

Synthesis and characterization of zinc oxide nanoparticle using *Chenopodium album* as a biotemplate and its larvicidal activity evaluation

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Abstract

The *Chenopodium album* plant was collected and the fresh plant extract plays a key role in zinc oxide nanoparticle synthesis. The reduction of Zn²⁺ ions to ZnO NPs by aqueous leaf extract of *Chenopodium album* was visually observed by color variation in the reaction mixture. The gradual color change in solution from light green to pale yellow. This indicates that the metal acetates were reduced to form its respective nanoparticles. The UV-visible absorption peak arises from 320-380 nm denote the development of ZnO NPs. In our study, the extreme absorption peak seemed at 374 nm directs the individual SPR band for ZnO NPs with lesser particle size.

The FT-IR spectrum noted, Scanning Electron Microscopy (SEM) investigation was performed to govern the size and morphology of the green synthesized ZnO NPs by *Chenopodium album* (aqueous leaf extract). The elemental composition of the synthesized ZnO NPs was confirmed by EDX analysis. The XRD pattern of aqueous leaf extract of *Chenopodium*

album derived ZnO NPs. In conclusion the zinc oxide nanoparticle was synthesized using fresh leaf extract of *Chenopodium album*. The synthesized nano particle was characterized and confirmed using UV-Visible, FT-IR, XRD, FE-SEM, EDX and SEM mapping analysis, the results showed that ZnO Nps were synthesized properly.

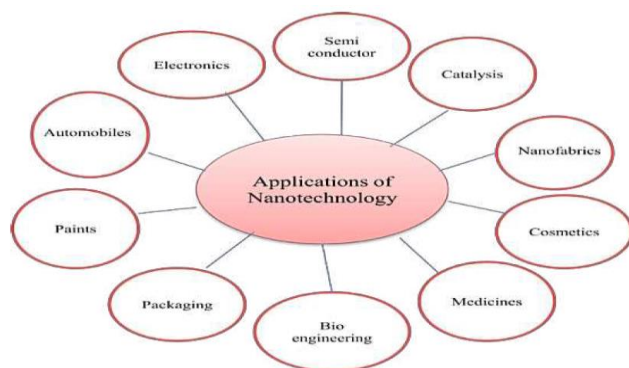
Keywords: *Chenopodium album*, ZnO NPs, larvicidal activity, aqueous leaf extract, UV-Visible, FT-IR, XRD, FE-SEM, EDX

Introduction

Mosquitoes are vectors for different frightful illnesses of humankind and are one of the most therapeutically huge vector transmitting pathogens and keep on devastatingly affecting people and creatures. Among the 3492 types of mosquitoes recorded around the world, in excess of a hundred animal types are equipped for transmitting different perilous illnesses like jungle fever, yellow fever, encephalitis, dengue, chikungunya and filariasis to human and different vertebrates [1] in tropical and subtropical nations and numerous pieces of the world [2]. Mosquito danger is especially high in South East Asian nations [3] and as of late an Earth-wide temperature boost has central to the mosquitoes blow out in calm nations and in higher elevation districts [4].

Worldwide utilization of substance bug sprays viz., organophosphates, for example, Temephos and Fenthion and bug development controllers, for example, diflubenzuron and methoprene are commonly utilized for the control of mosquito hatchlings [5]. The executives of illnesses vector utilizing engineered synthetic concoctions has flopped because of absence of novel bug sprays, significant expense of manufactured bug sprays, worry for ecological supportability, hurtful impact on human wellbeing and other non-target life forms, their non-

biodegradable nature, resurgences in mosquito populaces, higher pace of organic amplification through biological system and expanding bug spray obstruction on a worldwide scale [6-9]. These issues have featured the requirement for new systems for mosquito hatchlings control.



Plant extracts mediated synthesis of nanoparticles

At the beginning phase, microscopic organisms were utilized to combination nanoparticles and later continue with the utilization of infection, growths and actinomycetes and now the specialists have been concentrating on the common sources. Plant intervened engineered strategy is outstanding amongst other technique for the long-scale blend and nanoparticles delivered from plant removes are progressively steady with quicker manufactured rate contrasted and microorganism combination [10].

Also, it includes simple accessibility, ease and green methodology, more straight forward down gushing preparing and so on. A solitary advance plant intervened amalgamation proposes numerous courses to blend nanoparticles under surrounding conditions [11, 12]. In this specific situation, the plant-extricate helped biosynthesis of nanoparticles will be critical in different applications and discovering one of the most suffering methodologies towards natural generous course.

The writing review uncovered that they are distinctive sort of plants are being researched for their job in the blend of nanoparticles. It has been accounted for that silver nanoparticles have been orchestrated by the decrease of silver particles utilizing the concentrate of geranium leaves (*Pelargonium graveolens*). In comparison of same examinations with the utilizing of microbes and organisms, decrease process by utilizing plant extricate happens quickly [13]. The different plant materials, for example, *Morinda tinctoria* leaf extricate [14], *Ananas comosus* [15], *Cymbopogon flexuosus* extricate [16] and *Camellia sinensis* [17] are used to get ready Ag, Au nanoparticles. Correspondingly, *Musa balbisiana* peel extricate was utilized to union CuO [18], *Plectranthus amboinicus* leaf extract was used to blend ZnO [19], *Andean blackberry* leaf remove used to blend Fe₃O₄ [20], the leaf concentrate of *nyctanthes* was utilized to combination TiO₂, the blossom concentrate of *Achillea wilhelmsii* was used to union CdO [21] and the leaf separate of *Arachis hypogaea* was used to blend Cr₂O₃. So, flow research in organic technique utilizing plant extricates has opened another time in quick and nontoxic strategies for the creation of nanoparticles. During the combination, such a huge numeral of variables was impacting the arrangement of nanoparticles [22].

Depiction of Plant Material Used In the Present Study

There are different plant separates (root, stem, leaves, organic products, blossoms and seeds of the plant material) viably used for the planning of metal oxide nanoparticles. In the wake of dissecting an immense number of plant materials, we select *Chenopodium album* leaves since it contains higher constituents of polyphenols, which is answerable for hydrolysis of metal salts and shaping metal oxides. Additionally, it prompts squander minimization.

Objectives and Plan of Work

Objective:

- To synthesize zinc oxide nanoparticle by using fresh leaf of *Chenopodium album*
- To characterize the synthesized zinc oxide nanoparticle.
- To evaluate the larvicidal activity using synthesized nanoparticle.

Plan of work

- Literature survey of synthesis of zinc oxide nano particle using plant extracts.
- Selection of plant *Chenopodium album*
- Preparation of aqueous leaf extract
- Synthesis of zinc oxide nanoparticle can be done using fresh leaf extract of selected plant.
- Characterization was determined using UV-Visible, FTIR, SEM, EDX, SEM mapping and XRD analysis.

Material and Methods

Chemicals

All materials were purchased from Nice and Loba chemicals. Solvents used throughout the reactions were of high purity and used without further purifications.

Collection of plant materials

The plant material *Chenopodium album* was collected from the local places of Trichirappalli area. Freshly collected whole plant was used for the synthesis of zinc oxide nanoparticles.

Preparation of plant extract

Zinc acetate dehydrate salt and *Chenopodium album* plant extract were used as the preliminary materials. The extract solution was equipped by using leaves of *Chenopodium album* plant. The leaves of fresh plant that had been rinsed with de ionized water and finely cut into small pieces. Then the plant material was boiled with 100 mL of distilled water at 100°C, filtered by using whatmann No. 1 filter paper and stored at 4°C for further experimentation.

Synthesis of Zinc oxide nanoparticles

In the preparation of Zinc Oxide nanoparticles, samples $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1g) was first dissolved in enough quantity of de ionized water and mixed with 10mL of *Chenopodium album* plant extract solution under vigorous stirring with magnetic stirrer at 1000 rpm at room temperature for 3hr. Then 1ml of 10% NaOH solution was added to the reaction mixture to adjust the pH of the reaction mixture. The precipitated solid was filtered and dried. The crude product was maintained at 150°C for 12 hrs in oven. The obtained powder was calcined at 400 °C for 5 hrs and then crushed into fine powder by using pestle mortar.

Characterization studies

Zinc oxide nano particles synthesized by using green chemistry technique were confirmed with the help of UV-Visible spectrophotometer (Shimadzu) and FT-IR spectrophotometer (Shimadzu) spectrum in the range 4000-400 cm^{-1} , Powder XRD, SEM and EDX examination.

Larvicidal activity

The synthesized ZnO nanoparticle was further assessed for larvicidal activity against south urban mosquito larvae *Culex quinquefasciatus*. Appraisals were made on 'a dead/alive premise. Assessments depend on a rate size of 0–100, which 0 equivalents no action and 100 equivalents complete murder. The bioassay was rehashed multiple times, and the consequence of bioactivity was the normal of these reproduces. The qualities are contrasted and the positive control Permethrin. The LD50 values of some dynamic title mixes were assessed utilizing probit investigation and the outcomes were dissected utilizing the SPSS v16 programming.

Larvicidal Activity against Mosquito (*Culex quinquefasciatus*).

The aqueous leaf concentrate of *Chenopodium album* (1) and combined ZnO nanoparticle (2) were assessed for larvicidal activity against south urban mosquito larvae *Culex quinquefasciatus*. The larvicidal activity was assessed at the starter test convergence of 100 µg/mL against the fourth instar *Culex quinquefasciatus* by the water immersion strategy under states of (27 ± 2) °C, photoperiod of 10:14 (light:dark), and relative stickiness 50–70%. The test tests were set up at the convergences of 100, 75, 50, 25 µg/mL by utilizing dissolvable Dimethyl sulfoxide (DMSO). All the test measuring glasses containing twenty *Culex quinquefasciatus* were assessed for 24 hrs after treatment. The outcomes were documented by average percentage mortality.

Results and Discussion

The *Chenopodium album* plant was collected around Ariyalur and was identified using the *Flora of presidency of Madras* and the fresh plant extract plays a key role in zinc oxide nanoparticle synthesis.

Optical Characterization

The reduction of Zn^{2+} ions to Zn^0 NPs by aqueous leaf extract of *Chenopodium album* was visually observed by color variation in the reaction mixture. The gradual color change in solution from light green to pale yellow. This indicates that the metal acetates were reduced to form its respective nanoparticles.

UV-Visible Spectroscopy

The UV-visible absorption peak arises from 320-380 nm denote the development of ZnO NPs. In our study, the extreme absorption peak seemed at 374 nm directs the individual SPR band for ZnO NPs with lesser particle size. **Figure 1** displays UV-vis spectra of ZnO NPs synthesized by greener protocol.

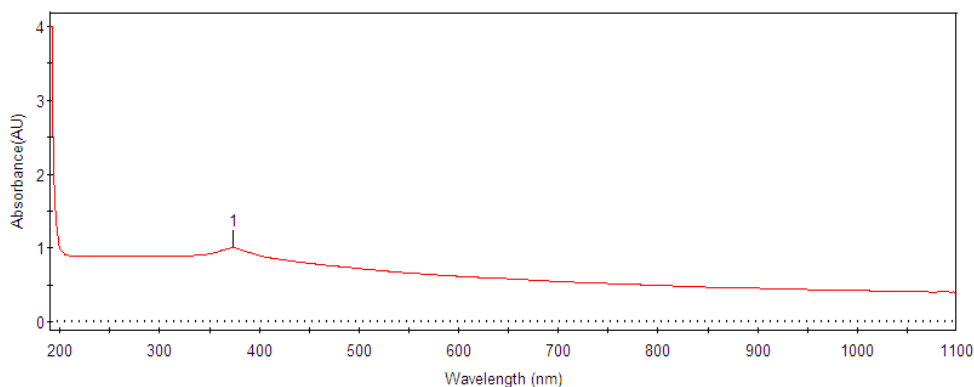


Figure 1 UV - Visible spectra of ZnO nanoparticle.

FT-IR Analysis of metal oxide nanoparticles synthesized by using aqueous leaf extract of *Chenopodium album*.

The FT-IR spectrum noted in the ranges from 400-4000 cm^{-1} . A wide peak at 3239.95 cm^{-1} agrees to the N-H group which may be appeared as a result of the manifestation of alkaloids [23]. The bands at $2800 - 3000 \text{ cm}^{-1}$ signify the existence of C-H functional group of alkanes [24]. The peaks (1583.84 cm^{-1}) showed the incidence of imine moiety (C=N) which confirms the leaf extract having enzymes or proteins [25]. The band at 477.41 cm^{-1} approves the existence of Zn-O vibrations [26]. FT-IR analysis confirmed the presence of functional groups in the capping agent and also the formation of ZnO NPs. FT-IR spectra of green synthesized ZnO NPs was represented in **Figure 2**.

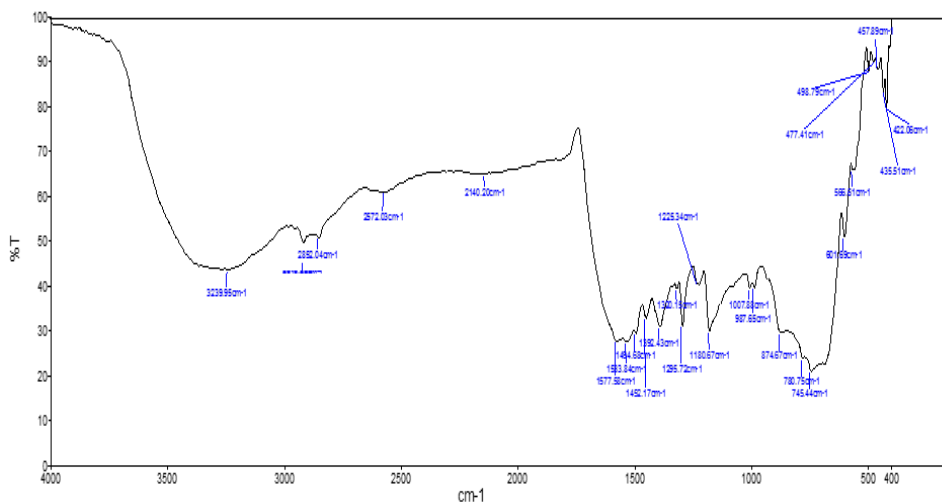


Figure 2. FT-IR spectra of ZnO nanoparticle

SEM and Mapping studies ZnO nanoparticle

Scanning Electron Microscopy (SEM) investigation was performed to govern the size and morphology of the green synthesized ZnO NPs by *Chenopodium album* (aqueous leaf extract). SEM image shown in Figure 3 confirmed that the obtained ZnO NPs were sponge like

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shaped. The green synthesized ZnO NPs were dispersed as distinct particles and mono dispersivity in nature. Phytochemicals in *Chenopodium album* aqueous leaf extract turn as a capping agent which prevents the aggregation of particles causes mono dispersivity of ZnO NPs. SEM mapping studies also conforms the synthesized nanoparticle was ZnO. The green dots corresponds to Zinc atom and red dots represents Oxygen atom. Figure 4 represents the SEM mapping studies of ZnO nanoparticle.

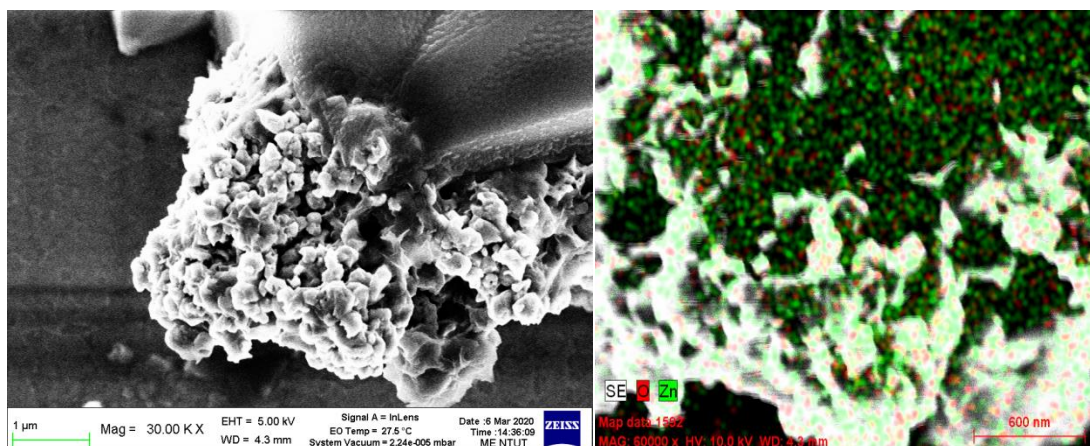


Figure 3. SEM image of ZnO nanoparticle. Figure 4. SEM image mapping of ZnO nanoparticle

EDX Analysis of ZnO nanoparticle

The elemental composition of the synthesized ZnO NPs was confirmed by EDX analysis. The manifestation of zinc and oxygen peaks in the EDX spectra confirmed that the synthesized material was ZnO NPs (Figure 5). The weight percentage of Zinc and Oxygen atoms were 69.25 and 18.70 respectively. The further peaks extant in the spectra may be as a result of the existence of bio organics or impurities in the solution. The elemental composition of ZnO nanoparticle was represented in Table 1.

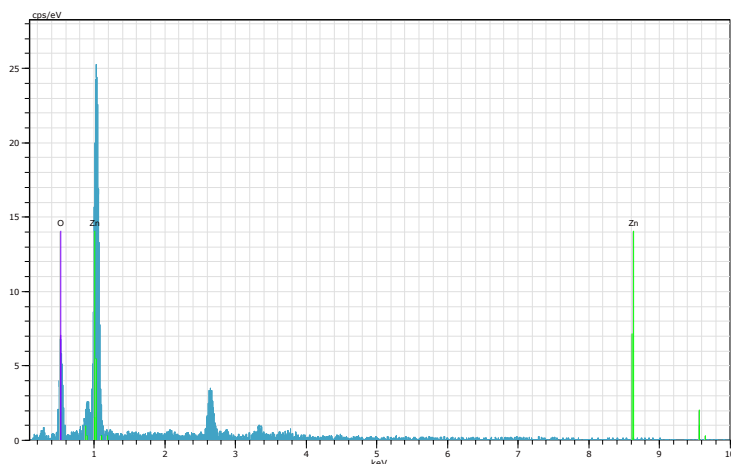


Figure 5. EDX spectra of ZnO nanoparticle.

Table 1. Elemental composition of ZnO nanoparticle

Element	Atomic Number	Weight %	Atom %	Weight % Error
O	8	18.70	52.46	4.5
Zn	30	69.25	47.54	4.6
Total	-	87.95	100.00	-

XRD Analysis

The XRD pattern of aqueous leaf extract of *Chenopodium album* derived ZnO NPs was represented in Figure 6. The diffraction peaks at $2\theta = 28.3^\circ, 31.7^\circ, 34.5^\circ, 36.3^\circ, 37.9^\circ, 44.9^\circ, 54.1^\circ, 56.7^\circ, 58.3^\circ, 61.1^\circ, 62.9^\circ$ and 67.9° were respectively indexed to (100), (002), (101), (104), (102), (110), (103), (200), (112), (201), (004) and (202) planes of hexagonal wurtzite structure of ZnO NPs. The obtained diffraction peaks were matched with of standard ZnO NPs. All the

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diffraction peaks are in good agreement with the standard pattern for pure face centered cubic phase of copper nanoparticles (JCPDS No. 043-0002). There is some impurity peaks were observed. The intense peaks indicate the highly crystalline nature of the formed nanoparticles. From the observed main diffracted peak, the average crystalline size can be calculated using the Scherer equation

$$D_{(hkl)} = \frac{k\lambda}{\beta \cos\theta}$$
 Where, $D_{(hkl)}$ is the average crystalline size, k is shape constant (0.89), λ is the wavelength of the incident x-ray (Cuk α source, $\lambda = 0.15405$ nm), β is the full width half maximum (FWHM), θ is the incident angle of x-ray. The average crystallite size of the synthesized ZnO nanoparticles was 19.52 nm.

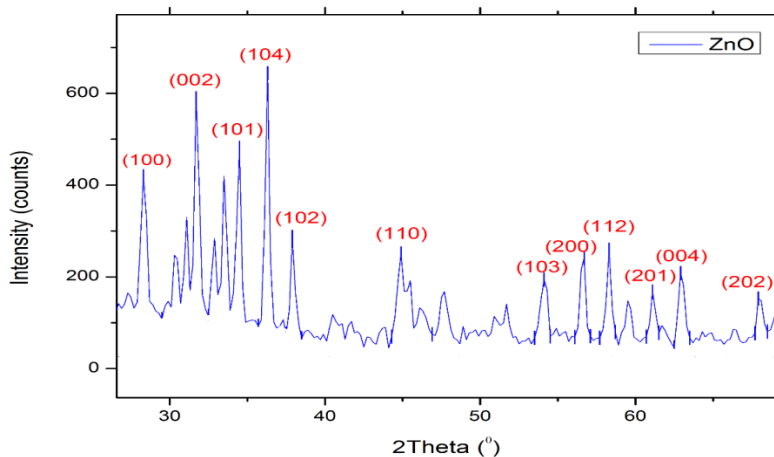


Figure 6. XRD spectra of ZnO nanoparticle.

Larvicidal activity

The synthesized ZnO particle(2) was highly active against *Culex quinquefasciatus* LD₅₀ value of 45.28 μ g/mL than aqueous leaf extract of *Chenopodium album* (1) and control permethrin with the LD₅₀ value of 84.73 and 69.83 μ g/mL. Among the test samples, aqueous

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leaf extract of *Chenopodium album* (1) was less active against *Culex quinquefasciatus* with the LD₅₀ values of 84.73 µg/mL respectively. The synthesized ZnO nanoparticle (2) was highly active and aqueous leaf extract of *Chenopodium album*(1) was moderately active compared to the positive control *Permethrin* with the LD₅₀ value of 69.83 µg/ml. The values are summarized in Table 2.

Table 2. Larvicidal activity of aqueous leaf extract of *Chenopodium album* (1)and synthesized ZnO particle (2)

Comp.No.	Mortality (%)Room temp				LD ₅₀ (µg /mL)
	Concentration(µg /mL) ^a				
	100	75	50	25	
1	60± 1.64	30± 0.54	10± 0.34	0± 0.00	84.73
2	80± 1.44	60± 1.30	40± 1.43	20± 1.29	45.28
Permethrin	68± 0.67	40 ± 1.29	24± 1.78	12± 0.98	69.83
DMSO	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0± 0.0

^aValue were the means of three replicates ± SD.

Conclusion

- In conclusion the zinc oxide nanoparticle was synthesized using fresh leaf extract of *Chenopodium album*.
- The synthesized nano particle was characterized and confirmed using UV-Visible, FT-IR, XRD, FE-SEM, EDX and SEM mapping analysis, the results showed that ZnO Nps were synthesized properly.
- The larvicidal assay depicts the effective larvicidal activity of ZnO Nps.
- This concludes that the further study on ZnO Nps helps for the insecticide development.

References

- [1] L. M. Rueda, Global diversity of mosquitoes (*Insecta: Diptera: Culicidae*) in fresh water. *Dev. Hydrobiol.*, 595(2008) 477-487.
- [2] A. H. Nour, S. A. Elhoussein, N. A. Osman, A. H. Nour, M. M. Yusoff, A study of the essential oils of four Sudanese accessions of basil (*Oimum basilicum* L.) against *Anopheles* mosquito larvae. *Am. J. Applied sci.*, 6(2009) 1359-1363.
- [3] M. S. Rao, U. S. N. Murthy, B. Gangadasu, B. C. Raju, C. H. Ramesh, S. B. Kumar, V. J. Rao, Larvicidal efficacy of neonicotinoid classes of compounds on *Culex quinquefasciatus*. *J. Entomol.*, 5(2008) 45-50.
- [4] L. S. J. Nerio, E. Olivero-verbal, Stashenko, Repellent activity of essential oils: A review. *Bioresour. Technol.*, 101(2010) 372-378.
- [5] Y. C. Yang, S. G. Less, H. K. Lee, M. K. Kim, S. H. Less, H. S. Lee, A piperidine amide extracted from *Piper longum* L. fruit shows activity against *Aedes aegypti* mosquito larvae. *J. Agri. Food. Chem.* 50(2002) 3765-3767.

- [6] A. W. A. Brown, Insecticide resistance in mosquitoes a pragmatic review. *J. Am. Mosq. Contr. Ass.* 22(1986) 123-139.
- [7] N. G. Das, D. Goswami, B. Rabha, *J. Vect. Borne Dis.* 44(2007) 145-148.
- [8] T. L. Russell, B. H. Kay, G. A. Skilleter, Environmental effects of mosquito insecticides on saltmarsh invertebrate fauna. *Aquatic. Biology.* 6(2009) 77-90.
- [9] M. Z. Ahmad, S. Akhter, G. K. Jain, M. Rahman, S. A. Pathan, F. J. Ahmad, R. K. Khar, Metallic nanoparticles: technology overview & drug delivery applications in oncology. *Expert. Opin. Drug. Deliv.* 7 (8)(2010) 927-42.
- [10] Siavash Irvani, 'Green synthesis of metal nanoparticles using plants', *Green Chemistry*, vol.1, (2011) pp.2638.
- [11] Narendra Kulkarni & Uday Muddapur, 'Biosynthesis of Metal Nanoparticles: A Review', *Journal of Nanotechnology*, vol. 2014, Article ID 510246, pp.8.
- [12] AK. Jha, K. Prasad, AR. Kamlesh Prasad & Kulkarni, 'Plant system: nature's nanofactory. *Colloids and Surfaces B: Biointerfaces*', vol.73, no.2, (2009) pp.219-223.
- [13] S. Shankar, A. Ahmad, & M. Sastry, 'Geranium leaf assisted biosynthesis of silver nanoparticles', *Biotechnology Progress*, vol.19,(2003) pp.1627-31.
- [14] M. Vanaja, K. Paulkumar, M. Baburaja, S. Rajeshkumar, G. Gnanajobitha, C. Malarkodi, M. Sivakavinesan, & G. Annadurai, 'Degradation of Methylene Blue Using Biologically Synthesized Silver Nanoparticles', *Bioinorganic Chemistry and Applications*, vol. 2014, Article ID 742346, (2014) pp.8.
- [15] Naheed Ahmad & Seema Sharma, 'Green Synthesis of Silver Nanoparticles Using Extracts of *Ananas comosus*', *Green and Sustainable Chemistry*, vol.2, (2012) pp.141-147.

- [16] SS. Shankar, A. Rai, B. Ankamwar, A. Singh, A. Ahmad, & M. Sastry, 'Biological synthesis of triangular gold nanoparticles', *Nature Materials*, vol.3, no.7, (2004) pp.482-488.
- [17] NC. Sharma, SV. Sahi, S. Nath, JG. Parsons, JL. Gardea-Torresdey, & T. Pal, 'Synthesis of plant-mediated gold nanoparticles and catalytic role of biomatrix-embedded nanomaterials', *Environmental Science and Technology*, vol.41, no.14, (2007) pp.5137-5142.
- [18] Chandan Tamuly, Moushumi Hazarika, Jadumoni Das, Manobjyoti Bordoloi, Dipankar J Borah & RD. Manash, 'Bio-derived Cu nanoparticles for the photocatalytic treatment of dyes', *Materials Letters*, vol.123, (2014) pp.202-205.
- [19] Li Fu & Zhuxian Fu, 'Ilex amboinensis leaf extract-assisted biosynthesis of ZnO nanoparticles and their photocatalytic activity', *Ceramics International*, vol.1, no.2, (2015) pp.2492-2496.
- [20] M. Sundrarajan, & S. Gowri, 'Green synthesis of titanium dioxide nanoparticles from Nyctanthes Arbor-tristis leaf extracts', *Chalcogenide Letters*, vol.8, no.8, (2011) pp. 447-451.
- [21] JK. Andeani, & S. Mohsenzadeh, 'Green synthesis of cadmium oxide nanoparticles from Achillea wilhelmsii flowers', *Journal of Chemistry*, vol.2013, Article ID 147613, pp.4.
- [22] SR. Senthil Kumar, 'Green synthesis of Silver (Ag) and Zinc oxide (ZnO) nanoparticles and studies on phytochemicals and antioxidants and antimicrobial potentials of Indian green tea (Camellia sinensis (L.) Kuntze)', Department of Botany, Annamalai University, Tamil Nadu, India, Ph.D Thesis, Chapter 1, (2014) pp.4.
- [23] B. M. Marsh, J. Zhou, E. Garand, Vibrational spectroscopy of isolated copper(II) complexes with deprotonated triglycine and tetraglycine peptides, *RSC Adv.*, 2015,5, 1790-1795.

Research Paper

- [24] G. Xiong, U. Pal, J. G. Serrano, K. B. Ucer, R. T. Williams, Photoluminescence and FTIR study of ZnO nanoparticles: the impurity and defect perspective, *phys. stat. sol. (c)* 3, No. 10, 3577–3581 (2006)
- [25] K. Handore, B. Kalpana, H. Sanjay, C. Amit, M. Prakash, A. Kakasaheb, P. Jalinder, N.C. Vasant, Novel Green Route of Synthesis of ZnO Nanoparticles by Using Natural Biodegradable Polymer and Its Application as a Catalyst for Oxidation of Aldehydes, *Journal of Macromolecular Science Part A Pure and Applied Chemistry*. 2014, 51, 941-952.