Hydrothermal Synthesis of SnO_2, TiO_2 Nanomaterials, its Characterization and pH Sensing Application

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Abstract

In this research, Hydrothermal method was used to synthesize titanium dioxide (TiO_2) and tin dioxide (SnO_2) as this method is cost effective and have ability to produce highly crystallized material. This research focuses on how synthesis conditions influence structural properties of material and their compatibility for ph sensing application. Characterization technique like XRD, FESEM, UV visible spectroscopy used to determine their crystal structure surface topography, optical properties etc. Crystalline phases of both TiO_2 and SnO_2 were confirmed by XRD analysis. FESEM technique revealed surface morphology and topography as it finds synthesized material are spherical particles, although some degree of accumulation was present. Optical studies analysis done with the help of uv spectroscopy gives us band gap energies of a 3.1 eV for TiO_2 and 3.2 eV for SnO_2 respectively. The range of band gap energies reflects the semiconducting nature of nanomaterials. pH sensing mechanism of synthesized nanomaterials were tested and it is observed that when the pH level changed both TiO_2 and SnO_2 showed changes in their electrical behaviour, so we can say that they are ph sensitive materials. Impedance plot also gives us an idea that these nanomaterials respond well to different pH values. Overall TiO_2 and SnO_2 prepared by hydrothermal method are reliable, stable, low-cost materials for pH sensing.

Keywords: Titanium oxide, Tin dioxide, Hydrothermal synthesis, Structural properties Optical properties, pH sensing

Introduction

Nanotechnology is a branch of science that deals with materials having size scale between 1 and 100 nanometres. Materials at such small size show different properties compared to their normal form. We can observed improved strength, unique chemical, electrical and optical properties in such nanoscale materials. These advantages make a nanomaterial demanding asset in many fields such as sensing applications, electronics, medicine etc. Titanium dioxide (TiO_2) and tin dioxide (SnO_2) which are metal oxides nanomaterials are affordable, stable and easy to prepare. These materials are suitable for sensor application because they have better surface properties. Whenever pH changes these nanomaterials can detect the changes easily which is useful factor in many chemical and environmental processes. We use Hydrothermal method to prepare nanomaterials although there are many methods but producing nanomaterials with

Published: 21 May 2026
DOI: https://doi.org/10.70558/IJST.2026.v3.i2.241258

controlled size, shape, and good purity is provided by hydrothermal route easily. It is process where the chemical reaction takes place at high temperature and pressure within a sealed container called as autoclave. Energy requirement for the process of hydrothermal synthesis is also less as compared to other methods. Characterization techniques like XRD, UV visible spectroscopy, FESEM used for study and analysis of properties of TiO_2 and SnO_2 nanomaterials. Structural properties like crystalline quality, lattice parameters, crystalline size, atomic spacing are being studied by XRD. Surface morphology, topography is analysed by high resolution images of FESEM. After having characterization techniques result and analysis, research motive move towards pH sensing application. For the purpose of analysing the pH sensing mechanism, we used EIS technique and Nyquist plot. Food processing unit, industrial operation, water quality testing needs pH sensors. We examine how the use Nanomaterials like SnO_2 and TiO_2 affects the sensitivity and response time of sensors. Overall, this research paper emphasis on the synthesis of nanomaterials, its application and showing their importance in developing effective, low-cost pH sensors.

Review of literature

The hydrothermal synthesis of SnO_2 nanorods studied by Caixin Guo and co-workers. They used relatively low temperature to produce nanostructure and demonstrated that by changing reaction condition we can control the shape and size of SnO_2 . Giglioa Lusvardi and team reported that TiO_2 nanoparticles have good photocatalytic, chemical properties and have higher efficiency as they worked on the synthesis and characterization of TiO_2 nanoparticles. Masanori Hirano and his colleagues studied TiO_2–SnO_2 composite nanoparticles they reported that structural, functional properties and surface activity could be improved by combining these two materials. Ultimately, composite nanoparticle could be very beneficial for the sake of sensing application. Muhammad Imran Khan and co-authors stated that SnO_2 and TiO_2 which are metal oxide have quick response, good sensitivity as compared to other pH-sensitive materials. Yali Wang and others studied SnO_2 nanoflower prepared by hydrothermal synthesis they showed that this structure can improve surface properties, gives us a larger surface area which provides more interaction with the environment and makes them beneficial for sensing application.

Objectives

1. To synthesize TiO_2 and SnO_2 nanomaterials using the hydrothermal method.
2. To study the structural and morphological properties using XRD and FESEM techniques.
3. To analyse the optical properties of the prepared nanomaterials using UV–visible spectroscopy.
4. To evaluate the pH sensing performance of TiO_2 and SnO_2 nanomaterials.
5. To know the compatibility of these nanomaterials for practical sensing application.

Methodology

We have used hydrothermal synthesis for the preparation of nanomaterials as this method produced high purity, crystalline nanoparticles in low cost set up.

ISSN: 3049-1118, Volume- 3, Issue- 2 (April - June 2026)

Hydrothermal Synthesis of SnO_2

Solution (A) - 0.36g of $SnCl_4.5H_2O$ + 7ml DI water

↓

Solution (B)- Separate solution of 1g of NaOH in 140ml of water

↓

Add solution A into B & Stir till clear solution is observed

↓

Then add 0.16g of CTAB in above solution and under water bath (60°C)

↓

Add in Teflon -lined autoclave and heat to 180°C for 12 hours

Hydrothermal Synthesis of TiO_2

Add Isopropyl alcohol (1cc0ml) + Titanium iso-propoxide(3ml) in beaker

↓

Add Acetic Acid (9ml) + Methanol (25ml)

↓

Dry at 90°C for 24 hours

↓

Yellow powder is observed

Characterisation technique - The synthesized SnO_2 and TiO_2 were characterized by different characterization techniques such as XRD, FESEM, UV to reveal their crystal structure, surface morphology, energy bandgap.

Results and discussions

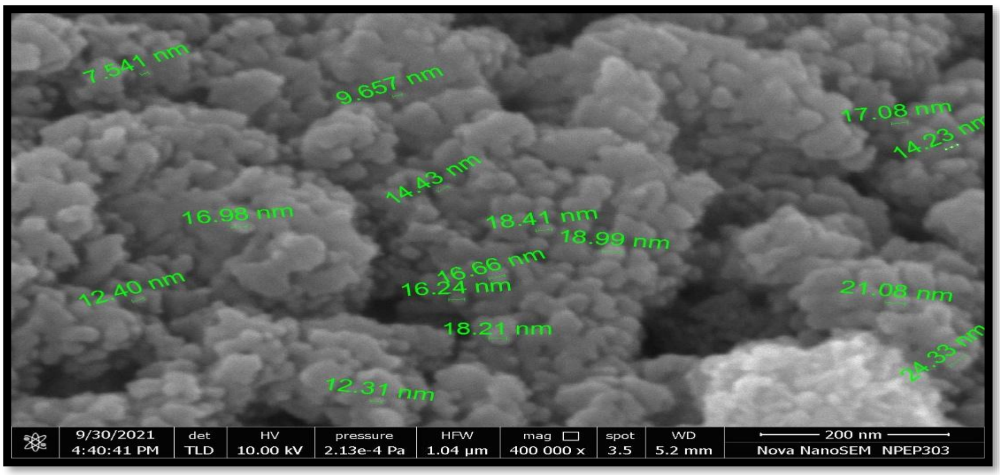


Figure1.0

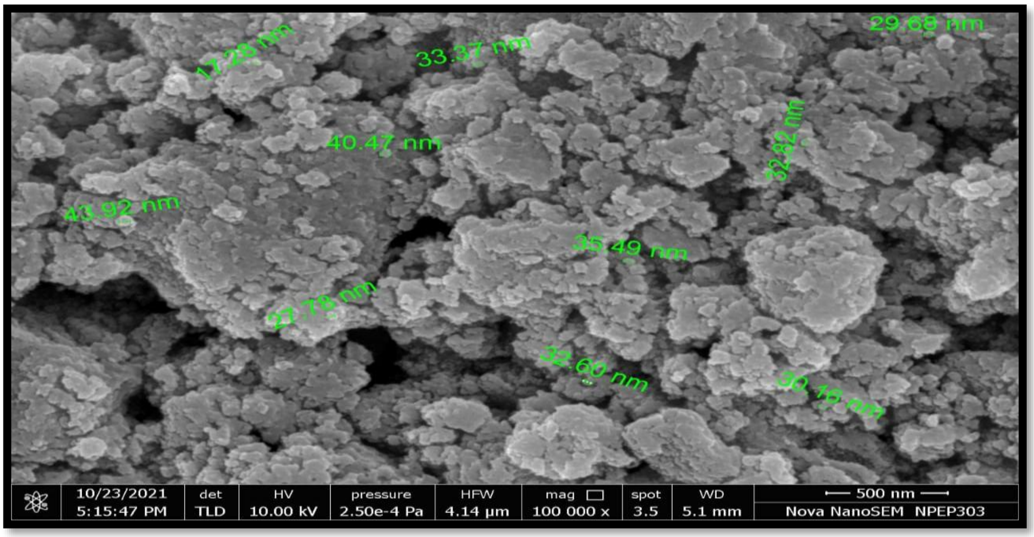
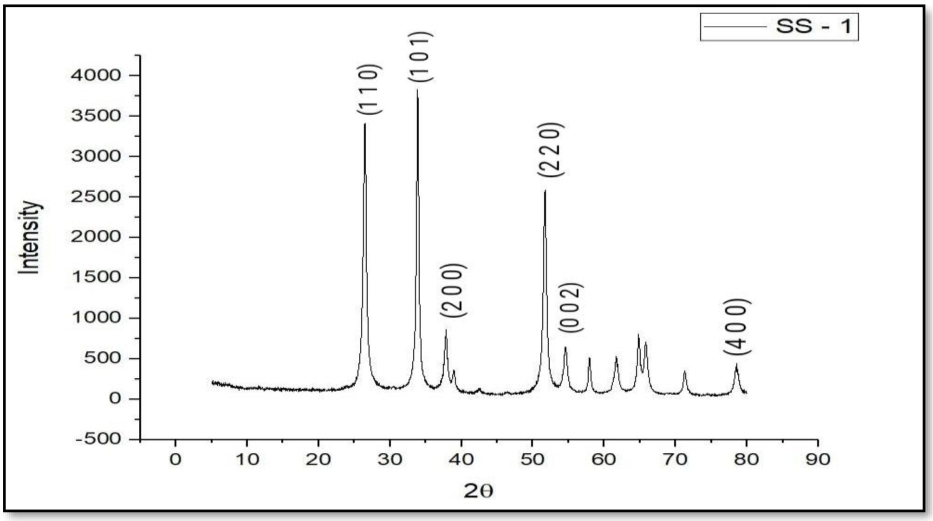


Figure2.0

The Figure 1.0 shows the FESEM image of TiO_2 with nano spherical morphology. Crystallites of TiO_2 are formed by synthesis of hydrothermal method with average size 16 nm.

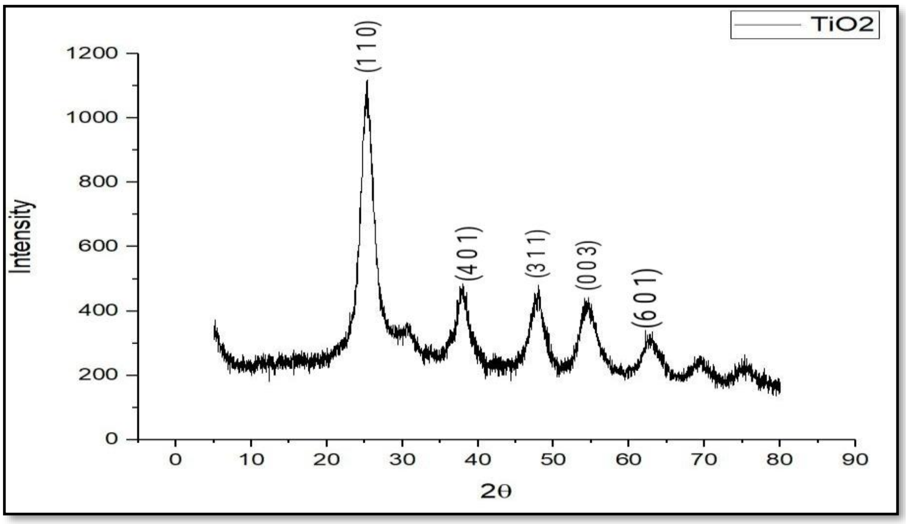
The Figure 2.0 represents the FESEM image of SnO_2 synthesized by hydrothermal method clustered to form micron size crystallites. The average size of 32.37nm of SnO_2 was successfully prepared.

XRD results



Graph 1.0 XRD Spectrum of stannic oxide (SnO_2)

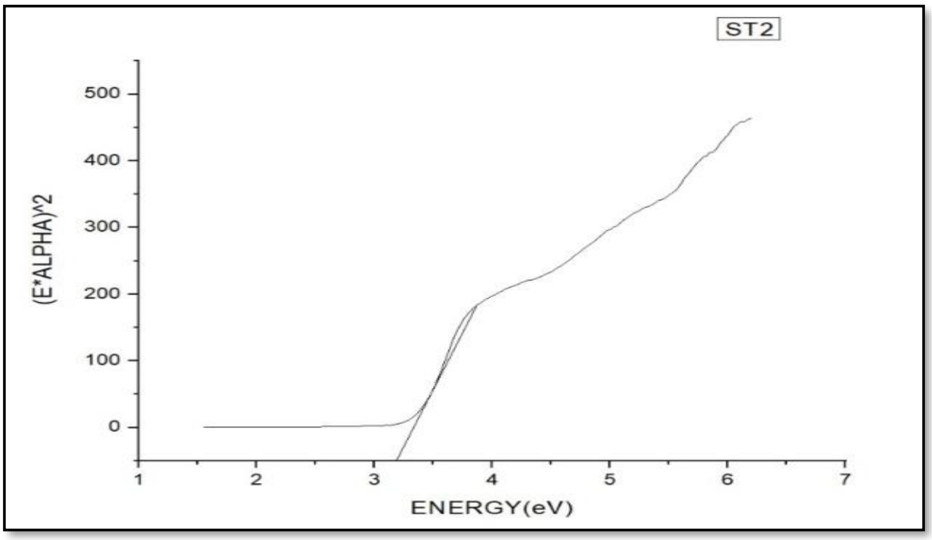
XRD was used to study the structural properties of SnO_2. Crystalline nature of SnO_2 was revealed by intense peak we got in spectrum of XRD. The XRD spectrum shows good intense peaks at 2Ø values (26.07°), (33.27°), (37.21°), (53.64°), (56.77°), (79.30°) following the planes (110), (101), (200), (220), (002), (400) respectively.



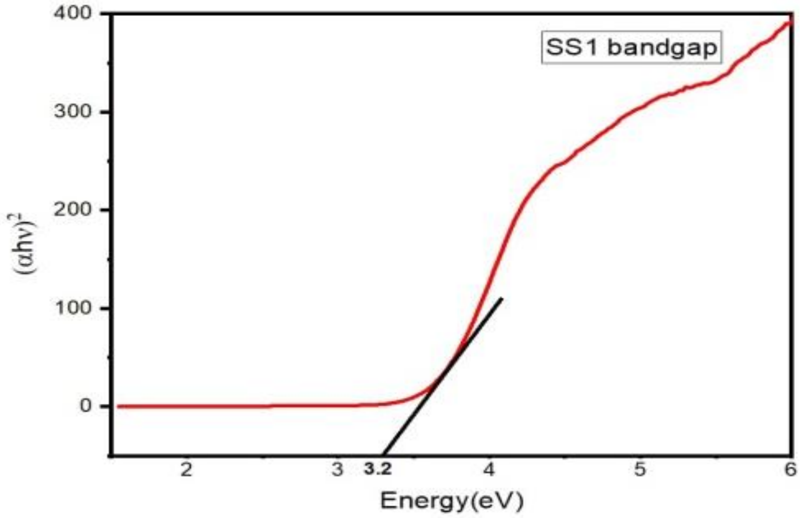
Graph 2.0 XRD spectrum of titanium dioxide

The structural properties of TiO_2 were studied with the help of XRD. We got to know about crystalline nature of TiO_2 by intense peak in the spectrum. The XRD spectrum shows intense peaks at 2Ø values (24.81°), (29.50°), (33.14°), (42.86°), (44.16°) corresponding the planes (110) (401) (311) (003) (601) respectively.

UV visible spectroscopy



Graph 3.0

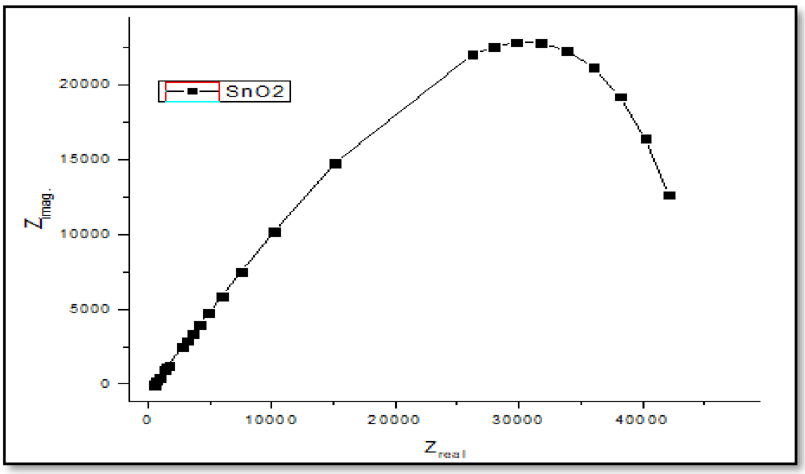


Graph 4.0

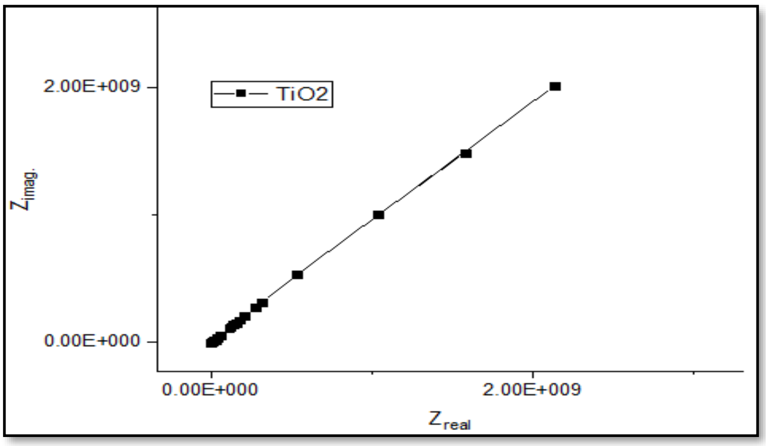
The Energy(eV) is plotted on X-axis and the Absorbance is plotted on Y-axis. We got Bandgap of 3.1eV and 3.2eV for nano spherical structure of TiO_2 and SnO_2 respectively. This band gap range shows material has semiconductor nature thus can have electron movement and it can be suitable for sensors.

pH Sensing Results

pH sensing instrument



Graph 5.0 Nyquist plot of SnO_2



Graph 6.0 Nyquist plot of TiO_2

The above graph shows the impedance expression is divided into a real part and an imaginary part. We got Nyquist plot by plotting Real part (Z_{real}) on the X- axis and the Imaginary part (Z_{imag}) on the Y-axis. The plot of SnO_2 shows a curved following by downward trend so we can conclude it shows both charge transfer and diffusion behaviour. It is Suitable for sensing applications as it responds to changes in pH. The plot of TiO_2 have straight line shows it have more resistance compared to SnO_2 but it shows faster diffusion so faster sensor response. We use EIS to study the sensing mechanism of our materials. This Nyquist plot show when a solution of particular pH comes in contact with material the changing charge transfer in the material takes place.

Application:

Healthcare monitoring, industrial applications have major use of pH sensors. Traditional glass pH electrodes are getting replaced by alternatives made by metal oxides materials because former have some limitation. Metal oxide-based electrochemical sensors like thick film sensors made by metal oxide are low-cost, easy to manufacture, suitable for large-scale production and have high performance. Materials like tin oxide (SnO_2) are widely used due to their high sensitivity to hydrogen ions, good conductivity, and chemical stability. Combining or doping materials, such as mixing SnO_2 with titanium dioxide (TiO_2) can improve sensors performances such as having longer life, better sensitivity, reduced interference from other ions and low fabrication cost etc. Test solutions with pH values ranging from 2 to 12 used to study sensing performance. A commercial glass pH electrode and conductivity meter were used to confirm the pH and conductivity of the solutions. The fabricated sensor was tested in distilled water, tap water, and lemon juice, and the results were compared with a standard glass pH electrode result. Investigation of effect of conductivity on sensor performance was tested by adding salt to distilled water. Metal oxide nanomateials also shows resistance to corrosion makes them suitable for harsh environment condition. Techniques like potentiometry, cyclic voltammetry, and electrochemical impedance spectroscopy (EIS) are used to analyse their behaviour and performance. Among these, to study the electrical properties of the sensor over a broad frequency range we used EIS method and with the help of result of Nyquist plot we can say that for modern pH sensing applications SnO_2–TiO_2 based sensors are promising and efficient alternative.

Conclusion:

The SnO_2 and TiO_2 nanomaterials were successfully formed by Hydrothermal Synthesis with average size of (32.37nm) and (16nm) respectively. Tauc plot obtained from UV visible spectroscopy got us the band gap of 3.1ev and 3.2 ev for TiO_2 and SnO_2 respectively. FESEM analysis shows that both materials have a spherical morphology. The electrochemical impedance analysis of these materials show they have good sensitivity towards hydrogen ion concentration. The SnO_2 and TiO_2 stands true for pH sensing process in real time.

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