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GREEN SYNTHESIS AND CHARACTERIZATION OF IRON AND ZINC NANO PARTICLES USING GRAPE EXTRACT

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Abstract

This study focused on the synthesis and characterization of Iron and Zinc nanoparticles using grape extract.. The nanoparticles were characterized using UV-VIS, FTIR, SEM-EDX and XRD spectroscopies. The UV-VIS spectroscopy of iron indicates that the iron nano particle absorb light at 770nm while the zinc nano particle absorb light at 380nm. The utilization of ATR-FTIR spectroscopy of iron revealed distinctive peaks at 3287 cm^{-1}, 2926 cm^{-1}, 1889 cm^{-1}, 1945cm^{-1}, 2012 cm^{-1} to 2072 cm^{-1}, 1919 cm^{-1} and 1817 cm^{-1}, 1379 cm^{-1} and 1038 cm^{-1}, 700 cm^{-1} corresponding to phenol, alkane, A hydride, A hydride, alkyle amine and Fe-bond functional groups respectively. These groups were identified as being responsible for the capping and stabilization of the nanoparticles. That of Zinc revealed distinctive peaks at 3276 cm^{-1} 2922 cm^{-1}, 2113 cm^{-1}, 1636 cm^{-1}, 1543 cm^{-1}, 14011 cm^{-1}, 237 cm^{-1}, and 1028 cm^{-1}corresponding to phenol, alkane, alkyne, alkene, aromatic compound alkene, inorganic carbonate, amine and alkyl amine respectively. Furthermore, EDX analysis confirmed the presence of iron with atomic and weight concentrations of (51.93 and 68.99), and zinc at (20.58 and 35.65) respectively. Scanning Electron Microscope (SEM) micrographs illustrated a uniform distribution and agglomeration of the synthesized iron nano particle (FeNPs) and zinc nano particle (ZnNPs), portraying a near-flat and plain shape with an average particle size of 100 nm. The XRD result revelled that the synthesized iron nano particle (FeNPs) has a crystalline structure with grain size of 23.5nm, while the synthesized zinc nano particle (ZnNP) has a hexagonal structure with grain size of 21.3nm.

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Keywords: Grape extract, Zinc nanoparticle, Iron nanoparticle.

1.0 Introduction

Nanotechnology can be defined as the manipulation of matter through certain chemical and physical processes to create materials with specific properties, which can be used in a particular application. A nanoparticle can be defined as a microscopic particle that has at least one dimension less than 100 nanometres in size [25] NPs industries are developing metals and metal oxides for the improvement of their services and products. There has been a tremendous increase in nanotechnology in the past decade due to it application in medicine, chemistry, biotechnology and agriculture[26], Whiteside 2005, [6]. These nanoparticles are synthesized using physical, chemical and biological methods. Various physical and chemical methods like hydrothermal, sol-gel synthesis, laser ablation, lithography, etc., require special equipment's and skilled labour. Moreover, they have toxic effects that are hazardous to health. The nanoparticles obtained via green synthesis method are found to be cost-effective, non-toxic, and biodegradable in nature [17], [30], [27], [38] This eco-friendly synthesis reduces the use of hazardous substances as the process utilizes the use of fruits, roots, leaves, flower extract and microorganisms like bacteria, fungi, algae etc [38], [28], [36], [14]

Different research works have shown how different plant extract have been used in the synthesis of iron and zinc nanoparticles such as the use of Andean blueberry extract in the synthesis of iron and zinc (Murguitio *et.al.,* 2022). The use of carica papaya leaf extract [29], Iraqi grape extract [44] , hibiscus rosa extract (Ibrahim *et.al.,* 2024), Artocarpus heterophyllus peel extract [37] and Mulberry fruit extract [35] in the synthesis of iron and zinc nanoparticles since they do not contain any hazardous chemicals and functions as a natural stabilizing, reducing and caping agent. Many plants sand their parts have evolved in into a superior platform from the production of nanoparticles. [28]

This research focused on the Green Synthesis of iron and zinc nanoparticles using grape extract, characterization by FTIR for functional groups, SEM for morphology, EDX for elements present with respect to atomic concentration and weight concentration, UV-VIS for atomic spectrum versus, XRD for crystalline structure and sizes

2.0 Materials and Methods

2.1 Method of extraction of grape juice

The grape extraction was prepared using the methodology outlined by Murgueitio *et al*. (2022), with slight modification (where grape juice was replaced by dried papaya leaf extract) made for optimization. The selection of fruits was based on their specific stage of maturity, and exclusively those exhibiting a deep black color were utilized. A thorough cleaning was implemented on the chosen fruits. Subsequently, both the fruits and stems were meticulously pounded in a mortar to extract the liquid juice. The obtained juice was sieved to eliminate any residual impurities, after which it was carefully transferred onto a filter paper to obtain the refined liquid extract.

2.2 Synthesis of Iron Nanoparticle

50 mL of grape extract was introduced into a 50 mL solution of 0.1 M $FeCl_3.6H_2O$, maintaining a precise 1:1 ratio, which resulted in the generation of a visually striking black-colored solution. The ensuing mixture underwent a heating process for a duration of 30 minutes, employing a magnetic stirrer. Subsequently, 1 M NaOH was added in droplet to adjust the pH to 11. The mixture turs deep black which indicated the successful formation of iron oxide nanoparticles.

The nanoparticle underwent separation process through centrifugation, operating at 4000rmp for a duration of 30 minutes. The isolated nanoparticles were subjected to a thorough drying process in an oven set at 80^{o}C, for a duration of three (3) hours. The nanoparticle was then stored in an airtight container for characterization [7].

2.3 Synthesis of Zinc Nanoparticle

30 mL of grape extract was introduced into 70 mL of a 0.1 M zinc oxide solution which result in the formation of a distinctive brown solution. Subsequently, the solution underwent a 30-minute heating process utilizing a magnetic stirrer 1 M NaOH was added in droplets to adjust the pH to 11. The solution turned dark brown after heating which indicate the successful formation of zinc oxide nanoparticles. The ensuing nanoparticle solution underwent separation by centrifugation at 4000 rpm for a duration of 30 minutes. The isolated nanoparticles were subjected to a final drying process in an oven set at 80°C for a period of 3 hours. The nanoparticles were then stored in an air-tight container for characterization [7].

2.4 Characterization of synthesized nanoparticles

2.4.1 Functional Group Analysis

FTIR spectra for both iron and zinc samples were meticulously captured using a cutting-edge FTIR Aligient Technology Cary630FTIR within the wavelength spectrum of 4000-650 cm^{-1}. (Murgueitio *et al.,* 2022).

2.4.2 Absorption Analysis of the Synthesized Nanoparticles

The UV-VIS transmission of both iron and zinc was diagnosed using CARY300 model between the range of 800 to 300nm. 800 to 300 nm. (Murgueitio *et al.,* 2022).

2.4.3 Scanning Electron Microscope (SEM) Analysis and Energy Dispersive X-ray (EDX) Analysis

The scanning electron micrograph (SEM) and EDX analysis of both iron and zinc was done using Phenom Prox [15], [34].

2.4.4 X-Ray Diffraction (XRD) Analysis

X-ray diffraction of both iron and zinc was carried out using (Rigaku Miniflex 600-C) to provide the X-ray patters with a 2Ɵ, scanning angle (2^o-70^o Braggs angle), x-ray tube (Cu abode), operating condition current applied 15Ma and voltage was 40kv. The crystalline size was calculated using Scherrer's equation $D = K\wedge/\beta Cos\ Ɵ$ (Wisam *et.al.,2020*)

3.0 Results and Discussion

3.1 ATR-FTIR analysis of synthesize sFeNPs and ZnNPs

The FT-IR spectra analysis of Figure 1A depicting FeNPs reveals a myriad of vibrational peaks associated with biomolecules within the range of 600-4000 cm^{-1}. These peaks play a pivotal role as capping and reducing agents in the formation of FeNPs. They shed light on the functional groups responsible for iron reduction and stabilization. Notably, characteristic peaks at 3287 cm^{-1} and 2926 cm-1 are ascribed to single bond of oxygen and hydrogen (O-H) stretching and carbon-hydrogen (C-H) stretching [45], [11] peak observed at 1889 and 1945 are attributed to carbon double bond oxygen (C=O) stretching vibration [45], 2012 cm^{-1} to 2072 cm^{-1} are attributed to C≡N [45] 1919 $cm^{-1}$and 1817 cm^{-1} are attributed carbon-carbon-carbon(C=C=C) and carbon doble bond oxygen (C=O) stretching [11],1379 cm^{-1} and 1038 cm^{-1} are attributed to(C-O) stretching and alkyl amine [32] 700 cm^{-1} ensure the present of Fe-O bond[5], [1]. These findings elucidate the diverse molecular interactions and bonds involved in the reduction and stabilization of iron, enhancing our understanding of the intricate processes underlying FeNP formation

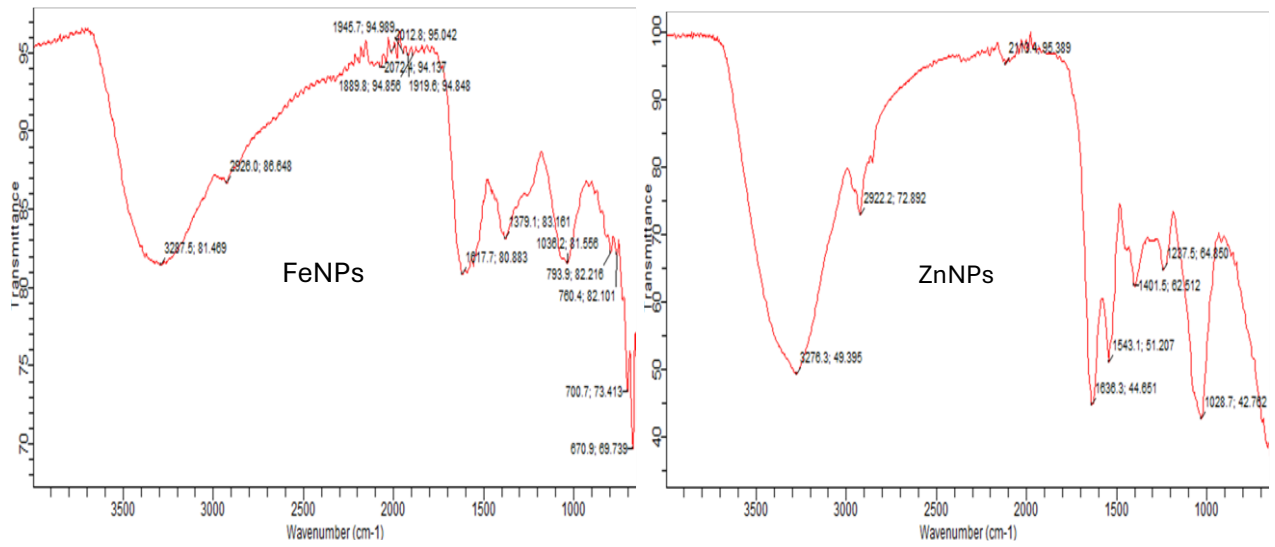

Figure 1.A and 1.B ATR- Fourier transform infrared spectroscopy of zinc nano particle (ZnNPs) and iron nano particle (FeNPs)

The Fourier Transform Infrared (FTIR) analysis, spanning the range of 600-4000 cm^{-1}, not only affirmed the successful synthesis of ZnNPs but also provided insights into the diverse functional groups of reducing agents present in the grape plant fruit extract (refer to Figure 1B). Noteworthy peaks at 3276 cm^{-1} and 2922 cm^{-1} are attributed to the single bonds of oxygen-hydrogen (O-H) stretching and carbon-hydrogen (C-H) stretching, respectively, in alignment with [1]. A distinct peak at 2113 cm^{-1} and 1636 are attributed to triple bond of carbon oxygen (C≡O) stretching vibration and carbon carbon (C-C) [1], 1543 cm^{-1} and 1401 cm^{-1} are attributed C-C and -C=O, peak observed at 1237 cm^{-1} is attributed to (C-N) stretching of amine [19] and 1028 cm^{-1} for (C-O), aligning with the findings of [5], [1]. The discernible shifts in peak positions, ranging from 600–4000 cm^{-1}, provide evidence that compounds containing these functional groups are bound to the zinc oxide surface.

The fictional groups found play a crucial role in the synthesis, stabilizing of FeNPs and ZnNPs. Hydroxyl (O-H) and carboxyl (C=O) groups are responsible for capping and reducing agents, stabilizing nanoparticle formation. Alkyl amine (C-N) and carbon-carbon (C-C) groups contribute to nanoparticle stability and biocompatibility. Fe-O and Zn-O bonds indicate nanoparticle formation and iron/zinc oxide

presence. Carbon-hydrogen (C-H) and carbon-nitrogen (C≡N) groups contribute to nanoparticle chemistry and potential bioactivity [5], [1].

From the result obtained for both iron and zinc the following functional groups are attributed to the plant extract, Hydroxyl (O-H) groups: polyphenols, flavonoid, and other plant derived compounds. Carbon-hydrogen (C-H) groups; plant derived organic compounds such as sugars and amine acids. Carboxyl (C=O) groups; organic acids, flavonoids, and phenolic compounds. Alkyl amine (C-N) groups; amino acids, and protein present in the plant extract [1].

3.2 Ultraviolet-Visible Spectroscopy analysis of FeNPs and ZnNPs

UV-VIS spectroscopy is a common technique used to analyze the concentration of a substance in a sample based on its absorbance of ultraviolet or visible light. In this case the FeNPs from fig2A absorbs light at the wavelength of 770nm which is relatively high suggesting a significant concentration of iron Nano particles in the sample. Higher absorbance value usually corresponds to higher concentration of analyte in the sample. The absorption peak could be related to the size, shape or composition of iron Nano particles. [43].

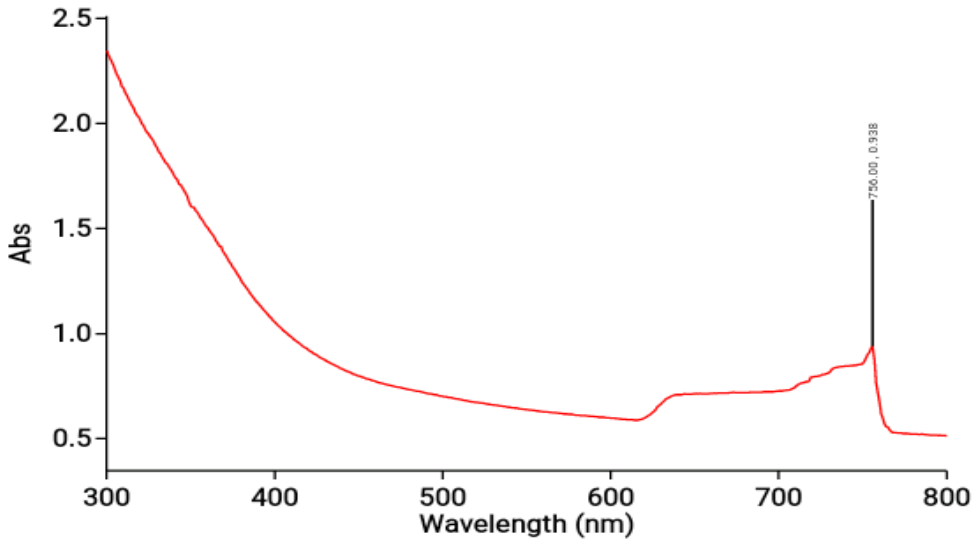

Figure 2.A-UV-VIS spectroscopy of iron nano particle (FeNPs)

From fig2B the absorbance value (Abs) indicates the extent to which the ZnNPs absorbs light at 380nm. Absorbance value can range from 0 (no absorption) to higher values with large values indicating strong absorption of light. The wavelength 380nm falls in the UV-visible range of the electromagnetic spectrum. This wavelength is in the visible light region which is typically associated with the absorption of light by materials that have electron transition within this range. The specific wavelength of 380nm could be indicative of certain electronic transitions or energy levels within the zinc Nano particles molecular or atomic structure. The absorption of light at this wavelength may be related to the presence of zinc (Zn) ions or nanoparticles. This result is in line with the actual band gap wave length of zinc oxide reported by [43].

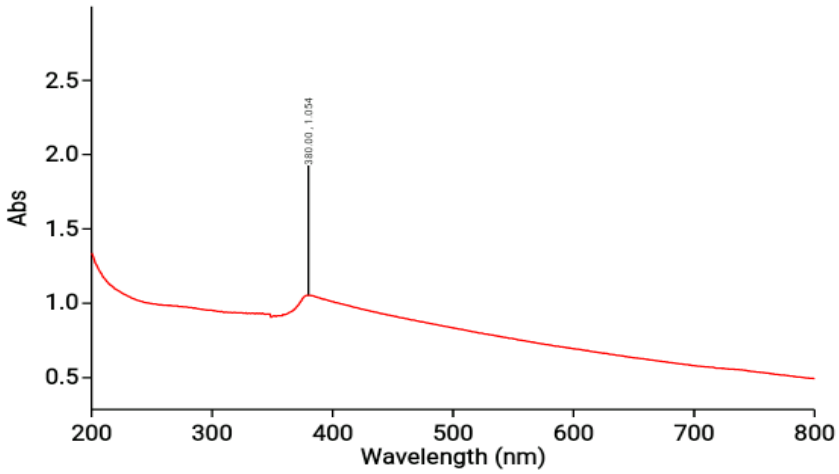

Figure 2.B-UV-VIS spectroscopy of zinc nano particle (ZnNPs)

3.3 Scanning electron microscope (SEM) micrographs and EDX analysis of synthesized of FeNPs and ZnNPs

The Scanning Electron Microscope (SEM) micrographs results of the synthesized FeNF and ZnNPs are presented in Table 1 and 2, along with Figures 3A and 3B. These images collectively convey that the synthesized nanoparticles exhibit a nearly flat and plain shape, characterized by an average particle size of 100 nm. Figure 3A displays a distinctive uniform distribution of nanoparticles, accompanied by noticeable agglomeration, possibly attributed to the concentration of the precursor employed.

Grape extracts were utilized for the biosynthesis of Fe nanoparticles, intended for applications in antibacterial, antioxidant, and cytotoxicity domains. The particle size reported in this study surpasses that documented by [16] and [2]while being inferior to the findings of [41] This variance could stem from disparities in the choice of precursors or the specific reaction conditions employed.

Furthermore, the SEM micrographs for ZnNPs from figure 3B reveal a non-uniform distribution and agglomeration of nanoparticles, likely influenced by the concentration of the precursor used grape extract [16]

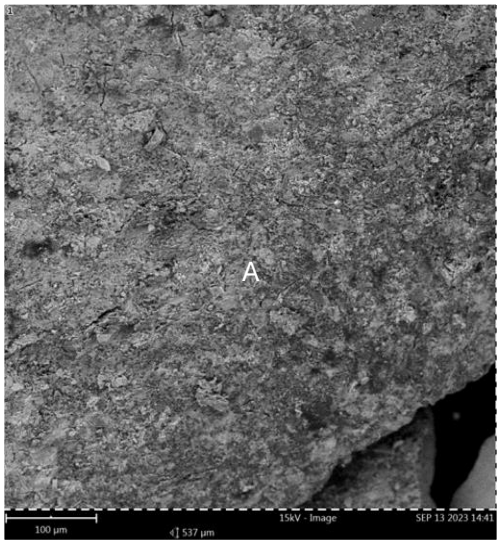

Plate 3.A-SEM, B-EDX micrograph of iron nano particle (FeNPs)

Table 1. EDX analysis of iron nano particle (FeNPs)

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
26	Fe	Iron	51.93	68.99
11	Na	Sodium	20.26	11.08
14	Si	Silicon	8.08	5.40
17	Cl	Chlorine	5.23	4.41
13	Al	Alluminium	5.40	3.47
12	Mg	Magnesium	3.79	2.19
15	P	Phosphorus	1.97	1.45
16	S	Sulfur	1.30	1.00
19	K	Potassium	1.03	0.96
20	Ca	Calcium	0.71	0.67
25	Mn	Manganese	0.29	0.38
22	Ti	Titanium	0.00	0.00

Energy dispersive X-ray (EDX) analysis was employed to elucidate the elemental composition of the synthesized nanoparticles. As depicted in plate 3 and 4, and detailed in Tables 1 and 2, the analysis revealed the prominent presence of iron (Fe) with atomic concentration and weight concentration of (51.93,68.99), Na (20.26,22.08), Si (8.08,5.40), Cl (5.23,4.41), Al (5.40,3.47), Mg (3.79,2.19), P (1.97,1.45), S (1.30,1.00), K (1.03,0.96), Ca (0.71,0.67) Mn (0.29,0.38), Ti (0.00,0.00)

From table 2 result of EDX for ZnNPs revealed Zn with the highest atomic concentration and weight concentration of (20.58,35.65), Ca (32.93,34.97), Na (36.46,22.22), Mg (4.97,3.20), Al (1.94,1.38), Si (1.41,1.05), P (0.81,0.66), S (0.43,0.36), Cl (0.21,0.20), K (0.16,0.17), Ti (0.11,0.13), Fe (0.00,0.00). where iron did not show any noticeable indication. The result obtained was similar to that of [18] ,[40].

The presence of sodium (Na) and calcium (Ca) can be as a result of impurities or from the grape extract. Potassium (K) is present from the precursor grape extract. Silicon (Si), Aluminum (Al) and Titanium (Ti) presence may be due to impurities from equipment or containers or environmental contamination.

Chlorine (Cl), Sulfur (S) and Phosphorus presence may be residual component from plant extract or impurities from starting materials [40]

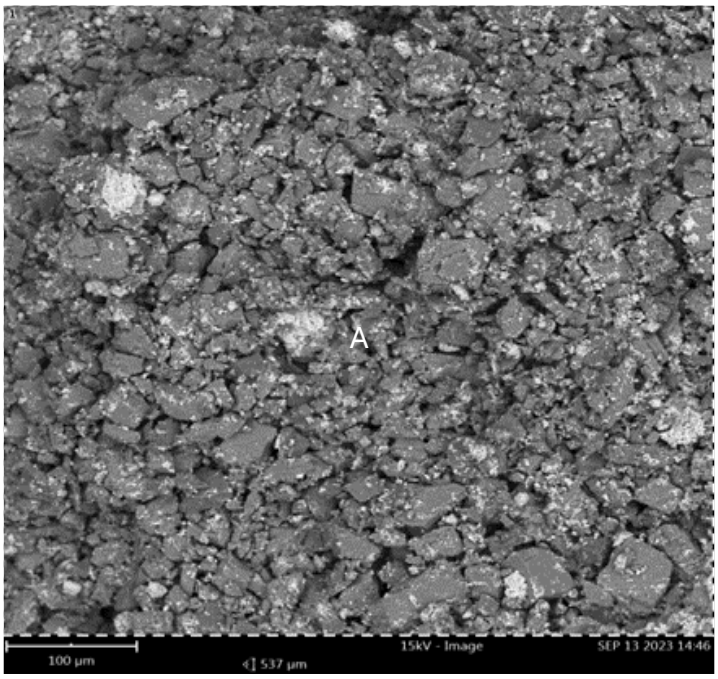

Plate 3. B-SEM, B-EDX micrograph of zinc nano particle (ZnNPs)

Table 2. EDX analysis of zinc nano particle (ZnNPs)

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
30	Zn	Zinc	20.58	35.65
12	Mg	Magnesium	4.97	3.20
13	Al	Aluminium	1.94	1.38
14	Si	Silicon	1.41	1.05
15	P	Phosphorus	0.81	0.66
16	S	Sulfur	0.43	0.36
17	Cl	Chlorine	0.21	0.20
19	K	Potassium	0.16	0.17
22	Ti	Titanium	0.11	0.13
26	Fe	Iron	0.00	0.00

3.4 X-ray Diffraction (XRD) analysis of synthesized FaNPs and ZnNPs

Plate 4A shows the XRD result of iron nano particle (FeNP) at 2Θdegree = 28.5º which matches the (012) plane of Hematite (Fe_3O_3) corresponding to the international center for diffraction data (ICCD) card number 00-001-1053. The result shows that the synthesized FeNP have a crystalline structure consistent with Hematite indicating a trigonal system [23]. The crystalline grain size of the FeNP was calculated and found to be 23.5nm [22]. Research have highlighted the potential of green synthesis methods such as using grape for production of Fe nano particles. These methods offer environmental benefits and produce nano particles with control size and shape[10] The unclarity of peaks may be due to crystalline impurities because present of impurities obscures peaks, or resolution, scanning rate or beam quality issues can also affect peak clarity [23] The noise may be a result of background radiation, instrumental noise or sample related noise (e.g., surface roughness) [23] Therefore, the peaks were identified using international center for diffraction database.

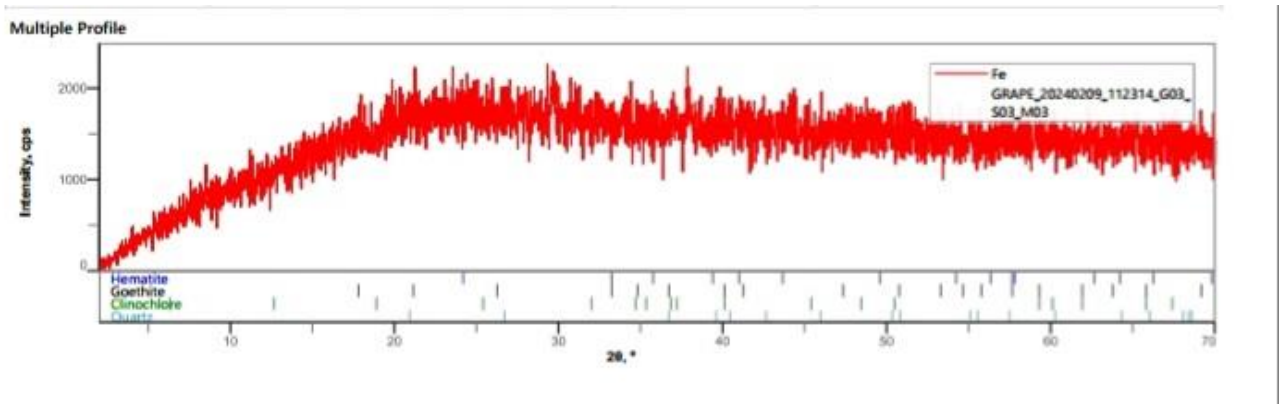

Plate 4.A- XRD analysis of iron nano particle (FeNPs)

Plate 4B shows the result of zinc nano particle (ZnNP) indicating diffraction peak at 2Θdegrre =26.66º,29.452º, 31.07º, 34.44º, 36.33º, 47.639º, 48.67º, 56.61º, 62.827º, and 69.052º corresponding to the lattice plane of (101) (104) (104) (100) (102) (101) (102) (116) (110) (103) (112) and (201) respectively (Jayachandra *etal,2022* , [39] The peaks were matched with the international center for diffraction data (ICCD) card number 04-013-6607. The synthesized zinc nano particle (ZnNP) has a hexagonal structure [3] The crystalline size was calculated and founded to be 21.3nm [39]., [3]

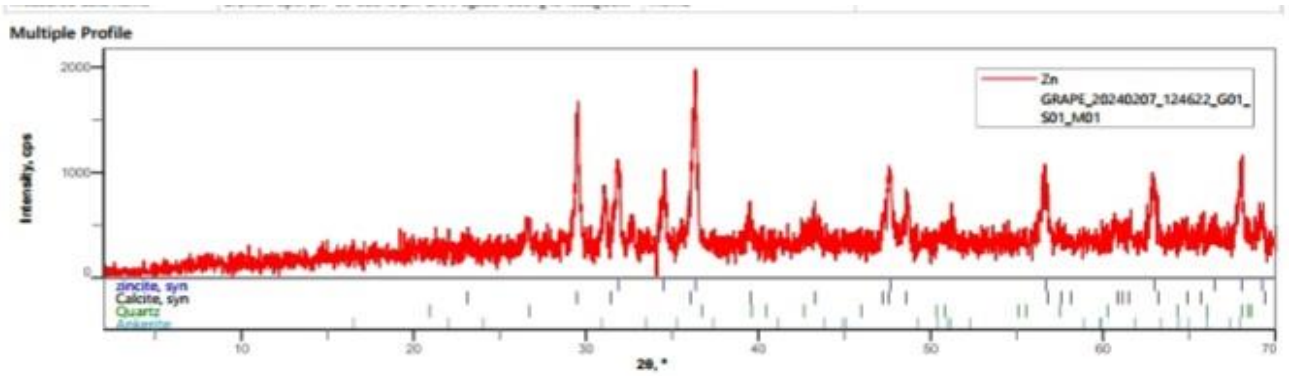

Plate 4.B-XRD analysis of zinc nano particle (ZnNPs)

4.0 Conclusion

In conclusion, the successful synthesis of iron, zinc nanoparticles were achieved through the utilization of grapefruit extract as both a stabilizing and capping agent. The UV-VIS of FeNP shows that it absorbed light at maximum peak of 770nm and ZnNP absorbed light at maximum peak of 380nm. The ATR-FTIR analysis of FeNP unveiled distinctive peaks at 3287 cm^{-1}, 2926 cm^{-1}, 1889cm^{-1}, 1945 cm^{-1} to 1919 cm^{-1}, and 1817cm^{-1}, 1379 cm^{-1} and 1038 cm^{-1} corresponding to alkyl halides, aldehydic, aromatic, amines, amine, and carboxylic acid functional groups, that of ZnNP is 3276, 2922,2113,1237,1028 which also correspond to These groups played crucial roles in the capping and stabilizing processes of the nanoparticles. The XRD result revelled that FeNP is trigonal in shape with grain size of 23.5nm while ZnNP is hexagonal with grain size of 21.3nm. Furthermore, EDX analysis exhibited the presence of iron with atomic and weight concentrations of (51.93 and 68.99), and zinc with concentrations of 20.58 and 35.65, respectively. Scanning Electron Microscope (SEM) micrographs of the synthesized FeNPs and ZnNPs demonstrated a homogeneous distribution and agglomeration of the nanoparticles. The morphology appeared nearly flat and plain, with an average particle size of 100 nm. Interestingly, the iron nanoparticles displayed superior features compared to zinc counterparts base on its characterization.

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Conflicts of Interest

The authors declare no competing financial interest.

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