



## GREEN SYNTHESIS OF ZINC CARBONATE HYDROXIDE NANO PARTICLES USING ELEOCARPUS TECTORIS

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### Abstract

The present study was aimed to prepare nano particles using the wild fruit *Eleocarpus Tectoris*[ECT]. The fruit has got so many health benefits and medical benefits. The nano particles was prepared by green synthesis method. The synthesized nano particles were characterized and thus the results were analysed. The particle size was calculated using XRD, and the particles were characterized by other studies like FT-IR, UV. The surface morphology of the particles were estimated using SEM and identification of compounds were studied by EDX. Since it is a wild fruit so the particles were studied for Anti-Microbial activity.

**Keywords:** Green synthesis, *Eleocarpus Tectoris*[ECT], Hydrozincate.

### Introduction

Nanotechnology is an important aspect in the growing research world. Nanoparticles play a major role in upcoming world. Synthesis of nano particles can be done by various methods<sup>[1]</sup>. Nowadays researchers use green synthesis method in various sectors. Zinc carbonate hydroxide was used in chemical industries to remove toxic gases. Zinc carbonate hydroxide or Hydrozincate nano particles has a wide application in the industry sectors<sup>[2]</sup>. There were various chemical and physical methods to synthesis Hydrozincate nanoparticles<sup>[3]</sup>. Green synthesis technique was widely used for the preparation of nano particles due to their low cost and eco-friendly nature<sup>[4]</sup>.

*Eleocarpus Tectoris*(vikki pazham) was mostly carved fruit by Nilgirians, Tamil Nadu, India during the summer seasons. It grows in hilly regions only in summer time from april to june. It was mostly seen in the month of may. It was thick green in colour. The fruit seems like thin layer of fleshy part surrounded on the seed where only the fleshy part is edible, which has a unique taste and a good aroma. The fruit was good for diabetic patients and also for skin allergy. In the present study *Eleocarpus Tectoris*[ECT] was used to prepare Hydrozincate nano particles.

The prepared Hydrozincate nanoparticles were studied for their physical properties like XRD, FT-IR, UV, EDX,SEM and Antibacterial studies.

## 2. Materials and Methods

### 2.1 Collection of Fruit

The fresh fruits of *Eleocarpus Tectoris* (Vikki pazham) were collected from the locality of Ooty, The Nilgiris, Tamil Nadu, India. The collected fruits were washed twice with running tap water to remove the dust and impurities present over it. Then it was washed with distilled water to remove the ions present in the tap water, that was present on the edible part of the fruit. This was done because only the edible part of the fruit was used for characterization studies. The edible part or the pulp of fruit was taken and shade dried at room temperature. It took 3-4 weeks to get dry completely. Then the dried pulps were made into powder using domestic mixer<sup>[5]</sup>. The ECT powder was stored in a airtight



container and used for further process. The ECT image of fresh fruit and the powder were given in Fig.1 and Fig.2 respectively.



**Fig.1: ECT Fruit**



**Fig.2: ECT powder**

## 2.2 Preparation of ECT Aqueous Extract

The aqueous extract of ECT was prepared by reflux method. For refluxing process, the 250ml round bottom flask was washed with tap water and with distilled water to remove the residues present in it. 10g of ECT powder was measured using electronic balance and was mixed with 200ml of distilled water<sup>[6]</sup>. The mixture was refluxed at 80°C for approximately for one hour. The ECT extract was collected and filtered twice using whatmann no. 1 filter paper to remove the impurities<sup>[1]</sup>. The filtered extract was pale yellow in colour, which was given in Fig.3.



**FIG.3: ECT extract**

## 2.3 Preparation of Hydrozincate Solution

0.1M of zinc acetate solution was prepared and the solution was stirred for 30-45 minutes for the salt to get dissolved completely. The ECT extract was added to zinc acetate solution at 1:1 ratio. The mixture was heated and stirred at 520-530 rpm for 1 hour. The colour of the solution has changed from pale yellow to brown. This brown solution was kept for a couple of days without disturbing for the particles to get sediment. The stirring process and the sedimented particles has been given in Fig.4 and Fig.5 respectively.





**FIG.4: Stirring processes of ECT extract Fig.5: Sedimentation of the Hydrozincate particles**

**2.4 Preparation of Hydrozincate Nanoparticles**

The sedimented particles was taken and centrifuged at 1000 rpm for 30minutes. The centrifuged particles were washed with distilled water for the removal of heavier water soluble bio organic molecules. This washed particle was again centrifuged for about 10minutes. The centrifuged particles were collected in a petri dish and dried in oven at 100°c for 15minutes. The sediment dried particles were collected and stored in a air tight vial for further studies. The collected dried particles were brown in colour and have been in given in Fig.6.



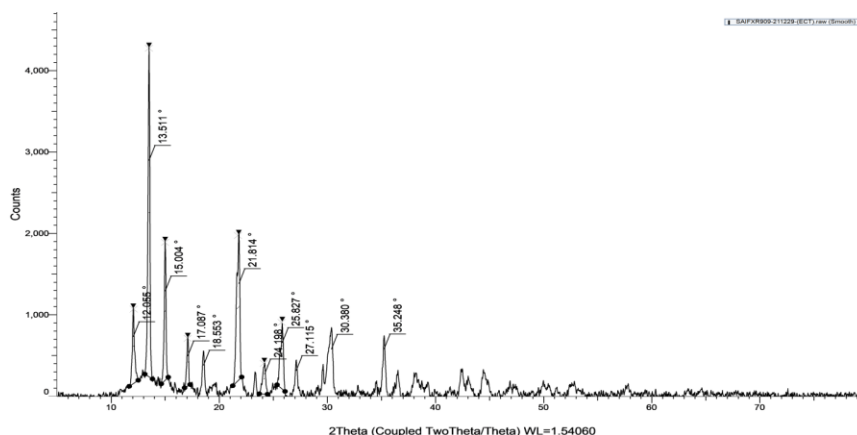
**Fig.6: Synthesized nano particle**

**2.5 Characterization Techniques**

The prepared nanoparticle powder was subjected to various studies to determine their properties and nature. All the studies were carried out from SAIF COCHIN, Kerala, India. The X-ray diffraction offers high quality diffraction data for powder specimen as well as single crystals. The Bruker D8 advance was used to record the XRD peaks. The UV absorbance graph was obtained using UV visible NIR spectrophotometer Perkin Elmer Lambda 365 with wavelength ranging from 200nm to 1000nm. The functional groups present in the prepared nano particles were characterized using FTIR spectrometer, Thermo Nicolet is-50 ranging from 4000cm<sup>-1</sup> to 100cm<sup>-1</sup> with resolution 0.2cm<sup>-1</sup>. The SEM-EDX are characterized using Jeol 6390 LA/ OXFORD XMX N with accelerating voltage 0.5 to 30 Kv. SEM has a magnification upto x300000 and EDX has a resolution of 136ev.

**3. Results and Discussion**

**3.1 XRD Analysis**





### Fig.7 : XRD Spectra of Hydrozincite Nanoparticles

X-ray diffraction is a powerful technique for the analysis of the crystalline material. Prepared Hydrozincate powder were analysed using x-ray diffraction spectrometer. The diffraction spectra for the prepared Hydrozincate powder was given in Fig.7. The peaks were noticed at  $2\theta$  values of 12.055, 13.511, 15.004, 17.087, 18.553, 21.814, 24.198, 25.827, 27.115, 30.380 and 35.243 the peaks were in well agreement with the Hydrozincate nano particles prepared from *Arachis Hypogaea*<sup>[3]</sup>. The peaks matched well with the JCPDS number 04-013-7572 which corresponds to confirmation of Zinc carbonate hydroxide or Hydrozincate. The peaks which are not matched with any of the reference may be due to the presence of bio molecules in the synthesized nano particles. This confirms that the particle thus formed is Hydrozincate or Zinc carbonate hydroxide. The high and sharp peak shows the crystallinity and the short distracted peaks shows the amorphous content of the particle<sup>[8]</sup>. The average particle size was calculated from Debye-Scherrer formula

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

Where k=debye constant

$\lambda$ =wavelength used

$\beta$ =Full width at half maximum

$\theta$ =diffraction angle

Therefore, the calculated particle size for prepared Hydrozincate nano particle was found to be 5.1712 nm.

### 3.2 UV Analysis

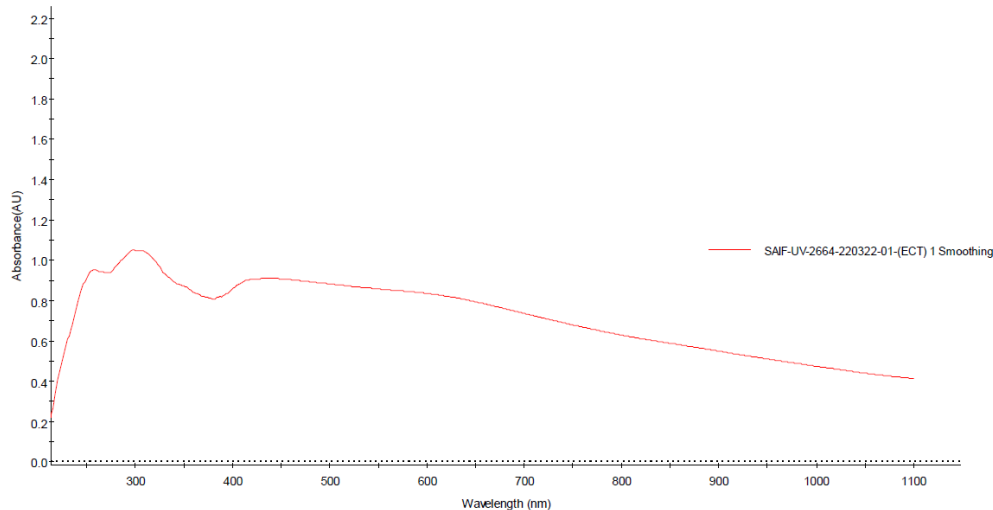
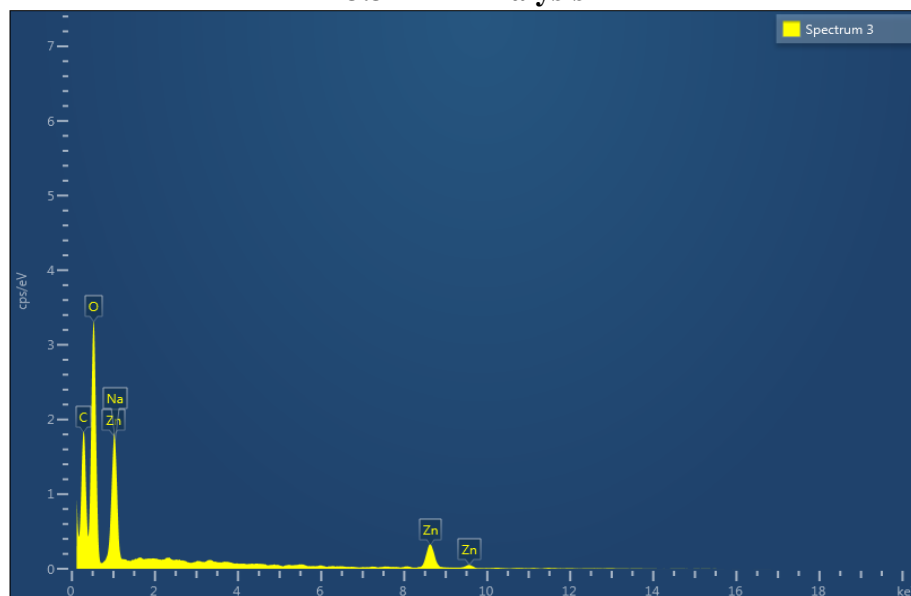


Fig.8 : UV Spectra of Hydrozincate Nanoparticles.

The UV-visible absorption spectra of the synthesized Hydrozincate nano particles was shown in the Fig 8. The obtained nano powder showed the transmittance in the UV region with maximum absorbance peak at 300nm. The peak at 300nm indicate that the absorbance is in blue shift region which confirms the particles are in nano range. The absorbance range from 300nm to 400nm indicates the presence of zinc<sup>[11]</sup>. There was a another small absorbance which was negligibly small noticed at 380nm and the absorption gradually decreases thereafter.

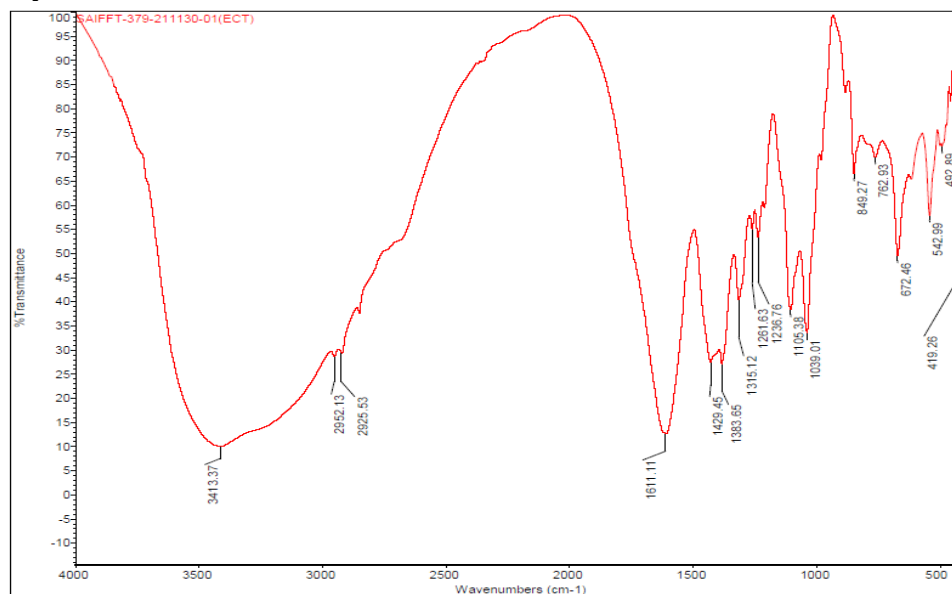
### 3.3 EDX Analysis



**Fig.9: EDAX Spectrum of Hydrozincate Nanoparticles.**

Energy dispersive X-ray Diffraction is known as EDX. It enables us to identify the elements present in the synthesized nano particles. According to EDX spectra the elements present in the synthesized nanoparticles are zinc, sodium, carbon, and oxygen. The traces of sodium may be due to the presence of bio organic metallo compounds present in the wild fruits. The atomic weight and molecular weight of carbon is 26.42% and 23.34%, oxygen is 48.23% and 49.98%, sodium is 1.76% and 2.63% and for zinc is 23.59% and 24.06% respectively.

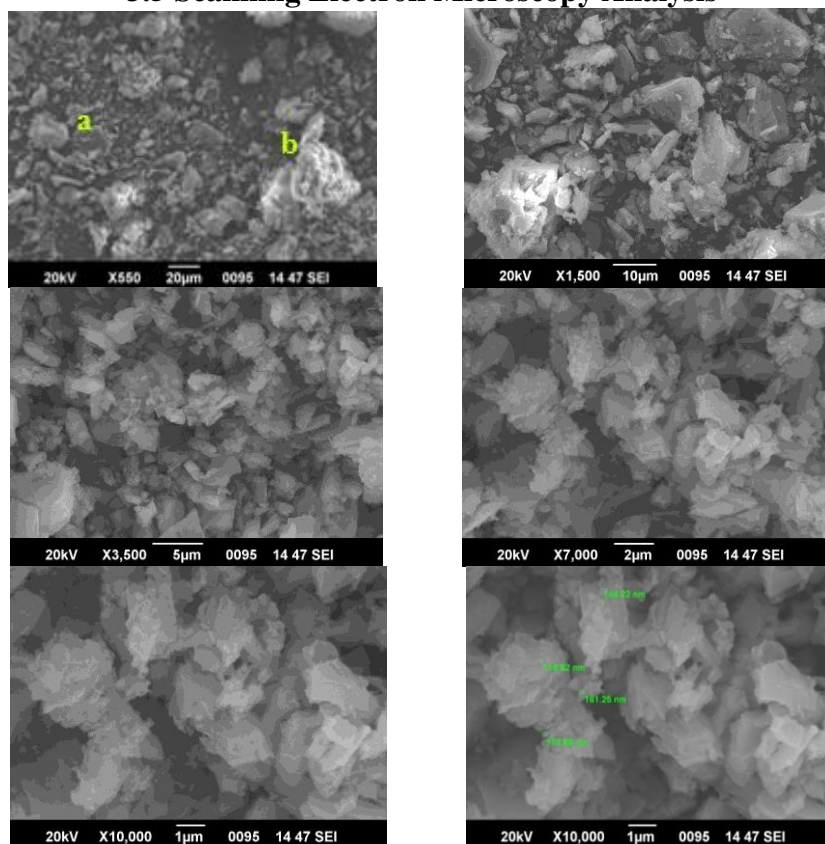
### 3.4 FT-IR Analysis



**Fig.10: FTIR Spectra of Hydrozincate Nanoparticles.**

The FT-IR analysis for the prepared Hydrozincate nano particles were performed to identify the functional groups present in the synthesized nano particles using ECT extract. The peak obtained at  $3413.37\text{cm}^{-1}$  corresponds to O-H stretching which indicates presence of Hydrozincate<sup>[7]</sup>. The peaks at  $2952.13\text{cm}^{-1}$  and  $2952.53\text{cm}^{-1}$  corresponds to C-H stretching. The sharp peak at  $1383.65\text{cm}^{-1}$  shows carbonate mode with C-H bending. The peaks at  $1315.12\text{cm}^{-1}$ ,  $1261.63\text{cm}^{-1}$  and  $1105.01\text{cm}^{-1}$  attributes to C-O stretching which indicates presence of carboxylic groups. The peaks observed at  $1383.65\text{cm}^{-1}$  and  $1511.11\text{cm}^{-1}$  attributes to zinc carbonate which was also confirmed from the previous work done by Veerendar Sharma et al.2018. The sharp peak at  $1039.01\text{cm}^{-1}$  shows the presence of amine group. The peaks obtained at  $849.27\text{cm}^{-1}$  and  $762.93\text{cm}^{-1}$  shows the strong absorbance of zinc<sup>[7]</sup>. The obtained peaks were in good agreement with M.T. Dieng et al. 2019 and N.Kanari et al.2017.

### 3.5 Scanning Electron Microscopy Analysis



**Fig.11 : SEM Images of Hydrozincate Nanoparticles.**

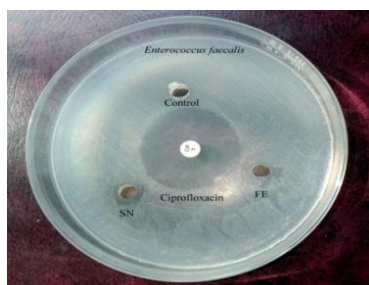
Fig.11 shows synthesized Hydrozincate surface morphology. The given SEM images are classified as (a) (b). The different morphologies were seen at different magnifications. The (a) type images were platelets in shape. The (b) type images were crumbled in shape. The (b) type images has got some luminescence. The luminescence may be due to the presence of the bio organic molecules present in the fruit. The particles are not in homogeneous. The (a) type images has not got the luminescence. As the magnification was done the surface gets agglomerated which may be due to presence of bio organic capping agents.



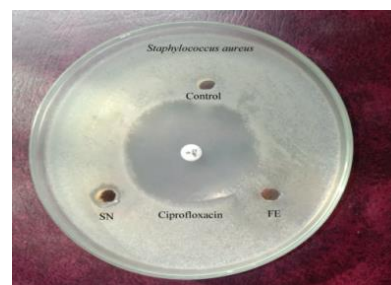
### 3.6 Anti-Microbial Activity

**Table 1: Anti-bacterial Results**

S.NO	Microorganisms	Control	NS	FE	Ciprofloxacin
		Zone of inhibition in mm			
1.	Enterococcus faecalis	-	10	07	30
2.	Staphylococcus aureus	-	8	-	35
3.	Escherichia coli	-	09	-	28
4.	Klebsiella pneumoniae	-	10	05	12



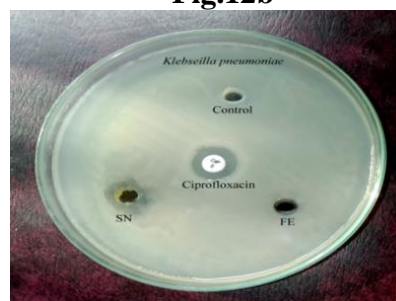
**Fig.12a**



**Fig.12b**



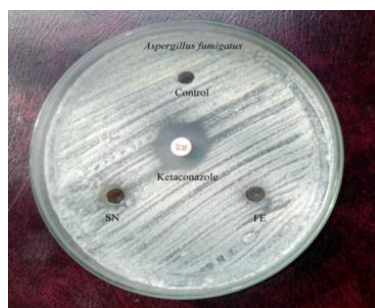
**Fig.12c**



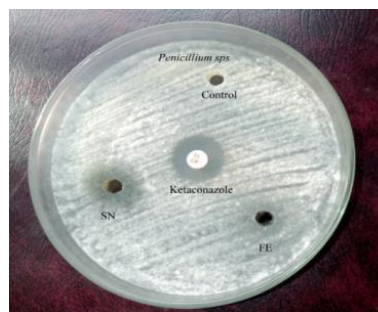
**Fig.12d**

**Table 2: Anti-fungal Results**

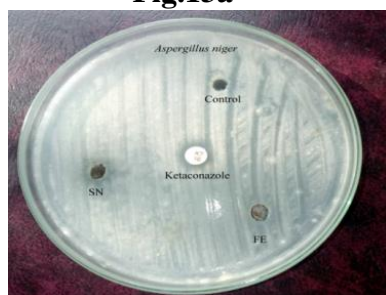
S.NO	Microorganisms	Control	NS	FE	Ketoconazole
		Zone of Inhibition in mm			
1.	<i>Aspergillus fumigatus</i>	-	08	-	15
2.	<i>Penicillium sps</i>	-	12	07	17
3.	<i>Aspergillus niger</i>	-	-	07	10
4.	<i>Candida albicans</i>	-	11	06	12



**Fig.13a**



**Fig.13b**



**Fig.13c**



**Fig.13d**

The prepared Hydrozincate nano particles were characterized for anti-bacterial activity for both gram positive and gram negative bacteria with Ciproflaxin as a reference. The synthesized nano particle showed a noticeable activity for Enterococcus faecalis, Staphylococcus aureus, Escherichia coli and Klebsiella pneumonia whereas the fruit extract showed activity against Enterococcus faecalis and Klebsiella pneumonia. From the overall study, the synthesized nano particle showed good activity against Klebsiella pneumonia. The anti-bacterial activity was shown in Fig.12(a,b,c,d).





Similarly, the synthesized nano particles were characterized for both gram positive and gram negative fungi with Ketoconazole as a reference. The synthesized nano particle showed a noticeable activity for *Aspergillus fumigates*, *Penicillium* sps and *Candida albicans*. The fruit extract showed activity for *Penicillium* sps, *Aspergillus niger* and *Candida albicans*. The fruit extract showed good activity for *Aspergillus niger* whereas the synthesized nano particle showed good activity for *Candida albicans*. The anti-fungal activity was shown in Fig.13(a,b,c,d).

## Conclusion

In the present study Hydrozincate nano particles were prepared using green synthesis method using *Eleocarpus tectorius*(ECT). The prepared nano particles were characterized by UV, FT-IR, SEM, EDX and Anti-microbial studies. Thus we can say that it is effective method to synthesize Hydrozincate nano particles using ECT.

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