

# SYNTHESIS AND CHARACTERIZATION OF DIFFERENT PHOTOCATALYTIC MATERIALS

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## Abstract

Access to clean water remains a major global challenge, affecting nearly 1 billion people. Photocatalytic materials, particularly semiconductors like TiO<sub>2</sub>, ZnO, CdS etc. offer promising solutions for wastewater treatment through light-induced redox reactions that degrade organic pollutants. Thin-film synthesis methods, such as sol-gel and spray pyrolysis, enable material optimization. Comprehensive characterization techniques including PEC, XRD, FESEM, EDX, XPS, and COD analysis are crucial for evaluating structural, chemical, and photocatalytic properties. These techniques facilitate the development of efficient photocatalysts, supporting environmental remediation and clean energy applications through enhanced light absorption, electron mobility, and surface reactivity.

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## Introduction

Today, nearly 1 billion people in the developing world don't have access to clean and potable water. One of the most persistent problems affecting the people throughout the world is inadequate access to clean water and its sanitation. Water is not only one of the most important resources for all human beings but also for all animals and plants present on the earth. All over the world one billion people per year are exposed to unsafe drinking water due to availability of poor source water quality and lack of adequate water treatment, although water covers nearly about 71% of the Earth's surface [1]. Due to these Photocatalytic materials attracts great attention for wastewater treatment, air purification, renewable energy production, Antibacterial coatings and self-cleaning surfaces etc. [2].

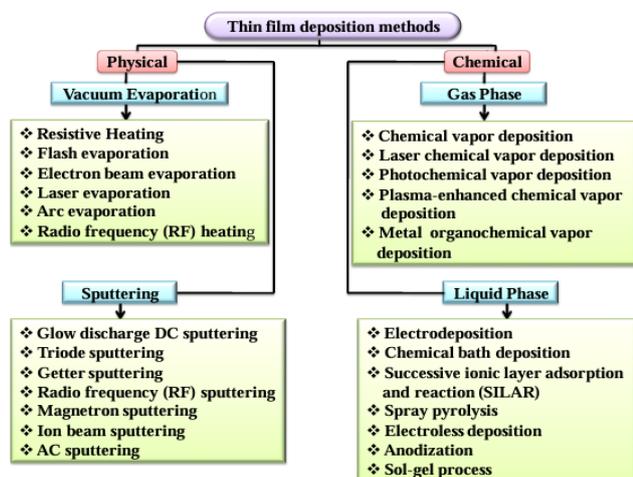
Photocatalysis is a process in which light energy, typically from UV or visible light, activates a semiconductor material that accelerates a chemical

reaction without being consumed in the process. A Photocatalytic material absorbs light (usually sunlight or UV light) and uses that energy to trigger or accelerate a chemical reaction. Incidence of light on the Photocatalytic material excites electrons from the valence band to the conduction band, creating electron-hole pairs [3]. These created charge carriers migrates to the surface of the material for conduction. During this process redox reaction occurs in which electrons can reduce oxygen to form superoxide radicals and holes can oxidize to split into the water molecules. Degradation of organic pollutants in water and air is the most studied Photocatalytic reaction. Titanium dioxide (TiO<sub>2</sub>), Zinc oxide (ZnO), graphitic carbon nitride, cadmium sulfide (CdS), and bismuth vanadate (BiVO<sub>4</sub>) are frequently studied Photocatalytic materials. These materials are known for their semiconducting properties and ability to generate electron-hole pairs upon light irradiation [4].



## Synthesis & Characterization of Photo catalytic Materials

The surface modification of these materials is important to improve the quality of material for this different characterization techniques are required to analyze the different physical, chemical and electrical properties of the materials. There has been significant interest in the preparation of good quality thin films where optimization of preparative parameters is of vital importance [5]. This chapter deals with the overview of thin film deposition techniques used to deposit or prepare different Photocatalytic materials using chemical a physical methods. Following figure gives the different techniques used for the preparation of Photocatalytic materials for various applications. Many catalysts, like ZnO, TiO<sub>2</sub>, ZrO<sub>2</sub>, WO<sub>3</sub>, SrO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub>, CeO<sub>2</sub>, CdS and ZnS have been attempted for the photocatalytic degradation of a wide variety of environmental contaminants [5]. Metal chalcogenides possess narrower band gap energy, which make them responsive to visible irradiation have be focussed to photocorrosion. The photocatalysts effectiveness for oxidation of organic compounds in water treatment is dependent on the oxidation potential of the valence band (VB) and the reduction potential of the conduction band (CB). The reduction potentials of TiO<sub>2</sub>, ZnO, ZrO<sub>3</sub>, SrTiO<sub>3</sub>, WO<sub>3</sub>, ZnS etc.could also be usedfor the photocatalytic oxidation of organic pollutants; however, it is often foundthat these are the most efficient semiconductor for the treatment of watercontaining organic pollutants and microorganisms [6].



The different characterization techniques such as photoelectrochemical (PEC) cell, X-ray diffraction (XRD), field emission, scanning electron microscope (FE-SEM), Atomic force microscopy (AFM), UV-Vis spectroscopy, Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), Photoluminescence spectroscopy (PL), Energy dispersive analysis by X- rays (EDAX), etc. have been employed for optimization of preparative parameters.

## Synthesis of Different Photocatalytic Materials

### 1. Synthesis of Titanium dioxide (TiO<sub>2</sub>) and doped TiO<sub>2</sub> thin films -

TiO<sub>2</sub> is a wide band gap, chemically stable and environmental eco-friendly semiconductor with good biocompatibility and stability. It exists in three phases, namely anatase (< 550 °C), rutile (>550 °C) and brookite (>1200 °C).TiO<sub>2</sub> film has the unique characteristics such as high optical transmittance over awide wavelength range and excellent adhesion to the substrates. TiO<sub>2</sub> is the most important material as a catalyst for PEC purification of wastewater because of its highoxidation potential in its valence band and high chemical stability [7].Titanium dioxide (TiO<sub>2</sub>) thin films can be synthesized using several chemical and physical deposition techniques such as sol–gel spin coating, dip coating, chemical vapor deposition (CVD), sputtering, or atomic layer deposition (ALD). Among these, the sol–gel spin coating method and spray pyrolysis techniques arethe simplest and most widely used. Doping of metal and non-metal in TiO<sub>2</sub> enhance the photocatalytic activity especially in the visible light by narrowing the band gap or reducing electron hole recombination. Doping can be done by using physical as well as chemical techniques depending on the application of TiO<sub>2</sub> thin films.Generally metals like Fe, Cu, Ag, Co are doped during the synthesis process in small concentrations typically 1-5mol%. Doping of non-metals like nitrogen (N), carbon (C) and sulphur (S) can be done by adding as precursor material during the deposition of TiO<sub>2</sub> [8].

### 2. Synthesis of Zinc oxide (ZnO) and doped ZnOthin films

Zinc Oxide (ZnO) is a wide band gap (3.37eV) semiconductor material with excellent photocatalytic

propertied under the incidence of Ultraviolet (UV) light. The physiochemical properties of synthesized thin films like, crystal structure, morphology, chemical and thermal stability depend on the synthesis condition. Many chemical and physical techniques are used for the synthesis of ZnO and doped ZnO thin films like radio frequency magnetron sputtering, hydrothermal method, sol-gel method and so on [9]. Depending on the application of ZnO and doped ZnO thin film in various fields, films can be synthesized and doped with metals and non-metals. Doping of pure ZnO thin films with metal or non-metal elements is an effective strategy to enhance its photocatalytic activity particular under visible and UV light. This can be done by narrowing the band gap and supressing charge carrier concentration [10]. Typically pure ZnO thin films doped with metals like Fe, Cu, Mn, Ag, Al by using various chemical and physical thin film deposition techniques. For the doping of these metals typically metal salts (concentration 1-5 mol% relative to Zn<sup>2+</sup>) are added to the solution during the preparation of precursor solution [11].

### 3. Synthesis of Cadmium Sulphide (CdS) and doped CdS thin films -

The synthesis of CdS (cadmium sulphide) and doped CdS thin films for photocatalysis applications is a highly specialized process that involves creating thin films of CdS or its doped variants to efficiently harness light energy for chemical reactions, often in water splitting, pollutant degradation, or carbon dioxide reduction applications. Due to suitable band gap (2.4eV) and good photocatalytic properties under visible light [12]. Several chemical and physical techniques are used for preparation of CdS and doped CdS thin films. Generally metal doping can create new energy states in the band gap, facilitate charge transfer, and improve the separation of electron-hole pairs, leading to better photocatalytic activity under visible light. Transition metals such as Cu, Ni, Fe, Co, and Ag are often used to dope CdS thin films. In case metal doped CdS thin films it shows enhancement in photocatalytic degradation of organic pollutants [13]. Non-Metal Doping such as Nitrogen (N), Sulfur (S), or Phosphorus (P) in CdS thin films can help to narrow the band gap of CdS, making it more efficient for visible light absorption. This type of doping improves the light absorption range and enhances photocatalytic efficiency under visible light.

### 4. Synthesis of Zirconium dioxide (ZrO<sub>2</sub>) and doped ZrO<sub>2</sub> thin films -

The synthesis of ZrO<sub>2</sub> (zirconium dioxide) and its doped variants for photocatalysis applications is a fascinating area of research, as these materials exhibit unique photocatalytic properties that are crucial for energy and environmental applications, such as water splitting, pollutant degradation, and CO<sub>2</sub> reduction. Doping ZrO<sub>2</sub> with various metal or non-metal elements can improve its photocatalytic efficiency by modifying its electronic structure, improving charge separation, and extending the absorption spectrum into the visible light region [14]. Several chemical and physical techniques are used for preparation of ZrO<sub>2</sub> and doped ZrO<sub>2</sub> thin films. Doping involves introducing various metal or non-metal elements into the ZrO<sub>2</sub> lattice to enhance its photocatalytic properties. Metal dopants Ti, Ce, Fe, Cu can reduce the bandgap of ZrO<sub>2</sub>, improve charge transfer, and create defect states that facilitate photocatalytic reactions [15]. Metal dopants in ZrO<sub>2</sub> thin films are typically introduced via co-precipitation, sol-gel, or hydrothermal methods. Non-metal dopants (especially nitrogen or carbon) can extend the absorption of ZrO<sub>2</sub> into the visible light region by creating impurity states within the bandgap. Nitrogen and carbon doping can be achieved by heat treatment in a nitrogen-rich or carbon-rich atmosphere [16].

## Characterization Techniques

### 1. Photoelectrochemical (PEC) characterization

Photoelectrochemical (PEC) technique is one of the useful technique for optimization of preparative parameters of photoactive semiconductor electrode, which is new, reliable and unique technique in thin film technology [6]. Fig.1 shows a schematic diagram of the PEC solar cell. A PEC cell consists of photoelectrode, proper electrolyte and counter electrodes, is illuminated with light consist of a suitable energy. For optimization of preparative parameters, the values of short circuit current (I<sub>sc</sub>) and open circuit voltage (V<sub>oc</sub>) are plotted against desired preparative parameter. The values of I<sub>sc</sub> and V<sub>oc</sub> go over the maximum for an optimized value of the desired parameter. It has been observed that, values of optimized preparative parameters obtained by PEC technique match well with the optimized values obtained by other techniques. The PEC

technique can be used for optimization of preparative parameters and to check the type of conductivity exhibited by semiconductor electrode thin films [17].

## 2. X-Ray diffraction (XRD) study

Phase identification and determination of crystal structure are important parameters while studying the thin films for various applications. X-ray diffraction is one of the techniques for determining crystal structure, lattice constant and so on. In this technique x-ray beam is incident on the material, then crystalline atoms diffracts this beam in particular directions. The pattern is produced by measuring the angle and intensity of diffracted beam. This technique was used to analyze different samples such as metals, semiconductors, various organic and biological samples and so on. The sample required for the analysis is in the powder or thin film form. Fig. shows the schematic of X-ray diffractometer used for the analysis.

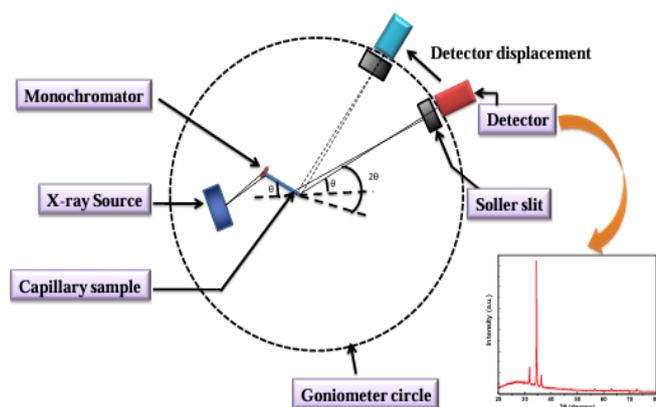


Fig 1: X-Ray Diffractometer

It consists of X-ray source for the production of x-rays. Different sources are used for the production of x-rays such as copper, tungsten and so on. The system consists of capillary or sample holder on which sample was mounted. Here monochromator is used to incident monochromatic x-ray beam and detector is used to observe and record the pattern. When the x-rays strikes on the sample, they obey Bragg's diffraction law [18].

## 3. Field emission scanning electron microscopy (FESEM) -

FESEM is one of the important techniques used for the morphological identification with magnification of 10x to 300,000x. FESEM is used to observe very

small topographical changes on the surface of the specimen sample. Different kinds of sample are characterized for using this technique such as powder, thin films, and biological samples etc. As compared with scanning electron microscopy (SEM), FESEM shows clear and less electrostatically distorted images. The working principle of FESEM is similar to that of SEM. Fig. shows the schematic representation of working of SEM. The main components of SEM systems are electron gun, electron column, detector, vacuum system and display. The vacuum is needed to accelerate the electrons without scattering and to avoid discharge inside the instrument. The free electrons are generated and accelerated to energies in the range 1-40 keV by using electron gun [19]. The main purpose of electron lenses is to incident small and focused beam on the specimen. Specimen stage is required to mount the sample with adjustment in three dimensions to obtain detailed over the active area of the sample.

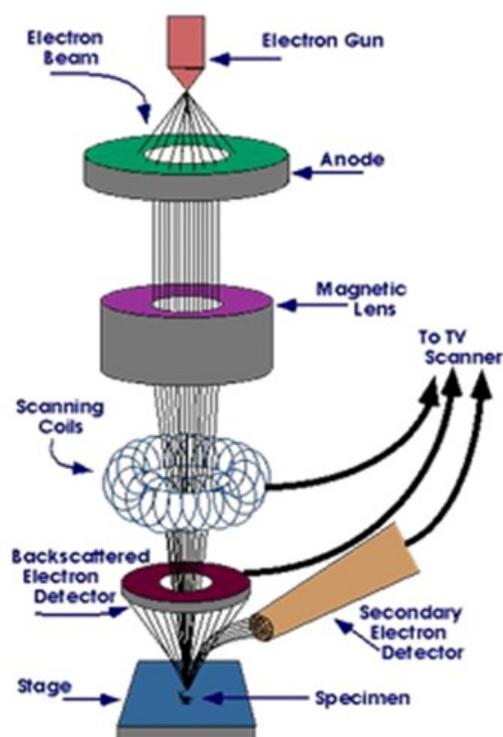


Fig 2: Schematic representation of scanning electron microscope

## 4. Energy dispersive X-ray analysis (EDX)

The EDX is one of the techniques used for the identification of the elements and their elemental composition of the sample. The EDX system is attached to the FESEM or TEM instrument. The characteristic EDX spectrum of particular sample

consists of corresponding peaks of elements, which take part in sample composition. In this technique an electrons are bombarded on the sample to analyze its elemental composition for analyzed volume. Then the x-rays are detected which are emitted from the sample surface. When the samples are bombarded by the electron beam, electrons are emitted from the atom comprising surface of sample. This electron vacancy is filled by electrons from higher state. To balance the energy difference between two electron states, x-rays are emitted [20]. This x-ray energy is the characteristics of the element from which it is emitted. All the elements from Be (4) and U (92) can be detected using this technique. EDX spectrum consists of various peaks resulting due to the electronic transition from different levels whose intensity is proportional to the density of that element. Elements having lower atomic number have less number of peaks in its EDX spectrum whereas element with higher atomic number have large number of peaks.

### 5. X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) is one of the most widely used surface characterization methods. It is also known as electron spectroscopy for chemical analysis (ESCA). It provides elemental analysis and electronic state of the elements. XPS spectra are attained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy (KE) and the number of electrons that escape from the top 1 to 10 nm of the material being analysed. Because the energy of a particular X-ray wavelength equals a known quantity, one can determine the electron binding energy (BE) of each emitted electrons. A typical XPS spectrum is a plot of a number of electrons detected as a function of their binding energy. Each element produces a characteristic set of XPS peaks at defined binding energy values; thus it is possible to detect each element on the surface or on the top layers of the material. These characteristic peaks correspond to the electron configuration of the electrons inside the atoms, e.g. 1s, 2s, 2p, 3s, etc. The number of electrons in each peak is related to the amount of the element within the irradiated area. The surface of the sample is placed in a vacuum environment and then irradiated with photons in the X-ray energy range [21].

### 6. Chemical Oxygen Demand (COD) analysis

Organic contaminants, including solvents, electrical insulators, lubricants, herbicides, and pesticides, can collect in aquatic environments and cause toxic effects on aquatic life and increase health risks of drinking water. These mentioned chemicals are at very low concentrations in the natural environment and they are typically introduced to surface waters as waste from human activities. If effluent with high COD levels is discharged into a stream or river, it will speed up bacterial growth in the river and consume the oxygen levels in the river. The oxygen may reduce to levels that are lethal to most fish and many aquatic insects present in the river. As the river re-aerates the water due to atmospheric involvement and as algal photosynthesis adds oxygen to the water, the oxygen levels will gradually increase downstream. Reduction in chemical oxygen demand (COD) is also studied of the same samples. The chloride interference is removed by using H<sub>2</sub>SO<sub>4</sub>; the mixture of diluted sample (before and after treatment) and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> is refluxed (acidic condition and Ag<sub>2</sub>SO<sub>4</sub> as catalyst) in a reflux flask equipped with condenser on a hot plate for 2 h. The refluxed sample is titrated against ferrous ammonium sulphate (FAS) as titrant. The NB medium is used as blank and similar condition was used for testing [22].

### Conclusion

Photocatalytic materials show great potential in addressing water pollution through advanced oxidation processes. Optimized synthesis and characterization significantly enhance photocatalytic efficiency. TiO<sub>2</sub>-based thin films are especially promising due to their stability and strong oxidation potential. Future research should focus on visible-light-active catalysts and scalable fabrication techniques.

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