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Analysis of PhotoElectro Catalytic Purification of Water

Bhishma Karki*, JeevanJyoti Nakarmi[1]
[1]Department of Physics,
Tribhuvan University, Kirtipur 44618,
Kathmandu, Nepal

Rhiddibir Singh[2]
[2]Department of Chemistry,
Tribhuvan University, Kirtipur 44618,
Kathmandu, Nepal

Amaresh Kumar Sahu [3]
[3] Sanjay Memorial Institute of Technology
,Bam, Odisha, India

***Abstract*— Advanced Oxidation Process is modern chemical method for the treatment of water containing non-biodegradable /toxic substances and to get decontaminated drinking water. Zinc acetate precursor is used to deposit zinc oxide thin films by Spray pyrolysis technique. Preperative parameters such as, substrate temperature, solution concentration and quantity of spraying solution were optimized to get good quality zinc oxide thin films. The prepared films were characterized by PEC, XRD, SEM, EDAX and UV-vis spectroscopy characterization techniques. Silver and gold deposited zinc oxide films were prepared to enhance photocatalytic inactivation of bacteria. The optimized stratified layer of TiO2 was given on zinc oxide thin films to improve its performance in photocatalytic process. The films prepared at optimized conditions of temperatures, concentrations are used in specially designed photoelectrochemical detoxification reactor. The prepared ZnO, Ag:ZnO , Au:ZnO and ZnO/TiO2 films are used to degrade Methylene blue (MB), textile effluent and inactivation bacteria such as E.coli, Bacillus, Salmonella, Staphylococcus etc.**

INTRODUCTION

Water is one of the world's most important resources for all human life, as with all animals and plant life on the planet. Civilization would be impossible without steady supply of fresh and pure water and it has been considered a plentiful natural resource because the sensitive hydrosphere covers about 71% of the Earth's surface. Its total water content is distributed among the main components of the atmosphere, biosphere, oceans and continents. However, 97% of the Earth's water is salty ocean water, which is unusable for most human activities. Much of the remaining 3% of the total global water resource, 2% is locked away in glaciers and icebergs and only 1% is thought to be easily accessible surface water located in biomass, rivers, lakes, soil moisture and distributed in the atmosphere as water vapor [1].

In the process of rapid development of science and technology, the demand for pure water is increasing to serve multifarious purposes in different types of industries. Human water consumption raised six fold in the past century, double the rate of population growth. In addition, the increasing population of the world is also escalating the requirements of pure water for drinking and household purposes. The high population density and the level of industrialization have triggered the hydrosphere to be polluted with inorganic and organic matter at an increasing rate. Moreover, to fulfillgrowing demand of food supply for increasing population of the world, a lot of pesticides, herbicides are used in agricultural purposes, which also cause the scarcity of clean water. Besides this, ground water has also been found contaminated in some parts of the world with some toxic metal ions both for natural and anthropogenic reasons. Therefore, specified quality of water for industrial use, household activities and drinking purpose is of great concern for the whole community of the world. More importantly, environmental regulation is also becoming stringent day by day to keep environment friendlier for human beings. To overcome the water pollution problems and to meet stringent environmental regulations, scientists and researchers have been focusing on the development of new and improvement of existing water purification processes. On the other hand, awareness has also been increased about water pollution all over the world and people have also started to realize that water is no longer an unlimited resource [2].

The photocatalysts effectiveness for oxidation of organic compounds in water treatment is dependent on the oxidation potential of the valance band (VB) and the reduction potential of the conduction band (CB). Fig. below shows the relative positions of the surface bands of the common semiconductors at pH 7 [5]. It can be seen that the CB of ZnO is sufficiently negative for the reduction of O_2, while the VB is sufficiently positive for the oxidation of $OH^{\bullet}$ making it an excellent semiconductor for oxidation of organic compounds in water.

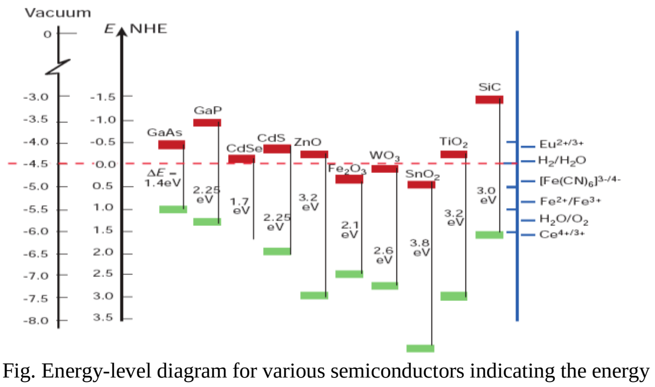

Fig. Energy-level diagram for various semiconductors indicating the energy positions of valence band and conduction band edges in aqueous media at pH 7.0.

I. PHOTOELECTROCHEMICAL PURIFICATION OF WATER USING OXIDE SEMICONDUCTORS

ZINC OXIDE (ZNO)

Zinc oxide is an n – type wide band gap (> 3 eV) semiconductor, which may be considered candidate for photocatalytic application, due to its high photosensitivity

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and non – toxicity [9]. ZnO nanoparticles have been used for photocatalytic degradation of pollutants and antibacterial treatment. Antibacterial agents are of relevance to a number of industrial sectors including environmental, food, synthetic textile, packaging, healthcare, medical, construction and decoration [10-11]. The antibacterial activity of ZnO powder and nanoparticles against various bacteria has been extensively investigated by Yang et.al [12].

Recently, bacterial activity of ZnOnanorod arrays fabricated by a hydrothermal method was also reported. Most applications of ZnOphotocatalysis have been concentrated on suspension of ZnO particles in a solution [21-28]. However, the use of ZnO catalytic slurries suffers limitations related to the filtering of particles. This requires either long time sedimentation or centrifugation process [29]. Thus there has been much interest in developing various supported ZnO nanostructured thin films for photocatalytic process. However, very few researchers have paid attention to use ZnOthin films as antimicrobial coatings. Xihong Li et.al [30] developed ZnO-coated poly (vinyl chloride) (PVC) packing film and tested its antibacterial property against the food pathogenic bacteria like *E. coli*, *Staphylococcus aureus*and fungi like *Aspergillusflavu*and *Penicilliumcitrinum.* The ZnO /PU (polyurethane) films and coats were fabricated by Li et.al [31] using solution casting and evaporation and tested their mechanical strength and antibacterial activity for *E.coli.* A layer of zinc oxide (ZnO) micro-grid was deposited on the surface of ZnO film using DC reactive magnetron sputtering and microsphere lithography on glass substrates and studied photocatalytic degradation experiments on methylene blue [32]. Textile effluent with diverse composition was effectively treated using hydrothermally synthesized ZnO [33].

II. CHARACTERIZATION TECHNIQUES

Photoelectrochemical (PEC) technique

PEC is a new, reliable and unique technique in thin film technology, which has proven to be the best tool for optimization of preparative and post preparative parameters of the semiconducting electrodes prepared by any deposition technique. A PEC cell consists of photoelectrode on conducting substrate, suitable electrolyte and counter electrode, is illuminated with light of an appropriate energy. For optimization of preparative parameters, the values of short circuit current (Isc) and open circuit voltage (Voc) are plotted against desired preparative parameter. The values of Isc and Voc go through 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 technique can be used for optimization of preparative parameters of any physical or chemical methods where PEC can be used also to check the type of conductivity exhibited by semiconductor electrode thin films [148]. In a spray pyrolysis technique, one can optimize preparative parameters such as substrate temperature concentration of solution and quantity of solution etc. by using PEC technique.

X-ray Diffraction (XRD)

The X-ray diffraction (XRD) is well-known technique for film samples. It is used to determine the crystal structure of materials. Asample is exposed to X-rays, which diffracts off the atomic planes of the crystalstructure. Constructive interference occurs when the atomic spacing is an integer multiple of the X-ray wavelength, resulting in a characteristic diffraction peak. By moving the detector and source across a range of different angles with respect to the sample, a diffraction pattern can be obtained. This diffraction pattern is characteristic of a material and the crystalline phases that are present in the sample identification and determination of lattice parameters are based on the interpretation of X-ray diffraction patterns.

The phenomenon of X-ray diffraction consists of reflection of X-rays from the different crystallographic planes of material and Bragg's law governs it,

$$2d_{hkl}Sinq = nl$$

where, *dhkl*= lattice spacing for (*hkl*) plane, l = wavelength of monochromatic X-rays, *n* = order of diffraction (*n* =1) In case of a polycrystalline sample having large number of grainsize is determined from Scherer's formula

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

where, *l* = wavelength of X- rays, *q* = Bragg's angle and *b* = full width at half maxima (FWHM) (in radian) of the peak intensity

III. THICKNESS MEASUREMENT BY INTERFEROMETRIC METHOD:

The determination of film thickness is of critical importance for the quantitative evaluation of certain parameters. The film thickness can be measured using several methods like multiple beam interferometry, film thicknes probe coupled with optical microscope, interference fringe patterns, Talystepand optical Nano scope, surface profiler, weight difference method, etc.

Interferometric method:

Large band gap oxide thin films show a pronounced interference maxima and minima in the spectral transmission. This is due to the interference between the waves reflected from film surface and the substrate surface. The interference peaks can be used as a tool to determine the film thickness. The oscillations after the edge are caused by the electron, which can be viewed as a wave, being scattered by the neighboring atoms causing constructive and destructive interference between the propagated and reflected waves. Fitting the observed transmittance data with the calculated one given
by Eq. (1.6), the thickness of the film can be estimated, [154]

$$T = \frac{t_1^2 t_2^2}{(1+2r_1r_2\cos 2\delta_1 + r_1^2 r_2^2)} \times \frac{n_2}{n_0}$$

Where

$$r_1 = \frac{n_0 - n_1}{n_0 + n_1}, r_2 = \frac{n_1 - n_2}{n_1 + n_2},$$

$$t_1 = \frac{2n_0}{n_0+n_1}, t_2 = \frac{2n_1}{n_1+n_2}, \text{and} \quad \delta = \frac{2\pi n_1 d}{\lambda},$$

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The available fresh water is not always clean as it may contain natural toxic impurities or be contaminated by human activities. Hence appropriate water treatment systems have to be provided urgently to save lives. Advanced Oxidation Process (AOP) is modern chemical method for the treatment of water containing non- biodegradable toxic substances andto decontaminated drinking water. With classical water treatment techniques such as flocculation, precipitation, adsorption on granular activated carbon, air stripping, reverse osmosis or combustion, pollutants are only transferred from one phase to another. Whereas AOPs degrade the organic pollutants in water with byproducts carbon dioxide and water. For disinfection of water AOPs are an alternative to conventional chlorination process, which leads to generate of toxic chlorinated byproducts in the presence of natural organic matter.

Among AOPs, heterogeneous photocatalysis seems to be an attractive method as it has been successfully employed for the degradation of various organic pollutants, including dyes and microorganisms. Heterogeneous photocatalysisto decontaminated drinking water. With classical water treatment

techniques such as flocculation, precipitation, adsorption on granular activated carbon, air stripping, reverse osmosis or combustion, pollutants are only transferred from one phase to another. Whereas AOPs degrade the organic pollutants in water with byproducts carbon dioxide and water. For disinfection of water AOPs are an alternative to conventional chlorination process, which leads to generate of toxic chlorinated byproducts in the presence of natural organic matter.

Among AOPs, heterogeneous photocatalysis seems to be an attractive method as it has been successfully employed for the degradation of various organic pollutants, including dyes and microorganisms. Heterogeneous photocatalysisis a process in which a combination of photochemistry and catalysis is operable and implies that light and catalyst are necessary to bring out a chemical reaction. The reason for the increased interest for the photocatalytic method is that the process use atmospheric oxygen as the oxidant and it can be carried out under ambient conditions. Among many oxide semiconductors, zinc oxide appears as a very promising photocatalyst for degradation of organic solutes in aqueous systems. Zinc oxide can be prepared by variety of techniques such as hydrothermal, sol–gel, RF magnetron sputtering, chemical vapor deposition, thermal vaporization, laser ablation,by E-beam evaporation, ion beam assisted deposition and spray pyrolysis [166-174]. Among these spray pyrolysis technique (SPT) is easy and economic, to deposit zinc oxide thin films. Zinc acetate precursor is used to deposit zinc oxide thin films by SPT. Preparative parameters such as, substrate temperature, solution concentration and quantity of spraying solution will be optimized to get good quality zinc oxide thin films. The prepared films will be characterized by PEC, XRD, SEM, EDAX and UV-vis spectroscopy characterization techniques.

Silver and gold deposited zinc oxide films will be prepared to enhance photocatalytic inactivation of bacteria. The optimized stratified layer of TiO2 will be given on zinc oxide thin films to improve its performance in photocatalytic process. The films prepared at optimized conditions of temperatures, concentrations will be used in specially designed photoelectrochemical detoxification reactor. This rector requires films of the size (10 X 10 X 1.5 cm^2). The prepared ZnO, Ag:ZnO , Au:ZnO and ZnO/TiO2 films will be used to degrade Methylene blue (BM), textile effluent and inactivation bacteria such as *E.coli*, *Bacillus*, *Salmonella*, *Staphylococcus* etc.

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