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Synthesis And Bio-Physical Characterization Of Mg Doped Zn Nanoparticles From Novel Plant

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Abstract
There is an increasing commercial demand for nanoparticles due to their wide applicability in various areas such as electronics, catalysis, chemistry, energy, and medicine. Metallic nanoparticles are traditionally synthesized by wet chemical techniques, where the chemicals used are quite often toxic and flammable. In this work, we describe a cost effective and environment friendly technique for green synthesis of magnesium doped zinc nanoparticles using the plant extract of Calotropis. Calotropis plant extract used first time as reducing agent as well as capping agent. The magnesium doped zinc nanoparticles were achieved by using high purity metal sulphates and Calotropis plant extract. The synthesis of nanoparticles was confirmed by changing the color from light brown to green within 1 hour of reaction. The further study of formation of magnesium doped zinc nanoparticles was confirmed by UV–Vis, FTIR, XRD, EDX, and SEM. The formation of zinc oxide phase was also confirmed by FT-IR. X-ray diffraction studies confirmed the crystalline nature of the nanoparticles indicating particle size within the range provided by electron microscopy data. The energy dispersive X-ray analysis confirmed the presence of Mg ion in the doped Zn lattice and scanning electron microscopy reveals the spherical morphology of nanoparticles. The antimicrobial activities of nanoparticles (NPs) were studied against fungi, gram-positive and gram-negative bacteria using the well diffusion method. The detailed characterization will be discussed here.
***Keywords: -** Green synthesis, Magnesium doped zinc nanoparticles, Calotropis*

1- INTRODUCTION

Nanobiotechnology represents the connection of nanotechnology and biotechnology which is an emerging field [1] and an important area of research for the synthesis of nanoparticles due to variety of their special characteristic such as their small dimensions, large surface area, distribution, high reactivity and morphology of particles [2]with different chemical compositions, size and shapes. In current years, the plant mediated synthesis of metal nanoparticles has received significant attention due to its ease, eco-friendliness and broad photo catalytic activity [3-6] and cost-effectiveness that can simply increase the bulk production of nanoparticles [7]. The rate of biosynthesis of nanoparticles is faster than other conventional methods and produces more stable materials [8-10]. The plants mediated biological synthesis of nanoparticles recommended as valuable and beneficial approach [11-13], safe, dirt free, reliable, bio-compatible, and rapid and lead to ecofriendly byproducts. The character and size of nanoparticles is depending on the type of the plant extract with temperature of oxide formation.

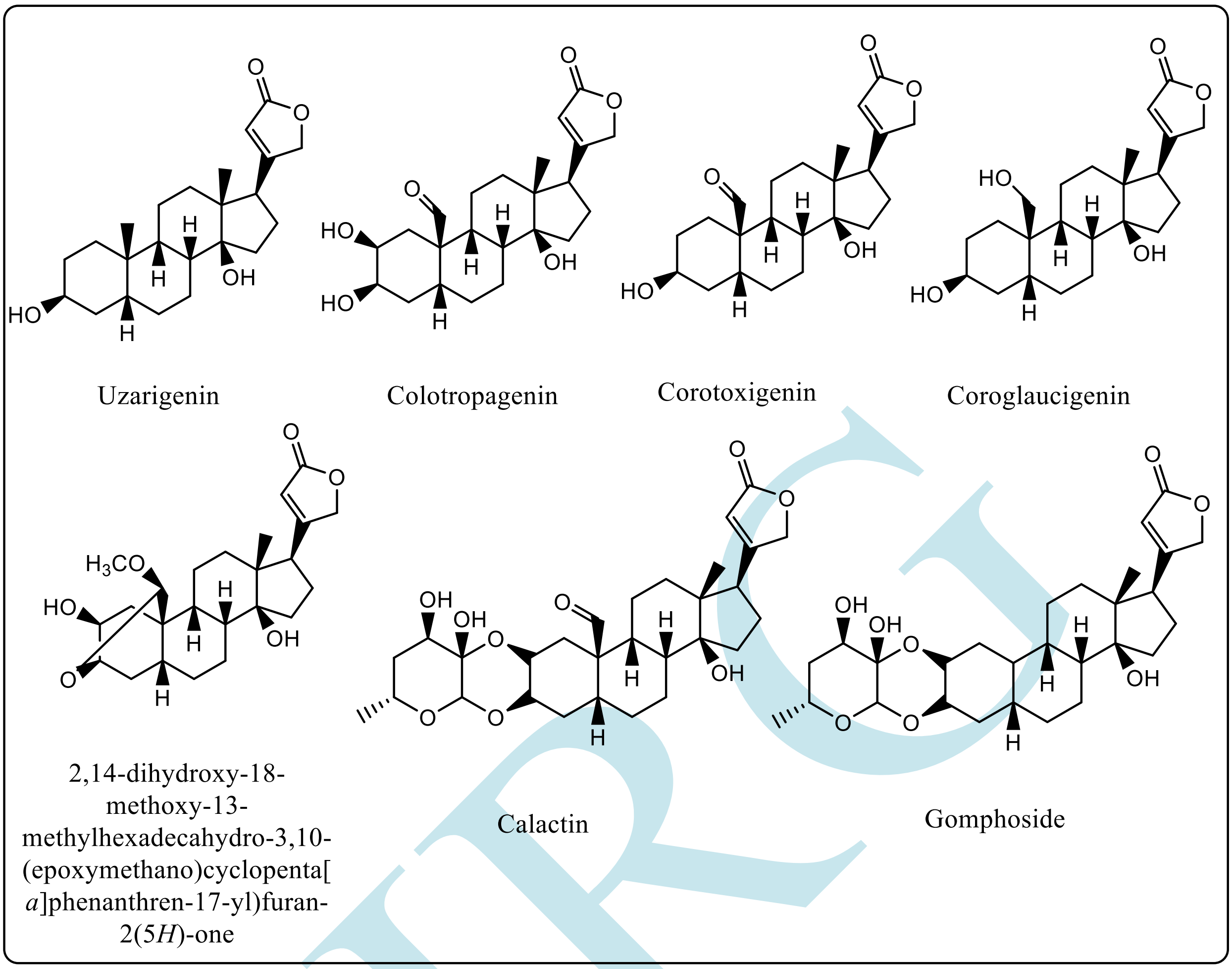

Fig. 1:- Some bioactive drugs isolated from Calotropisgigantean.

The scientific name of the Calotropis is Calotropisgiganteanbelong to the family Asclepiadanceae, also called as Alarka, Shwetarka, Mandara, and Vasuka. Calotropis gigantea is a species of Calotropis native to Cambodia, Indonesia, Malaysia, the Philippines, Thailand, Srilanka, India, China, Pakistan, Nepal and tropical Africa [14-16].Calotropisgigantean has considerable interest due to its biological activities such as antibacterial [17], antifungal [18], antiviral[19], anticandidal [20] and anticarcinogenic activities[21-22]and used for the cure of toothache, earache, headache, sprain, stiff joints. It is a well known cardenolides drug for its cardioactive phytochemic property a wide range of cardenolides drug were isolated from plant Calotropisgigantean (Fig. 1).It has biologically active compounds like cardenolides, triterpenoids, resins, proteolytic enzymes, flavonoids, tannins, sterol, and terpenes which help in the reduction of metal.

Zinc oxide (ZnO) is a potential photo catalyst, due to its band gap energy and stability for the reason that of its band gap, the ZnO has comprehensive applications, including self-cleaning, photo catalysis, environmental purification and high quantum efficiency. The formation of ZnO nanoparticles in which Zn makes complex with plant extract, the method is ecological friendly and sustainable. In this method, ZnO nanoparticles were synthesized through green method, and its band gap values were varying by the Mg content. The effect of Mg doping on the crystal structure of ZnO nanoparticles were using spectroscopic and microscopic techniques. In this present work, we describe the effect of Magnesium doped on the Zinc Oxide nanoparticles by using aqueous extract of Calotropis gigantean. In this synthesis, the zinc oxide nanoparticles were synthesized by using Zinc sulphate as an initial precursor and Calotropisgigantean is used as an reducing and stabilizing agent. The capping agents present in Calotropisgigantean control the size of nanoparticles, increase the surface energy of nanoparticles and prevent it from agglomeration. It has been used for the first time as a reducing material as well as surface stabilizing agent for the synthesis of spherical-shaped ZnO-NPs. The UV-visible (UV-VIS) spectroscopy measured the optical properties of synthesized nanoparticles. The Characterization of synthesized NPs had been done by X-ray diffraction (XRD), Fourier transformed infrared spectroscopy (FTIR),Electron dispersive X ray spectroscopy (EDX).

2- METHODOLOGY

2.1- MATERIAL AND METHODS

The chemicals were used for the Magnesium doped zinc oxide nanoparticlesare of Analytical grade, purified solvents and double distilled water. Magnesium Sulphate (Merck), Zinc Sulphate (Merck), concentrated hydrochloric acid (Merck) was purchased from Hi-media. All the glassware was washed with distilled water before use.

2.2- SYNTHESIS OF MG DOPED ZN NPS

The synthesis method contains two steps –

I. Preparation of Calotropisgigantean plant extract
II. Green synthesis of Mg doped Zn NPs

2.3- PREPARATION OF CALOTROPIS GIGANTEAN PLANT EXTRACT

Fresh leaves of *Calotropis gigantean* plant was collected from Jaipur national university, Campus. The collected leaves were washed under running water 2-3 times to remove the dust particles, then dried in oven at 60°C for 2 hours and converted into powdered form by crushing. 5 gm of crushed powder and 250ml of deionised water was taken into a 500ml beaker. Then, the solution was boiled for 2-3 hrs and was filtered through Whatman filter paper. Hence, that extract was used further for the synthesis of ZnO nanoparticles.

2.4- GREEN SYNTHESIS OF ZNONPS

The zinc oxide nanoparticles were synthesized by adding 50ml of Calotropisgigantean plant extract solution and 5gm of zinc sulphate in a 250ml beaker. The pH value of the solution was adjusted by adding concentrated HCl.The acidic solution of Zinc (II) sulphate and leaf extract of Calotropisgigantean plant extract heated at 60˚C for 2 hrs under vital stirring by using magnetic stirrer. After 2 hours of continuous stirring, solution was allowed to settle down overnight and then centrifuge the solution at 15000 rpm for 10 min to obtain the ZnO nano- particles. The collected nanoparticles were washed repeatedly for 2-3 times with ethanol and water, finally separated nanoparticles were dried in hot air oven at 80˚C and then calcinated at 600˚C for 2 hrs in Muffle Furnace. Now, the particles were grinned by motor pistol to get the finest particle size suitable for further Characterization.

3- RESULT AND DISCUSSION

The Zinc Oxide nanoparticles were obtained by green synthetic method using Magnesium Sulphate **(**$MgSO_4$**)**, Concentrated Hydrochloric acid and Calotropis gigantean Plant extract. The synthesized nanoparticles were characterized by different analytical techniques such as UV-Vis, FTIR, SEM and XRD-EDX.

3.1- UV-VISIBLE ABSORPTION SPECTROSCOPY

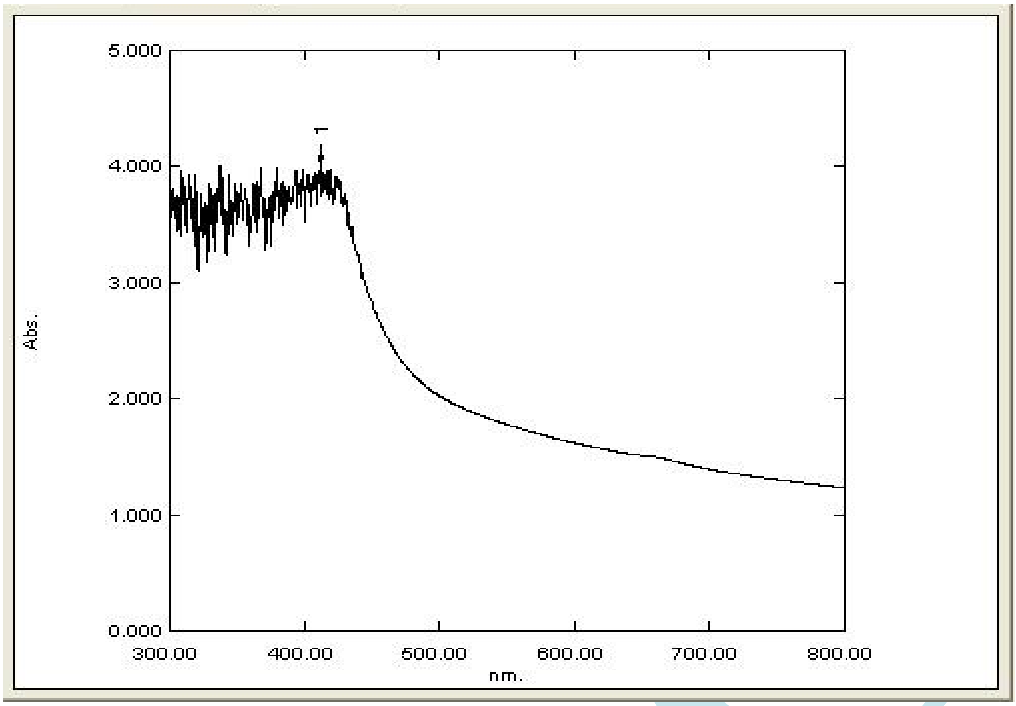

Fig. 2:- UV visible Analysis

The synthesis of zinc Oxide nanoparticles by reduction of aqueous metal ion by Calotropisgigantean plant extract can easily be monitored by using double beam Uv-visible spectrophotometer (Shimadzu Model Number. 1800). The absorption peak obtained in UV-Vis absorption spectra in visible range of wavelength that is 413 nm, (Fig. 2) which confirmed the presence of Zinc. The absorption peak at 413.0 nm confirmed the formation of Zinc oxide nanoparticles.

3.2- FTIR ANALYSIS

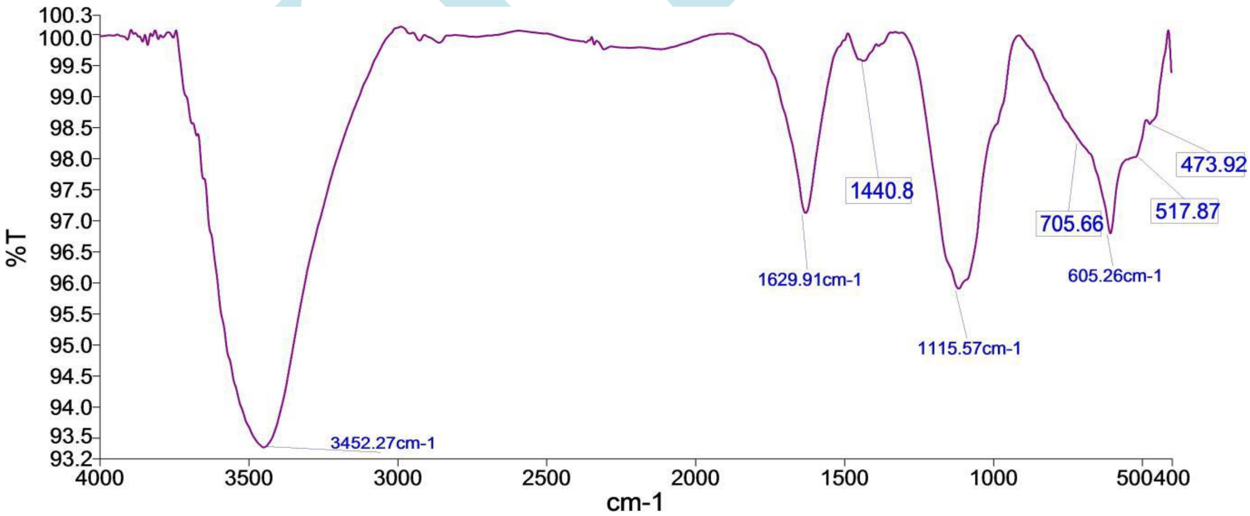

Fig. 3:- FTIR Analysis

The dual role of the plant extract as a bioreductant and capping agent was confirmed by FTIR analysis of the prepared Mg doped ZnNPs. The results of the FTIR spectral analysis of leaf extract of Calotropisgigantean plant and their Mg doped Zn NPs synthesized after the bioreduction recorded in the range of 400 to 4000 cm-1. The KBr technique has been used to record the spectra of ZnO nanoparticles. The band located near 605 cm^{-1} corresponds to the Zn-O stretching mode, 3200 to 3600 cm^{-1} corresponds to the stretching vibration of –OH bond which show peak 3452.27 $cm^{-1}$and at 1630 cm^{-1} has been assigned to the first overtone of fundamental stretching mode of –OH (Fig. 3). Stretching due to vibrations of water molecule gives information of the surface of sample. The band at 1440.8 cm -1 is attributed to the Mg-O stretching vibration and it is absent in case of ZnO.

3.3- X-RAY DIFFRACTION ANALYSIS (XRD)

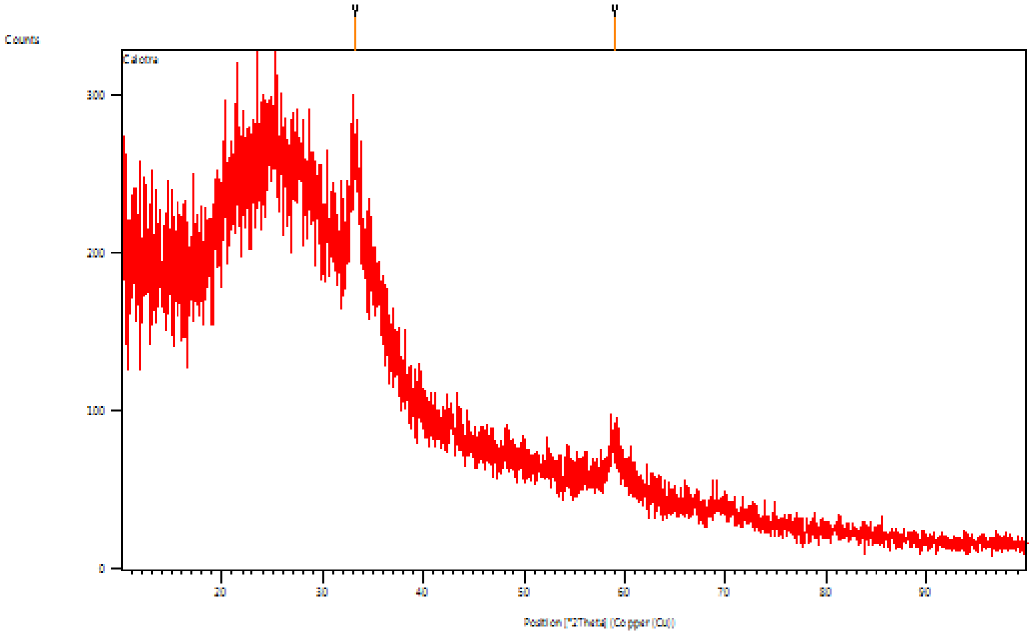

Fig. 4:- X-ray Diffraction Analysis (XRD)

X-ray diffraction analysis identifies the phase and crystalline nature of nanoparticles. It is found that zinc oxidenanoparticles were formed in the form of nanocrystals, and sample exhibited at 2θ = 33.21° and 58.97°are associated with the (111) and (220) crystal planes respectively (Fig. 4).X-ray diffraction results showed that the strong intense and sharp peaks of ZnO indicate that the nanoparticles are highly crystalline in nature. The XRD pattern having only zinc oxide phase and no other impurity peaks were observed. The crystalline size of the pure and Mg doped Zn nanocrystals were projected from higher intense peak of XRD pattern using Debye Scherrer's equation

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

Where D is the crystallite size of ZnNPs, λ is the wavelength of the X-ray source (0.1541 nm) used in XRD, β is the full width at half maximum of the diffraction peak, K is the Scherrer's constant with a value from 0.9 to 1, and θ is the Bragg angle.

3.4- ENERGY DISPERSIVE X-RAY ANALYSIS

Energy Dispersive X-Ray Analysis (EDX), referred to as EDS or EDAX, is an x-ray technique used to identify the elemental composition of materials. The experimental data obtained by Energy Dispersive X-Ray spectroscopy, shows high intense peak of spectra corresponding to the true element, made by the sample.

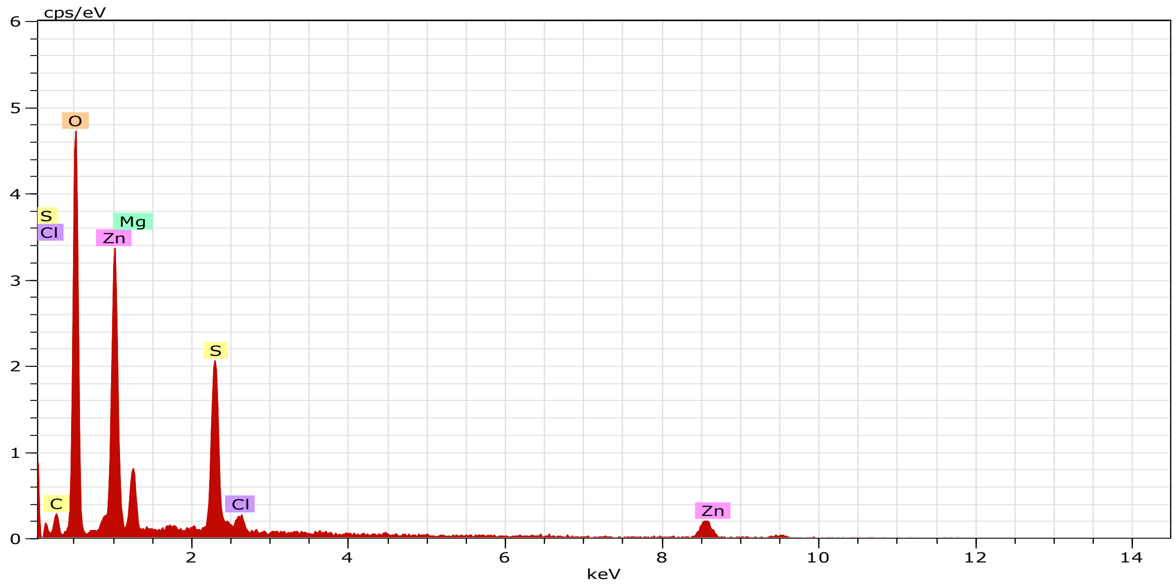

Fig. 5:- Energy Dispersive X-Ray Analysis (EDX)

The data generated by EDX analysis consist of spectra showing peaks corresponding to the elements making up the true composition of the sample being analyzed. The Fig.5 shows the EDAX analysis, confirmed the presence of metallic zinc oxide in biosynthesized ZnO NPs. The composition obtained from EDAX analysis was Zinc, Magnesium, Oxygen, Chlorine, Sulphur and Carbon. The presence of carbon in trace amount indicates the involvement of plant phytochemicals groups in reduction and capping of the synthesized ZnO NPs. (Fig. 5).

3.5- SEM ANALYSIS

The Scanning microscopy images show the formation of Mg doped Zn Nanoparticles using Calotropis gigantean plant extract. The cluster of aggregates at the scale bar of 500 nm and 1µm appears like the spherical shape having particle size less than 100nm confirms the formation of Nanoparticles. The SEM image of the Mg doped Zn sample is shown in (Fig.6,7).

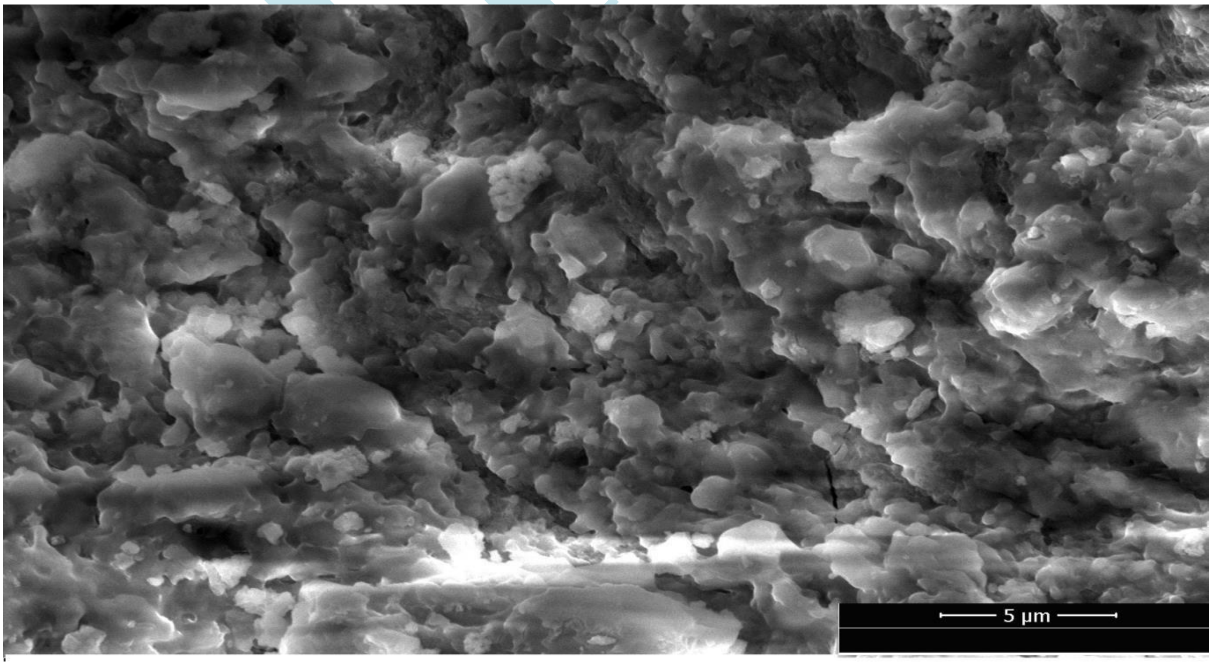

Fig. 6:- SEM images of Mg doped Zn Nanoparticles

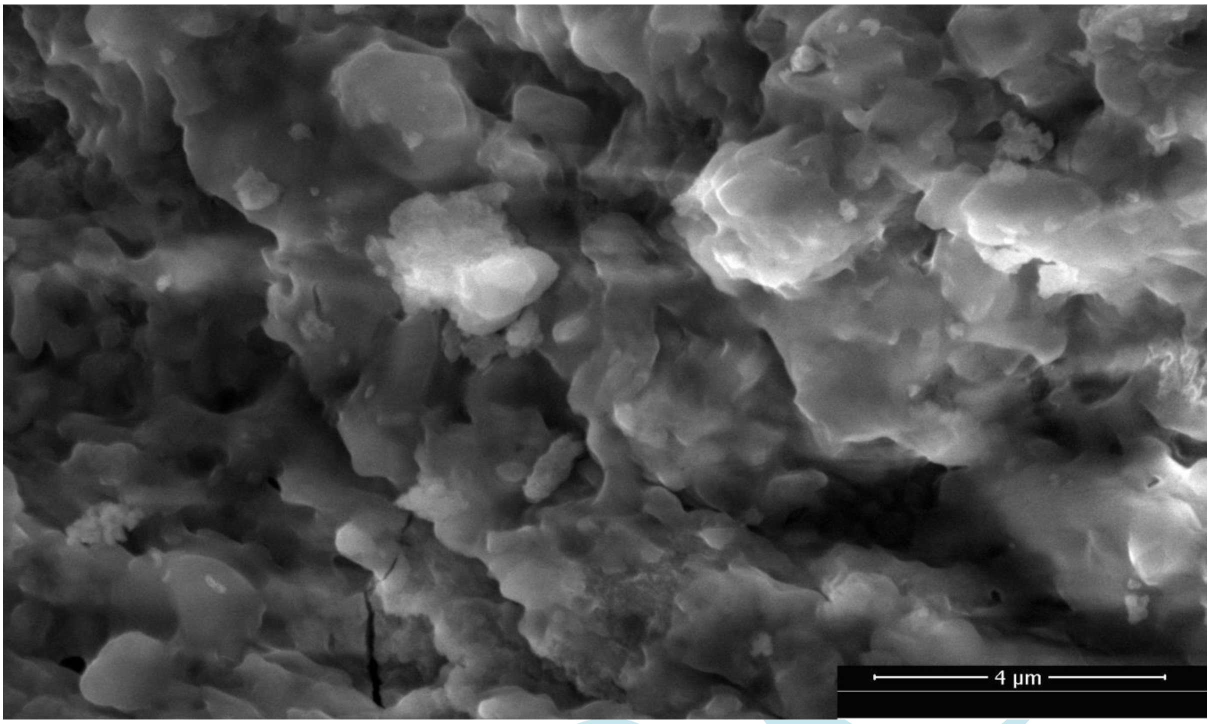

Fig. 7:- SEM images of Mg doped Zn Nanoparticles

4- CONCLUSION

In summary, we have developed convenient, eco – friendly and less hazardous procedure for the synthesis of Mg doped Zn Nanoparticles from novel plant Calotropis giganteanfor the first time. The employed plant plays important role as capping agent on the surface of metal nanoparticles of Mg doped Zn. During this study, the UV – Vis absorption spectra in the visible range of wavelength were observed at 413.0 nm. The further studies FTIR, EDX and XRD, SEM confirmed the formation of zinc oxide nanoparticles.

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