

Fabrication of Different SnO₂ Nanorods for Enhanced Photocatalytic Degradation and Antibacterial Activity

Govindhan Gnanamoorthy

University of Madras

Virendra Kumar Yadav

Jaipur National University

Krishna Yadav (✉ envirokrishna@gmail.com)

Bundelkhand University Faculty of Science <https://orcid.org/0000-0002-4228-2726>

Kandasamy Ramar

Jamal Mohamed College

Javed Alam

King Saud University

Arun Kumar Shukla

King Saud University

Fekri Abdulraqeb Ahmed Ali

King Saud University

Mansour Alhoshan

King Saud University

Research Article

Keywords: Hydrothermal method, Acid-mediated, Malachite Green dye, Photocatalyst, Antibacterial

Posted Date: February 8th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-157108/v1>

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Version of Record: A version of this preprint was published at Environmental Science and Pollution Research on April 10th, 2021. See the published version at <https://doi.org/10.1007/s11356-021-13627-w>.

Abstract

The acid-mediated (Oxalic acid [OXA], Cinnamic acid [CA], and itaconic acid [IA]) SnO₂ nanorods were synthesized by the hydrothermal method. The synthesized SnO₂ nanorods, in turn, were analyzed with various physic-chemical techniques such as the X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), scanning electron microscope (SEM), and Raman spectroscopy. Furthermore, the photocatalytic activity of the different SnO₂ nanorods was investigated with the malachite green (MG) dye under visible light illumination. The OXA-SnO₂ nanorods displayed an excellent degradation performance with observed values of 91% compared to other nanomaterials' (CA and IA-SnO₂) photocatalytic activity. The different synthesized SnO₂ materials were tested for antibacterial and antifungal studies; this result may be used or developed in future research activities.

1. Introduction

A novel nanomaterial have most attractive and considered due to the photocatalytic applications. recently, food and pharmaceuticals, textile, leather, and paper industries reported materials contains several organic aromatic groups and molecules are produced toxic gas and other moieties, which is highly damage in an environment (Bouras et al. 2019). However, these dyes are highly toxic and affect human, animal, and bird health disorders and damaging ecosystems (Gruzeł et al. 2019). Nowadays, pollution can be controlled with soluble dyes and used in various fields in the emerging world. For instance, Sirajul (2019) reported that the Cd ions incorporated into the SnO₂ nanoparticles are favoured for the absorption process and thermodynamic studies. Meanwhile, the researchers developed SnO₂ based nanomaterials for several applications such as in electrochemical sensors, solar cells, battery studies, fuel cells, supercapacitors, biological studies and photocatalytic activities (Chen et al. 2019; Dilip and Jayaprakash 2018; Honarmand et al. 2019; Mao et al. 2019; Liu 2019; Saravanakumar et al. 2019; Singh et al. 2019; Sousa et al. 2019; Tammina et al. 2019; Wang et al. 2019). In case some people used different toxic chemicals and synthesis methods, it has been explained by the morphology and band gap energy. Therefore, the photocatalytic activity of different acid-mediated SnO₂ nanorods was tested.

Moreover, several metal oxides are currently reported to be used in the field of photocatalysis, such as ZnS based materials, WO₃, (Sn(IV)/TiO₂/AC), Ag/ZnO, ZnSn(OH)₆, N-doped ZnS, PVDF/TiO₂ nanofibers, 3D g-C₃N₄, β-Bi₂O₃@Bi₂S₃, BiOCl nanocubes, MoS₂@Fe₃O₄, and rGO/CdWO₄. However, these types of materials, created for the first time, displayed a better efficiency in photocatalytic performances with a well-known photo-generated recombination used in different light sources with respect to the various organic pollutants and dyes. These semiconductor materials possess more favorable photocatalytic studies boosting the degradation efficiency as well as exhibiting the synergistic effects (Dong et al. 2017; Yu et al. 2018; Sun et al. 2018; Cai et al. 2019; Sun et al. 2006; Tie et al. 2018; Tan et al. 2018; Tie et al. 2019; Zhang et al. 2017; Tie et al. 2019; Heo et al. 2019; Dong et al. 2019a; Dong et al. 2019b).

This study aims to introduce the SnO₂ nanorods which have a tetragonal crystal system with the materials having been characterized by the surface morphology and various physic-chemical methods. The SnO₂ nanorod has a few advantages including being facile, eco-friendly, easily available, having mild reactions and reduced toxicity, and lower costs of the synthesized materials. The synthesized OXA, CA, and IA-SnO₂ nanomaterials were used for the photocatalytic studies under the visible light source with the malachite green dye. However, the OXA-SnO₂ nanomaterials gained a higher efficiency when compared with the CA and IA-SnO₂ nanomaterials.

2. Materials And Methods

2.1. Materials

The number of standard chemicals required for the material synthesis was the recommended analytical grade chemicals, such as stannous chloride (SnCl₂·2H₂O), Oxalic acid (C₂H₂O₄), Cinnamic acid (C₉H₈O₂), and Itaconic acid (C₅H₆O₄), with NaOH materials with Methanol and Ethanol used as solvents.

2.2. Synthesis of SnO₂ nanorods

The SnO₂ nanorods were first synthesized by the hydrothermal method. In a typical procedure, 1 mm of the SnCl₂·2H₂O was dissolved in methanol, making three sets with each set of the solution added dropwise in different acids such as oxalic acid, cinnamic acid, and itaconic acid. These precursors were stirring for 1 hour, then, the liquid NaOH (0.1M) was added and the solution stirred overnight for 12 hours. The obtained homogeneous solution was then transferred to the stainless-steel autoclave, maintaining the temperature at 180 °C. The obtained product was washed with ethanol and water several times, followed by drying at a vacuum oven at 25 °C overnight.

2.3. Antibacterial studies

The antibacterial activity of the SnO₂ nanorods was evaluated for the different pathogens by using the agar well diffusion method (Gnanamoorthy et al. 2020). This bacterial analysis was performed by the antibiotic condition. The synthesized SnO₂ nanorod was tested to the *Staphylococcus aureus* (Gram-positive), *Escherichia coli* (Gram-negative), *Pseudomonas aeruginosa* (Gram-negative), *Candida albicans* (Antifungal), and *Enterococcus faecalis* bacteria (Gram-positive)—all cultured from the Mueller-Hinton agar with incubated temperatures at 32–35 °C for 48 hours. The 0.9% saline solution was used for washing with observed bacterial strain intensity OD values for 0.5 at 571 nm, and then different higher concentrations (100 µg/mL, 200 µg/mL, and 500 µg/mL) were added in the well and different positive controls (20 µg/mL) (*S. Aureus*, *E. Faecalis* – Amoxicillin, *E. Coli*, *P. Aeruginosa* – Levofloxacin, *C. Albicans* – Fluconazole) were used, after being measured for the zone of inhibition.

2.4. Characterization

The three different synthesized SnO₂ nanomaterials were characterized and confirmed by XRD (Rigaku, Dmax-2500) with the surface morphology images captured by SEM (Hitachi, S-4800). The phase orientation confirmation was recorded by FT-IR and Raman spectroscopy (WQF-410 and LABRAM-HR system with laser excitation of 514.5 nm). The dye degradation studies were carried out by UV-Visible spectroscopy (Hitachi U-3010). The photocatalytic measurement was recorded by the photocatalytic reactor (Techinstro).

3. Results And Discussion

3.1. Structural analysis

The three different SnO₂ nanorods were synthesized by the hydrothermal method. Fig.1 (a-c) shows the XRD patterns of the synthesized materials and observed all the diffraction Bragg peak positions compared to the reference and well-matched with JCPDS card no 10–1854. The sharp peaks seem to assemble SnO₂ nanorods with different acids' sample methods. The obtained diffraction peaks at 26.0°, 33.9°, 38.1°, 51.8°, 54.2°, 57.9°, 61.9°, 64.7°, 65.9°, 71.3°, and 78.6° corresponding to the (110), (101), (200), (211), (220), (002), (120), (112), (301), (250), and (321) planes. The synthesized SnO₂ nanorods were identified the tetragonal system in all three samples. The CA and IA-SnO₂ samples obtained for the lower intensity had some differences when compared to the OXA-SnO₂ nanorods. These three acid-mediated SnO₂ nanorods showed diffraction peaks without shifting 2θ values than the lattice parameter which also increased. The OXA, CA, and IA-SnO₂ nanorods crystallite size were calculated by the Debye-Scherrer formula (Eqn-1),

$$D = K\lambda / \beta \cos \theta \quad 1$$

Here, K is the shape factor, λ the wavelength, and θ the diffraction angle. The evaluated crystallite size is in decreasing order of the materials, OXA-SnO₂ > CA-SnO₂ > IA-SnO₂ nanomaterials, 38 nm, 37 nm, and 32 nm, respectively.

3.2. FT-IR spectroscopy

The chemical composition of the three different synthesized SnO₂ nanorods was characterized by the FT-IR spectroscopy method. The SnO₂ nanorods' FT-IR comparison spectra is shown in Fig. 2 (a-c) and evaluated by the several vibration peaks. The vibration peak at 400–620 cm⁻¹ corresponds to the O-Sn-O and Sn-O stretching vibrations which is similar to the previously reported work (Ali et al. 2019; Chen et al. 2012). The peak 1608 cm⁻¹ is attributed to the bending vibration modes of the N-H group and 3340 cm⁻¹ can be ascribed to the O-H stretching or N-H stretching vibrations of absorbed water molecules (Liu et al. 2019). The CA-SnO₂ nanoparticles' vibration peak transmittance has been decreased depending on the formation of molecules. Hence, synthesized SnO₂ functional groups were confirmed and the results coincide with the Raman spectroscopy. Therefore, the materials' functional groups were confirmed, which has been used for the subsequent application process.

3.3. Raman spectroscopy

Fig. 3 (a-c) shows the Raman spectra of the OXA, CA, and IA-SnO₂ nanorods, the three peaks appeared with good agreement results. The overall characteristic peaks are at 240, 472, and 627 cm⁻¹ which are associated with the A_{1g} vibration mode at 625 cm⁻¹. The peak 240 and 472 cm⁻¹ are the E_g vibration modes of different SnO₂ nanorods (Hui Liu et al. 2019). The other peak at 240 cm⁻¹ is the companion peak associated with the vibration $\nu_1^c(A_1^c)$ of the edge-sharing of the SnO₂ structure.

3.4. DRS UV-Visible spectroscopy

The OXA, CA, and IA-SnO₂ nanorods' diffuse reflectance spectroscopy results were shown in Fig. 4 (a-c). The SnO₂ nanorods were synthesized at a temperature of 180°C and the optical band gap energy was evaluated using the Kubelka-Munk equation (given below Eqn-2).

$$\alpha h\nu = A(h\nu - E_g)^n \quad 2$$

Where α is the proportionality constant, A the absorption coefficient, $h\nu$ the Planck constant, and E_g the band gap respectively. The obtained DRS-UV results show that the OXA, CA, and IA-SnO₂ nanorods have identified red shift regions of the transition. The OXA, CA, and IA-SnO₂ nanoparticles' band gap energies were at 2.5 eV, 2.8 eV, and 3.0 eV, while preparing the low band gap energy compared to the reported band gap values (Chen 2019 et al.; Kar et al. 2019). The OXA-SnO₂ nanoparticles exhibited lower band gap energies when compared to the CA and IA-SnO₂ materials. As a result, all these SnO₂ nanomaterials were used to enhance the photocatalytic performances.

3.5. Morphology studies

Fig. 5 (a-d) shows the scanning electron microscope images of OXA-SnO₂ and these images explained by the nanorod like structure with bundles of rods edge-to-edge carefully merging with each other and obtained with a diameter range of 3-1 μ m. The CA-SnO₂ surface morphology is shown in Fig. 6 (a-d), the synthesized material has shown layers like structure with diameter range is 1 μ m. The IA-SnO₂ surface morphology is shown in Fig. 7 (a-d) and the morphology illustrated that the tube tablet-like structure with a diameter range of 300 to 500 nm. All the synthesized SnO₂ nanomaterials were confirmed by various techniques and have shown low band gap energy, therefore, the photocatalytic activity should be enhanced.

3.6. Photocatalytic activity

Fig. 8-10 (a-d) explains that the absorption spectra of the MG photodegradation in the presence of different SnO₂ nanorods showed a maximum absorption range at 630 nm. Fig. 8-10 (a) shows the evidence of the photocatalyst dye degradations of the MG under visible light illumination of 500 nm, Fig. 8-10 (b) displays the function of time (C_t/C_0) and the photodegradation with respect to concentration and

the reaction time. Fig. 8-10 (c) illustrates the calculated degradation percentage, here the OXA-SnO₂ nanorods have been shown an excellent degradation compared to other mediated SnO₂ nanomaterials, the kinetic first order scan rate was calculated and the value found to be R²-0.997, 0.997, and 0.988 and the slope value 0.0299, 0.0184, and 0.034 for OXA-SnO₂, CA-SnO₂ and IA-SnO₂ respectively. However, the intensity of the adsorption peak decreased within 90, 60, and 50 minutes, the monitored degradation (Fig. 8-10 (d)) process percentages at 91, 78, and 66. Summarized, the synthesized different SnO₂ nanorods evaluated an excellent photocatalytic performance under the visible light source.

The obtained degradation efficiency of the SnO₂ nanomaterials such as OXA-SnO₂ and CA-SnO₂ nanoparticles are 78% and 66%, the results of which show low degradation efficiency with electron transfer when compared to the OXA-SnO₂ due to it having low bandgap energy. This band gap energy plays a key role in the formation of desirable defects of suitable photocatalytic behavior. In the presence of visible light, the acid-mediated SnO₂ nanomaterials produced charge recombination barriers in the valance and conduction bands. For the conduction band, the whole pair of H₂O/OH⁻ interacted with the Hydroxyl (OH[•]) in a radical formation, the conduction band of O₂ produced to the O₂^{-•} and HO₂[•] were converted to H₂O₂ and OH[•] formation which strongly separates the radical formation. The detailed mechanism has been displayed in Fig.11.

Therefore, the above experimental results suggest that the synthesized SnO₂ nanomaterial was enhanced to the photocatalytic dye degradation and the OXA-SnO₂ nanorods have enhanced the photocatalytic activity when compared to other reported materials (Kaviyarasu et al. 2016).

3.7. Antibacterial and antifungal activities

SnO₂ nanoparticles were analyzed for antibacterial and antifungal activities, which corresponds to the *Staphylococcus aureus* (Gram-positive), *Escherichia coli* (Gram-negative), *Pseudomonas aeruginosa* (Gram-negative), *Candida albicans* (Antifungal), and *Enterococcus faecalis* bacteria (Gram-positive) zones of inhibition shown in Fig. 12 (a-e). Here, the first three bacteria (*Staphylococcus aureus* (Gram-positive), *Escherichia coli* (Gram-negative), *Pseudomonas aeruginosa* (Gram-negative) have higher antibacterial activity due to the particle size and Sn²⁺ ions (Hada et al. 2018). The *Candida albicans* (Antifungal) and *Enterococcus faecalis* bacteria (Gram-positive) are not inhibited by the activity (Table-1). Among them, the strains were tested only for higher concentrations, as the lower concentration did not support the activity. Therefore, this SnO₂ material was enhancing antibacterial and antifungal activities.

Table 1. Antibacterial activity against different pathogens with positive controls of Amoxicillin, Levofloxacin, Fluconazole.

S. No	Pathogens	1mg/mL	2mg/mL	5mg/mL	Positive Control	Negative Control
1	S. Aureus	28	30	32	32	-
2	E. Coli	28	28	30	34	-
3	<i>P. Aeruginosa</i>	25	27	32	38	-
4	<i>C. Albicans</i>	-	-	-	-	-
5	<i>E. Faecalis</i>	-	-	-	-	-

* Zone of inhibition: Concentration (20 µg/ml) used for different positive control.

4. Conclusion

All three different SnO₂ nanorods were synthesized by the hydrothermal method. The prepared nanomaterial structure, functional groups, and the surface morphology were investigated and confirmed by the XRD, FT-IR, SEM, and Raman analysis. The XRD results confirmed the tetragonal structure of the SnO₂ nanorods and the metal oxide functional groups were identified and confirmed by the FT-IR analysis. Additionally, the samples showed nanorod like structures. All the obtained SnO₂ nanorods applied the photocatalytic performances with malachite green dye. The synthesized SnO₂ nanomaterials enhanced the degradation; meanwhile, the OXA-SnO₂ nanorods displayed an excellent photocatalytic performance when compared to the other materials. The OXA-SnO₂ nanomaterials enhanced the antibacterial activity.

Declarations

Ethical Approval

Not applicable

Consent to Participate

Not applicable

Consent to Publish

Not applicable

Authors Contributions

Govindhan Gnanamoorthy: Conceptualization, Methodology, Writing- Original Draft, Writing- Review and Editing. **Virendra Kumar Yadav:** Data Curation, Writing- Original Draft, Review and Editing. **Krishna Kumar Yadav:** Resources, Writing- Original Draft, Writing- Review and Editing. **Kandasamy Ramar:** Writing- Original Draft, Review and Editing. **Javed Alam:** Project Administrator, Resources, Writing- Review and Editing. **Arun Kumar Shukla:** Validation, Writing- Original Draft, Formal analysis. **Fekri Abdulraqeb Ahmed Ali:** Formal analysis, Writing- Original Draft, Review and Editing. **Mansour Alhoshan:** Writing- Original Draft, Writing- Review and Editing.

Funding

The authors extend their appreciation to the Deputyship for Research & Innovation, “Ministry of Education” in Saudi Arabia for funding this research work through the project number IFKSURG-1439-085.

Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Availability of data and materials

Data sharing not applicable to this article as no datasets were generated or analyzed during the current study.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgment

The authors are indebted to the Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai, for all the valuable support and needful facilities.

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Figures

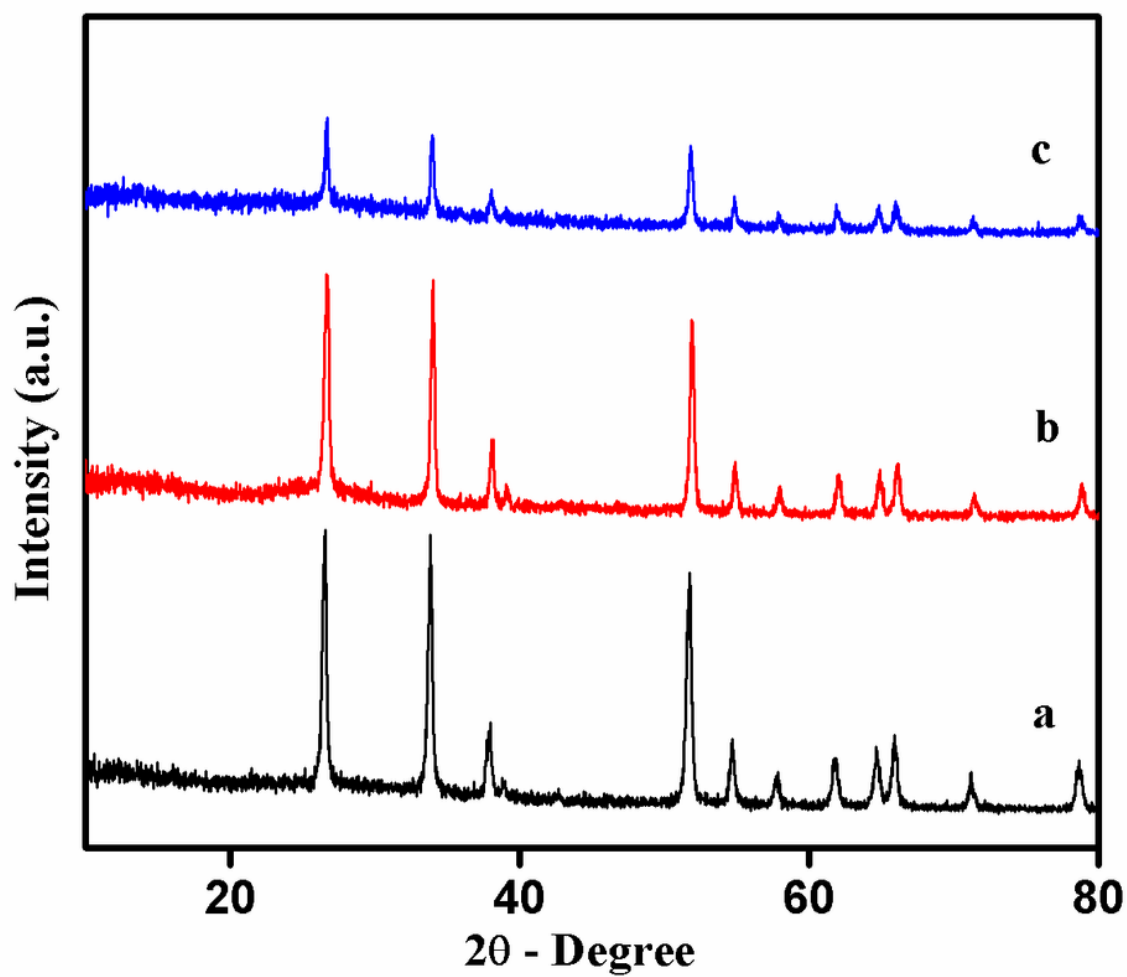


Figure 1

(a-c) XRD patterns of (a) OXA, (b) CA, and (c) IA-SnO₂ nanorods.

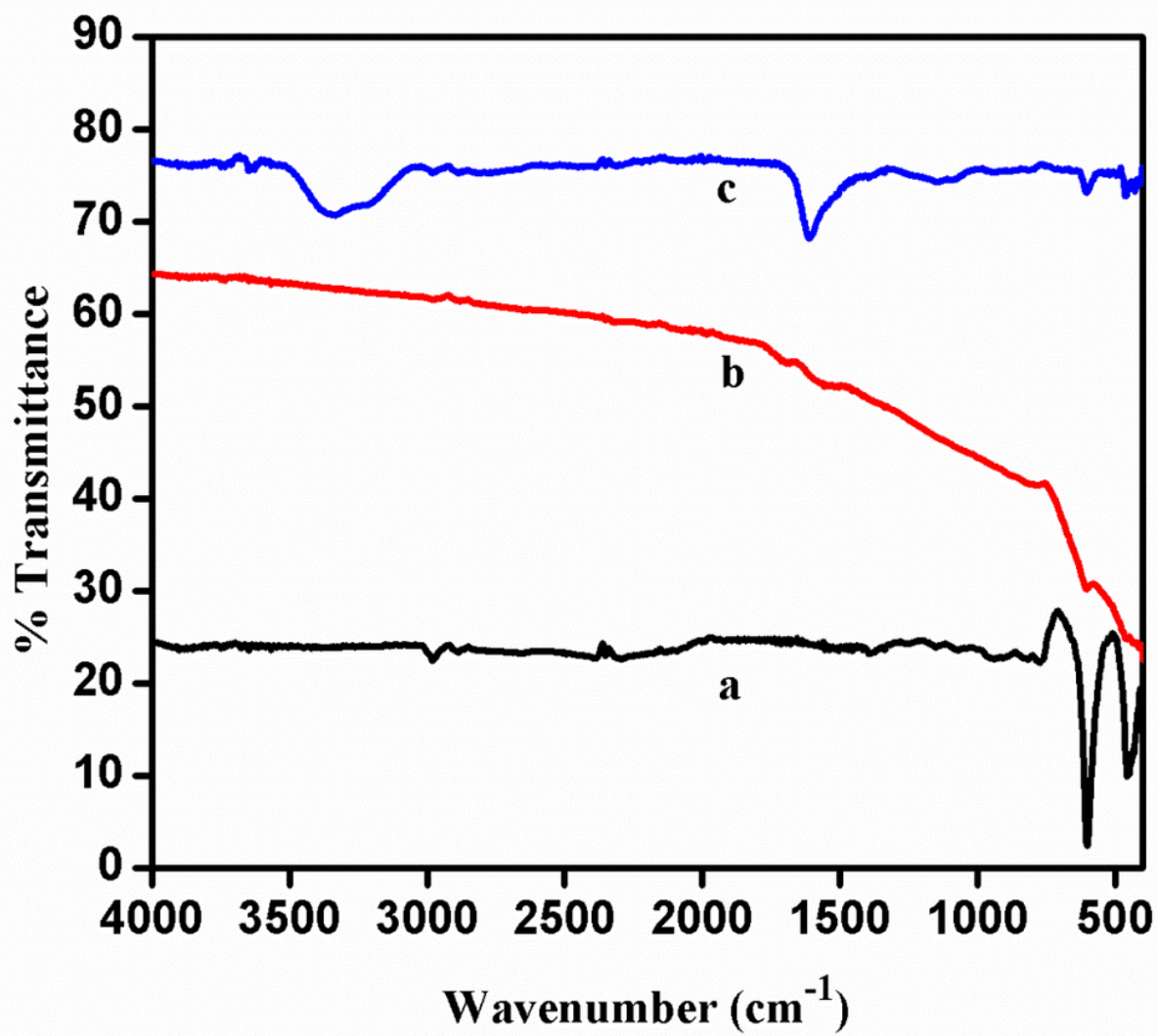


Figure 2

(a-c) FT-IR spectra of (a) OXA, (b) CA and (c) IA-SnO₂ nanorods.

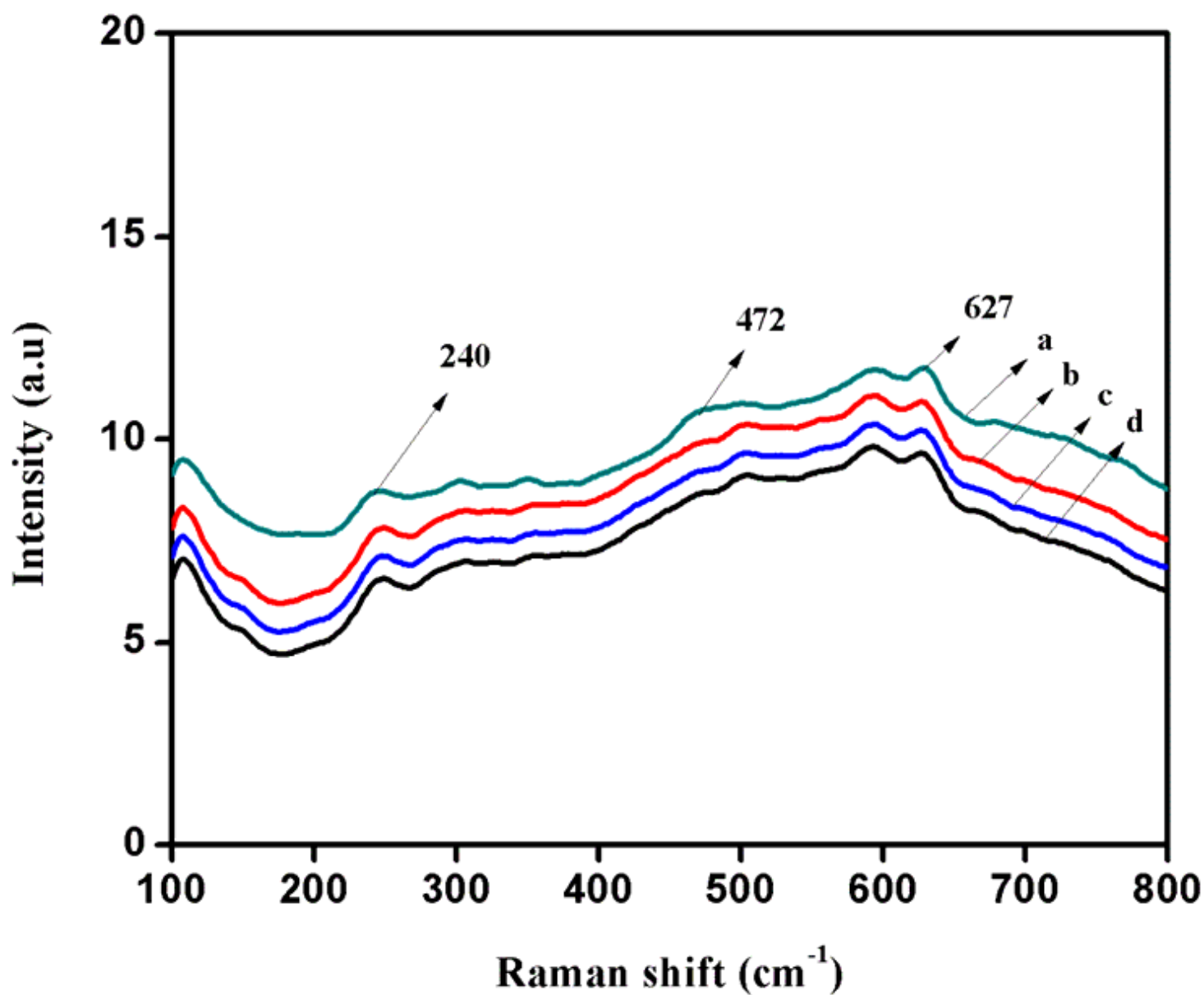


Figure 3

(a-c) Raman spectra of (a) OXA, (b) CA and (c) IA-SnO₂ nanorods.

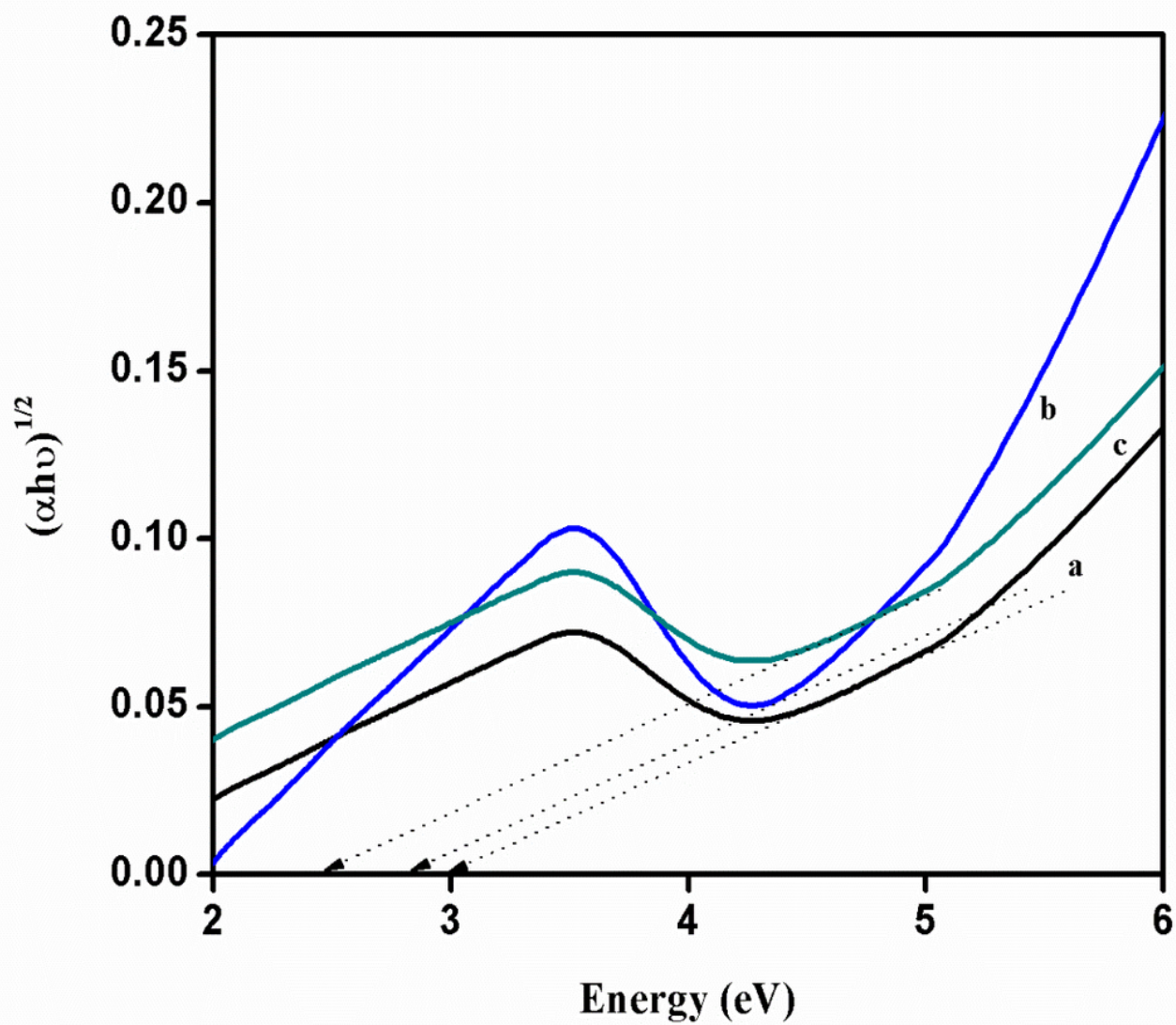


Figure 4

(a-c) Band gap energy of acid mediated (a) IA, (b) CA and (c) OXA-SnO₂ nanorods.

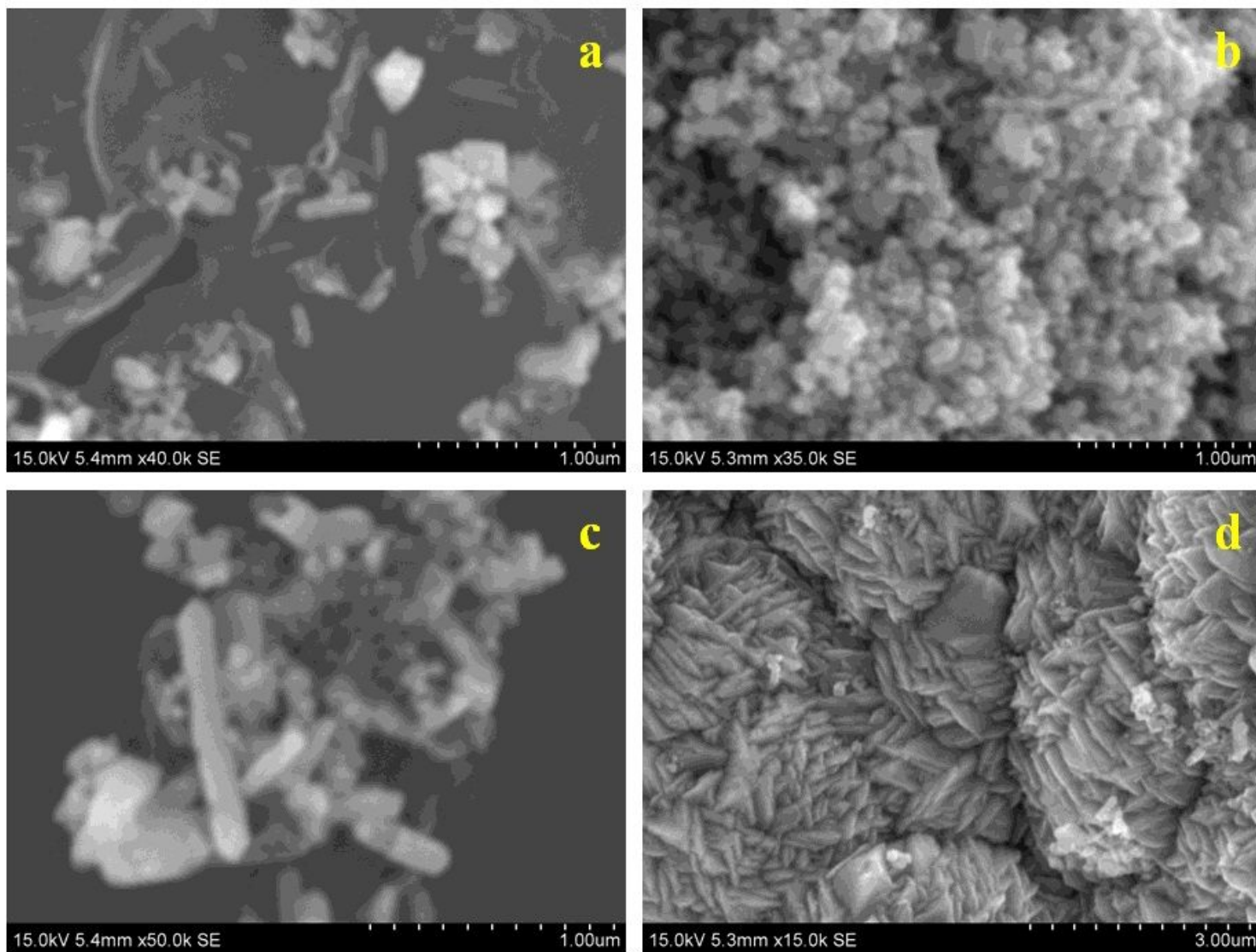


Figure 5

(a-d) SEM images of OXA-SnO₂ nanorods.

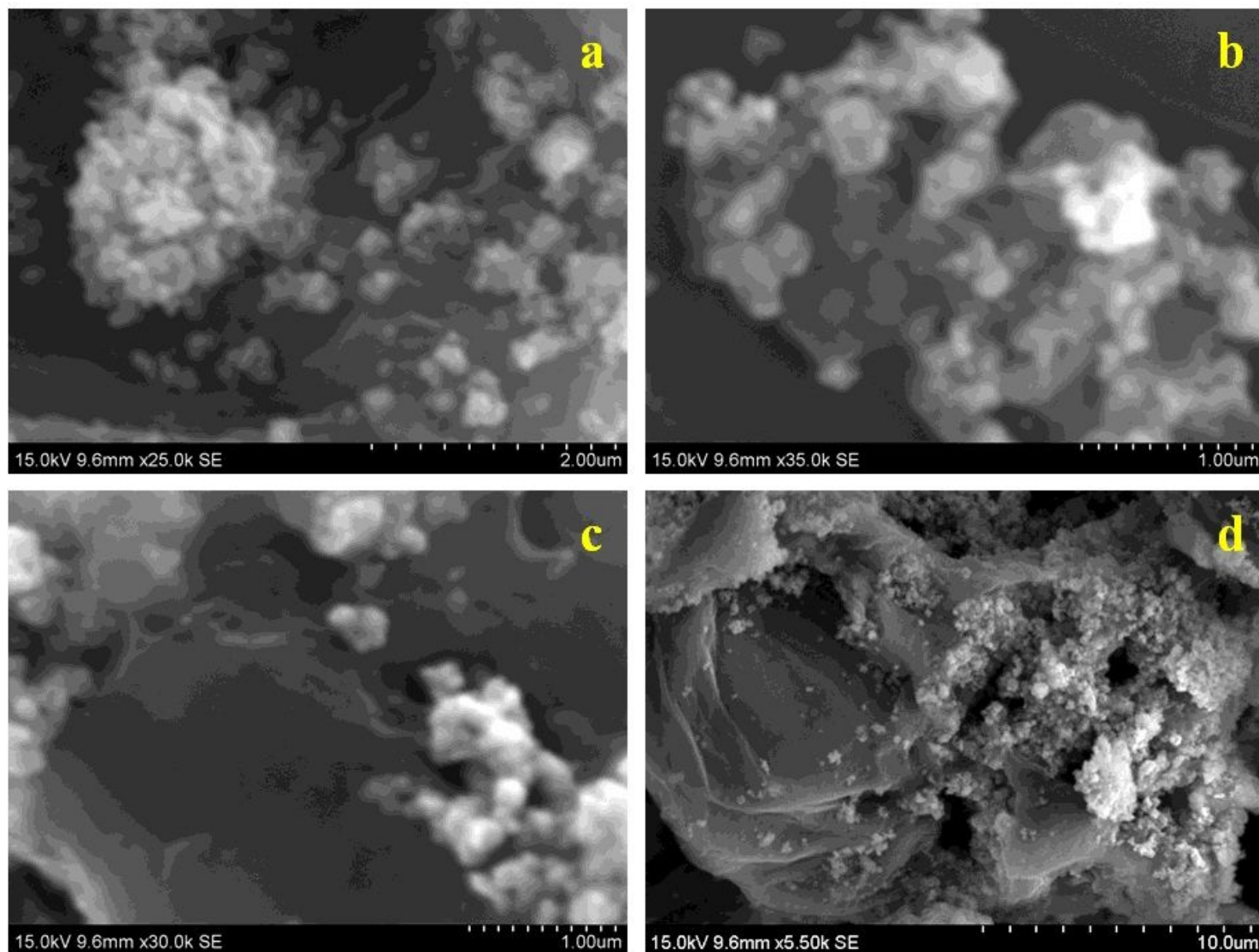


Figure 6

(a-d) SEM images of CA-SnO₂ nanorods.

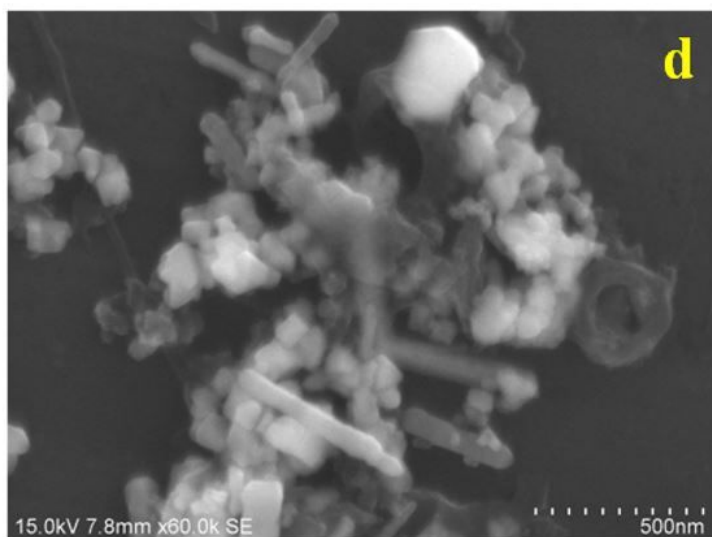
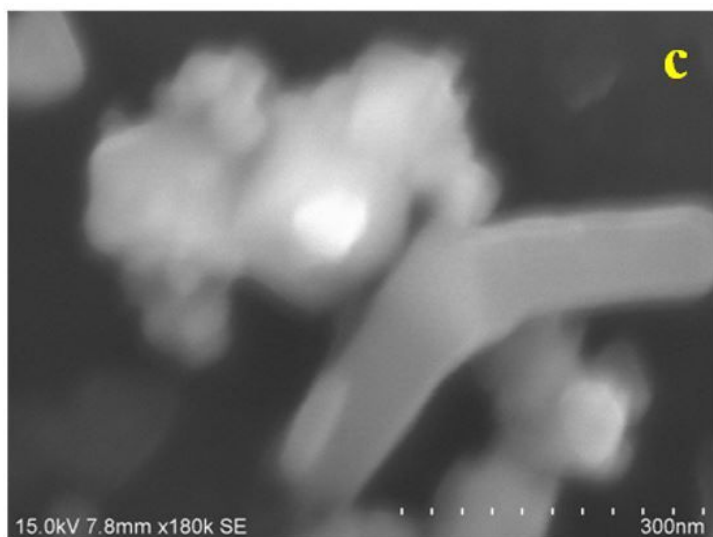
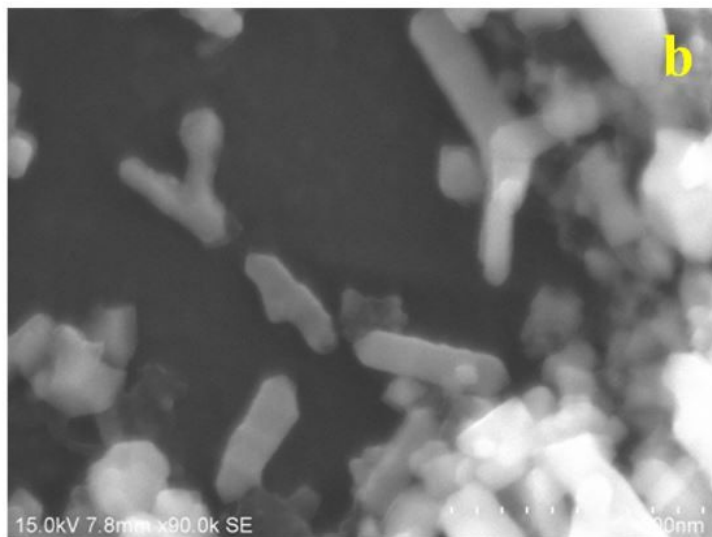
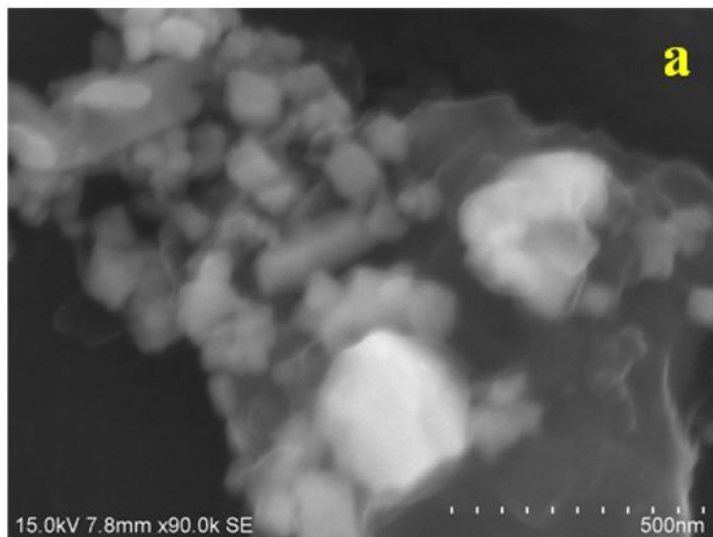


Figure 7

(a-d) SEM images of IA-SnO₂ nanorods.

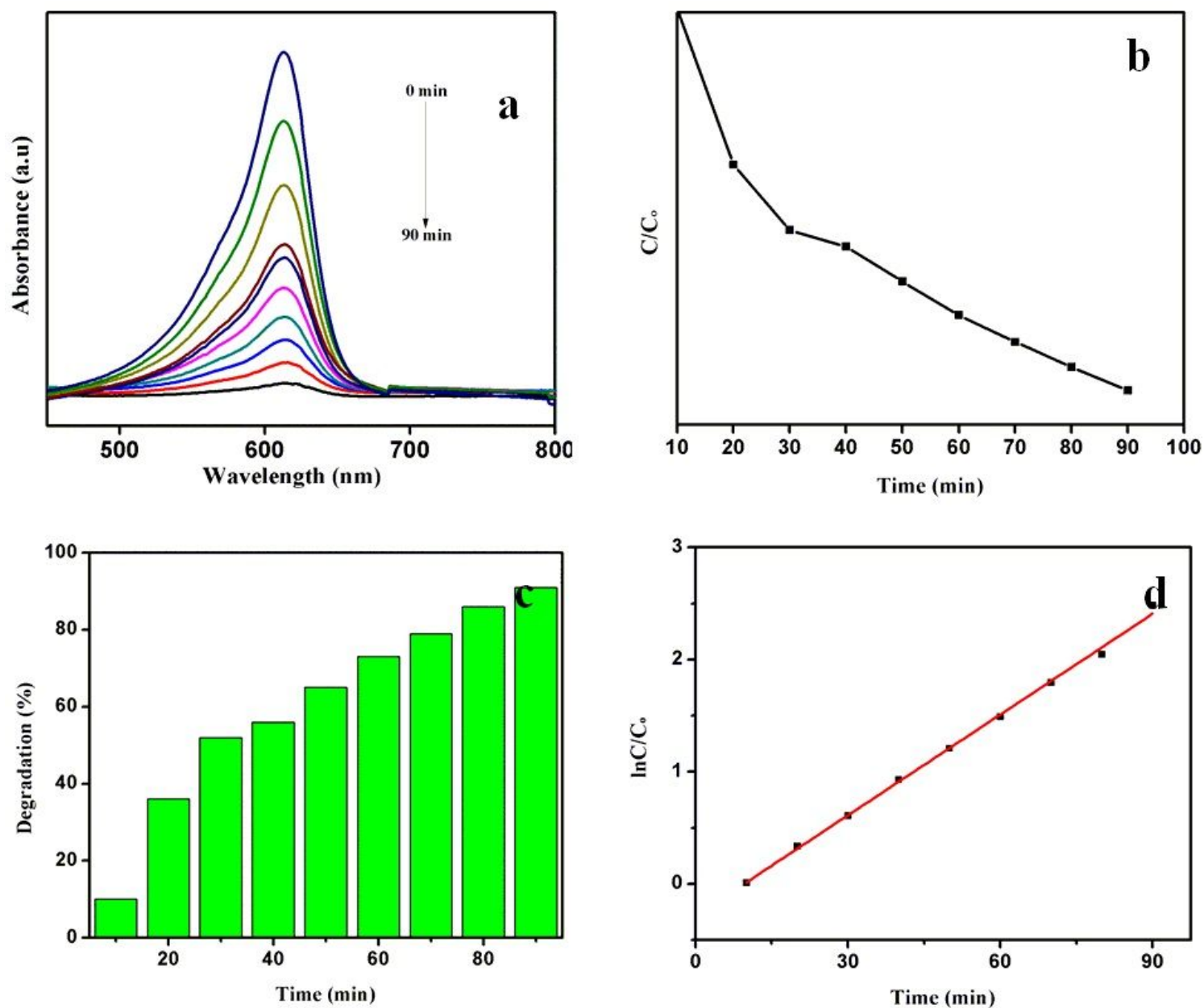


Figure 8

(a) Dye degradation (b) Concentration of C/C_0 (c) percentage of degradation and (d) Rate constant of OXA-SnO₂ nanorods.

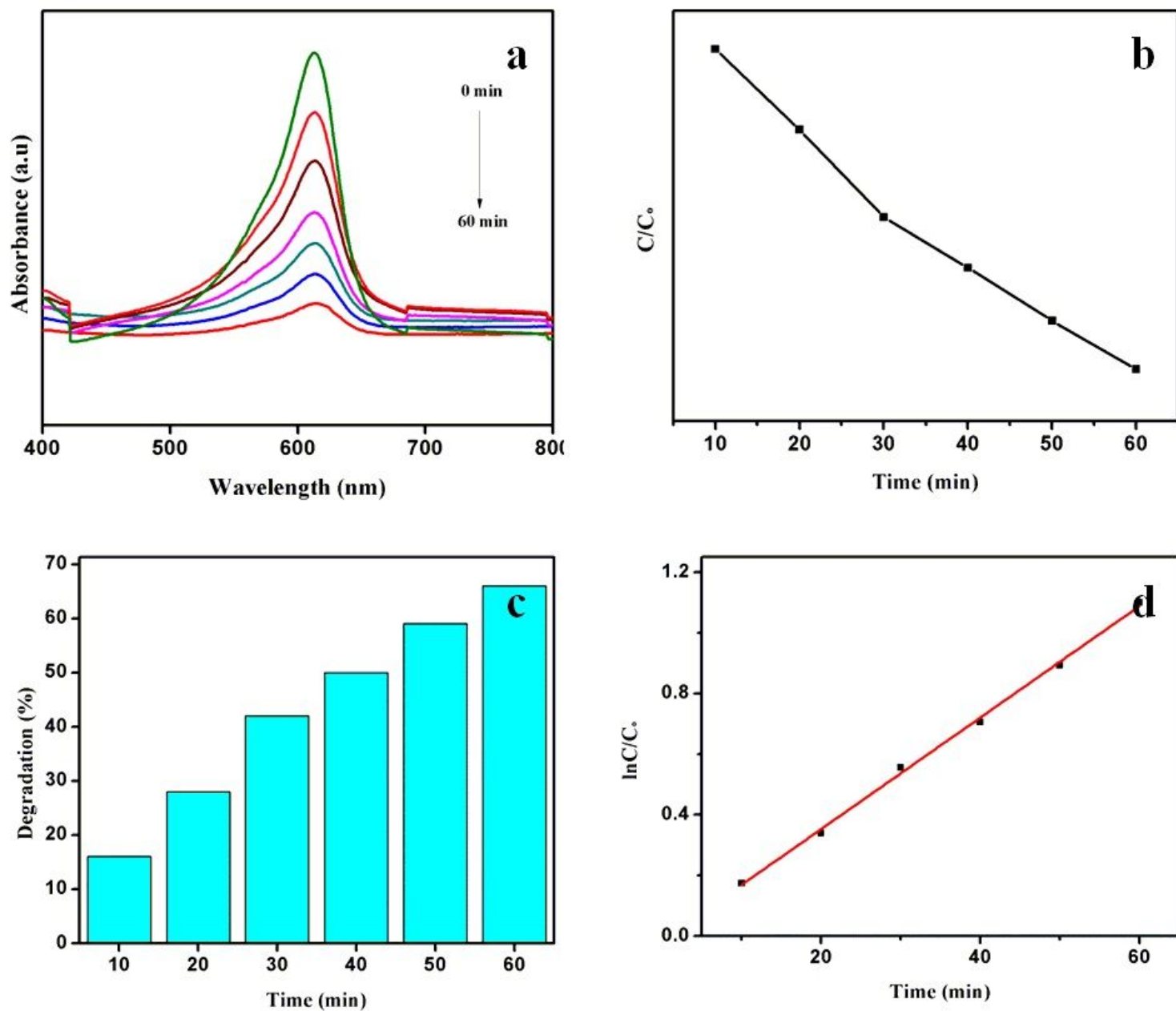


Figure 9

(a) Dye degradation (b) Concentration of C/C_0 (c) percentage of degradation and (d) Rate constant of CA-SnO₂ nanorods.

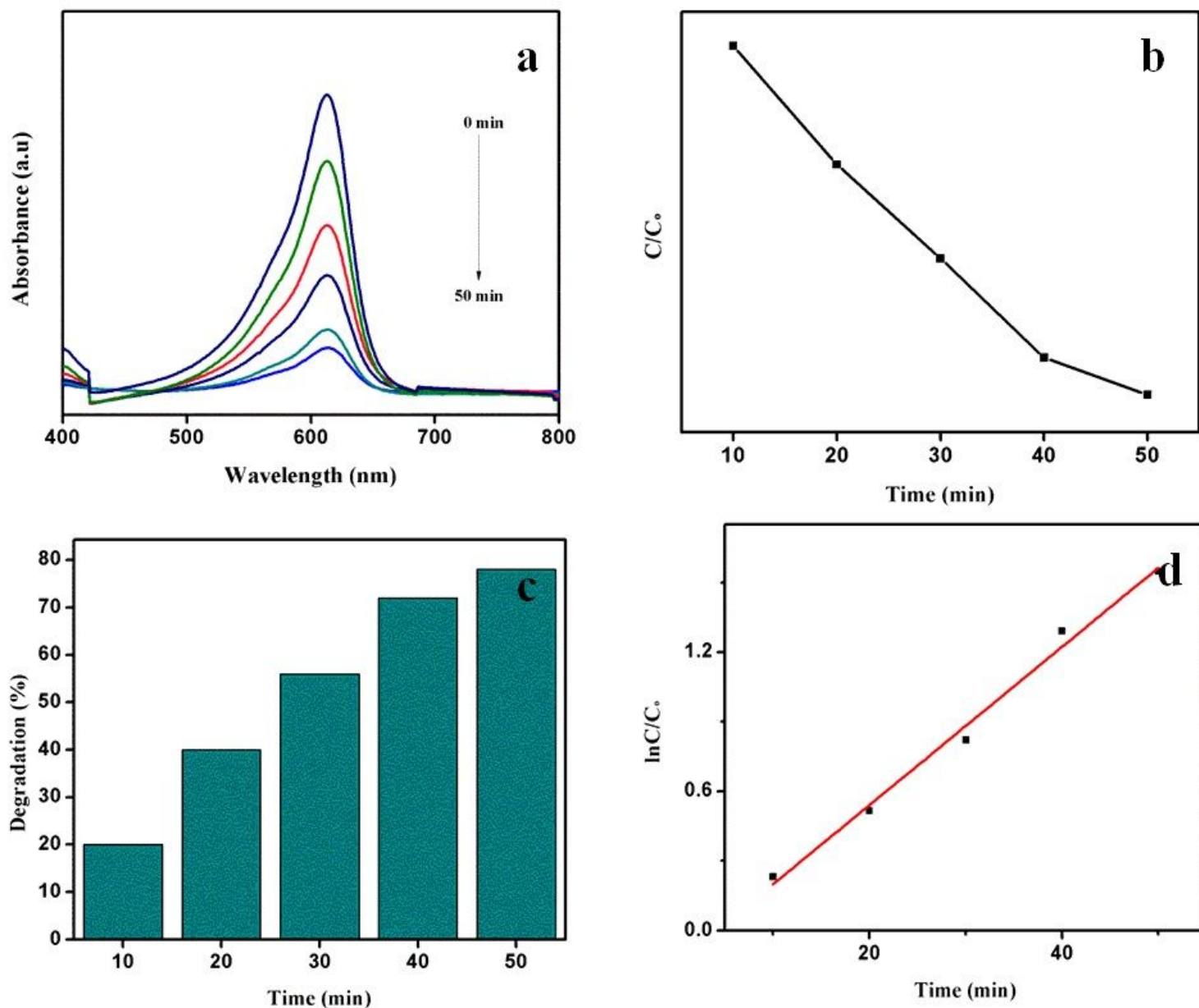


Figure 10

(a) Dye degradation (b) Concentration of C/C_0 (c) percentage of degradation and (d) Rate constant of IA-SnO₂ nanorods.

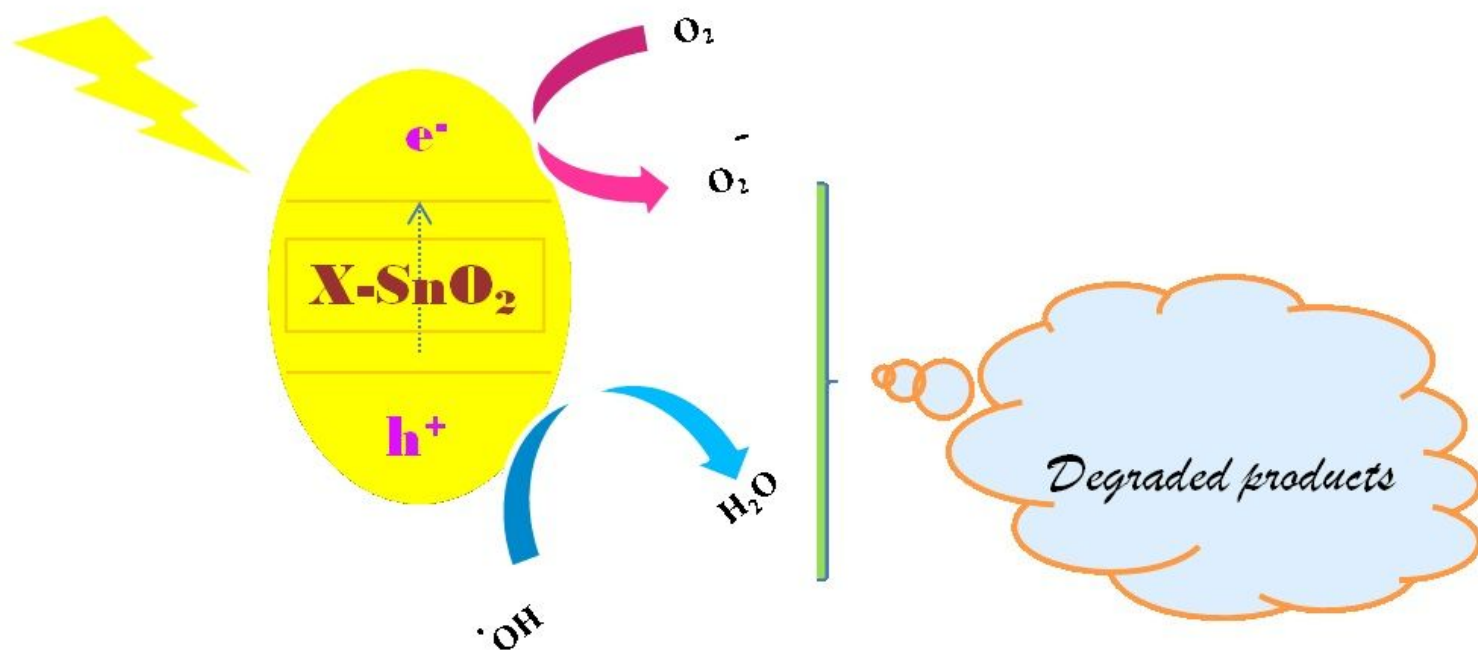


Figure 11

Degradation mechanism of X-SnO₂ nanomaterials.

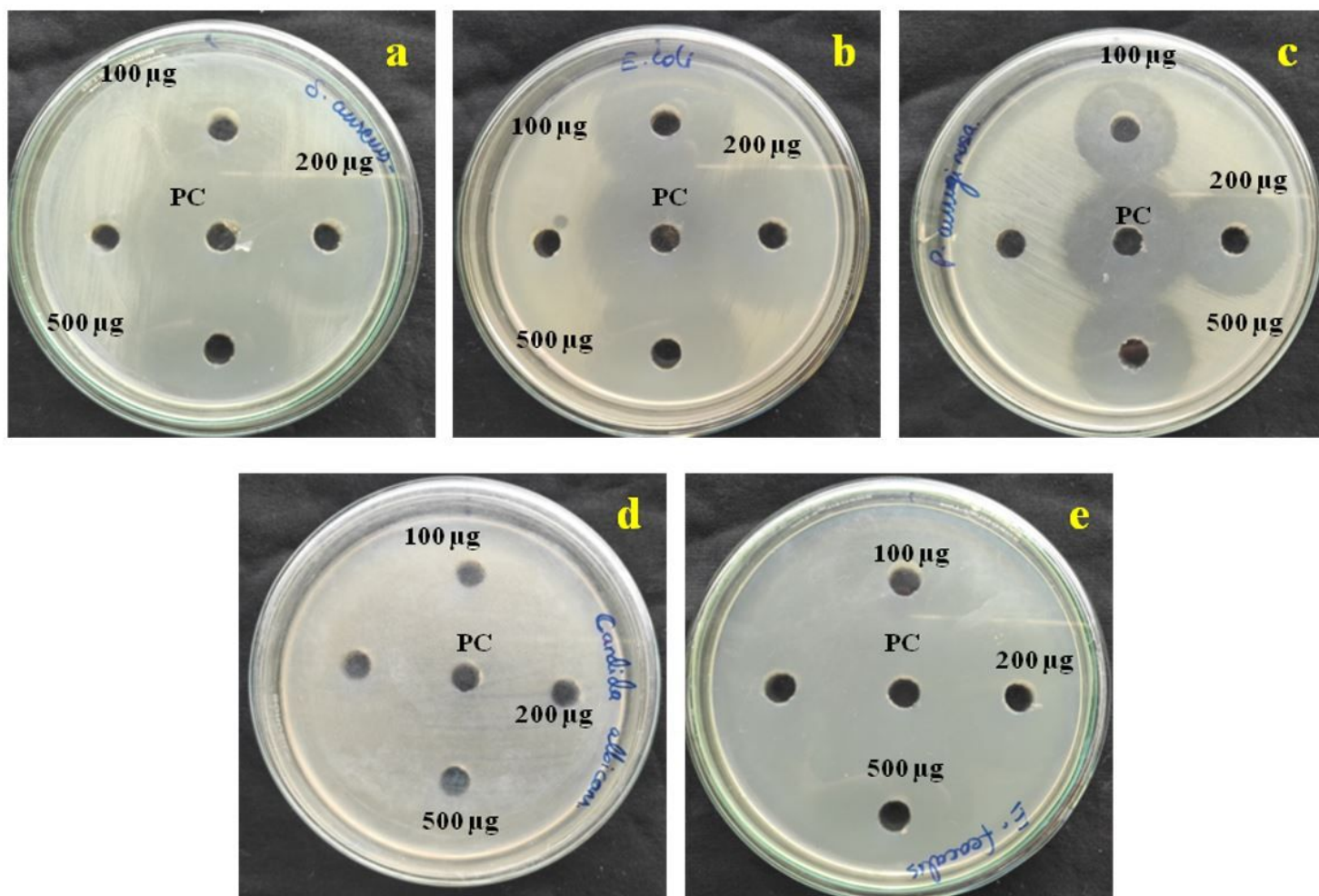


Figure 12

SnO₂ nanoparticles Antibacterial and Antifungal studies: (a) *S. Aureus* (Gram-positive), (b) *E. Coli* (Gram negative) (c) *P. Aeruginosa* (Gram-negative) (d) *C. Albicans* (Antifungal) and (e) *E. Faecalis* bacteria (Gram-positive).