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SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY OF NiO NANOPARTICLES

Dhanuja B. S[1,2], Dedhila Devadathan[1], Baiju V[1], Biju R[1], Raveendran R[1]

[1]Nanoscience Research Laboratory, Department of Physics, Sree Narayana College,
Kollam, Kerala, (India)
[2]Department of Physics, Sree Narayana College, Sivagiri, Sreenivasapuram P.O, Varkala
Thiruvananthapuram , Kerala, (India)

Abstract

In the present study, nanoparticles of NiO were synthesised through controlled co-precipitation method and microwave assisted fast synthesis method. In both methods NiO was synthesised in presence of citric acid and also in presence of polyvinyl alcohol (PVA). Structural characterizations of all the four synthesized samples were carried out using XRD. Comparison of XRD's of metal oxides with JCPDS confirmed that the formed metal oxide was NiO. Phase purity and crystallinity of these samples were confirmed from XRD. Crystallite sizes were calculated using Scherrer equation. Nickel oxide nanoparticles showed visible range absorption in addition to UV range. From band gap calculation it was seen that all of them showed subgaps. The present study investigates the antimicrobial activity of the synthesized samples as antibacterial agents. The gram positive bacteria: Staphylococcus aureus and the gram negative bacteria: Escherichia coli are the microbes used in the present study. The study confirms that the nickel oxide samples prepared in the present study can be used as antibacterial agent against Escherichia coli. The results suggest that proper tuning can make them good antimicrobial agent.

Keywords:* *Nanoparticles, Antibacterial Agents

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i.INTRODUCTION

Among transition metal oxides, nickel oxide (NiO) bulk and nano size have received considerable attention due to their wide range of applications in different fields [1-3]. Because of the quantum size and surface effects, NiO nanoparticles exhibit catalytic, optical, electronic, and magnetic properties that are significantly different than those of bulk-sized NiO particles [4, 5]. The novel properties of nanomaterials such as large surface area, potential for self assembly, high specificity, high reactivity and catalytic potential make them an excellent candidate for various applications [6]. In the present work nickel oxide (NiO) nanoparticles is prepared in presence of capping agent, citric acid using co-precipitation method. The aim of the work is to study the variations in the properties obtained when NiO is prepared using microwave assisted fast synthesis method. Also when NiO was prepared in presence of polyvinyl alcohol instead of citric acid using co-precipitation method and microwave assisted fast synthesis method. The NiO nanoparticles synthesized is characterized using X-ray Diffraction Spectroscopy (XRD). The optical studies of the prepared samples are done using UV/Vis spectroscopy. In the present work there is a pressing demand to develop new antimicrobial agents and discover novel strategies due to the emerging infectious diseases and the development of drug resistance in the pathogenic bacteria and fungi. The present study investigates the antimicrobial activity of the synthesized samples as antibacterial agents. The results of all the four samples are compared.

2 EXPERIMENTAL

AR grade chemicals obtained from Merck were used for the preparation of NiO nanoparticles. NiO was prepared in presence of citric acid by the co-precipitation (NCA) and microwave method (NCAM) and in presence of polyvinyl alcohol (PVA) NPVA and NPVAM. The samples annealed at 500^0c were used for analysis. XRD study was carried out using XPERT-PRO model powder diffractometer (PAN analytical, Netherlands) employing Cu-K_α radiation ($\lambda = 1.54060A^o$) operating at 40kV, 30mA. The UV/Vis spectrum was obtained using JASCO V-650 UV visible spectrophotometer.

3 RESULTS AND DISCUSSIONS

3.1 XRD Analysis

Figure 1 shows the XRD of NCA. From the XRD itself it is clear that all the samples formed shows crystalline nature with sharp crystalline peaks. The broader diffraction peaks obtained indicated the smaller crystallite size. In order to confirm the phase purity of the samples prepared, the interplanar spacing (d_{hkl} values), 2θ values and relative intensity values of nickel oxide corresponding to the observed diffraction peaks were compared with the standard values of nickel oxide as reported by JCPDS-International Centre for Diffraction Data. From the comparison of diffraction peaks NF and NS with JCPDS-ICDD pattern number #78-0423 it is clear that NiO formed was a cubic system with FCC lattice. The well defined X-ray diffraction peaks in the case of all samples indicate that NiO formed are crystalline in nature. Also, the diffraction peaks are notably broadened indicating the smaller crystallite size.

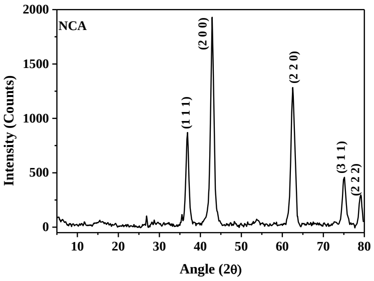

Fig.1 XRD of NCA

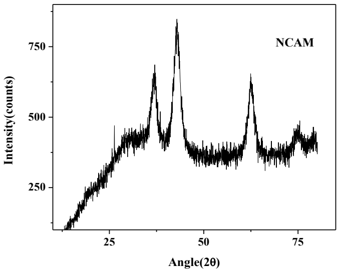

Fig.2 XRD of NCAM

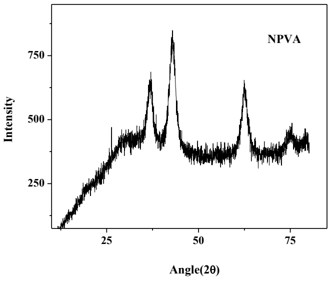

Fig.3 XRD of NPVA

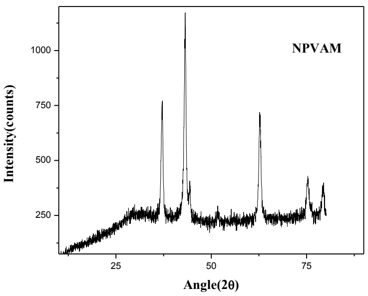

Fig.4 XRD of NPVAM

For NPVA, NCAM and NPVAM the diffraction peaks in addition to the 2θ values seen in NCA additional peaks were found. The presence of unremoved PVA might have created these impurity peaks. Annealing the samples at higher degrees can remove PVA completely and can give pure crystalline peaks. Using curve fitting the full width at half maximum of the major peaks was found and from this the crystallite size of all the samples were found. The result obtained NCA was 10.84 nm For NCAM 18.49 nm, NPVA 30.86 nm and NPVAM 17.81 nm respectively.

3.2 UV/Vis Spectrum Analysis

The absorbance spectrum of all the four samples were studied and compared. It is reported in literature that optical energy band gap of NiO lies in the range (3.6–4.0 eV) and is attributed to charge-transfer transition between the valence bands of O (p) states to conduction bands of Ni d (e_{2g}) states. Figure 5 shows the absorption spectrum of NCA. All the samples showed absorbance in both UV and visible region. The multiple peaks obtained which shows the presence of subgaps.

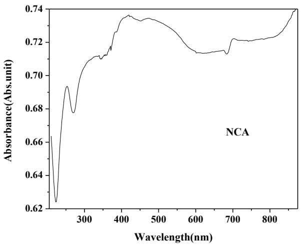

Fig.5 UV Absorbance spectrum of NCA

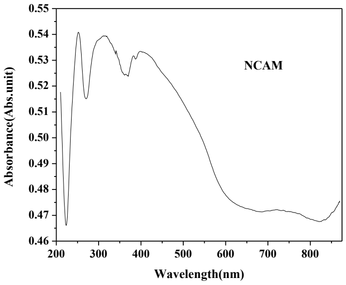

Fig.6 UV Absorbance of spectrum NCAM

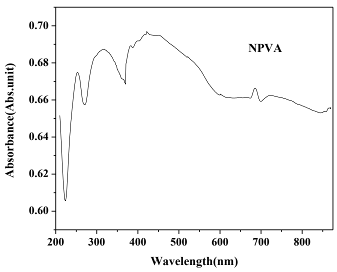

Fig. 7 UV Absorbance of spectrum NPVA

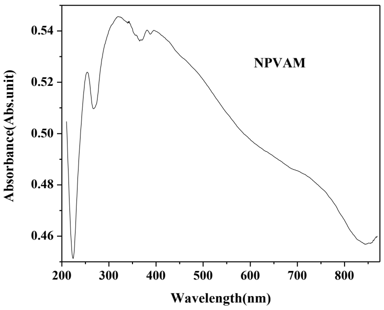

Fig. 8 UV Absorbance spectrum of NPVAM

From absorbance spectrum, Tauc plot was drawn and the corresponding energy gap was found. The energy band gap of the material is related to the absorption coefficient α by the Tauc relation, $\alpha = A\ (h\nu - E_g)^n$, where A is a constant, hν is the photon energy ($\nu = c/\lambda$), E_g is the band gap and n is either 2 for an indirect transition or ½ for a direct transition. The $(\alpha h\nu)^2$ vs hν for all the samples were also plotted. Figure 9 shows Tauc plots drawn for NCA. Similar results were obtained for other three samples.

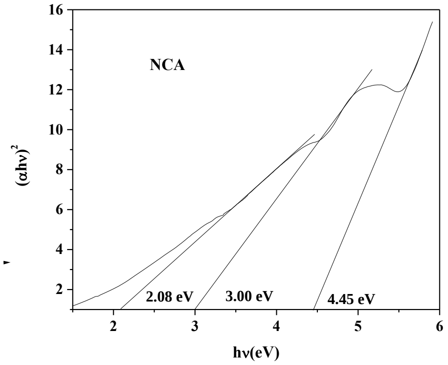

Fig.9 Tauc plot for NCA

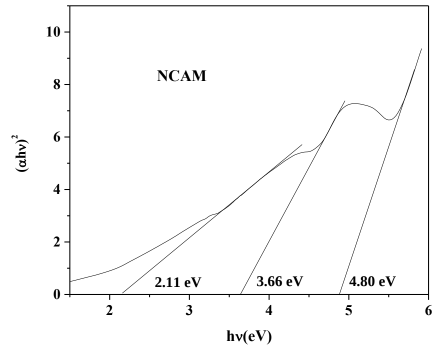

Fig.10 Tauc plot for NCAM

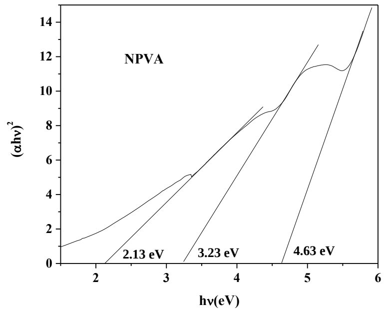

Fig.11 Tauc plot for NPVA

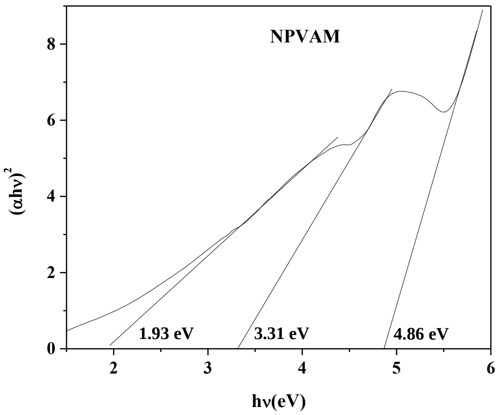

Fig.12 Tauc plot for NPVAM

3.3 Antibacterial Studies

The antibacterial activities of all the four samples were studied in detail. The antimicrobial activity of the nanoparticles is generally known to be a function of the surface area which is in contact with the microorganisms. Reactions take place at the surface of a chemical or material. Hence, the smaller size and the higher surface to volume ratio i.e., larger surface area, enhanced interaction with the microbes is seen. Figure 13 shows the comparison of antibacterial activity of NCA, NCAM, NPVA and NPVAM. Antimicrobial effects of NiO nanoparticles can be attributed to several mechanisms:

1. Induction of oxidative stress due to ROS (reactive oxygen species) generation,
2. Membrane disorganization due to accumulation of nanoparticles in the bacterial membrane and also their cellular internalization,
3. Release of metal ions that may be responsible for antimicrobial activity by binding to the membrane of microorganisms.

The antibacterial mechanism can also be attributed to the destruction of the outer membrane of bacteria by the generated superoxide anion radicals ($\bullet O^{2-}$) as the reactive species. The reactive species such as •OH and $\bullet O^{2-}$ are generated at the catalyst's surface, hence the high surface area is very beneficial for degradation of bacteria. The activity was found to be highest in case of gram negative bacteria. All the four samples showed no activity for S.aureus. The variation in the sensitivity or resistance to both gram positive and gram negative bacteria populations could be due to the differences in the cell structure, physiology, metabolism or degree of contact of organisms with nanoparticles.

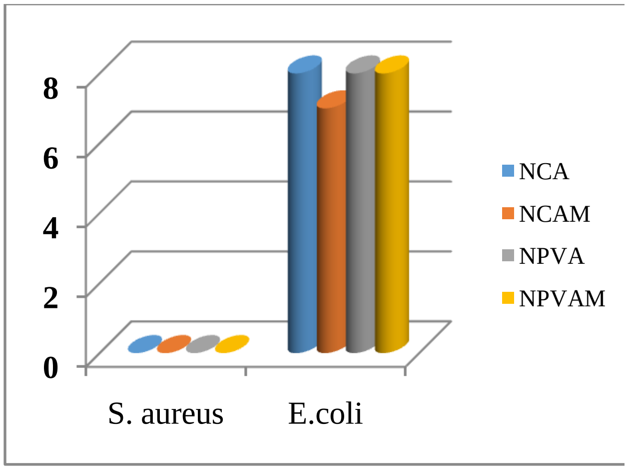

Fig.13 Comparison of Antibacterial activity of NCA, NCAM, NPVA and NPVAM

4 CONCLUSIONS

Comparison of XRD's of metal oxides with JCPDS confirmed that the formed metal oxide was NiO. Crystallite sizes were calculated using Scherrer equation. All the four samples prepared in this work showed strong UV as well as visible absorption. Hence all these materials could be suitable for anti UV applications. Also these materials could be a good candidate as photocatalyst for photocatalysis in presence of UV, as antibacterial agents, etc. From band gap calculation it was seen that all of them showed subgaps. The antibacterial activity was found to be highest in case of gram negative bacteria. All the four samples showed no activity for S.aureus. Nickel oxide samples prepared in present study showed comparable activity towards E.coli. The results suggest that proper tuning can make them good antimicrobial agent.

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