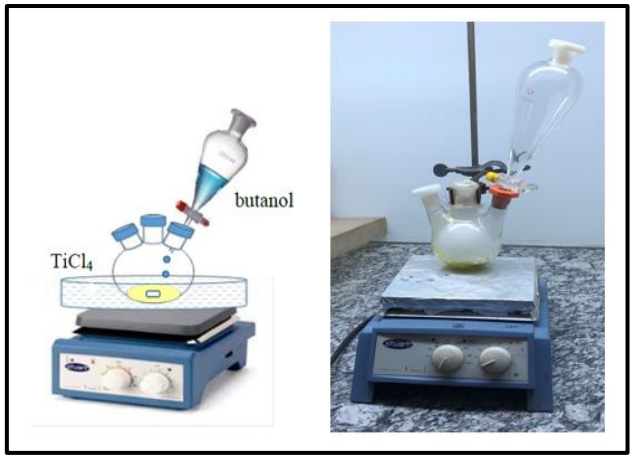

Figure1: Preparation of Titanium (IV) butoxid

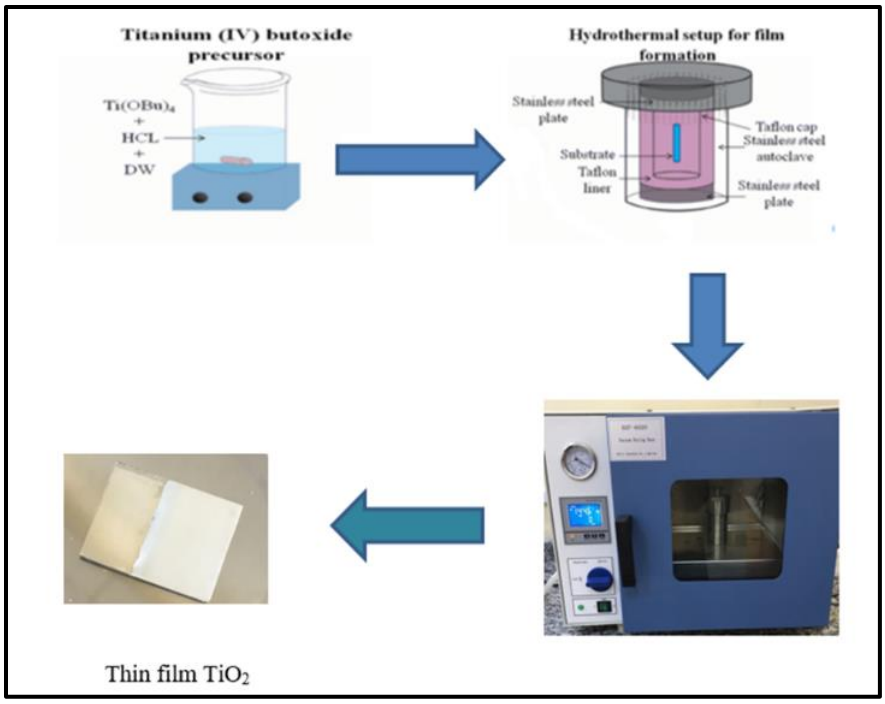

Figure 2: Steps of prepration TiO_2 nanorods

2.2 Preparation of Tin Sulfide SnS_x

SnS_x thin films were prepared by SILAR method by dissolving 0.2 M of $SnCl_2.2H_2O$ in 50 ml of distilled water with 0.1M of Na_2S in 50 ml of distilled water. The glass is then rinsed in distilled water for 10 seconds, then the glass is immersed in Na_2S solution for 20 seconds, the sulfide ions react with the tin ions absorbed on the glass, and then it is rinsed in water for 10 seconds to remove impurities, thus one SILAR cycle is completed and after 125 cycles are completed. We obtained SnS_x membranes and the experiment was conducted at room temperature as shown in Fig. 3a. When the SnS_x film is annealed at a temperature of 400°C, it turns into a SnO_2 compound and its color changes to golden, as shown in Fig. 3b.

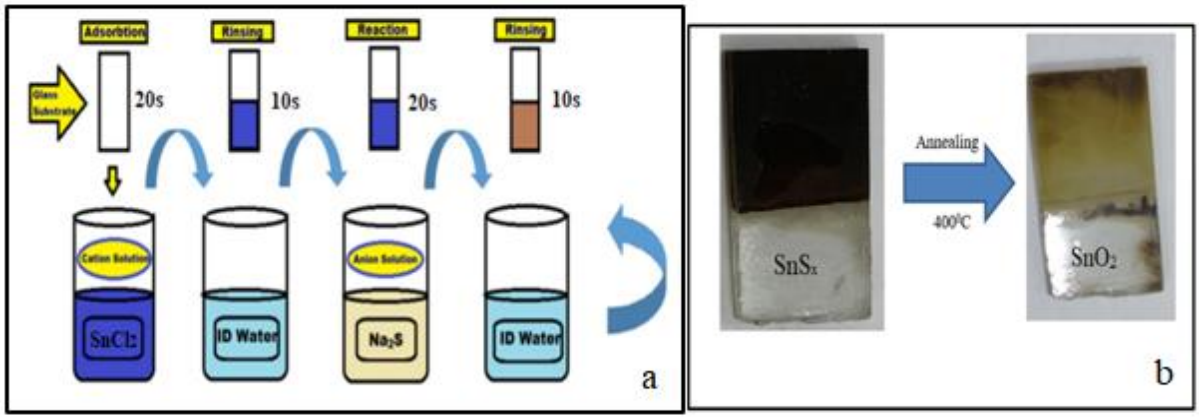

Figure 3: (a) Scheme for preparing SnS_x ,(b) Photographs of SnSx sample before annealing and SnO_2 after annealing

2.3 Fabrication of the Photodetector

The TiO_2/SnS_x photodiode was fabricated by depositing the SnS_x films prepared by SILAR method on the thin TiO_2 films prepared on FTO substrates by the hydrothermal method, while the TiO_2/SnO_2 photodiode is made by depositing the SnS_x films prepared by the SILAR method on the thin TiO_2 films prepared on the TiO_2 thinner bases. At 400°C, its color turns golden, and then a layer of aluminum (Al) is deposited by thermal evaporation method on the surface of the SnS_x films in the TiO_2/SnS_x duo and on the surface of the SnO_2 films. after annealing as shown in Fig.4. Then the connecting wires were attached to the FTO base and aluminum metal using Silver paste. The FTO substrate was used as the electrode with dimensions of 1.5 x 1cm, and the effective area of the photodetector manufactured was $1cm^2$.

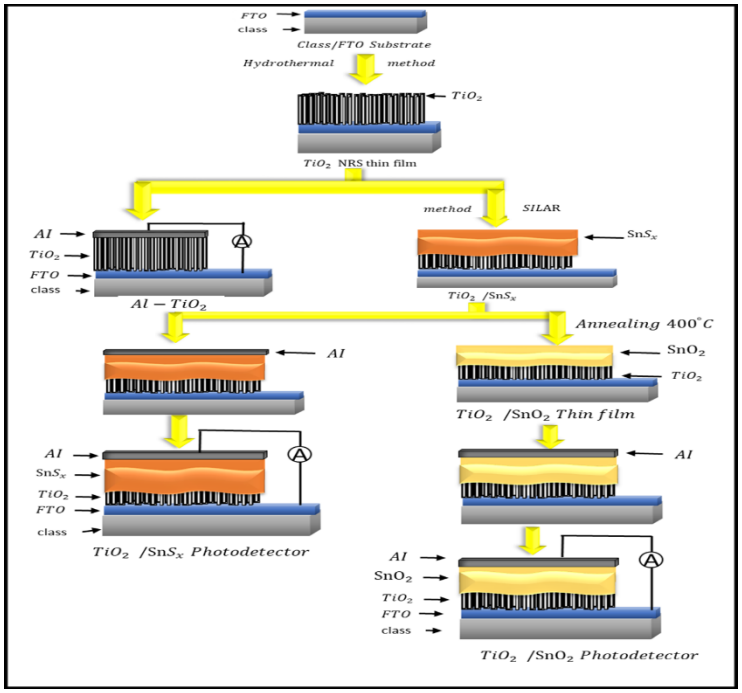

Figure 4: Schematic diagram of the manufactured reagents FTO/TiO_2/Al, FTO/n-TiO_2/p-SnO_2/Al .FTO/ n-TiO_2/p-SnO_2/Al,

3. Results and discussion

3.1 Structural analysis: X-ray diffraction

The structural properties of TiO_2 films prepared by hydrothermal method at different temperatures of 150,160,170,180, and 190°C were studied, and SnS_x films prepared by SILAR method were prepared using X-ray diffraction technique and its diffraction pattern is shown in Fig. 5A where we notice the peaks (110), (101) (200), (211). , (002) and (221) corresponding to the diffraction angles 2θ (26.69, 36.22, 37.93, 51.58, 62.94, 65.70) respectively at temperatures (150, 160, 170, 180)°C where the peaks (200), (211) belong to FTO and the other values belong to TiO_2 has a quaternary structure of a rutile type, and this result is consistent with (JCPDS 21-1272), but we note the preferred growth direction at (101) and at a temperature of 190°C. Furthermor, there are no peaks are appeared due to the increase in temperature, which causes rapid growth that leads to irregular formation The crystalline works on the peeling of the membrane where the crystal growth rate starts to decrease and as a result of these increases in the lengths of the rods leads to the breakage of the rods according to the results [13, 16]. The (c/a) approximate to the typical ratio of

the quaternary TiO_2 compound and this result with (JCPDS 21-1272). Figure 5B shows the X-ray diffraction pattern without the film of the compound SnS_x prepared by SILAR method for 30 cycles. We notice the appearance of the first two peaks at (002) that belong to the compound SnS_2 according to the card JCPDS (001-0687), while the other peak (201) belongs to the compound SnS according to the card JCPDS(73-1859), this shows that the prepared compound is SnSx(x=1,2) which consists of two compounds, SnS_2,SnS.

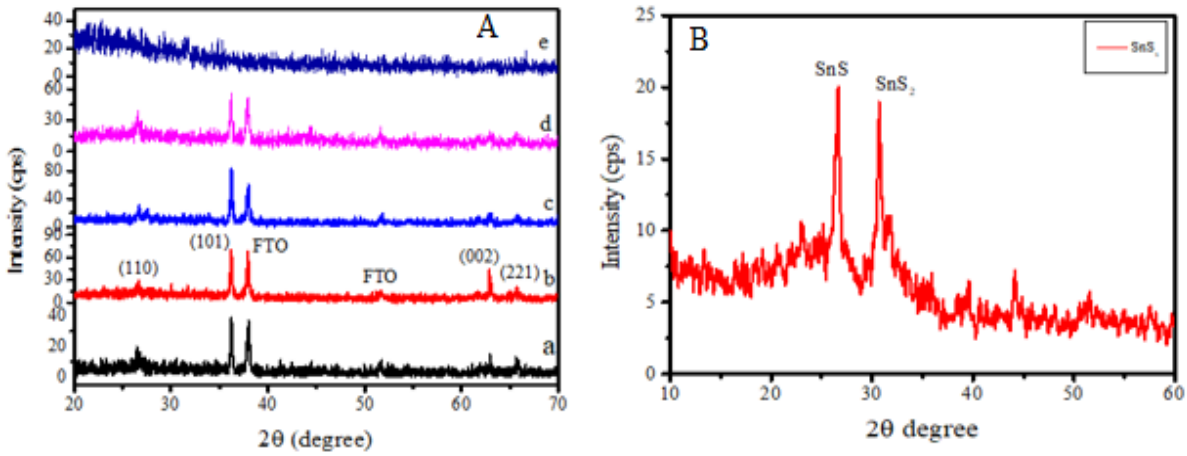

Figure 5: (A) X-ray diffraction pattern of TiO_2 model prepared by hydrothermal method on FTO substrate at temperatures (a)150°C (b)160°C (c)170°C (d)180°C (e) 190°C,(B) X-ray diffraction pattern with no fading SnS_x prepared by SILAR method and with 30cycles

Table 1: The values of the lattice constants and particle size of the prepared samples

compound	G.S(nm)	c/a	c(nm)	a(nm)	Sample
TiO_2 Rutile	42.5	0.6468	2.9534	4.5656	150°C
	84.9	0.6428	2.9576	4.6009	160°C
	38.6	0.6467	2.9527	4.5652	170°C
	53.1	0.616	2.923	4.744	180°C

3.2 Surface Morphology properties

The surface properties of TiO_2 films prepared by hydrothermal method were studied on FTO substrates at different temperatures 150°C, 160°C, and 170°C and SnS_x films prepared by SILAR

method. Rectangular and perpendicular to the base as shown in Fig. 6, and through the results that the size of the nanorods began to grow with the increase in the preparation temperature from 150°C to 170°C and continued to increase at a decreasing rate until the TiO_2 nanorods began to break at a high temperature about 180°C, where it begins to peel As a result, it begins to degrade, which causes the membranes to peel off early, and this is consistent with [13, 16–19]. Using the Image J program, the average diameter of the nanorods was calculated, as it was observed that the diameter of the nanorods increased with increasing temperature, as the diameter of the nanorods at a temperature of 150°C (20-30) nm and a temperature of 160°C would be (30-40)nm, The temperature is 170°C and the diameter of the bars is (50-60)nm. Figure 7 shows FESEM images of SnS_x films prepared by the SILAR method on glass bases with 30 cycles. We note from the figure that they are rod-shaped and the average diameter of the rods is (10-20) nm**.**

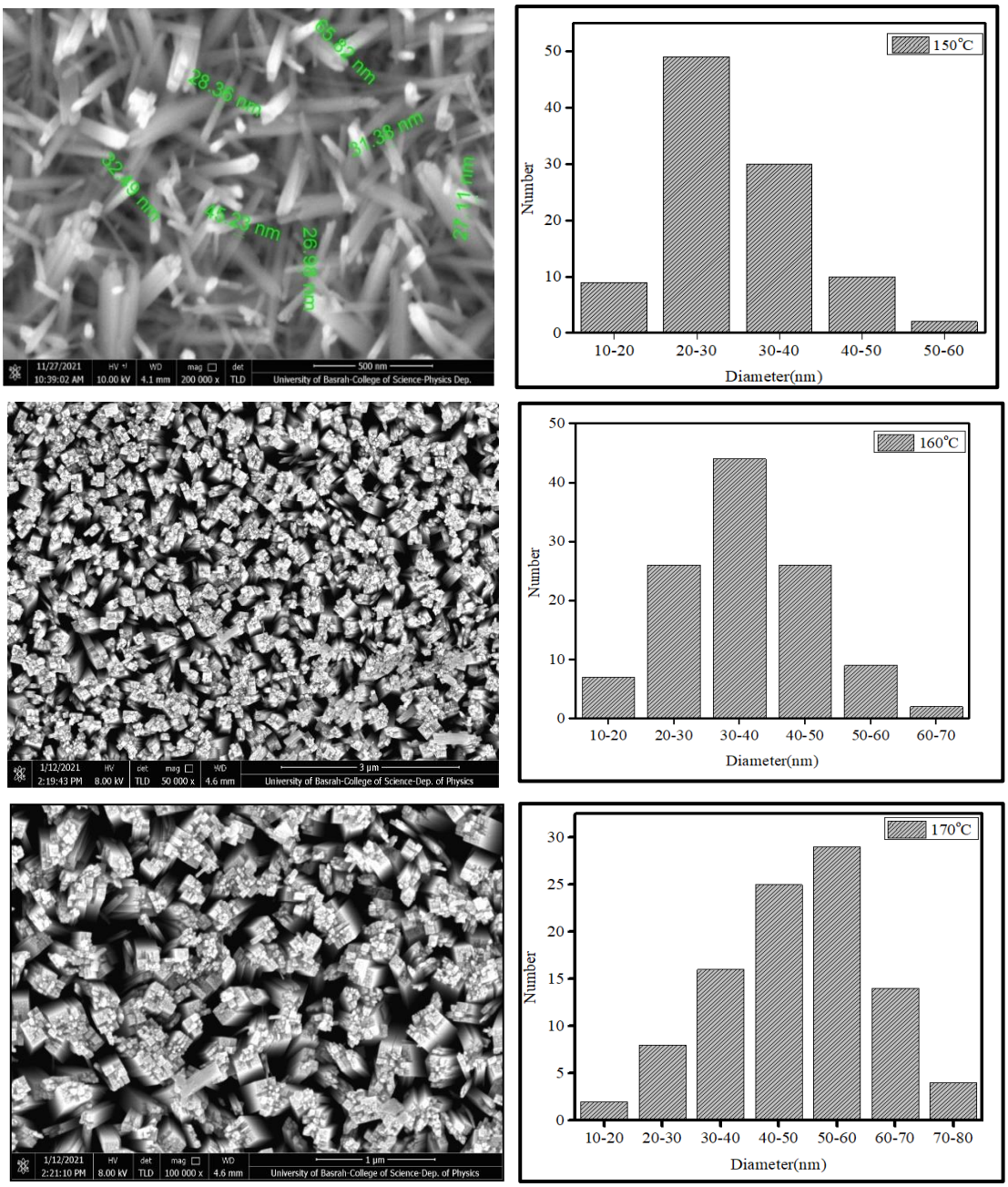

Figure 6: SEM and Diameter distribution of TiO_2 thin films at different temperatures

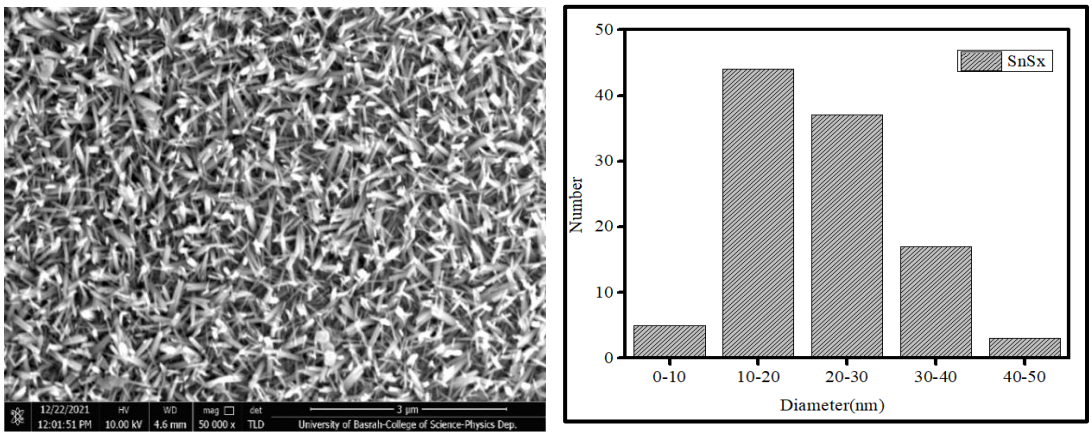

Figure 7: SEM and Diameter distribution of SnS_x thin films

3.3 Optical properties

The optical properties of the nanostructured thin films were studied using the absorption spectra of a wavelength range (300-1000). Figure 8 shows the absorption spectra of the films of the composite TiO_2 prepared by the hydrothermal method and at different temperatures (150,160,170,180,190)^{o}C, where they show strong absorption in the UV region between (300-400) nm and weak absorption in the visible region indicating that it has a wide energy gap and the absorbance increases with increasing temperature and at high temperatures the absorption was observed in the visible region $\lambda > 410$ which is generally related to the absorption of visible light through surface defects TiO_2 NRs and the energy gap was calculated. The Eg for films prepared using the absorbance spectrum from plot $(\alpha h\nu)^2$ versus $(h\nu)$ by extrapolating the straight-line curve to the photon energy axis intercept at $((\alpha h\nu)^2 = 0)$ as shown in Fig. 9 which is consistent with the results [18–21]. We noted that there is little difference in the energy gap value. This difference is due to the difference in the size of the thin-film nanorod according to the increasing concentration and deposition temperature. The increase in crystallinity and regularity in the thin-film crystal structure may also be one of the reasons. In Fig.10, the absorption spectrum of the SnS_x films prepared by the SILAR method and 30 cycles on the bases of the glass and by annealing at a temperature of 400°C turns into the compound SnO_2, where the absorption appears in the range (350-480) and the energy gap is calculated by extrapolating the straight line on the x-axis, which be consistent with research [22, 23].

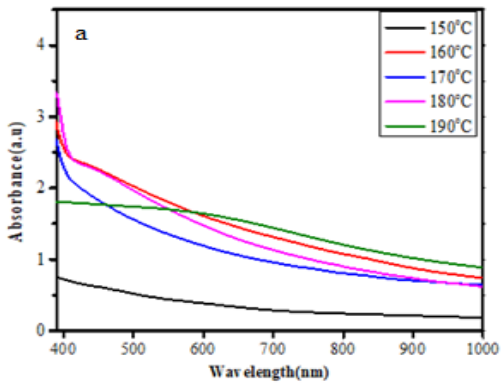

Figure 8: Absorption spectrum of TiO_2 films prepared by hydrothermal method on FTO substrate at different temperatures

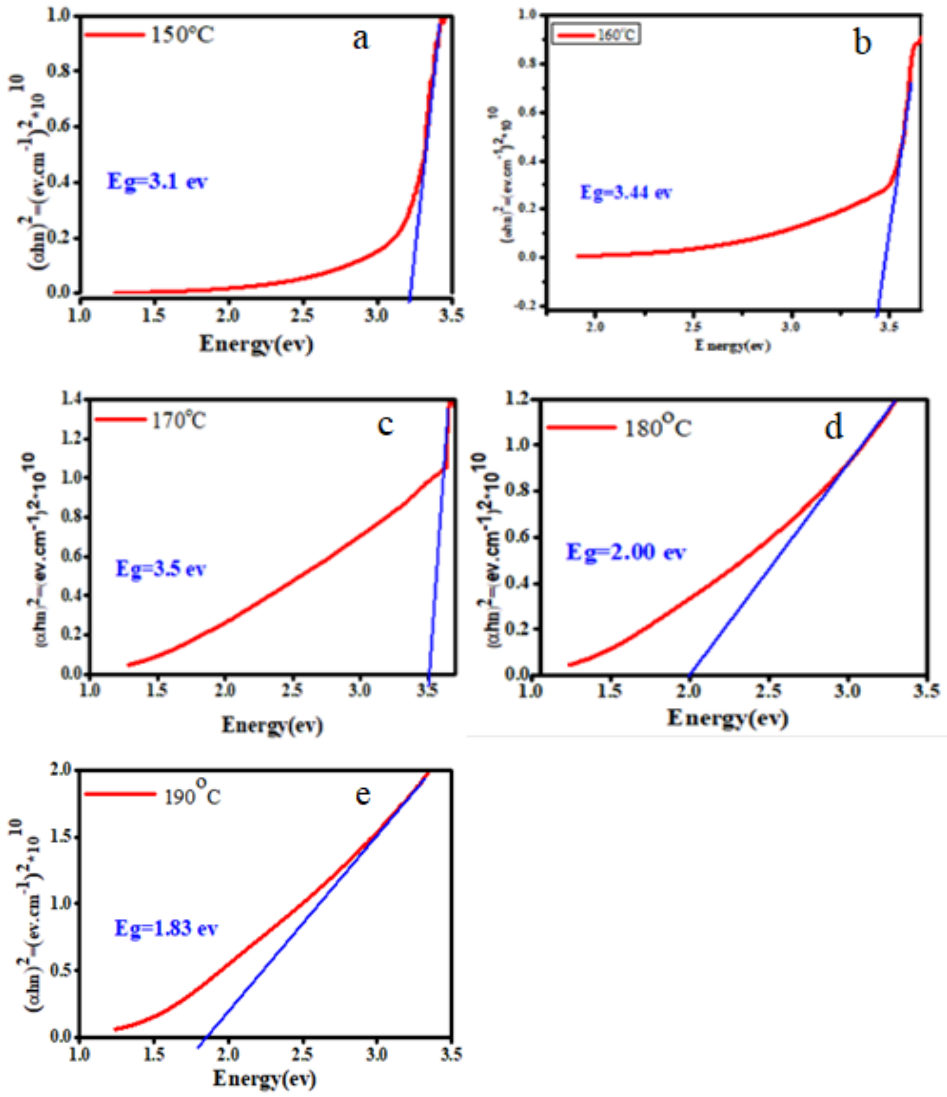

Figure 9: Energy gap values of TiO_2 films prepared by hydrothermal method on FTO substrates at different temperatures (a)150°C,(b)160°C,(c)170°C,(d)180°C,(e)190°C.

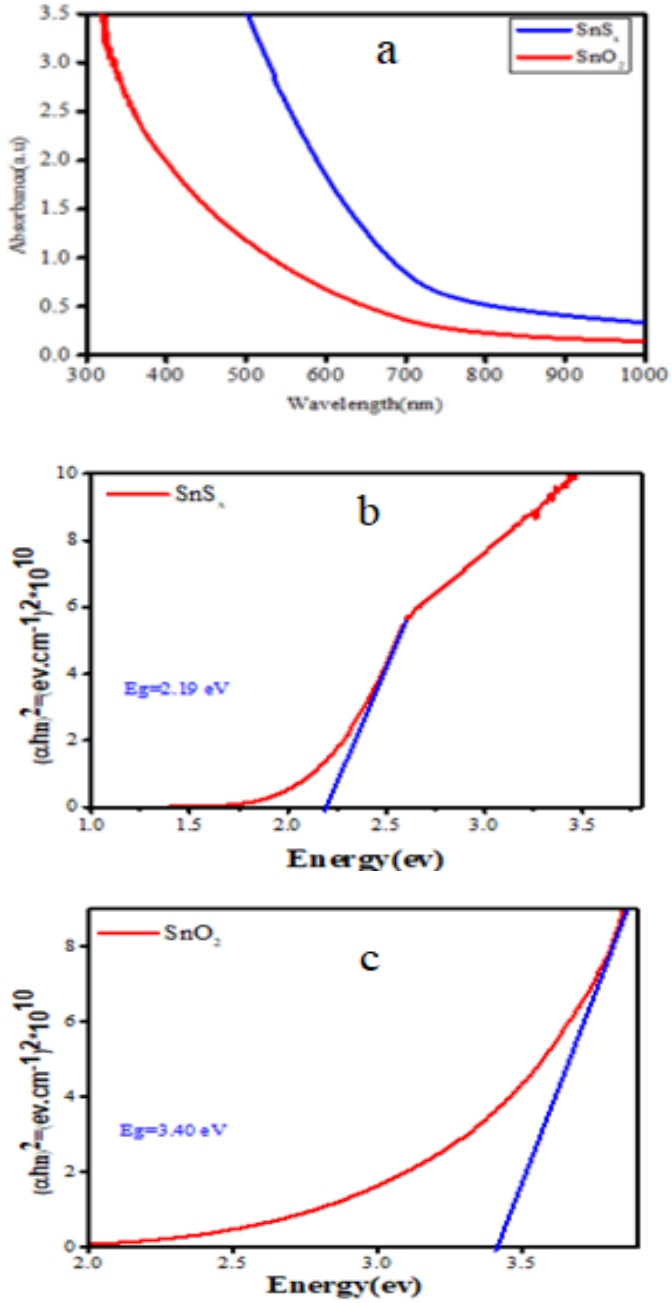

Figure 10: (a) Absorption spectrum of the films of the SnS_x prepared by SILAR method on glass bases at 30 cycles and of the SnO_2 compound prepared from the heat treatment of SnS_x at 400°C,(b) Energy gap values for SnS_x,(c) Energy gap values for SnO_2

Table 2: Energy gap values

substrate	compound	Eg(ev)	T(°C)
FTO	TiO_2 Rutile	3.1	150
		3.44	160
		3.5	170
		2.00	180
		1.83	190
		2.53	160
		3.05	
		3.01	
Glass	SnS_x	2.15	-------
	SnO_2	3.40	----

3.4 Photoluminescence (PL)

Photoluminescence spectrum (PL) at room temperature for TiO_2 films prepared on FTO substrates by hydrothermal method at temperatures 150°C, 160°C using an excited wavelength of 325 nm. The spectra show an emission peak at 396 nm as shown in Figure 11. upon excitation with bandgap energy the photoexcited electrons relax to the lowest level of the excited state [24], that is, when TiO_2 is exposed to ultraviolet rays, its gap is recombined with the electron and emits light photons [25]. We observe a decrease in the intensity with increasing temperature and therefore the decrease in the intensity of the PL peak confirms the decrease in the recombination rate for electron gap pairs [26].

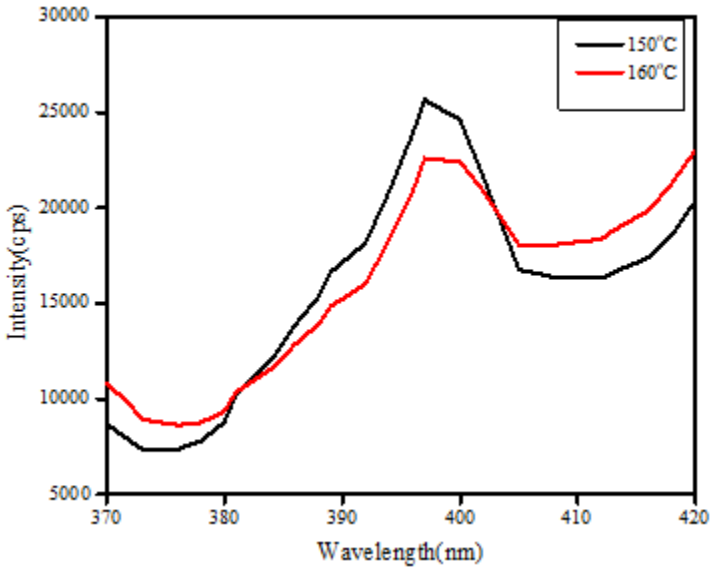

Figure 11: Photoluminescence spectra of TiO_2 films prepared by hydrothermal method at 150°C, 160°C**.**

3.5 Current-voltage Characteristics

The electrical properties of the photodetector in the dark and in the light were studied using Keithly 2400 device under applied forward and reverse bias voltage from -4 to +4 at room temperature. The figure shows 13a the change of the dark current as a function of the bias voltages applied to the two ends of the detector. The current increases linearly with the increase of the voltage applied to the membranes, meaning that the mechanical is non-ohmic. Figures 12,13, and 14 show the curves of the values of the current in the dark and the illumination passing through the detector as a function of the forward and reverse bias. In illumination, the detector was exposed to white light of intensity (0.67 mW/cm^2). We note that all detectors show a non-linear (Schottky) behavior. In the forward bias, we notice a large increase in the value of the current with an increase in the applied voltage. The reason for the large increase in the current is due to the fact that the forward bias voltage will reduce the voltage barrier, that is, a decrease in the width of the depletion region, which leads to an increase in the speed of electrons. As for the reverse bias, the width of the depletion region will increase, which will lead to an obstruction in the movement of electrons,. a large voltage barrier that works to straighten the current. We also note that the dark current is small, while the light current generated under illumination was high. In order to calculate the idealization factor (n) by drawing the relationship between LnI with the forward bias voltage in the dark state and the illumination at room temperature and from the slope of the straight line, the

idealization factor (n) can be calculated using Equation 1. As for calculating the voltage barrier height, the resulting saturation current I0 was calculated From the extension of the logarithmic curves of the current at its value (V = 0) and from an equation 2, the height of the voltage barrier was calculated, as shown in Table 3.

$$n = \frac{q}{k_B T}\left(\frac{dv}{d(\ln(I))}\right) \quad (1)$$

$$\varphi_b = \frac{k_B T}{q} = \ln\left(\frac{AA^* T^2}{I_o}\right) \quad (2)$$

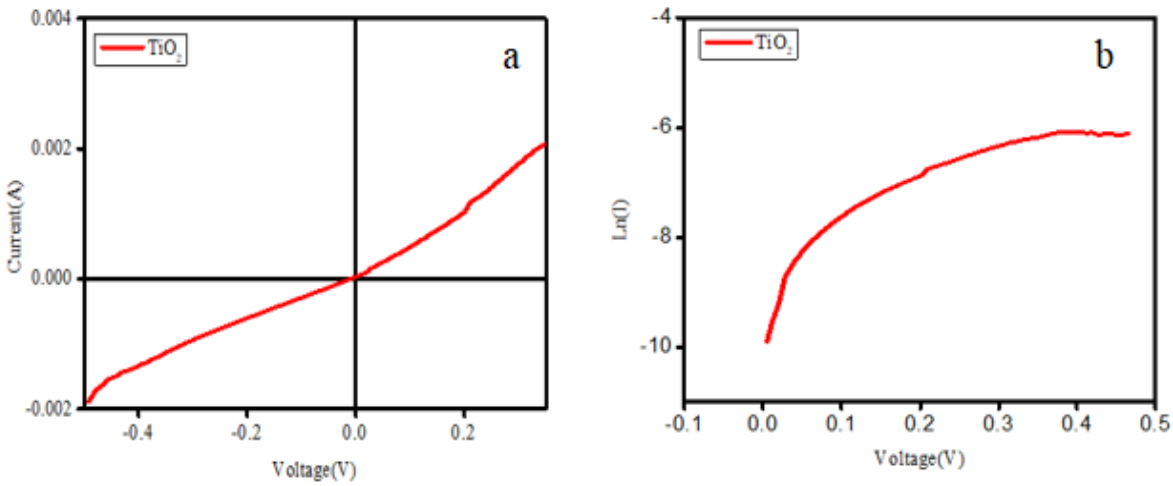

Figure 12: (a) Characteristic (I-V) for TiO_2 under darkness, (b) Ln(I) versus voltage (V)

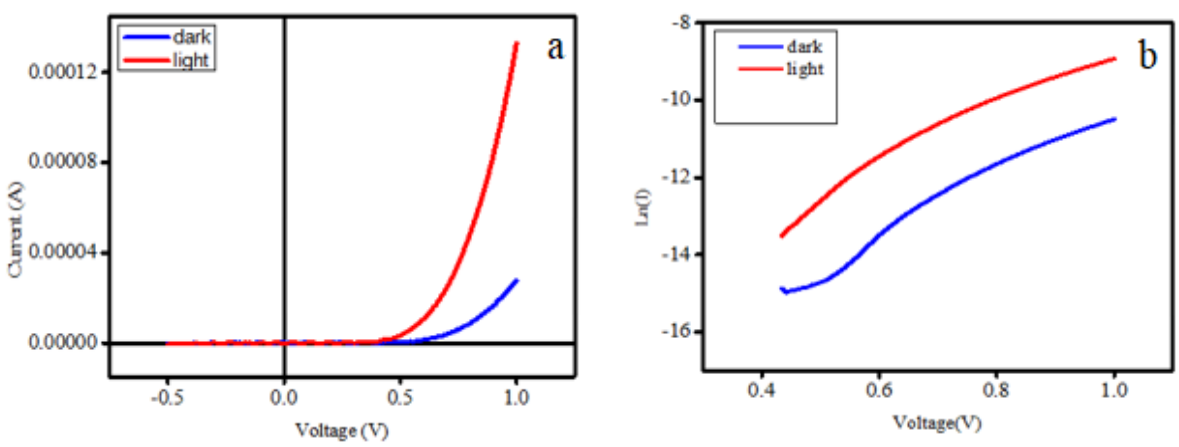

Figure13: (a) Characteristic (I-V) Under Darkness and Illumination of the FTO/n-TiO_2/p-SnSx/Al Detector, (b) Ln(I) versus voltage (V)

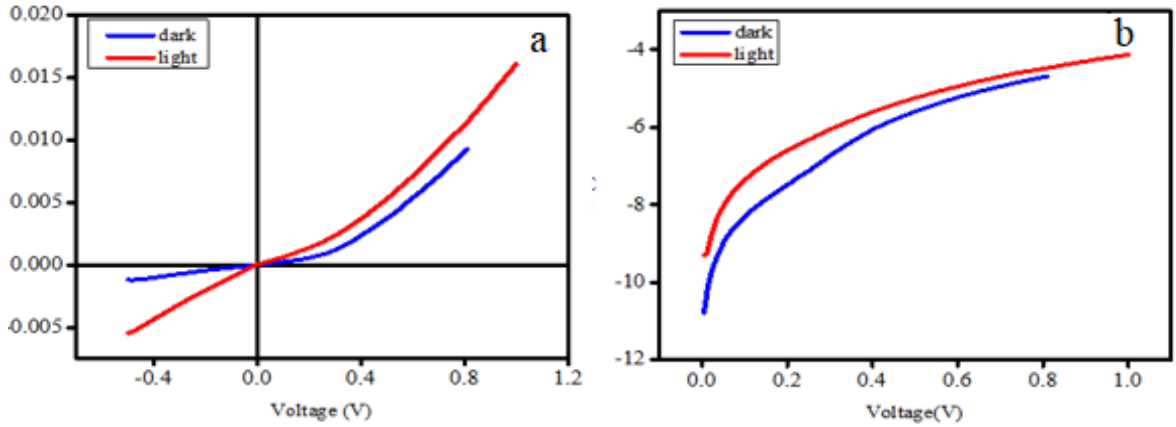

Figure 14: (a) Characteristic (I-V) Under Darkness and Illumination of the FTO/n-TiO_2/p-SnO_2/Al Detector, (b)Ln(I) versus voltage (V).

Table 3: values of the ideal factor, saturation current, and voltage barrier

Photodetector		TiO_2	n-TiO_2/p-SnS_x	n-TiO_2/p- SnO_2
n	dark	6.154	4.452	7.673
I_0(A)	dark	$3.562x10^{-6}$	$4.563x10^{-7}$	$1.165x10^{-5}$
Φ_B(ev)	dark	0.684	0.827	0.773
n	Light	--------	4.980	7.437
I_0(A)	Light	--------	$2.93x10^{-6}$	$1.224x10^{-5}$
Φ_B(ev)	Light	-------	0.809	0.772

3.6 Photosensing properties

The optical sensing properties of the FTO/TiO_2/Al detector as well as the n-TiO2/p-SnSx, n-TiO_2/p-SnO_2 photodetectors prepared at different temperatures (150ºC, 160ºC, 170ºC) were studied using ultraviolet light with a wavelength of 385nm and intensity of 0.05 mW. cm^2 at room temperature and by applying a different bias voltage. These rays were directed perpendicular to the photodetectors with a distance of (5 cm) and the response and sensitivity were calculated using the equations 3,4 respectively. The performance mechanism of photodiodes can be explained by the following steps: (1) generation of electron hole pairs by absorbing the incident light, (2) separation of electron hole pairs by the internal electric field of the junction and (3) current flowing in the external circuit to generate the output signal [27] . The rise time and the landing time were also calculated as shown in the table 3. The results obtained through the optical sensing properties of the FTO/TiO_2/Al detector prepared by the hydrothermal method, at a temperature of 160ºC, and for different bias voltages, as in the figure 14a. at 0V bias voltage, the light produces an electric field that gives an optical current and grows until it reaches saturation The current increases from 0.001µA to 0.100µA by highlighting and it showed a high sensitivity of 9900%.

$$R_\lambda = \frac{I_{ph}}{P_{in}} \qquad (3)$$

$$S = \frac{I_{ph} - I_d}{I_d} 100\% \qquad (4)$$

We note from the results that when comparing the n-TiO_2/p-SnS_x reagents at different temperatures (150ºC, 160ºC, 170ºC) at 0V, the photocurrent increases with increasing temperature, and the response is quick as the temperature increases, as shown in the figure 16d.As well as in the aforementioned detectors, the photocurrent increases with the increase in the applied voltage and the sensitivity decreases, as well as in the FTO/TiO_2/Al detector the response is weak, but when a layer of SnS_x,SnO_2 compounds is deposited, its response is fast and improves the performance of the photodetector as found in [20, 28]. It was observed in the FTO/n-TiO_2/p-SnO_2/Al FTO/n-TiO_2/p-SnS_x/Al reagents that the highest sensitivity is 40400% at a temperature of 170ºC with a voltage of 0 V and a rise time of 0.17s with a drop time of 0.3s as well Sensitivity decreases with increased voltage applied and its response is rapid. We note that the best reagent is FTO/n-TiO_2/p-SnS_x/Al as in the Fig. 15b,c.

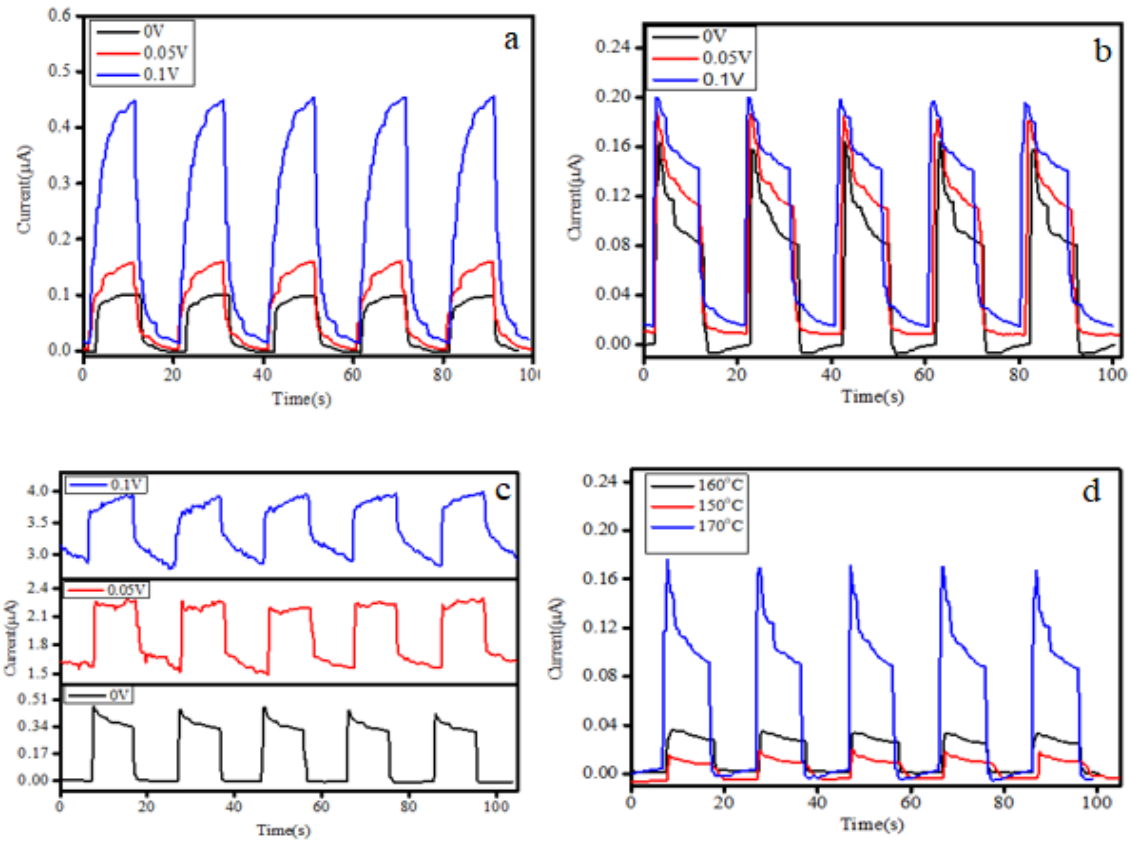

Figure15: Time response (a) of the FTO/TiO_2/Al reagent prepared at 160°C (b) of the n-TiO_2/p-SnS_x reagent (c) of the n-TiO_2/p-SnO_2 reagent (d) of the n-TiO_2/p-SnS_x detector prepared at temperatures (150°C,160°C,170°C) at 0V.

Table 3: Photodetector parameters

Photodetector		τ_{rise} (s)	τ_{fall} (s)	I_{light} (µA)	I_{dark} (µA)	S_{ph} %	R (mA/W)	V (volt)
TiO_2	160°	1.57	2.70	0.100	0.001	9900	1.98	0
		2.58	3.75	0.158	0.003	5166	3.1	0.05
		4.73	2.86	0.446	0.016	2687	8.6	0.1
TiO_2/SnS_x	150°	0.4	0.57	0.024	0.001	2300	0.46	0
		0.56	0.77	0.03	0.002	1400	0.56	0.05
		0.57	0.92	0.036	0.003	1100	0.66	0.1
TiO_2/SnS_x	160°	0.18	0.75	0.042	0.011	250	0.62	0
		0.19	0.31	0.059	0.018	227	0.82	0.05
		0.194	0.316	0.081	0.027	200	1.08	0.1
TiO_2/SnS_x	170°	0.17	0.32	0.162	0.0004	40400	3.23	0
		0.179	0.53	0.184	0.008	2200	3.52	0.05
		0.21	0.98	0.199	0.015	1226	3.68	0.1
TiO_2/SnO_2	160°	0.61	1.36	0.034	0.009	277	0.5	0
		0.4	0.8	19.09	16.02	19	61	0.05
		0.8	1.13	39.3	33.9	15	108	0.1

Conclusions

XRD assays showed that the shape of the TiO_2 compound is a tetra-rutile type in all samples and has a preferred growth direction towards (101). We note that when the temperature increases, the intensity of the peaks decreases. The XRD tests confirmed that the samples prepared by the SILAR method that the compound SnS_x consists of the two compounds SnS and SnS_2, as the shape of the compound SnS is a structure The rhombic compound SnS_2 has a hexagonal shape. The FE-SEM surface tests showed that the TiO_2 films are rod-shaped and the upper surfaces are square and perpendicular to the FTO substrate. The optical results of the films of the compound SnS_x prepared by the SILAR method showed that the energy gap increases when annealing at a temperature of 400°C and turns into the compound SnO_2. The highest sensitivity is 40400% for the FTO/n-TiO_2/p-SnS_x/Al detector with a voltage of 0 V and a rise time of 0.17s with a drop time of 0.3s. Also, the sensitivity decreases with the increase in the applied voltage and its response is fast. We note that the best reagent is FTO/n-TiO_2/p-SnS_x/Al.

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تحضير ودراسة خصائص اغشية TiO_2 ، SnS_x و SnO_2 واستعمالها ككواشف للاشعة فوق البنفسجية

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المستخلص

في هذا البحث تم استخدام طريقه Hydrothermal في تحضير اغشية TiO_2 بدرجات حرارة (150,160,170,180)^oC مع ثبوت الوقت 6h وطريقة SILAR في تحضير غشاء SnS_x. ومن خلال فحوصات XRD يكون شكل المركب TiO_2رباعي نوع Rutile بكل العينات وذو اتجاه نمو مفضل باتجاه (101) ونلاحظ عند زيادة درجه الحرارة تقل شده القمم واما من ناحية التركيز نلاحظ النمو المفضل يكون باتجاه (002) أي عند زيادة التركيز تزداد شده القمم. اكدت فحوصات XRD ان العينات المحضرة بطريقه SILAR ان المركب SnS_x يتكون من المركبين SnS,SnS_2 اذ يكون شكل المركب SnS هيكل معيني والمركب SnS_2 يكون شكله سداسي. نلاحظ جميع الكواشف$Al\text{-}TiO_2,TiO_2/SnS_x,TiO_2/SnO_2$ الضوئية المصنعة لها سلوك لا خطي شوتكي. ان الكواشف $Al\text{-}TiO_2$ بدرجات حرارة مختلفة تمتلك حساسيه عالية هي حوالي 9900% وتقل حساسيه الكاشف بزيادة الجهد المسلط، وذات استجابة بطيئة وبذلك نقوم بتشويبها بالمواد SnS_x,SnO_2. اما في الكواشف $TiO_2/SnS_x,TiO_2/SnO_2$ نلاحظ ان اعلى حساسيه تكون 40400%عند درجه الحرارة 160^oC وبجهد 0 V وكذلك تقل الحساسية بزيادة الجهد المسلط وتكون استجابته سريعة.