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Novel synthesis of silver nanoparticles using leaf ethanol extract of *Pisonia grandis* (R. Br)

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ABSTRACT

An effective and versatile technique was implemented for the synthesis of silver nanoparticles using the ethanolic extract of leaves of Pisonia grandis (R. Br). The silver nanoparticles were synthesized by three different methods and the completion of the reaction was monitored with UV-visible spectrophotometer. The XRD, Scherrer's equation and SEM analysis was employed for the characterization of synthesized silver nanoparticles. The particle size was found to be less than 150 nm, possessing spherical shape as confirmed from XRD and SEM analysis.

Key words: *Pisonia grandis,* XRD, Scherrer's equation, SEM.

INTRODUCTION

In recent years, noble metal nanoparticles have been the subject of focused research due to their unique optical, electronic, mechanical, magnetic, and chemical properties that are significantly different from those of bulk materials [1]. Nanotechnology is a field that is mushrooming, making an impact in all spheres of human life. A number of approaches are available for the synthesis of silver nanoparticles viz, reduction in solutions, chemical and photochemical reactions in reverse micelles, thermal decomposition of silver compounds, radiation assisted, electrochemical, sonochemical, microwave assisted process and recently via green chemistry route [2].

The biosynthetic method employing plant extracts [3] has received much attention recently owing to its simplicity, eco-friendliness and economically viable nature, compared to the other existing methods such as using bacteria and fungi [4] and the chemical [5] [6] and physical methods used for synthesis of metal nanoparticles. Understanding the biochemical processes that lead to the formation of nanoscale inorganic material is potentially appealing as an environment-friendly alternative to chemical methods [7].

Nanoscale materials have emerged as novel antimicrobial agents owing to their high surface area to volume ratio and the unique chemical and physical properties, which increases their contact with microbes and their ability to permeate cells [8]. Also, nanotechnology has amplified the effectiveness of silver particles as antimicrobial agents [9].

Silver is also the only material whose plasmon resonance can be tuned to any wavelength in the visible spectrum. Fundamental studies carried out in the last three decades show that silver nanoparticles exhibit a rare combination of valuable properties, namely, unique optical properties associated with the surface plasmon resonance (SPR), welldeveloped surfaces, catalytic activity, high electrical double layer capacitance, etc. Nanosilver has been used extensively as anti-bacterial agent in the health industry, food storage, textile coatings and a number of environmental applications [10].

The plant *Pisonia grandis* R.Br (*Nyctaginaceae*) synonym *Pisonia alba* is widely distributed throughout India and is a widespread evergreen commonly grown lettuce tree. Leaves, stem and root of this species are extensively used by the tribal's in the preparation of several folk medicines. It has been extensively used in Indian traditional medicine as an antidiabetic, anti-inflammatory agent, and used in the treatment of algesia, ulcer, dysentery and snake bite. The presence of pharmacologically important compounds like allantoin and pinitol in *Pisonia grandis* makes the plant more noteworthy in the field of nanochemistry also. The process of isolation of the above compounds from leaves of Pisonia grandis has been reported from this laboratory [11 & 12] (Patent pending no.385/CHE/2010 and 3606/CHE/2010).

The present study was aimed at the synthesis of silver nanoparticles using the ethanolic extract of leaves of *Pisonia grandis* under three different experimental conditions *viz* i) room temperature ii) higher temperature and iii) sonication. The particle size of the synthesized silver nanoparticles was characterized by XRD and SEM analysis.

MATERIALS AND METHODS

1.1. Plant collection
Leaves of fresh plant were collected from the residential areas of Coimbatore.

1.2. Preparation of the plant extract
The ethanolic leaf extract of *Pisonia grandis* (200mg) was weighed and taken in a 250 ml beaker and 100 ml of Millipore water was added to it. The solution was sonicated using ultrasonic bath (PCI Ultrasonics 1.5 L (H)) for 15 mins to disperse the extract in water. The solution was filtered thrice using Whatman filter paper to get a clear solution.

1.3. Synthesis of silver nanoparticles
The silver nanoparticles were synthesized using a constant volume of the plant extract under various experimental conditions *viz*, room temperature (28 - 30°C), higher temperature (90°C) and sonication using ultrasonic bath (PCI Ultrasonics 1.5 L (H)) with different volumes of 3mM silver nitrate solution. The appearance of reddish brown colour after 3 h indicates the formation of silver nanoparticles. The completion of the reaction was monitored by UV-visible spectroscopy.

1.4. Separation of silver nanoparticles
The synthesized silver nanoparticles were separated by centrifuging (Spectrofuge 7M) at 13,000 rpm for 15 mins. The process was repeated by dispersion of pellets in water, to obtain coloured supernatant solutions. The sample was then stored at -4°C for further use.

1.5. Characterization of silver nanoparticles
a)UV-visible spectroscopy

The formation and completion of silver nanoparticles was characterized by UV-visible spectroscopy using a Double beam spectrophotometer 2202- (SYSTRONICS).

b)XRD analysis

A drop coated film of synthesized silver nanoparticles was characterized using SHIMADZU Lab X XRD-6000 with a Cu Kα radiation monochromatic filter in the range 10–80°. Debye-Scherrer's equation was used to determine the particle size of the silver nanoparticles from the 2θ values of the X-ray diffraction peaks is given below

Debye-Scherrer's equation:

$D = k \lambda / \beta \cos \theta$

K – constant, λ – wavelength of the X-ray, β – full width half maximum of the XRD peak (radians), θ - Bragg's angle of the XRD peak.

c) Scanning Electron Microscopy
The synthesized AgNP's were fabricated on a glass substrate. The morphology and size of silver nanoparticles was investigated using TESCAN make Scanning Electron Microscope provided with Vega TC software. Further Secondary electron sputtering at an applied potential of 20 kV was adopted prior to recording SEM.

RESULTS AND DISCUSSION

The silver nanoparticles synthesized with different volumes of silver nitrate for a constant volume (5ml) of leaf ethanol extract of *Pisonia grandis* under various experimental conditions responded to the following analyses.

1.6. UV- Visible spectroscopy
Reduction of silver ions into silver nanoparticles using ethanolic extract of leaves of *Pisonia grandis* was evidenced by the visual change of colour from yellow to reddish brown due to excitation of surface plasmon vibrations [13] in silver nanoparticles as shown in figure 1. The UV-visible spectra show an absorption band at 421 nm which corresponds to the absorbance of silver nanoparticles (figure 2). After 3 hrs, no significant colour change was observed. Increased concentrations of silver nitrate resulted in a brown solution of nanosilver indicating the completion of reaction. Comparative study on different methods of synthesis revealed that the sonication method of production of the AgNP's was efficient and was complete in 30 mins (fig 3). Also, uniformity in the shape of synthesized silver nanoparticles was noted in sonication due to the acceleration effect in chemical dynamics and rates of the reactions. Ultrasonic energy might have changed the chemical route of synthesized particles by generating free radicals [14].

Fig 1. Photograph of AgNP's of varying concentrations of silver and leaf ethanol extract of *Pisonia grandis*

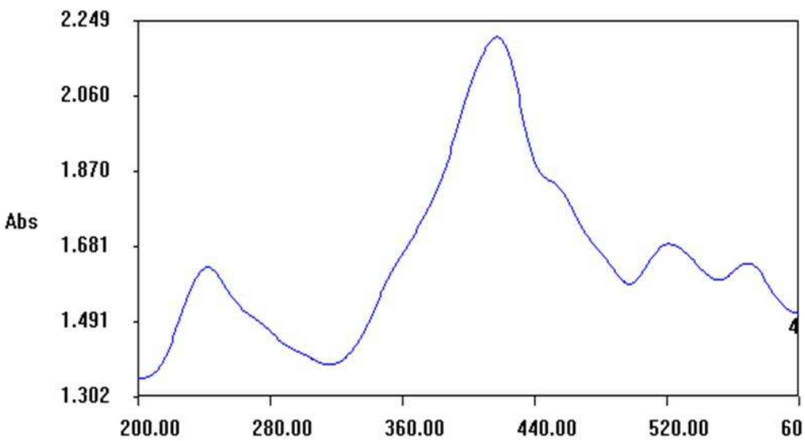


Fig 2. UV-visible spectra of synthesized silver nanoparticles using the leaf ethanol extract of *Pisonia grandis*

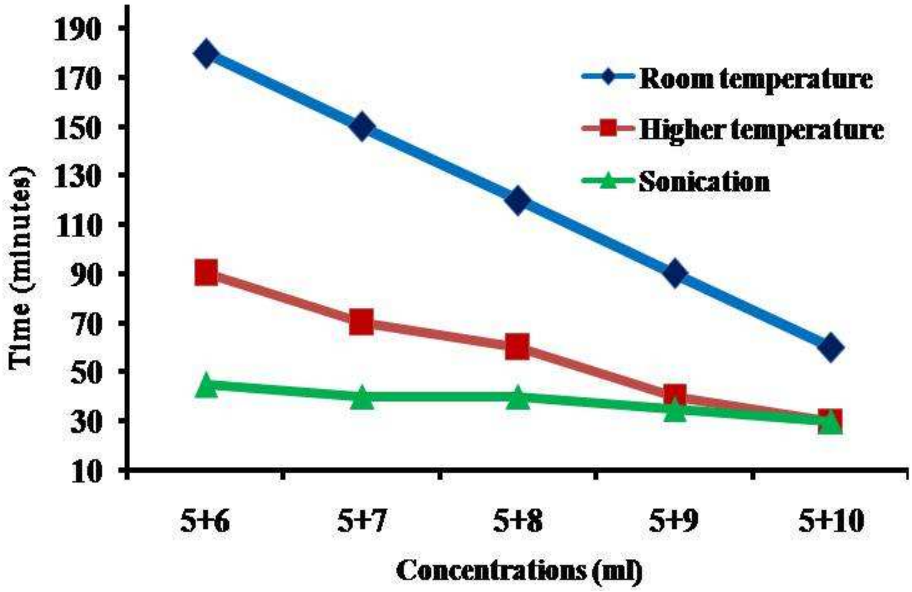
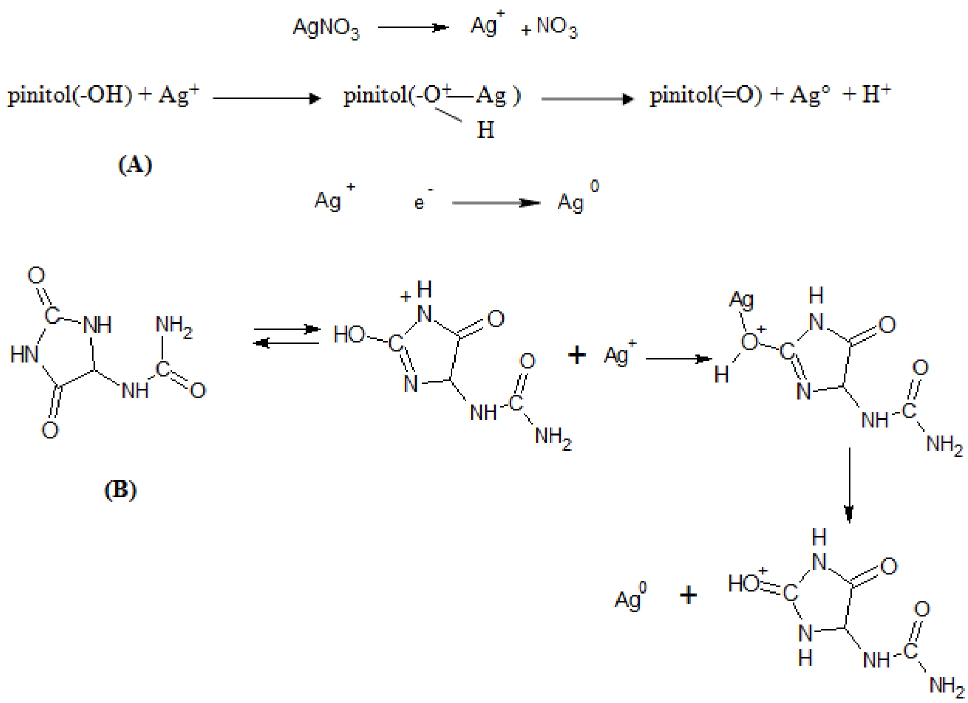


Fig 3. Comparison of different experimental conditions for the synthesis of silver nanoparticles

1.7. Mechanism of reduction of silver to AgNP's

Formation of AgNP's was noted over a time period of 30 minutes to three hours for the various concentrations. Sinorhizobial octasaccharide [15], geraniol [16], glutathione [17], curcacycline A and curcacycline B [18], polyols [19], polyphenols, glutathiones, metallothioneins, and ascorbates [20] are known to reduce silver producing silver nanoparticles. The presence of metabolites like pinitol and allantoin in *Pisonia grandis* is reported [11 & 12]. A probable method of reduction mechanism of pinitol and allantoin with silver nitrate producing silver nanoparticles is given in scheme 1.



Scheme 1. Probable mechanism of reduction silver to silver nanoparticles by the bioactive molecules: pinitol (A) and allantoin (B)

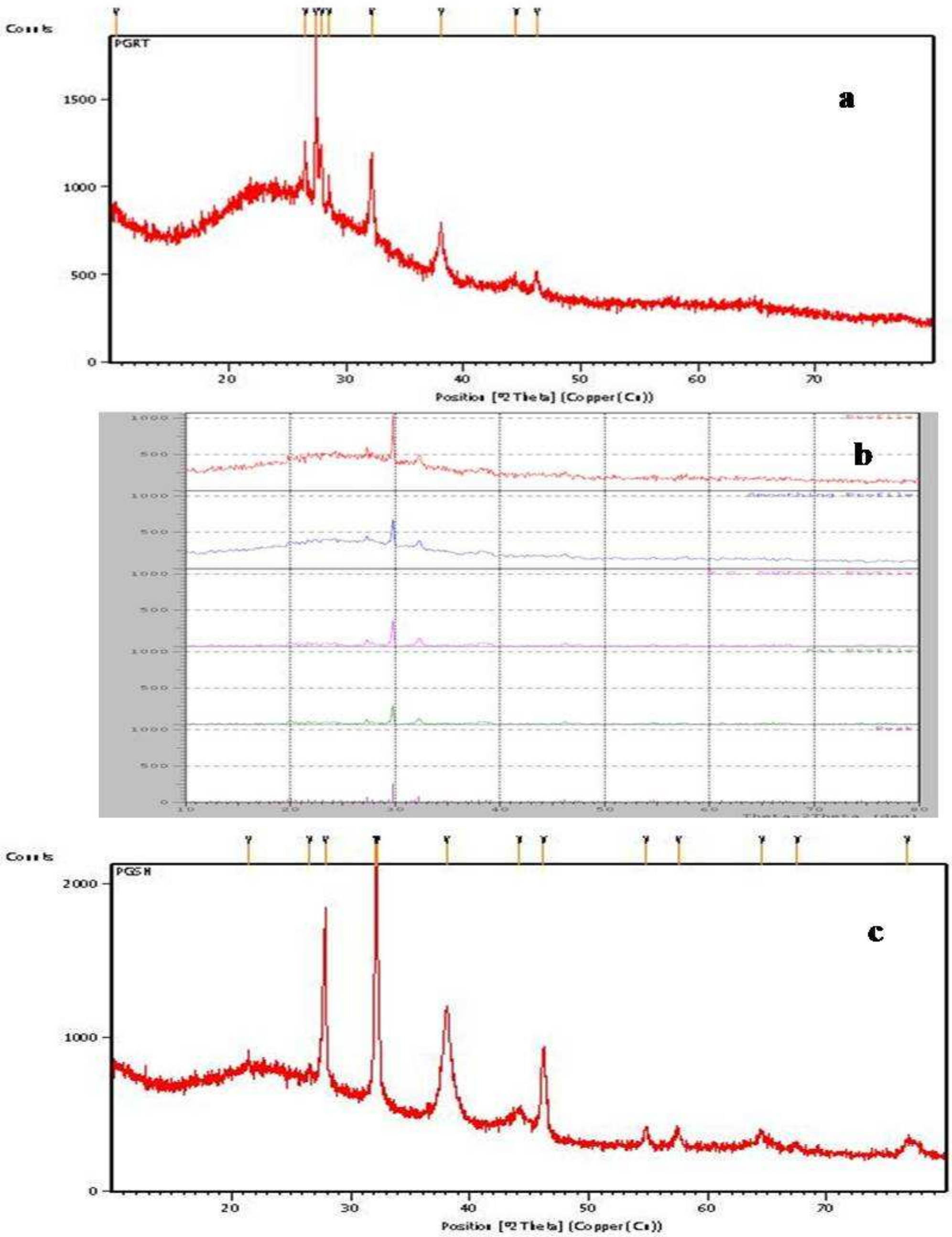


Fig 4. XRD patterns of synthesized silver nanoparticles using the ethanolic extract of leaves of Pisonia grandis at (a) room temperature, (b) higher temperature and (c) sonication

1.8. XRD analysis

The particle size of the synthesized silver nanoparticles was characterized by the XRD analysis. The XRD patterns of the synthesized silver nanoparticles using the ethanolic extract of leaves of *Pisonia grandis* is shown in figure 3. The X-ray diffraction peaks at 2θ = 38.08°, and 44.47° indexed as 111, 200 planes of FCC of silver [21] at room temperature conditions are shown in figure 4a.

In higher temperature conditions (90°C) (fig 4b), the peak appearing at 32° is assigned to 101 facets of silver [22] whereas; for sonication (fig 4c), three intense and sharp peaks at 2θ = 32.23°, 38.08°, 44.18° and 64.51° were obtained corresponding to the 101, 111, 200 and 220 planes of Bragg's reflection of silver respectively [23]. The particle size of the synthesized silver nanoparticles calculated using Debye-Scherrer's equation is listed in table. 1.

Table. 1. Determination of crystalline size of AgNP's by using Debye-Scherrer's equation - Experimental conditions

S.No	2θ (degrees)	FWHM (degrees)	β = π*FWHM/180 (radians)	D = k λ / β. Cosθ (nm)
Room temperature				
1.	32.16	0.1171	0.0020	70.68
2.	38.08	0.1338	0.0023	62.96
3.	44.47	0.8029	0.0140	36.96
Higher temperature				
4.	32.27	0.4500	0.0078	29.14
Sonication				
5.	32.23	0.1004	0.0017	82.45
6.	38.08	0.3011	0.0052	27.94
7.	44.18	0.3011	0.0052	12.90
8.	64.51	0.2676	0.0046	35.19

The average particle size of the synthesized silver nanoparticles are 56.86 nm, 29.14 nm and 39.62 nm obtained for room temperature, higher temperature and sonication respectively. The study shows that smaller silver nanoparticles can be obtained at elevated temperatures.

1.9. SEM analysis

Scanning electron microscopy has provided further insight into the morphology and size details of the synthesized nanoparticles. SEM micrographs of the synthesized silver nanoparticles using the ethanolic extract of leaves of *Pisonia grandis* fabricated on a glass substrate are shown in figure 4. The synthesized silver nanoparticles were well dispersed without aggregation, possessing spherical shape. The particle size was found to less than 150 nm in all three experimental conditions.

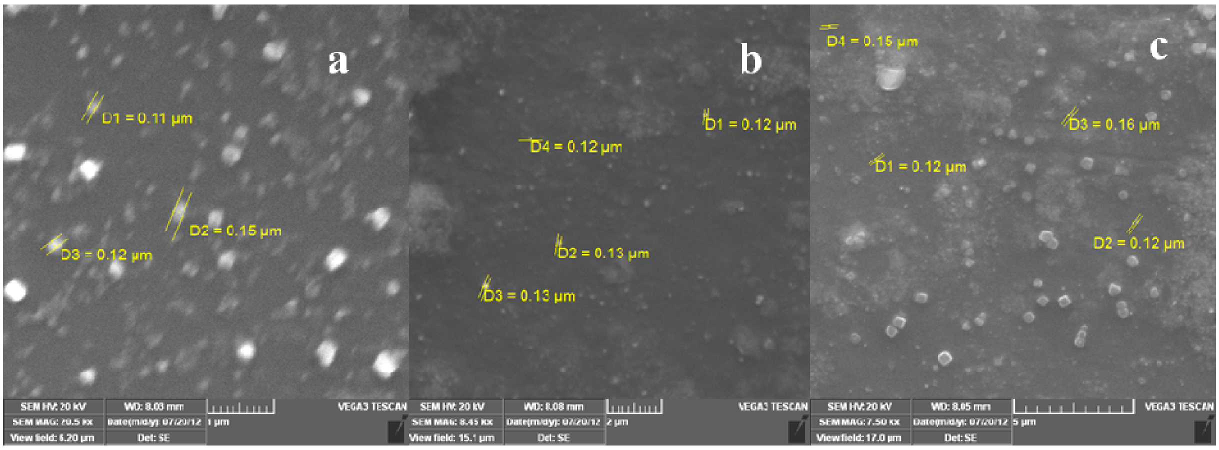


Fig 4. SEM micrographs of synthesized silver nanoparticles using the ethanolic extract of leaves of *Pisonia grandis* at (a) room temperature, (b) higher temperature and (c) sonication

CONCLUSION

The reduction of silver into silver nanoparticles using the ethanol extract of leaves of *Pisonia grandis* was carried out under room temperature, elevated temperatures and under sonication condition. Among these methods of synthesis, sonication proves to be efficient, rapid and inculcates green chemistry. The particle size of the synthesized silver nanoparticles was found to be 20-150 nm possessing spherical shape. The XRD and SEM results confirmed that the morphology of synthesized AgNP's is analogous in three experimental conditions. Hence the complimentary properties of silver nanoparticles obtained from *Pisonia grandis* can unfasten newer pathways in drug development. Studies on the medicinal properties of the AgNP's enriched extracts of *Pisonia grandis* are underway in our laboratory (Patent pending no. 85/CHE/2012).

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REFERENCES

[1] A. J. Gavhane, P. Padmanabhan, S. P. Kamble and S. N. Jangle, *Int J Pharm Bio Sci*., **2012**, 3(3), 88 – 100.
[2] V.Manonmani, Vimala Juliet, International Conference on Innovation, Management and Service, IPEDR, IACSIT Press. Singapore. 14, **2011.**
[3] T. Dhanalakshmi and S. Rajendran, *Archives of Applied Science Research*, **2012**, 4 (3), 1289-1293.
[4] G. Oza, S. Pandey, R. Shah, M. Sharon, *Advances in Applied Science Research*, **2012**, 3 (3), 1776-1783.
[5] A. A. El-Kheshen and S. F. Gad El-Rab, *Der Pharma Chemica*, **2012**, 4 (1), 53-65.
[6] H. U. Igwe, E. I. Ugwu, *Advances in Applied Science Research*, **2010**, 1 (3), 240-246.
[7] S. R. Bonde, D. P. Rathod, A. P. Ingle, R. B. Ade, A. K. Gade and M. K. Rai, *Nanoscience Methods*, **2012**, 1, 25–36.
[8] K. Lamsal, S. W. Kim, J. H. Jung, Y. S. Kim, K. S. Kim And Y. S. Lee, *Mycobiology*, **2011**, 39(3), 194-199.
[9] L. Christensen, S. Vivekanandhan, M. Misra, A. K. Mohanty, *Adv. Mat. Lett*., **2011**, 2(6), 429-434.
[10] J. E. Mary and L. Inbathamizh, *Asian J Pharm Clin Res.*, **2012**, 5(1), 159-162.
[11] K.S. Shubashini, G. Poongothai and P. Lalitha, *International Journal of Pharmacy and Life Sciences*, **2011**, 2(6), 815-817.
[12] K.S. Shubashini, G. Poongothai and P. Lalitha, *Journal of Natural Remedies*, **2011**, 11(1), 39-43.
[13] A. Power, J. Cassidy, T. Betts, *The Analyst*, **2011**, 136, 2794-2801.
[14] A. Hassanjani-Roshana, S. M. Kazemzadeha, M. R. Vaezi and A. Shokuhfar, *Journal of Ceramic Processing Research*, **2011**, 12(3), 299-303.
[15] C. Kwon, B. Park, H. Kim, and S. Jung, *Bull. Korean Chem. Soc.*, **2009**, 30(7), 1651-1654.
[16] M. Safaepour, A. R. Shahverdi, H. R. Shahverdi, M. R. Khorramizadeh, and A. R. Gohari, *Avicenna J Med Biotech.*, **2009**, 1(2), 111-115.
[17] B. Baruwati, V.Polshettiwar and R. S. Varma, *Green Chem.*, **2009**, 11, 926–930.
[18] H. Bar, D. Kr. Bhui, G. P. Sahoo, P. Sarkar, S. P. De, A. Misra, *Colloids and Surfaces A: Physicochem. Eng. Aspects*., **2009**, 339, 134–139.
[19] D. Jain, H. Kumar Daima, S. Kachhwaha, S. L. Kothari, *Digest Journal of Nanomaterials and Biostructures*, **2009**, 4(3), 557 – 563.
[20] A. K. Jha and K. Prasad, *International Journal of Green Nanotechnology: Physics and Chemistry*, **2010**, 1, 110–117.
[21] N. Vigneshwaran, A. A. Kathe, P. V. Varadarajan, R. P. Nachane, and R. H. Balasubramanya, *Langmuir*, **2007**, 23, 7113-7117.
[22] R. Varshney, A. N. Mishra, S. Bhadauria, M. S. Gaur, *Digest Journal of Nanomaterials and Biostructures*, **2009**, 4(2), 349 – 355.
[23] R. Sarkar, P. Kumbhakar, A. K. Mitra, *Digest Journal of Nanomaterials and Biostructures*, **2010**, 5(2), 491 – 496.