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Green Synthesis and Characterization of Hydrozincite Nanoparticles from Wild Passion Fruit

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Abstract: ***Green synthesis of nano particles involves the use of whole plant or parts of plant for the bioreduction of metal ions into their elemental form. Hydrozincite nano particles were synthesized using the wild passion fruit. The fruit has got so many health benefits. The synthesized nano particles were characterized by X-Ray diffraction(XRD), Fourier Transform Infra-Red spectroscopy(FT-IR), UV-Vis absorption spectroscopy(UV), Scanning Electron Microscope(SEM) and Energy Dispersive X-Ray Diffraction(EDX). Using XRD the average particle size was found to be 14.032nm. The functional groups of the prepared sample were identified using FT-IR analysis. From UV the absorption peak was noticed at 302.199 nm which confirms the particle was in nano range. The surface morphology of the synthesized particles was noticied as bread spongy from the SEM analysis. The chemical compounds in the nano particles were identified using EDX analysis. Anti-microbial resistance is one of the greatest challenges to global health. The prepared nano particle shows a good effect for both gram-positive and gram-negative anti-bacterial activity.***

I. INTRODUCTION

In this growing science world, Nanotechnology has produced a number of nanoparticles. There are numerous ways to produce the nanoparticles such as chemical, hydrothermal, electrochemical, microwave assisted, green synthesis etc. Green synthesis method of producing nano particles has gained a much impact on today's world due to less toxic chemicals and it has became a friendly method for the synthesization of nanoparticles. Green synthesis or Biosynthetic method employs whole plant or some parts of plant for the production of nanoparticles. This method was emerged as a simple and a best alternative method of chemical and physical methods[1]. Metal nanoparticles has incorporated a much interest in medical field. Metal nanoparticles have surface to volume ratio, surface energy, spatial confinement and reduced imperfections. Also metal nanoparticles have their own unique electrical, optical properties, physical, chemical, magnetic, thermal and biological properties.[2]

Nanoparticles depend on many factors such as surface area, shape, size, cyrstallinity, functional groups, release of by-products, concentration and etc. Biological methods are user friendly, cost-effective and solves drawback of the chemical synthesis[2].

Nanoparticles has a wide applications in industrial sectors, in which Zinc carbonate Hydroxide has a major role. It was also used in respirators as it reduces the toxic gases. It was also used as a precursor material for the synthesis of ZnO nanoparticles which has a adverse effect in solar cells, electronics and industrial catalyst[3]. Zinc Carbonate Hydroxide is also termed as Hydrozincite or Zinc Carbonate in basic form.

Hydrozincite nanoparticles were synthesized from Wild Passion fruit, which grows in Ooty, The Nilgiris, Tamil Nadu, India. The plant of Passion fruit is a shrub and it is a climber type, the fruit is in oval shape which is pale yellow in colour. The edible part of the fruit is yellowish orange in color. The fruit is best for controlling diabetics and blood pressure.

II. MATERIALS AND METHODS

A. Collection of Fruit

The fresh fruits of Passion fruit (PF) were collected from the campus of Government Arts College, Ooty, The Nilgiris, Tamil Nadu, India. The collected fruits were peeled off and the edible part was taken. This was done because the edible part of the fruit was used for characterization studies. The edible part of the fruit was taken and shade dried at room temperature. It took 4-5 weeks to get dry completely. Then the dried pulps were made into powder using domestic mixer. The powder was stored in an airtight container and used for further studies. The image of fresh fruit and the powder were given in Fig.1 and Fig.2 respectively.

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Fig.1: Fresh fruits


Fig.2: Dried fruit powder

B. Preparation of PF Aqueous Extract

The aqueous extract of PF 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 PF powder was measured using electronic balance and was mixed with 200ml of distilled water. The mixture was refluxed at 90^{o}c for approximately for one hour. The PF extract was collected and filtered twice using whatmann no. 1 filter paper to remove the impurities. The filtered extract was yellowish orange in colour. The refluxing process and the filtered extract was shown in Fig.3 and Fig.4.


Fig.3: Reflux process


Fig.4: Filtered extract

C. Preparation of Hydrozincite 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 PF extract was added to zinc acetate solution at 1:1 ratio. The mixture was heated and stirred at 500 rpm for 1 hour. The colour of the solution has changed from yellowish orange to brown. This brown solution was kept for a couple of days without disturbing for the particles to get sediment. The colour change of the solution before and after stirring was shown in Fig.5 and Fig.6 respeectively.

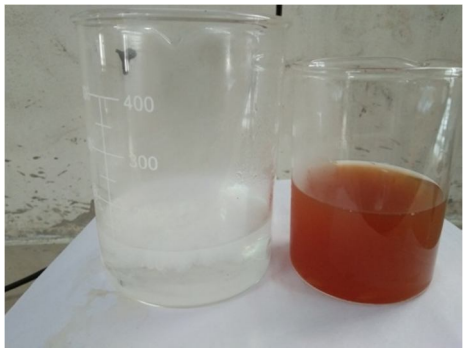


Fig.5: Before stirring



Fig.6: After stirring

D. Preparation of Hydrozincite Nanoparticles

The sedimented particles was taken and centrifuged at 1000 rpm for 45minutes. 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 sedimented dried particles were collected and stored in a air tight viale for further studies. The collected dried particles were brownish black in colour. The sedimented and collected particles was given in Fig.7 and Fig.8



Fig.7: Sedimented particles



Fig.8: Collected Hydrozincite particles

E. Characterization Techniques

The prepared nanoparticle powder was subjected to various studies to determine their properties and nature. All the studies were carried out at 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^{-1} to 100cm^{-1} with resolution 0.2cm^{-1} . The SEM-EDX were characterized using Jeol 6390 LA/ OXFORD XMX N with accelerating voltage ranging from 0.5 to 30 Kv. SEM has a magnification upto x300000 and EDX has a resolution of 136ev.

III. RESULTS AND DISCUSSION

A. XRD Analysis

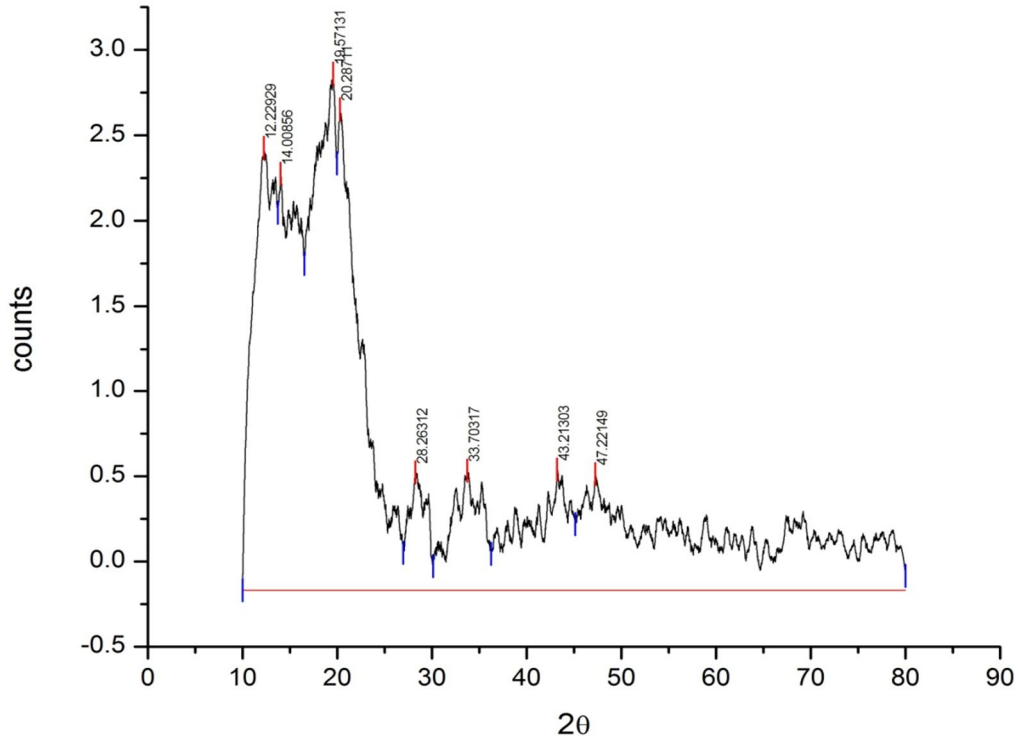


Fig.9: XRD spectra of synthesized nanoparticles

X-ray diffraction is a powerfull technique for the analyzation of the crystalline material. Prepared Hydrozincite powder were analysed using x-ray diffraction spectrometer. The diffraction spectra for the prepared Hydrozincite powder was given in Fig.9. The diffraction peaks were noticed at 2θ values of 12.229, 14.008, 18.571, 20.871, 28.263, 33.703, and 43.213. The obtained peaks were in well agreement with card number 00-019-1458 and 04-013-7572 which corresponds to Zinc carbonate hydroxide or Hydrozincite element. This confirms that the particle thus formed is Hydrozincite or Zinc carbonate hydroxide. The high and sharp peak shows the crystallinity and the short distracted peaks shows the amorphous content of the particle[3]. The average particle size was calculated from Debye-Scherrer formula

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

Where k=debye constant (0.94)

λ=wavelength used (1.54060)

β=Full width at half maximum

θ=diffraction angle

Therefore, the calculated particle size for prepared Hydrozincite nano particle was found to be 14.032nm.

B. UV Analysis

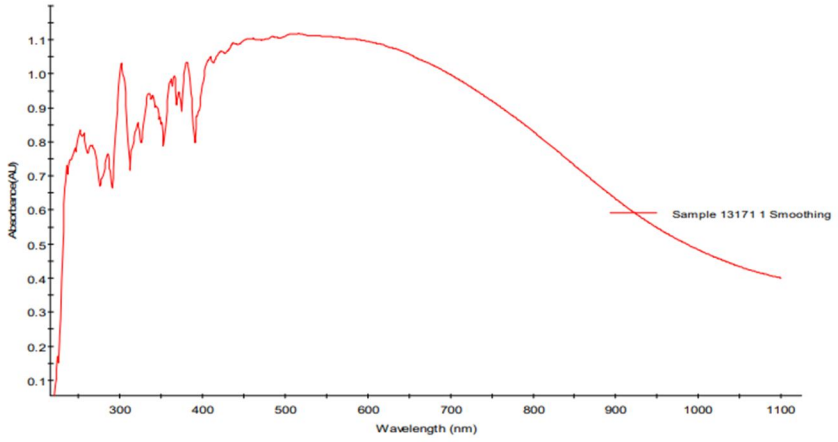


Fig.10: UV spectrum for synthesized nanoparticles

The UV-visible absorption spectra of the synthesized Hydrozincite nano particles was shown in Fig.10. The absorption region ranges from 200nm. The obtained nano powder showed the transmittance in the UV region with maximum absorbance peak at 302.199nm. The band gap energy for the maximum absorption was 410eV. The peaks indicate that the absorbance is in blue shift region which confirms the synthesized particles were in nano range. The colour change from yellowish orange to brown colour is due to Surface Plasmon Resonance, which indicates the reduction in the size of nanoparticles.

C. FT-IR Analysis

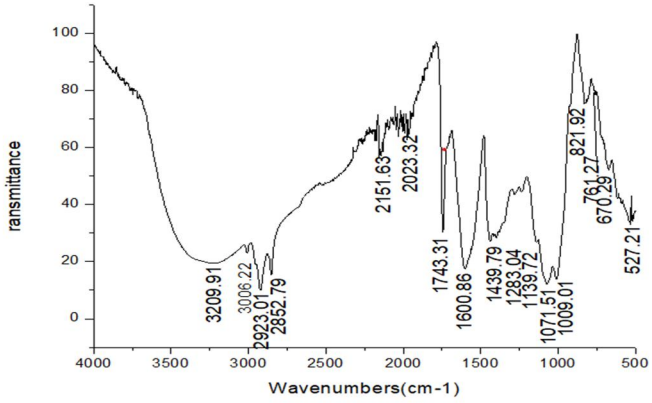


Fig.11: FT-IR spectrum for prepared nanoparticles

The FT-IR analysis for the prepared Hydrozincite nano particles were performed to identify the functional groups present in the synthesized nano particles using PF extract. The peak obtained at 3209.91cm^{-1} corresponds to O-H stretching vibrations which indicates prescence of Hydrozincite[4]. The peak at 2923.01cm^{-1} corresponds to C-H stretching. The double peak at 1439.79cm^{-1} shows carbonate mode with O-H bending. The peaks at 1439.79cm^{-1} attributes to the potassium hydroxide peaks. The peaks ranging from 2923.01cm^{-1} – 1743.31cm^{-1} are the satellite peak regions which are due to the various combination modes in Hydrozincite[5]. The peaks obtained at 821.92cm^{-1} and 761.27cm^{-1} are assigned as the bending mode of carbonate[6].

D. EDX Analysis

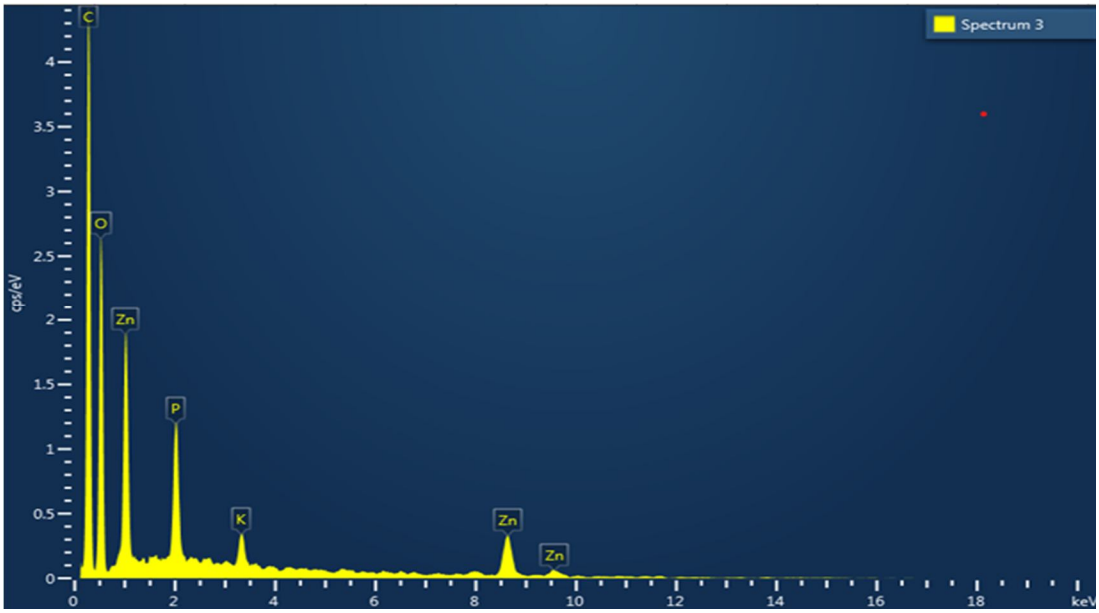


Fig.12: EDX analysis for the prepared nanoparticles

Energy dispersive X-ray Diffraction spectrum (EDX) 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, carbon, oxygen, phosphour and potassium. The traces of potassium and phosphor may be due to the presence of bio organic metallo compounds present in the wild fruits[7]. The atomic and molecular percentage of carbon was 60.3% and 50.68% , oxygen was 10.9% and 15.61%, zinc was 27.26% and 30.22%, Phospour was 1.22% and 2.62% and Potassium was 0.32% and 0.87% respectively. The percentage of potassium and phospour was negligible.

E. Scanning Electron Microscopy Analysis

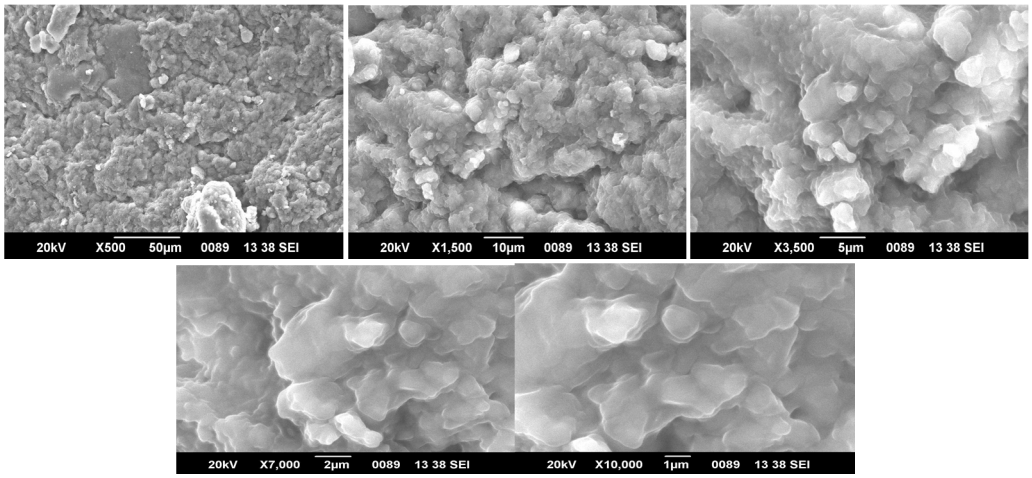


Fig.13: SEM images for prepared nanoparticles

Fig.13 shows the surface morphology of the prepared nano particles. It is found to be planar bread spongy in shape. As the magnification was done, the surface seems to be fully spongy. The individual particles show spherical shape and some larger aggregates were also observed which might be the interparticle interactions found in nano-scale particles[3]. Luminiscene is noticed which may be due to presence of bio organic compounds like Potassium and Phosphur present in the wild fruit[7].

F. Anti-Microbial Activity

Table.1: Anti-Bacterial activity

S.no.	Microorganisms	Control	A1	A2	Ciproflaxin
		Zone of inhibition in mm			
1.	*Staphylococcus aureus*	-	12	10	37
2.	*Enterococcus faecalis*	-	15	12	25
3.	*Escherichia coli*	-	15	12	29
4.	*Klebsiella pneumonia*	-	13	11	28

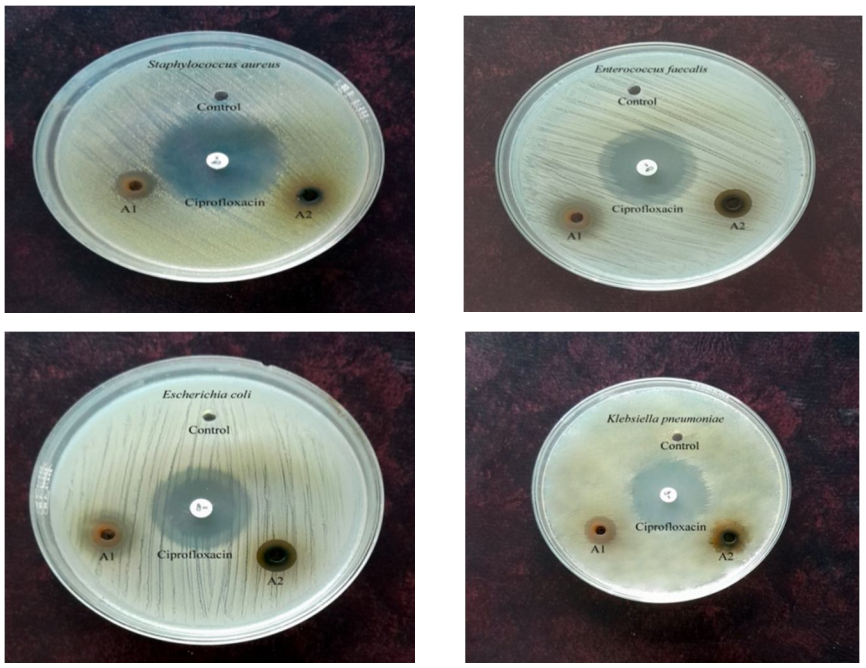


Fig.14: Anti-bacterial activity for synthesized nanoparticle and the fruit powder

The prepared Hydrozincite nanoparticles were tested for anti-bacterial activity. Synthesized nano particle and the PF fruit powder was first studied for both gram-positive and gram-negative bacteria with Ciprofloxacin as a reference. The synthesized nano particle showed a noticeable high activity when compared with fruit powder against all the four bacteria's. A1 indicates the fruit powder and A2 indicates the synthesized nano particles. A1 showed the zone of inhibition upto 15mm for the bacteria Enterococcus faecalis and Escherichia coli. For the Staphylococcus aureus the zone of inhibition was 12mm and for Klebsiella pneumoniae showed the zone of inhibition upto 13mm. A2 showed the zone of inhibition upto 12mm for Enterococcus faecalis and Escherichia coli.

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For Staphylococcus aureus the zone of inhibition was 10mm and for Klebsiella pneumoniae showed the zone of inhibition upto 11mm. The A1 showed the activity 32%, 60%, 51%, 46% respectively and A2 showed their activity as 27%, 48%, 41%, 39% respectively. From the above analysis the overall activity of A1 showed a good activity when compared to A2.

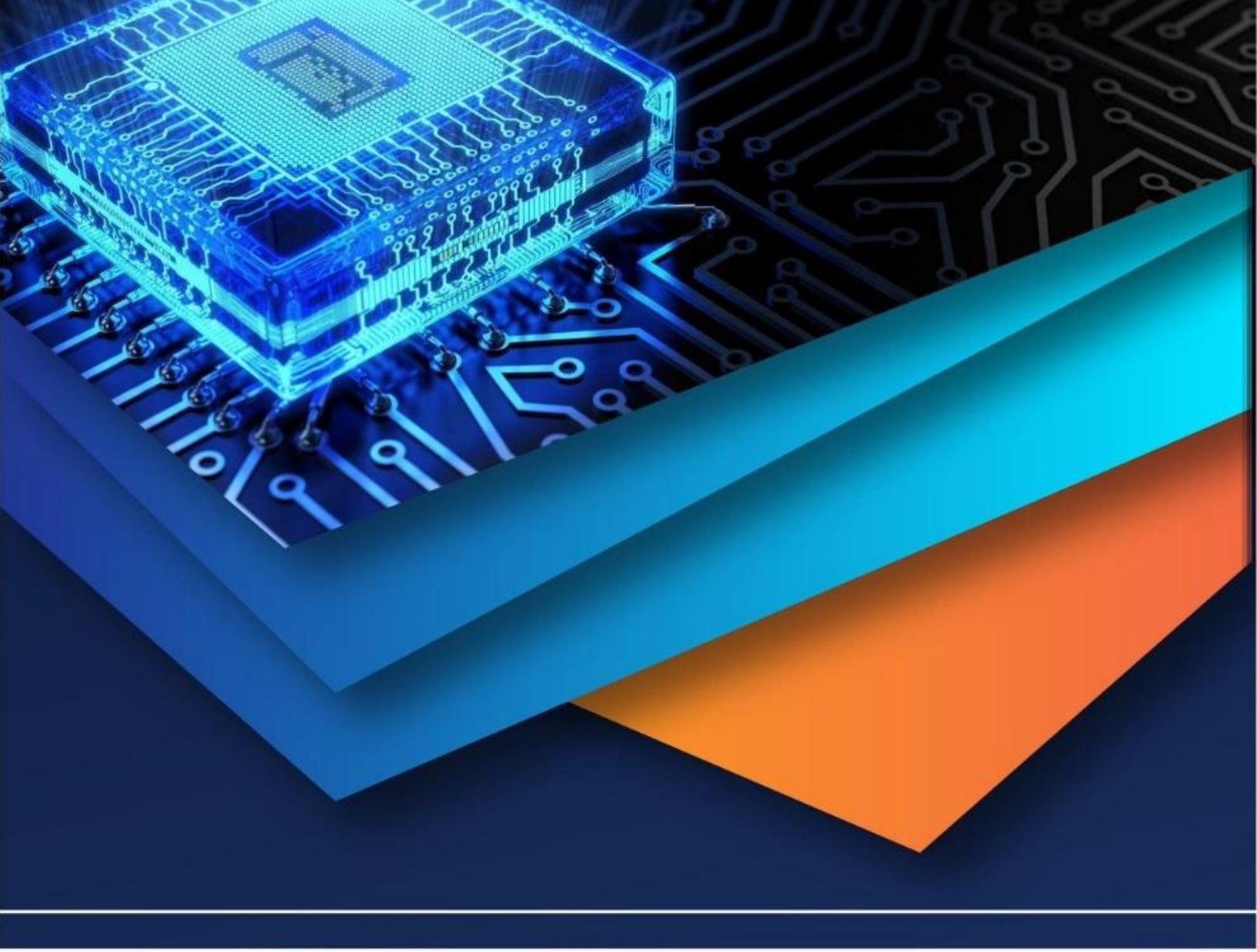
IV. CONCLUSION

The rapid synthesis of Hydrozincite nano particles using Passion Fruit (PE) extract solution has been demonstrated. Structural and morphological properties of the synthesized nano particles were characterized. The present work proves that the PE fruit extracted solution synthesis is a new useful method for the preparation of Hydrozincite nano particles. This simple, cost-effective, time saving and environmental friendly synthetic method gives a potential avenue for various applications. The eco-friendly green approach for the synthesis of nano particles will increase their economic and sustainable management.

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