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One Pot Synthesis of Zinc Oxide Nanoparticles via Chemical and Green Method

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Abstract

Presently the progress of green chemistry in the synthesis of nanoparticles with the use of plants has engrossed a great attention. This study reports the exploit of aqueous leaf extract of Corriandrum sativum as an eco-friendly agent for the pattern of Zinc Oxide nanoparticle using zinc acetate and sodium hydroxide as a surrogate for Chemical method. The present exploration describes the synthesis and characterization of ZnO nanoparticles prepared by green and chemical technique (XRD, SEM, FTIR and EDAX). ZnO nanoparticles have found fabulous application in biomolecular detection, diagnostics, micro electronics and water remediation. Though chemical and green methods are trendier for nanoparticles synthesis, the biogenic green fabrication is a better choice due to eco-friendliness.

Keywords: *Corriandrum sativum,* Characterization, Zinc oxide nanoparticles, Zinc acetate.

Introduction

Green synthesis techniques make use of moderately pollutant free chemicals to synthesis nanometerials and embrace the use of benign solvents such as water, natural extracts. Green chemistry seeks to reduce pollution at source[1, 2]. It is enhanced to prevent waste than to treat or clean up waste after it is formed. This principle focuses on choosing reagents that facade the least risk and generate only benevolent by products. Though physical and chemical methods are trendier for nanoparticles synthesis, the biogenic fabrication is a better choice due to eco-friendliness[3, 4]. Nanoparticles due to their smaller size and large surface to volume ratio exhibit remarkable novel properties and methodical applications in the field of biotechnology, sensors, medical, catalysis, optical devices, DNA labeling, drug delivery[5] and they are rewardingly treated as a bridge between bulk material and atomic and molecular structures. ZnO nanoparticles have found fabulous application in biomolecular detection, diagnostics, and micro electronics[6]. Green synthesis of ZnO nanoparticles were agreed out using *Corriandrum Sativum* leaf extract for the eco-friendly development of novel technologies. We have developed a facile and eco-friendly method for the synthesis of Zinc Oxide nanoparticles using aqueous leaf extract of *Corriandrum Sativum* with Zinc acetate dihydrate as precursor. ZnO have extensive applications in water purification[7]. ZnO nanoparticles have been used to remove arsenic, sulphur from water even though bulk zinc oxide cannot absorb arsenic. It is because nanoparticles have much larger surface areas than bulk particles[8]. The plant phytochemical with antioxidant properties is accountable for the preparation of metal and metal oxide nanoparticles. Recently nano particles synthesis was achieved with *bacteria, fungi, actinomycetes*[9, 10, 11] and use of plant extract such as *neem, camellia sinensis, Corriandrum, nelumbo licifera, ocimum sanctum* and several others which is compatible with the green chemistry principles[12,13]. Among the diverse biosynthetic approaches, the use of plant extracts has compensation such as easily available, safe to handle and possess a broad viability of metabolities. The phytochemicals responsible for the synthesis of nanoparticles are terponoids, flavonoids, carbohydrates, saponins, alkaloid and protein[14]. *Coriandrum sativum* also known as cilantro, Chinese parsley or dhania is an annual herb in the family Apiaceae[15]. It is a soft, hairless plant growing to 50 cm. The leaves are uneven in shape, broadly lobed at the base of the plant and slender. The flowers are borne in small umbels, white asymmetrical with the petals pointing away[16]. Genus name comes from the Greek word *koris* (bedbug) in reference to the purported resemblance of the smell of fresh leaves to bedbug infested linen. The therapeutic properties[17] of Coriander include being, aphrodisiac, carminative, depurative, digestive, analgesi fungicidal, revitalizing, antispasmodic, stomachic and stimulant. Coriander is constructive to refresh and awake the mind. It can be used for mental fatigue, migraine pain, tension and nervous weakness[18-25]. Approaches such as simple solution-based methods, chemical precipitation[26,27], sol-gel, solvothermal[28-31] electrochemical and photochemical reduction techniques are more widely used[32, 33]. Chemical method leads to the presence of some toxic chemicals adsorbed on the surface that may have adverse effects[34]. Increasing awareness towards green chemistry and biological processes has led to the development of an eco-friendly approach for the synthesis of nanoparticles. The use of environmentally benign plant leaf extract[35-38] for the synthesis of zinc oxide nanoparticle offers copious profit of eco-friendliness where toxic chemicals are not used[39, 40]. ZnO is non toxic it can be used as photocatalytic degradation materials of environmental pollutants.

Material and Methods

Zinc acetate dihydrate (99%purity) and sodium hydroxide (pellet.99%) was used as the introductory material was supplied by Sigma-Aldrich chemicals. A fresh leaf of *Corriandrum sativum* were washed thoroughly with double distilled water, grinded and was filtered through Whatman filter paper was used for further studies. Identification of active phytoconstituents (Table-1) was done by the methods of Trease and Harbourne[41, 42].

Table-1
Qualitative Phytochemical Analysis of *Corriandrum sativum*

S.No	Phytoconsitituents	Reagents	Aqueous
1	Alkaloids	Mayer's	+
		Wagner's	+
2	Carbohydrates	Molisch's	+
		Benedict's	+
3	Glycosides	Legal's	+
		Borntrager's	+
4	Steroid	Libermann burchard's	+
5	Fixed oils	Spot test	+
6	Saponins	Gelatin	-
		Lead acetate	-
7	Tannins	Ferric chloride	+
		Wagner's	+
8	Protein	Millon's	+
		Biuret	+
9	Flavonoids	Alkaline Reagent	+
		Shinoda's	+
10	Terpenoids	Thionyl chloride	+

+ Presence **-** Absence

ZnO nano particle was prepared by two different methods. In synthesis I (Green synthesis method) to 50 ml of distilled water 0.02M aqueous Zinc acetate dihydrate was added under constant stirring. Aqueous leaf extract of *Corriandrum* were introduced into the above solution after 10min stirring at different sets (0.25,0.5,1ml).To the same 2.0M NaOH was added to make pH 12 resulted in a pale white aqueous solution . This was then placed in a magnetic stirrer for 2hrs. The pale white precipitate was then taken out and washed over and over again with distilled water followed by ethanol to get free of the impurities. Then a pale white powder of ZnO nanoparticles was obtained after drying at 60˚C in vacuum oven over night. In synthesis II (Chemical method), 0.02M aqueous Zinc acetate dihydrate was dissolved in50 ml distilled water under vigorous stirring. At room temperature, aqueous 2.0M NaOH was added drop by drop to reach pH 12. Which was then placed in a magnetic stirrer for 2hr.After completion of reaction, the white precipitate formed was washed thoroughly with distilled water followed by ethanol to remove the impurities. The precipitate was dried in a hot air oven for overnight at 60˚C. Complete conversion of $Zn(OH)_2$ into ZnO NPs took place during drying.

The external morphology and particle size of the sample were characterized by (SEM) (LEO 1530FEGSEM) Scanning Electron Microscope. FT-IR spectra (Fourier Transform Infrared Spectrometer) were recorded on Jasco FT-IR5300 model spectrophotometer in KBr pellets in the range of 4000-400 cm^{-1} for Green and Chemical methods. Crystal phase identification of the samples were characterized by powder X-Ray Diffractometer (XRD, PW 3040/60 Philips X'Pert, Holland) with Cu (Kα) radiation (λ =0.15416 nm) operating at 40 kv and 30 mA with 2θ ranging from 10- 90˚ for Chemical and Green method.

Results and discussion

The end product in Green synthesis method- I was pale white precipitate where in chemical synthesis method –II white precipitate appeared. The X-Ray powder Diffraction pattern of the synthesized sample from aqueous leaf extract of *Corriandrum sativum* by Green synthesis method – I was recorded on an X-ray diffractometer using Cu(kα) radiation ($\lambda = 1.5415 \times 10^{-10}$) operating at 40 kv and 30 mA with 2θ ranging from 10˚-90˚ figure-2 shows distinctive peak at (100), (002), (101), (102), (110), (103), (200), (112) and (201) that are in good agreement with wurtzite ZnO(JCPDS CARD NO: 36- 1451)[43]. High purity and crystallinity of the prepared ZnO NPs confirms the sturdy and clear peak. For other impurities no characteristic peak was accessible[44]. Zinc oxide NPs synthesized by Chemical method -II using X-ray diffraction of the Figure-3 using zinc acetate dihydrate and sodium hydroxide shows 2θ values at 32.1, 34.7, 36.6, 47.9, 56.9, 63.2, 66.7, 68.3, 69.4, 77.3 equivalent[45] to (100), (002), (101), (102), (110), (103), (200), (112), (201) and (202) planes in that order. The results confirmed that the ZnO nanoparticles are of wurtzite hexagonal type structure[46]. Using Debye Scherer's equation $d = k\lambda / \beta \cos\theta$ the average particle size obtained for ZnO nanoparticle is 66nm in Green synthesis method -I while the size was found to increase to 81nm in Chemical method – II.

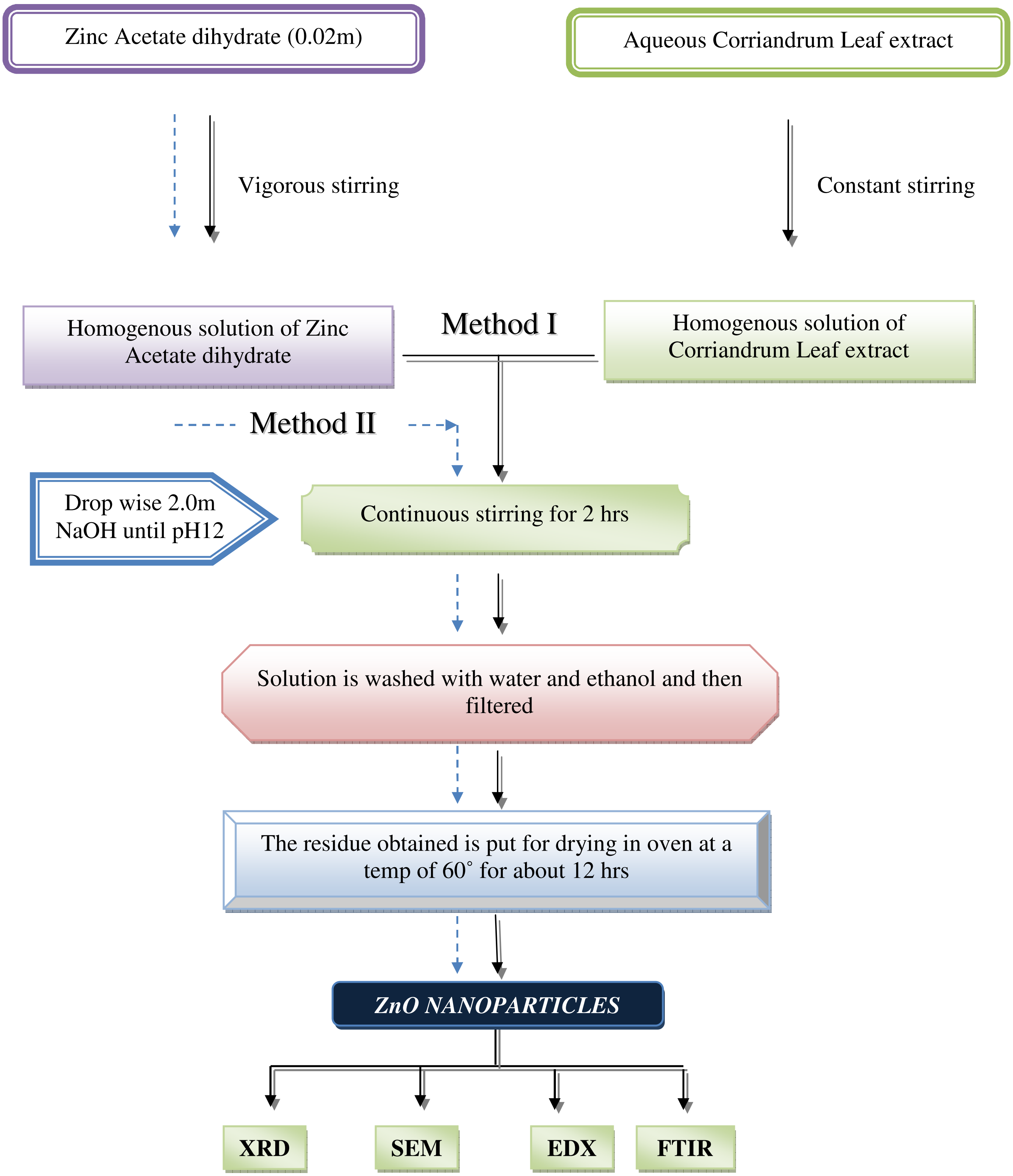


Figure-1
Synthesis by Green and Chemical Method

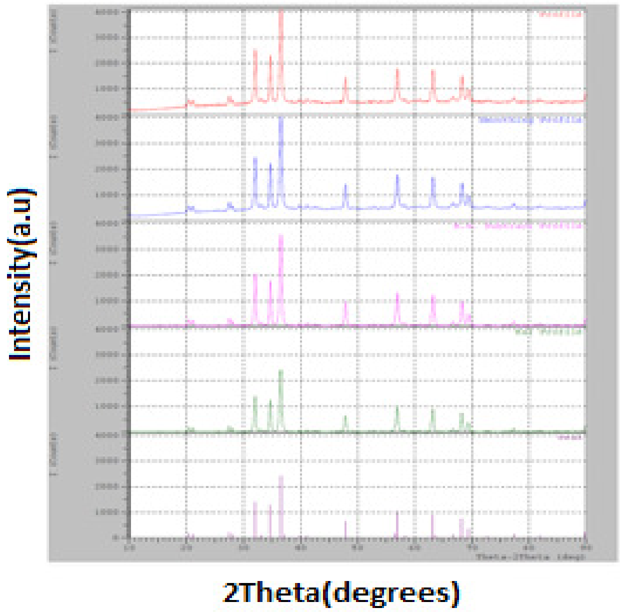


Figure-2
XRD spectra of ZnO nps of aqueous leaf extract of *Corriandrum sativum* by Green synthesis method-I

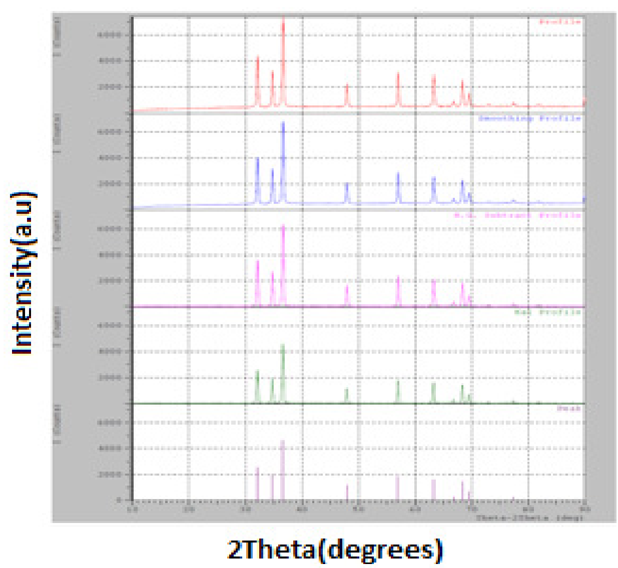


Figure-3
XRD spectra of ZnO nps using Chemical method-II

The samples synthesized by green (method I) and chemical (method II) separately were analysed on a Jasco FT-IR5300 model operating at a resolution of 4000-400cm^{-1} in the percent transmittance mode. Figure-4 shows the FT-IR spectra of ZnO nanoparticles prepared from aqueous coriander leaf extract by green synthesis method-I. The pattern of absorption at 710 cm^{-1} and 768 cm^{-1}, 849 cm^{-1} aromatic C-H out of plane bending is a typical mono substituted benzene ring 1,2,3 tri-substituted benzene ring and 1, 4 di-substituted benzene ring. Band 1029 cm^{-1} corresponds to C-N stretching vibration of amine[47].

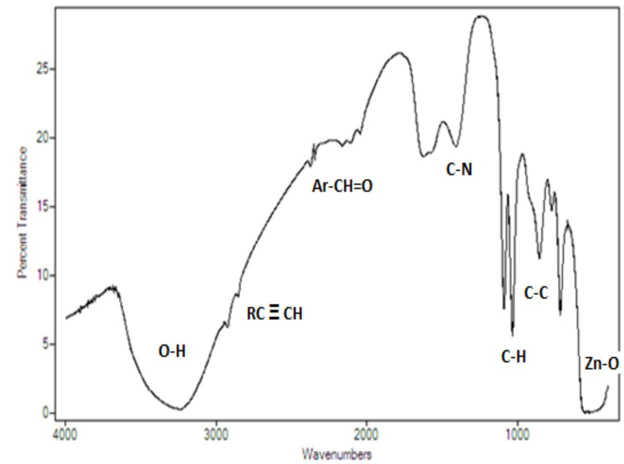


Figure-4
FT–IR spectra of ZnO nps of aqueous leaf extract of *Corriandrum Sativum;*

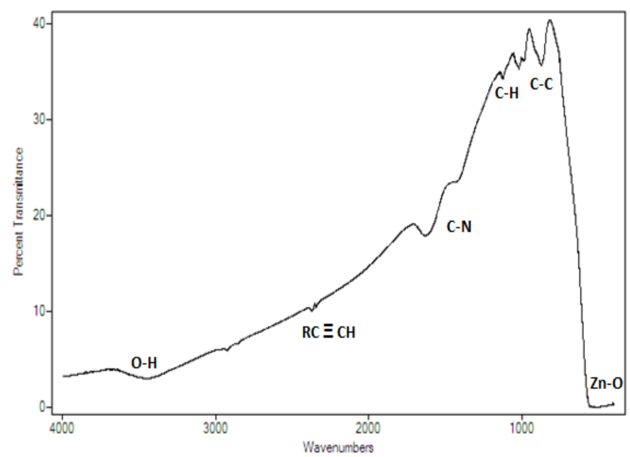


Figure-5
FT–IR spectra of ZnO nps using chemical method

Medium absorption in the region 1617- 1411cm^{-1} often implies an aromatic ring. Two weak absorptions near 2900-2750 cm^{-1} indicates the presence of aromatic aldehydes[48]. The weak absorption at 2049 is due to CΞC stretching vibration. The broad and intense band at 3237-3565 cm^{-1} is owing to OH stretching. Absorption at 450-540cm^{-}1 identifies the presence of ZnO nanoparticle. Broad IR bands at 3237, 1614, 2900 cm^{-1} show the presence of hydroxyl group, aldehydes, amines and aromatic ring confirms that the phytoconstituents could possibly enhance the stabilization of ZnO nanoparticles in the aqueous medium. Figure-5 shows the FT-IR spectra of ZnO nanoparticles prepared for chemical method-II. Intense, broad band near 3360 cm^{-1} represents the hydrogen bonded O-H stretching vibration[49]. The band at 2990 cm^{-1} represents asymmetric $C_{sp}{}^{3}$-H stretching frequency of alkyl group. The band at 540- 417 cm $^{-}$1 point out ZnO nanoparticles[50-52]. Regardless of repeated washing the surveillance proves the subsistence of aldehydes, amines, terpenoids, phenolic compounds were bounded to the surface of ZnO nanoparticle enhances stabilization by covering the metal nanoparticles[46]. The physicochemical properties of *Corriandrum sativum* acts as a bio template which prevents the particles formed from aggregating.

SEM image Figure-3 (a,b,c,d) by green synthesis method-I dogged the particle size and external morphology of the ZnO nanoparticle . It can be seen from the image that the zinc oxide nanoparticles range from 100-190 nm. Figure-3c and 3d shows cubic structure composed of quite a lot of individual small nanoparticles. The morphology of ZnO nanoparticle prepared by chemical method-II is shown in the Figure-6 (e,f,g,h) represents that the obtained products are composed of near flower shape morphology with the average size in the range of 0.5µm. The profile (3g) of .5µm clearly pictures like a flower. Nanoparticles in profile (6e, 6f) have been agglomerated into bigger ones. Profile (g, h) confirms the structure of nanoflowers of 100-200nm which is in close agreement with green method.

To further confirm the structure elemental analysis of aqueous extract of *Corrianndrum* ZnO NPs by green synthesis-I and Chemical method -II was carried. EDX spectrum Figure-7 peaks of zinc and oxygen elements 55.92 and 44.08% proves ZnONPs prepared is essentially free from impurities. EDX pattern displayed in Figure-8 indicates that the as-prepared products of chemical method are composed of zinc and oxygen of 68.30 and 31.70 %. We have developed a green and chemical route for zinc oxide nanoparticles synthesis. But preparation of zinc oxide nanoparticles using *Corriandrum sativum* is being ecofriendly and can be an effective alternative for the large scale synthesis of zinc oxide nanoparticles. Among the diverse synthetic approaches, the plant phytochemical with antioxidant property is accountable for the preparation of Zinc oxide nanoparticle as the byproducts are benign. . Green method provides progression over chemical method as it is cost effective, environment friendly and scaled up for large scale synthesis.

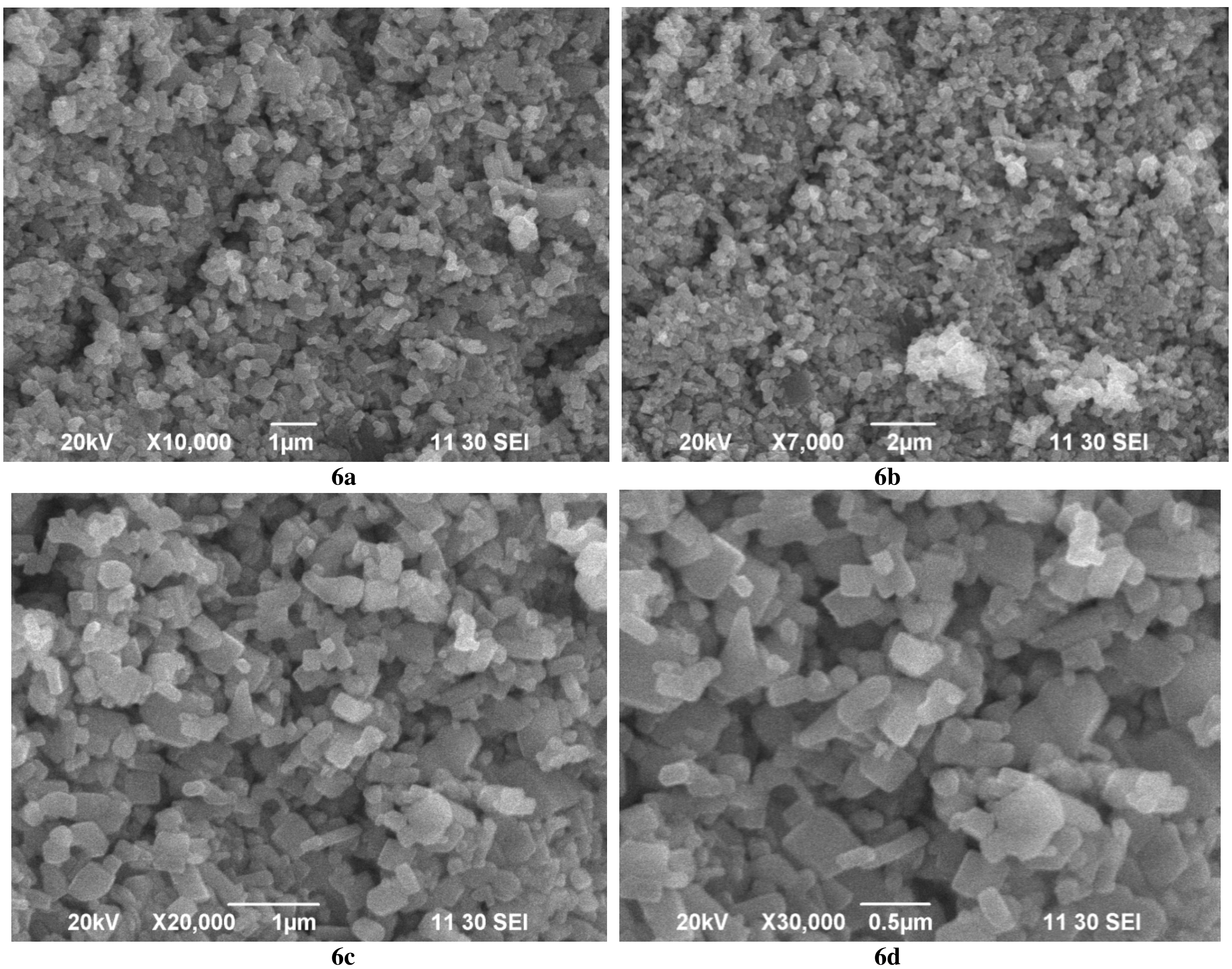
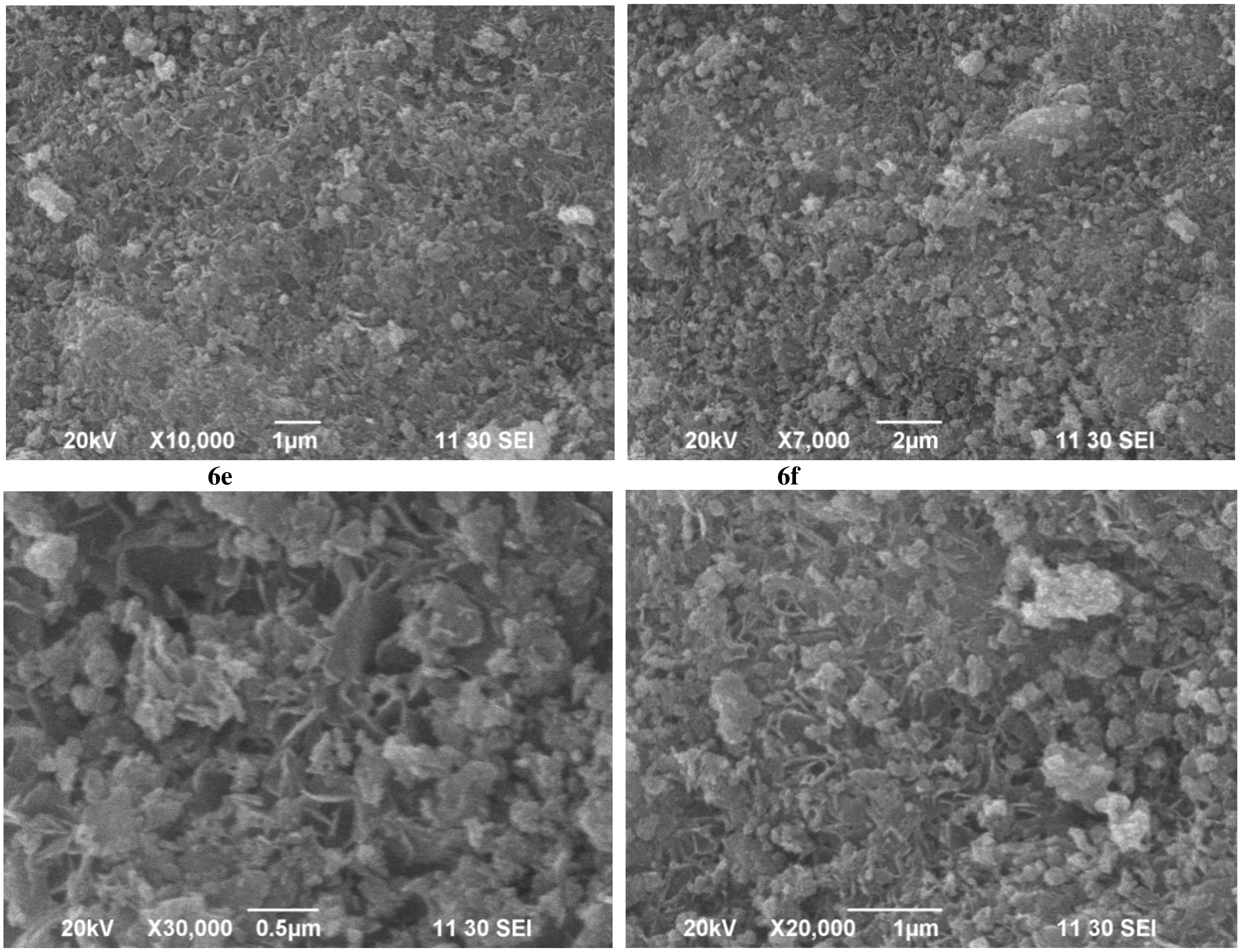


Figure-6 (a,b,c,d)
SEM pictures of aqueous extract of *Corriandrum* ZnO NPs at different magnifications by green synthesis method-I



6e 6f

6g 6h

Figure-6 (e, f, g, h) SEM Image of synthesized ZnO NPs using chemical method-II

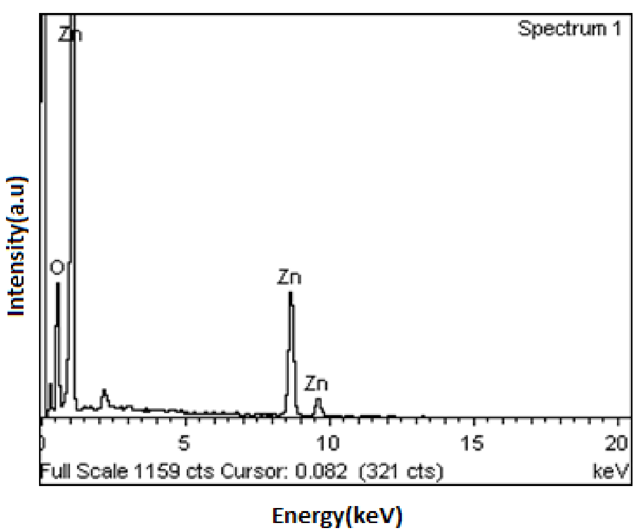


Figure-7
Elemental Spectra of aqueous extract of *Corrianndrum* ZnO NPs by Green synthesis method-I revealed by EDX analysis,

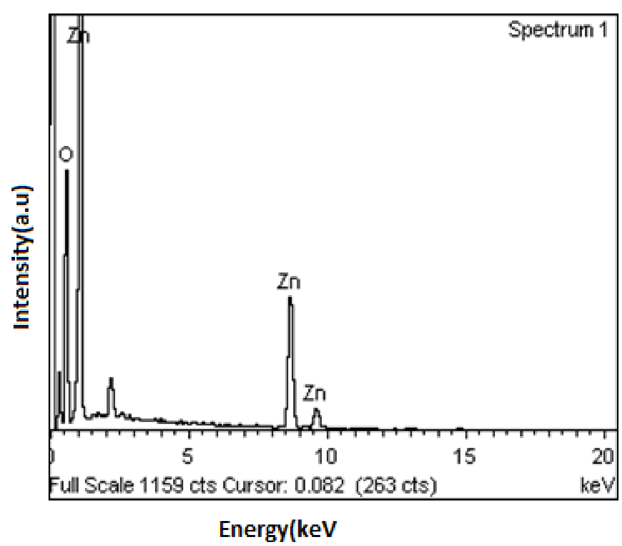


Figure-8
EDX spectra of ZnO nps using Chemical method-II

Conclusion

FT-IR studies of aqueous *Corriandrum* leaf extract reveals the presence of phytoconstituents like alcohol, aldehyde and amine which were the surface active molecules stabilized the nanoparticles and this phytochemicals have interacted with the zinc surface and aids in the stabilization of zinc oxide nanoparticles. The plant phytochemical with antioxidant properties is accountable for the synthesis of Zinc oxide nanoparticles. XRD study reveals that the average size was 66nm in Green synthesis method -I while the size was found to increase to 81nm in Chemical method – II. Though chemical and green methods are trendier for nanoparticles synthesis, the biogenic green fabrication is a better choice due to eco-friendliness. The explored eco-friendly high efficient ZnO nanoparticles prepared from *Corriandrum* leaf extract are expected to have more extensive application in biotechnology, sensors, medical, catalysis, optical devices, DNA labeling, drug delivery and water remediation.

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