International Journal of Advanced Technology in Engineering and Science
Vol. No.6, Issue No. 07, July 2018
www.ijates.com
ijates
ISSN 2348 - 7550

SYNTHESIS AND CHARACTERIZATION OF METAL OXIDE IN NANOPARTICLES

[a.] *Shivander Parmar ,*

Research Scholar, Mechanical Engineering Department, Adesh Institute of Technology, Gharuan,

[b]. *Er. Balwinder Singh ,*

Head and Assistant Professor, Mechanical Engineering Department, Adesh Institute of Technology, Gharuan,

ABSTRACT

RGO can be covalently functionalized either by π-π stacking, hydrogen bonding, and van der Waals interaction. RGO shows unique photoluminescence properties which occurs from the near-UV- visible to near-infrared wavelength range. It is used in various optoelectronics applications. RGO exhibits excellent charge mobility. Due to the high specific surface area, RGO can accommodate active species on its surface and facilitates the electron transfer at the surface of the electrode. RGO stable dispersion can be used to deposit on various substrates to make thin films in electrode materials. Thus an inexpensive electrode with the large surface area can be obtained with the use of RGO based conductive film.

Incorporation of RGO into metal oxides (i.e. TiO_2, ZnO etc.) leads to improvesphotocatalytic activity due to its ability to enhance charge mobility and lowers the recombination rate at the surface of the conductive glass. It also reduces the back electron at the surface as it acts as a barrier between the Photo electrode and conducting surface and leads to increase in the photocurrent

1.0. INTRODUCTION

Nanomaterial may be defined as a low dimension nanostructures having at least one dimension less than 100nm. Nanomaterials have found applications in almost every field of science and technology owing to their extraordinary and highly desirable physical, chemical, structural and optical properties. Nanomaterials are of great scientific interest as they serve as a narrow bridge between bulk materials and atomic or molecular structures.

The bulk material exhibits constant physical properties irrespective of its size, but at the nanoscale this is also often not the case. Nanomaterials have a high aspect ratio, size-dependent optical and electronic properties contrary to their bulk counterparts. Size-dependent properties like quantum confinement in semiconductor particles (quantum dots), surface plasmon resonance (SPR) in some metal particles and super-paramagnetism in magnetic materials are interesting and important for scientific and technological applications. The properties of materials change as their size approaches the nanoscale (<100nm) and the percentage of atoms at the surface of a material becomes significant [1].

The interesting and sometimes astonishing properties of nanoparticles are not partly due to the aspects of the surface of the material dominating the properties in lieu of the bulk properties. Nanoparticles exhibit a number of special properties relative to bulk material [2].

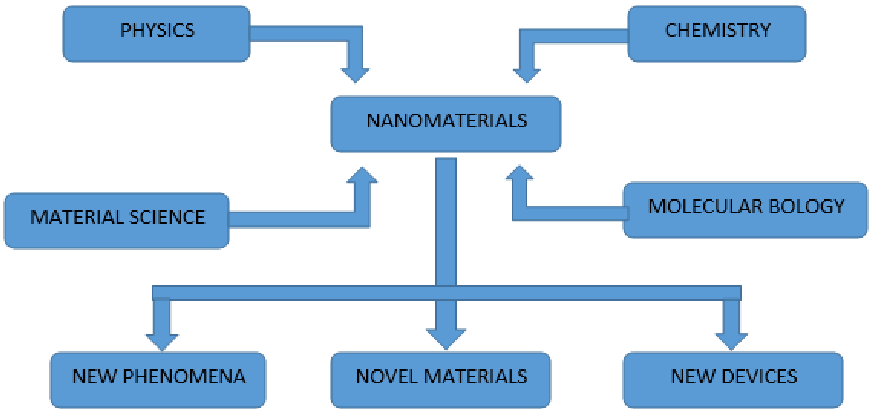

Fig. 1.1.Nanomaterials in different areas of science and Technology

1.1. Nano Composite

Inorganic and organic composite materials have been widely studied for a long time. When there is a nano sized inorganic phase in compound they are called nanocomposites. Organic inorganic nanocomposites are generally organic polymer composites with inorganic nanoscale fillers. They shows the advantages of both the organic material and inorganic material. They contains special nanofillers properties leading the materials with enhanced properties [3].

Inorganic nanoscale building blocks include nanotubes, layered silicates, nanoparticles of metal, metal oxides, semiconductors, and so forth, among all these, carbon is viewed as being very important. Various allotropes of carbon include fullerenes, carbon nanotubes, and graphene. Therefore, carbon composites gained substantial, academic and industrial interest. In fact, among the numerous inorganic/organic nanocomposites, carbon composites are most commonly reported in the literature. They exhibits a variety of applications so they have received much attention in recent years [4].

Nano-composites generally of three type:

2.0. LITERATURE SURVEY

Lagemant et al. [5]studied Electron surface state distribution on electron transport in DSSC having TiO_2photoelectrode effect the power conversion efficiency by controlling the electron transport. Electron transport should be fast because the slow transport of electrons led to increase in the recombination rate that decreases the efficiency of dye sensitized solar cells. For improvement in efficiency of DSSCs, further recombination rate should be minimum. Recombination occurs at the interfaces of cell, if the mobility of electron is high, then it increases the photon current density of electrons that further improves the PCE. It gives an idea about the electron transport rate nonlinear depend on electron concentration.

Rani et al. [6] and Manca et al. [7] analyzed TiO_2nanorod based photo-electrodes for dye solar cell with tunable morphology. Dye adsorb on the surface of TiO_2 depend on its surface properties. Electrons generated by striking of photons moves towards the TiO_2 NPs and generated holes are absorbed by the electrolyte. Surface area of TiO_2 should be maximized so that higher amount of dye is loaded on its surface, which increases the absorption of photon by DSSCs. So that fast electron transfer by changing the morphology of TiO_2 led to increase in the efficiency of the cell. For fast electrons diffusion rate and long electron lifetime, TiO_2 is used in the form of rod, wire.

Yao et al. [8] Proposed multilayered TiO_2photoelectrode for enhancing the photo conversion efficiency of DSSCs. Multilayered TiO_2 is easy to fabricate, compatible with current deposition process and can be modified by simply adjusting the process parameters of the system. It has wider spectral response and good molar extinction that attributes to improve power conversion efficiency. TiO_2 blocking layer is used to prevent from back scattering of the electrons from the electrolyte. By using a single layer of TiO_2-MWCNT nano-composite on TiO_2 film by spin coating process, TiO_2 blocking layer can be synthesized.

Rani et al. [9] fabricate, characterized the photo-conducting behavior of mesoporousZnO, TiO_2 and TiO_2/ZnO Bilayer System. Semiconductors are used for the photo-anode materials in DSSCs are mainly TiO_2 and ZnO. Both are nontoxic, highly stable, easy to prepare by sol-gel process and cost effective therefore used as cost effective solar cells. Both shows higher current density because they are n-type semiconductor. There is some limitation of photo-anodematerials used, to overcome this problem different band photo-anode materials is used and arrange in such a way that CB of materials used is higher than the CB of semiconductor material used.ZnO has a lower CB than the TiO_2 so that ZnO NPs are used first on the TCO followed by TiO_2 NPs. It reduces the backscattering of charge carriers and improves the PCE.

Fang et al. [10] studied grapheme quantum dot optimization of DSSCs. Low recombination rate and high surface area for absorption of photon is required for enhancing the performance of solar cells. Grapheme is used to improve these properties. Grapheme has good conductivity, high specific area. Due to the quantum confinement edge effect of grapheme quantum dots it shows (1) strong photo luminescent (2) good chemical stability (3) high optical absorptivity. Grapheme QD improves the photovoltaic performance.

Tripathi et al. [11] synthesized and characterized ZnO:Ag and TZO:Ag for plasmonicnanocomposite for enhanced dye synthesized solar cells. Silver nanoparticles are used as a dopant in TiO_2 and ZnO that produces the surface plasmon effect in the nano-materials. TZO:Ag bilayer of silver doped TiO_2nanomaterials and ZnOnano-materials fabricated on the TCO by doctor blade technique. Noble metal produces SPR that increases the light harvesting efficiency by increasing the photon path in the solar cells and decreases the recombination of electrons. Silver doped TiO_2 has improved electron collection efficiency as a result faster electron transfer.

Rani et al. [12] studied the influence of annealing temperature on sol-gel derived TiO_2 films. There is a great effect on TiO_2nanomaterials by changing the annealing temperature during the synthesis process. By structure modification, position and intensity of photo generated charge carriers transfer transition can be changed. Use of nano-composite materials as doping in the TiO_2nanomaterials changes the morphology. By increasing the annealing temperature the grain size of the TiO_2 nano-materials increases, but micro strain and stacking fault of the photo-anode film decreases. It reduces the concentration of lattice imperfections and improves the crystallinity of the TiO_2nanomaterials.

3.0. PROBLEM FORMULATION

Metal oxides have following limitations:

- Limited absorption spectra: Metal oxides shows the only absorption of UV-visible light from the whole spectra.
- Charge recombination: It is one of the major problems in photosensitive materials. It would create obstacles in the path of charge transport and charges no more available to pass through the external circuit.
- Electron Transport limitation: The charges excited by photons moves to the valence band but have limited transportation to flow and lead to surface trapping of electrons.

To overcome these problems, RGO is added to the TZO. TZO is the mixture of TiO_2nanomaterials and ZnOnanomaterials.

RGO increases the surface area of the TZO which lead to more mobility to the charges. RGO addition to TZO shows:

- High absorption solar spectra
- Reduces the backscattering of electron
- Reduces recombination of electrons
- Electron transport medium between nanomaterials

4.0. OBJECTIVES

1. Synthesis and characterization of TiO_2, ZnO and TZO nanomaterials samples.
2. Structural, optical and electrical properties of of TiO_2, ZnO and TZO nanomaterials samples.
3. Effect of rGO on TZO sample.
4. Structural, optical and electrical properties of ofrGO/TZO at 0.75, 1.25 and 2.0 wt% of rGO.
5. Optimization of sensitivity of TZO by adding 0.75, 1.25 and 2.0 wt% of rGO.

5.0. EXPERIMENTAL DETAILS

This includes materials selection, preparation, fabrication and characterization techniques are described. The materials characterization methods include XRD, FTIR, UV -Visible spectroscopy, SEM and Electrical Characterization.

5.1. Synthesis of Titanium di-oxide (TiO_2)

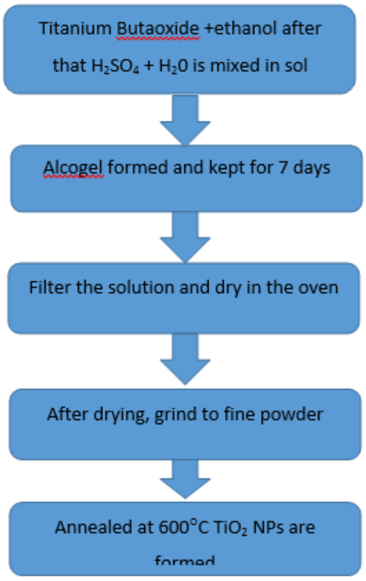

Fig. 1.1: Synthesis of TiO_2 nanomaterial

5.2. Synthesis of Zinc Oxide (ZnO)

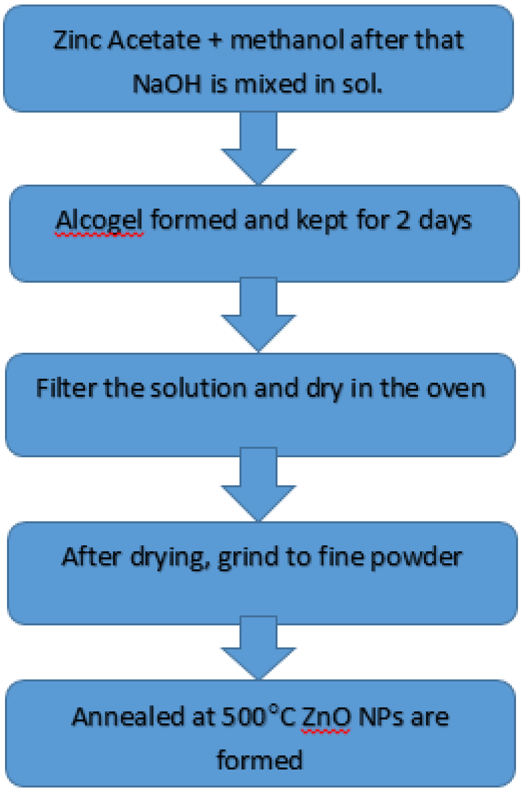

Fig. 2.3. Synthesis of ZnO nanomaterials

5.3. Synthesis of Reduced Graphene Oxide (RGO)

Graphene oxide (GO) was synthesized by modified Hummer's method using graphite flakes. The material required for GO formation was used in the following ratio

Table 1 Ratio of the materials for GO formation.

Material	Ratio
Graphite flakes and H_2SO_4	1:100
H_3PO_4 and H_2SO_4	1:4
$KMnO_4$ and graphite flakes	6:1
Graphite flakes and H_2O_2	1:6

5.4. **Composite Fabrication**

Photo electrode is prepared by doctor blade technique. TiO2 nanomaterial and ZnOnanomaterials are used to form photo electrode. Both metal oxides nanomaterial are mixed together by mortar cluster by continues grinding for several hours and TZO nano-sized powder is obtained. Further RGO is mixed in the TZO at different compositions by mortar cluster by continues grinding to obtain RGO/TZO composite. The samples formed are corresponding to three different concentration of 0.75%, 1.25% and 2% by weight composition [i].

Total six samples are prepared three of oxide nanomaterials each and three of RGO/TZO composite of different concentrations. In the paste formation all the powdered oxides are dispersed by grinding in the mortar and then particle stabilizer such as PEG (polyethylene glycol) is added to prevent the re-aggregation. These nanomaterials are deposited on the ITO glass substrate by doctor blade technique. After drying in the air for approximately 30 minutes the films annealed at 673K for 30 min to burn organic binders. After that silver electrode is deposited on the prepared film.

5.5. **Characterization Techniques**

Various techniques employed for the optical characterization, which were performed for the different samples are discussed below in brief.

X-ray powder diffraction (XRD)

Secondary Electron Microscopy (SEM)

Ultraviolet/visible spectroscopy

FTIR Spectroscopy

ijates
ISSN 2348 - 7550

6.0. CONCLUSION

We study the effect of addition of rGO to electrical properties of metal oxide semiconductors (TiO_2 and ZnO), which was fabricated by mixing of the materials by mortar cluster and deposited on an ITO substrate by doctor blade method. rGO, TiO_2, and ZnO were synthesized in lab using wet chemical method. The structural and morphological characterization was done by X-ray Diffraction, Fourier transformed infrared spectroscopy, UV-visible spectroscopy and scanning electron microscopy. Electrical characterization was done by plotting I-V characteristics, Dark Conductivity and Photoconductivity.

We studied that mixing of TiO_2 and ZnO gives us a mixture which exhibits properties of both the nano-materials. Both the nano-materials were mixed to give unique optical and electrical properties. It gives us more amount of photocurrent than ZnO and provides more stable persistent current than TiO_2. The activation energy and band gap both decreases by mixing of two nanoparticles, make this material perfect for photovoltaic properties.

We studied the effect of rGO on the metal oxide semiconductor's physical and electrical properties. rGO incorporated TZO nanomaterials shows immerse changes in the physical and electrical properties. The band gap and activation energy decreases by the mixing of rGO to metal oxides, as the concentration of rGO (by wt%) increases the value of band gap and activation energy decreases, resulting in the changing in crystallite size by increasing the concentration of rGO (by wt%) into metal oxide mixture, this is due to quantum confinement effect.

rGO incorporation to metal oxides leads to drastic change in dark conductivity and photo conductivity of the nanomaterials as studied above. In conclusion we can say that:

The addition of rGO lowers the recombination rate of electrons and also lowers the backscattering of electrons resulting in the increase in the photocurrent.

rGO incorporation leads to yield more amount of current as it acts an electron transport layer between the nanomaterials.

rGO addition to the TZO leads to a huge persistent photocurrent resulting in the improving in the photo response, which gives us a suitable photo electrode.

REFERENCES

1. Tanaka, Toshikatsu, G. C. Montanari, and R. Mulhaupt. "Polymer nanocomposites as dielectrics and electrical insulation-perspectives for processing technologies, material characterization and future applications." *IEEE transactions on Dielectrics and Electrical Insulation* 11.5 (2004): 763-784.
2. Safdari, Masoud, and Marwan S. Al-Haik. "Synergistic electrical and thermal transport properties of hybrid polymeric nanocomposites based on carbon nanotubes and graphite nanoplatelets." *Carbon* 64 (2013): 111-121.
3. Kesong, et al. "Graphene-polymer nanocomposites for structural and functional applications." *Progress in Polymer Science* 39.11 (2014): 1934-1972.

4. Peponi, Laura, et al. "Processing of nanostructured polymers and advanced polymeric based nanocomposites." *Materials Science and Engineering: R: Reports* 85 (2014): 1-46.
5. Van de Lagemaat, J., N-G. Park, and A. J. Frank. "Influence of electrical potential distribution, charge transport, and recombination on the photopotential and photocurrent conversion efficiency of dye-sensitized nanocrystalline TiO_2 solar cells: a study by electrical impedance and optical modulation techniques." *The Journal of Physical Chemistry B*104.9 (2000): 2044-2052.
6. Rani, Mamta, KiratBir Kaur, and S. K. Tripathi. "Synthesis and Characterization of Dye Sensitized TiO_2Nanorods." *AIP Conference Proceedings*. Vol. 1393. No. 1. AIP, 2011.
7. De Marco, Luisa, et al. "Novel preparation method of TiO_2-nanorod-based photo-electrodes for dye-sensitized solar cells with improved light-harvesting efficiency." *The Journal of Physical Chemistry C* 114.9 (2010): 4228-4236.
8. Yao, B. D., et al. "Formation mechanism of TiO_2 nanotubes." *Applied physics letters* 82.2 (2003): 281-283.
9. Rani, Mamta, and S. K. Tripathi. "Fabrication, characterization and photo-conducting behavior of mesoporous ZnO, TiO_2 and TiO_2/ZnO bilayer system: comparative study." *Energy and Environment Focus* 2.3 (2013): 227-234.
10. Fang, Zheyu, et al. "Plasmon-induced doping of graphene." *Acs Nano* 6.11 (2012): 10222-10228.
11. Tripathi, S. K., Mamta Rani, and Navjot Singh. "ZnO: Ag and TZO: Ag plasmonicnano composite for enhanced dye sensitized solar cell performance." *ElectrochimicaActa* 167 (2015): 179-186.
12. Rani, Mamta, Saeed J. Abbas, and S. K. Tripathi. "Influence of annealing temperature and organic dyes as sensitizers on sol–gel derived TiO_2 films." *Materials Science and Engineering: B* 187 (2014): 75-82.