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DEPARTMENT OF PHYSICS & RESEARCH CENTRE NATIONAL SEMINAR ON FUNCTIONAL MATERIALS AND ITS APPLICATIONS



This is to certify that Mr./Ms./Dr./Prof. Dr. J.R. Sheeba
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Tin Oxide Nano Particles added with Ocimum Sanctum leaf extract for Instant Degradation of Methylene Blue Dye

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Abstract

The synthesis of Tin oxide nano particles were carried out by co-precipitation method with EDTA as surfactant. The Powder XRD analysis confirms the tetragonal (rutile) structure. The size of the nano particles were confirmed using XRD and SEM. EDAX confirms the presence of SnO₂. The tin oxide nano particles added with Ocimum Sanctum shows instant degradation of Methylene blue dye.

Introduction

In the recent past synthetic organic dyes were widely used in various industries and factories like textiles, leathers, cosmetics, paints etc. During the dyeing process, the left over residues of these dyes enter into the environment, especially water streams. The waste water from the industries contaminates the fresh water resources. Since these dyes are colouring agents, when they are passed into the water bodies, they trap the sunlight, thus affecting the aquatic eco system. This results in decrease in dissolved oxygen content in the water bodies [1]. Therefore the removal of these dye contaminants in the waste water is very much essential before discharging it into the water bodies to prevent health hazards. Semiconductor metal oxides are widely used in these photo catalysis applications in the waste water treatment [2]. Among the various semiconducting metal oxides, SnO₂ is a promising element considered for photo catalytic area which can be controlled by the growth parameters [3]. In the present work, co-precipitation method is used to synthesis the nano crystalline tin oxide, because it is simple, inexpensive, does not require high temperature and pressure and impure materials are eliminated by filtration and washing. Leaf extracts of ocimum sanctum were added with the synthesized tin oxide nano materials and used for photocatalytic degradation of methylene blue dyes.

Experimental Procedure

Preparation of tin oxide nano particles

Analytical grade reagents were used for the synthesis of the sample. SnO₂ samples were prepared using Tin chloride (IV) Penta hydrate (SnCl₄.5H₂O, Sigma Aldrich), with 1M as a precursor and was slowly added into 50 ml of de-ionized water at room temperature under constant stirring to produce a transparent colloidal solution. EDTA was used as surfactant. Aqueous NaOH was added drop wise to the resulting solution in order to modulate the pH at 3 uniformly throughout the reaction. The colloidal solution was then left for constant stirring for about 3hours. The temperature was maintained at 60 °C. A white precipitate was formed and collected and was filtered using Whatman filter paper and washed with de-ionized water several times until the effluent pH was neutral and then with ethyl alcohol. After drying at 170°C in hot air oven for 2 hours, the SnO₂ nano particles was obtained.

Preparation of ocimum sanctum leaf extract

Fresh leaves of ocimum sanctum were harvested and washed well with distilled water. The leaves were ground well into a fine paste with the help of mortar and pestle. The paste was crushed well and the extract is filtered. The filtrate was dried in hot air oven at 70°C for 1 hour. The prepared tin oxide samples were added with ocimum sanctum leaf extract.

Characterization techniques
XRD Analysis

The determination of crystallite size and phase were carried out using X'Pert Pro X-ray diffractometer. XRD data were collected in the 2theta range of 20°–80° and the diffraction pattern of SnO₂ is shown in figure 1. The diffraction peaks are in harmony with JCPDS card no: 41-1445 and it belongs to tetragonal rutile structure [4]. The major peaks appear at 26.98°, 34.92°, 51.88° and 65.08°. The cell parameters, a = 4.81974Å and c = 3.06311Å and cell volume V = 71.1557 Å³ were calculated using the formula

$1/d_{hkl}^2 = (h^2+k^2)/a^2 + l^2/c^2$ where h,k,l miller indices and d_{hkl} is the distance between two consecutive planes (m=1) with plane index (hkl) and the unit cell volume of the Tetragonal system is, V = a²c. The crystalline size, D, was estimated from the peak width with the Scherrer's formula:

$$D = (K\lambda) / (\beta \cos\theta_B)$$

Where, λ is X-ray wavelength, β is the full width half maximum (FWHM) of a diffraction peak θ_B is the diffraction angle and K is the Scherrer's constant of the order of units [5]. The average crystalline size of tin oxide nano particles using Scherrer's formula was 6.365 nm.

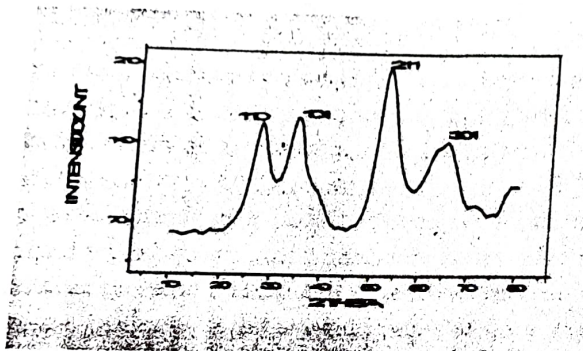


Figure :1 XRD pattern of SnO₂ nano particles

SEM and EDAX Analysis

Pure tin oxides are synthesized by co-precipitation method and the morphology and microscopic structure are characterized by using Scanning Electron Microscope(SEM) as shown in figure(2). In the present study, the synthesized tin oxide is stabilised in tetragonal phase. When the crystallite size exceeds certain size, transformation of tetragonal to monoclinic can be observed. But no such phase transformation could be observed. The SEM analysis depicts the tetragonal arrangement of atoms.

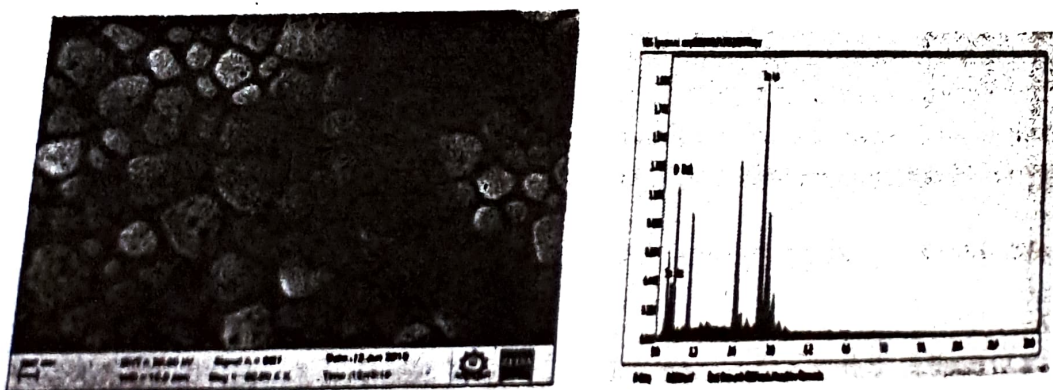


Figure: 2 SEM and EDAX of SnO₂ nano particles

The EDAX spectrum confirms the presence of Sn and O elements of the synthesised compound which is shown in the figure (2). The EDAX spectrum also confirms the absence of impurities that arise from the precursor and during the synthesis process. The atomic percentages of Sn and O elements present in the as-prepared powder are 74 and 26 mass.%, respectively, which indicates that the synthesised SnO_2 powder is very close to the stoichiometry.

Photodegradation of methylene blue dye

0.1 ml of methylene blue solution was added in 100ml of distilled water. Now 0.5 gm of tin oxide was mixed with Ocimum Sanctum leaf extract and added to the above solution. It was noticed that the solution decolorized instantly. The pH of methylene blue solution was 6. The pH of tin oxide mixed with Ocimum Sanctum leaf extract was 7. The colour of dispersion disappears indicating that the chromophoric structure of the dye was destroyed. The photo catalytic activity of pure SnO_2 and SnO_2 added with ocimum sanctum leaf extract was examined by the changes in the absorption spectra of methylene blue dye solution during their photo degradation process. Fig (3) compares the absorption spectra of methylene blue dye, pure SnO_2 added with methylene blue dye and SnO_2 mixed with ocimum sanctum leaf extract added with methylene blue dye.

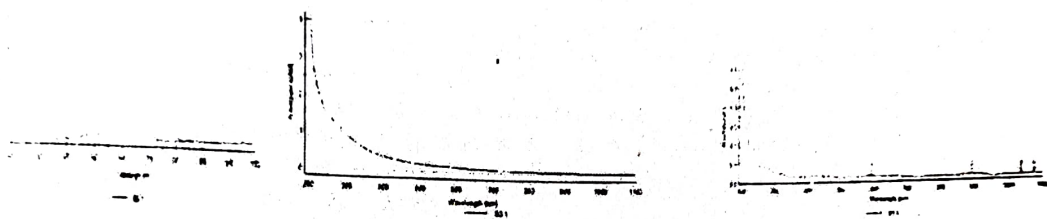


Figure: 3 Comparison of absorption spectra

It was noticed that the SnO_2 mixed with ocimum sanctum leaf extract added with methylene blue dye solution showed the better degradation efficiency.

Conclusion

In the present work nano crystalline tin oxide was synthesized using inexpensive co-precipitation method. The synthesized samples were annealed using hot air oven. The annealed samples were characterized using XRD, SEM and EDAX. From the XRD analysis the crystalline size of tin oxide nano particles was calculated using Scherrer's formula and was 6.365 nm. The SEM analysis confirmed the tetragonal arrangement of atoms. The presence of Sn and O elements of the synthesized compound was confirmed using EDAX spectrum. Photo catalytic degradation of methylene blue dye was noticed instantly when SnO_2 mixed with ocimum sanctum leaf extract added with methylene blue dye solution. The absorption spectra of methylene blue dye, pure SnO_2 added with methylene blue dye and SnO_2 mixed with ocimum sanctum leaf extract added with methylene blue dye was examined and found that SnO_2 mixed with ocimum sanctum leaf extract added with methylene blue dye solution gave the best degradation efficiency.

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