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Synthesis and Characterization of Nanoparticles of Zno, Carbon Dot and Zno-Carbon Dot Nanocomposite from Groundnut Shell Wastes

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ABSTRACT: The objective of this paper was to biosynthesis and characterize nanoparticles of zinc oxide (ZnO NPs), carbon dots (C.dot), and zinc oxide carbon dot (ZnO-C.dot) nanocomposite from Groundnut Shells. The nanomaterials were characterized using different analytical techniques such as UV-visible spectrophotometry, Fourier Transform Infrared Spectrophotometry (FTIR), Energy DispersiveSspectrometry (EDX), Powdered X-ray Diffractometry (PXRD), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM) respectively. The UV-visible spectra revealed that ZnONPs, C.dot, and ZnO-C.dot exhibited maximum absorption peaks at 372 nm, 235 nm, and 283 nm, respectively. The FTIR results of ZnONPs, C.dot, and ZnO-C.dot revealed strong reactive functional groups for OH and C=O with a high electron density. The EDX results revealed the elemental composition of the nanomaterials in weight percentages for each element. The SEM images of the synthesized nanomaterials revealed that ZnONP had a spherical shape, C. dot had a network-like shape, and ZnO-C dot had an irregular shape. According to TEM, the average particle size of the nanoparticles was 3.42 nm for ZnONP, 2.89 nm for C.dot, and 3.47 nm for ZnO-C.dot. The XRD spectra results showed that all of the nanomaterials were crystalline, with the exception of C. dot, which is amorphous.

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Nanotechnology has emerged as a ground breaking domain, driving innovation across various scientific disciplines, particularly in materials science and environmental sustainability. One intriguing facet within this realm is the synthesis and characterization of nanoparticles from biomass waste materials. Groundnut shell waste, a byproduct of agricultural activities, has drawn significant attention as a sustainable precursor for the production of valuable nanomaterials owing to its rich carbon content and widespread availability (Akpeji and Okewale, 2022; Akpeji *et al*., 2024). Groundnut shells, abundant in agricultural regions, are characterized by their high carbon content and unique chemical composition, making them ideal materials for nanoparticle synthesis. By harnessing this readily available biomass waste, researchers aim to develop eco-friendly and cost-effective methodologies for producing nanoparticles with tailored properties.

The synthesis of nanoparticles from groundnut shell waste involves innovative approaches encompassing chemical, physical, and biological methodologies. Chemical methods entail the reduction or decomposition of precursor materials using chemical agents, enabling precise control over nanoparticle size, morphology, and surface chemistry (Yang *et al.*,

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2020). Physical methods, on the other hand, rely on physical processes such as vapor condensation or mechanical milling to generate nanoparticles with distinct structural characteristics (Mishra *et al.*, 2019). Additionally, biological approaches leverage the catalytic activity of microorganisms or enzymes from biospecimens, to synthesize nanoparticles in an environmentally benign manner (Gupta *et al.*, 2018).

Characterization of nanoparticles derived from groundnut shell waste is paramount for elucidating their structural, chemical, and optical properties. Transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and UV-visible spectroscopy are among the key characterization techniques employed to assess the morphology, crystallinity, surface chemistry, and stability of synthesized nanoparticles (Kumar *et al*., 2021; Elemike et al., 2022; Akpeji and Okewale, 2022).

Zinc oxide (ZnO) nanoparticles, carbon dots, and their nanocomposites of (ZnO-C.dots) derived from groundnut shell waste hold immense potential for diverse applications spanning catalysis, sensors, electronics, and biomedical fields. ZnO nanoparticles exhibit exceptional optical, electronic, and photocatalytic properties, rendering them suitable for applications such as solar cells, gas sensors, and antimicrobial agents (Liu *et al*., 2019). Carbon dots, fluorescent carbon nanoparticles with low toxicity and excellent biocompatibility, find utility in bioimaging, drug delivery, and sensing applications owing to their unique photoluminescent properties (Zhu *et al.*, 2020). ZnO-carbon dot nanocomposites, amalgamating the advantageous properties of both constituents, offer synergistic enhancements in various applications such as photocatalysis, energy storage, and environmental remediation (Zhang *et al.*, 2021). Hence, this work is aimed to synthesis and characterize nanoparticle of zinc oxide (ZnO-NPs), carbon dots (C.dot) and zinc oxide carbon dot (ZnO-C.dot) nanocomposite from Groundnut Shells waste.

MATERIALS AND METHODS

Bio-synthesis of Zinc oxide nanoparticles from Groundnut shell: The extraction and synthesis processes were similar to Okewale and Akpeji (2022); with a few minor modifications. Groundnut shell was obtained at Effurun market in Warri metropolis from groundnut sellers. To eliminate any debris or particles brought along during leaf collecting, the shells were washed with distilled water before being air-dried for five days at room temperature. The shells were then ground with an electric blender and sieved using a Laboratory Test sieve with a 600-microns-wide aperture. The aqueous extract was made using 400 mL of distilled water and 10g of dried, ground plant material that boiled at 80 °C for 2h. The mixture changed from being colorless to brown in its aqueous solution. In order to be employed for the manufacture of ZnO nanoparticles, the extract was obtained after filtration using WhatmanTM filter paper and kept in a sample bottle. In this synthesis, 50mL of the aqueous extract and 500mL of the 0.01M zinc acetate solution were combined at a ratio of 1:5, and 20mL of NaOH solution was added gradual while stirred with a magnetic stirrer for 2h at 70° C.

Fig. 1: Process flow diagram for the preparation of groundnut shell extract & synthesis of ZnO NPs as modified from Okewale and Akpeji (2022)

The color of the colloidal solution steadily changed from light brown to cream throughout the reaction period, indicating a quicker rate of bio-reduction of the zinc acetate and the precipitation of zinc hydroxide Zn $(OH)_2$. A UV-Vis spectrophotometer was used to detect the formation of zinc oxide nanoparticles after the fluid underwent further churning on the magnetic stirrer. The $Zn(OH)_2$

precipitate was cleaned by washing with distilled water and centrifuging for 30 minutes at 5000 rpm. In order to obtain the necessary ZnO Nanoparticles, the supernatant was removed, leaving a clear white precipitate of ZnO-NPs that was dried in an LD-201-E Vision Scientific drying oven at 70^{o}C. The following diagram provides a summary of the protracted process (Fig 1):

Synthesis of Carbon dots (C-dots) from Groundnut shell using Hydrothermal Method: Following the description and contribution of Bibekananda De and Niranjan Karak (2013) in the synthesis of carbon quantum dot (C.dot), this C-dots was synthesized as described but with some variations in methods. The method of synthesis of carbon dots described in this work is the hydrothermal carbonization method at 300^{o}C. 80g of the Groundnut shell was carbonized in a murfle furnace for 2 hours. The solid carbon was removed after cooling and then pulverized using morta and pestle. The powdered carbon obtained was submerged into an empty container and 1000ml of distilled water was added. The mixture was boiled on the furnace for another 1hour at 300^{o}C. The resulting mixture was filtered, and the filtrate was transferred into a conical flask and properly sealed with foil paper to make air-tight.

The formation of the carbon dot showed green luminescence behavior under the UV-lamp. The filtrate was centrifuged and the residue transferred into petri dishes and dried in an oven at 105^{0}C. After drying, the carbon dots were deposited at the surface of the petri dishes which was gently scrapped and stored in sample bottle. The synthesized carbon dot was then characterized by instrumental analysis like the UV-Visible spectrophotometry, Fourial Transform Infrared Spectrophotometry (FTIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), X-ray Diffractometry (XRD) and Energy Dispersive X-ray techniques. The step by step procedure is diagrammatically represented below (fig. 2):

Fig. 2: Process flow diagram for the synthesis of carbon dot from groundnut shell

Synthesis of Zinc oxide carbon dot Nanocomposite (ZnO-C.dot): Nanocomposite is a step wise mixture of different nanomaterials at different proportion. The past work of Elemike *et al.* (2021) reported a proportion of 2:1 but in this work, a reverse ratio of (1:2) of ZnONP and carbon dot were separately mixed in distilled deionized water and stirred at 80^{o}C for 2 hours.

The progress of the synthesis was monitored by checking the maximum absorbance on the UV-Vis Spectrophotometer at regular interval. Each of the nanocomposites was then recovered by gentle drying in LD-201-E Vision Scientific drying oven. The nanocomposites of ZnO-C.dot was then scrapped and stored for necessary characterization and heavy metal remediation. (fig.3)

Fig. 3: Process flow diagram for the synthesis of ZnO-C.dot from groundnut shell

RESULT AND DISCUSSION

After synthesis of nanoparticles, it is necessary to elucidate the physical morphology and chemical components of the nanoparticle. The final products were analyzed using UV-Visible spectrophotometry, FTIR, SEM, TEM, EDX, and PXRD, which can provide helpful information on the chemistry of the biosynthesized nanomaterials, to resolve the conundrum of whether nanoparticles and nano-composites were created in this technique.

UV-visible Spectrophotometry: The initial instrumental method used to monitor the development of nanoparticle production is often UV-V spectroscopy. This is because it clarifies important information about how nanoparticles are formed Jimoh *et al.,* (2022). A Perkin Elmer Lambda 40 UV-visible spectrometer was used to monitor the synthesis and progress of the nanoparticles and nanocomposites. Fig. 4 (a-c) showed the UV-vis spectral result for ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot nanocomposite respectively with maximum absorbance at different wavelength due to varying characteristics of the nanoparticles. Fig. 4a showed the UV-vis spectral result of the biosynthesized ZnO-NPs. The absorbance of the nanoparticles is observed at 372 nm. This gives excellent agreement with those in literatures previously reported by Hairui *et al.* (2020), who reported 371nm as the maximum absorbance peak of ZnONP in their findings. Groundnut shell extract contains some phytoenzymes which has helped in the bio reduction of the zinc acetate precursor into ZnONPs Hairui *et al.,* (2020). Fig. 4b reflects the UV-Visible spectrum of Carbon quantum dots made from groundnut shell showed that the carbon quantum dot absorbed at 235 nm which is in accordance with reports from literature Rifat *et al.,* (2021). Carbon dots are generally known to exhibit broad absorption spectra that extend from the ultraviolet to visible range. This result corresponds to the UV and blue light regions of the electromagnetic spectrum reported by (Bibekananda and Niranjan, 2013). Fig. 4c showed the UV-Visible spectrum for the doped nanoparticles of ZnO-C.dot with maximum absorption at 285 nm. When zinc oxide nanoparticles is combined with carbon dot to form a nanocomposite of ZnO-C.dot, the absorption spectrum can be influenced by various factors, including the concentration and interaction between the individual components. The presence of the zinc oxide nanoparticles may introduce additional absorbance features in the UV range, and the combination of the absorbance characteristics of both components can result in a composite spectrum. The doping of ZnO-NP with C.dot showed that there is an interaction between the two nanomaterials with a different optical property and broader range of wavelength from their individual components. This means that the new nanocomposite of ZnO-C.dot may absorb light over a wider spectrum, encompassing the wavelengths of ZnO-NPs and C.dot respectively. Muhammad *et al.,* (2021) had similar view in their findings in the UV-Visible characterization of nanocomposite of Ag-ZnO with observation showing a broader range of wavelength in the ZnONP due to the doping effect of AgNP.

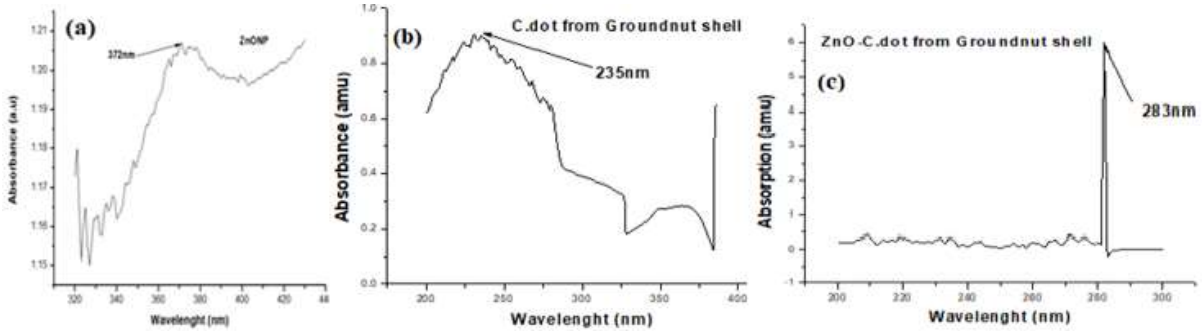

Fig. 4: (a-c): UV-Visible Spectra of the synthesized nanoparticles and nanocomposite

Fourier Transform Infrared (FTIR) Spectroscopic Analysis: Fig. 5 (a-c) revealed the chemical properties of the biosynthesized nanoparticles of ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot nanocomposite, in form of the functional groups present on the surface of the nanoparticles. The FTIR spectrophotometry of model Nicolet iS10 FT-IR Spectrometer was used for this analysis and has performed in the range of 4000 to 450 wave number. Fig. 5a is the FTIR spectrum for ZnO-NPs which revealed band peaks and characteristics functional group present in the synthesized zinc oxide nanoparticles. This is to conclude that the nanoparticles have absorption peaks in the range of 3437.8 cm^{-1}, 2625.8 cm^{-1}, 1500 cm^{-1} and 863.3 cm^{-1}. The peak at 863.3cm^{-1} can be ascribed to the

vibration of C-N bond of the primary amine. The peak at 1500 cm^{-1} could be ascribed to the vibration of the nitro compounds. In fact, these reactive groups at the surface of the nanoparticles can have important role as a reaction active sites Donya *et al.,* (2013). The peaks at 2625.8 cm^{-1} and 3437.8 cm^{-1} are ascribed to C-H stretch of alcohols and the stretching vibration of hydroxyl (-OH) functional group of the alcohols. Fig. 5b described the FT-IR spectrum of the carbon quantum dots which showed obvious absorption bands within the range of 600-4000cm^{-1}. In this particular spectrum, stretching frequencies at 1500 cm^{-1} and 1370cm^{-1} were observed, indicating the presence of C–O and –C–H of aliphatic. Figure. 5c is the FTIR spectrum of the nano-composites of ZnO-C.dot with band peaks observed at 3500 cm^{-1}, 1729cm^{-1}, 1264 cm^{-1} and 918cm^{-1} respectively which showed the presence of -OH stretch vibration of the alcohols, C=O bond of carbonyl and C-N bond of the amine functional group. It is seen that the nanocomposite shows some similar traits and increased reactive functional groups that can show good application in Adsorption studies Muhammad *et al.,* (2021).

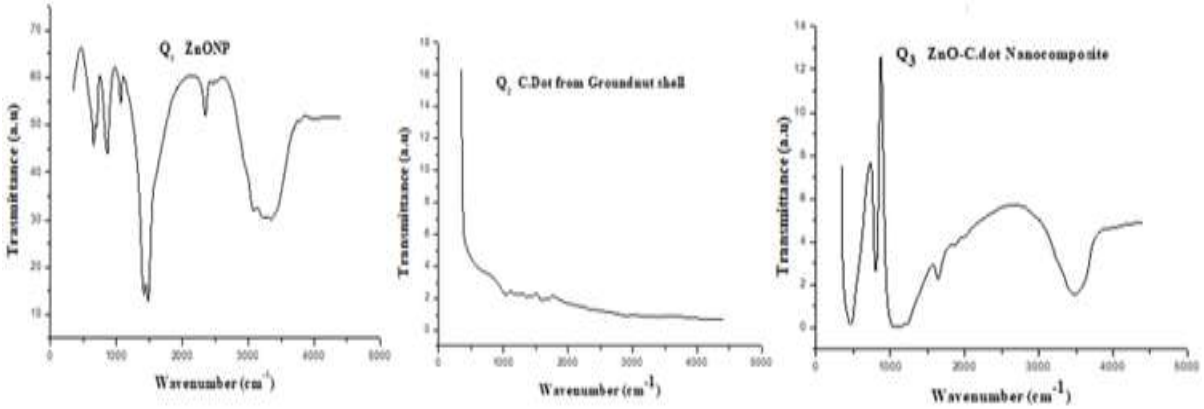

Fig. 5: (a-c): FTIR Spectral for nanoparticles and nanocomposites

Scanning Electron Microscopy (SEM): Figure 6 (Q_1-Q_3) depicts the Scanning Electron Microscope (SEM) micrographs of the different nanomaterials of ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot. The scanning electron microscope (SEM) is a highly specialized instrument that captures images of a specific sample by utilizing a focused electron beam to scan its surface. Fig. 6 Q_1 revealed the micrograph of ZnONPs synthesized through plant assistance. The fundamental objective of the SEM is to reveal the surface structure and actual morphology of the nanoparticles. The micrograph which acts as the adsorbent in this study revealed that ZnONPs is seen to be spherical and porous to adsorb molecule Jan *et al.,* (2020). The porous surface seen in the micrograph gives an idea of the applicability of the nanoparticles in the adsorption of molecules. This means that the nanoparticles will be a source of good adsorbent material to adsorb molecules onto its surface. Past research works of Kovo *et al.,* 2021; Jimoh *et al.,* 2022 had explained similar observations with respect to its morphology and significance in application. Fig. 6 Q_2 reveals the SEM micrograph for carbon dot with a rough surface over and wire like networks pores in every side for adsorption molecules onto the surface of the carbon dot. The porous surface seen in the micrograph explains their high reactivity in the adsorption of molecules like heavy metals. Past research works of Rifat *et al.,* (2021) had explained similar observations with respect to its morphology and significance in application. Fig. 6 Q_3 showed the SEM micrograph for ZnO-C.dot nanocomposite which shows the rough and porous nature of the composite. The state of the surface morphology is being contributed by the ZnO-NP which has spread its morphology on the surface of the C.dot and has disabled the network. This better explain how these two components have been doped to form a nanocomposite materials and evidences seen on the micrograph. Also, the porous nature of both nanomaterials became more intense after the conjugation process. This increase in the porosity means that the nanocomposite will be more efficient in the adsorption of molecules as deposited by Elemike *et al.,* (2021).

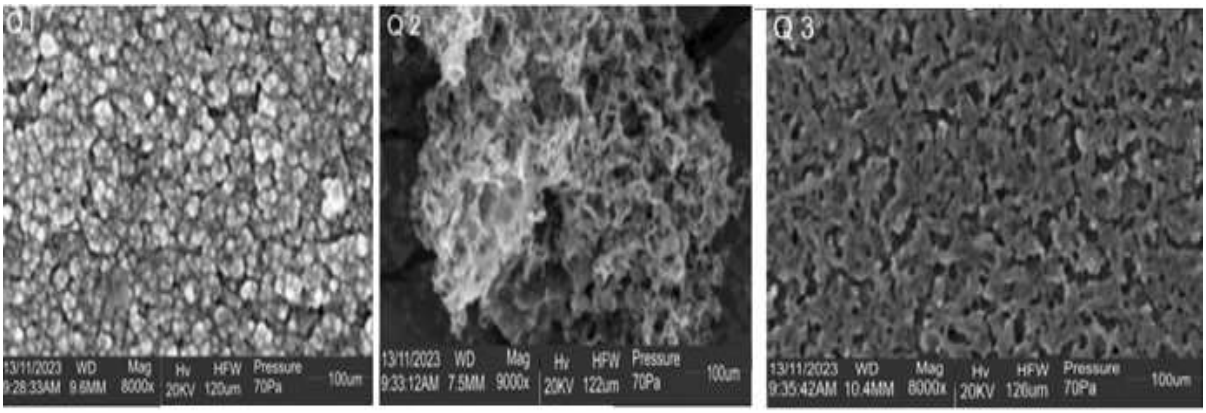

Fig. 6: (Q_1-Q_3): SEM Micrographs of ZnONP, Carbon dot, ZnO-C.dot nanocomposites

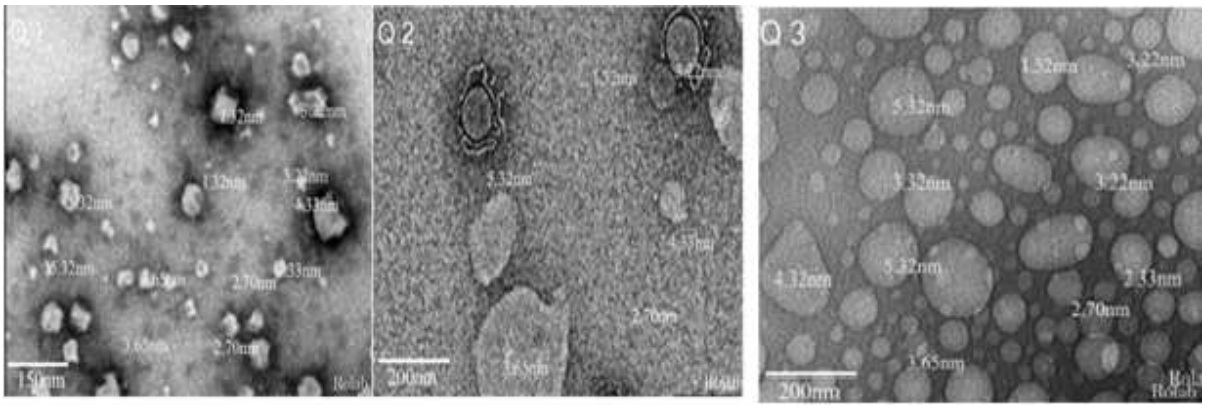

Fig. 7: (Q_1-Q_3) TEM Micrographs of ZnONP, C.dot and ZnO-C.dot nanocomposites

Transmission Electron Microscope (TEM): The significance of TEM with model JEOL2100 is to reveal the particle size of the nanoparticles and nanocomposites within the nanometer range as revealed in fig. 7 (Q_1-Q_3). The nanomaterials in Fig. 7(Q_1-Q_3) revealed the TEM micrograph of ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot. The micrographs of all the nanomaterials all showed sphericity and well dispersed, separated and without agglomerations, which gives an idea on the quality control observed in the course of the synthesis of the nanoparticles. The average particle sizes of these nanomaterials are 3.42 nm, 2.89 nm and 3.47 nm respectively for ZnO-NPs, C.dot and ZnO-C.dot, which are between the nano-range of 1-100 nm. This actually showed the successful synthesis of these nanoparticles owing to their particle size. The significance of the tiny sizes of these nanomaterials is owed to their increasing rate of reaction especially for the carbon dots due to their high surface area which explains its applicability in the increased rate of adsorption of molecules hea and other application in chemical reactions (Tratnyek *et al.*, 2006).

Energy Dispersive X-ray analysis (EDX): The Energy dispersive X-ray Diffraction (EDX) study is one of the instrumental techniques that reveals the elemental constituents present in materials in their weight percentages. When an element is activated by an external source, usually an electron beam in an electron microscope, it emits the distinctive X-rays that are detected by the device. These x-rays' energies match the particular elements in the sample, making it possible to identify and analyze individual components quantitatively. The EDX for the nanomaterials presented in this work are shown in Fig. 8 (Q_1-Q_3) which signifies the energy dispersive x-ray spectra for ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot.

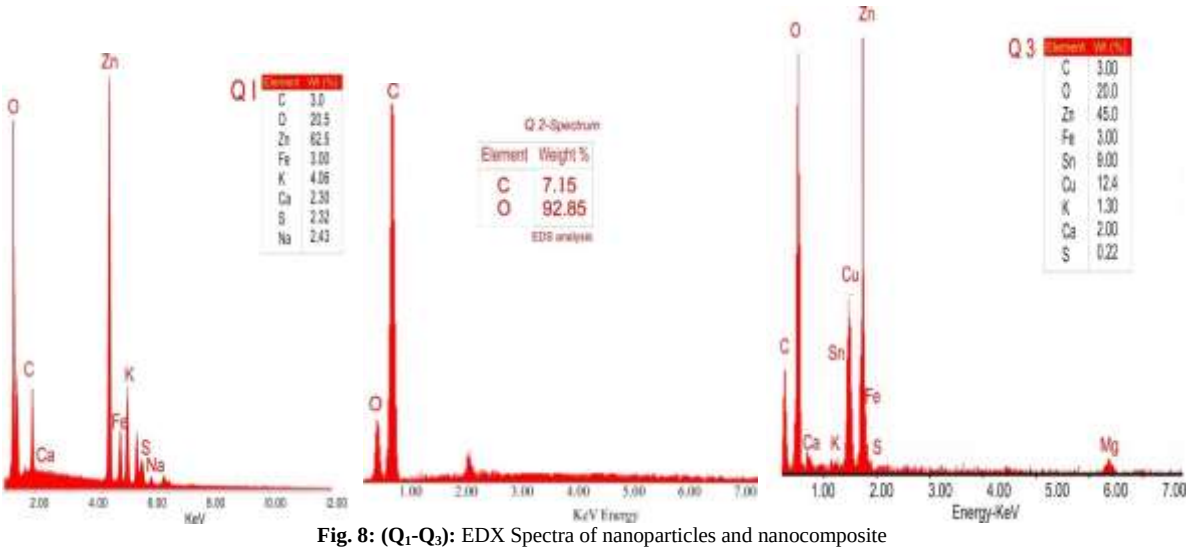

Fig. 8: (Q_1-Q_3): EDX Spectra of nanoparticles and nanocomposite

The spectra of the ZnO-NPs in Fig. 8 Q_1 confirms the presence of elements like zinc and oxygen with peaks corresponding to the optical absorption 62.5% (1.012kev) and 20.5% (0.525kev) respectively which proves that the synthesized ZnO-NP. Other elements like carbon, iron, potassium, calcium, sulphur and sodium were present in trace amount which may be coming from the plant material of the groundnut shell or from external sources of the reagents used (Okewale and Akpeji, 2022). Fig.8 Q_2 revealed the EDX spectra of the carbon dot made from the hydrothermal carbonization of groundnut shell. The spectra of the carbon dot showed optical absorption carbon 7.15% (0.275kev), oxygen 92.85% (0.525kev). No other element was found as impurity which shows the successful preparation of the carbon dot. The elemental composition revealed in the spectra of ZnO-C.dot in Fig. 8Q_3 showed carbon, zinc and oxygen with optical absorption of 3% (0.277kev), 45% (1.780kev), 20% (0.525kev) which explains the complete synthesis of the ZnO-C.dot nanocomposite. Other elements that surfaces like copper, tin, iron, sulphur and calcium could have emanated from the plant materials or the reagents used for the synthesis of the nanoparticles. In all of the spectra of the nanocomposites from 8(Q_1-Q_3), the elemental component in each composite could easily be identified at different percentage weight concentration. This showed that there was a good doping of the nanoparticle with the ZnO NP and successful synthesis of the nanocomposites.

Powdered X-ray diffraction techniques (pXRD): Powder X-ray diffraction techniques (PXRD). The spectral information helps to determine the state of crystallinity and non-crystallinity of the nanoparticles with diffraction peaks obtained accordingly. The X-ray diffraction (XRD) technique is commonly used to determine the crystalline structure and degree of crystallinity of a substance. By examining these characteristics of XRD peaks, such as peak sharpness, number, intensity, positions, and spacing, one can assess the state of crystallinity of a substance. Crystalline materials typically exhibit well-defined, sharp peaks with multiple distinct peaks and high intensity, indicating a higher degree of crystallinity. In contrast, amorphous materials or substances with lower crystallinity may exhibit broad or diffuse peaks with lower intensity.

Fig.9 (a-c) showed the XRD spectrum with diffraction peaks for ZnO-NPs, Carbon dot (C.dot) and ZnO-C.dot. Fig.10 a represents the spectrum of ZnO-NPS with diffraction peaks seen at 2 theta values of 26.30°, 35.61°, 47.42°, 53.30, 55.72°, 68.01° corresponding to (101), (004), (200), (105), (211), (204) and () indices. These peaks are indicative of pure crystalline wurtzite structure of ZnO (JPCDS 36–1451). The observed peaks are in tandem with the ZnO nanoparticles synthesized by Mahamuni *et al.,* (2019).

Figure 9 b revealed the XRD spectrum of carbon dot. The pattern showed sharp peaks at 2θ index of 16.81° and 36.85° corresponding to the (002) and (101) respectively. The broadness, non-sharpness and low intensity of the peaks confirms the non-crystallinity of the carbon dots. Different observation was noticed by Bibekananda and Niranjan Karak, (2013) in their past work where peak of 21.7° was noticed which is equivalent to (002) in the 2theta plane with broad peaks all over the spectra. This explains the non-crystalline state of the C.dot. This could also help

improve its adsorption of metals from solutions. Fig 9c represents ZnO-C.dot nanocomposite and the observed 2θ peaks are 27.45°, 33.36° , 44.39° and 62.55 ° which could be matched to (111), (200), (220) and (311) respectively. There is a successful mixture and doping of the zinc oxide into the carbon dot as the number of peaks were raised and the peak sharpness was increased for the XRD spectrum of carbon dot. This explains the state of crystallinity of the nanocomposite. The sensitization of the ZnO NP with C.dot was evident in the slight differences in the peak values observed in the composite material.

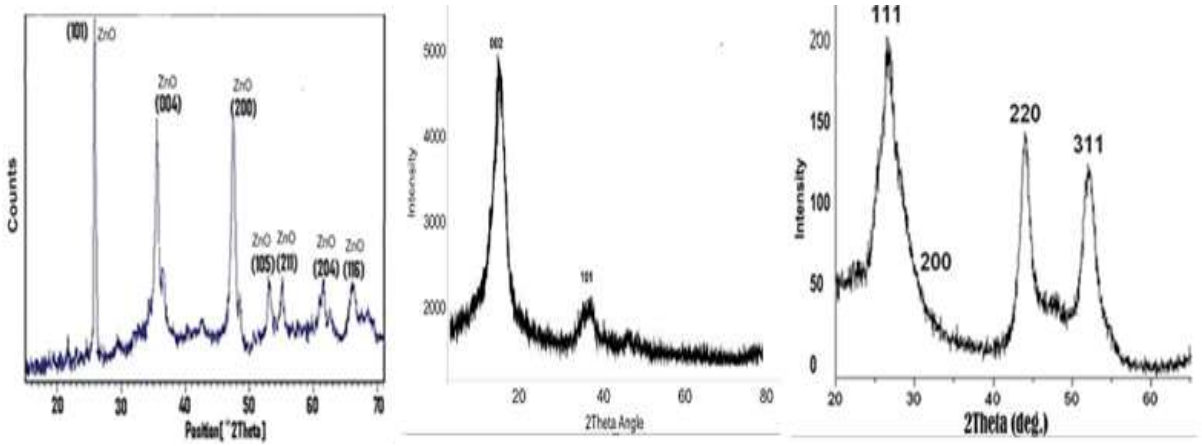

Fig. 9: (a-c): XRD Spectra of the synthesized ZnO-C.dots nanocomposite

Conclusion: This study successfully biosynthesized and characterized zinc oxide nanoparticles (ZnO NPs), carbon dots (C.dot), and zinc oxide carbon dot (ZnO-C.dot) using groundnut shells as waste materials. Through UV-visible spectrophotometry, FTIR, EDX, PXRD, SEM, and TEM, distinct properties and morphologies of the nanomaterials were elucidated. The nanoparticles exhibited promising features such as strong reactive functional groups, crystallinity, and efficient adsorption capabilities. These findings promotes the significance of utilizing waste materials for nanoparticle synthesis and suggest avenues for exploration in environmental remediation strategies.

*Conflict of Interest***:** The authors declare no conflict of interest. Every author participated well according to their own area of interest and research and work amicably.

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