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ORIGINAL ARTICLE

OPTICAL AND THERMO GRAVIMETRIC ANALYSIS ON METAL OXIDE NANOPARTICLES

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ABSTRACT

Metal Oxide nanomaterials are important and excellent materials, because of its special properties like chemical stability, high photocatalytic activity, high electrical permittivity and non toxic nature. ZnO, MgO and CaO nanoparticles were synthesized by using chemical precipitation method and it has been characterized for its optical properties by UV-Vis, to detect the presence of functional group by FTIR and phase formation, decomposition by TGA.
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Keywords: ZnO, MgO, CaO, Chemical Precipitation, UV-Vis, FTIR, TGA

1.INTRODUCTION

Nanotechnology is the technological innovations of the 21st century. We can greatly expand the range of performance of existing chemicals and materials by reducing the structure to a nanoscale. Materials reduced to the nanoscale can show different properties compared to what they exhibit on a macro scale, enabling unique applications. At this scale the surface to volume ratio of materials becomes large leading to unique properties [Albrecht et al., 2006]. Nanotechnology can provide unparalleled understanding about materials and devices and is likely to have impact in many fields. In recent years a number of investigations have focused on II-VI group metals. In particular, inorganic oxide nanomaterials like ZnO, MgO and CaO have shown potential as effective alternatives in ad- dressing some of the challenges [Stoimenov et al., 2002; Nasibulin et al., 2009; Zhang et al., 2012]. In particular, inorganic metal oxide nano material like CaO, ZnO and MgO are of particular interest because they are not only stable under harsh process conditions but also generally regarded as safe materials to human beings

In the present study we have reported the synthesis of the ZnO, MgO and CaO nanoparticles by chemical precipitation method and the optical and thermal properties of the synthesized nanoparticles were studied using Ultraviolet Visible Spectroscopy (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR) and Thermo Gravimetric Analysis (TGA).

2. EXPERIMENTAL PROCEDURE

ZnO, MgO and CaO nanoparticles were synthesized by chemical precipitation method using Zinc Chloride, Magnesium Nitrate Hexahydrate, Calcium Chloride and Sodium Hydroxide as the precursors. The solutions of the materials were made by dissolving them in the milliq water.

The precipitation was induced by drop wise addition of the sodium hydroxide into Zinc Chloride, Magnesium Nitrate Hexahydrate and Calcium Chloride solution under constant stirring for 2 hours. After the completion of the reaction, the solution was allowed to settle for overnight. Then the precipitate formed was washed several times with milliq water to remove the impurities formed during precipitation. Then the precipitate was separated and kept in the hot air oven for overnight to dry away the water. Then the obtained particles were grinded using mortar and pestle and kept for calcination at 450^0C for 3 hours. During this process the complete conversion of hydroxide into oxide takes place.

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3. RESULTS AND DISCUSSION

UV –VIS ANALYSIS

The UV-Vis reflectance spectroscopy is used to study the optical properties of the synthesized nanoparticles. ZnO nanoparticles were suspended in isopropanol and MgO, CaO nanoparticles were suspended in water and kept in ultrasonication for 5 min. The absorption spectra of ZnO, MgO, CaO nanoparticles was recorded using UV-Vis spectrophotometer and the UV-Vis spectrum is shown in Fig.1, Fig.2, Fig.3. From this spectrum, it has been inferred that the nanoparticles has sufficient transmission in the entire visible and IR region. The absorption peak of ZnO was found in the wavelength of 212.11 nm. For ZnO the absorption peak is expected at around 378 nm. Thus there is a strong blue shift in the absorption spectra of the ZnO sample indicating that particles must be smaller than the bohr radius of exciton which is for ZnO [Rupesh Kumar et al., 2013]. The absorption peak of MgO was found in the wavelength of 213.60 nm and the absorption peak of CaO was found in the wavelength of 212.11 nm.

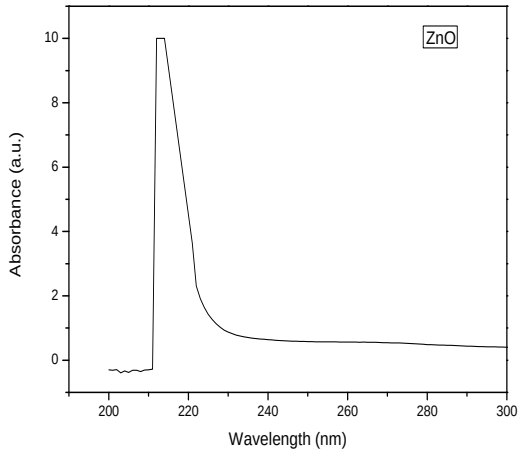

Fig.1. UV-Vis Spectrum of ZnO Nanoparticles

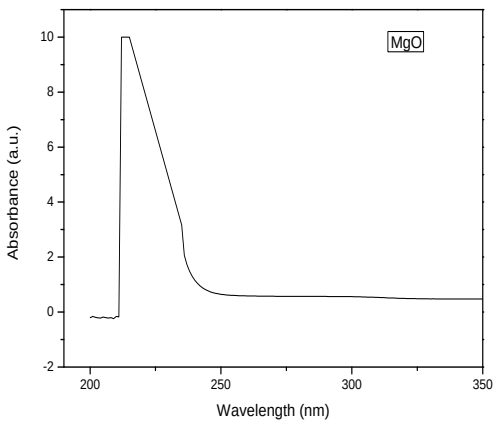

Fig.2. UV-Vis Spectrum of MgO Nanoparticles

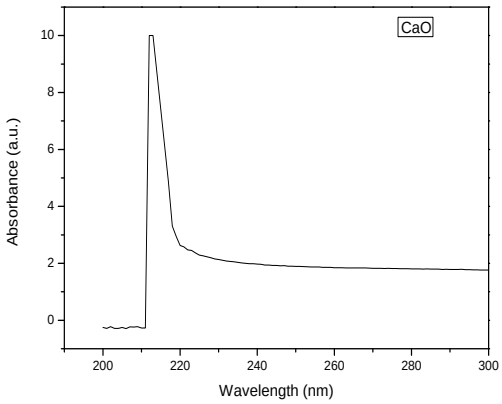

Fig.3. UV-Vis Spectrum of CaO Nanoparticles

FOURIER TRANSFORM INFRARED SPECTROSCOPY

Fourier transform infrared spectroscopy was used to detect the presence of functional groups adsorbed on the surface of synthesized nanoparticles during precipitation process. The FTIR spectrum of the synthesized ZnO nanoparticles was recorded in the range 500 - 4000 cm^{-1} using KBr pellets and the spectrum has been shown in Fig.4. The absorption peak at 3658.80 cm^{-1} corresponds to the O-H stretching vibration indicating the existence of hydroxyl group. The peak observed at 3514.31 cm^{-1} is assigned to O-H stretching indicating the presence of H-bonding. The peak observed at 2880.65 cm^{-1} is attributed to C-H stretching. The peaks absorbed at 1751.33 cm^{-1},1735.64 cm^{-1}, 1702.24 cm^{-1} correspond to C=O stretching vibration. The peaks absorbed at 1655.21 cm^{-1}, 1637.89 cm^{-1} is assigned to N-H bending vibrations. The peak observed at 1586.81 cm^{-1} is attributed to C-C stretching vibration. The peak observed at 1552.93 cm^{-1} corresponds to N-O asymmetric stretching vibration. The peak observed at 1468.84 cm^{-1}, 620 cm^{-1} is attributed to C-H bending vibrations. The peak observed at 1049.19 cm^{-1} is assigned to C-N stretching. The peak observed at 911.64 cm^{-1} is due to O-H bending vibration. The peak observed at 726.91 cm^{-1} corresponds to C-H rocking vibrations. The peak observed at 571.86 cm^{-1} is attributed to Zn-O stretching band.

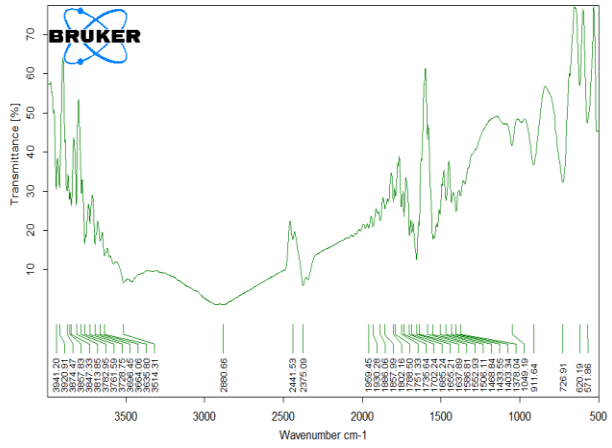

Fig.4. FTIR Spectrum of ZnO Nanoparticles

The FTIR spectrum of the synthesized MgO nanoparticles was recorded in the range 500 - 4000 cm^{-1} using KBr pellets and the spectrum has been shown in Fig.5. The absorption peak at 2922.90 cm^{-1} corresponds to the C-H stretching vibration. The absorption peak at 1741.60 cm^{-1} is attributed to C=O stretching vibrations. The absorption peak at 1676.01 cm^{-1} is assigned to –C=C- stretching vibrations. The absorption peak at 15334.39 cm^{-1}, 1516.27 cm^{-1} is due to C-C stretching vibrations. The absorption peak at 836.97 cm^{-1} corresponds to Mg-O vibrations.

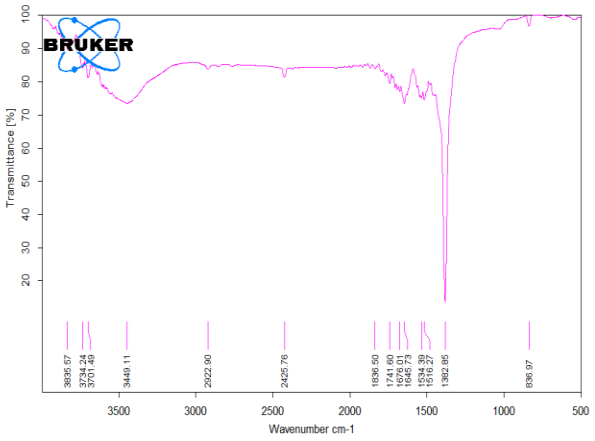

Fig.5. FTIR Spectrum of MgO Nanoparticles

The FTIR spectrum of the synthesized CaO nanoparticles was recorded in the range 500 - 4000 cm^{-1} using KBr pellets and the spectrum has been shown in Fig.6. The absorption peak at 3642.80 cm^{-1}, 3442.86 cm^{-1} corresponds to the O-H stretching vibration. The absorption peak at 2869.61 cm-1 is assigned to C-H stretching vibrations. The absorption peak at1742.18 cm-1, 1708.37 cm-1 is attributed to C=O stretching vibrations. The absorption peak at 1676.52 cm-1 is due to -C=C- stretching vibrations. The absorption peak at 1448.02 cm-1 is assigned to C-C stretching vibration. The absorption peak at 847.51 cm-1,713.40 cm-1 corresponds to C-Cl stretching vibration. The absorption peak at 587.36 cm-1 is attributed to Ca-O vibrations.

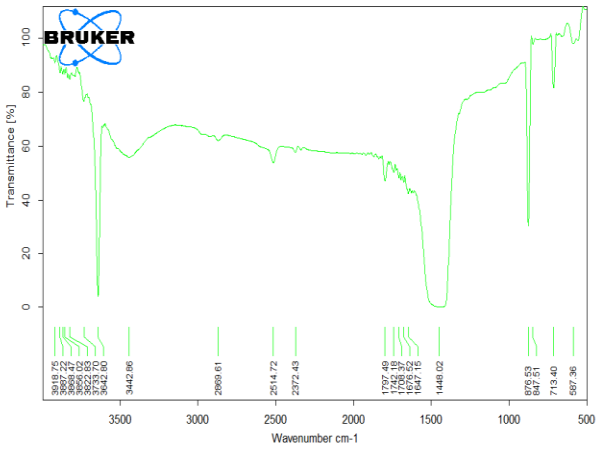

Fig.6. FTIR Spectrum of CaO Nanoparticles

THERMOGRAVIMETRIC ANALYSIS

Thermogravimetric analysis helps to know about the phase formation and decomposition of the sample that occurs during the heat treatment. The thermal analysis of the synthesized ZnO, MgO, CaO nanoparticles was carried out over the range 0^oC-1000^oC. The Fig.7 shows that for ZnO the mass loss takes place over the range 140^oC-210^oC. This loss of mass is attributed to the removal of surface adsorbed water from the precursor solution and the removal of organic species present in the sample. The phase transition occurs from 210^oC-610^oC.

Total mass of the sample kept for analysis = 6.4344 mg

Total mass decomposed according TG = 2.1336 mg

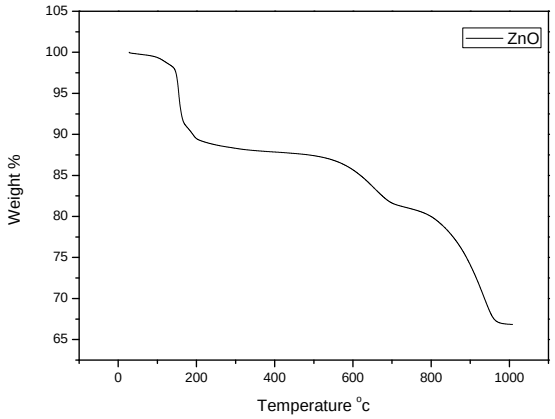

Fig.7. TGA Curve of ZnO Nanoparticles

For MgO nanoparticles the first mass loss takes place over the range 10^oC-140^oC and is shown in the Fig.8. This loss of mass is attributed to the removal of surface adsorbed water from the precursor solution. The second decomposition takes place over the range 270^oC-370^oC implies that the removal of organic species presents in the sample and the third decomposition of the sample take place over the range 470^0C-780^0C implies the removal of remaining organic species present in the sample. The phase transition occurs from 780^oC-1000^oC.

Total mass of the sample kept for analysis = 6.8373 mg

Total mass decomposed according TG = 3.8531 mg

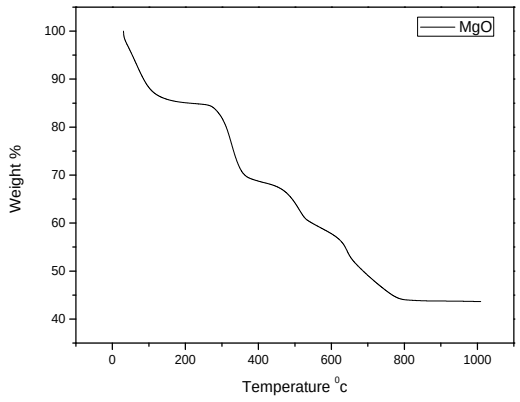

Fig.8. TGA Curve of MgO Nanoparticles

The thermal analysis of the synthesized CaO nanoparticles was carried out in the range 0^oC-1000^oC. The first mass loss takes place over the range 370^oC-430^oC and is shown in the Fig.9. This loss of mass is attributed to the removal of surface adsorbed water from the precursor solution. The second decomposition takes place over the range 570^oC-690^oC implies that the removal of organic species presents in the sample. The phase transition occurs from 690^oC-1000^oC.
Total mass of the sample kept for analysis = 3.4867 mg
Total mass decomposed according TG = 1.2282 mg

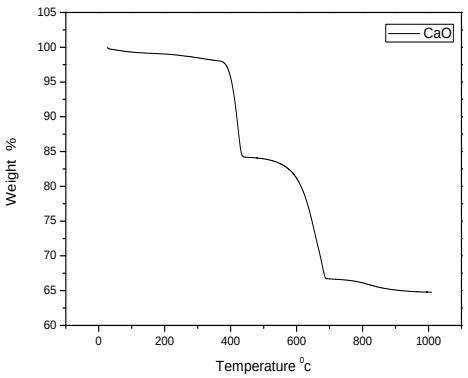

Fig.9. TGA Curve of CaO Nanoparticles

4. CONCLUSION

ZnO, MgO and CaO nano particles was synthesised by chemical precipitation method. The UV –Vis analysis shows that the excitonic peak of ZnO nano particles occurs at 212.75 nm which shows that there is a strong blue shift for the synthesized nano particles and for MgO, CaO nano particles the peak occurs at 213.60 nm,212.11 nm respectively. The FTIR spectrum shows the existence of functional groups in the synthesized ZnO MgO, CaO nano particles. The TGA results show that the phase formation occurs at 210^oC-610^oC for ZnO, 780^oC -1000^oC for MgO and 690^oC-1000^oC for CaO nano particles.

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