Synthesis and Characterization of $Ca(OH)_2$ Nanoparticles in Different Media

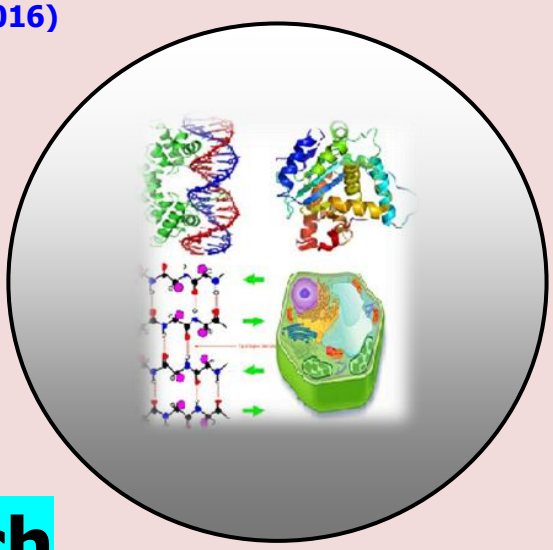
By

B. Sathya, K. Rajathi and S. Sridhar

ISSN 2319-3077 Online/Electronic
ISSN 0970-4973 Print

UGC Approved Journal No. 62923
MCI Validated Journal
Index Copernicus International Value
IC Value of Journal 82.43 Poland, Europe (2016)
Journal Impact Factor: 4.275
Global Impact factor of Journal: 0.876
Scientific Journals Impact Factor: 3.285
InfoBase Impact Factor: 3.66

J. Biol. Chem. Research
Volume 35 (2) 2018 Pages No. 877-882



Journal of Biological and Chemical Research

An International Peer Reviewed / Referred Journal of Life Sciences and Chemistry

Indexed, Abstracted and Cited in various International and National Scientific Databases

Published by Society for Advancement of Sciences®

J. Biol. Chem. Research. Vol. 35, No. 2: 877-882, 2018
(An International Peer Reviewed / Refereed Journal of Life Sciences and Chemistry)
Ms 36/01/13/2019

ISSN 2319-3077 (Online/Electronic)
ISSN 0970-4973 (Print)

Dr. B. Sathya
http:// www.sasjournals.com
http:// www.jbcr.co.in
jbiolchemres@gmail.com

RESEARCH PAPER

Received: 01/10/2018 **Revised: 10/12/2018** **Accepted: 11/12/2018**

Synthesis and Characterization of Ca $(OH)_2$ Nanoparticles in Different Media

B. Sathya, K. Rajathi and **S. Sridhar

Department of Chemistry, Government Arts College, Thiruvannamalai - 606603, Tamil Nadu, India
***Department of Botany, Government Arts College, Thiruvannamalai - 606603, Tamil Nadu, India**

ABSTRACT

Nanoparticles, of either simple or composite nature, display unique physical and chemical properties and represent an increasingly important material in the development of novel nano devices which can be used in numerous physical, biological, biomedical and pharmaceutical applications. The synthesis of uniform nanosized drug particles using green chemicals like ionic liquids with specific requirements in terms of size, shape and physical and chemical properties (especially non-toxic nature) is of great interest in the formulation of new pharmaceutical products. Room temperature ionic liquids (RTILs), a class of green and neoteric solvents have attracted increasing interest over recent years as environmentally benign excellent alternatives to organic solvents in homogeneous and biphasic processes synthesis of several nanoparticles. The use of plant extracts in the synthesis of nanoparticles have proven to be cost effective and opens up a wide area in non-toxic nanoparticle synthesis. In the present investigation, different medias were used for the synthesis of $Ca(OH)_2$ nanoparticles and the structures were confirmed by elemental and spectral studies such as FT-IR spectroscopy, X-Ray Diffraction (XRD) analysis, Scanning Electron Microscope (SEM) analysis, Energy Dispersive X ray specroscopy (EDX) and Transmission Electron Microscopy (TEM) analysis.

Key words: Green synthesis, Ionic Liquid, Plant Extract and Calcium Hydroxide nanoparticles.

INTRODUCTION

Nanotechnology has dynamically developed as an important field of modern research with potential effects in electronics and medicine (Glomm, 2005). Nanobiotechnology combines biological principles with physical and chemical procedures to generate nano-sized particles with specific functions. Nanobiotechnology represents an economic alternative for chemical and physical methods of nanoparticles formation. These methods of synthesis can be divided into intra cellular and extracellular (Ahmad et al., 2003). Due to the outbreak of the infectious diseases caused by different pathogenic bacteria and the development of antibiotic resistance, the pharmaceutical companies and the researchers are searching for new antibacterial agents (Rai *et al*., 2009). Ionic liquids have emerged as a class of material, having properties that make them green solvents which had found applications in a large number of organic transformations (Rajendran and Priyadarshini, 2010; Yung *et al*., 2011; Shelton *et al*., 2009). Even though, ionic liquids were initially introduced as an alternative green media because they are room temperature molten salts that are nonvolatile, thermally stable, recyclable, and easy to handle. Ionic liquids have high nucleation rates beyond showing their significant catalytic activities, electronic and steric stabilizer for many reactions and depress particle growth (Sahoo *et al*., 2006). The main

goal of this work was to investigate the role of different media (without media, ionic liquid media, plant extract media) for the formation, stabilization and morphology of Ca $(OH)_2$ nanoparticles.

MATERIALS AND METHODS

Synthesis of ionic liquid

Synthesis of 1, 1 Bis (3-Methylimidazolium -1-yl) Methane iodide

1-Methylimidazole (16.4 g, 0.2 mole) was mixed with CH_2I_2 (26.7g, 0.1 mole) in 100 ml of Toluene and refluxed for 2 hours (Qingbin et al, 2006). After the reaction was over as monitored on TLC, the mixture was cooled to room temperature and the solid product was separated by filtration, washed with toluene and dried in vacuum oven at 40^0C for 10 h.

^{1}H-NMR (400 MHz, $CDCl_3$): δ 9.51 (s, 2H), 7.92 (s, 2H), 7.79 (s, 2H), 4.29 (s, 2H), 3.88 (s, 6H).

^{13}C-NMR (75 MHz): δ = 151.87, 137.11, 123.99, 122.67, 48.25, 36.25, 26.41.

Synthesis of *Ormocarpum sennoides* (Elumbotti) aqueous leaves extract

About 100g of fresh leaves of *Ormocarpum sennoides* were shade dried and the leaves were powdered using kitchen blender. The powdered leaves were soaked in the 200ml of double distilled water for 48 hours and then filtered with Watmann No. 42 filter paper. The resulting extract was used as the plant extract solution.

Synthesis of Ca $(OH)_2$ Nanoparticles in different media

(i) Without media

In a typical synthesis procedure, calcium dihydrate (3.3g, 0.3mole) was dissolved in 25ml of deionised water under magnetic stirring at 90^0C. Then, sodium hydroxide (2.4g, 0.6mole) was dissolved in 25ml of water was slowly added into the solution of cadmium chloride under magnetic stirring at 90^0C. The solution was continuously stirred for 30 minutes. The formed colorless $Ca(OH)_2$ suspension was centrifuged to get the precipitate which was then washed several times with double distilled water to remove the basicity of the solution and dried in air oven for 24 hours for further characterization.

(ii) In Ionic liquid media

Calcium chloride dihydrate (3.3 g, 0.3mole) was dissolved in 15ml of deionized water and sodium hydroxide (2.4 g, 0.6 mole) was dissolved in 15ml of deionised water .Then ionic liquid (0.6mole) was dissolved in 20ml of deionized water was slowly added to the above solution. The solution was stirred for 30 minutes. The formed colorless $Ca(OH)_2$ suspension was centrifuged to get the precipitate which was then washed several times with double distilled water to remove the basicity of the solution and dried in air oven for 24 hours for further characterization.

(iii) In Plant extract media

Calcium chloride dihydrate (3.3 g, 0.3 mole) was dissolved in 15ml of deionised water, which was then added to 20ml of plant extract and 15ml of sodium hydroxide (2.4 g, 0.6mole) was added to the above solution under the magnetic stirring at 90^0C. The solution was continuously stirred for another 30 minutes. The formed colorless $Ca(OH)_2$ suspension was centrifuged to get the precipitate which was then washed several time with double distilled water to remove the basicity of solution and dried in air oven for 24 hours for further characterization.

CHARATERIZATION

The FT - IR spectra were obtained on a Varian 800 FT-IR as thin films. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded in $CDCl_3$ and DMSO-d_6 on a Joel JNN ECX 400P spectrometer. Nanoparticles were well characterized by FT - IR spectra, Powder X-ray Diffraction (powder XRD), Energy Dispersive X-ray Spectroscopy (EDX) and Transmission Electron Microscope (TEM). The phase, purity and crystalline size of the $Ca(OH)_2$ nanoparticles were studied by XRD. The X-ray diffraction (XRD) patterns were recorded on a Philips Xpert X-ray diffractometer with Cu Kα radiation (λ = 0.15406 nm) employing a scan rate of 1º/min in the 2θ range from 20^o to 80^o.Surface morphology, size and the distribution of particles were characterized by a LEO 1430VP Transmission Electron Microscope (TEM) using an accelerating voltage of 15 kV.

RESULTS AND DISCUSSION

FT-IR Spectral Comparison Studies

Table 1. Interpretation of FT-IR spectrum of $Ca(OH)_2$ nanoparticles synthesized by different media.

Peak Assignment	Frequencies of absorption bands, cm^{-1} of $Ca(OH)_2$ nanoparticle in absence of media	Frequencies of absorption bands, cm^{-1} of $Ca(OH)_2$ nanoparticle in ionic liquid media	Frequencies of absorption bands, cm^{-1} of $Ca(OH)_2$ nanoparticle in plant extract media
O-H stretching Vibration mode	3636	3640	3616
O-H bending vibration mode	1629	1638	1635
Asymmetric stretching vibration mode of C-O bond	1478, 1016	1497, 1055	1416, 1018
Symmetric deformation vibration mode C-O bond	873	872	865

The FTIR spectral data of $Ca(OH)_2$ nanoparticle synthesized in different medias are listed in table-1. Present findings showed similarity to the results previously reported (Liu et al., 2010; Blesa et al., 2003).

XRD analysis of $Ca(OH)_2$ nanoparticles

The phase, purity and crystallite size of the $Ca(OH)_2$ nanoparticles synthesized in different media were studied by XRD (Fig.1a,1b,1c). Based upon peak width, it can be observed that they are in agreement with the diffraction patterns of neat high crystalline $Ca(OH)_2$, with a hexagonal primitive structure (JCPDS card No. 84-1263). No intense peaks of other phases of $Ca(OH)_2$ and impurities were found. The sharp diffraction peaks manifest that the $Ca(OH)_2$ nanostructure possesses high crystallinity. On the whole, XRD results indicate that the products are crystalline in nature, high purity and free of impurities. From the XRD analysis, it is visible that, media does not play a major role for the determination of crystallite nature, but, it produces the remarkable change in the size of the nanoparticles. The crystallite size of the $Ca(OH)_2$ samples for without media , ionic liquid media and plant extract media was determined from the most intense diffraction peak (101) by using the Debye-Scherrer's equation was 30, 28 & 24 nm respectively.

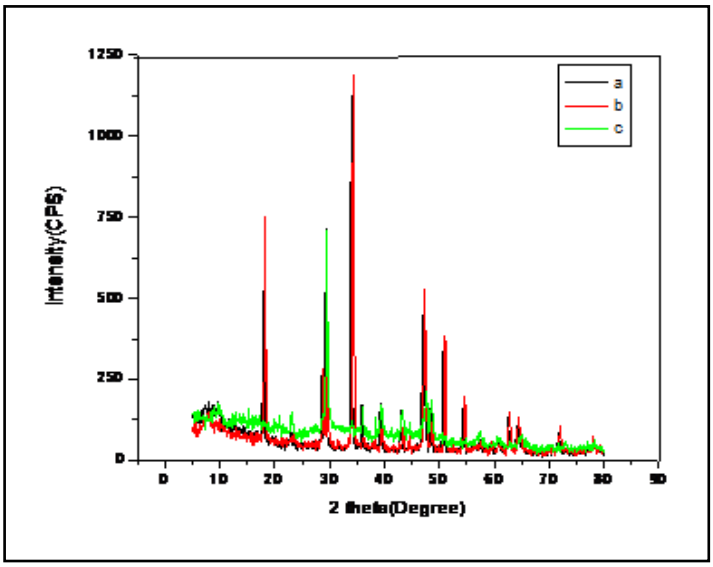


Figure 1. XRD pattern of: (a) $Ca(OH)_2$ nanoparticle without any media; (b) $Ca(OH)_2$ nanoparticle in ionic liquid media; (a) $Ca(OH)_2$ nanoparticle in plant extract media.

SEM analysis of $Ca(OH)_2$ nanoparticles

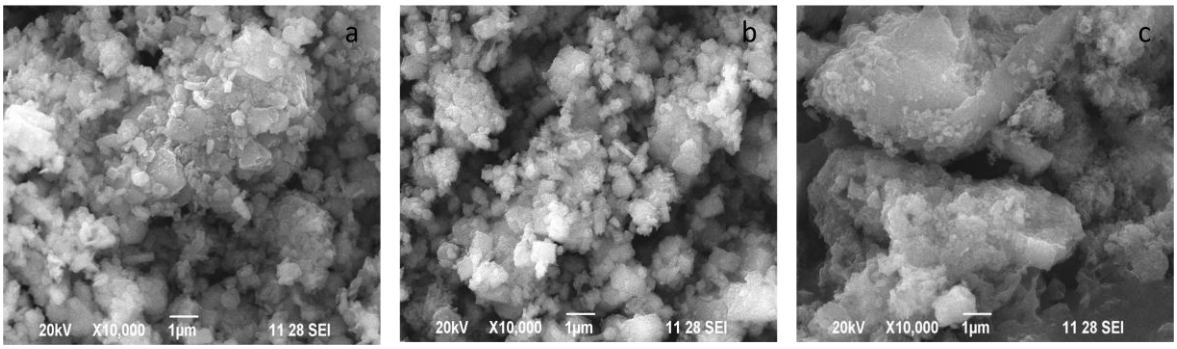


Figure 2. SEM images of: (a) Ca $(OH)_2$ nanoparticle without any media; (b) Ca $(OH)_2$ nanoparticle in ionic liquid media; (a) $Ca(OH)_2$ nanoparticle in plant extract media.

The morphology, structure and size of the samples are investigated by scanning electron microscope (SEM). The morphology of $Ca(OH)_2$ nanoparticles without and with media were investigated using SEM and presented in the figure 2a-c. The SEM results shown in figure 2a revealed granular stone shaped nanoparticle was obtained when the synthesis was performed in the absence of media. It is evident that structure of the nanoparticles produced in IL and plant extract media (Figure 2b-c), were flakes in shape.

EDX pattern of $Ca(OH)_2$ nanoparticles

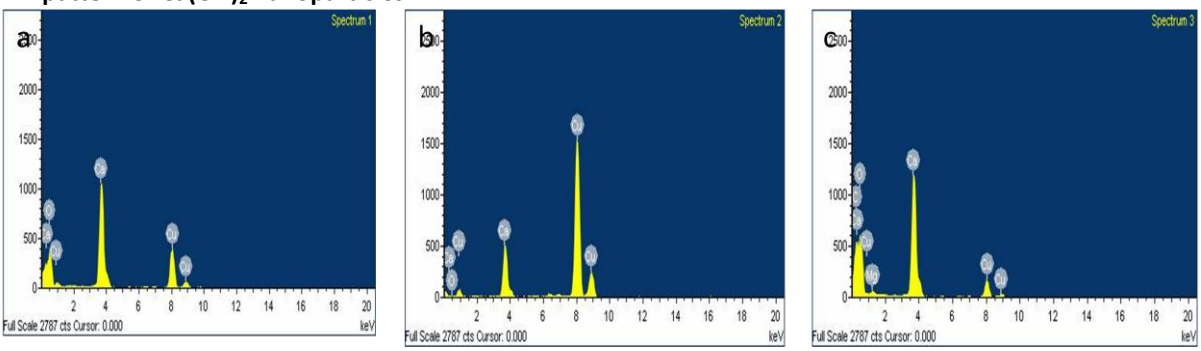


Figure 3. EDX patterns of: (a) $Ca(OH)_2$ nanoparticle without any media; (b) $Ca(OH)_2$ nanoparticle in ionic liquid media; (a) $Ca(OH)_2$ nanoparticle in plant extract media.

The purity and composition of the products (($Ca(OH)_2$ samples for without media , ionic liquid media and plant extract media) were studied by energy dispersive X-ray spectroscopy (EDX). The results are displayed in figures 3a-c. Only calcium and oxygen signals are observed in the spectral studies. The other peaks in the figures corresponded to gold, palladium, and silicate which were due to sputter coating of the glass substrate on the EDX stage and these were not considered in the elemental analysis of $Ca(OH)_2$nanoparticle. It implies that the nanoparticles are made up of only Ca, H and O. It is clear that the $Ca(OH)_2$ nanoparticles prepared were sufficiently pure.

TEM analysisof $Ca(OH)_2$ nanoparticles

The morphologies and dispersity of synthesized nano structured $Ca(OH)_2$ samples from different media were investigated by Transmission electron microscope (TEM). A typical transmission electron microscope (TEM) image of $Ca(OH)_2$nanoparticles derived from without any media is presented in figure 4a. Analysis of the micrographs indicated that the morphology of the nanoparticles was hexagonal shape with smooth surfaces. When the reaction was carried out in the presence of ionic liquid, the morphology of nanoparticles was turned as spherical shaped particles (Figure 4b). The TEM images of $Ca(OH)_2$ nanoparticles synthesized in plant extract media (Figure 4c) confirmed the capsule morphology of the nanoparticles. It is evident from the result that, the morphology of nanoparticles changes by changing the media. Presence of media plays an important role in the size as well as shapes of the nanoparticle formed.

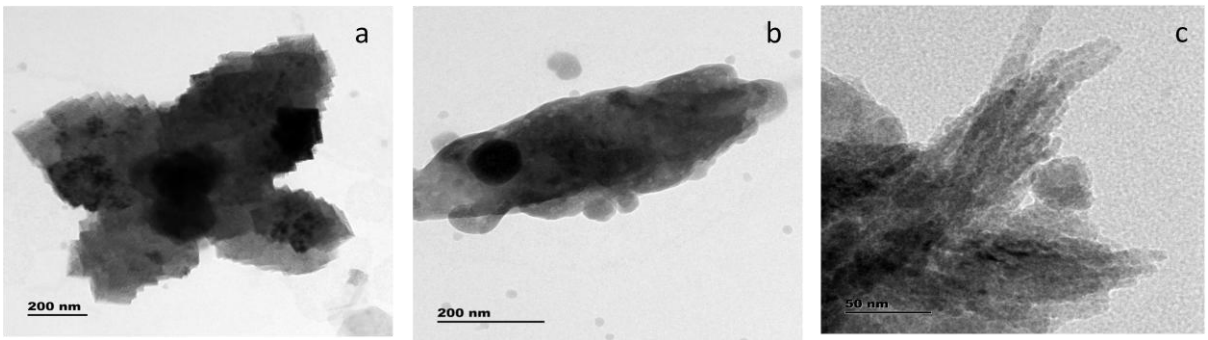


Figure 4. TEM images of: (a) $Ca(OH)_2$ nanoparticle without any media; (b) $Ca(OH)_2$ nanoparticle in ionic liquid media; (a) $Ca(OH)_2$ nanoparticle in plant extract media.

CONCLUSION

In the present work, an attempt was made to synthesize an imidazolium based ionic liquid called 1,1-Bis(3-methylimidazolium-1-yl) methane iodide. Under the given optimal conditions, synthesis of this ionic liquid gave excellent yield and the product was identified and characterized by FT-IR, ^{1}H-NMR and ^{13}C-NMR studies. Next the synthesis of nanostructured $Ca(OH)_2$ nanoparticles, in template solvents, imidazolium based ionic liquid, plant extract by simple chemical co-precipitation method was explored. Formation of nanoparticles was confirmed by FT-IR spectroscopy method. It was confirmed in all the three cases. The phase, purity and crystalline size of the $Ca(OH)_2$ nanoparticles were studied by XRD. In all the three methods, higher level of crystallinity and no contamination by other components were proved from the XRD and EDX analysis of synthesized $Ca(OH)_2$ nanoparticles. Comparison of the SEM images shows that the smallest and the flake shaped $Ca(OH)_2$ particles were obtained, when the synthesis of $Ca(OH)_2$ nanoparticles was carried out in the presence of ionic liquid and plant extract media. TEM observations of $Ca(OH)_2$ in absence of any media reveal the hexagonal shaped morphologies, although the TEM images of $Ca(OH)_2$ synthesized in ionic liquid and plant extract media exhibit the spherical and capsule shaped structures respectively. When the synthesis of $Ca(OH)_2$ nanoparticles was carried out in the absence of any media, the size of the particle was hexagonal. If the synthesis was carried out in the presence of some media the size of particle was constricted and the shape changed from hexagonal to spherical and capsule structures.

ACKNOWLEDGEMENTS

The authors thank the principal and the head of the department, Government Arts College, Thiruvannamalai for the constant encouragement given.

REFERENCES

Ahmad, A., Mukherjee, P., Senapati, S., Mandal, D., Khan, M.I., Kumar, R. and Sastry, M. (2003). Extracellular biosynthesis of silver nanoparticles using the fungus *fusarium oxysporum*. Colloids Surfaces, B: *Biointerfaces*; 27: 313-318.

Blesa, M.J., Miranda, J.L. and Moliner, R. (2003). Micro-FTIR study of the blend of humates with calcium hydroxide used to prepare smokeless fuel briquette, Vib. Spectrosc.; 33: 31-35.

Glomm, R.W. (2005). Functionalized Nanoparticles for Application in Biotechnology. Dispersion Sci. Technolog; 26: 389-314.

Liu, T., Zhu, Y., Zhang, X., Zhang, T. and Zhang, T. (2010). Synthesis and characterization of calcium hydroxide nanoparticles by hydrogen plasma-metal reaction method, Mater. Lett.; 64: 2575- 2577.

Qingbin, L., Fred van R. and Roger, A.S. (2006). Synthesis and application of dicationic ionic liquids. J. Chem. Technol. Biotechnol.; 81: 401-405.

Rai, M., Yadav, A. and Gade, A. (2009). Silver nanoparticles as a new generation of antimicrobials. Biotechnol. Adv.; 27: 76 - 83.

Rajendran, A. and Priyadarshini, M. (2010). Synthesis and characterization of a novel ionic liquid (TBA-AMPS) and its applications in Mannich condensation reactions under solvent free conditions. Afri. J. of Pure and Appl. Chem.; 4(9): 182-187.

Sahoo, S., Joseph, T. and Halligudi, S.B. (2000). Mannich reaction in Bronsted acidic ionic liquid: a facile synthesis of amino carbonyl compounds. J. of Molecular Catal. A: Chemical; 244: 179-182.

Shelton, R.A., Arends, I. and Hanefeld, U. (2009). Green Chemistry and Catalysis, Wiley-VCH, New York.

Yung, K.Y., Schadock-Hewitt, A.J., Hunter, N.P., Bright, F.V. and Baker, G.A. (2011). 'Liquid litmus': chemosensory pH-responsive photonic ionic liquids. Chem. Comm.; 47(16): 4775-4777.

Corresponding author: Dr. K. Rajathi, Department of Chemistry, Government Arts College, Thiruvannamalai - 606603, Tamil Nadu, India.
Email: rajathi_sridhar@rediffmail.com

Indexed, Abstracted and Cited in Indexed Copernicus International and 20 other databases of National and International repute