International Journal of Current Science Research
www.drbgrpublications.in

Volume: 5; Issue: 8; August-2019; pp 1782-1792. ISSN: 2454-5422

Synthesis and Characterization of SnO_2 Nanoparticles by Electrolysis

Mamata Baliarsingh* [1] and Dr. Padmaja Pattnaik[2]

[1]M.Phil Scholar, Department of Physics, School of Applied Science, Centurion University of Technology and Management, Bhubaneswar, Odisha, India

[2]Head, Department of Physics, School of Applied Science, Centurion University of Technology and Management, Bhubaneswar, Odisha, India

*Corresponding author email: mamatabaliarsingh3@gmail.com

Abstract

The main objective of this work is to synthesize SnO_2 nanoparticles by electrolysis. The experiment is conducted taking dil.HCl of different concentrations and two electrodes [Tin (Sn)] are used for electrolysis .The effect of electrolyte and potentials is studied. The particles obtained after electrolysis are characterized using XRD.The structure of SnO_2 by diffractogram is Tetragonal which is in agreement with standard diffraction pattern. From UV-Visible spectrophotometer, the absorption peak of SnO_2 is about 207 nm. The product has the size distribution in a range of 25-150 nm.

Keywords: Tin oxide, SnO_2 nanoparticles, diffractogram, X-ray diffractometer, Fourier Transform Infrared.

Introduction

Today material nanoparticles are used in commercial coatings that absorbs at particular wavelengths. Recently, the optical absorption of holder metal nano particles has been employed as the basis for novel sensors, the enhance local fields close to particle surface makes it possible to detect single molecules by surface enhanced spectroscopy.While designing optical coating, it is vital to have accurate model of their optical properties. The optical properties of nanoparticles are important for both traditional and emerging technologies. Nanoparticles have long been used as colouring agent is glass and points. Research in nanoparticle optics soarced in 1970s due to increased societal interest in solar energy application. Nanoparticles based coating can be optimized for specific application by modifying their particles-size, shape, volume fraction, micro structure etc. Because the optics of nanoparticles is a complex subject, different classes of models are needed in different situations. We have developed a comprehensive set of working models for sub micron particles embedded in supporting media. We also carried out experimental studies on metal insulator composite solar absorbing coatings, nano structuring transparent conductors, pigmented polymers and parent coatings. The experiments of results are close up related to the prediction of the appropriate.

Theoretical models

Nano is a prefix that is used as a microscopic unit to change its value by orders of magnitude. Nano means one billionth (10^{-9}). Nanoparticles are defined as the smallest particle ever found as the result of molecular processing and nano technology. These are the particles with extra ordinary properties of deliberating new components and transmitting energies at nano meter scale. The small size of nano particles is responsible for their unusual properties. Nano materials are important due to their large surface area ratio compared to bulk materials and fundamental size effects.

SnO_2 has been applied as a catalyst, gas sensor-battery. These applications are used in the form of nano particles i.e nano sheets, nano rod clusters, nano rod array, nano rod bundles, nano spheres which were prepared by different methods.

Several scientists reported that the Sno_2 nano particles could be synthesized using wet chemical method. Those method are biogenic, synthesis, gel combustion, hydrothermal route, sol gel, micro emulsion, solvo-thermal, thermal decomposition, so no chemical, precipitation. All methods were proved to produce SnO_2 nanoparticles. Unfortunately, these methods are time-consuming and complicated. They are not suitable for fabrication cost and facile preparation process. Another method is electro chemical method which it is proposed in this work. The several advantages of this procedure include control of particles size, excelled yields, operational simplicity and minimum environmental. In sonication on nickel hydroxide nanoparticles and influence CTAB It has been applied. Synthesis, synthesis nickel hydroxide by electrolysis at high voltage and influence of $Ni(OH)_2$ nanoparticles on insulin sensor sensitivity. Based on the explanation, most of researchers produced SnO_2 nano particles using techniques which require Tin (IV) chloride as a reagent. This reagent is relatively expensive and difficult to be found in Indonesia. There have been limited studies concerned an synthesis of Sno_2 from tin bare which is cheaper and easier to be found in Indonesia. Therefore, this research intent to produce a nano size SnO_2 using Tin bear as regent by high potential electrolysis.

Nano Materials

Nano crystalline materials are the solids composed of nano meter sized particles, or grains or crystallites. Recent progress in nano material research is regarded as one of the most advanced as one of the most advanced and interdisciplinary area of research in physics, chemistry, bioscience and engineering (Edelstein and Commeratra, 1998). The application of nano materials permits the alteration of fundamental physical and chemical properties of conventional materials as their size is reduced to the nano scale, offering new materials with unique electrical, optical and mechanical properties.

Nano materials are used for commercial purpose such as absorbents, fillers, catalysts, semiconductors, photo catalysts, cosmetics, micro-electronics, pharmaceuticals, drug carriers, energy storage and antifriction coatings. The unique properties of nano materials which arise due to domination of surface effects over interior volume effect. Due to their nano dimension, can be sued to design novel technologies or to improve the performance of existing processes. Nano materials have found multi applications in water treatment energy production and contaminant sensing. Metal oxide nano particles are attractive from both scientific and technological point of view. Metal oxide nano materials can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites. Metal oxides nano materials have attracted a great deal of attention because of their large application in various files like catalysis, sensors, and environmental remediation like absorption and degradation of various contaminants from aqueous media.

Nano rods materials

1D nano materials including nano tubes, nano wires, nano fibers and nano rods have attracted a great deal of attention due to their combination of superior proportion like small dimension structure, high aspect ratio and unique device function that lead to a large range of promising applications in electronics, photonics. Chemicals sensor, field emission devices, solar cell, Lithium ion-battery, hydrogen storage and drug delivery.

Nano composite materials

A composite is considered to be any multiphase material that exhibits a significant proportion of the properties of all constituents' phases such that a better combination of Properties is realized. According to the principle of combined action, better property combinations are fashioned by the judicious combination of two or more distinct materials. Nano composites are composites in which at least one of the phases shows dimensions in the nano meter range. Nano composite materials have emerged as suitable alternatives to overcome limitations of micro composites and monolithic while posing preparation challenges related to the control of elemental composition and stoichiometry in nano cluster phases.

Properties of Nanoparticles

Nanoparticles often have different properties than the bulk material due to high surface area which results in high reactivity and quantum effects. It alters the electrical and visual properties due to confinement of electrons which changes the wavelength observed as well as the conduction band gap. Nano particles take several forms including emulsion, aerosol or a solid suspension in liquid. The nanoparticle also acts arts with other nano particles in unique ways depending on their particular form. They can elect magnetically attract or repel each other or be governed by weak vander Walls forces. Nano materials are important due to reason that their increased surface to area ratio compared to bulk materials and others is due to more fundamental size effects. As the size of a material is decreased below 100nm, if becomes similar to many physical phenomenon such as the mean free path of electrons, photons, the wave length of light and energy transfer distances. The surfaces play a very significant role in the properties of nano materials. The other factor is the relationship between the electronic properties of the material and the critical dimension of particle.

Physical properties of Nanoparticle

Nano particles are unique because of their large surface area and this dominates the contribution made by the small bulk of the materials.

Optical properties: - Nanoparticles pass unexpected optical properties as they are small enough to confine their electrons, and produce quantum effect. One example of this is that gold nano particles appear deep red to black in solution. Distribution of non-agglomerated nano particles in a polymer are used to time the index of refraction. Additionally, such a process may produce materials with non-linear optical properties. Semiconducting nano particles and some oxide polymer nano composite exhibit fluorescence showing blue shift with decreasing particle size.

Electronic properties: - As the size of the nano particles deceases to their Bohr radius all the electronic properties begin to change. The electronic properties of nano particles depend on size and their shape.

Mechanical properties: - The large amount of grain boundaries in bulk materials made of nano particles allows extended grain boundary sliding leading to high plasticity.

Catalytic properties: - Due to their large surface, nano particles of transition element oxide exhibit interesting catalytic properties. In some cases, catalysis may be enhanced by changing composition or other parameters.

Magnetic properties: - In magnetic nano particles the energy of magnetic aristocracy may be small that the vector of magnetization fluctuates thermally and this is called super paramagnetism. Such materials are free at romance and correctively.

Diffusion properties: - Diffusion properties of nanoparticles at elevated temperative, nanoparticles posses the property of diffusion.

Materials and Methods

The most challenging part of resort in the field of nano science and nano technology is the cost effective and environmentally safe procedure for synthesizing desired nano materials. The properties of nanoparticles depend largely on their synthesis procedures. Several physical chemical and biological synthesis method have been developed to improve the properties of nano particles. Synthesis of nano particles to have a better control over particle size, distribution morphology, quantity, quality and purity by employing environment friendly process has always been a challenge for the researchers.

The two basic strategies used to produce nano particles “top down” and “botton up”. The term “top down” refers to the mechanical crushing of source materials using a milling process. In the “botton up” strategy structures are built up by chemical processes.

Top down approach

In top down approach nano objects and materials are created by larger entities without bouncing its atomic reactions usually top down approach are practiced less as compared to the bottom up approach

Bottom up approach

In the bottom up approach different materials are constructed from molecular components of their own which are not required to assemble by an external source. They chemically assemble themselves themselves by recognizing the molecules of their own breed. Two or more components can be combinend to form a new one or to be used as whole. Some of the examples of molecular self assembly are Watson Crick base pairing and enzyme substrate. The above method is time taking.Therefore the best method for synthesis of SnO_2 nanoparticles is High Potential electrolysis.

Electrolysis method

Materials:

To conduct the experiment, tin metal sheet which is pure (99.9%) is purchased from market. The thickness of the sheet is 1mm and dimension is adjusted into 4mm*10c.m.The two sheets of equal size are used as cathode and anode. For chemical preparation and cleaning, demineralized water is used.

Method:

The SnO_2 nanoparticles were synthesized by electrolysis with a tin sheet as anode and cathode. The electrolysis is performed at various potential in 100 ML 0.02 M HCL as electrolyte solution to see the influence of the potential applied. Various concentration of electrolyte solution is also applied to observe the formation of SnO_2 nanoparticles during electrolytes. The concentrations are 0.005, 0.01, 0.02, 0.03, 0.04, 0.05 & 0.06, HCL at fixed potential.

Continuous string is also applied during all the electrolysis experiment. Electrolysis was stopped after 15 minutes and both the electrodes are released from the electrolysis cell dried at room temperature and weighed to observe the weight different of both the electrodes before and after electrolysis. The solution obtained is cooled at room temperature and ready for characterization. The plasma band peak of SnO_2 nanoparticles solutions is observed using GENESYS IOS UV-Vis spectrophotometer. The nanoparticles structure was analyzed by XRD. Malvern zetasizer nano series instruments were used to measure the particle size obtained.

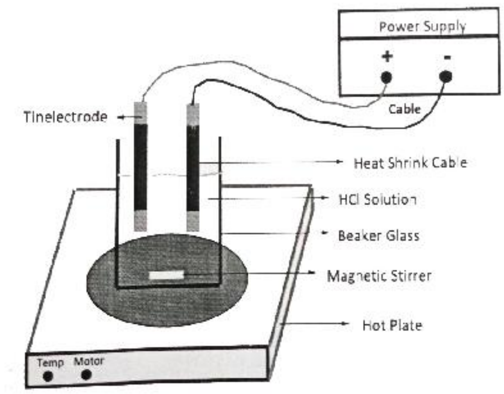

Figure: 1 Electrolysis cell of SnO_2

Result and Discussion

Weight of cathode =1.83 gm

Weight of anode =1.128 gm

Size of cathode and anode =4mm*10c.m

The reaction on cathode occurs as

Cathode (Sn):

$$2H_2O_{(l)} + 2e^- \rightarrow 2OH^-_{(aq)} + H_{2(g)} \quad \ldots(1)$$

H_2 production at the cathode can be observed by bubbled formation during electrolysis process. At that time the reaction on the anode have three possibilities as described in Eqns. (2), (3), or (4);

Anode (Sn):

$$Sn_{(s)} \rightarrow Sn^{2+}_{(aq)} + 2e \quad \ldots(2)$$

$$Sn_{(s)} \rightarrow Sn^{4}_{(aq)} + 4e^- \quad \ldots(3)$$

$$2H_2O_{(1)} \rightarrow 4H^+_{(aq)} + O_{2(g)} + 4e^- \quad \ldots(4)$$

The oxidation of Sn was observed by the lost weight of the Sn.The Sn can be oxidized to be Sn^{2+} or $Sn^{4+.}$ Formation of O_2 as Eqn. (4) was also observed by the bubbles produced at the surface of the anode.

XRD Analysis:

The nanoparticles were obtained characterized by using XRD. All diffraction peaks are in good agreement with the SnO_2 standard diffraction pattern. The results indicate the product obtained in SnO_2 nanoparticles with a tetragonal structure. The product was analyzed by zeta sizer.

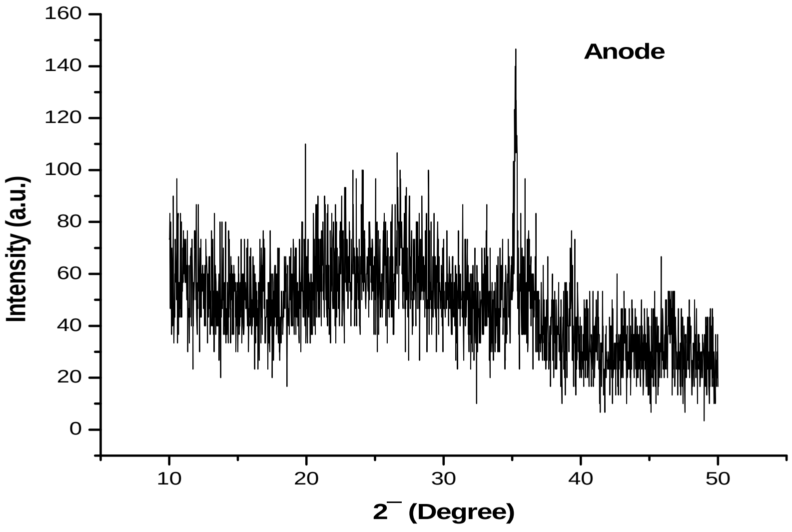

Figure: 2 XRD spectrum of Anode

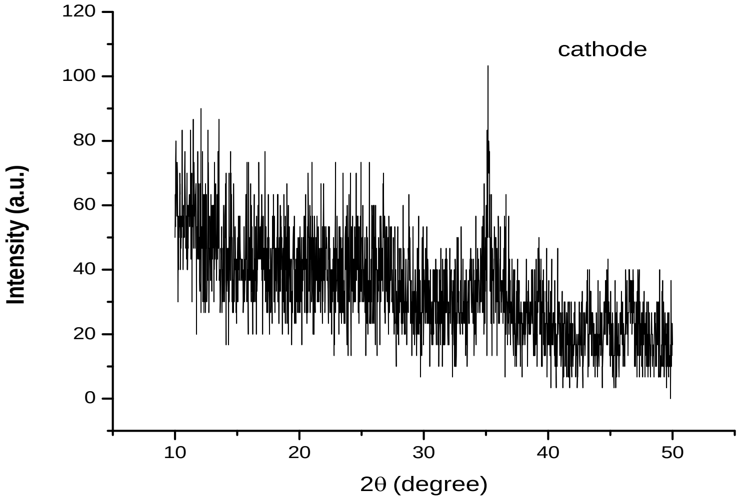

Figure: 3 XRD spectrum of Cathode

The effect of potential:

Taking different potential, the loss of mass reduction occurs at both cathode and anode. But greatest mass reduction takes place at anode at 60v if applied potential increases, there is increase of absorbance of maximum wave length. That means more dissolution of electrodes with more potential. Hence potential act as a controller parameter in electron pressure that causes reduction or oxidation. The equation E=I.R is satisfied that means increase in potential increase current. There is formation of ion during electrolysis under the effect at electric field. Mass of anode is oxidized into ions directly proportional to mass of anode.

Table 1. Reduced mass of tin after electrolysis process at various concentration at potential 60 V

Concentration (M)	Tin Mass (g)
0.005	0.0199
0.01	0.0499
0.02	0.1785
0.03	0.1810
0.04	0.2623
0.05	0.2897
0.06	0.2985

Table: 2 Reduced mass of anode at different potential

Potential (Volt)	Mass of Anode(gm)	Reduce Mass of Anode
05	1.018	0.110
10	0.932	0.086
15	0.748	0.184
20	0.579	0.169
25	0.350	0.229
30	0.169	0.181

Table: 3 Reduced mass of cathode at different potential

Potential (Volt)	Mass of cathode(g)	Reduce Mass of cathode(g)
05	1.083	0.747
10	1.041	0.042
15	0.795	0.137
20	0.656	0.139
25	0.404	0.252
30	0.189	0.252

The effect of electrolyte solution:

Different concentration of acid was taken. When acid concentration increases, dissolution of electrolyte increases. The greatest mass reduction occurs at 0.06 M HCl. During electrolysis HCl ionized into H^+ and Cl^- ions. At anode dissolution action is activated by Cl^- ions into Sn^{2+} or Sn^{4+}. From UV-Vis spectra, absorbance value increase with increasing of the acid concentration. It was achieved at 0.06 M HCl with the absorbance and wavelength values are 3.068 and 207 nm, respectively.

The SnO_2 nanoparties were formed (the oxidation number of tin increase from 0 to 2) in solutions as follows:

$$2H_2O_{(l)} + Sn_{(s)} \rightarrow 2OH^-_{(aq)} + Sn_2^+ (aq) + H_2 (g) \quad \ldots (5)$$

$$Sn^{2+}_{(aq)} + 2OH^-_{(aq)} \rightarrow Sn(OH)_{2(aq)} \quad \ldots (6)$$

This reaction can be continued to oxidize Sn2+ to Sn4+ as the following:

$$Sn(OH)_{2(aq)} \rightarrow SnO_{2(s)} + H_{2(g)} \quad \ldots (7)$$

The other possibility reaction can also occur. The oxidation number of tin increases from 0 to +4, the SnO_2 nanoparties was formed with the following reaction:

$$4H_2O_{(l)} + Sn(s) \rightarrow 4OH^-(aq) + Sn^{4+}_{(aq)} + 2H_{2(g)} \quad \ldots (8)$$

$$Sn^{4+}_{(aq)} + 4OH^-_{(aq)} \rightarrow SnO_2O_{(l)} \quad \ldots (9)$$

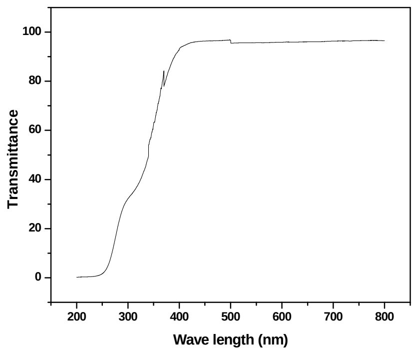

Figure -4 UV-visible spectrum of SnO_2 at 0.05M HCl conc.

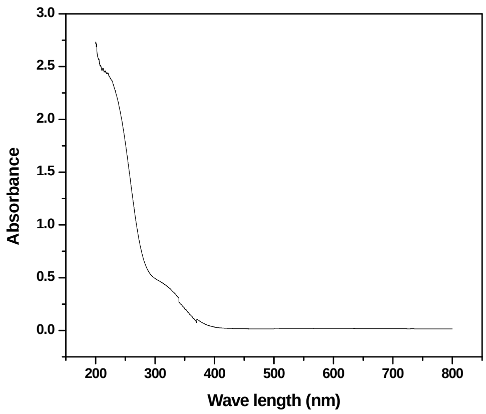

Figure-5 UV-visible spectrum of SnO2 at 60V potential

Conclusion

Based on the results, it is concluded that SnO_2 nanoparticles can be synthesized from the tin metal using an electro chemical method in HCL solution. The absorbance value of the conditions is 3.068 at 207nm. Characterization using XRD indicates the diffractogram pattern of product synthesis is SnO_2 nanoparticles. The structure is tetragonal. There is a loss of weight of Sn bare that dissolve during synthesis process indicates oxidation of Sn. At anode bubbles are formed which indicates formation of O_2. Potential difference affects the mass of cathode and anode. Applied potential is directly proportional to absorbance at maximum wavelength. Electric current is proportional to mass of anode which is oxidized into ions. When acid concentration increases, dissolution of tin metal increases.

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