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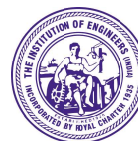
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EMERGING TRENDS IN SCIENCE AND TECHNOLOGY (ETIST-2021)

27th October 2021

Jointly Organized by

Department of Biological Science, Physical Science and Computational Science

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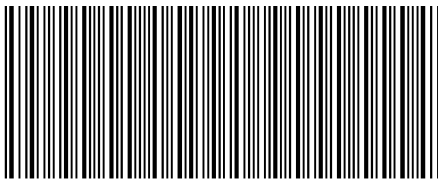
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ABOUT THE INSTITUTION

A nation's growth is in proportion to education and intelligence spread among the masses. Having this idealistic vision, two great philanthropists late. S.P. Nallamuthu Gounder and Late. Arutchelver Padmabhushan Dr.N.Mahalingam formed an organization called Pollachi Kalvi Kazhagam, which started NGM College in 1957, to impart holistic education with an objective to cater to the higher educational needs of those who wish to aspire for excellence in knowledge and values. The College has achieved greater academic distinctions with the introduction of autonomous system from the academic year 1987-88. The college has been Re-Accredited by NAAC and it is ISO 9001 : 2015 Certified Institution. The total student strength is around 6000. Having celebrated its Diamond Jubilee in 2017, the college has blossomed into a premier Post-Graduate and Research Institution, offering 26 UG, 12 PG, 13 M.Phil and 10 Ph.D Programmes, apart from Diploma and Certificate Courses. The college has been ranked within Top 100 (72nd Rank) in India by NIRF 2021.

ABOUT CONFERENCE

The International conference on “Emerging Trends in Science and Technology (ETIST-2021)” is being jointly organized by Departments of Biological Science, Physical Science and Computational Science - Nallamuthu Gounder Mahalingam College, Pollachi along with ISTE, CSI, IETE, IEE & RIYASA LABS on 27th OCT 2021. The Conference will provide common platform for faculties, research scholars, industrialists to exchange and discuss the innovative ideas and will promote to work in interdisciplinary mode.

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Effect of capping agents on the Morphology of Nanostructured NiO

N. Neelakandeswari^{1*} – R. Vasanthapriya² – M. Chitra³ – K. Uthayarani⁴

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ABSTRACT:

Nickel Oxide (NiO) is a cost-effective semiconducting material finds its applications in many areas like catalysis, electrolysis, photovoltaics and sensors. Herein we report the hydrothermal preparation of nanostructured NiO in the presence and in the absence of capping agents and their characterization by various state of the art techniques like Fourier-transform Infra Red spectroscopy (FT-IR), Ultra – Violet and Visible Spectroscopy (UV-Vis), photoluminescence (PL), Powder X-Ray diffraction (XRD) and Scanning Electron Microscopy (SEM). Effect of capping agents on the structural and optical properties of NiO nanostructures is studied and the results are reported here.

Keywords: Nickel oxide, capping agent, size control, structural properties.

1. INTRODUCTION

Innovations in the field of nanotechnology in the recent years motivate the researchers to produce nano sized particles of metals, metaloxides and metalsulphides. Among the iron group (Fe, Co, Ni) metal oxides, NiO is attractive owing to its peculiar properties of wide band gap (3 eV), NaCl type crystal lattice, special mechanical, optical and antiferromagnetic properties [1]. As a result, NiO finds applications in catalysis, electrode materials, sensors, magnetic materials, supercapacitors and solar cells [2]. Literature revealed that the properties and applicability of NiO purely depends upon the size and morphology of the nanostructures and can be controlled by the preparation protocol [3]. Various techniques are reported for the synthesis of NiO in different forms such as nanoparticles, sheets, fibres, ribbons, belts, wires and tubes [4] using spray pyrolysis, sol-gel, co-precipitation, electrospinning, hydrothermal, solvothermal and microemulsion techniques [5]. Based on this, it is understood that, apart from the technique employed for the synthesis of nanomaterial, surfactant or a capping agent used also play a major role in controlling the size and shape [6]. Hence, in this paper, it is aimed to investigate the role of surfactant on the structural and optical properties of NiO nanostructures prepared via solvothermal technique using various capping agents each in a category of neutral, cationic and anionic.

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2. EXPERIMENTAL DETAILS

Nickel chloride, cyclohexane, sodium dodecyl sulphate (SDS), cetyl trimethyl ammonium bromide (CTAB), starch, hexamethylene tetramine (HMT), ethanol and ammonia were used are of analytical grade and used as received without any further purification. 0.1M aqueous solution of nickel chloride was mixed with cyclohexane and aqueous SDS with constant stirring at room temperature (25°C). 0.2 M aqueous solution of HMT was added to the above mixture with constant stirring at pH – 9 and the pH of the suspension was adjusted with the addition of ammonium hydroxide at room temperature. This suspension was stirred for 1h, carefully transferred into a Teflon lined stainless steel autoclave and maintained at 100° C for 2h. The autoclave was cooled to room temperature, gel obtained was filtered, washed several times with distilled water and ethanol separately, dried at 100° C for 3 hrs and calcined at 500° C for 3hrs. The procedure was repeated without capping agent and with starch and CTAB as capping agents in place of SDS. Nanostructures prepared without surfactant and with starch, SDS and CTAB were named respectively as N1, N2, N3 and N4 for convenience.

The diffraction pattern of all samples were recorded using a PANalytical X'pert PRO X-ray diffractometer using Cu-K α radiation ($\lambda = 1.5406\text{\AA}$) as the Xray source at room temperature. Attenuated total reflection infrared spectra of the samples were recorded on a Perkin Elmer RXI FT – IR spectrometer with ATR attachment. Photoluminescence spectra (PL) of the powdered samples were recorded using AGILENT fluorescence spectrometer. Ultra violet- visible spectra of the powdered samples were recorded with Analytic Jena UV-Vis spectrometer. The surface morphology of NiO nanoparticles was characterized by FE-SEM (ZEISS ultrafield emission Scanning Electron Microscope).

3. RESULTS AND DISCUSSION

ATR spectrum of the NiO nanoparticles prepared by solvothermal route is used to identify the formation of NiO nanoparticles. Presence of vibrations around 3600 cm^{-1} and 3738 cm^{-1} in all the samples correspond to the O-H Stretching vibrations of interlayer bounded water molecules and surface water molecules respectively. Band observed around 3860 cm^{-1} is attributed to the terminal Ni (OH) $_2$ and is associated with NiO network. Intense bands observed around 1690 & 1525 cm^{-1} are assigned to the O-H bending vibrations of surface Ni (OH) $_2$ and surface bound water molecules [7]. Band observed around 701 cm^{-1} is due to the vibration of Ni - O bond [8]. Absence of other organic functionalities in the IR revealed the decomposition of organic capping agents during the calcination process.

UV-Visible and photoluminescence spectra of prepared NiO nanostructures are given in figure-1. Band gap was calculated using the formula $E=hc/\lambda$ and the calculated band gap for all the prepared nanostructures for N1, N2, N3 and N4 are 4.01, 3.76, 3.92 and 3.92 eV respectively. The results are in agreement with the literature [9].

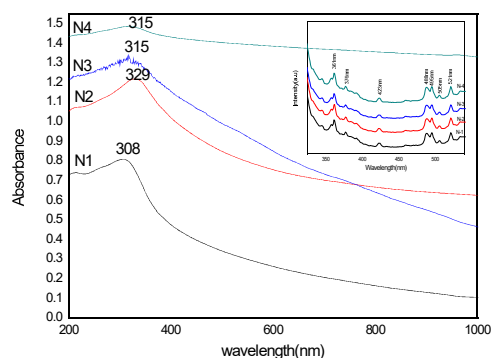


Figure 1 UV-Visible spectrum of NiO nanostructures (inset PL spectrum)

PL spectra recorded at 310 nm excitation (Figure-1(inset)) possesses 7 emissions at 361nm, 376 nm, 423 nm, 488 nm, 495 nm, 505 nm and 521 nm for all the four NiO nanostructures. Two UV emission peaks correspond to the near band edge emission due to the intraionic $3d^8-3d^8$ transitions of Ni^{2+} [10]. Visible light emission observed in both green and blue regions might be due to intrinsic defects arising from Ni and O vacancies [11].

To find out the crystallite size and morphology, powder XRD and FE-SEM were recorded. Powder XRD pattern of the prepared nanostructures are presented in figure-2. Crystalline nature of the prepared samples are identified from the sharpness of the peaks. All the diffraction peaks are compared with the standard JCPDS card (File No: 47-1046) and found that the prepared NiO unit cell is cubic with face centred phase (fcc) and are indexed as (111), (200), (220), (311) and (222) respectively. The absence of diffractions corresponding to $Ni(OH)_2$ and Ni_2O_3 confirms the formation of phase pure NiO [7]. Crystallite size was calculated using the Scherer's formula [12] and it was found to be 35, 30, 43 and 44 nm respectively for N1, N2, N3 and N4. Report revealed that CTAB has a poor effect on the size control, which is evidenced from the larger crystallite size observed for N4 (43 nm) [13]. Calculated crystallite size for N1 is in accordance with the UV results. But for samples N2, N3 & N4, variation of band gap and crystallite size do not follow the common trend. The observed variation might be correlated to the morphology (Figure-3) of the prepared nanostructures and presence of chemical defects or vacancies in the crystal structure introduced during the aggregation of crystallites to various morphologies [14].

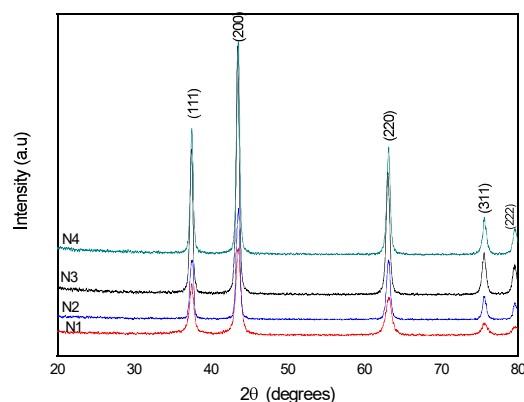


Figure 2 XRD pattern of NiO nanostructures

Morphology of the nanostructures were imaged using FE-SEM and the images are presented in figure – 4. Morphology varies from spherical, flakes, bipyramidal and pyramid like structures for NiO prepared without surfactant and with starch, SDS and CTAB surfactants respectively. FE-SEM image of N1 is in accordance with the literature [9] and the band gap reported for spherical morphology is also 3.99 eV, which is almost close to the calculated band gap for N1. Neutral capping agent directed the formation of tetrahedral faceted flake like nanostructures whereas anionic and cationic surfactants assisted the growth of bipyramidal and pyramidal morphologies. Formation of such morphologies can be explained based on the mechanism of formation of NiO nanostructures proposed by Jia Hong Pan et al., [15]. According to them, mechanical agitation, rate of formation of Ni(OH)₂ from the precursor and the anions associated with lattice of Ni(OH)₂ greatly influence the morphology. Generally, chloride ions will lead to the formation of sheet or flake like morphology and sulphate ions lead to hierarchial sheet like morphology. Another report by D. Mohammadyani [16] and team revealed that the appearance of various morphologies is due to the aggregation of particles and might be due to the influence of magnetic interaction between the particles as well as the interfacial energies associated with them.

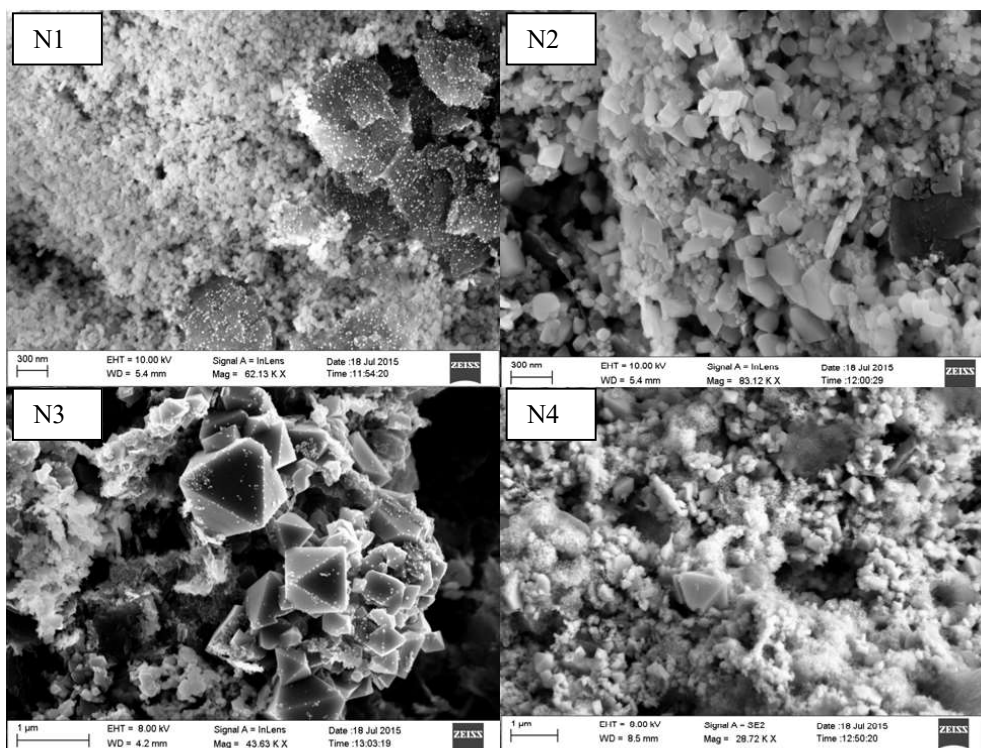


Figure 3 FE-SEM images of NiO nanostructures prepared

Hence in the present work, formation of different morphologies could be explained as, capping agents used during solvothermal synthesis of NiO, greatly controlled the size of Ni(OH)₂ formed and resulted in the formation of very small nanocrystals. As the organic moiety went off during calcination, the nanocrystals formed possess large surface area and hence surface energy, which directs them to aggregate so as to and henceforth minimize the surface energy [15,17]. Since there was no anion available in neutral surfactant used (starch), aggregation of nanocrystals in N2 leads to the formation of flakes whereas SO₄²⁻ in SDS and Br⁻ in CTAB direct the aggregation towards bipyramidal

and pyramidal microstructures respectively. This could be correlated to the appearance of same band gap for both N3 and N4, besides the difference observed in average crystallite size.

4. CONCLUSION

NiO nanostructures were prepared via solvothermal techniques using neutral, anionic and cationic capping agents. Morphology varies from sphere, flakes, bipyramidal and pyramidal respectively for without capping agent and prepared with starch, SDS and CTAB as capping agents. Reason behind the formation of different morphologies was discussed.

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DSSC PERFORMANCE OF ZINC - TIN - VANADIUM OXIDE NANOCOMPOSITE USING BEETROOT (*BETA VULGARIS*) AS DYE SENSITIZER

M. Chitra¹ - K. Uthayarani^{1*} - R. Vasanthapriya¹ and N. Neelakandeswari²

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ABSTRACT: In this present work, zinc - tin - vanadium oxide (ZTVO) nanocomposite prepared using hydrothermal route was systematically characterized. Dye-sensitized solar cell (DSSC) was fabricated using this ZTVO nanocomposite as photoanode and betalain from beetroot (*Beta vulgaris*) as natural dye. This simple protocol was formulated at a low cost for the first time for DSSC fabrication and it has attained the efficiency of 3.41%. This better efficiency of ZTVO might be due to larger surface area, presence of pores in addition to smaller band gap (1.97 eV).

Keywords: nanocomposites, solar energy materials, zinc-tin-vanadium oxide, beetroot, natural dye, band gap

1. INTRODUCTION

The low-cost, high-efficiency and non-pollution aspects of Dye-sensitized solar cells (DSSCs) has considered as one of the most promising alternatives to silicon based photovoltaic cells in the last two decades [1]. DSSCs became more interesting since after the phenomenal work carried out by O'Regan and Gratzel in 1991. In the present scenario, natural dyes are employed as light harvesting elements to provide the charge carriers. These natural dyes provide a feasible alternative to expensive organic based DSSCs (rare metal complexes) owing to its low cost, non-toxicity, ease of extraction using simple chemical procedures, environmental friendly, easily biodegradable, abundance and large absorption coefficient. The pigmented dyes such as chlorophyll from fig leaves exhibited an efficiency of 0.64 %, anthocyanin extracted from eggplant and that of pomegranate records an efficiency of 0.64 % and 2.00 % respectively, betalain from red turnip gives out 1.7 % efficiency etc. Among them in this present work,

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