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Publications

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ONLINE ISSN: 2992-5819
PRINT ISSN: 2992-6149

NEWPORT INTERNATIONAL JOURNAL OF SCIENTIFIC AND EXPERIMENTAL SCIENCES (NIJSES)

Volume 4 Issue 2

https://doi.org/10.59298/NIJSES/2023/11.5.5181

Synthesis and characterization of zinc oxide nanoparticles

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ABSTRACT

Zinc Oxide (ZnO) nanoparticles were synthesized by hydrothermal method and analyzed for various properties. Analysis of the XRD spectra of the synthesized nanoparticles showed 46 % crystalline zincite and the average grain size was calculated to be around 39.63 nm, studies of the FTIR spectra measured over the range between 4000–500cm^{-1} proved the presence of Zn Obonding of the synthesized nanoparticles at 734.26 to 1473.71 cm^{-1}.SEMimage of the synthesized nano particles showed the formation of agglomerated nanoparticles of different shapes whose size are within the nanometer range while, cyclic voltamo gram of the immobilized ZnO nanoparticles on GCE also showed significant increase in redox peak current when compared to the bare (un-immobilized) GCE. It can be concluded from the obtained results that ZnO nanoparticles have been successfully produced which can be used to improve the performance of electrochemical sensors.

Keywords :- *XRD analysis, FTIR studies, SEM image analysis, Cyclic voltammogram*

INTRODUCTION

Metal oxide nanoparticles have found a wide range of applications in the fields of science and innovation nowadays, owing to their distinctive electrical, mechanical, optical, and magnetic characteristics [1]. Because of its vital applications as catalytic agents, photovoltaic cells, textile fabrics, and polymers, zinc oxide nanoparticles (ZnO NPs) have garnered a considerable deal of interest from researchers in recent years [2]. Zinc oxide (ZnO) is a wide band gap (3.37 eV) semiconductor with a large exciton binding energy (60 mV) and one of the most widely used and studied functional oxides [3], it is also one of the most preferred materials in material science researchfield [4]; [5] Zinc oxide is a low-cost material and easily available in nature[6]. Zn On a noparticles are transparent to visible part of light and absorbing UV radiations. It is less toxic, high resistant and durable material [7]. Morphology of ZnO nanoparticles can be easily modified [8]. Electron mobility is very high for ZnO nanostructures [9]. Several techniques such as chemical vapour deposition, spray pyrolysis, sol-gel method, hydrothermal method et care readily available for the synthesis of nano materials. However, preparation techniques play a very important role in determining the size and shape of nanoparticles [10]. Hydrothermal method has been attracted many researchers due of its distinct advantages likes imple equipment, low cost and mild preparation in conditions [3].It is an environment friendly technique. Size and shape of nanomaterials can be modified by hydrothermal method. Morphology of nanoparticles mainly depends on reaction time, temperature and concentration of reacting solutions. This in turn affects the physical and chemical properties of nanoparticles. Particles are choosing for various applications according to their morphology [11]. The present work presents the synthesis and characterization of ZnO nanoparticles obtained through a homogeneous phase reaction between zinc acetate and sodium hydroxide at high temperature. The particles were then characterized, by evaluating their crystallinity through X-ray diffractometry, their chemical composition through FTIR spectroscopy, their shape

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and size via SEM microscopy, and the specific surface area. The electrochemical behavior of the synthesized ZnO nanoparticles were also studied using cyclic voltammetry

Experimental

Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) and sodium hydroxide were purchased from Yola scientific, yola Adamawa state. Double distilled water was used as the solvent throughout the experiment.

Synthesis of ZnO nanoparticles

ZnONPs were synthesized according to the method described by [3]. 0.070 g of $Zn(CH_3COO)_2{\cdot}2H_2O$ and 0.400 g of NaOH were dissolved into 40 mL of distilled water. After the mixture was magnetically stirred for 20 min and this solution was transferred into a 50 mL Teflon-lined stainless steel autoclave. It was then sealed and maintained at 180 °C for 2 h. After slowly being cooled to room temperature, obtained powders were collected by centrifugation and washed with distilled water and absolute ethanol. The powders were finally dried at 60 °C for 12 h.

Characterization methods

The crystal structure of ZnO nanoparticles was analyzed by a Rich Siefert 3000 diffractometer with Cu-Kα1 radiation (λ = 1.5406 Å). FT-IR spectrum of the ZnO was recorded on Schimadzu FT-IR 8300 series instrument by using potassium bromide pellets. The morphology of the materials was analyzed by SEM HITACHI SU6600 scanning electron microscopy respectively. The electrochemical experiments were performed on electrochemical work station AUTOLAB Metrohm Dropsens using bare GCE as working electrode, a platinum wire was the counter electrode and saturated calomel electrode (SCE) was the reference electrode.

Electrode modification

Ultrasonically dispersed ZnO nanopowder in 5 mL of water was drop coated onto the GCE and dried at room temperature. CV's were run in electrochemical cell containing 5 M Ferri/Ferro solution, 1M KCl at 50 mV^{-s}. in presence of SCE as a reference electrode and Pt wire as counter electrode.

RESULTS AND DISCUSSION

XRD Analysis of ZnO nanoparticle

Crystal structure of the sample was analysed by the use of XRD spectrometer. XRD patterns of the ZnO nanoparticles prepared by hydrothermal method is shown in Figure 1.The intense peaksappearedatdifferent2θ,°values for hexagonal Zincite structure of ZnO nanoparticles. Other peaks and relative intensities obtained for the ZnONPs revealed the presence of franklinite, Gahnite, Cristobalite and illite at different percentage as depicted in figure 2. This result matched with other literatures[3];[12]. There were little characteristic peaks of impurity observed. Average grain size of the ZnONPs was found to be around 39.63 nm. Average grain size of ZnO was determined using Debye Scherrer formula:

$$D = 0.9\,\lambda / \beta \sin\theta \quad \text{---------- equ. 1}$$

Where Disthecrystallitesize, λ is the incident radiation wavelength, βisthefull widthat half-maximum of the ZnO lineand θ is the diffraction angle.

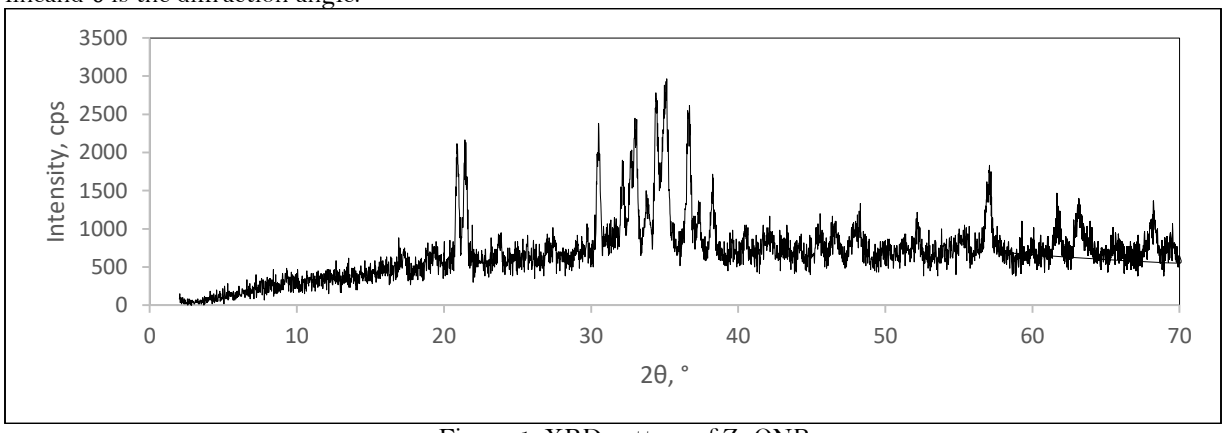

Figure 1. XRD pattern of ZnONPs

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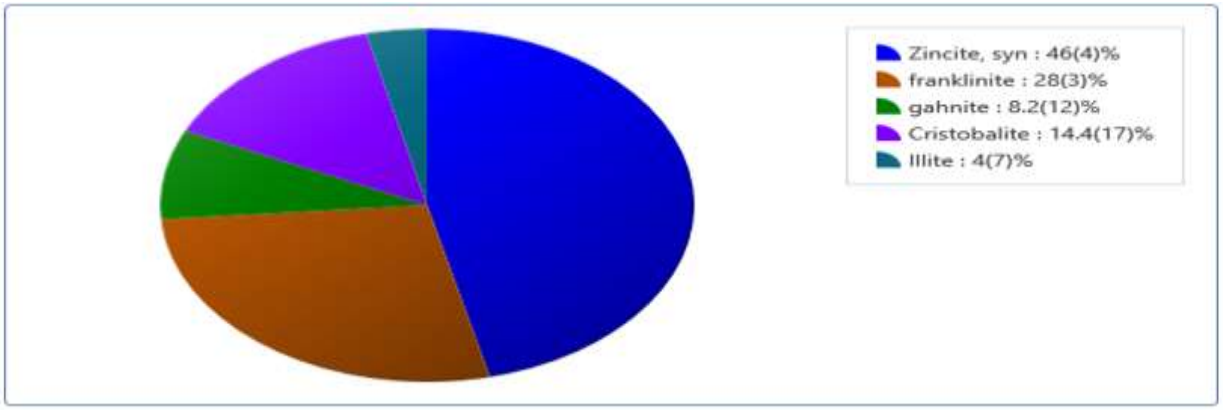

Fig. 2 percentage composition of components of the ZnONPs

FT IR analysis

FTIR analysis offers a valuable means to examine the bonding between neighboring structures in practical applications.[13] Measurements were conducted in the range of 4000–500 cm−1, revealing significant absorption peaks at various wave numbers, including 3383.75, 2985.87, 2482.25, 2318.97, 1740.82, 1649.41, 1563.00, 1473.71, 914.76, and 734.26 cm−1, as illustrated in Fig. 3, for the ZnO nanoparticles synthesized via the hydrothermal method. These spectra showcased distinct peaks corresponding to different ion vibrations within the crystal lattice[14];[15]. Specifically, peaks within the range of 734.26 to 1473.71 cm−1 were attributed to Zn-O stretching vibrations[14];[3].

The range from 2361.97 to 2482.25 cm−1 might be associated with CO2; atmospheric carbon dioxide could cause absorption in this region if not properly eliminated. Additionally, the band at 3383.75 reflects C-H stretching, while the peak around 2985.87 indicates asymmetric and symmetric bending of carbonyl groups. These functional groups suggest the presence of organic impurities in the synthesized ZnO nanoparticles. A weaker band near 1740.82 cm−1 is assigned to H-O-H stretching vibrations, potentially due to water molecule oscillation [16];[17]. Moisture might be present from when the FTIR sample disks were prepared in an open atmosphere, indicating hydration within the structure [18]. Nevertheless, FT-IR analysis has confirmed the presence of metal oxide bonding and the purity of the ZnO nanoparticles. Additionally, the spectra indicated a crystalline structure for the synthesized ZnO nanoparticles, with some chemical substances absorbed on the surface. These findings confirm the potential use of metallic oxide (ZnO) nanoparticles to enhance biosensor performance.

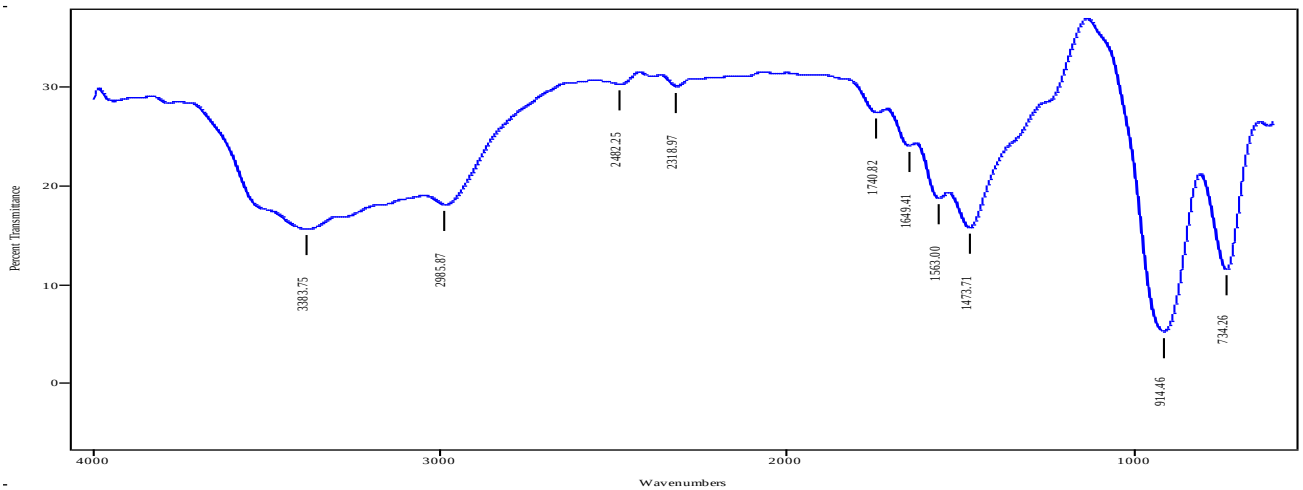

Fig. 3. FT-IR Spectra of ZnO nanoparticles

SEM Analysis of ZnO Nanoparticles

Fig.4. A and B showed the particle size distribution and SEM microgram of ZnO nanoparticles synthesized through the hydrothermal method. Analysis of the SEM images reveals that the particles adopt irregular morphology with different sized particle in the nanometer range, average particle size of 41.18 ± 0.71 nm was obtained using image J software. It is also observed that the formation of agglomerates were purely due to the

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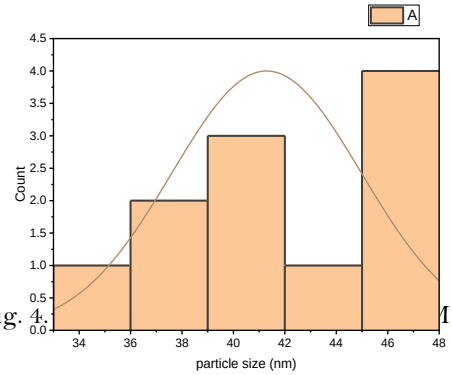
synthetic condition as reported by [3] in a similar work titled "Hydrothermal Synthesis of Hydrated Zinc Oxide Nanoparticles and its Characterization". Agglomeration is a known property of a nanoparticles which is attributed to their large surface area to volume ratio and high surface energy, the interlinked porous network may provide many more pathway through the network to make contact between the electrode materials and the electrolyte ions which may lead to improved electrochemical performance of electrodes [19].



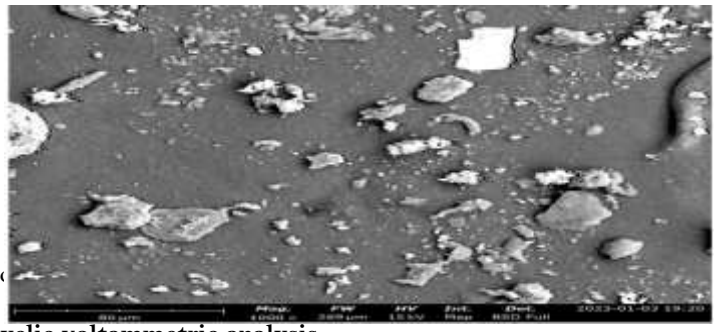


Fig. 4. M mi

Cyclic voltammetric analysis

The electrochemical behaviour of ZnO/GCE was examined by cyclic voltammetry. Figure 5. showed the cyclic voltamogram of bare and ZnO/GCEat a fixed scan-rate of 50 mVs^{-1} in 5 mM [$Fe(CN)_6$]containing 0.1 M KCl solution as a supporting electrolyte. It can be seen that the ZnO is non-electroactive in the selected potential region. However, it shows enhanced peak current than the bare GCE which indicated that the ZnONPs can be used for enhancement of modified electrode in electrochemical sensors.

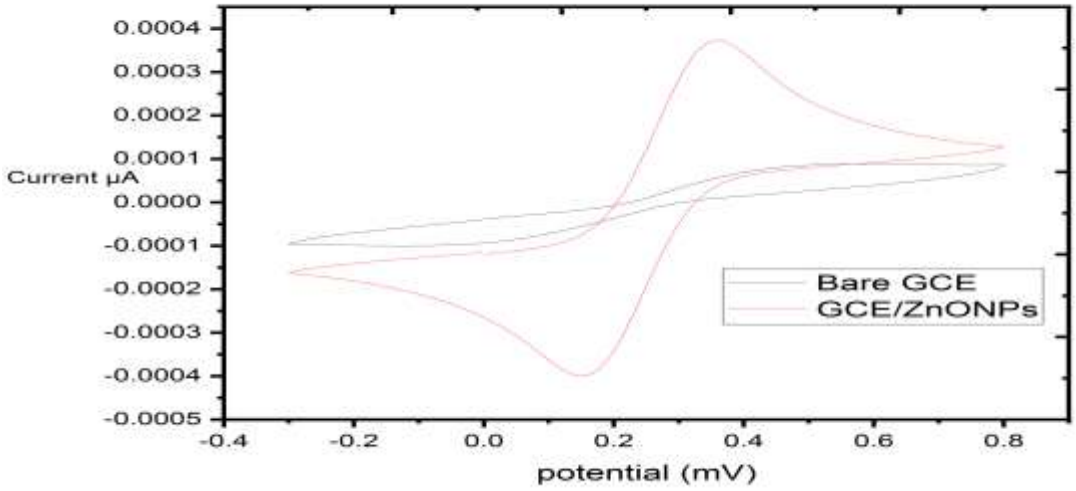


Fig. 5. Cyclic voltamogram of bare and ZnONPs immobilized on GCE

CONCLUSION

ZnO nanoparticles were successfully synthesized by hydrothermal method. The XRD confirms the crystal structure and phase purity of the sample. FT-IR analysis confirms the formation of the Zn-O bond in the ZnO nanoparticles. The SEM of ZnO nanoparticles shows the formation agglomerated particles of different shapes within the range of nanometer. The electrochemical behaviour of the ZnO nanoparticles modified GCE was investigated by cyclic voltammetry. The results conclude that the ZnO nanoparticles will have potential application in the electrochemical sensor.

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CITE AS: Buba Mohammed, Maitera O.N, and Bala Suleiman (2023). Synthesis and characterization of zinc oxide nanoparticles. NEWPORT INTERNATIONAL JOURNAL OF SCIENTIFIC AND EXPERIMENTAL SCIENCES 4(2):40-44. https://doi.org/10.59298/NIJSES/2023/11.5.5181