

RESEARCH ARTICLE

Synthesis and Characterization of Nano Porous Nickel Oxide Particles using *Eclipta prostrata* Plant Extract

Meyyappan Revathi*, D. Malathy, A. Nancy, Sivagama Sundari

Department of Chemistry, School of Basic Sciences, Vels Institute of Science, Technology and Advanced studies, Velan Nagar, Pallavaram 600117

*Corresponding Author E-mail: mrev80@gmail.com

ABSTRACT:

Biosynthesis of nano materials is a better alternative to physical and chemical methods because of eco-friendly, cost effective and less time requirement. In the present study nano porous nickel oxide particles were prepared by novel green synthesis method using aqueous and alcoholic extract of *Eclipta prostrata* plants. The plant extract behaves both as a strong reductant and capping agent for the synthesis of nano particles. 10ml of the plant extract was mixed with 90ml 0.1M of nickel nitrate aqueous solution. The reaction mixture was heated at 80°C for one hour. The change in the colour of the mixer confirms the formation of nano particles and completion of the reaction, after that the entire content was kept in a muffle furnace at 300°C. The synthesized nano particles will be physico chemically characterised by XRD, SEM and TEM.

KEYWORDS: Biosynthesis, Nanotechnology,

INTRODUCTION:

Due to swift industrialization and urbanization, our environment is undergo huge smash up and a large amount of perilous and superfluous chemical, gases or substances are released, and so now it is our need to learn about the secrets that are present in the Nature and its products which leads to the growth of advancements in the synthesis processes of nanoparticles. Nanotechnology applications are highly suitable for biological molecules, because of their exclusive properties. The biological molecules undergo highly controlled assembly for making them suitable for the metal nanoparticle synthesis which was found to be reliable and eco friendly [1-3].

Nanoparticles:

The term “nanoparticles” is used to describe a particle with size in the range of 1nm-100nm, at least in one of the three possible dimensions.

In this size range, the physical, chemical and biological properties of the nanoparticles changes in fundamental ways from the properties of both individual atoms/molecules and of the corresponding bulk materials.

Nanoparticles can be made of materials of diverse chemical nature, the most common being metals, metal oxides, silicates, non-oxide ceramics, polymers, organics, carbon and biomolecules. Nanoparticles exist in several different morphologies such as spheres, cylinders, platelets, tubes etc. Generally the nanoparticles are designed with surface modifications tailored to meet the needs of specific applications they are going to be used for.

Synthesis of nanoparticles:

Nanotechnology is an innovative and budding field of science that is clear to have marvelous impact on mankind by helping to solve major dares in health and energy. There is a fabulous research interest in the area of nanotechnology to extend consistent processes for the synthesis of nanomaterials over a range of sizes. This is due to the realistic applications of metal nanoparticles in various areas such as medicine [4], chemical sensing, catalysis, and electronics [5].

There are two basic approaches used in nanoparticle synthesis: the top-down (communication and dispersion) approach and the bottom-up (nucleation and growth) approach.

While the usual methods of synthesis of metal sols persist to be used for producing metal nanoparticles, there have been numerous upgrades and modifications in the methods which provide a better control over the size,

shape, and other characteristics of the nanoparticles [6]. Various chemical, physical, and biological methods have been developed to synthesis nanoparticles.

Physical approaches:

Most important physical approaches include evaporation-condensation and laser ablation. Various metal nanoparticles such as silver, gold, lead sulfide, cadmium sulfide, and fullerene have previously been synthesized using the evaporation-condensation method. The absence of solvent contamination in the prepared thin films and the uniformity of nanoparticles distribution are the advantages of physical approaches in comparison with chemical processes. [7,8].

Chemical approaches:

The most common approach for synthesis of nanoparticles is chemical reduction by organic and inorganic reducing agents. In general, different reducing agents such as sodium citrate, ascorbate, sodium borohydride (NaBH_4), elemental hydrogen, polyol process, Tollens reagent, N, N-dimethylformamide (DMF), and poly (ethylene glycol)-block copolymers are used for reduction of metal ions (Ag^+) in aqueous or non-aqueous solutions. However chemical and physical methods are produce toxic byproducts which are hazardous [9].

Biological approaches:

In recent years, the development of efficient green chemistry methods employing natural reducing, capping, and stabilizing agents to prepare silver nanoparticles with desired morphology and size have become a major focus of researchers. Biological methods can be used to synthesize silver nanoparticles without the use of any harsh, toxic and expensive chemical substances [10,11]. The bioreduction of metal ions by combinations of biomolecules found in the extracts of certain organisms (*e.g.*, enzymes/proteins, amino acids, polysaccharides, and vitamins) is environmentally benign, yet chemically complex. Many studies have reported successful synthesis of silver nanoparticle using organisms (microorganisms and biological systems) [12,13].

Need for green synthesis:

Biosynthesis of nanoparticles is a kind of bottom up approach where the main reaction occurring is reduction/oxidation. The need for biosynthesis of nanoparticles rose as the physical and chemical processes were costly. Often, chemical synthesis method leads to presence of some of the toxic chemical absorbed on the surface that may have adverse effect in the medical applications [14]. This is not an issue when it comes to biosynthesized nanoparticles via green synthesis route [15]. So, in the exploration of low cost pathways for nanoparticles synthesis, scientists have been used microbial enzymes and plant extracts (phytochemicals).

With their antioxidant or reducing properties they are usually responsible for the reduction of metal compounds into their respective nanoparticles. Green synthesis provides advancement over chemical and physical method as it is cost effective, environment friendly, easily scaled up for large scale synthesis and in this method there is no need to use high pressure, energy, temperature and toxic chemicals. Plants provide a better platform for nanoparticles synthesis as they are free from toxic chemicals as well as provide natural capping agents. Moreover, use of plant extracts also reduces the cost of microorganisms isolation and culture media enhancing the cost competitive feasibility over nanoparticles synthesis by microorganisms [16]

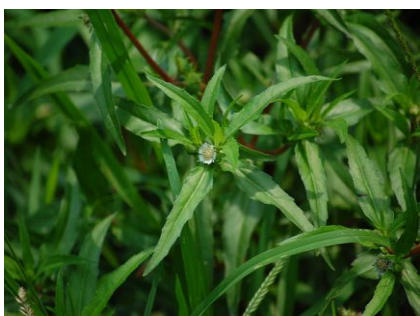
Sometimes the synthesis of nanoparticles using various plants and their extracts can be advantageous over other biological synthesis processes which involve the very complex procedures of maintaining microbial cultures [17,18]. Many such experiments have already been started such as the synthesis of various metal nanoparticles using fungi like *Fusarium oxysporum* [19], *Penicillium* sp. [20] and using some bacteria such as *Bacillus subtilis* etc. [21,22]. But, synthesis of nanoparticles using plant extracts is the most adopted method of green, eco-friendly production of nanoparticles and also has a special advantage that the plants are widely distributed, easily available, much safer to handle and act as a source of several metabolites [23]. There has also been several experiments performed on the synthesis of silver nanoparticles using medicinal plants such as *Oryza sativa*, *Helianthus annuus*, *Saccharum officinarum*, *Sorghum bicolor*, *Zea mays*, *Basella alba*, *Aloe vera* *Capsicum annum*, *Magnolia kobus*, *Medicago sativa* (Alfalfa), *Cinamomum camphora* and *Geranium* sp. in the field of pharmaceutical applications and biological industries. Besides, green synthesis of silver nanoparticles using a methanolic extract of *Eucalyptus hybrida* was also investigated [24]. In the recent days, silver nanoparticles have been synthesized from the naturally occurring sources and their products like green tea (*Camellia sinensis*), Neem (*Azadirachta indica*), leguminous shrub (*Sesbania drummondii*), various leaf broth, natural rubber, starch, *Aloe vera* plant extract, lemongrass leaves extract, etc. [25]. With respect to the microbes, the silver nanoparticles get attached to the cell wall, thereby disturbing the permeability of cell wall and cellular respiration. The nanoparticles may also penetrate deep inside the cell wall, thus causing cellular damage by interacting with phosphorus and sulfur containing compounds, such as DNA and protein, present inside the cell. The bacteriocidal properties of silver nanoparticles are due to the release of silver ions from the particles, which confers the antimicrobial activity [26]. Besides, the potency of the antibacterial effects corresponds to the

size of the nanoparticle. The smaller particles have higher antibacterial activities due to the equivalent silver mass content. With respect to the clinical applications of nanoparticle, microorganisms including diatoms, fungi, bacteria and yeast producing inorganic materials through biological synthesis either intra or extracellular made nanoparticles more biocompatible [27]. Hence, the objective of this present study is to synthesis nano porous nickel oxide particles by green synthesis method using *Eclipta prostrata* plant extract.

MATERIALS AND METHODS:

Eclipta prostrata:

Eclipta prostrata commonly known as false daisy, yerba de tago, Karisalankanni, and bhringraj, is a species of plant in the sunflower family. It is widespread across much of the world. This plant has cylindrical, grayish roots. The solitary flower heads are 6–8 mm in diameter, with white florets.



Scientific name:

Eclipta prostrata
Kingdom-Plantae
Clade –Angiosperms
Order –Asterales
Family-Asteraceae
Genus- *Eclipta*
Species -*E. Prostrata*

Synthesis of nano porous nickel oxide particles:

The leaves of *Eclipta prostrata* plant were collected and dried at room temperature, grinded well. The 10g of dried powder was mixed with 100ml of ethanol and water separately. The mixture was heated at 60°C for 2 hours. It was filtered by using whatmann filter paper and the filtrate was collected. The ethanolic and water extract of the plant were used as a stock solution for the study. 10ml of *Eclipta prostrata* plant ethanolic extract and water extract were separately mixed with 90ml of 0.1M nickel nitrate aqueous solution in a 200ml beaker. The reaction mixture was stirred and heated at 80°C for one hour. The change in colour of the reaction mixture was

noted. The ethanol medium colour was changed from green to greenish yellow colour and the water medium colour was changed from green to brown colour which showed the formation of NiONPs.

Characterization:

Green synthesized NiONPs samples were used to observe Fourier transform infrared spectra study to identify the presence of functional groups. FT-IR spectra were measured by Perkin Elmer 360 model spectrometer having a 4000 to 400cm⁻¹ with 4cm⁻¹ resolution over 40 scans. X-ray diffraction measurement was done with Cu-K α radiation of 0.154187 nm wavelength to investigate the formation, crystalline behavior and the quality of synthesized NiONPs powder. The scanning was done in the region of 2 θ from 30° to 80° at 0.02/min and the time constant was 2 Sec. The size of NiONPs was calculated by the Debye-Scherrer equation. The surface morphology and particle size of synthesized NiONPs was investigated by scanning electronic microscope.

RESULT AND DISCUSSION:

The green synthesis of nickel nano particles by plant extract is studied in this study. The Present study deals with the nickel particles using leaf extract of *Eclipta prostrata* and *Ocimum tenuiflorum*. The result related to the synthesis of nickel nano particles and their characterization is explained here.

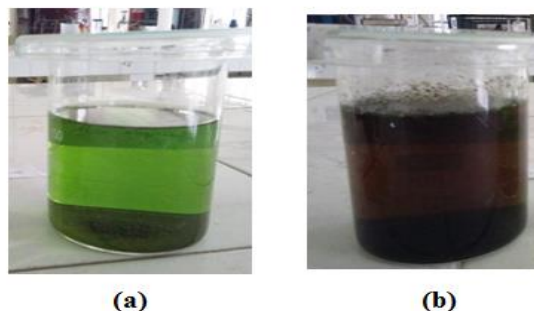


Figure. 1 (a) Reaction mixture before nano particle formation (b) After nano particle formation

PHYSICAL CHANGE:

When the plant extract was mixed in the aqueous solution of the nickel, the plant extract acted as an reducing agent and reduces the nickel ion and produces NiONPs the formation of nano particles was confirmed by the colour change of the reaction mixer (greenish to brownish colour). Figure. 1 shows the change in colour of the reaction mixture which shows the formation of NiONPs.

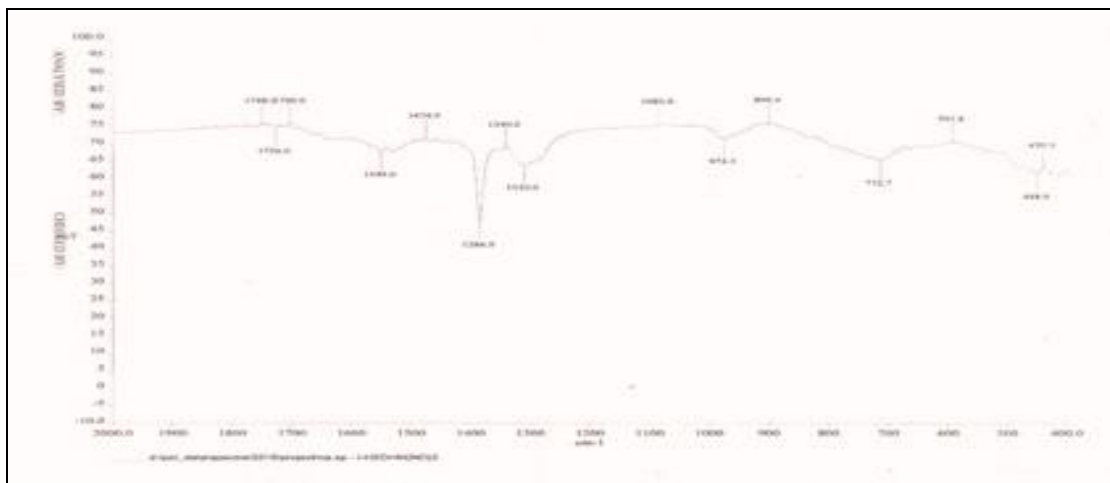
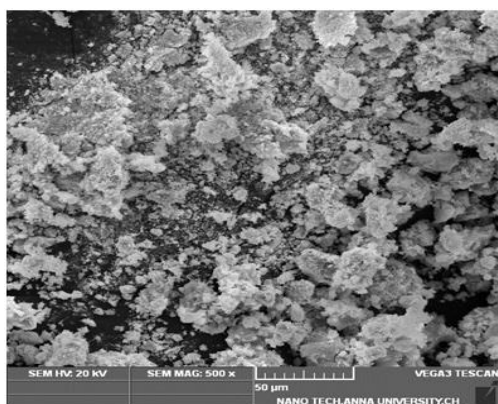
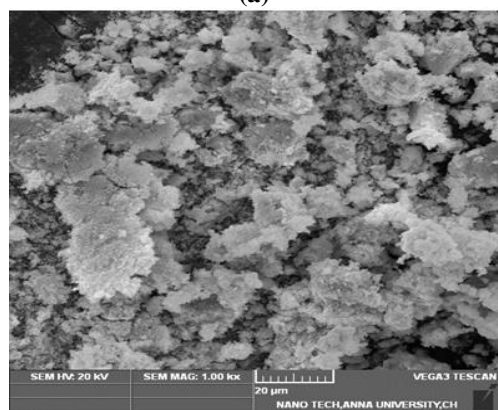


Figure: 2 FTIR spectrum of synthesized nano porous NiONPs



(a)



(b)

Figure: 3 SEM images of synthesized nano porous NiONPs

FTIR:

The peak observed at approximately 1644cm^{-1} is assigned to the bending vibration of water molecules. The peak so Observed at approximately 1390cm^{-1} , 1300cm^{-1} and 1030cm^{-1} are respectively, assigned to the O-C=O symmetric and asymmetric stretching vibrations and the C-O stretching vibration originating from the adsorption of atmospheric CO_2 or ethanol, but the intensity of the band has weakened, which indicated that

the ultra fine powers tend to strong physically absorption to H_2O and CO_2 Figure. 2. After calcination, the FT-IR Spectra NiO nanoparticle shows strong band at 425 cm^{-1} corresponds to the vibration of Ni-O bond.

SCANNING ELECTRON MICROSCOPE:

The morphological structure of the green synthesized NiONPs materials scanning was observed using Hitachi S-4800 field emission scanning electron microscope. Fig. 3a, b represents low and high magnification of SEM micrographs of synthesized NiONPs which confirms the formation of pebble like sheet. Most of the NiONPs are predominately spherical in shape having smooth surface and well dispersed with close compact arrangement. The average particle size was found around 7.26 nm using an advanced software named “IMAGEJ”, the obtained particle size is almost similar to XRD.

X-RAY DIFFRACTION STUDIES:

The XRD pattern of synthesized NiONPs using *Eclipta prostrata* leaf extract was shown in Fig. 4. The XRD was done to determine the crystalline nature of AgNPs and the resulted peaks were found at 13.5° , 28.64° , 38.15° , 44.35° , 60.49° and 64.60° correspond to the (001), (002), (111), (200), (220), and (222) lattice planes confirms the formation of face centered cubic crystal structured Ni (111), Ni (200), Ni (012), Ni (222), NiO (111), NiO (012) and NiO (220), respectively [28,29]. XRD results also suggested that the crystallization of the bio-organic phase occurs on the surface of the NiONPs or vice versa. Generally, the broadening of peaks in the XRD patterns of solids is attributed to particle size effects. Broader peaks signify smaller particle size and reflect the effects due to experimental conditions on the nucleation and growth of the crystal.

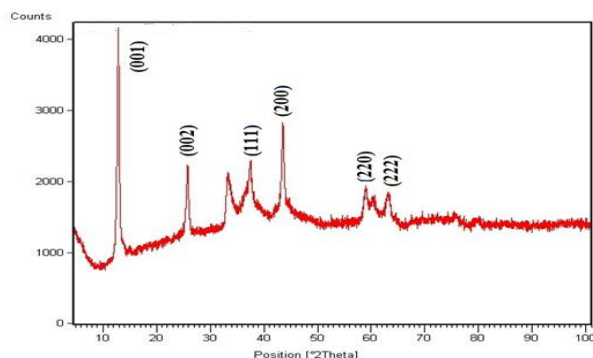


Figure: 4 XRD patterns of synthesized NiONPs

CONCLUSION:

The NiO nanoparticles have been successfully synthesized by using *Eclipta prostrata* plant extracts. The formation of these nanoparticles has been confirmed by XRD, SEM. The results reveal that the synthesized NiONPs are in spherical structure. The surface morphology of the NiONPs was studied using scanning electron microscopy which recorded the maximum of 1mm size. The size range studied through SEM results range from 5µm to 500µm. The FT-IR spectrum ascribed the biological molecules which perform dual functions of formation and stabilization of NiONPs in the aqueous medium. So, it can be summarized that, green synthesis is an effective and eco-friendly method of producing metal nanoparticles.

REFERENCES:

- Resmi, C.R., Sreejamol, P. and Prita Pillai, Green synthesis of silver nanoparticles using *azadirachta indica* leaves extract and evaluation of antibacterial activities, International journal of Advanced biological research, 4(3), 300-303, 2014.
- Amiya kumar Prusty, Prasanna parida, Green Synthesis of Silver Nanoparticle Using *Eichhornia crassipes* and Study of in-vitro Antimicrobial Activity, Scholars Academic Journal of Pharmacy (SAJP), 3(6): 504-509, 2014.
- Monaliben Shah, Derek Fawcett, Shashi Sharma, Suraj Kumar Tripathy and G errard Eddy Jai Poinern, Green Synthesis of Metallic Nanoparticles via Biological Entities, Materials, 7278–7308, 2015.
- OV Salata, Applications of nanoparticles in biology and medicine, J Nanobiotechnology, 2(3),1- 6, 2004.
- Wim H De Jong and Paul JA Borm, Drug delivery and nanoparticles: Applications and hazards, International Journal of Nanomedicine, 3(2), 133–149, 2008.
- Xi-Feng Zhang, Zhi-Guo Liu, Wei Shen and Sangiliyandi Gurunathan, Silver Nanoparticles: Synthesis, Characterization, Properties, Applications, and Therapeutic Approaches, International Journal of Journal of Molecular Sciences, 17, 1534-1568, 2016.
- S. Iravani, H. Korbekandi, S.V. Mirmohammadi, and B. Zolfaghari, Synthesis of silver nanoparticles: chemical, physical and biological methods, Research in Pharmaceutical Sciences, 9(6): 385–406, 2014.
- Jun Natsuki, Toshiaki Natsuki, Yoshio Hashimoto, A Review of Silver Nanoparticles: Synthesis Methods, Properties and Applications, International Journal of Materials Science and Applications, 4(5), 325-332, 2015.
- Essam A. Makky, Siti H. Mohd Rasdi, J.B. Al-Dabbagh, G.F. Najmuldeen and Ab Rahim Mohd Hasbi, Bioremediation of hazardous waste for silver nanoparticles production, International Journal of Current Microbiology and Applied Sciences, 2014, 3(12), 364-371.
- Payal N. Agrawal, Nikhilesh S. Kulkarni, Biosynthesis of Silver Nanoparticles from Silver Resistance Bacteria Isolated From Metal Contaminated Soil, Scholars Academic Journal of Biosciences, 2017,

- 5(3), 187-191.
- IbrahimKhan, KhalidSaeed, IdreesKhan, Nanoparticles: Properties, applications and toxicities, Arabian Journal of Chemistry, 2017, <https://doi.org/10.1016/j.arabjc.2017.05.011>
- Hassan Korbekandi, Zeynab Ashari, Siavash Iravani, and Sajjad Abbasi, Optimization of Biological Synthesis of Silver Nanoparticles using *Fusarium oxysporum*, Iran Journal of Pharmaceutical Research, 2013, 12(3), 289–298.
- Ahmed A. Moosa, Ali Mousa Ridha and Mustafa Hameed Allawi, Green Synthesis of Silver Nanoparticles using Spent Tea Leaves Extract with Atomic Force Microscopy, International Journal of Current Engineering and Technology, 2015, 5(5), 3233-3242.
- M. Ramya and M. Sylvia Subapriya, Green synthesis of silver nanoparticles, Int. J. Pharm. Med. & Bio. Sc. 2012, 1(1), 1-10.
- Sukumaran Prabhu and Eldho K Poulouse, Silver nanoparticles: mechanism of antimicrobial action, synthesis, medical applications, and toxicity effects, International Nano Letters 2012, 2:32, 1-10.
- P. Shanmuga Praba, V. S. Vasantha, J. Jeyasundari and Y. Brightson Arul Jacob, Synthesis of plant-mediated silver nanoparticles using ficus microcarpa leaf extract and evaluation of their antibacterial activities, Eur. Chem. Bull., 2015, 4(3), 117-120.
- G. Geoprincy, B. N. Vidhya Sri, U. Poonguzhali, N. Nagendra Gandhi, S. Renganathan, A Review On Green Synthesis Of Silver Nanoparticles, Asian Journal of Pharmaceutical and Clinical Research, 2013, 6(1), 8-12.
- Shakeel Ahmed, Mudasir Ahmad, Babu Lal Swami, Saiqa Ikram, A review on plants extract mediated synthesis of silver nanoparticles for antimicrobial applications: A green expertise, Journal of Advanced Research, 2016, 7, 17–28.
- Swarup Roy and Tapan Kumar Das, Plant Mediated Green Synthesis of Silver Nanoparticles-A Review, International Journal of Plant Biology & Research, 2015, 3(3), 1044-1055.
- Alka Yadav, Kateryna Kon, Gabriela Kratosova, Nelson Duran, P. Avinash, Ingle, Mahendra Rai, Fungi as an efficient mycosystem for the synthesis of metal nanoparticles: progress and key aspects of research, Biotechnol Letters, 2015, 37, 2099–2120.
- Khawaja Salahuddin Siddiqi and Azamal Husen, Khawaja Salahuddin Siddiqi and Azamal Husen, Fabrication of Metal Nanoparticles from Fungi and Metal Salts: Scope and Application, Nanoscale Research Letters, 2016, 11, 98-113.
- N. Saifuddin, C. W. Wong And A. A. Nur Yasumira, Rapid Biosynthesis of Silver Nanoparticles Using Culture Supernatant of Bacteria with Microwave Irradiation, E-Journal of Chemistry, 2009, 6(1), 61-70.
- Mina Sorbiun, Ebrahim Shayegan Mehr, Ali Ramazani, Asemeh Mashhadi Malekzadeh, Biosynthesis of metallic nanoparticles using plant extracts and evaluation of their antibacterial properties, Nanochem Research, 2018,3(1): 1-16.
- Manish Dubey, Seema Bhadauria, B.S. Kushwah, Green Synthesis of Nanosilver Particles From Extract Of *Eucalyptus Hybrid* (Safeda) Leaf, Digest Journal of Nanomaterials and Biostructures, 2009, 4 (3), 537 – 543.
- D. Gnanasangeeth, T.S.R. Umamageshwari, Green Synthesis of Zinc Oxide Nanoparticles for Water Remediation, International Journal of Chem Tech Research, 2017, 10 (15), 101-107.
- Amal Kumar Mondal, Sanjukta Mondal (Parui), Sumana Samanta and Sudebi Mallick, Synthesis of Ecofriendly Silver Nanoparticle from Plant Latex used as an Important Taxonomic Tool for Phylogenetic Interrelationship, Advances In Bioresearch, 2011, 2 (1), 122 – 133.
- Ratul Kumar Das, Vinayak Laxman Pachapur, Linson Lonappan, Mitra Naghdi, Rama Pulicharla, Sampa Maiti, Maximiliano Cledon, Larios Martinez Araceli Dalila, Saurabh Jyoti Sarma, Satinder Kaur Brar, Biological synthesis of metallic nanoparticles: plants, animals and microbial aspects, Nanotechnol. Environ. Eng. 2017, 2(18), 1-21.
- Priyabrata Mukherjee, Absar Ahmad, Deendayal Mandal, Satyajyoti Senapati, Sudhakar R. Sainkar, I. Mohammad, Khan, Renu Parishcha, P. V. Ajaykumar, Mansoor Alam, Rajiv Kumar, and Murali Sastry, Fungus-Mediated Synthesis of Silver Nanoparticles and Their Immobilization in the Mycelial Matrix: A Novel Biological Approach to Nanoparticle Synthesis, *Nano Letters*, 2001, 1 (10), 515–519.
- N. Subash And C. Sasikumar, Eco - Friendly Synthesis Of Silver Nano Particle From Leaf Extract Of *Azadirachta indica* and *Phyllanthus emblica*, International Journal of Advanced Research 2014, 2(7), 986-990.