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VARIATION OF ZINC OXIDE NANOPARTICLE DOPED WITH BURNT BAMBOO NANOCOMPOSITE USING SOL – GEL METHOD

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Abstract*: This study provided us with a significant insight into the versatile nature of metal oxides. And when dopped with biochar improves its capabilities more efficiently, Here nanoparticles of zinc oxide (ZnO), nanocomposites of ZnO, doped with burnt bamboo stem (BB:ZnO,) were synthesized using sol gel techniques. Zinc (II) acetate dihydrate, and sodium hydroxide as precursors of zinc. Absolute ethanol and ethylene glycol were used as nanoparticle stabilizer and capping agent respectiviely. Bamboo stem (BB) biochar has absorbance that ranged from 29.07 – 36.61%. Transmittance of Bamboo stem (BB) biochars ranged from 40.04 – 50.15%. ZnO nanoparticles, BB:ZnO nanocomposite possessed absorbance and transmittance values that ranged from 4.22 – 45.08%. The results showed that absorbance values of the sample are high within UV and VIS regions but low within NIR region while transmittance are low in UV and VIS regions but reached maximum values within NIR region. Addtion of biochars of BB was found to increase the absorbance of these metal oxide nanoparticles which in turn resulted in decrease in transmittance. Band gap energy values of BB biochar were found to be 2.15 eV. Energy band gap of ZnO, were found to be 3.25 Ev. while that of BB:ZnO, were found to be 3.20. Presence of biochar (BB) was found to decrease the energy band gap of this metal oxide nanoparticles. These results obtained in this work showed the possibility of modifying the optical properties of ZnO, using BB biochar. These energy band gap values showed that this metal oxide and it's nanocomposite is wide band gap semiconductor (WBGS) material. Structural analyses of the BB biochar revealed formation of hexagonal carbon structure. XRD spectra of the nanoparticle and nanocomposite confirmed the formation of hexagonal, monoclinic and tetragonal phases of ZnO. Additon of BB biochar led to increase in peak intensity and crystallite sizes of the nanocomposite with corresponding decrease in dislocation densities and microstrains. Crystallite sizes of ZnO, was found to be 17.693 nm. while that of BB:ZnO, was found to range from 18.613 – 20.126 nm. Surface morphology study revealed that this nanoparticle and nanocomposite is made up of particle of different sizes, orientations and shapes. The SEM image revealed that the surfaces are made up tiny spherical – like particles and sometimes made up of nanoplates and nanorod. Analyses of EDS spectra obtained showed that these nanoparticle and nanocomposite are made up of desired element such as zinc (Zn) with trace of some other elements which may be as a result of compositions of precursors used during the experiments. The results of characterizations and analyses of this nanoparticle and nanocomposite showed that it could be use in photovoltaic, photodetector, LASER based and optoelectronics devices.*

Keyword*: Zinc Oxide Nanoparticle, Nanocomposite of BBZincs, Biochar, Absorbance, Transmittance, Energy bandgap, Electromagnetic spectrum.*

1.0 INTRODUCTION

The domination of fossil fuel-based power generation in recent years have led to a growing demand for energy resulting in global challenges associated with climate change, and with a rapid growth in carbon dioxide (CO_2) emissions (Asumadu-Sarkodie and Owusu, 2016). Today, the world emphasizes much on the need for cheap, available, efficient and environmental friendly energy found in renewable energy sources, (Okpala, 2013),. According to Hens and Quynh (2016), renewable resources are natural resources that can be regenerated or replaced by ecological processes on a relevant timescale. They include biological resources such as biomass, plants, and animals and elements like carbon and nitrogen. Renewable resources also include inexhaustible solar energy. Of this wide range of energy choice, sunlight or solar energy is certainly one of the most attractive and also much abundant (Ezenwaka *et al*., 2014).

The ultimate solutions of the energy crisis will be through the discovery of methods of harnessing these non-conventional energy sources. Zinc oxide nanoparticles (ZnONPs) represent a remarkable class of nanomaterials with diverse and promising applications across various scientific and technological fields. These nanoparticles consist of zinc and oxygen atoms arranged in a crystalling lattice structure and have gained significant attention due to their unique properties at the nanoscale. One of the most striking features of ZnONPs is their exceptional optical properties, They are transparent in the visible region of the electromagnetic spectrum but can absorb and emit light in the ultraviolet (UV) range. This property makes them valuable for applications like UV-blocking sunscreens, UV sensors, and optoelectronic devices. They have a wide range of applications due to their size-dependent properties, their versatility and tunable characteristics continue to drive innovation in numerous scientific and industrial fields and when doped with the biochar of bamboo stem their capabilities were found to enhanced and significantly improved it's utilization more efficiently.

MATERIALS

1.1

Materials used for this work are grouped into three, which are reagents, apparatus and biological dopanp (extracts of burnt bamboo stem).

1.2 **Reagents**

The reagents used for the synthesis of zinc oxide are zinc (II) acetate dihydrate, ethanol, ethylene glycol, sodium hydroxide, acetone and distilled water.

1.3 Apparatus

Laboratory apparatus and equipment used are;

(i) Digital weighing balance
(ii) Glass beakers (100 mL, 250 mL and 500 ml)
(iii) Measuring cylinder (50 mL and 100 mL)
(iv) Magnetic stirrer with hot plate and magnetic bead
(v) Electric oven
(vi) Whatman filter paper (110 mm)

1.4 Biological dopant

Biochar of bamboo stem was used as dopant in this work. Details of the preparation of these biochars of bamboo stem were given in section 1.5 below.

1.5 Preparation of reagent and dopant solutions

(a) Zinc (II) acetate

Zinc acetate is a salt with the formula $Zn(CH_3CO_2)_2$, which commonly occurs as the dihydrate $Zn(CH_3CO_2)_2 \cdot 2H_2O$. The dihydrate form has molar mass of 219.50 g/mol. In this work, zinc acetate served as precursor for Zn ion. 0.20 M of zinc (II) acetate dihydrate was prepared by dissolving 43.9 g of the compound in 1000 mL of distilled water.

(b) Ethylene glycol

Ethylene glycol is an organic compound with the formula $(CH_2OH)_2$. In synthesis of metal oxide by sol gel, ethylene glycol can serve as stabilizing agent, complexing agent as well as solvent medium. In this work, ethylene glycol was used as a stabilizing agent.

(c) Sodium hydroxide

Sodium hydroxide (caustic soda) is an inorganic compound with the formula NaOH. Sodium hydroxide has a molar mass of 40 g/mol. In this work, sodium hydroxide was used to adjust the pH of the reaction medium to desired alkaline region. 0.5 M of sodium hydroxide was prepared by dissolving 20 g in 1000 ml of distilled water.

(d) Preparation of burnt bamboo fine powder (BB

Unburnt bamboo stems were obtained by cutting the stem into small pieces (1–4 cm). The cut bamboo stems were allowed to dry in an open air for 30 days. To produce burnt sample of bamboo, some quantities of dried unburnt bamboo stem were subjected to combustion without using any external means such as petrol or kerosene. The burnt bamboo stem were ground into fine powder and labelled BB. The fine powdered BB were stored in a clean container for further use.

METHODS

1.6 Synthesis of zinc oxide nanoparticles

Zinc oxide nanoparticles were formed in a 250 ml beaker at 60 °C. The reaction mixture contained zinc (II) acetate, ethanol, ethylene glycol and sodium hydroxide molar solutions. Firstly, 100 ml of 0.2 M of zinc (II) acetate was transferred into the beaker, followed by addition of 20 ml of absolute ethanol. The mixture was stirred with a magnetic stirrer for 10 minutes. After that, 10 ml of ethylene glycol was added to the mixture under continuous stirring for 10 minutes before drop-wise addition 100 ml of 0.5 M of NaOH. Addition of NaOH led to the formation of gelatinous precipitate which are white in colour. The final mixture was allowed to stir for 60 minutes at temperature of 60 °C and aged for 24 hours. After 24 hours, the gel – like precipitates were filtered then washed three times with alcohol and with water for many times. Finally, the filtrate was dried in an electric oven at 100 °C for 1 hour to produce metal oxide nanoparticle. Equations (3.1) and (3.2) shows the chemical equation for the formation of zinc oxide nanoparticle. Table 3.1 shows the bath constituent for sol gel synthesis of zinc oxide nanoparticles.

$$Zn(CH_3COO)_2 \cdot 2H_2O_{(aq)} + 2NaOH_{(aq)} \rightarrow Zn(OH)_{2_{(s)}} + 2Na(CH_3COO)_{(l)} + 2H_2O_{(s)} \quad 1.7$$

$$Zn(OH)_{2(s)} \xrightarrow{heat(oxidation)} ZnO_{(s)} + H_2O_{(s)} \qquad 1.8$$

Table 1.0: Sample preparation table for ZnO nanoparticles

Sample name	0.2 M of Zn (ace) (ml)	Absolute Ethanol (ml)	Ethylene glycol (ml)	0.5 M of NaOH (ml)
Zn	100	20	10	100

1.9 Synthesis of bamboo stem biochar (BB) doped zinc oxide nanocomposites

For synthesis of burnt bamboo stem doped zinc oxide nanoparticles, similar procedures used for synthesis of zinc oxide was used with introduction of dopant extract. In this procedure, 100 ml of 0.2 M of zinc (II) acetate was transferred to 250 ml beaker, followed by dropping of 0.5 ml of BB extract into the solution. The mixture was stirred for 30 minutes to allow for solution homogeneity. This follows with addition of 20 ml of ethanol to the mixture and stirred with a magnetic stirrer for 10 minutes. 10 mL of ethylene glycol was added to the mixture under continuous stirring for 10 minutes. Finally, 100 ml of NaOH (0.5 M) was added to the mixture. Addition of NaOH caused the precipitation of gel – like zinc hydroxide nanoparticles. The final mixture was allowed to stir for 60 minutes under continuous heating at 60 °C and aged for 24 hours. The heat treatment of the zinc hydroxide precipitates led to formation of zinc oxide nanoparticles as precipitates. After 24 hours, the gel – like precipitates were filtered then washed with ethanol and water for many times. Finally, the filtrate was dried in an electric oven at 100 °C for 1 hours to produce the desired nanocomposites. Three other samples of BB doped zinc oxide nanocomposites were synthesized with different volume of 1.0 ml, 1.5 ml and 2.0 ml of BB respectively. Table 1.1, shows the bath constituent for sol gel formation of BB doped zinc oxide nanocomposites.

Table 1.1: Sample preparation table for BB doped ZnO nanocomposites

Sample	0.2 M of Zn (ace) (ml)	Absolute Ethanol (ml)	Ethylene glycol (ml)	0.5 M of NaOH (ml)	BB dopant conc. (ml)
ZB_1	100	20	10	100	0.5
ZB_2	100	20	10	100	1.0
ZB_3	100	20	10	100	1.5
ZB_4	100	20	10	100	2.0

RESULT

1.10 Optical properties

Optical absorption spectra of charred bamboo stem (BB), zinc oxide (ZnO), BB doped zinc oxide nanoparticle was studied using spectrophotometer within the far end of ultraviolet (UV) region of (300 nm to 395 nm), visible light (VIS) region of (400 nm to 700 nm) and near infrared (NIR) region of (705 nm to 1100 nm) with a scanning speed of 400 nm/min and interval of 5 nm. Other optical properties such as transmittance, reflectance, refractive index, extinction coefficient, and energy band gap were evaluated.

2.0 Optical properties of ZnO nanoparticles, charred bamboo stem (BB) doped ZnO composites

Optical properties of charred bamboo stem (BB) particles, charred oil bean seed particles, zinc oxide nanoparticles, BB doped zinc oxide nanocomposite are presented in this section.

(a) Optical properties of ZnO and charred bamboo stem (BB) particles and charred bamboo stem doped ZnO

Figure 1.0 shows the graph of absorbance against wavelength for the charred bamboo stem extract (BB), ZnO nanoparticles and charred bamboo stem doped ZnO nanocomposites synthesized by sol gel approach. Absorbance of the samples were found to decrease as wavelength increases from 300 nm to 1100 nm. Absorbance of BB was found to be greater than that of ZnO nanoparticles and BB doped ZnO nanocomposites. The graph shows that the absorbance of the nanocomposites increases as the amount of charred bamboo stem extract increased from 0.5 ml to 2.0 ml.

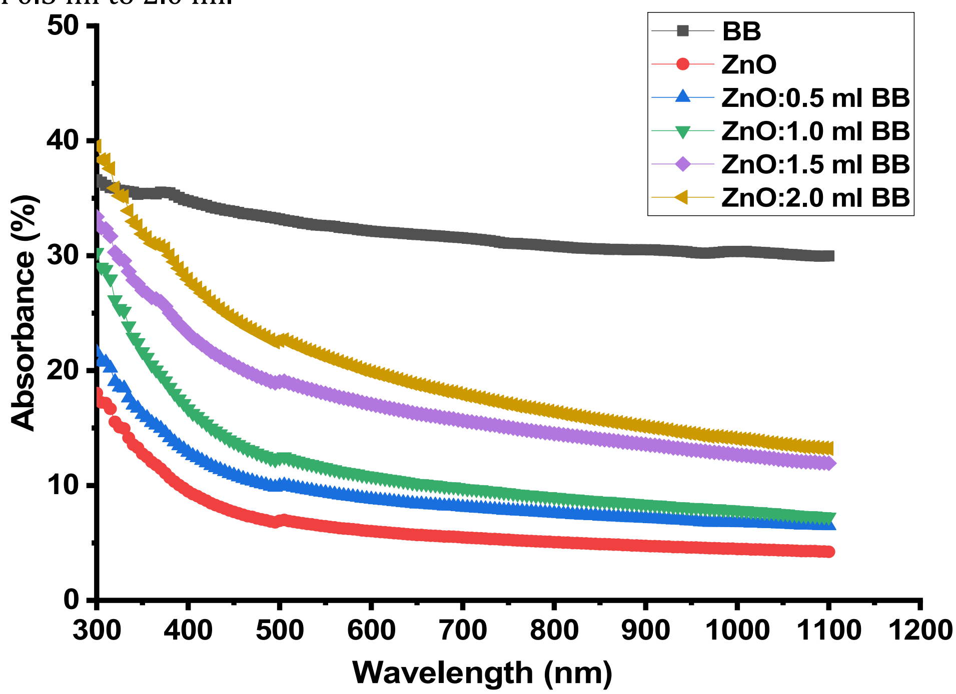

Figure 1.0: Absorbance against wavelength for BB, ZnO and BB doped ZnO nanoparticles

Absorbance values of BB particles ranged from 36.61% at 300 nm to 29.97 % at 1100 nm. ZnO nanoparticles has absorbance values that ranged from 18.02% at 300 nm to 4.22% at 1100 nm. ZnO doped with 0.5 ml of BB extract has absorbance values that ranged from 21.59% at 300 nm to 6.49% at 1100 nm. ZnO doped with 1.0 ml of BB extract has absorbance values that ranged from 30.28% at 300 nm to 7.25% at 1100 nm. ZnO doped with 1.5 ml of BB extract has absorbance values that ranged from 33.38% at 300 nm to 11.93% at 1100 nm.

ZnO doped with 2.0 ml of BB extract has absorbance values that ranged from 39.53% at 300 nm to 13.21% at 1100 nm.
Figure 1.2, shows the graph of transmittance against wavelength for the charred bamboo stem extract (BB), ZnO nanoparticles and charred bamboo stem doped ZnO nanocomposites synthesized by sol gel approach. Transmittance of the sample was found to increase as wavelength increases from 300 nm to 1100 nm. Transmittance of BB was found to be less than that of ZnO nanoparticles and BB doped ZnO nanocomposites.

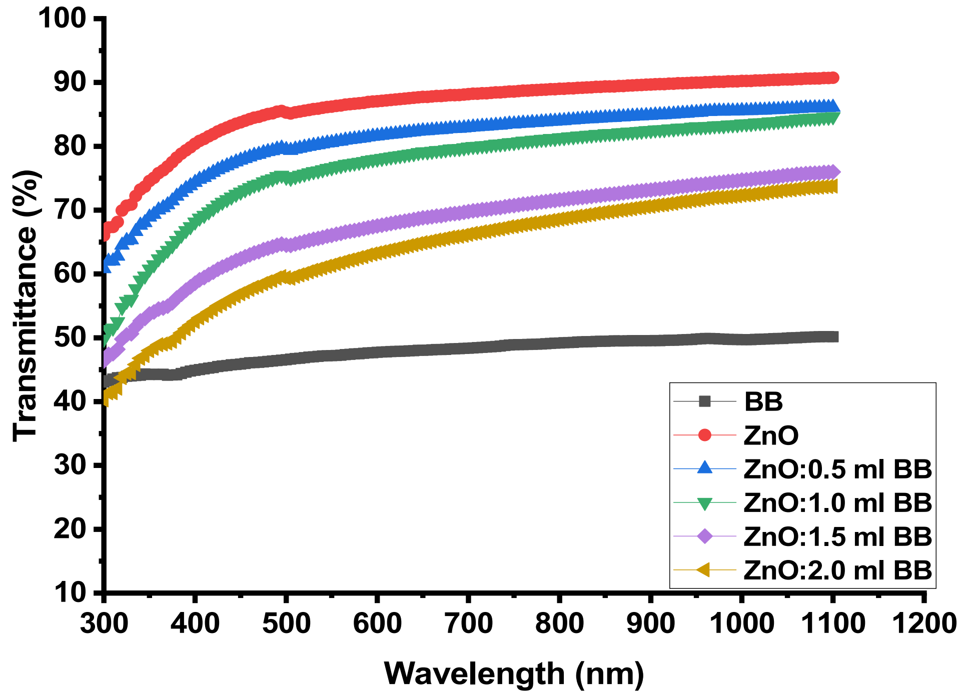

Figure 1.2. Transmittance against wavelength for BB, ZnO and BB doped ZnO nanoparticles

The graph shows that the transmittance of nanocomposites decreased as the amount of charred bamboo stem extract was increased from 0.5 ml to 2.0 ml. Transmittance values of BB particles ranged from 43.04% at 300 nm to 50.15 % at 1100 nm. ZnO nanoparticles has transmittance values that ranged from 66.02% at 300 nm to 90.75% at 1100 nm. ZnO doped with 0.5 ml of BB extract has transmittance values that ranged from 60.83% at 300 nm to 86.12% at 1100 nm. ZnO doped with 1.0 ml of BB extract has transmittance values that ranged from 49.80% at 300 nm to 84.63% at 1100 nm. ZnO doped with 1.5 ml of BB extract has transmittance values that ranged from 46.36% at 300 nm to 75.99% at 1100 nm. ZnO doped with 2.0 ml of BB extract has transmittance values that ranged from 40.25% at 300 nm to 73.77% at 1100 nm.
Figure 1.3, shows the graph of reflectance against wavelength for the charred bamboo stem extract (BB), ZnO nanoparticles and charred bamboo stem doped ZnO nanocomposites synthesized by sol gel approach. Reflectance of the samples were found to decrease as wavelength increases from 300 nm to 1100 nm except for BB particles that has reflectance values that are approximately the same throughout the wavelength.

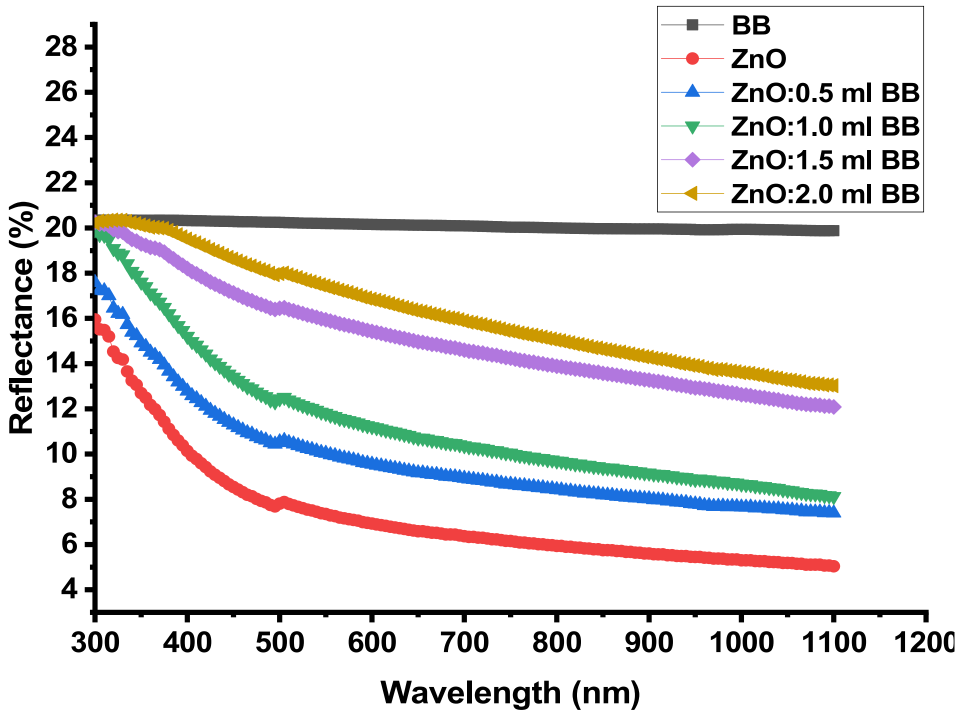

Figure 1.3. Reflectance against wavelength for BB, ZnO and BB doped ZnO nanoparticles

Reflectance of BB was found to be greater than that of ZnO nanoparticles and BB doped ZnO nanocomposites. The graph shows that the reflectance of the nanocomposites increases as the amount of charred bamboo stem extract increased from 0.5 ml to 2.0 ml. Reflectance values of BB particles were found to be approximately the same throughout the wavelength. ZnO nanoparticles has reflectance values that ranged from 15.95% at 300 nm to 5.04% at 1100 nm. ZnO doped with 0.5 ml of BB extract has reflectance values that ranged from 17.58% at 300 nm to 7.39% at 1100 nm. ZnO doped with 1.0 ml of BB extract has reflectance values that ranged from 19.92% at 300 nm to 8.12% at 1100 nm. ZnO doped with 1.5 ml of BB extract has reflectance values that ranged from 20.25% at 300 nm to 12.09% at 1100 nm. ZnO doped with 2.0 ml of BB extract has reflectance values that ranged from 20.23% at 300 nm to 13.02% at 1100 nm.

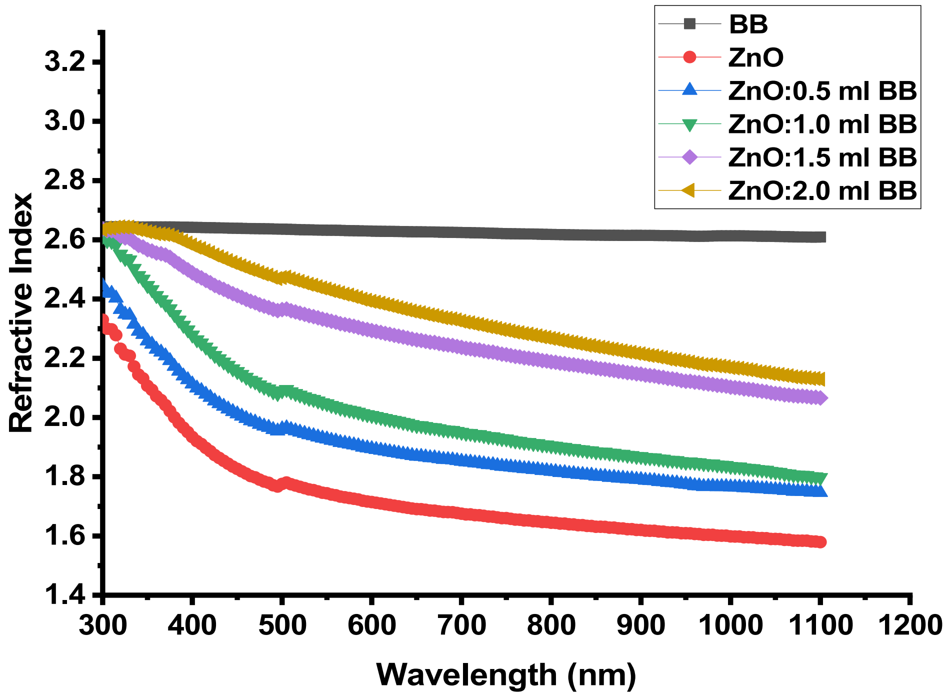

Figure 1.4, Refractive index against wavelength for BB, ZnO and BB doped ZnO nanoparticles

Figure 1.4, shows the graph of refractive index against wavelength for the charred bamboo stem extract (BB), ZnO nanoparticles and charred bamboo stem doped ZnO nanocomposites synthesized by sol gel approach. Refractive index of the samples were found to decrease as wavelength increases from 300 nm to 1100 nm except for BB particles that has refractive index values that are approximately the same throughout the wavelength. Refractive index of BB was found to be greater than that of ZnO nanoparticles and BB doped ZnO nanocomposites. The graph shows that the refractive index of the nanocomposites increases as the amount of charred bamboo stem extract increased from 0.5 ml to 2.0 ml. Refractive index values of BB particles were found to be approximately the same throughout the wavelength. ZnO nanoparticles has refractive index values that ranged from 2.33 at 300 nm to 1.58 at 1100 nm. ZnO doped with 0.5 ml of BB extract has refractive index values that ranged from 2.44 at 300 nm to 1.75 at 1100 nm. ZnO doped with 1.0 ml of BB extract has refractive index values that ranged from 2.61 at 300 nm to 1.80 at 1100 nm. ZnO doped with 1.5 ml of BB extract has refractive index values that ranged from 2.64 at 300 nm to 2.07 at 1100 nm. ZnO doped with 2.0 ml of BB extract has refractive index values that ranged from 2.63 at 300 nm to 2.13 at 1100 nm.

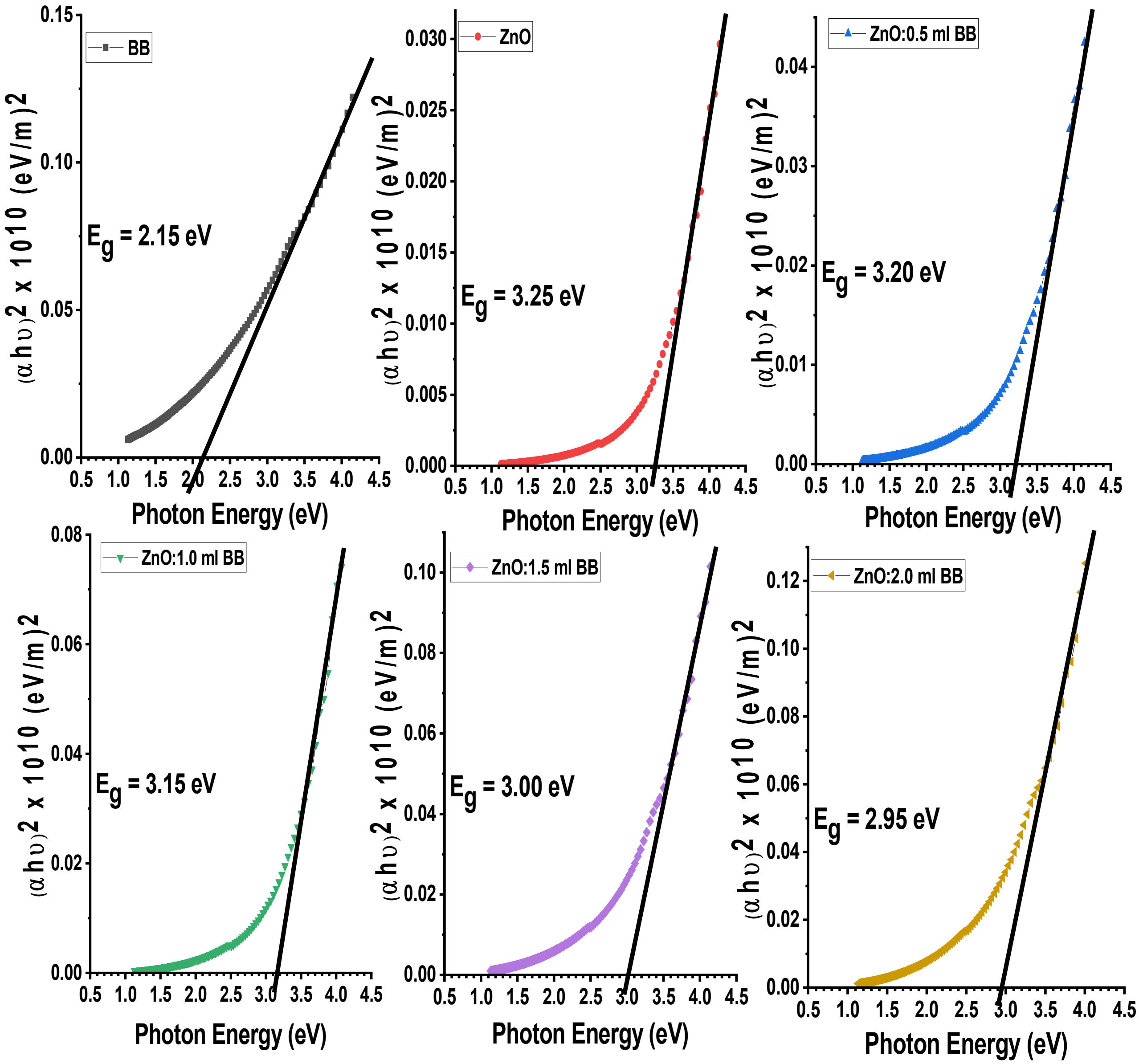

Figure 1.5, Graphs of $(\alpha hv)^2$ against photon energy for BB, ZnO and BB doped ZnO nanoparticles

Figure 1.5, shows the plots of (αhv) squared plotted against photon energy (hv). The direct band gap energy was extrapolated at the axis of the photon energy (hv) where $(\alpha hv)^2 = 0$. Energy band gaps of BB particles was observed to be equals to 2.15 eV. ZnO nanoparticles has energy bandgap of 3.25 eV. ZnO doped with 0.5 ml of BB has energy bandgap of 3.20 eV. ZnO doped with 1.0 ml of BB has energy bandgap of 3.15 eV. ZnO doped with 1.5 ml of BB has energy bandgap of 3.00 eV. ZnO doped with 2.0 ml of BB has energy bandgap of 2.95 eV. These values of energy bandgap were found to decrease as amount of BB extract increases from 0.5 ml to 2.0 ml. Similar energy bandgap values for zinc oxide nanoparticles have been obtained by (Aga *et al.,* 2022; Gautam *et al.,* 2020; Davis *et al*., 2019; Sáenz-Trevizo *et al.,* 2016). Aga *et al.,* (2022) obtained energy bandgap of ZnO that ranged between 3.27 – 3.34 eV. Gautam *et*

al., (2020) obtained energy bandgap of ZnO that ranged between 3.22 – 3.25 eV. Sáenz-Trevizo *et al.,* (2016) obtained energy bandgap of ZnO that ranged between 3.17 – 3.24 eV while Davis *et al*., (2019) obtained energy bandgap of ZnO that ranged between 3.10 – 3.37 eV. Jing *et al.,* (2021) obtained similar decrease in energy bandgap (redshift) of ZnO due to introduction of biochar. He *et al.,* (2021) and Akir *et al.,* (2017) attributed the redshift due to chemical interaction between the ZnO and biochar. The interaction is as a result of chemical bonding between ZnO and carbon. Also, the decrease may be as a result of presence of narrow bandgap substance with reduced energy necessary for electrons of ZnO to transition from valance band to conduction band.
Figure 1.6, shows the graph of extinction coefficient against wavelength for the charred bamboo stem extract (BB), ZnO nanoparticles and charred bamboo stem doped ZnO nanocomposites synthesized by sol gel approach. Extinction coefficient of the samples were found to increase as wavelength increases from 300 nm to 1100 nm. Extinction coefficient of BB was found to be higher than that of ZnO nanoparticles and BB doped ZnO nanocomposites. The graph shows that the extinction coefficient of nanocomposites increased as the amount of charred bamboo stem extract increased from 0.5 ml to 2.0 ml.

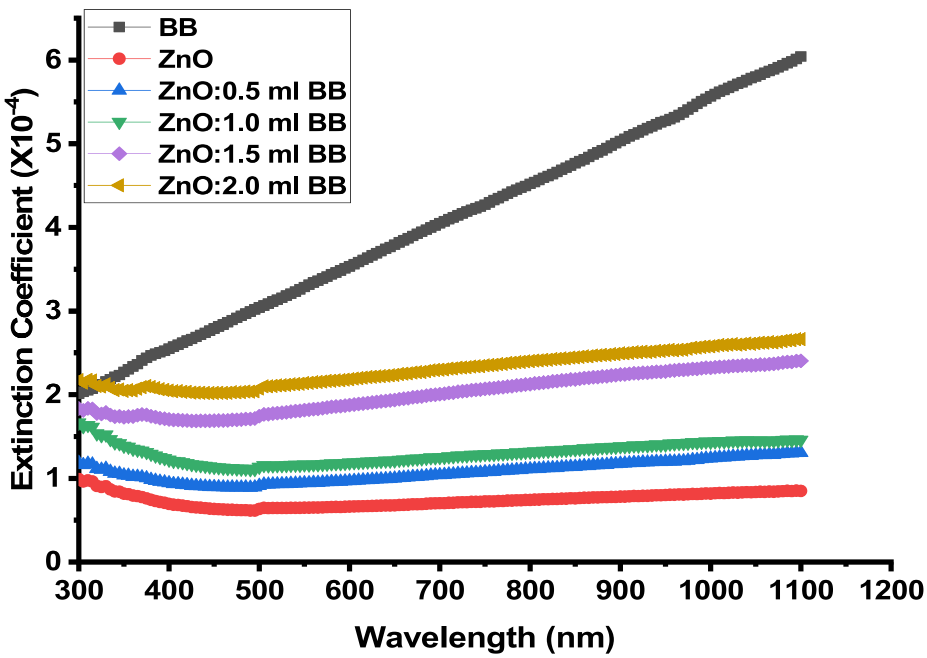

Figure 1.6, Extinction coefficient against wavelength for BB, ZnO and BB doped ZnO nanoparticles

Extinction coefficient values of BB particles ranged from 2.01×10^{-4} at 300 nm to 6.04×10^{-4}at 1100 nm. ZnO nanoparticles has extinction coefficient values that ranged from 9.92×10^{-5} at 300 nm to 8.50×10^{-5} at 1100 nm. ZnO doped with 0.5 ml of BB extract has extinction coefficient values that ranged from 1.20×10^{-4} at 300 nm to 1.31×10^{-4}at 1100 nm. ZnO doped with 1.0 ml of BB extract has extinction coefficient values that ranged from 1.66×10^{-4} at 300 nm to 1.46×10^{-4} at 1100 nm. ZnO doped with 1.5 ml of BB extract has extinction coefficient values that ranged from 1.84×10^{-4} at 300 nm to 2.40×10^{-4} at 1100 nm. ZnO doped with 2.0 ml of BB extract has extinction coefficient values that ranged from 2.17×10^{-4} at 300 nm to 2.66×10^{-4}at 1100 nm.

2.1 Structural properties

X-ray diffraction analyses were carried out on BB particles, BB doped ZnO nanocomposites, The x-ray diffraction spectra of these samples are represented in Figures 1.7,Structural information of this biochar, metal oxide nanoparticles and it's corresponding nanocomposites were obtained at two theta (2θ) interval between 15 ° and 70 °. Observed (2θ) peaks and their corresponding full width half maxima (FWHM) and inter-planar spacing (d – spacing) were recorded. These information were used to determine the crystallite size (D), dislocation density ($\boldsymbol{\delta}$) and microstrain ($\boldsymbol{\varepsilon}$) of the deposited of the samples.

2.2 Structural properties of charred bamboo stem (BB) particles

Figure 1.8, shows the x – ray diffraction pattern of bamboo stem biochar (BB) particles. The result shows weak diffraction peaks in the XRD pattern of BB. A weak dome shape between 20 ° and 32 ° in the crystal pattern suggest that BB particles are amorphous in nature. Broad peak angle of 26.77° was observed which correspond to 2theta value for hexagonal graphite of JCPDS file number of 01 – 075 – 1621 with miller index of [002] reflections. The BB structural pattern obtained in this work is similar to broad amorphous scattering with 2θ angle between 20 ° and 30 ° obtained by (Armynah *et al.,* 2019). Sackey *et al.,* (2021) and Değermenci *et al.,* (2019) suggested that the amorphous nature of the Bamboo biochar is due to the presence of hemicellulose, lignin and other similar components.

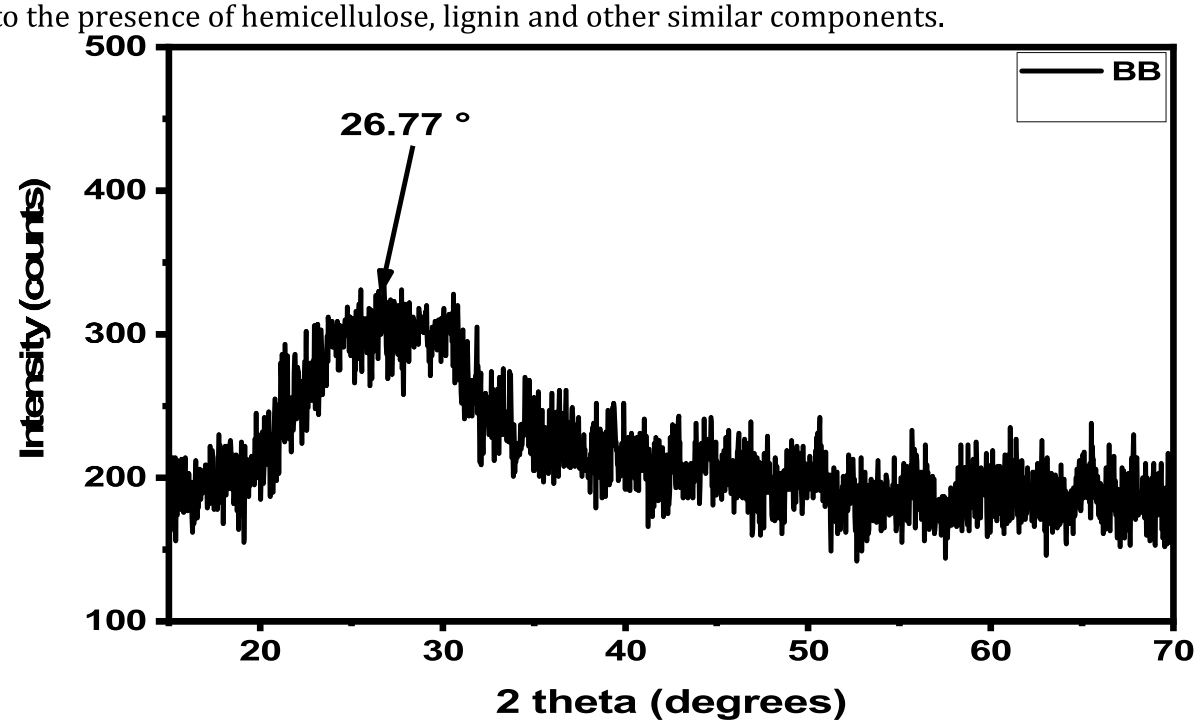

Figure 1.7: XRD pattern for charred bamboo stem (BB) particles

Figure 1.8: XRD pattern for zinc oxide nanoparticles

Table 1.1, Crystal structural properties of ZnO nanoparticles

2θ (º) Observed	D – spacing (Å) Observed	[hkl]	FWHM (º)	Crystallite Size (nm)	$\delta \times 10^{15}$ lines/m^2	$\varepsilon \times 10^{-3}$
31.788	2.813	100	0.458	18.818	2.824	7.025
34.445	2.602	002	0.360	24.128	1.718	5.068
36.276	2.474	111	0.511	17.087	3.425	6.806
47.576	1.91	102	0.603	15.026	4.429	5.973
56.621	1.624	110	0.545	17.278	3.350	4.418
62.878	1.477	103	0.594	16.367	3.733	4.241
66.348	1.408	200	0.506	19.572	2.610	3.381
67.990	1.378	112	0.611	16.369	3.732	3.956
69.094	1.358	201	0.69	14.592	4.697	4.375
			Average	**17.693**	**3.391**	**5.027**

Figure 1.8 shows the x – ray diffraction pattern of zinc oxide nanoparticles. The result shows that the fabricated zinc oxide nanoparticles are of hexagonal crystal phase belonging to the JCPDS file number (00-036-1451). Two theta angles of 31.788 °, 34.445 °, 36.276 °, 47.576 °, 56.621 °, 62.878 °, 66.348 °, 67.990 °and 69.094 ° were obtained corresponding to miller indices of [100], [002], [111], [102], [110], [103], [200], [112] and [201] respectively. Preferential orientation was observed at [111] plane. The structural parameters such as full width half maximum (FWHM), miller indices (hkl), observed 2 theta angles, observed d – spacings, and crystallite sizes of the synthesized zinc oxide nanoparticles were presented in table 1.1. From table 1.0, the average crystallite size of 17.693 nm was obtained for zinc oxide. Dislocation density and microstrain were found to be 3.391×10^{15} lines/m^2 and 5.027×10^{-3} respectively. Yudasari *et al.,* (2021), Ahmed *et al.,* (2018) Brzezińska *et al.*, (2018) and Darezereshki *et al.,* (2021) obtained the same structural phase for their synthesized zinc oxide nanoparticles.

2.3. Structural properties of charred bamboo stem doped ZnO composites

Figure shows the x – ray diffraction pattern of BB doped ZnO nanocomposites fabricated with different amount of BB ranging from 0.5 to 2.0 ml. The patterns show that the BB doped ZnO nanocomposites formed have structural phase of hexagonal zinc oxide with JCPDS file number (00-036-1451). The intensities of the x – ray diffraction spectra were found to increase as amount of BB extract increased from 0.5 to 2.0 ml.

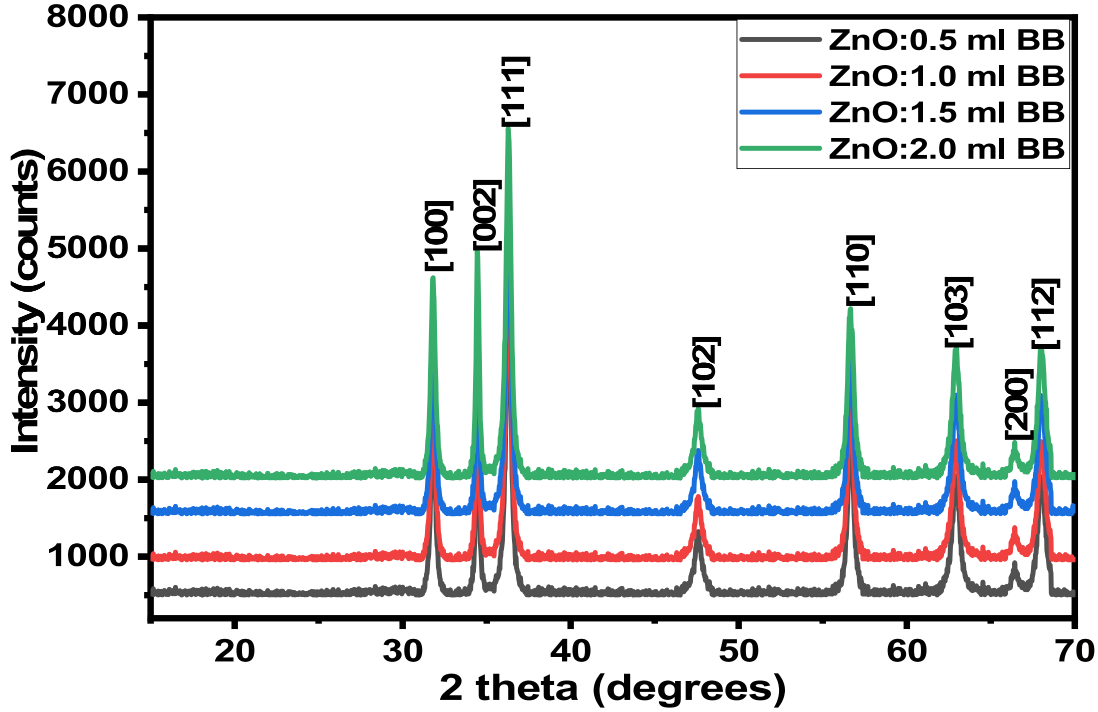

Figure1.7 : XRD pattern for BB doped zinc oxide nanoparticles

Similar peaks observed for zinc oxide were seen the previous figure. No shift in peak positions of zinc oxide nanoparticles was observed as a result of addition of BB rather increase in peak intensity was observed as shown in figure 1.6. Peak at 69.094 ° was found to have disappeared due to the introduction of BB in the structure of zinc oxide. The average crystallite size of 18.613 nm, 19.012 nm, 19.784 nm and 20.126 nm were obtained for zinc oxide doped with 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml of BB respectively. Dislocation densities were found to be 2.886×10^{15} lines/m^2, 2.766×10^{15} lines/m^2, 2.555×10^{15} lines/m^2 and 2.469×10^{15} lines/m^2 respectively. Microstrain values were found to be 4.827×10^{-3}, 4.672×10^{-3}, 4.329×10^{-3} and 3.983×10^{-3} respectively.

2.3 Morphological analyses

The micrographs were taken at magnification of I microns. Generally, it could be observed that all the SEM images exhibits irregular particle – like morphologies. Also, some degree of connectivity of individual particle with one another was observed. ImageJ software for microscopy image analysis was used to determine the average particle sizes of the nanoparticles and nanocomposites from the micrograph images (Abramoff *et al.,* 2004).

2.4 Micrograph of charred bamboo stem (BB) particles

BB

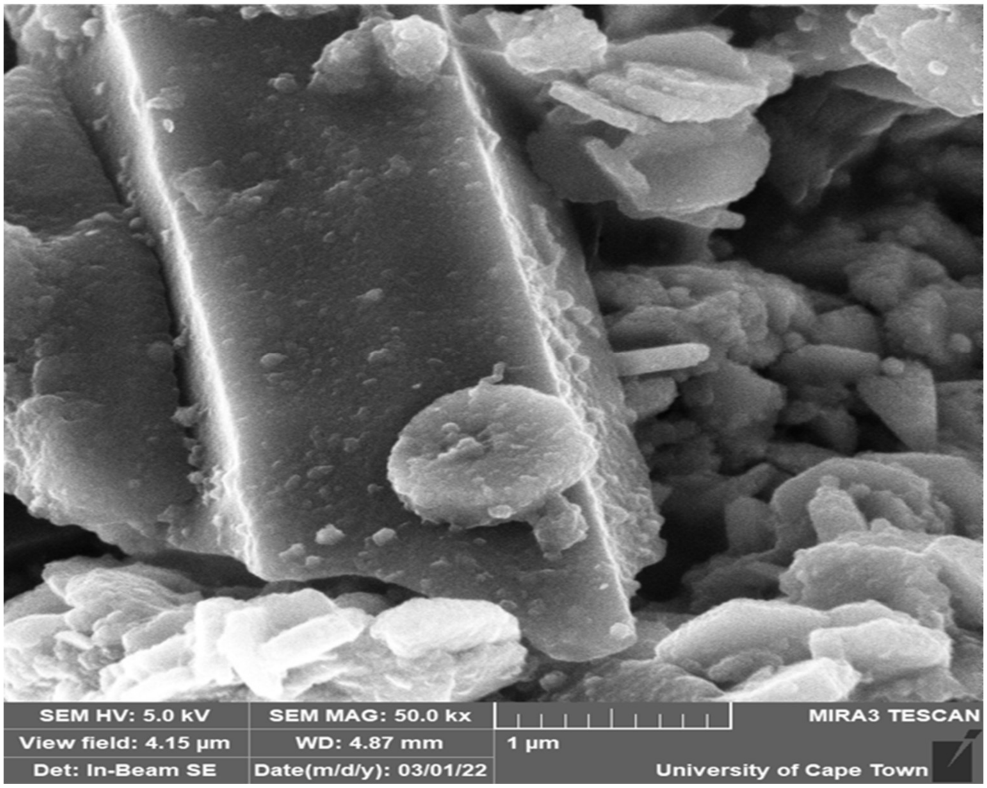

Figure 1.8: SEM image of charred bamboo stem (BB) particles

Figure 1.8 shows the SEM image of charred bamboo stem (BB) particles. The SEM image revealed that particles of BB are of different sizes and shape. Average particles size of 88.45 nm was obtained. Also, the SEM image revealed some degree of agglomeration.

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2.5 Micrograph of ZnO nanoparticles

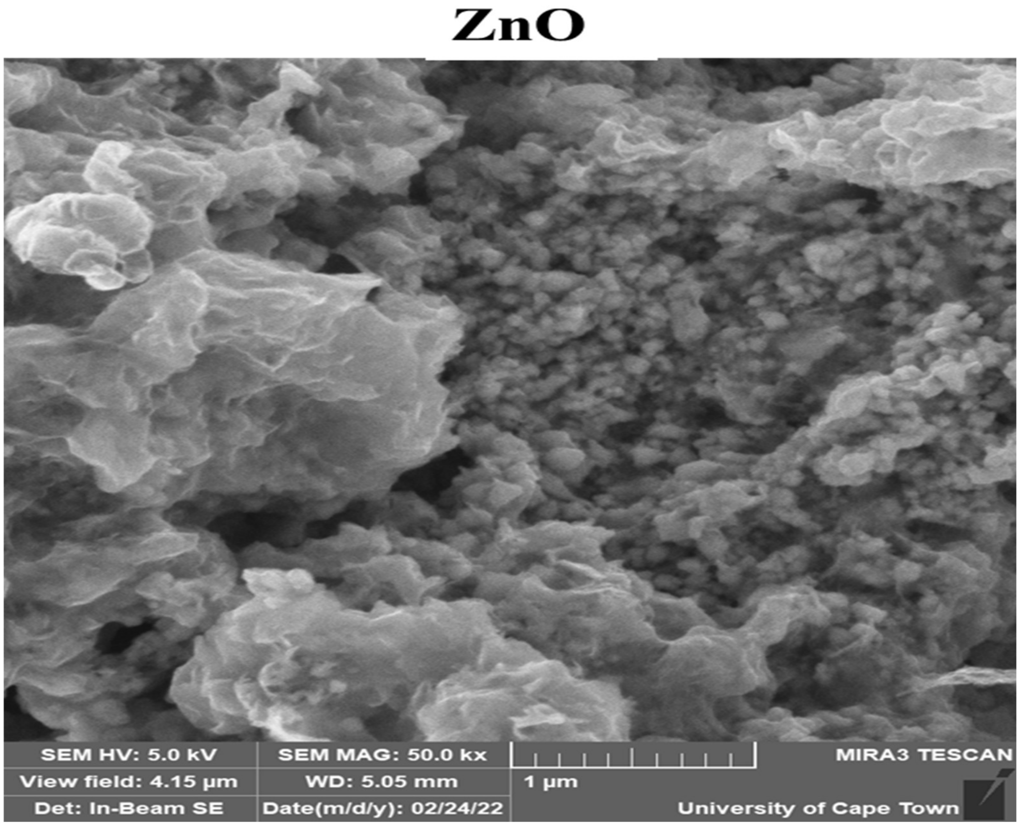
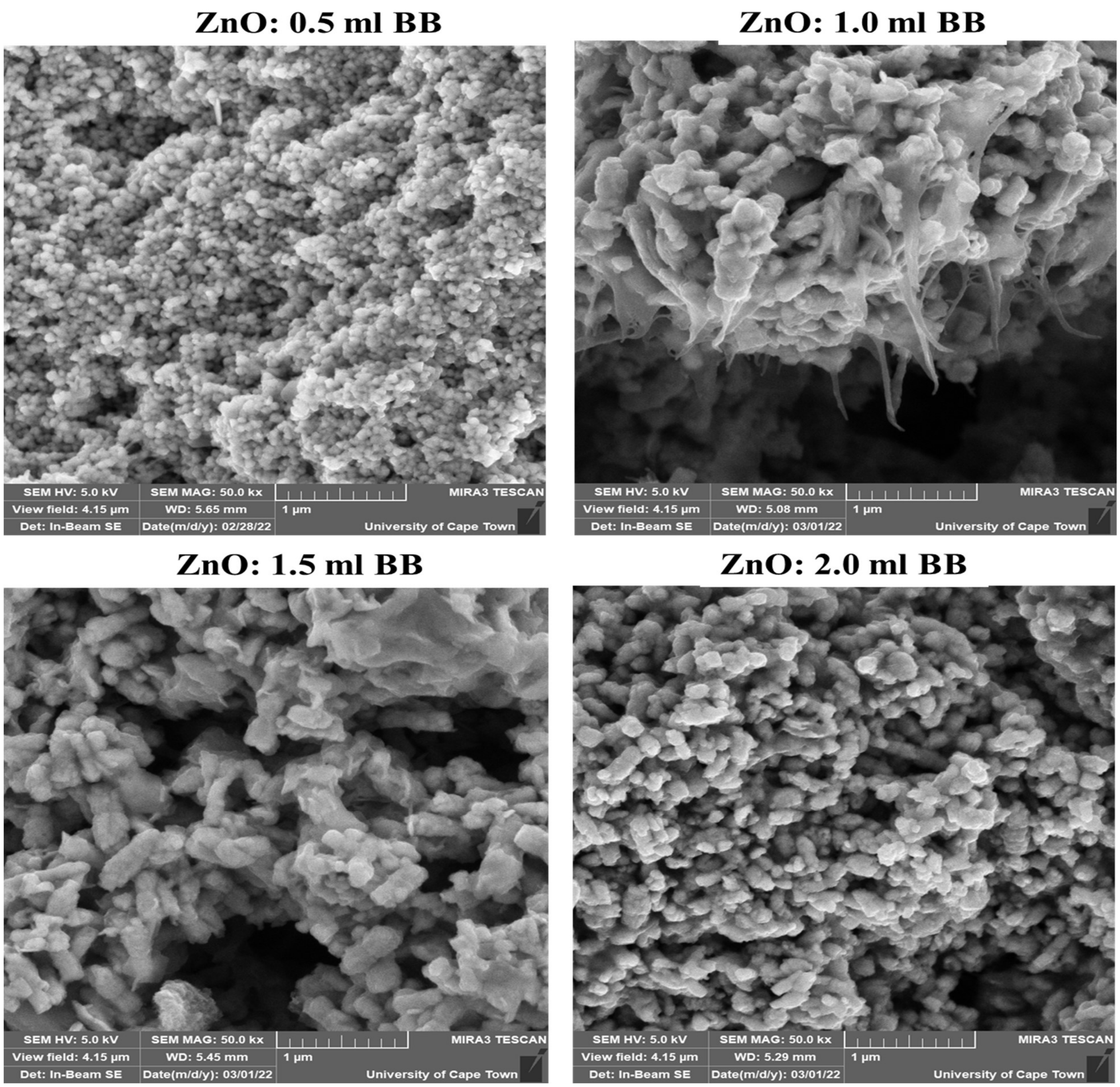


Figure 1.9: SEM image of zinc oxide nanoparticles

Figure 1.9 shows the SEM image of zinc oxide nanoparticles. The SEM image revealed the formation of agglomerated tiny spherical – like nanoparticles of different sizes. Average particle

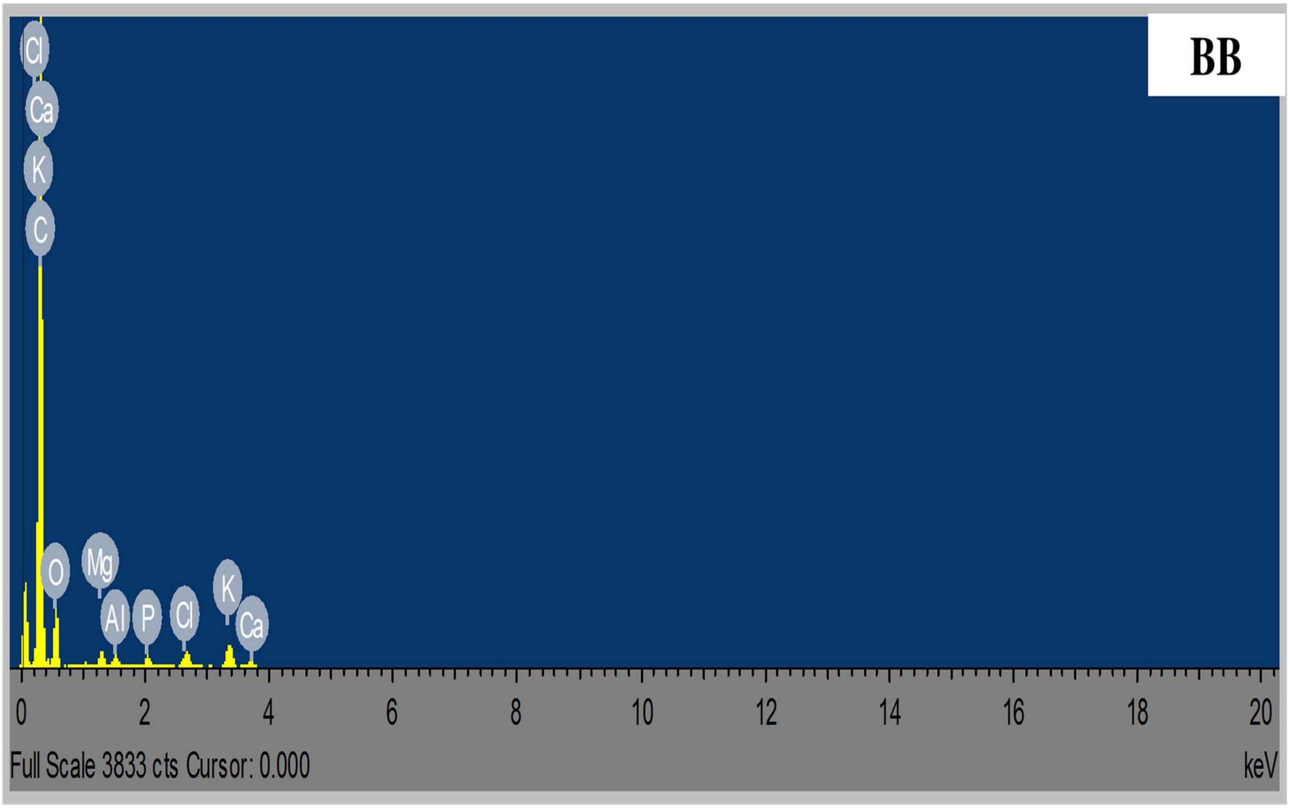
2.6. Micrographs of charred bamboo stem doped ZnO composites

Figure 1.10, 1.11, 1.12,and 1.13 shows the SEM images of zinc oxide nanoparticles doped with charred bamboo stem (BB) extract of 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml respectively. The SEM images revealed the formation of tiny nanospheres and nanorods. The transformation from nanospheres to nanorods was observed as amount of BB increased from 1.0 ml to 2.0 ml. zinc oxide nanoparticles doped with 0.5 ml of BB contained nanopheres with 27.88 nm. Zinc oxide doped 1.0 ml of BB contained more of nanospheres with some few nonarod – like structures. Zinc oxide doped with 1.5 ml and 2.0 ml revealed the formation of more of nanorod – like structure with little or no nanospheres present. This result shows that addition of BB to the zinc oxide has significant influence in the morphology of zinc oxide nanoparticles. Average particle sizes of 25.11 nm, 27.69 nm, 38.85 nm and 31.54 nm were obtained for zinc oxide doped with 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml of BB respectively.


ZnO: 0.5 ml BB
SEM HV: 5.0 kV
SEM MAG: 50.0 kx
MIRA3 TESCAN
View field: 4.15 µm
WD: 5.65 mm
1 µm
Det: In-Beam SE
Date(m/d/y): 02/28/22
University of Cape Town
ZnO: 1.0 ml BB
SEM HV: 5.0 kV
SEM MAG: 50.0 kx
MIRA3 TESCAN
View field: 4.15 µm
WD: 5.08 mm
1 µm
Det: In-Beam SE
Date(m/d/y): 03/01/22
University of Cape Town
ZnO: 1.5 ml BB
SEM HV: 5.0 kV
SEM MAG: 50.0 kx
MIRA3 TESCAN
View field: 4.15 µm
WD: 5.45 mm
1 µm
Det: In-Beam SE
Date(m/d/y): 03/01/22
University of Cape Town
ZnO: 2.0 ml BB
SEM HV: 5.0 kV
SEM MAG: 50.0 kx
MIRA3 TESCAN
View field: 4.15 µm
WD: 5.29 mm
1 µm
Det: In-Beam SE
Date(m/d/y): 03/01/22
University of Cape Town

2.7 Elemental compositional analyses

2.8 Elemental compositional analyses of charred bamboo stem (BB) particles

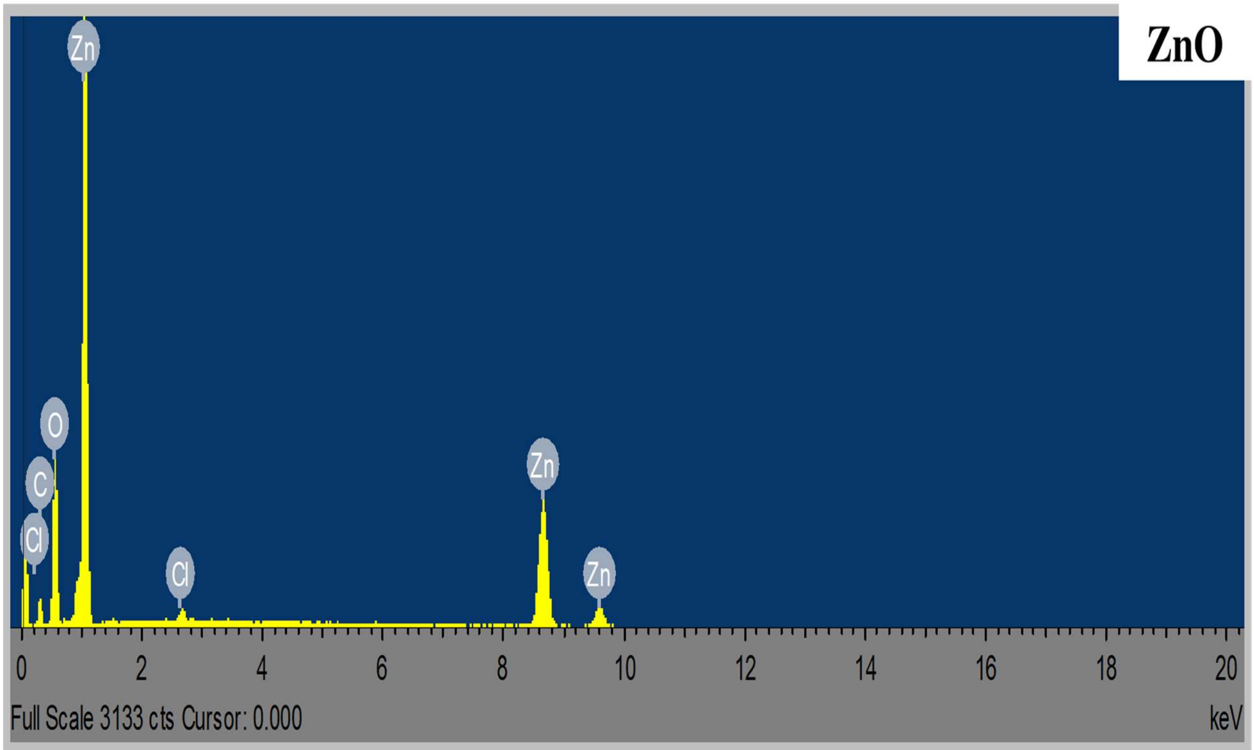


Elements	C	O	Mg	Si	P	S	Cl	K	Ca	Total
Atomic %	79	13.94	0.17	1.72	0.56	0.11	0.63	3.62	0.25	100

Figure1.10 : EDS spectrum of charred bamboo stem (BB) particles

Figure 1.10 shows the elemental compositions of charred bamboo stem (BB) particles. The result shows that BB particles contained 79.00% of carbon (C), 13.94% of oxygen (O), 0.17% of chlorine, 3.62% of potassium and 0.25% of calcium. The result showed that the particles is rich in carbon.

2.9 Elemental compositional analysis of ZnO nanoparticles

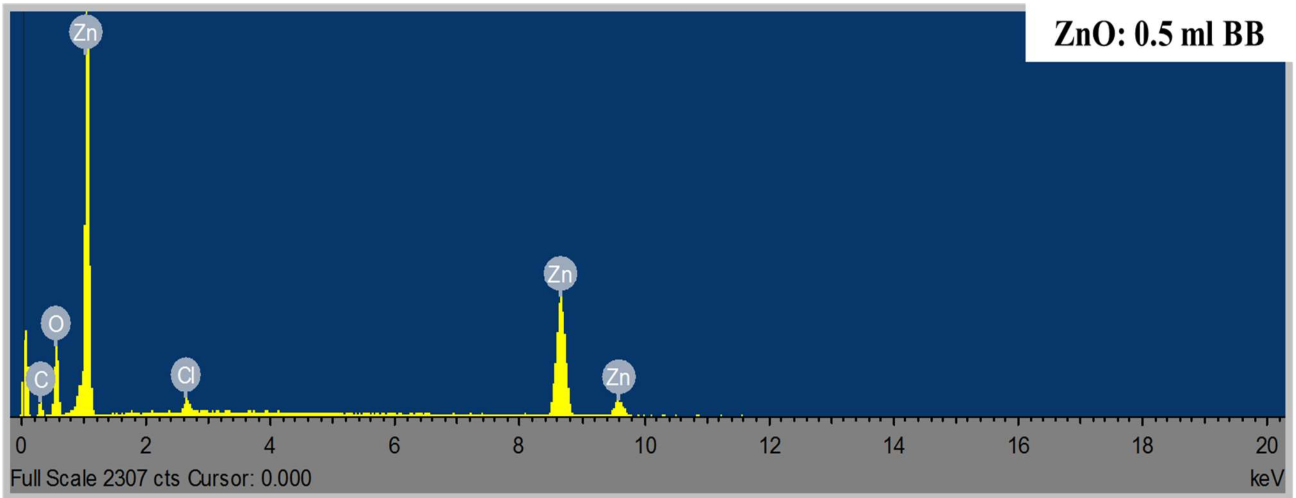


Elements	C	O	Cl	Zn	Total
Atomic %	5.89	20.64	0.7	72.77	100

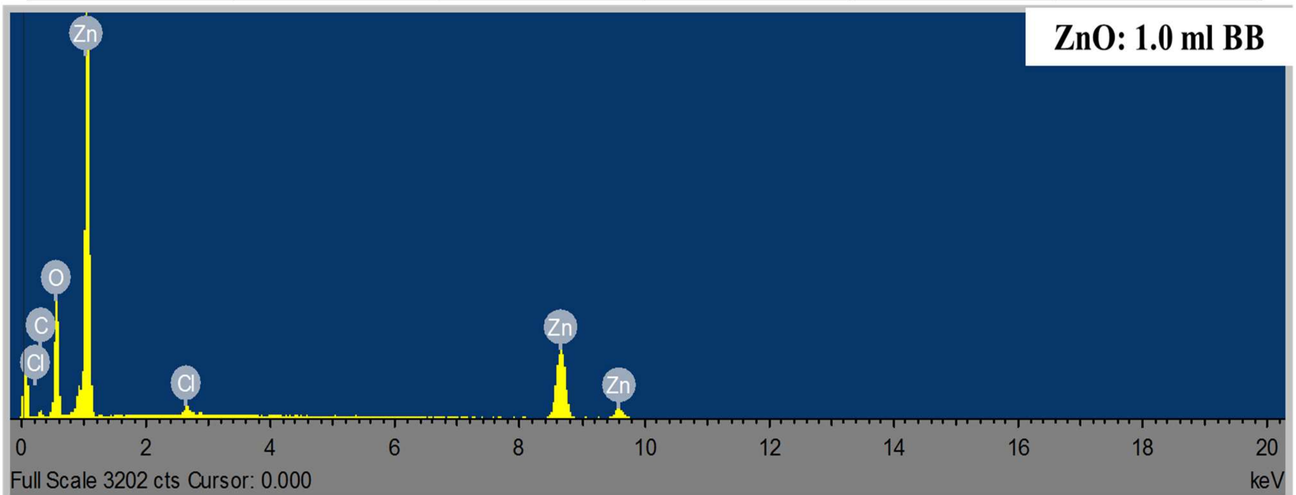
Figure 2.0: EDS spectrum of zinc oxide nanoparticles

Figure 2.0 shows the elemental compositions of zinc oxide nanoparticles. The result shows that the nanoparticles contained 72.77% of zinc (Zn), 20.64% of oxygen (O), 5.89% of carbon (C) and 0.70% of chloride (Cl). The result showed that the nanoparticle is rich in zinc.

3.0 Elemental compositional analyses of charred bamboo stem doped ZnO composites



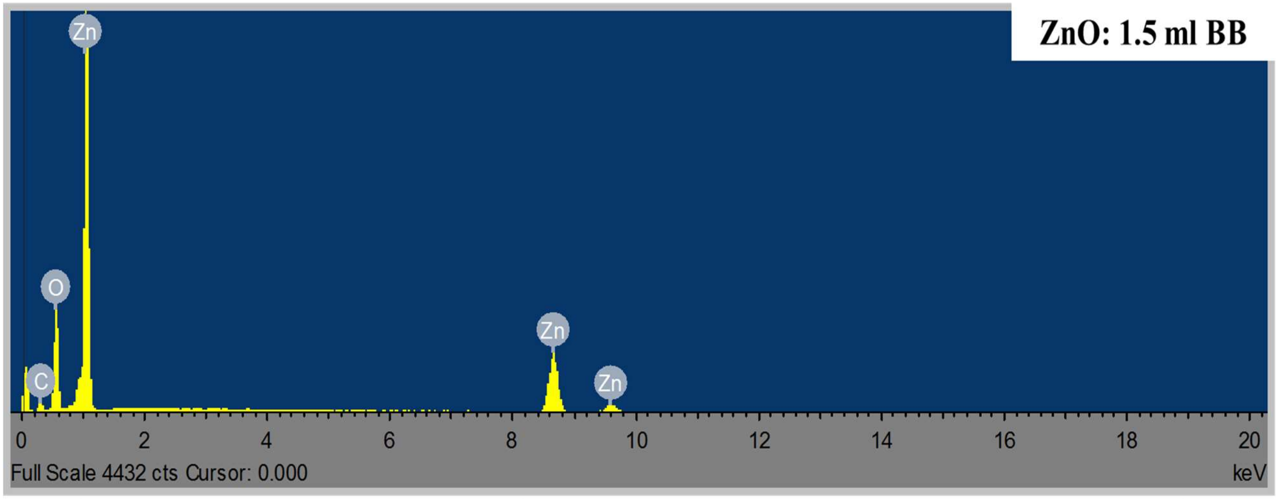
Elements	C	O	Cl	Zn	Total
Atomic %	13.52	12.25	1.58	72.65	100



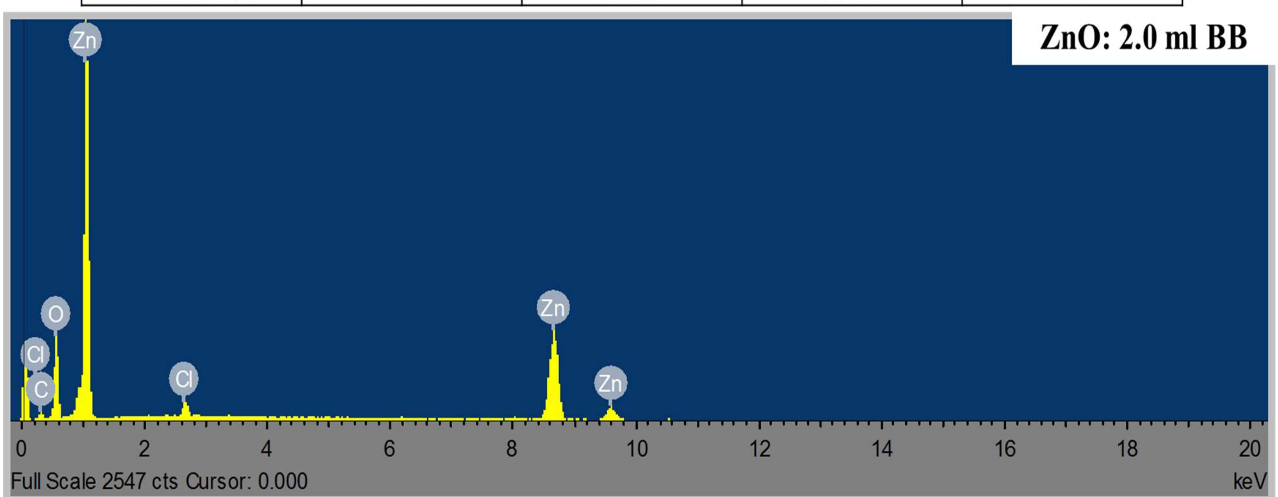
Elements	C	O	Cl	Zn	Total
Atomic %	7.51	31.02	1	60.47	100

Figure 2.1: EDS spectra of BB doped zinc oxide nanoparticles formed with 0.5 ml and 1.0 ml of BB

Figures 2.1 and 2.2. show the elemental compositions of zinc oxide doped with charred bamboo stem particles. Figure 2.3. gives the EDS spectra of zinc oxide doped with 0.5 ml and 1.0 ml of BB extract. The results showed that zinc oxide doped with 0.5 ml of BB extract contained 72.65% of zinc (Zn), 12.25% of oxygen (O), 13.52% of carbon (C) and 1.58% of chloride (Cl). Zinc oxide doped with 1.0 ml of BB extract contained 60.47% of zinc (Zn), 31.02% of oxygen (O), 7.51% of carbon (C) and 1.00% of chloride (Cl). Figure 1.17 gives the EDS spectra of zinc oxide doped with 1.5 ml and 2.0 ml of BB extract. The results showed that zinc oxide doped with 1.5 ml of BB extract contained 60.19% of zinc (Zn), 31.69% of oxygen (O) and 18.12% of carbon (C). Zinc oxide doped with 2.0 ml of BB extract contained 59.05% of zinc (Zn), 32.14% of oxygen (O), 8.14% of carbon (C) and 0.67% of chloride (Cl).



Elements	C	O	Zn	Total
Atomic %	8.12	31.69	60.19	100



Elements	C	O	Cl	Zn	Total
Atomic %	8.14	32.14	0.67	59.05	100

Figure 2.4: EDS spectra of BB doped zinc oxide nanoparticles formed with 1.5 ml and 2.0 ml of BB

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DISCOSION

Metal oxide nanoparticles such as zinc oxide (ZnO), have been synthesized through sol gel approach. Nancomposite formed by doping the metal oxide with biochar were ZnO doped with bamboo stem biochar (BB:ZnO) (BB:SnO_2), Successful synthesis of this metel oxide was done using aqueous solutions of zinc (II) acetate dihydrate, and sodium hydroxide as precursors of zinc. Absolute ethanol and ethylene glycol were used as nanoparticle stabilizer and capping agent. Biochar of bamboo stem was prepared by pyrolysis of the air dried bamboo stem. Aqueous solutions of these biochar was prepared and used as dopant solution. Synthesized ZnO nanoparticle and it's corresponding nanocomposites was subjected to optical, crystal structural, morphological, compositional analyses. Optical properties were determined using UV-VIS spectrophotometer. Crystal structural properties was carried out using x-ray diffractometer. Morphological and compositional properties were obtained using scanning electron microscope (SEM) equipped with energy dispersive x-ray spectroscope (EDS).

Optical properties of synthesized nanoparticles and nanocomposites studied include absorbance, transmittance, reflectance, refractive index, and energy bandgap. X-ray diffraction spectra of nanoparticles and nanocomposites were obtained between the 2 theta angle range of 15 ° to 70 °. Crystal phases of the nanoparticles and nanocomposites were determined. From the x-ray diffraction spectra, crystallite size, dislocation density and microstrain were obtained. SEM images obtained for the nanoparticles and nanocomposites were used to determine average particle sizes of the samples. Also, morphology of the nanoparticles and nanocomposites were observed. EDS results of the samples obtained at 20.0 Kx magnification confirmed the presence of elements such as zinc (Zn), copper (Cu), tin (Sn), oxygen (O) and traces of other elements in the synthesized nanoparticles and nanocomposites.

CONCLUSION

Studied properties of synthesized ZnO nanoparticle and it's biochars doped nanocomposites showed that this nanoparticles and nanocomposites possessed a good attributes for industrial applications. Bamboo stem (BB) biochar has absorbance that ranged from 29.07 – 36.61%. Transmittance of Bamboo stem (BB) biochar ranged from 40.04 – 50.15%. ZnO nanoparticles,and BB:ZnO nanocomposites possessed absorbance and transmittance values that ranged from 4.22 – 45.08%. Absrobance values of the nanoparticles and nanocomposites were found to decrease as wavelength increased from 300 – 1100 nm. Also, transmittance of the nanoparticles and nonacomposites were found to increase as wavelength increased from 300 – 1100 nm. This implies that the absorbance values of the nanoparticles and nanocomposites were observed to be high within UV and VIS regions and low within NIR region. Transmittance values of the nanoparticles and nanocomposites are observed to be low in UV and VIS regions but reached maximum values within NIR region. Addtion of biochar of bamboo stem was found to increase the absorbance of these metal oxide which in turn resultes in decrease in transmittance of the metal oxides. This implies that increased presence of biochar advanced the possibility of using this biogenic metal oxide nanocomposite in absorber layer of solar cell and other optoelectronic devices that required high absorbing layers.

This nanoparticles and nanocomposites showed low reflectance values in all wavelength regions studied with maximum reflectance values always observed within UV region. The overall reflectance values of the nanoparticles and nanocomposites fall between ≥5.04% and ≤20.35%. Refractive index values of the nanoparticles and nanocomposites ranged from 1.58 to 2.64.

Band gap energy values of bamboo stem biochar were found to be 2.15 eV. Energy band gap of ZnO was found to be 3.25 eV while that of BB:ZnO was found to range from 3.20 – 2.95 eV. Presence of biochar (BB) was found to decrease the energy band gap of ZnO nanoparticles, while that of BB:ZnO was found to range from 3.30 – 3.10 eV. This results obtained in this work showed the possibility of modifying the optical properties of ZnO, using biochar derived from biomass materials of bamboo stem. These energy band gap values showed that these metal oxide and their biochar based nanocomposites are wide band gap semiconductor (WBGS) materials which are found applicable in light emitting diodes, photovoltaic devices, laser band devices and other high-temperature optoelectronic devices.

Structural analyses of the Bamboo stem (BB) biochar showed that they are of hexagonal carbon structure. XRD spectra of the nanoparticles and nanocomposites confirmed the formation of hexagonal phase of ZnO. Additon of BB biochar led to increase in peak intensity and crystallite sizes of the nanocomposites. Also, decrease in dislocation densities and microstrains were observed as amount of BB increased, showed a decrease in crystallite size with corresponding increase in dislocation density and microstrain. Crystallite sizes of ZnO, was found to be 17.693 nm. Dislocation densities of ZnO, was found to be 3.391×10^{15} lines/m^2. while microstrain values of ZnO was found to be 5.027×10^{-3}. Crystallite sizes of BB:ZnO nanocomposites ranged from 18.613 – 20.126 nm while values of dislocation density ranged from 2.886×10^{15}. Microstrain values of ZnO based biochar nanocomposites was found to be 4.827×10^{-3} Structural results of the nanoparticles and nanocomposite showed that addition of biochars derived from bamboo stem (BB) could be used to alter the crystal structural parameters of ZnO nanoparticles.

Surface morphology study revealed that this nanoparticle and nanocomposite are made up of particles of different sizes, orientations and shapes. Majority of the SEM images revealed that the surfaces are made up tiny spherical – like particles and sometimes made up of nanoplates and nanorods. Average particle sizes of nanoparticles and nanocomposites obtained using ImageJ software for image analysis showed that the particles of the metal oxides and their corresponding composites are in nanoscale.

The results of characterizations and analyses of this nanoparticles and nanocomposites showed that it could be use in photovoltaic devices, light emitting diode (LED), photodetector, LASER based and optoelectronics devices.

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