

SYNTHESIS CHARACTERIZATION AND OPTICAL PROPERTIES OF CONDUCTING POLYANILINE DOPED WITH MGO NANOCOMPITES

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Abstract:- MgO nanoparticles were prepared by using wet chemical methods such as co-precipitation and hydrothermal method. The synthesized MgO nanoparticles were characterized by X – ray diffraction analysis. PXRD result reveals that the nanoparticles are in crystalline form with cubic phase and the crystalline size was calculated using Debye Scherrer formula. UV results shows that absorption peak at wavelength of 295 nm and 297 nm were obtained for co-precipitation method and hydrothermal method respectively and their band gap values were also calculated and reported. The characteristic irrational frequency of Mg=O at 548 cm⁻¹ agrees well with the literature. The morphology of the synthesized nanoparticles was studied by using Scanning Electron Microscopy (SEM).

Key words: Nanoparticle, MgO, PANI (6000), Hcl, Powder X-ray diffraction, Scanning electron microscopy, FTIR analysis, UV-analysis.

I. Introduction

MgO were prepared by a low-temperature combustion method. The crystalline nature and particle size of the samples were characterized by Powder X-ray diffraction analysis (XRD). The morphology of prepared nanocrystals was studied by scanning electron microscope (SEM) The presence of functional groups in the samples was analyzed using FT-IR analysis. The optical absorption of the synthesized samples was studied using UV absorption spectral analysis [1,5].

II. Experimental procedure

2.1 Preparation of Magnesium Oxalate of the MgO is synthesized through self-propagating low temperature combustion. This precursor is prepared by dissolving equimolar quantity of magnesium nitrate and oxalic acid in 100ml distilled water. This mixture was well stirred in a beaker. The white precipitate of magnesium oxalate obtained was filtered through sintered glass funnel and washed with double distilled water.

2.2 Synthesis Of Magnesium Oxide of the Thermal decomposition of magnesium precursor with a fuel leads to the formation of high surface area of MgO. The above prepared magnesium oxalate was mixed with polyethylene glycol in the weight ratio 1:5 and ground in a pestle and mortar. The resultant solid was placed in a crucible and heated in air. It was observed that initially PEG melted then frothed and finally ignited to give MgO as a residue. On cooling to room temperature, no traces of carbon impurities were observed in the final residue of MgO. As the reaction is fast, i.e. going to completion within 10 min, and ignites automatically .

2.3 Synthesis of Pani was synthesized by chemical oxidative polymerization method using aniline as monomer in the presence of hydrochloric acid as a catalyst and ammonium per sulphate as oxidant. Aniline and HCL are taken in a proper ratio and dissolved in 100ml of distilled water. APS was added drop wise into the mixture. The reaction mixture continuously stirred in a magnetic stirrer for 6 hours, under ice cold temperature. After 6 hrs the precipitate was separated by filtering, dried. The final product was grounded into fine powders.

2.4 Powder X-ray diffraction:

The X-ray diffraction patterns of the samples in this present study are obtained on Philips X-ray diffract meter using CuK α radiation ($\lambda = 1.5406 \text{ \AA}$). The diffract grams were recorded in terms of 2θ in the range $10 - 90^\circ$ with a scanning rate of 2 per minute.

Polyaniline is used Figure1. Shows X-ray diffraction pattern of Polyaniline. Careful analysis of X-ray diffraction of polyaniline suggests that it has amorphous nature with a broad peak centered on 2θ is 25.49° which corresponds to (111) diffraction planes of pure PANI.

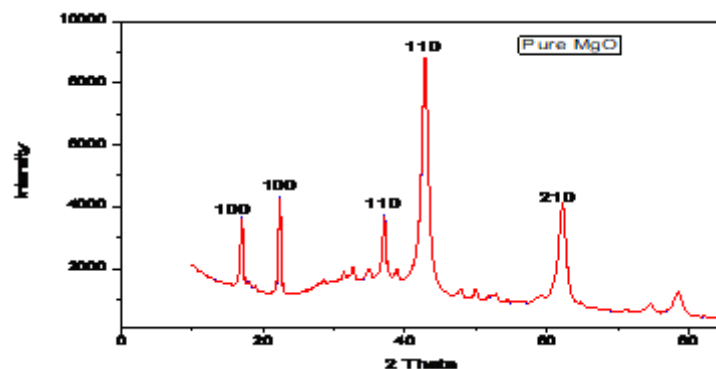


Fig.1 XRD Pattern

2.5 Scanning Electron Microscopy (SEM):

The powder morphology of Polyaniline and its composites sintered in the form of pellets (to measure grain size) are investigated using Phillips XL30 ESEM scanning electronic microscope (SEM). The samples in the form of pellets are mounted on aluminum platform; conducting gold is sputtered on the sample to avoid charging at the sample surfaces. They were examined under SEM and selected areas were photographed

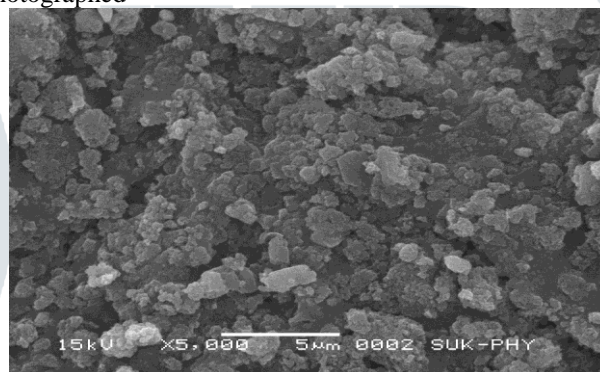


Fig.2 SEM Image for Pure MgO

2.6 Optical Absorption Spectroscopy (UV-analysis):

The synthesized nanoparticles of MgO – PANI Nanocomposites were used to study the optical absorbance in the wave length range between 200nm and 800 nm. Absorbance curves are shown in fig.3. The absorbance curve for PANI with 10 wt% of a composites shows an absorption edge at wave length 285nm which corresponds to an optical band gap of nearly 4.3 eV. It is observed that as the wt% of MgO was increased the absorption edge found to shift towards the longer wave length indicating the decrease in band gap to the order of 2.8eV.

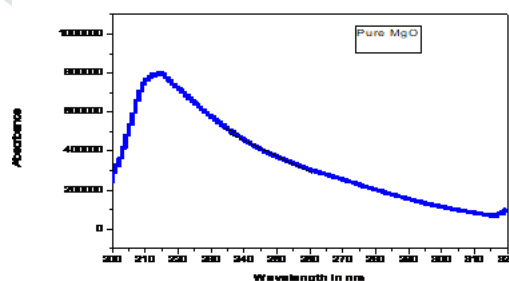


Fig.3 UV for Pure MgO

2.7 FTIR Analysis:

The IR spectra of all the samples are recorded on Perkin Elmer (model 783) IR spectrometer in KBr medium at room temperature. For recording IR spectra, powders are mixed with KBr in the ratio 1:25 by weight to ensure uniform dispersion in KBr pellets. The mixed powders are pressed in a cylindrical dye to obtain clean discs of approximately 1mm thickness. The characterization of polyaniline and its composites by spectroscopic method is important, as it gives information not only about various molecular – levels interactions but also on the type of charge carriers. Infrared spectroscopy is a powerful tool to determine the structural changes that occurs during doping and dedoping process [6,7,8]. Various groups have reported IR results of polyaniline, but the IR results of composites are scarce. Figure 4. Shows the FTIR spectra of pure Magnesium oxide.. It is observed from the figure that the peaks from 410 cm⁻¹ to 700cm⁻¹ are due to metallic stretch. The peak at 1490 cm⁻¹ is due to C-N stretching of quinoid rings. It has predominant peaks at the wave number 3705cm⁻¹ and 3425 cm⁻¹ corresponds to O-H vibrations and it is formed due to water molecules.

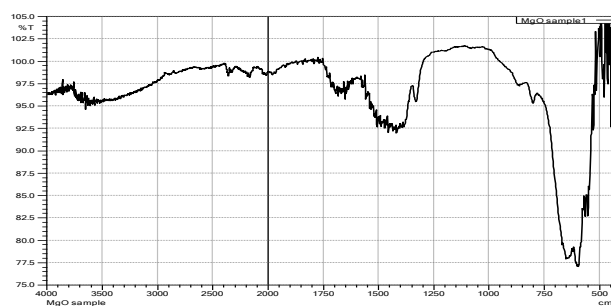


Fig. 4 . FTIR graph of MgO sample

3. Conclusions

MgO nanoparticles have been prepared by chemical precipitation method. The crystallite size of synthesized nanoparticles calcined at 600 °C for 4 hour and 6 hour was estimated by using Debye-Scherrer formula. The average crystallite size (19 nm) was observed to increase with increase of the calcinations temperature and duration. FTIR Spectra of the MgO nanoparticles claimed at 600 °C for 4 hrs and 6 hrs show presence of the IR peaks band at around 3399 cm^{-1} , 1467 cm^{-1} , 1058 cm^{-1} , 864 cm^{-1} and 668 cm^{-1} . These peaks are attributed to stretching mode of -OH group, adsorption of CO_2 and different Mg-O-Mg vibration mode of MgO nanoparticles respectively. The energy band gap of the calcined samples, determined by Tauc plot, was found decrease as the duration of calcination increases. It might be due to quantum confinement effect i.e. increase in the crystallite size, decreases the band gap. From absorption spectroscopy results, it has been found that firstly the absorbance decreases sharply with an increase in wavelength near the band edge (367 nm) indicating the formation of nanostructure samples and thereafter the value of absorption coefficient becomes more or less constant indicating the uniformity of size of synthesized nanoparticles.

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