ZnO nanoparticles preparation from spent zinc–carbon dry cell batteries: studies on structural, morphological and optical properties

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ABSTRACT
Recovery and recycling of valuable materials from battery waste is challenging as well as important from economic and environmental point of view. In this work, spent zinc–carbon batteries were processed with HCl and NaOH to synthesize ZnO nanoparticles. ZnO nanopowders were also prepared by using ZnCl2 as precursors with varying amount of NaOH using sol–gel method. In both cases, ZnO nano particles were characterized with XRD, XRF, FESEM, EDAX, particle size analyzer and UV–visible (UV–Vis) spectroscopy. The purity of the synthesized ZnO from spent zinc–carbon batteries was found to be 99.69%. FESEM micrographs showed the reduction in particle size from 50 nm to 20 nm with a decrease in NaOH concentration. UV–Vis study showed significant peak shift with maximum absorption between 365 nm and 359 nm region of the visible spectrum. Because of these remarkable properties, as prepared ZnO nanoparticles can find applications in biomedicine, nanoelectronic devices, sensors, nanomedicine, GATE dielectrics, photovoltaic devices, etc.

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1. Introduction
Zinc–carbon-type dry-cell batteries are frequently used to operate many electronic and electrical appliances in our daily life with a limited life. These wastes batteries usually contain valuable materials. Recovery and recycling of valuable materials from these batteries reduces the amount of wastes to be finally disposed and thus reduce the waste disposal problems [1]. However, recycling is still not a favorable process particularly in developing countries where economic interests supersede environmental obligations. In this scenario, the recovery of valuable products from the spent batteries not only reduces the environmental hazards but also minimize the loss of these valuable metallic elements [2].

According to Environmental Protection Agency (USA), zinc can be marked as one of the 129 priority pollutants. In human body, prolonged excessive exposure to zinc can cause deficiencies in iron and copper, and also causes nausea, fever, headache, tiredness, and abdominal pain [3]. As a consequence of these efforts, zinc is now being extracted from several sources, i.e. from zinc scrap, galvanizing plant dross and ash, leach residues, spent primary batteries, etc. From these resources spent zinc–carbon-type batteries are important to recover zinc because they constitutes almost 25% of the total weight of these type of batteries [1].

Nowadays, there has been an increasing demand for the development of multifunctional semiconductors which are nanosized due to their significant electrical and optical properties. They are highly significant in fabricating nano-scaled optoelectronic and electronic devices with multifunctionality [4–6]. Among various semiconducting materials, zinc oxide (ZnO) is a distinctive electronic and photonic n-type semiconductor material with a wide direct band gap of 3.37 eV and a high exciton binding energy (60 meV) at room temperature, which make it attractive for potential use in electronics, optoelectronics and laser technology [7]. The piezo- and pyroelectric properties of ZnO reveals that it can be used as a sensor, converter, energy generator and photocatalyst in hydrogen production [8,9]. Metal and metal oxide nanoparticles when embedded and smeared on to surfaces, can find immense applications in water treatment, industrial applications, synthetic textiles, biomedical and surgical devices, food processing and packaging [10]. Because of its distinctive properties such as: hardness, rigidity, low toxicity, biocompatibility and biodegradability, ZnO became a material of interest for biomedicine and in pro-ecological systems [11–13].

Profound studies have been done on various factors, such as precursor concentrations, temperature, duration, dopant concentration and solvent medium, pH of the reaction mixture which individually and collectively affects the shape, size and optical

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properties of ZnO nanoparticles. Various researchers revealed that NaOH concentration plays a vital role in determining the morphology, size, shape and optical band gap of obtained nanostructures [14].

It was found that various approaches for the preparation of ZnO nanopowders have been developed, namely, sol–gel, micro emulsion, thermal decomposition, spray pyrolysis, electrodeposition, ultrasonic, chemical vapor deposition, hydrothermal and precipitation methods. Most of these techniques were not thoroughly used on a large scale, but chemical synthesis has been extensively used due to its simplicity and low cost [15–17]. In the present study, synthesis of ZnO nanoparticles was done from spent batteries and comparison of the characteristics of these nanoparticles were made with particles obtained from analytical grade ZnCl₂ precursor in order to understand the quality of the products. After optimizing the process parameter with analytical grade ZnCl₂ precursor initially, ZnO nanoparticles were finally obtained and characterized from battery waste in this work. The novelty of this work is to utilization of battery waste materials into valuable nanosized particles which are environmentally feasible. Also shorten steps in the synthesis process of ZnO nanoparticles for reducing overall cost during synthesis and produce high purity of products.

2. Experimental

2.1. Materials

Spent Zinc–carbon batteries of DC 1.5 V size C (R14 P) batteries were collected after their end-of-life in electronic appliances. All of the reagents are of analytical grade (i.e. purity higher than 99.9%) and used without further purification. Hydrochloric Acid (HCl, ≥37%, ACS, ISO, reagent grade, Merck-KgaA, Germany) and sodium hydroxide (NaOH, ≥97%, Merck, Germany) were used as received. Analytical grade Zinc chloride (ZnCl₂, ACS reagent, grade, Merck-KgaA, Germany) was also used. The solutions were prepared by using di-mineralized water.

2.2. Synthesis of zinc oxide nanoparticles from analytical grade precursors

In order to synthesize the ZnO nanoparticles by wet chemical method, stock solutions of zinc chloride was prepared by dissolving ZnCl₂ salt in 100 mL of di-mineralized water in a 250 mL beaker. It was stirred continuously with magnetic stirrer and its temperature was raised to 80°C. Once the temperature of zinc chloride solution was reached to 80°C, then 2–3 drops of fuming hydrochloric acid were added drop wise for completing dissolution of zinc chloride in di-mineralized water. The pH of solution was kept ~2 and stirred continuously for 1 h. Meanwhile, 10 g of NaOH was dissolved in 100 cc of di-mineralized water in a separate vessel. From the prepared solution, 20 mL of 2.5 M sodium hydroxide was added slowly in the solution with constant stirring. In this case, the solution pH was maintained ~12. The aqueous solution turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide. After the completion of reaction, solution was allowed to settle and the supernatant was removed by centrifuge at 8000 rpm for 10 min washing with distilled water five times. After centrifugation, the powder was allowed to dry in vacuum drier at 70°C for 12 h.

The same procedure was followed to prepare zinc oxide using 5 M and 7.5 M of NaOH concentrations while keeping zinc chloride dihydrate concentration fixed at 0.4 M as shown in the flow chart below in Figure 1. All the samples were centrifuged, dried and the powders were then kept in air-tight vials.

2.3. Synthesis of ZnO nanoparticles from spent batteries

According to the exploration and finding of 2.2 in case of concentration variation from 2.5 to 7.5, 2.5 M concentration was chosen for synthesis of ZnO nanoparticles from spent batteries.

In this case, synthesis of ZnO nanoparticles by wet chemical method, the whole battery was dismantled manually and weight distributions of different component parts were measured. The amount of zinc metal scrap was 11.2 g present in each battery shell and from which weighed 5.4 g has taken for the procedure.

According to work plan small amount of Zn metal shred reacts with concentrated HCl for the formation of ZnCl₂ and H₂ gas.

\[
\text{Zn} + 2\text{HCl} \rightarrow \text{ZnCl}_2 + \text{H}_2
\]

The reaction was quite vigorous. After 4 h from the beginning of the process, a pH meter in the solution indicates that the content is still strongly acidic and pH was ~1, even though the unreacted zinc have stopped bubbling. The produced ZnCl₂ was very hygroscopic and difficult to found in solid form. In this case, adding 40 cc of di-mineralized water was added to form 0.2 M 30 cc of prepared ZnCl₂ solution, and was stirred continuously with magnetic stirrer until its temperature was raised to 80°C. Meanwhile 10 g of NaOH was dissolved in 100 cc of di-mineralized water in a separate vessel. From the prepared sodium hydroxide solution 20 ml of 2.5 M sodium hydroxide was added slowly in the solution with constant stirring, once the temperature of acidic zinc chloride solution was reached to 80°C. In this
case, the solution pH was maintained ~12. The aqueous solution turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide. After the completion of reaction, solution was allowed to settle and the supernatant was removed by centrifuge at 8000 rpm for 10 min washing with distilled water five times. After centrifugation, the powder is allowed to dry in Vacuum drier at 70°C for 12 h. Overall reaction in the process:

\[
\begin{align*}
\text{Zn} + \text{HCl}_{(\text{conc.})} & \rightarrow \text{ZnCl}_2 + \text{H}_2 \\
\text{ZnCl}_2 + 2\text{NaOH} & \rightarrow \text{Zn(OH)}_2 + 2\text{NaCl} \\
\text{Zn(OH)}_2 & \xrightarrow{70\degree C} \text{ZnO}
\end{align*}
\]

2.4. Nanoparticles characterization

The prepared ZnO nanoparticles from both samples were characterized for their optical and nanostructural properties. X-ray diffraction patterns for the ZnO NPs were recorded using an X-ray diffractometer (D8 Advance, Bruker AXS GmbH, Germany) with Cu Kα radiation of wavelength \(\lambda = 1.5406\ \text{Å}\), applied voltage 40 KV, tube current 40 mA for a scan range of \(2\theta = 20°\) to 90°. From the XRD analysis, full-width at half-maximum (FWHM) data were used to determine average crystallite size. The average crystallite size of the ZnO particle was calculated using Debye-Scherrer’s formula:

\[D_p = \frac{0.9\lambda}{\beta \cos\theta}\]

where \(\beta\) is the line broadening at FWHM.

The strain can be calculated by using the following formula:

\[\varepsilon = \frac{\beta}{(4 \times \tan \theta)}\]

The dislocation density, \(\delta\), defined as the length of dislocation lines per unit volume of the crystal can be evacuated from the following formula:

\[\delta = \frac{1}{D_p^2}\]

3 Results and discussion

3.1. X ray diffraction characterization

Figure 2 shows that combined XRD patterns of ZnO samples synthesized from ZnCl₂ at 2.5 M, 5 M and 7.5 M concentration of NaOH. Figure 3 shows that XRD pattern of ZnO sample synthesized from battery waste at 2.5 M concentration of NaOH.

XRD peaks indicate that synthesized material consist of particles with high purity as no impurity phases were detected. The diffraction peaks were located at 31.63°, 34.3°, 36.20°, 47.24°, 56.35°, 62.51°, 66.21°, 67.52°, 68.7°, 72.1° and 76.63°. These were indexed as hexagonal zincite phase of ZnO particles match with JCPDS-
ICDD card no 00-036-1451. The diffraction peaks for ZnO synthesized from battery waste shown in Figure 3 are also analogous with diffraction peaks obtained from analytical grade ZnCl$_2$ (Figure 2). However, two additional peaks before 30° and around 45° are evident in Figure 3. These two peaks could be attributed to the impurity phases like SiO$_2$ as this produces diffraction peaks around the above mentioned regions as per PDF 01-087-2096. Yet it is difficult to trace the impurity phases with two peaks only.

Comparing Figures 2 and 3, it is substantiate that both XRD patterns almost analogous with each other and all the samples showed major ZnO phase with clear diffraction peaks corresponding to the lattice planes in the zincite with a hexagonal structure.

Figure 2 also shows the X-ray diffractograms of the samples produced from different concentration of NaOH using ZnCl$_2$ salt precursors. This analysis was carried out to evaluate the crystalline nature and size of ZnO nanoparticles with increase in concentration of NaOH in the solution. The average crystallite size of the samples S-1, S-2 and S-3 with different concentration was calculated from Equation (1) using FWHM data to be 16.34 nm, 21.11 nm and 25.81 nm, respectively, obtained from XRD diffractograms data analysis.

### 3.2. Effect of concentration on crystallite size

Figure 4 shows the variation of crystallite size of as prepared ZnO nanoparticle samples with respect to concentration variation of NaOH from 2.5 M to 7.5 M.

Figure 4 reveals that a linear and systematic increase was observed in the crystallite size with an increase in NaOH concentration from 2.5 M (sample S1) to 7.5 M (sample S3).

This is because during the synthesis process, with the increase in NaOH concentration the rate of reaction also increases and there is more chance for the formation of Zn(OH)$_2$ nuclei. Therefore more chance for the growth of the crystals. This phenomenon is also based on Ostwald ripening [18,19].

### 3.3. Effect of concentration on lattice strain

Figure 5 shows that effect of concentration varying from 2.5 M to 7.5 M of NaOH in the reaction mixture on the lattice strain of ZnO.

It reveals that the values of lattice strain in the samples decreases with respect to the NaOH concentration in the reaction mixture. Various researchers have reported that in the samples strain is inversely proportional to the crystallite size [20], which also corresponds to Figures 4 and 5.

### 3.4. Effect of concentration on dislocation density

Figure 2. Combined XRD pattern of ZnO from ZnCl$_2$ at different concentrations of NaOH.

Figure 3. XRD pattern of ZnO obtained from battery waste.

Figure 4. Variation of crystallite size of as-prepared ZnO nanoparticles with respect to concentration variation of NaOH.
Figure 6 shows that effect of NaOH concentration which varies from 2.5 M to 7.5 M on dislocation density in the synthesis of ZnO nanoparticles. It reveals that dislocation density of as-prepared nanoparticles increases with increase in the concentration of NaOH in the reaction mixture. This is based on the fact that, the amount of defects in the sample is defined as the length of dislocation lines per unit volume of the crystal. This dislocation density was calculated using Equation (3) represents atomic disorder at the boundary causes discontinuity in crystal planes. Thus lower concentration provided finer the crystallite size (Figure 4), larger grain boundary area that impedes dislocation density (Figure 6). Consequently, grain-size reduction usually improves toughness of nanoparticles as well as lower dislocation density [21].

3.5. X-ray fluorescence characterization

Table 1 shows the XRF characterization of ZnO nanoparticles obtained from zinc carbon battery source.

| Serial no. | Analytes | Composition % |
|------------|----------|---------------|
| 1          | ZnO      | 99.6918       |
| 2          | SiO$_2$  | 0.2089        |
| 3          | Fe$_2$O$_3$ | 0.0502      |
| 4          | MnO      | 0.0330        |
| 5          | NiO      | 0.0161        |

It is revealed that synthesis of ZnO nanoparticles from spent zinc carbon battery source contains 99.69% ZnO sample and very minor percentage of SiO$_2$, Fe$_2$O$_3$, MnO and NiO in the sample. Thus from this result it was substantiate that synthesized ZnO nanoparticles was highly pure.

3.6. Particle size distribution

Figure 7 shows the volume density–size curves for different concentration of NaOH of ZnO samples. It reveals that, for 2.5 M and 5 M concentration, volume density is slightly increased but in case of 7.5 M concentration, the peaks were shifted towards right with increasing the concentration of NaOH. Thus higher concentration of NaOH in the reaction mixture resulted in wider size distributions. Similar result was obtained for ZnO synthesized from battery waste as well. From the morphological characterization (3.6) it was shown that most of the particles were distributed in nanoscale range but in this case for the concentration of 2.5 M, there is an extra peak at higher size (Figure 7); which is higher than that of ZnO sample prepared using 5 M concentration. This may happen because of the moisture absorption and agglomeration of particles. Therefore, in the case of particle size distribution, micron sizes of the particles were obtained.

3.7. Morphological characterization

Figure 5. Effect of concentration on lattice strain.

Figure 6. Effect of concentration on dislocation density.

Table 1. XRF analysis for ZnO nanoparticles from spent zinc carbon battery source.

Figure 7. Particle size Analysis of ZnO samples from various concentrations.
Figure 8 shows that the morphology of the as-prepared zinc oxide from spent batteries was examined and compared with the morphology of a synthesized ZnO nanoparticles obtained from analytical grade ZnCl$_2$ at varying the concentration of NaOH. Thus, morphology of the particles from Figure 8(a$_1$) was very similar with the morphology of the particles from Figure 8(b$_1$, c$_1$, d$_1$).

These micrographs substantiate that the sample consist of particles dispersed within the range of

Figure 8. (a$_1$, a$_2$) FE-SEM micrograph and energy-dispersive analysis X-ray (EDAX) of ZnO nanoparticles from battery waste at 2.5 M concentration. (b$_1$, b$_2$, c$_1$, c$_2$, d$_1$, d$_2$) FE-SEM micrographs and energy-dispersive analysis X-ray (EDAX) of as-prepared ZnO nanoparticles from ZnCl$_2$ at 2.5 M, 5 M, and 7.5 M concentration.
20 nm to 50 nm at 2.5 M concentration of NaOH (Figure 8(a₁,b₁)) and most of the particles exhibit similar morphology. It can also be shown that the size of the nanoparticles is less than 20 nm at 2.5 M concentration of NaOH which was in good agreement with the crystallite sizes (18.9 nm) calculated from the Debye–Scherrer formula. Figure 8(c₁,d₁) also show that the rise in concentration of NaOH from 5 M to 7.5 M, also increased the particles size.

The elemental compositions of the ZnO nanoparticles were also observed using EDX. The EDX data showed almost the same peaks for all samples. Figure 8 (a₂, b₂, c₂, d₂) represents EDAX patterns which indicate that the as prepared ZnO nanoparticles were composed of only zinc and oxygen. No evidence of other impurities was found and the ZnO nanoparticles were nearly stoichiometric. This observation was in good agreement with XRD results, which confirmed the phase purity of ZnO nanoparticles.

### 3.8. Effect of base concentration on the particle size

Figure 9 shows that effect of NaOH concentration at 2.5 M, 5 M and 7.5 M on particle size in the reaction mixture. It reveals that increasing the concentration from 2.5 M to 7.5 M the particle size increases from 20 nm to 50 nm. In this case it was evident from Figure 7 that the average particle size at 2.5 M, 5 M and 7.5 M concentration were 20.33 nm, 40 nm and 50.33 nm, respectively.

This is because supersaturation is a driving force for crystal nucleation and growth with concentration. At low supersaturation crystals can grow faster which results in a larger crystal size. But at higher supersaturation, crystal nucleation dominates crystal growth, resulting in smaller crystallite size. These obtained results are in fair agreement with the observation (Figure 9) that by decreasing concentration increases the level of supersaturation, thus crystallite size is reduced [14].

### 3.9. Optical characterization

Figure 10 shows that UV–Vis absorption spectrum of ZnO nanopowders using 2.5 M, 5 M and 7.5 M concentration of NaOH synthesized from analytical grade ZnCl₂ and using 2.5 M concentration of NaOH synthesized from battery waste.

Figure 10 exhibits a strong absorption band at about 365 nm for 7.5 M and 360 nm for both 5 M and 2.5 M concentration of NaOH. It was also observed that significantly sharp absorption of ZnO indicates the monodispersed nature of the nanoparticles distribution. The absorption spectrum of ZnO nanoparticles prepared with various NaOH concentrations decreases as the concentration of NaOH decreases in the reaction mixture. Thus from figure it can be observed that UV absorption peak at 365 nm, 361 nm, 360 nm were exhibited for 7.5 M, 5 M and 2.5 M concentration of NaOH, respectively. And UV absorption peak at 360 nm was exhibited for 2.5 M concentration of NaOH obtained from battery waste was very similar to the analytical grade results.

Thus energy band gap for ZnO nanoparticles at 7.5 M, 5 M and 2.5 M concentrations was calculated using Equation (4) as 3.43 eV, 3.41 eV, and 3.39 eV, respectively, and from battery waste was 3.37 eV in case of 2.5 M concentration of NaOH which was very close with analytical grade concentration(2.5 M) and from this the standard band gap energy for ZnO nanoparticle is 3.37 eV. The absorption in the ZnO above 3.3 eV assembled to the band-to-band transition. An increase in the band gap with decrease in particle size is observed which is assigned to the quantum confinement effect. The spacing of the electronic levels and the band gap increases with decreasing particle size.

![Figure 9](image_url)  
**Figure 9.** Effect of concentration on particle size.

![Figure 10](image_url)  
**Figure 10.** UV–Vis absorption spectrum of ZnO nanoparticles at different concentration of NaOH.
This is because the electron hole pairs are much nearer together. Furthermore, the coulombic interaction between them cannot be ignored, thus giving an overall higher kinetic energy \[22\].

### 3.10. Effect of concentration on band gap

Figure 11 shows the effect of concentration variation from 2.5 M to 7.5 M on band gap in the synthesis of ZnO nanoparticles from analytical grade precursors and battery waste as well.

It reveals that increasing the concentration of NaOH in the reaction mixture increases particle size. This results in decreases the band gap energy of ZnO nanoparticles.

This is because lattice reorganization closely related to the electron-hole density which enhances the density. Hence the value of band gap energy lies in the lower wavelength region results in grain size decreases. When concentration was increased grain size of the nano particle was also increased. This is also a result of quantum confinement effect. In case of a metal oxide, the quasi-continuous density of states in the valence and the conduction bands splits into discrete electronic levels, the spacing between these levels and the band gap increasing with decreasing particle size \[22\].

### 4. Conclusions

In this study, ZnO nanoparticles have been synthesized from dry cell zinc–carbon batteries and from analytical grade reagent (ZnCl₂) using wet chemical synthesis method. XRD studies confirmed that zincite structure was obtained for prepared nanoparticles from both procedures. FESEM micrographs shows the particles were of different shapes and having particle size approximately 20 nm–50 nm from both cases. The base concentrations have profound effect on the size and shape of the nanoparticles. Thus the particle size increases with increase in base concentration in the reaction mixture. From UV–Vis absorption spectrum, the prepared ZnO nanoparticles exhibit sharp UV absorption peak corresponding to near band gap exciton emission. The band gap of the samples shows increases with decreases in the concentration of NaOH in the reaction mixture indicating an increase in particle size due to quantum confinement effect. From these results 2.5 M concentration of NaOH is most accurate for the synthesis of desired product because in this case nanosized of the particles are obtained which have good UV-shielding properties. It can be concluded that the synthesis of ZnO nanoparticles from spent battery is of most ecological and economical benefits.

Thus, the above study reveals that recovered nanoparticles with the effect of NaOH concentration, particularly on their size, may find applications in different industrial applications, namely, nano-electronic devices, UV sensors and detectors, active medium for lasers, photovoltaic devices, bio-medical engineering etc.

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### Disclosure statement

No potential conflict of interest was reported by the authors.

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