www.sphinxsai.com

ChemTech

International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.7, No.2, pp 734-739, 2014-2015

ICONN 2015 [4th -6th Feb 2015]
International Conference on Nanoscience and Nanotechnology-2015
SRM University, Chennai, India

Biological synthesis of palladium nanoparticles using leaf extract of *Sebastiania chamaelea*(L.) Muell. Arg.

Malathi.R[1] and Ganesan. V*[1]

[1]Centre for Research and PG Studies in Botany, Ayya Nadar Janaki Ammal College (Autonomous),Sivakasi- 626 124, Tamil Nadu, India.

Abstract : We report the biological synthesis of palladium nanoparticles with the help of *Sebastiania chamaelea*(L.) Muell. Arg. (Family: *Euphorbiaceae*) leaf. Aqueous leaf extract of *Sebastiania chamaelea* was employed for the bioreduction of Pd^{2+} ions to Pd^0. The leaf was collected from the campus of Ayya Nadar Janaki Ammal College, Sivakasi, Tamil Nadu. 10ml leaf extract was prepared, resuspended in 90ml of palladium chloride solution and it is known as reaction medium. The colour change of the reaction medium from pale yellow to dark brown during the incubation period is due to the vibrations in Surface Plasmon Resonance (SPR). It indicates the formation of palladium nanoparticles. From this reaction medium, a small aliquot of the sample was used for the characterization of palladium nanoparticles through UV-Visible (UV-Vis) Spectroscopic analysis, Fourier Transform Infra Red (FTIR) Spectral analysis, X- Ray Diffraction (XRD) analysis and Scanning Electron Microscope (SEM) with Energy Dispersive X-ray (EDX) analysis.The SPR with λ max at 475nm and the absorbance was raised up to 0.78a.u. The FTIR analysis explains that the biomolecules responsible for the stability of palladium nanoparticles that are synthesized by the leaf broth. The XRD analysis gives the structural information of nanoparticles. The SEM and EDAX analyses confirmed the significant presence of palladium nanoparticles. The size of the particle ranged from 50 to 80 nm. Thus the synthesis of palladium nanoparticles with various sizes is achieved using the leaf broth of *Sebastiania chamaelea*, as a green route due to its eco-friendly nature and does not involve any toxic methods and chemical in the synthesis of palladium nanoparticles.
Key words: *Sebastiania chamaelea*, leaf broth, reaction medium, eco friendly synthesis, palladium nanoparicles.

Introduction

Nature has many natural stabilizing and capping agents in the form of biomolecules that can produce the stabilized nanoparticles. Biological sources such as plants and microbes have these biomolecules as secondary metabolites, proteins and enzymes. This served as an inspiration for biological synthesis of metal nanoparticles[1]. Recently, the researchers concentrate this type of synthesizing nanoparticles synthesis because this method involves in the utility of environmentally benign solvents and non-toxic chemicals[2,3]. This is also alternative to physical and chemical methods. Different types of metal nanoparticles such as silver, gold, platinum and palladium were obtained using plants and microbes[4].These metal nanoparticles have been used in many applications in different field. Among them, palladium nanoparticles have unique properties and assorted applications[4]. Recently, we demonstrated the eco-friendly synthesis of silver nanoparticles using leaves of *Sebastianiachamaelea*[5-12]. There are very few reports available for synthesis of palladium nanoparticles[5-12]. Hence, the present is aimed to design a protocol for the biological synthesis of palladium nanoparticles using leaf extract of *Sebastiania chamaelea*.

Experimental

All the reagents used in the present study were obtained from Himedia Laboratories Pvt. Ltd., (Mumbai, India). *Sebastiania chamaelea*(L.)Muell. Arg. (Family: *Euphorbiaceae*), is anerect or ascending herb to 80cm; latex milky. Leaves alternate, linear-lanceolate, 05-10×0.4-0.69 (1) cm. Unisexual monoecious male flowering are minute, in clusters of threeTepals unequally 05-partite/lobed. Stamens 03, free, alternate with tepals, exserted and female flowering are three Tepals with Ovary echinate, Tri-locular;styles 3. Disc 0.Capsule 3-lobed, 5mm across of 2-valved cocci, each with 2 vertical rows of soft prickles; seeds oblong, carunculate; testa smooth- Plains from the coast, especially on the hills to 1200m, on the thin layer of soil by exposed rocks; robust specimens in fallow fields.Flower and fruit 1-12- India and Sri Lanka to Australia and Pacific[13]. Fresh and healthy leaves of *Sebastiania chamaelea*(Fig. 1) were collected from the campus of AyyaNadar JanakiAmmal College, Sivakasi, Tamil Nadu, India.

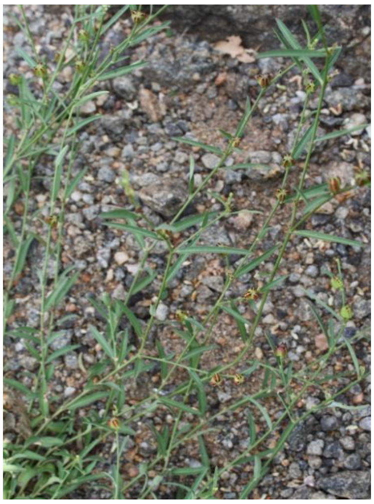

Figure 1: *Sebastianiachamaelea* Leaves

The collected leaf samples were thoroughly washed with tap water followed by distilled water to remove the surface contaminants and dried for 48 hours under shade. The dried leaves were ground to make fine powder using mortar and pestle and sieved using 20 mesh sieve to get uniform size range. For the preparation of leaf broth, the sieved leaf powder of *Sebastiania chamaelea*(10g) added to 100ml of distilled water and boiled at 70^0C for ten minutes. The freshly prepared leaf broth (10 ml) was re-suspended in 90 ml of aqueous solution of palladium chloride and this mixture is used as reaction medium. This reaction medium was kept at room temperature for 24 hours. From these reaction media a small aliquot of the samples was collected separately to characterize the palladium nanoparticles that were synthesized during the above reaction. The characterization was performed through the following analyses: UV-Visible spectroscopy (UV-Vis), Fourier Transform Infra-Red Spectroscopy (FTIR), X-ray diffraction (XRD) analysis, Scanning Electron Microscopy (SEM) and Energy Dispersive X- ray analysis (EDX).

Results and discussion

In biological synthesis, natural materials act as reducing agents for the production of metal nanoparticles[14]. In this present work, we have shown the ability of *Sebastiania chamaelea*leaf extract to reduce palladium chloride ions and form palladium nanoparticles.

UV-Visible spectrum of silver nanoparticles

UV-Visible spectral analysis was carried out on a Labomed (Model UV-D3200) UV- Visible spectrophotometer. The formation of palladium nanoparticles was monitored by UV-Visible spectroscopy in the

350-700nm range. The colour of the reaction medium changed from transparent yellow to brown, which is due to the excitations of surface plasmon vibrations. It indicates the formation of palladium nanoparticles and it was confirmed by UV-Visible analysis. Fig. 1 shows the UV-Vis spectra of reaction medium with respect time. The maximum absorbance peak was observedat 475nm at 24hrs of incubation and the absorbance was raised up to 0.78a.u. The absorption bands appearing in the contrast spectrum of $PdCl_2$ solution were ascribed to the ligand-to-metal charge-transfer transition of Pd (II) ions [15,16,17]. The maximum absorbance peak for synthesized palladium nanoparticles using aqueous extract of soybean (*Glycine max*) leaf was 420nm [19].

In the reaction medium, fifty percent of reduction completed in the first three hours and the rest of the reduction occurred in the next three hours (Figure 2a). The reduction is very quick and the reduction was completed at24 hours.

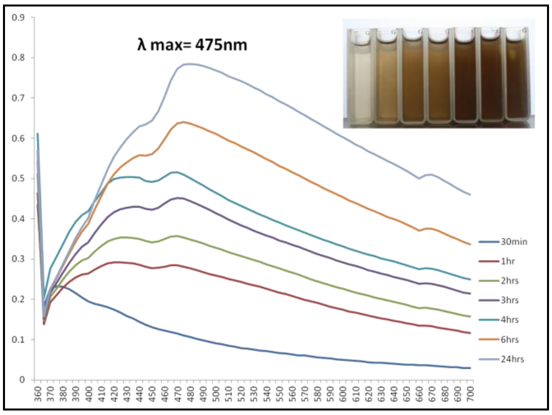

Figure 2: UV –Visible absorption spectra of palladium nanoparticles synthesized by leaf broth of *Sebastianiachamaelea*. The inset shows the colour change of the reaction medium- 30min, 1hr, 2hrs, 3hrs, 4hrs, 6hrs, and 24hrs (left to right).

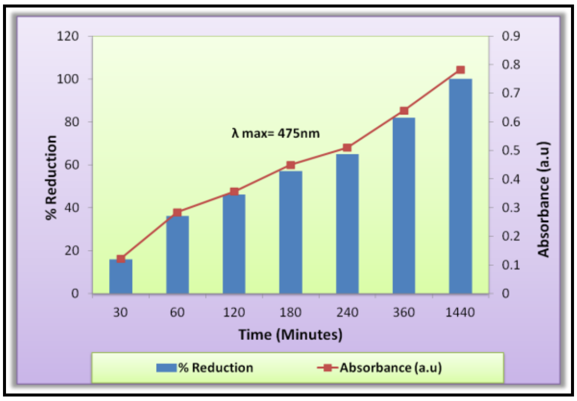

Figure 2a:Spectral plots of absorbance at λ max of SPR bands and percentage reduction of palladium chloride as a function of time

FT-IR Spectroscopic Analysis

FT-IR measurements (using Shinmadzu FT-IR spectrophotometer through KBR pellet method) identified the biomolecules in the *Sebastiania chamaelea* leaf broth, which are responsible for reduction and stability to the biosynthesized palladium nanoparticles. The IR spectra analysis provides an idea about biomolecules bearing different functionalities [14]. Fig. 3 shown the FT-IR spectrum bands of palladium nanoparticles at 840.90 cm^{-1}, 972.12 cm^{-1}, 995.27 cm^{-1} and 1458.18 cm^{-1} correspond to C-H bend (alkenes), 2877.79 cm^{-1}, 2916.37 cm^{-1} and 2954.95 cm^{-1}correspond toC-H stretch (alkanes), 2129.41 cm^{-1} corresponds to -C≡C- stretch (alkynes),671.23 cm^{-1} corresponds to =C-H bend (alkynes), 501.49 cm^{-1} and 578.64 cm^{-1} correspond to –C-Br stretch (alkyl halides), 748.38 cm^{-1} corresponds to –C-Cl stretch (alkyl halides), 1735.93 cm^{-1} corresponds to C=O stretch (aldehydes), 3194.12 cm^{-1} corresponds to N-H stretch (amines), 3441.01 cm^{-1} corresponds to N-H stretch (amides), 1064.71 cm^{-1}, 1165.00 cm^{-1} and 1249.87 cm^{-1} correspond to C-N stretch (aliphatic amines), 1373.32 cm^{-1} corresponds to N=O stretch (nitro compounds), 2222.00 cm^{-1} and 2337.72 cm^{-1} correspond to C≡N stretch (nitriles), 2576.90 cm^{-1}, 2723.49 $cm^{-1}$and 2839.22 cm^{-1} correspond to O-H stretch (carboxylic acid).[14]have synthesized palladium nanoparticles using *Solanum* leaf extract and assigned theFT-IR bands at 1637 and 2675cm^{-1}to the carbonyl groups and secondary amines respectively.

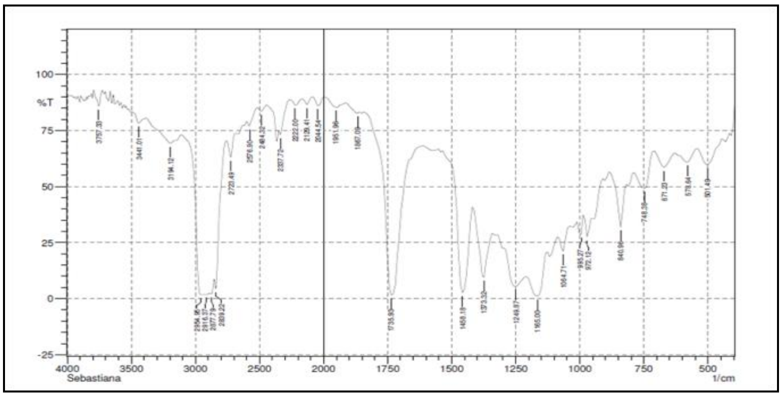

Figure 3: FTIR spectrum of synthesized palladium nanoparticles using leaf aqueous broth of *Sebastiania chamaelea* with palladium chloride.

XRD analysis

The X-ray pattern of synthesized palladium nanoparticles was shown in Fig.4.Crystalline metallic palladium nanoparticles were examined by X-ray diffractometer Shimadzu (XRD 6000). The number of strong Bragg's diffracted peaks was observed at 28.45^{o}, 33.0^{o}, 35.93^{o}and 40.0^{o} corresponding to the (100), (200), (220) and (111) and facets of the face-centered cubic lattice of palladium were obtained. The crystallite size of palladium nanoparticles was calculated using peak broadening profile of (111) peak at 40.0^{o}. The calculated crystallite size of the synthesized palladium nanoparticles is 70nm.

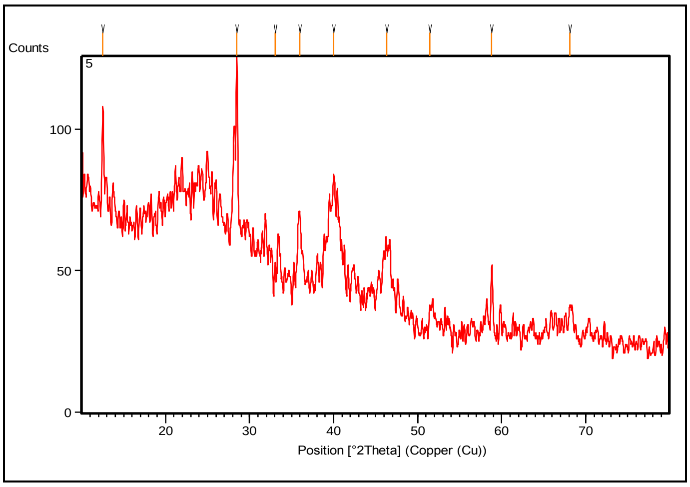

Figure 4: XRD pattern of palladium nanoparticles formed after reaction of leaf broth of *Sebastianiachamaelea*.

SEM and EDAX analysis

SEM images provided information about the morphology and size of the biosynthesized palladium nanoparticles. Fig. 5 shows the SEM image of palladium nanoparticles, these particles are in different shapes such as relatively spherical and cubic within the size range of 50-80nm. Recently,[4] reported that the fabrication of spherical shaped palladium nanoparticles using aqueous extract of fruits of *Pistacia atlantica* with the average size of about 60nm, while it was spherical shape within the size range 60-70nm of palladium nanoparticles using *Solanum trilobatum*leaf extract[14].Fig. 6 shows the EDAX patterns of synthesized palladium nanoparticles using *Sebastiania chamaelea* leaf extract. The EDAX showed peak that confirmed presence palladium in the suspension.

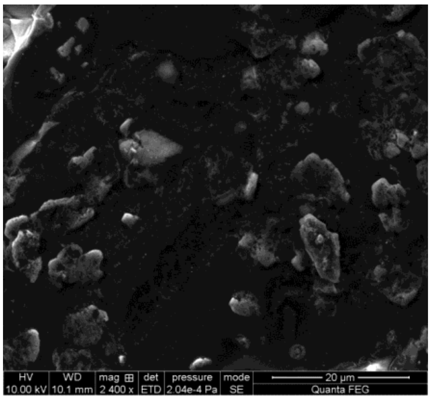

Figure 5: SEM images of palladium nanoparticles synthesized from the *Sebastiania chamaelea* leaf broth.

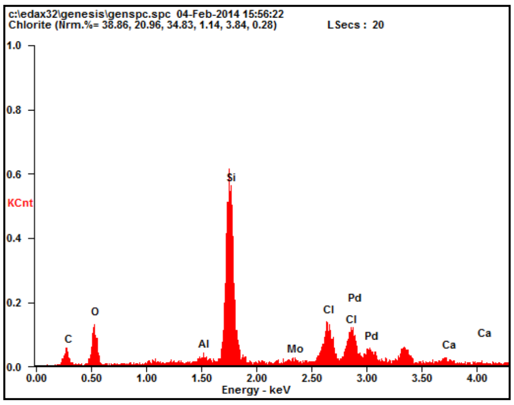

Figure 6: EDX images of palladiumnano particles synthesized from the *Sebastiania chamaelea* leaf broth.

Conclusion

We reported the biological synthesis of palladium nanoparticles with a help of *Sebastiania chamaelea* leaf extract. It has been achieved with the rapid reduction of palladium chloride into palladium nanoparticles. The reaction medium changed its color from transparent yellow to brown during the incubation period. The UV-Visible spectrum of the reaction medium has λ max at 475 nm and the absorbance raised up to 0.78a.u. The FT-IR spectrum showed the bands of palladium nanoparticles at 840.90 cm^{-1}, 972.12 cm^{-1}, 995.27 cm^{-1} and 1458.18 cm^{-1} correspond to C-H bend (alkenes), 2877.79 cm^{-1}, 2916.37 cm^{-1} and 2954.95 cm^{-1}correspond toC-H stretch (alkanes), 2129.41 cm^{-1} corresponds to -C≡C- stretch (alkynes),671.23 cm^{-1} corresponds to =C-H bend (alkynes), 501.49 cm^{-1} and 578.64 cm^{-1} correspond to –C-Br stretch (alkyl halides), 748.38 cm^{-1} corresponds to –C-Cl stretch (alkyl halides), 1735.93 cm^{-1} corresponds to C=O stretch (aldehydes), 3194.12 cm^{-1} corresponds to N-H stretch (amines), 3441.01 cm^{-1} corresponds to N-H stretch (amides), 1064.71 cm^{-1}, 1165.00 cm^{-1} and 1249.87 cm^{-1} correspond to C-N stretch (aliphatic amines), 1373.32 cm^{-1} corresponds to N=O stretch (nitro compounds), 2222.00 cm^{-1} and 2337.72 cm^{-1} correspond to C≡N stretch (nitriles), 2576.90 cm^{-1}, 2723.49 $cm^{-1}$and 2839.22 cm^{-1} correspond to O-H stretch (carboxylic acid)after bio-reduction. The synthesized particles ranged in size from 50-80 nm and were different shapes such as relatively spherical and cubic as shown by the SEM images. The strong palladium peak obtained from the EDAX spectrum confirms the significant presence of elemental palladium. The XRD analysis determines the average size of the nanoparticles is 70 nm. Thus we succeeded in designing a green approach which is simple and cost-effective and an alternative to physical and chemical methods in the synthesis of palladium nanoparticles.

Acknowledgement

This work is supported by Science and Engineering Research Board, Department of Science and Technology, Government of India, New Delhi. Authors thank the Principal and Management of AyyaNadarJanakiAmmal College, Sivakasi for providing facilities.

References

1. Mallikarjuns, K., John Sushma, N., Narasimha, G., Rao, K.V., Manoj, L., and Deva, B., Prasad Raju., Synthesis and spectroscopic characterization of palladium nanoparticles by using broth of edible mushroom extract. Proceedings of the "International Conference on Nanoscience, Engineering and Technology" (ICONSET 2011) Organized by Sathyabama University in association with Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, November, 2011, 28-30.
2. Antastas, P.T., and Warner, J.C., Green chemistry: Theory and practice, Oxford University Press, Oxford, UK, 1998.
3. Gong, C.Y., Wang, Y., Wang, X., Wei, X., Wu, Q., Wang, B., Dong, P., Chen, L., Luo, F., and Qian, Z., Biodegradable self-assembled PEG-PCL-PEG miscelles for hydrophobic drug delivery, Part 2: in vitro and in vivo toxicity evaluation. J. Nanopart. Res., 2011, 13, 721-731.
4. Molaie, R., Farhadi, K., Forough, M., and Sabzi, R.E., Biological and Green synthesis of palladium nanoparticles using aqueous extract of *Pistacia atlantica* plant's Fruit; A facile biological approach Proceedings of the 4th International Conference on Nanostructures (ICNS4), Kish Island, I.R. Iran 12-14 March, 2012.
5. Ganesan, V., Astalakshmi, A., Nima, P., Arunkumar, C., Synthesis and characterization of silver nanoparticles using *Merremia tridentata* (L) Hall. F., International Journal of Current Science,2013, 6, 87-93.
6. Arun kumar, C., Astalakshmi, A., Nima, P., and Ganesan, V., Plant mediated Synthesis of Silver nanoparticles using leaves of *Odina wodier* Roxb. International Journal of Advanced Research, 2013, 1(5), 265-272.
7. Nima, P., and Ganesan, V., Eco-friendly synthesis and characterization of silver nanoparticles synthesized at different pH using leaf broth of *Hyptissuaveolens* (L.) PoitInt.J. Nanotechnol. Appl, 2013, 3, 19-30.
8. Arunkumar, C., Nima, P., Astalakshmi, A., and Ganesan, V.,Green synthesis and characterization of silver nanoparticles using leaves of *Tecomastans* (L.) Kuth. Int. J. NanotechnolAppl, 2013, 3, 1-10.
9. Ganesan, V., Aruna Devi, J., Astalakshmi, A., Nima, P., and Thangaraja, A., Eco-friendly synthesis of silver nanoparticles using a sea weed, *Kappaphycus alvarezii* (Doty) Doty ex P. C. Silva. Int.J. Eng. Adv. Technol., 2013, 2, 559-563.
10. Astalakshmi, A., Nima, P., and Ganesan, V., A green approach in the synthesis of silver nanoparticles using bark of *Eucalyptus globulus*, Labill. International Journal of Pharmaceutical Sciences Review and Research, 2013, 23(1), 47-52.
11. Malathi, R., and Ganesan, V., Biogenic Synthesis of Silver Nanoparticles usingleaves of *Crinum asiaticum* Linn. International Journal of Science and Research, 2014,3(10), 2238- 2243.
12. Astalakshmi,A., Nima, P., Malathi, R., and Ganesan, V., Evaluating the potentiality of leaves of *Manilkarazapota* (L.) P. Royan and *Mimusopselengi* L. in the synthesis of silver nanoparticles. International Journal of Metallurgical & Materials Science and Engineering, 2014,4(2), 25-36.
13. Mathew, K.M., An excursion flora of central Tamil nadu India, The Rapinat Herbarium, Tiruchirapalli, 1991, 470.
14. Kanchana, A., Devarajan, S., and Ayyappan,S.R., Green synthesis and characterization of palladium nanoparticles and its conjugates from *Solanum trilobatum* leaf extract, Nano-Micro Letters, 2010, 2(3), 169-176.
15. Yonezawa, T., Imamura, K., and Kimizuka, N., Direct preparation and size control of palladium nanoparticle hydrosols by water-soluble isocyanide ligands, Langmuir, 2001, 17, 4701–4703.
16. Luo, C., Zhang, Y., and Wang, Y., Palladium nanoparticles in poly(ethyleneglycol): the efficient and recyclable catalyst for Heck reaction, 2005, J.MolCatal A, 2005, 229, 7–12.
17. Ho, P.F., and Chi. K.M., Size-controlled synthesis of Pd nanoparticles from b-diketonato complexes of palladium, Nanotechnology, 2004, 15, 1059–1064.
18. Yang, X., Li, Q., Wang, H.,Huang, J.,Lin, L., Wang, W., Sun, D., Su,Y.,Opiyo, J.B., Hong, L., Wang, Y., He, N., and Jia, L., Green synthesis of palladium nanoparticles using broth of *Cinnamomum Camphora*leaf," Journal of Nanoparticle Research, 2010, 12(5), 1589-1598.
19. Petla, R.M., Vivekanandhan, S., Misra, M., Mohanty, A.K., and Satyanarayana, N., Soybean (Glycine max) Leaf Extract Based Green synthesis of palladium nanoparticles. Journal of Biomaterials and Nanobiotechnology, 2012, 3, 14-19.

*****