

Influence of SnO₂ Prepared by Rosemary Extract to Improve Porous Silicon for Photodetector Applications

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ABSTRACT

The goal of this work is to determine whether SnO₂ made from rosemary extract at concentrations of 0.1M and 0.2M may be used to enhance PSi for photodetector applications. Electrochemical etching was used to prepare the porous silicon samples. To identify the chemical linkages created during the etching process, PSi subjected to FTIR spectroscopy. The energy gap from PL emission was 1.75 eV. Using the drop casting technique, SnO₂ films produced on substrates (glass and PSi). The films are polycrystalline and crystal with structure was tetragonal rutile, according to XRD pattern. It was discovered that the produced samples' crystallite sizes ranged from 9 to 12 nm. The films' surface at 0.1M found to be smooth by AFM, with a mean grain size of 73.76 nm, according to the study, the SnO₂ film's surface exhibits good grain distribution and homogeneity at concentrations of 0.2 M, and the grain size grows to 83.87 nm. As the molar concentration of precursor rises, the band energy of SnO₂NPs tends to decrease. Presence of SnO₂ nanoparticles made from *Salvia rosmarinus* extract at two different concentrations 0.1M and 0.2M verified by FTIR analysis. It was discovered that current-voltage (I-V) characteristics showed Schottky-like features, with photocurrent growing exponentially as bias voltage and light intensity rose. The synthesized reagents' suitability for detection in the UV and NIR areas confirmed by their good responsivity and selective detectivity.

Keywords: Tin oxide, plant extract, nanostructure, UV-photodetector.

INTRODUCTION

Over time, metal oxide nanoparticles have been shown to play an important function in both scientific and technological sectors. the many

applications for metal oxides such as ; Lithium ion batteries, field effect transistors, fuel cells, solar cells, light-emitting diodes, cancer cell therapy, biosensors, and antibacterial agents. The metal oxide (SnO₂) is of

particular interest because of its n-type semiconductor, large band gap (3.6 eV), and wavelength greater than 400 nm. Because excellent optical and electrical qualities are linked to superior chemical and mechanical stabilities [2]. Additionally, SnO₂ has a high mobility of electron ($\sim 100\text{--}200\text{ cm}^2\text{ V}^{-1}\text{s}^{-1}$), which suggests that photoexcited electrons are transported more quickly. Moreover, injecting or inhaling SnO₂ has no negative health effects and is biocompatible [1-3]. SnO₂ is a promising semiconducting material for real-world use in self-powered UV photo-detectors under ambient circumstances. It has a lot of potential for use in energy of renewable and renewable energy consumption to meet carbon neutrality goals. A photodetector (PD) is a device that uses the electrical impact of individual photons to detect incident light. The "5S" requirements high sensitivity, high spectral selectivity, high signal-to-noise ratio, high speed and high stability are widely recognized to be met by a typical photodetector [4]. Flame sensing, satellite launching, optical communication, biological and chemical analysis, and astronomical research are only a few of the commercial and scientific uses for self-powered UV photodetectors [4]. Researchers have used a change of physical and chemical techniques over the past few decades to synthesize SnO₂-NPs, including chemical precipitation, sol-gel, hydrothermal, microwave-assisted, ultrasonic atomization and electrochemical deposition [5]. However, because of its benefits such as the need for basic equipment, low operating costs, non-toxicity, and superior homogeneity—the plants technique has drawn the interest of numerous researchers. While microorganisms like bacteria, fungi, and algae require extensive procedures to produce nanoparticles, plants provide one-step approaches. Plant extracts were shown to be ideal for producing nanoparticles because of their rapid development, non-pathogenic reaction conditions, and environmental safety[6-8]. Two crucial elements of green synthesis are the a natural

source or extract as the primary ingredient and solvent system use of water.. Plant components like leaves, seeds, flowers, and roots are needed to produce nanoparticles from plants. Green chemistry principles, such as the use of non-toxic chemicals, renewable resources, environmentally friendly solvents, and the production of biodegradable waste products during the research and design of nanoparticle plant extracts, are upheld in this procedure [8-10]. Our review of previous studies indicates that extensive research on SnO₂ -based photodetectors has predominantly relied on conventional physical and chemical synthesis routes. In contrast, limited attention has been paid to environmentally friendly SnO₂ preparation methods and their integration with porous silicon for photodetector fabrication. Therefore, driven by the growing demand for sustainable and cost-effective electronic devices, our aim was to combine an environmentally friendly approach with porous silicon-based photodetectors and systematically correlate device properties under preparation conditions.

PREPARATION OF SALVIA ROSMARINUS EXTRACTS.

Salvia rosmarinus was bought at Baghdad market, cleaned two or three times under running water, sanitized once with Milli-Q water, and then allowed to dry for a week at RT in a dust-free environment. The process was similar to that described by Balaji Boya et al.[11]), who used agro-waste cotton boll peel to create tin oxide nanoparticles. An electric mixer was used to grind the dried Salvia rosmarinus into a powder. The aqueous extract was made by adding around 3 g of powder to 100 mL of deionized water and boiling it for 35 minutes at 60 °C in a water bath. After cooling to ambient temperature, the extract was filtered using Whatman no. 1 filter paper and refrigerated at 4 °C. 3.5g and 7g of SnCl₄ · 5H₂O were added to 100 ml of deionized water while being continuously stirred at 70C for 30 minutes in order to

create the SnO₂ NPs at two different concentrations (0.1M and 0.2M). Each of the produced SnCl₄ • 5H₂O (0.1M and 0.2M) received a drop wise addition (30 ml) of *Salvia rosmarinus* extract. For an hour, the solutions were continuously stirred at 70 degrees Celsius. As seen in figure 1, the solution was pale yellow at 0.1M and amber at 0.2M after 30 minutes.

PREPARATION OF POROUS SILICON AND FABRICATION OF PHOTODETECTOR

Silicon wafer (P-type) with an orientation of <111> used in preparation of Psi .. The wafer was cut into tiny, 2 cm by 2 cm pieces. The oxide layer has been removed from the wafer surface by cleaning it with appropriate solutions including acetone, hydrofluoric acid, and ethanol. Use silver paste as an electrode on the back Si surface using a mask to determine the effective area, then dry at 60C to ensure good adhesion and electrical conductivity. The effective area reached 0.09cm² and is constant for all samples. As seen in Figure 2, the samples were manufactured using an electrochemical cell composed of Teflon, which is resistant to assault by the hydrofluoric acid electrolyte. The silicon wafer, which acts as anode, is positioned between Teflon top and bottom part. A

round gold cathode immersed in the electrolyte of hydrofluoric acid. As seen in Figure 2, the hydrofluoric acid electrolyte is inserted into the upper portion of the Teflon cell. There must be enough electrolyte to cover the gold cathode and provide the necessary fluorine ions. The area circular window on the top of the Teflon cell exposes the silicon to hydrofluoric acid was 1 cm², to create porous silicon substrates. The electrochemical process lasts roughly ten minutes at a steady current of 10 mA. Two different of PD devices were made Ag/SnO₂(0.1M)/PSi/p-Si/Ag and Ag/SnO₂(0.2M)/PSi/p-Si/Ag nanostructures as shown in fig.2e. Ag electrode was placed on back side of PSi and SnO₂ was deposited using drop casting (5 drops, each measuring 20 µl) at 80 C. Ag electrode placed on front of side of samples for measurement of PD device characterize. To minimize variation between the prepared heterogeneous , all experimental steps were carried out meticulously and under identical experimental conditions. The Jobin Yvon-miniature monochromator (model: LEOI-92) was used to assess the PD's spectral responsivity in the 200–1250 nm spectral range at 1 V reverse bias voltage following power calibration using a Sanwa silicon power meter.



Fig.1. preparation of SnO₂ by green synthesis method at different concentration: (a) The *Salvia rosmarinus* (b) SnCl₄ • 5H₂O (c)SnO₂at 0.1M and 0.2M during work under 80C. (d) from left: distill water , *Salvia rosmarinus* extract , SnCl₄ • 5H₂O solution ,SnO₂ colloidal at 0.1 and 0.2M respectively.

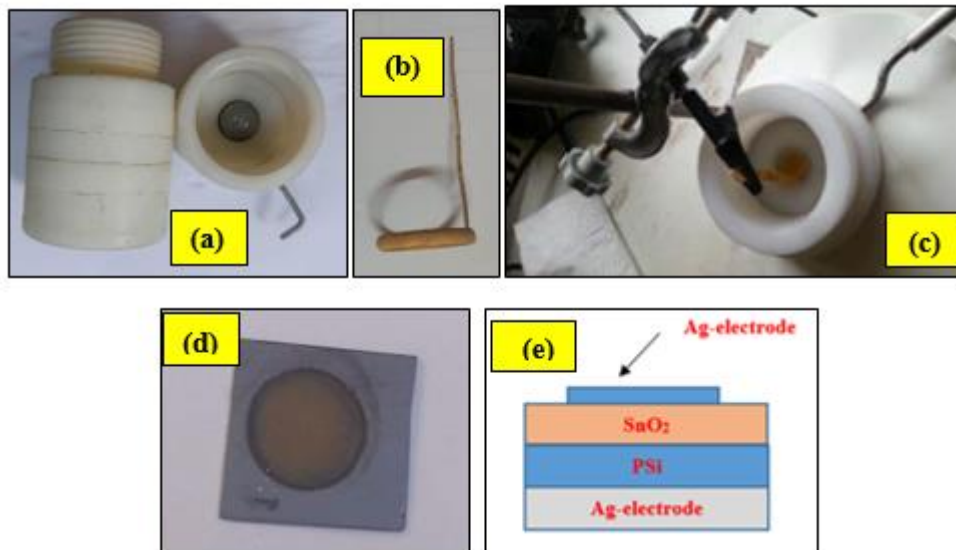


Fig2.preparation of Psi by electrochemical etching : (a) Teflon cell (b) The cathode is a circular gold (c) process of etching exposes the silicon to hydrofluoric acid (d) the porous silicon by constant current 10mA and period of about 10min. (e) schematics of Ag/SnO₂(0.1M)/PSi/p-Si/Ag.

RESULT AND DISSECTION

According to the 3D AFM of the morphological surface of the Psi layer and histogram presentation in Fig. 3a,b, the pores are randomly dispersed and have comparatively varied sizes and shapes. The porous layer's valed average particle size was 66.83 nm. The Ssk value (-1.44) verifies that the surface leans more toward pits or depressions than elevations, and the Sku value (4.49) shows that the surface is neither flat nor nearly Gaussian due to its deep pits. Photoluminescence (PL) emission at ambient temperature for the p-type PSi sample after a 10-minute etching time shown in Figure 3c. The resulting PL spectrum shows blue shift with an intensity maximum at 705 nm and an energy gap of

1.75 eV. Strong evidence for the effect of quantum confinement in PSi through shown by PL emission in the visible range and a blue shift.. It has already been documented that PL peak positions move to the blue shift when pore size increases [12-14]. The FTIR spectra of the recently etched PSi nanoparticles after 10 minutes of anodic etching displayed in Figure 4. The band of vibrational modes Si-O-Si is visible in the absorption spectra of the original porous silicon samples at 783 cm⁻¹, 923.99 cm⁻¹, 472.82 and 1106 cm⁻¹. Additionally, spectrum shows bands at 2853 and 2923 and 3449, 1634 cm⁻¹ that are typical of CH and OH stretching vibration. The Si-Si bond is responsible for the other peak at about 613 cm⁻¹ [15-17].

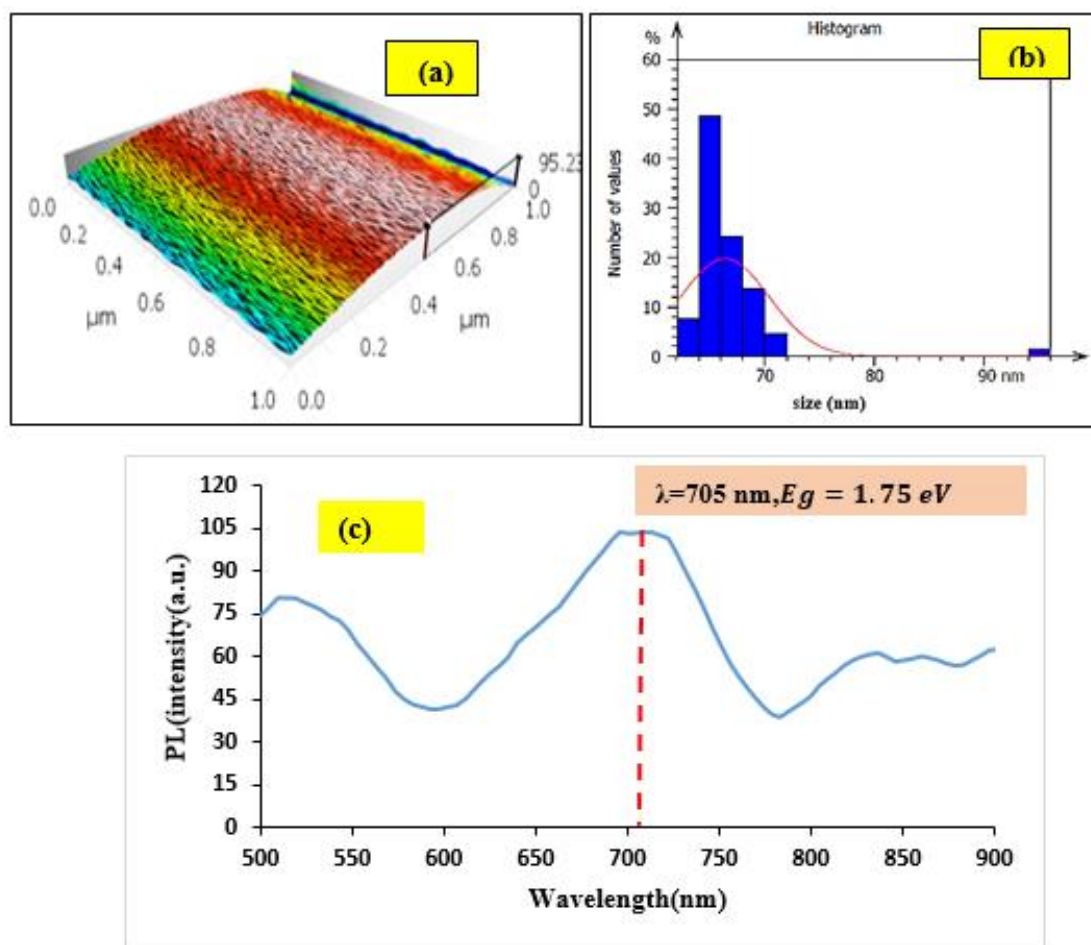


Fig.3(a,b) images of AFM of PSi and (c) photoluminescence (PL) emission of Psi at 10 min etching time

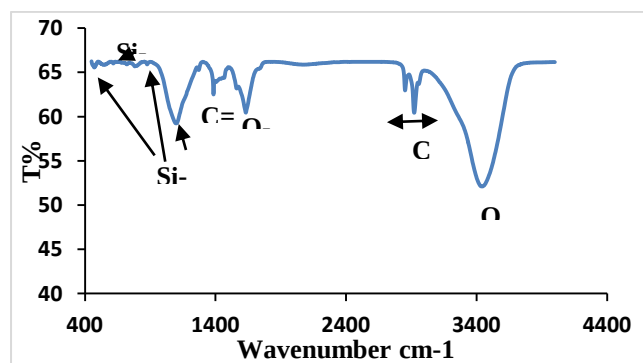


Fig.4 FT-IR spectra PSi.

The planes of SnO₂ at (110), (101), (211), and (112) at 2θ diffraction angle 26.60°, 33.75°, 51.98°, and 64.52°, respectively, as shown by the XRD results of the SnO₂ sample generated by Salvia rosmarinus extract at different concentrations (0.1M and 0.2M) in Figure 5a,b. The four peaks are match of the tetragonal rutile structure for the cassiterite phase

according to card (JCPDS No. 41-1445). The absence of any impurity peaks indicates that the SnO₂ nanoparticles as synthesized are crystalline and pure. The both samples displayed a significant SnO₂ distinct diffraction peaks have same phase as demonstrated by the comparison of the two XRD patterns[18,19]. The Scherrer formula used to estimate the average crystallite size, which was found between 9 and 12 nm. boosting the concentration from 0.1 to 0.2 molar did not alter the material's structure, according to X-ray diffraction, but it did improve the degree of crystallinity by boosting peak intensity. This suggests a decrease in surface imperfections due to a rise in the number of crystals as well as the uniformity and arrangement of atoms in the crystal lattice.

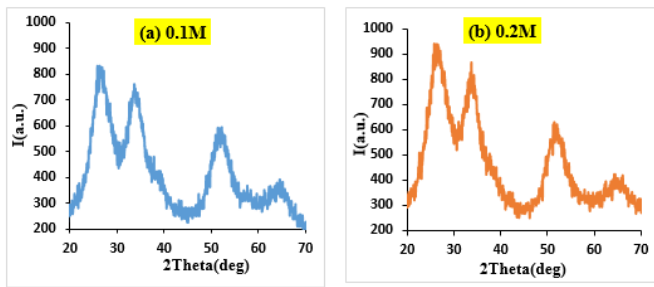


Fig.5. XRD analysis of SnO₂ films at concentrations (a)- 0.1M and (b)0.2M.

The AFM topographies of SnO₂ at different concentrations (0.1M and 0.2M, respectively) using a scanning area of X:2.0 × Y:2.0 μm² are displayed in Figure 6a,b. As the 3-D AFM image of the surface

makes evident, morphology of SnO₂ surface films have a well uniform surface homogeneity and provides a suitable pointer for the building of the SnO₂ film. The films' surface at 0.1M was smooth, with a mean grain size of 73.76 nm, a roughness and RMS 2.17 nm 2.67 nm respectively. The study discovered that at concentrations of (0.2) M. SnO₂ surface film has well distribution of grain and homogeneity, and the size increases to 83.87 nm with the increase of SnO₂ concentration due to the improvement of the film's crystallization state and the growth of the crystallite; additionally, roughness 3.58 nm and RMS 5.61 nm increase with increasing concentration.

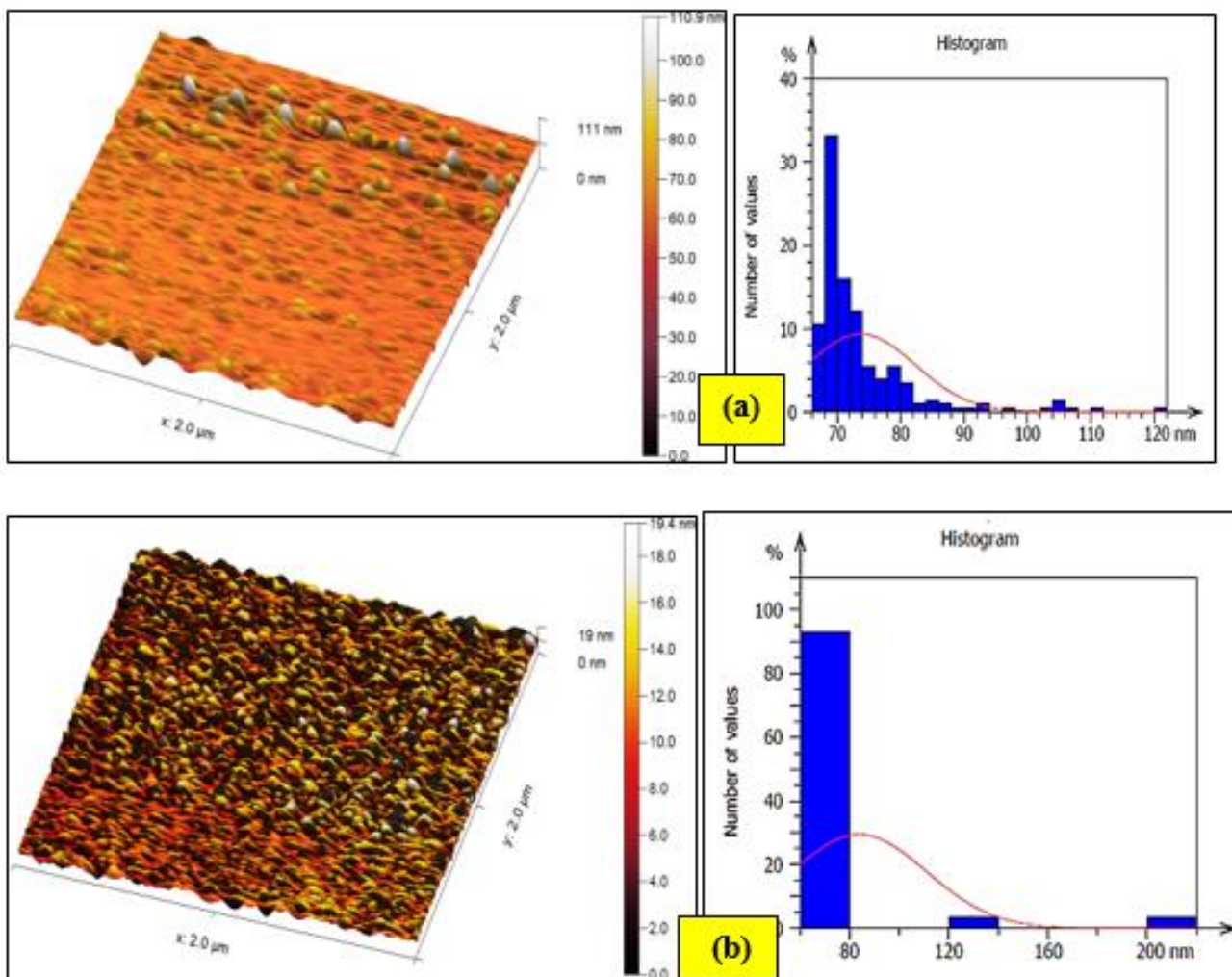


Fig. 6. Image of AFM for films : (a)SnO₂ at 0.1M and (b)SnO₂ at 0.2M .

SEM is a perfect method for evaluating the degree of dispersion, homogeneity, aggregation, and purity of

nanoparticles. SEM is used to examine the samples' morphology. Figure 7a,b illustrates this for SnO₂ at

concentrations of 0.1M and 0.2M, respectively. At a concentration of 0.1, the surface has small, scattered particles without any agglomeration, as seen in the image. Since the concentration is low, particle development is limited, resulting in tiny. However, because of the higher growth rate with increasing concentration, we see the formation of bigger particles at a concentration of 0.2, with distinct agglomerations taking the form of oval and rod forms. Consequently, the extract's ability to prevent agglomeration is diminished, leading to an increase in surface roughness. The AFM findings are supported by these results. It was discovered that the size fell within the real nanoparticle size range 1 -100 nm.

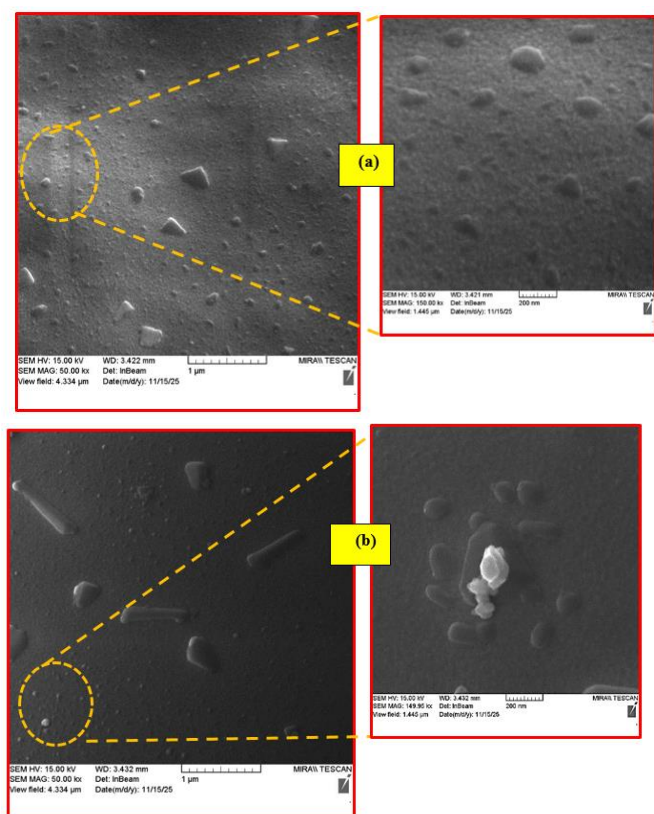


Fig. 7. Image of SEM for SnO₂ films : (a) SnO₂ at 0.1M and (b) SnO₂ at 0.2M .

A relatively simple technique for highlighting the optical features of nanomaterials is absorption spectroscopy. Figure 8 showed the optical absorption spectra of pure SnO₂ nanoparticles at two distinct molar concentrations (0.1 and 0.2 M). The significant UV absorption of the SnO₂ confirmed by the fact

that the two samples show the absorption maxima at 200–400 nm, which denotes an electronic transition from the valence to conduction band (bandgap transition)[20]. Tin oxide becomes transparent in the visible and infrared spectrum because of the absorption quickly decreasing with increasing wavelength. The absorption spectra at a concentration of 0.2M exhibits more absorption than the absorption spectrum at a concentration of 0.1M. This suggests that there are more tin oxide particles in the solution, which leads to higher absorption since there are more active light-absorbing centers.

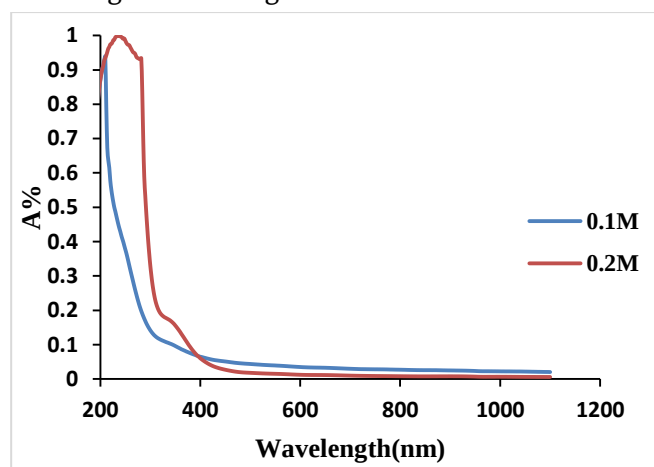


Fig. 8. Absorption spectrum of SnO₂ at concentration 0.1 and 0.2 M.

The gap of energy (E_g) for produced samples calculated from their absorption spectra using Eq. (1) [21]:

$$[(ah\nu)^{1/n} = A(h\nu - E_g)] \dots \dots (1)$$

where (α) optical density, photon energy ($h\nu$), E_g , A , and n represent band gap energy, a constant, and a semiconductor-specific parameter, respectively. As seen in figure 9a,b, the band gap energies (E_g) of the SnO₂ nanoparticles (0.1 and 0.2M) can be obtained by estimating the optical band gap energies of SnO₂NPs for direct transition ($n = 1/2$) from a plot of $(\alpha h\nu)^2$ vs $h\nu$ the intercepts of the tangent with the abscissa axis. By extrapolating the absorption edge onto the energy axis, the band energy of SnO₂ determined to be 3.95 eV at 0.1M and 3.74 eV at 0.2M. As the molar concentration of precursor rises, gap energy of

SnO₂NPs tends to decrease. In contrast, SnO₂ nanoparticles show an increase in energy gap when compared to bulk SnO₂ (3.6 eV). It is commonly known that quantum confinement effects(QCE) cause a blue shift in the spectra of semiconductors of nanoscale sizes [20].

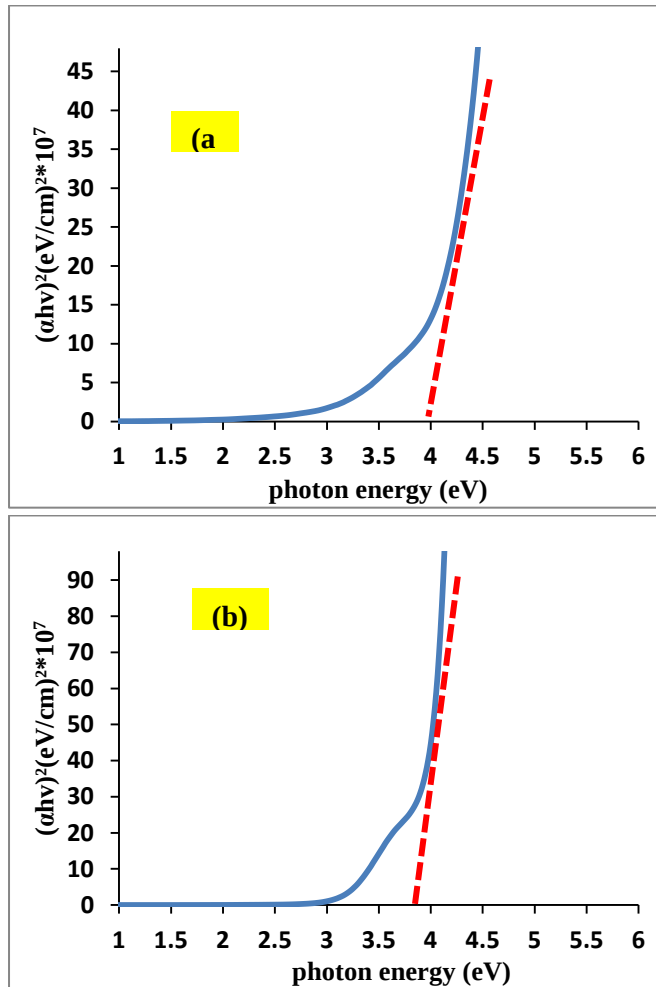


Fig.9. Tauc plot of SnO₂ at concentrations (a) 0.1M and (b) 0.2M.

Figure 10 displays the FTIR analysis of SnO₂ nanoparticles prepared from *Salvia rosmarinus* extract at two distinct concentrations (0.1M and 0.2M) in the (wavenumber =k) range of 450-4000 cm⁻¹. Based on variation in signal intensity, the acquired FTIR data were enough for distinguishing between concentrations. The distinctive band from 615.13 to 870.11 cm⁻¹ created by Sn-O-Sn bond of SnO₂ [37, 38]. The hydroxyl group (-OH) is primarily responsible about band at 3437 cm⁻¹ and 1,636 cm⁻¹

[40]. The presence of these bonds is crucial for reducing ions and stabilizing nanoparticles because they reveal the phenolic group in the extract. In the band region between 2853.33 - 2924.67 cm⁻¹ present of C-H group. The peak at 1384 cm⁻¹ may be caused by Sn-OH vibrations, band region at 1127 cm⁻¹ displays C-O-C group[22-25]. It is noted from the SnO₂ spectrum for the two different concentrations that at 0.2M, the bonds are more pronounced and the transmittance intensity is lower, indicating an increase in the molecular density or Sn-O bonds due to the increased solution concentration. Increasing the concentration (0.2M) improves the interaction between the extract and the tin ions, thus forming a clearer SnO₂ network. While at 0.1M the peaks are smaller and less pronounced, indicating the formation of smaller particles or in smaller quantities.

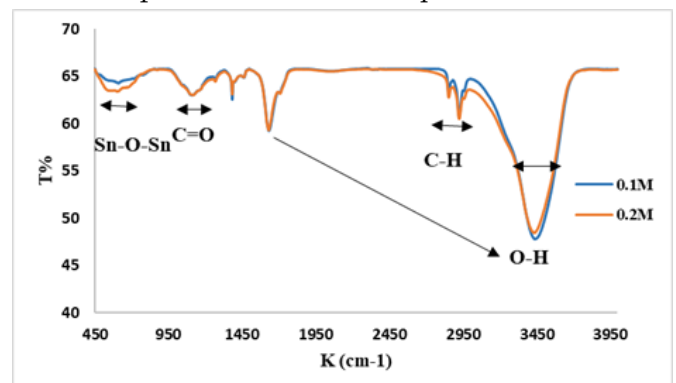


Fig.10. Spectra of FTIR for SnO₂ nano-particles at different concentration.

Fig. 11a,b displays typical I-V features of the Ag/SnO₂/PSi/Si/Ag devices at varied concentrations of 0.1M and 0.2M recorded in the dark at bias voltages(forward and reverse). The forward current rises as the voltage rises due to decrease in the charge trapping centers. As the bias voltage increased, the dark current increased as well. Additionally, the current measured in absence of light for the SnO₂ at 0.2M was greater than that of the SnO₂ at 0.1M device because the built-in potential decreased due to a decrease in the depletion region caused by an increase in the majority carriers via the applied voltage. When illuminated by a halogen lamp (Philips,

100 mW/cm²), the photocurrent rises due to an increase in the reverse voltage, which causes the depletion area to rise with an increase in bias voltage. In reverse-bias, the strong electric field quickly eliminated the generated (e-h) pairs in the depletion width, causing the photocurrent increase through the incident light due to the growing number of carriers (photogenerated) in the depletion layer [26,27].

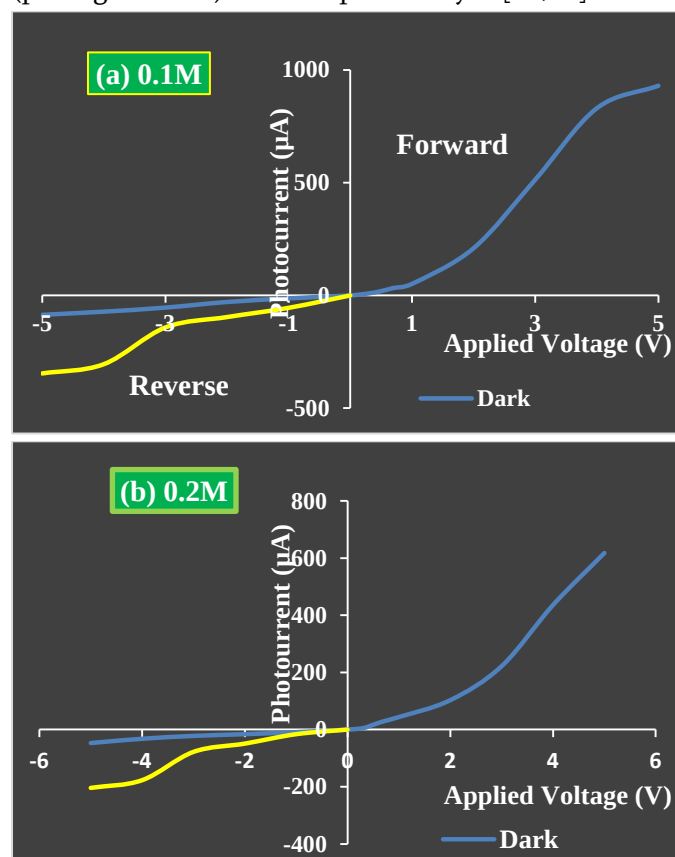
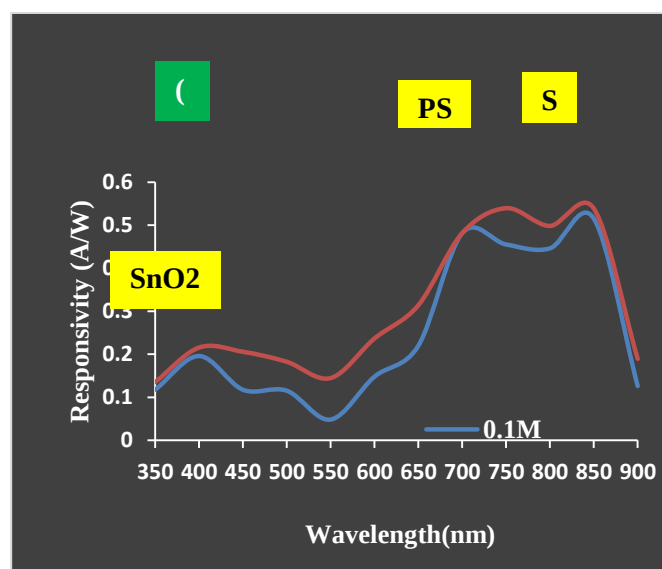


Fig.11. I-V measurement of Ag/SnO₂/PSi/Si/Ag devices at concentrations (a) 0.1M and (b) 0.2M.

Important factors influencing the detectors' actions were responsiveness and particular detectivity[2]. Fig. 12a,b shows the responsivity and specific detectivity of Ag/SnO₂/PSi/Si/Ag at 0.1M and 0.2M in the 350–900 nm range. It is evident that every sample responded well in the three regions of 350–400 nm, 700–750 nm, and 800–850 nm. Due to its huge energy gap, which causes SnO₂ to function in the ultraviolet, the first region linked to the absorption SnO₂ edge. Second region linked to the absorption porous silicon edge at both concentrations. Because porous

silicon may trap light inside its pores, this increases the electron-gap pair. The third region ultimately returns to the silicon edge. Based on the two spectra, it can be shown that the concentration of SnO₂ has no effect in the 800–850 nm range since silicon absorption predominates in this area. Increasing the concentration improves light absorption and electron transport efficiency across the interfaces between the SnO₂, Psi, and Si layers, resulting in improved spectral responsiveness. The responsivity spectra of SnO₂ at 0.2 M concentration are higher than those at 0.1 M concentration. For 0.1M and 0.2M, the greatest photoresponsivity peaked at 400, 750, and 850 nm, respectively, at 0.19, 0.45, and 0.51 A/W, and at 400, 750, and 850 nm, at 0.22, 0.53 A/W. Three zones of specific detection— 1.46×10^{11} jones, 3.08×10^{11} jones, and 3.49×10^{11} jones at concentration 0.1M—emerge because specific detectivity acts similarly to responsivity. These regions achieve their highest values at the same wavelengths, 400, 750, and 850 nm. Figure 12b illustrates 1.72×10^{11} jones and 4.26×10^{11} jones for 400 nm, 750 nm, and 850 nm at concentration 0.2M.



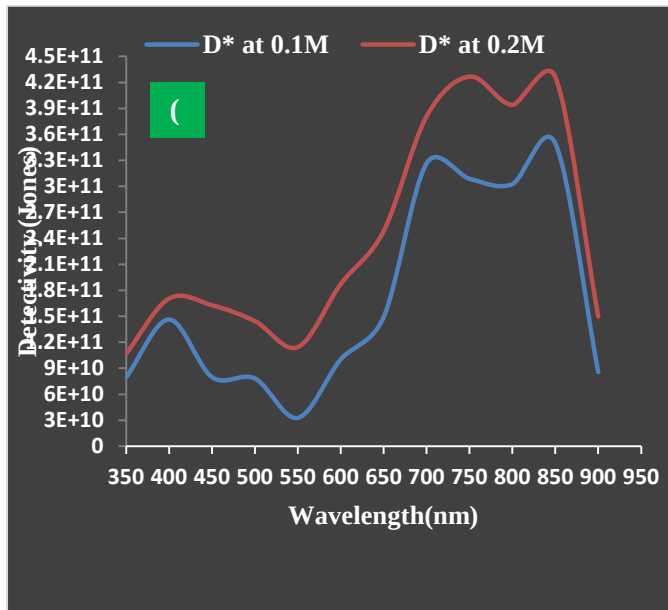


Fig. 12(a) Responsivity and (b) specific detectivity of Ag/SnO₂/Psi/Si/Ag photodetectors at different concentration 0.1M and 0.2M.

CONCLUSIONS

Using the plant extraction approach (rosemary extract), SnO₂ nanoparticles were successfully produced at concentrations of 0.1M and 0.2M. The enhanced peak intensity at the higher concentration suggested a minor improvement in crystallinity and an increase in crystalline size, but XRD findings showed that the crystal structure did not change between the two concentrations. This conclusion was corroborated by AFM data, which showed an increase in density surface, roughness, and grain size at 0.2M, indicating the formation of bigger grains and increased interparticle cohesion with increasing SnO₂ concentration. Although the gap value remained higher than in the bulk state due to surface/defect effects and structural restrictions in the nanolayers, a decrease in the energy gap was observed with increasing concentration, which is expected with increased grain size and decreasing quantum confinement. Furthermore, porous silicon's PL emission revealed that the band gap of 1.75 eV, this value is larger than that of regular silicon and lower than that of SnO₂. This suggests that porous silicon could be used as an efficient absorption layer or base

to improve energy compatibility when combined with SnO₂ films in photodetector applications. The higher concentration of Ag/SnO₂/Psi/Si/Ag was found to provide better performance, higher responsiveness, and specific detectivity when photodetector parameters like responsivity and detectivity were measured. This is explained by the improvement in crystallinity, the decrease in grain barriers that obstruct carrier transport, and the increased light absorption linked to increased roughness, surface area, and defects that produce free carriers (like oxygen vacancies).

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