Int.J.Nano Dim. 2(3): 171-176, Winter 2012
ISSN: 2008-8868

Contents list available at **IJND**

International Journal of Nano Dimension

Journal homepage: *www.IJND.ir*

Synthesis and characterization of zinc oxide – agar nanocomposite

A. Jegan[1,3]
A. Ramasubbu[2]
K. Karunakaran[1]
S. Vasanthkumar[3,*]

[1]Department of Chemistry, Sona College of Technology, Salem, India.
[2]Post graduate and Research department of Chemistry, Government Arts College, Coimbatore, India.
[3] Director, School of Science and Humanities and Head of Department of Chemistry and Director i/c of School of Nano Sciences and technology, Karunya University, Coimbatore -641114. India.

Received: 18 February 2011
Accepted: 20 May 2011

* Corresponding author:
S.Vasanthkumar
Director, School of Science and Humanities and Head of Department of Chemistry and Director i/c of School of Nano Sciences and technology, Karunya University, Coimbatore -641114. India.
Tel +91 442429024
Fax +91 422-2615615
Email kumar2359@yahoo.com

ABSTRACT

A novel zinc oxide - agar nanocomposite was successfully synthesized using zinc chloride as a precursor. The nanocomposite was prepared using a domestic microwave oven. The prepared nanocomposites have been characterized by Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) and UV- visible spectroscopy. The crystalline properties of the synthesized materials were investigated by XRD and Raman spectroscopy. The UV- visible spectra shows a characteristic the absorption peak at 356 nm. The morphological observation of the SEM results reveals that the ZnO nanostructures are between 50 to 100 nm in size, and are embedded in the agar matrix.

Keywords: *Agar; Zinc Oxide; Embedded; Nanocomposite.*

INTRODUCTION

Zinc oxide is an important n-type semiconductor with a direct band gap of 3.37 eV. Zinc oxide nanoparticles are widely used in various applications such as optical devices [1], catalysis [2], light emitting diodes [3], photo detectors [4, 5], solar cells [6] and gas sensors [7]. Zinc is an essential nutrient in humans and animals for many physiological functions, including immune and antioxidant function, growth, Skeleton development, skin growth, appetite, wound healing and reproduction [8]. Zinc oxide (ZnO), a safe source for Zn supplementation and it is commonly used to fortify foodstuff in the food industry. ZnO will decompose into Zn ions after consumption [9]. A variety of methods have been used for the synthesis of zinc oxide nanoparticles such as direct precipitation [10], homogeneous precipitation [11], solvothermal method [12], sonochemical method [13], reverse micelles [14], sol gel method [15], hydrothermal [16], thermal decomposition [17], and microwave irradiation [18].

In recent times bionanocomposites are given great attention by of all researchers because they exhibit an enhancement in mechanical and thermal properties. Organic-inorganic hybrid materials, has a great potential to food industry application. Agar is a natural, biocompatible and biodegradable carbohydrate derived from marine algae. This is used as a gelling agent in food industry, impression materials in dental and salt bridges in electrochemistry. Agar is composed of Agarose and Agaropectin [19]. Previously L. Shi.et.al reported the preparation of ZnO-Pectin [20] and ZnO – whey protein [21] nanocomposites. However, review of literature, confirmed that the synthesis of Agar – ZnO nanocomposite has been unexplored, which aroused our interest in the present investigation. Herein we report the simple ecofriendly microwave assisted synthesis of Agar – ZnO nanocomposite using zinc chloride as a precursor, which can be used potentially in food industry. The nanocomposite was characterized by UV- visible spectroscopy; X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM), and Fourier transform infrared (FTIR) spectroscopy.

EXPERIMENTAL

Materials

$ZnCl_2$ and NaOH (Qualigens, India), Agar agar (Hi Media, India), and all other reagents were of Analar Grade. The Agar was washed with distilled water repeatedly to remove the impurities, if any. Doubly distilled water was used throughout the experimental studies.

Preparation of Zinc oxide – agar nanocomposite

ZnO – agar nanocomposite were synthesized by a microwave method. About 1 g of the purified Agar was dissolved in 100 ml of distilled water at 95ºC under magnetic stirring. After complete dissolution, 100ml of 0.1 M Zinc chloride solution was added in to the Agar solution drop wise. 0.1 M NaOH was prepared and added with stirring to zinc chloride agar mixture solution to the set pH of 10. The mixture was kept in a domestic microwave oven at 170 W for 25 minutes. The reaction was carried at 3 minutes interval. After the reaction a milky white composite was obtained which indicates the formation of ZnO nanoparticles. The composite was washed with distilled water repeatedly in order to remove the excess agar. The obtained sample was dried at 80ºC in an Oven.

Characterization of samples

Fourier transform infrared (FT-IR) analysis was performed using a Perkin Elmer spectrophotometer. X-ray powder diffraction (XRD) datas were collected using an X-ray diffractometer (Bruker, AXS) with Cu-Kα radiation (0.15406 nm).The surface morphologies of the particles were observed by a field-emission scanning electron microscope (SEM) (JEOL, JSM-6700F). UV–visible absorption spectra's were measured for the colloidal solution, which were prepared by dissolving the precipitates in distilled water and kept in a 1 cm optical path length quartz cuvette, using a UV-visible spectrophotometer (Elico BL 198 Biospectrophotometer) in the wave length of 200-900 nm under room temperature.

RESULTS AND DISCUSSION

FT-IR

Figure 1 shows the typical FTIR spectrum pattern of the ZnO -Agar nanocomposite in the range of 400–4000cm^{-1}. The characteristic FTIR spectra shows peaks at 873 and 929 cm^{-1}, due to 3, 6-anhydro - β- galactose skeletal bending in agar [22]. The peaks at 1060 and 1150 cm^{-1} corresponds to the ester-sulfate link vibrations and the broad band starting at 3500 cm^{-1} corresponds to the O-H bond. These bands are associated with Agar. The peak at 1630 cm^{-1} corresponds to O-H bending of absorbed water. The broad band between 400 and 600 cm^{-1} was attributed to the Zn-O stretching mode frequency [23]. All the peaks clearly illustrates that the ZnO particles are present in the agar matrix.

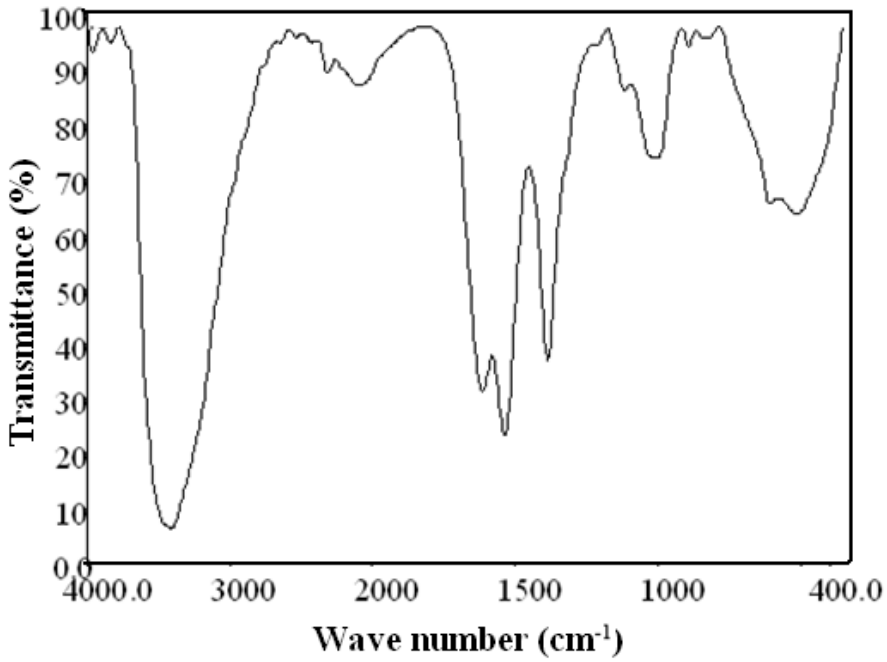

Fig.1. FT-IR spectra of the microwave assisted synthesized nanocomposite.

X- ray diffraction

Figure 2 shows X-ray diffraction pattern of ZnO- Agar nanocomposite. The XRD pattern indicates the face centered cubic structure of Zinc oxide nanoparticles. The presence of peaks at 2Θ values of 32.08, 34.74, 36.64, 48.04, 57.04,63.22,68.28 and 69.24 correspond to (100), (002), (101), (102), (110), (103), (200), (112) and (201) planes of Zinc oxide respectively. The strongest peak at 2 Θ = 36.64 belongs to the (101) plane of the products. No impurity peaks were detected in the products. The XRD results were in close agreement with the previous report [10].

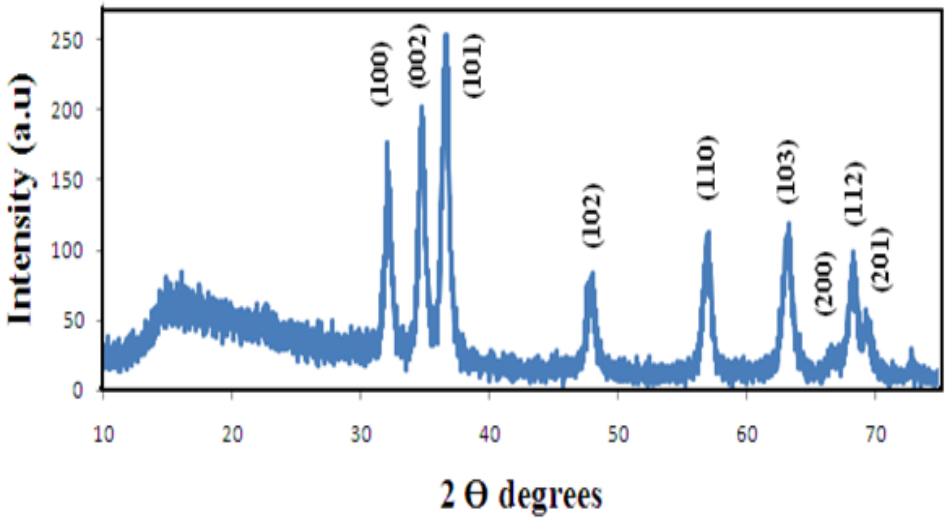

Fig. 2. X- ray diffraction of zinc oxide - agar nanocomposite.

Scanning electron microscope

The particle morphologies of the prepared ZnO - agar nanocomposite were observed by SEM. Figure 3 (a)-(d) show the SEM images of ZnO – agar nanocomposite at different magnifications. The SEM observation clearly illustrates that the Zinc oxide nanoparticles are formed in the agar matrix. Also it is to be noted that the nanoparticles varied in size from 50 nm – 100 nm. The low-magnification images demonstrate that the ZnO nanoparticles are dispersed in the agar matrix (Figure 3 (a) and (b)).

UV–visible absorption spectroscopy

The room temperature UV–vis absorption spectrum of the ZnO – agar nanocomposite was recorded in the wavelength range of 200–900 nm. Figure 4 represents the UV–vis absorption spectra of the ZnO – agar nanocomposite. The composite shows a broad adsorption band at 356 nm. The UV–vis absorption spectrum illustrates the nanocomposite which has a blue-shift (356 nm) compared to that of bulk ZnO (~370 nm), due to the quantum confinement effect.

The mechanism for the formation of ZnO – agar nanocomposite may be proposed as follows: The agar should be mixed with distilled water and heated at 95 ºC. During this heating, the agar is completely dissolved in the distilled water, and the Zn ions bind with the functional group of agar and form a complex. The Zn ions are converted to Zinc hydroxide with NaOH addition, while the pH is maintained around 10. After the microwave heating the ZnO is formed in the agar matrix. The natural polymer has a strong affinity for the oxide surface due to the binding of the carboxylate groups to sites along the oxide surface.

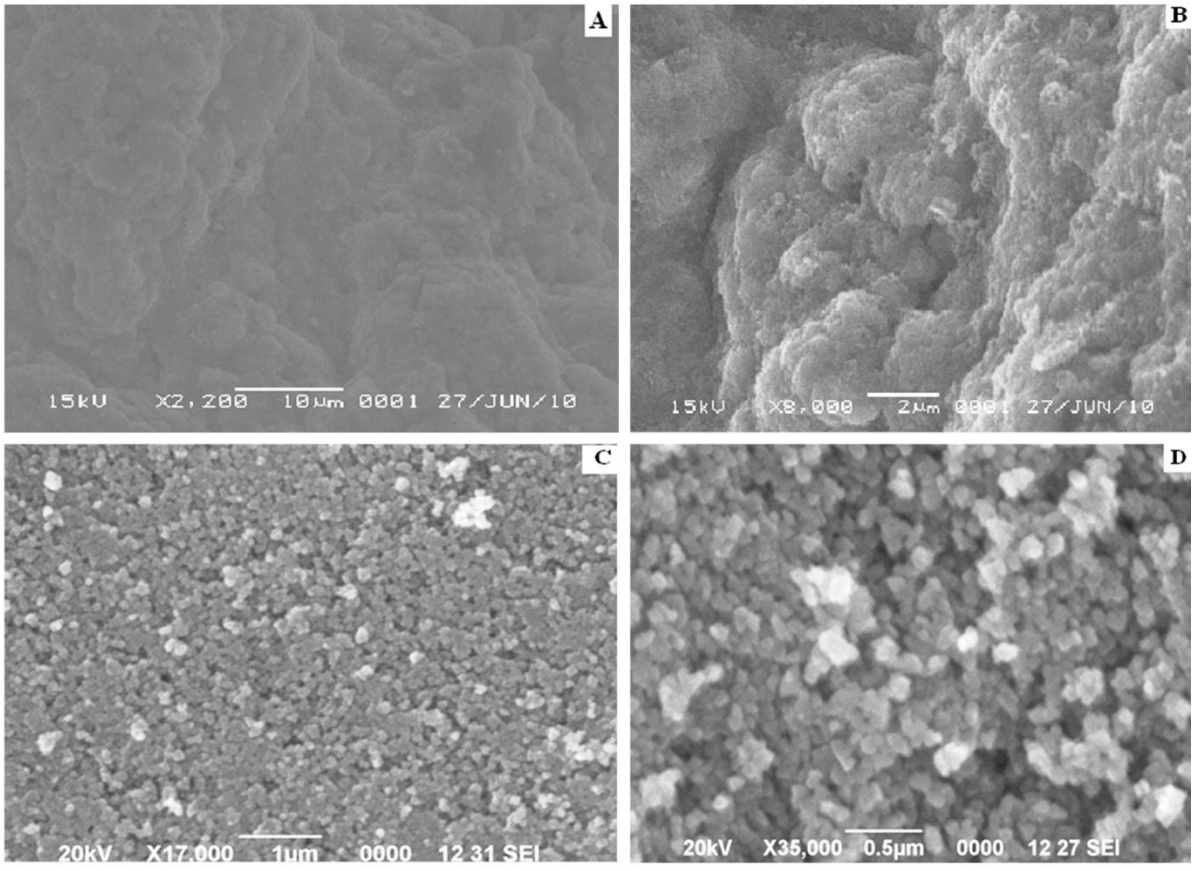

Fig. 3. SEM images of zinc oxide - agar nanocomposite at different magnifications.

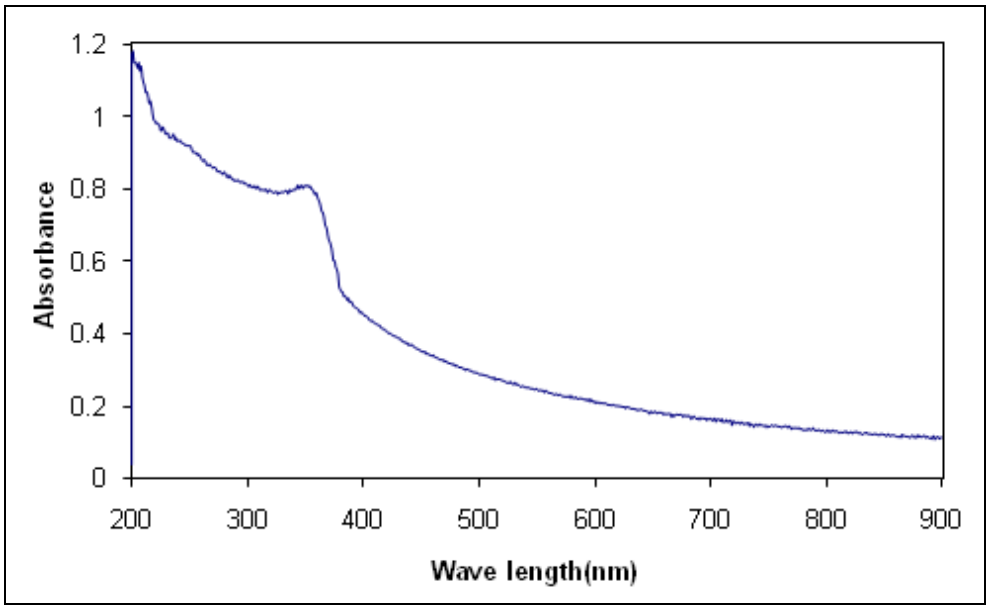

Fig. 4. UV- spectra of zinc oxide - agar nanocomposite.

CONCLUSION

We have successfully synthesized a novel agar – zinc oxide nanocomposite by using a simple domestic microwave oven. The microwave heating plays an important role in the formation of the nanocomposites. The XRD results confirm the presence of ZnO nanoparticles. The FT-IR results point to ZnO nanoparticles being distributed in agar matrix. The scanning electron microscope images confirm the size of ZnO nanoparticles, which varies from 50 to 100 nm. A great deal of potential applications in food industry can be expected for the ZnO – agar nanocomposite.

ACKNOWLEDGEMENTS

Dr. S.V.K. thanks the management and authorities of Karunya University for their support and encouragement. Dr. A.R., and Dr. S.V.K., thank the UGC, New Delhi, for providing the financial assistance as UGC – MRP. (F. No. 39 – 723 /2010 (SR)). The authors are thankful to Dr. S. Saravanan, Department of physics, Sona College of Technology, Salem for his assistance.

REFERENCES

[1] Ogata, K., Maejima, K., Fujita, S.Z., Fujita, S.G., (2001). ZnO growth toward optical devices by MOVPE using N_2O. *Journal of Electronic Materials*, 30: 659 - 661.

[2] Moghaddam, F. M., Saeidian, H., (2007). Controlled microwave-assisted synthesis of ZnO nanopowder and its catalytic activity for O-acylation of alcohol and phenol. *Materials Science and Engineering B*, 139: 265 - 269.

[3] Liu, C. H., Zapien, J. A., Yao, Y., Meng, X. M., Lee, C. S., Fan, S. S., et al., (2003). High-density, ordered ultraviolet light-emitting ZnO nanowire arrays. *Advanced materials*, 15: 838 - 841.

[4] Kind, H., Yan, H., Messer, B., Law, M., Yang, P., (2002). Nanowire ultraviolet photodetectors and optical switches. *Advanced materials*, 14: 158 - 160.

[5] Keem, K., Kim, H., Kim, G.T., Lee, J. S., Min, B., Cho, K., et al., (2004). Photocurrent in ZnO nanowires grown from Au electrodes. *Applied Physics Letters*, 84: 4376 – 4378.

[6] Beek, W. J. E., Wienk, M. M., Janssen, R.A. J., (2005). Hybrid polymer solar cells based on zinc oxide. *J. Mater. Chem.* 15: 2985 - 2988.

[7] Roy, S., Basu, S., (2002). Improved Zinc Oxide Film for Gas Sensor Applications. *Bull. Mater. Sci.*, 25:513 - 515.

[8] Shay, N. F., Mangian, H. F., (2000). Neurobiology of zinc-influenced eating behavior. *J. Nutrition.* 130: 1493S – 1499S.

[9] Hotz, C., Brown, K.M., (2004). Assessment of the risk of deficiency in populations and options for its control. *Food Nutr. Bull.* 25: S95 – S203.

[10] Perez-Lopez, O.W., Farias, A.C., Marcilio, N.R., Bueno, J.M.C., (2005). The catalytic behavior of zinc oxide prepared from various precursors and by different methods. *Mater. Res. Bull.* 40: 2089-2099.

[11] Kim, J.H., Choi, W.C., Kim, H.Y., Kang, Y., Park, Y.K., (2005). Preparation of mono-dispersed mixed metal oxide micro hollow spheres by homogeneous precipitation in a micro precipitator. *Powder Technol.*153: 166 - 175.

[12] Na Ayudhya, S. K., Tonto, P., Mekasuwandumrong, O., Pavarajarn, V., Praserthdam, P., (2006). Solvothermal synthesis of ZnO with various aspect ratios using organic solvents. *Cryst. Growth Des*, 6: 2446 – 2450.

[13] Kandjani, A. E., Tabriz, M. F., Pourabbas, B., (2008). Sonochemical synthesis of ZnO nanoparticles: The effect of temperature and sonication power. *Mater. Res. Bull.* 43: 645 - 654.

[14] Hirai, T., Asada, Y., (2005*).* Preparation of ZnO nanoparticles in a reverse micellar system and their photoluminescence properties. *J. Colloid Interf. Sci.* 284: 184 - 189.

[15] Ristic, M., Music, S., Ivanda, M., Popovic, S., (2005). Sol–gel synthesis and characterization of nanocrystalline ZnO powders. *J. Alloy Compd.*, 397: L1- L4.

[16] Liu Bin, Zengl Hua Chun. (2003). Hydrothermal Synthesis of ZnO Nanorods in the Diameter Regime of 50 nm. *J Am Chem Soc Commun*, 125; 4430 - 4431.

[17] Baskoutas, S., Giabouranis, P., Yannopoulos, S. N., Dracopoulos, V., Toth, L., Chrissanthopoulos, A., et al., (2007). Preparation of ZnO nanoparticles by thermal decomposition of zinc alginate. *Thin solid films*, 515: 8461 - 8464.

[18] Seungho Cho, Dae-Seob Shim, Seung-Ho Jung, Eugene Oh, Bo Ram Lee, Kun-Hong Lee, (2009). Fabrication of ZnO nanoneedle arrays by direct microwave irradiation. *Mater. Lett.* 63: 739 - 741.

[19] Marc Lahaye and Cyrille Rochas, (1991). Chemical structure and physico-chemical properties of agar. *Hydrobiologia*, 221: 137 - 148.

[20] Shi, L., Zhou, J., Gunasekaran, S., (2008). Low temperature fabrication of ZnO-whey protein isolate nanocomposite. *Mater. Lett.* 62, 4383 - 4385.

[21] Shi, L., Gunasekaran, S., (2008). Preparation of Pectin–ZnO Nanocomposite. *Nanoscale Res Lett.* 3: 491 - 495.

[22] Leonel Pereira, Ana Sousa, Helena Coelho, Ana M. Amado, Paulo J.A. Ribeiro-Claro, (2003). Use of FTIR, FT-Raman and 13C-NMR spectroscopy for identification of some seaweed phycocolloids. *Biomol. Eng.* 20: 223 - 228.

[23] Z. Jiang, Z. Huang, P. Yang, J. Chen, Y. Xin, J. Xu, (2008). High PL-efficiency ZnO nanocrystallites/PPV composite nanofibers. *Compos. Sci. Technol*. 68, 3240 - 3244.