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Optical Investigation of ZnO Samples Using X-Ray Fluorescence

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ABSTRACT

The problem of the high cost of the materials that used in different devices and the increasing demand for new substituted materials has enhanced the interest in replacing these materials by semiconductors materials to be used. In this study the chemical structure and optical properties of Zinc oxide samples were studied to be used as substituted material using X-Ray Fluorescence. Zinc metal, Zinc sulphide, zinc sulphate and commercial oxide in a powdered form were collected from different areas in Khartoum State, Sudan. These samples were subjected to heat treatment at 1000^{O}C in furnace under ambient oxygen for 3, 6 and 9 hours respectively except the commercial oxide sample which was used as a control. These samples were pressed in a pellets form using press machine under 15 tons pressure. The spectra are recorded at an incident angle θ 45± 5 with respect to the surface of the sample and revealed that the spectrum Zn Kα is 8.638 KeV for all samples and Zn k β is 9.572 KeV lines and signals from the Cu source of the x-ray tube. Peaks of ZnO samples obtained from Zn annealed at 1000ºC for 3 hours and from ZnS annealed at 1000ºC for 9 hours represented sharp and smooth peaks, while the other samples represented sharp and rough peaks. Among the samples annealed at 1000ºC for 3 ours, ZnO obtained from ZnSO4 recorded the highest intensity value (9.9x10^{3}a.u) followed by ZnO (8.1x10^{3}a.u) obtained from Zn annealed at 1000ºC for 9 hours and then ZnO (7.8x10^{3}a.u) sample obtained from Zn annealed at 1000ºC for 6 hours.

Keywords: *chemical structure, High temperature, Optical investigation, XRF, ZnO.*

INTRODUCTION

Zinc-base oxide is a substituted material of transparent conducting oxide that can be applied in different technologies (Patel, 2014). The wide band gap (3.37eV at 3.00K) of zinc oxide (ZnO) and high excition energy (60 meV) make high quantum efficiency at room temperature (Meyer 2004), due to these properties ZnO can be applied in different devices as laser diodes (LDs) (Ohta , 2001), light emitting diodes (LEDs) (Kang , 2007; Jeong 2007), field emission (Kim , 2004) and optical detectors in ultra violet region (Liu , 2000). Usually ZnO is a good choice to be used and preferable materials because of its low cost and environmental friendly nature (Singh , 2011). It is the most attractive material due to its high theoretical Curie temperature above 300 K (Dietl , 2000) and can be applied in spintronics area as diluted magnetic semiconductors (DMSs). Recently Zinc oxide has been intensively investigated using different methods such as x-ray diffraction (XRD), particle size analyzer (PSA), scanning electron microscopy (SEM) for morphological properties, photoluminescence (PL) spectra, Hall-ect measurements, UV-VIS spectroscopy and x-ray fluorescence (XRF). XRF is a method used for chemical analysis of different materials, which is typically applied for bulk analysis. It is a widely method used because of the relative ease and low cost of sample preparation, stability and ease of use (Kari Wirth, 2013). XRF can be used to analyze different types of materials including metal, oil, polymer, plastic, food industries, mining, mineralogy, geology, environmental analysis of water waste materials and even for pharmacy researches (Brouwer, 2006), also it can be used to investigate solids, liquids, powder, filtered and other forms (Luice , 2012). This research deals with the preparation of ZnO from different samples by heat treatment at 1000ºC for 3, 6 and 9 hours respectively and investigated its chemical composition and measured the intensity using XRF method.

Materials and Methods

Samples collection

Samples include Zinc metal (ZnO), Zinc sulphide (ZnS), zinc sulphate ($ZnSO_4$) and commercial oxide in a powdered form were collected from different areas in Khartoum State, Sudan.

Samples preparation

Three grams of each sample except commercial zinc oxide (as a control sample) were put in a crucible and subjected to heat treatment at 1000ºC in a furnace (England, Maximum Temperature 1200ºC) for 3, 6 and 9 hours respectively.

X-Ray Fluorescence (XRF) Investigation

Samples for element structure investigation by XRF were firstly pressed into a pellet form using a pressing machine (CABRRA, Series 35 pulse) under 15 tons pressure using the self-binder properties of the samples (Bruker, 2006). A pressure of about 15 tons is usually applied to obtain good pellets (Abdalla, 1980). All samples were fabricated at Department of Physics at University of Khartoum.

The purpose of XRF spectrometer was to detect photons of x-rays, sort them out of energy, display, memorize and process the spectrum. It consist of Si (Li) detector, pre amplifier (P.A), canberra amplifier (Apm), multi-channel analyzer (M.C.A) and PC. Each sample was put on the detector for processing. After ionization (creation of electron-hole iron pairs) an electric pulse (signal) for each sample will be produced proportional to the energy absorbed. Pulse for each sample was collected, magnified in P.A and then transmitted to the Amp to magnify again and carry out some other electric functions ensuring the shape and the size of each pulse to be suitable for processing by multi-channel analyzer. The M.C.A builds up a sort of graph with energy on x-axis and the number of counts or pulses on the y-axis and then displays the spectrum as digital data on the screen. At the end of data collection, the spectrum transferred to the P.C. to run a program of data analysis as well as quantitative analysis. The exciting x-ray source was Cd-109 of 25 mm Si activating and annular shape.

RESULTS AND DISCUSSION

Results revealed that the spectra are corrected at an incident angle θ 45± 5 with respect to the surface of the sample. Figures 1,2, 3, 4, 5, 6, 7, 8, 9, and 10 showed the typical XRF signals of ZnO powder samples obtained from the samples (Zn, ZnS, ZnSO4) annealed at 1000ºC for 3, 6 and 9 hours. The ZnO spectrum consists of a well-design background on which sharp characteristics and spectral artifacts are superimposed.

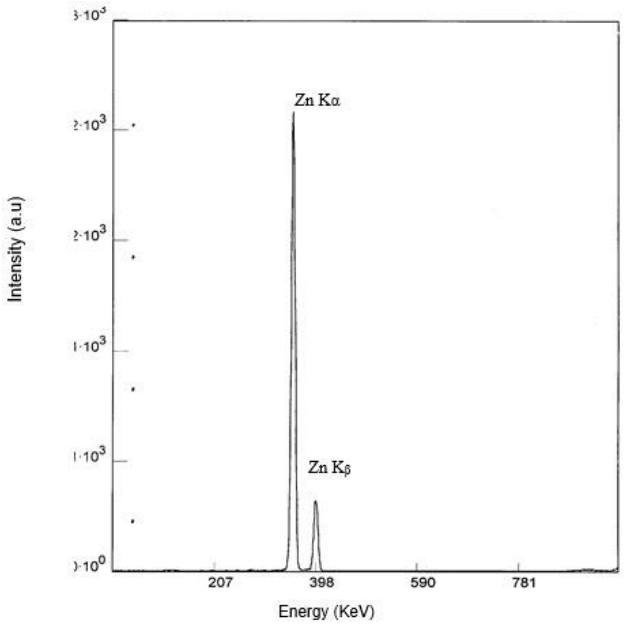

Figure 1. XRF investigation of ZnO obtained from Zn annealed at 1000ºC for 3 hours

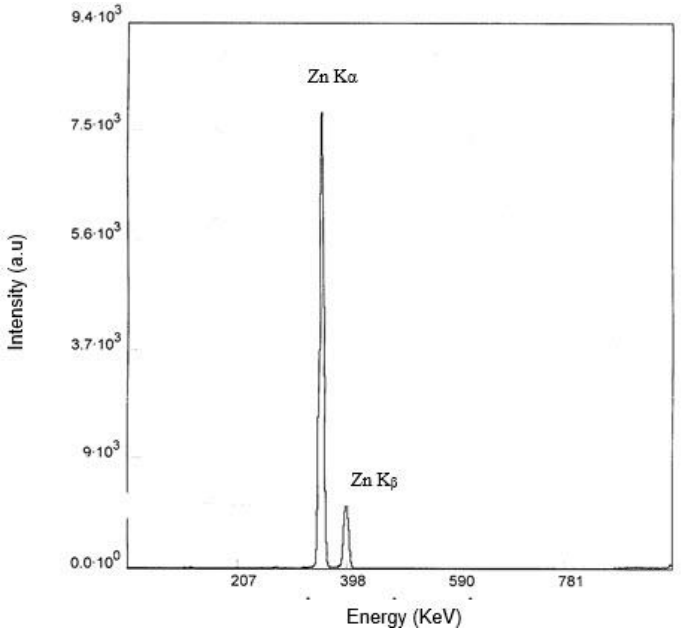

Figure 2 . XRF investigation of ZnO obtained from Zn annealed at 1000ºC for 6 hours

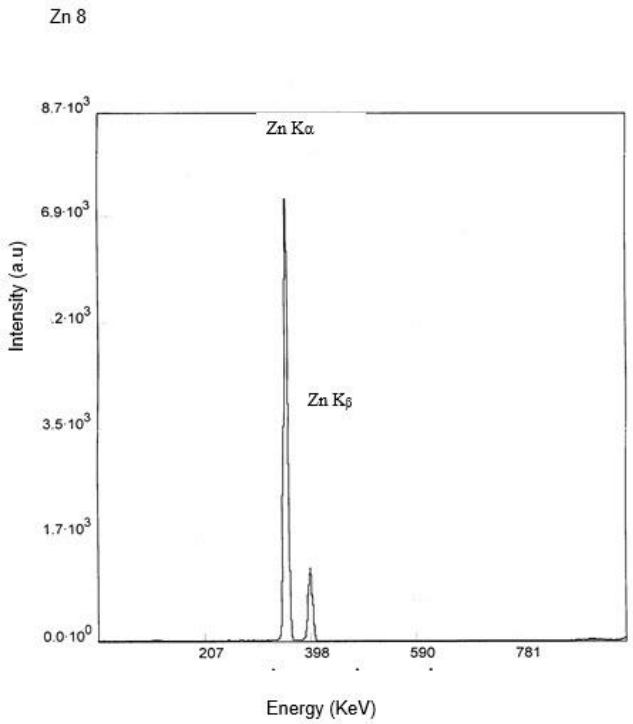

Figure 3. XRF investigation of ZnO obtained from Zn annealed at 1000ºC for 9 hours.

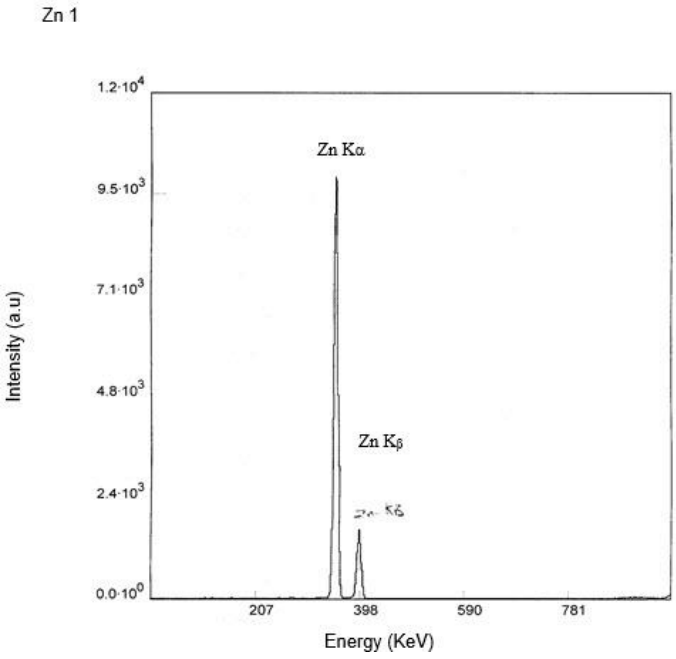

Figure 4. XRF investigation of ZnO obtained from ZnS annealed at 1000ºC for 3 hours

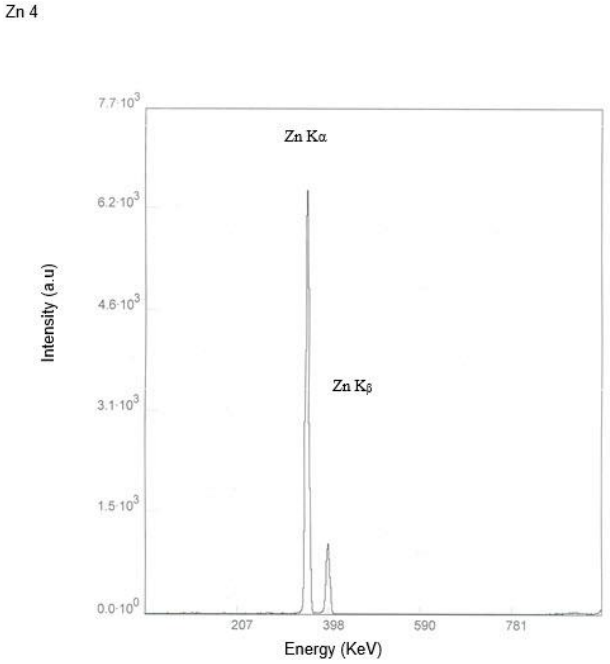

Figure 5. XRF investigation of ZnO obtained from ZnS annealed at 1000ºC for 6 hours

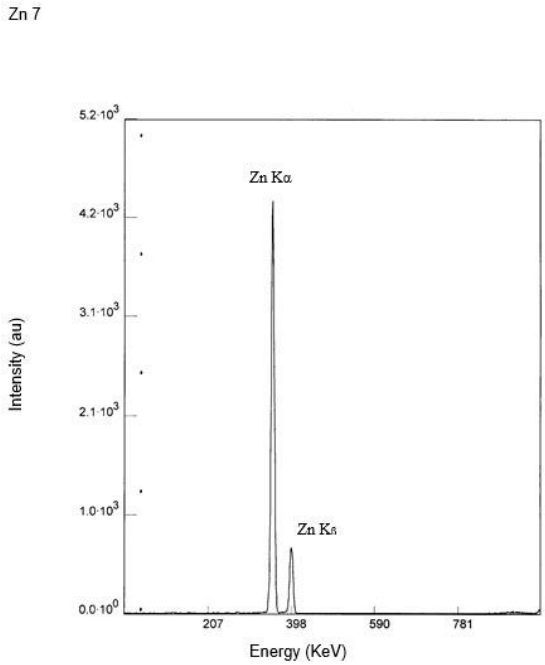

Figure 6. XRF investigation of ZnO obtained from ZnS annealed at 1000ºC for 9 hours

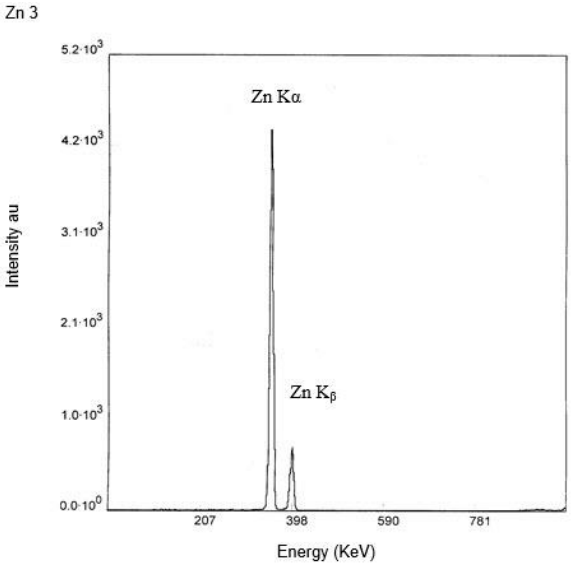

Figure 7. XRF investigation of ZnO obtained from ZnSO4 annealed at 1000ºC for 3 hours

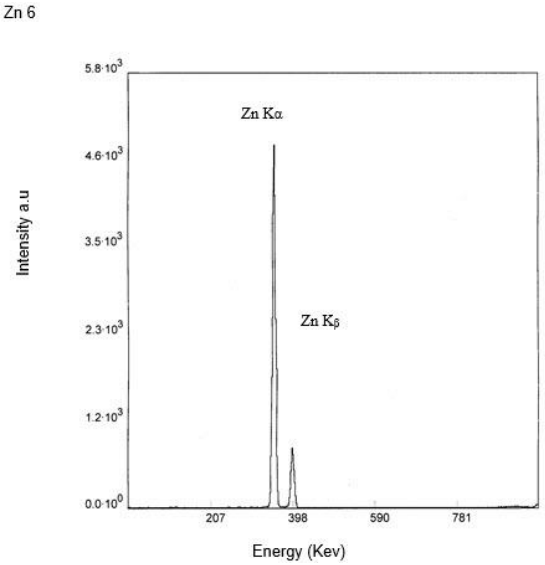

Figure 8. XRF investigation of ZnO obtained from ZnSO4 annealed at 1000ºC for 6 hour

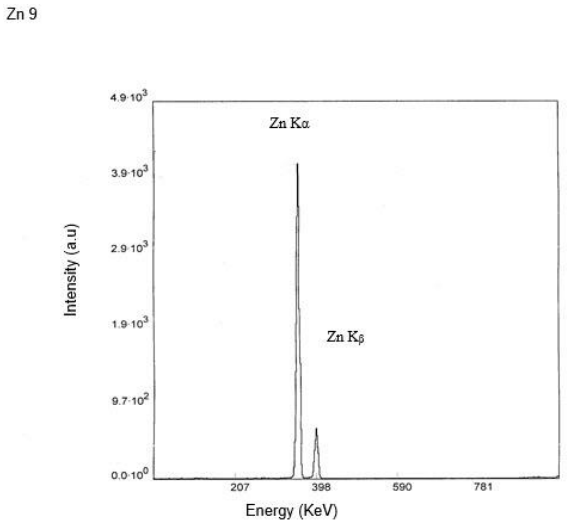

Figure 9. XRF investigation of ZnO sample obtained from ZnSO4 annealed at 1000ºC for 9 hours

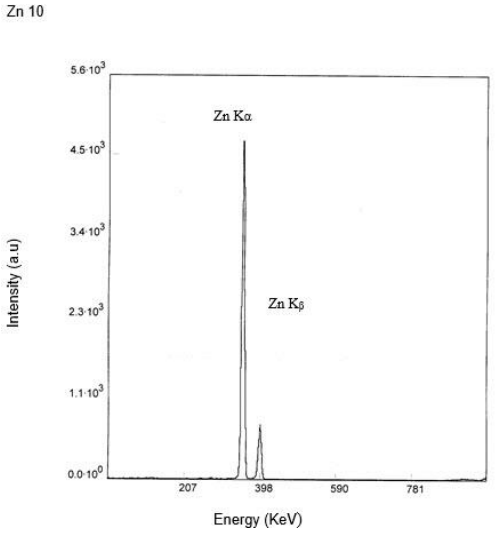

Figure 10. XRF investigation of commercial ZnO

The spectrum revealed that Zn Kα is 8.638 KeV for all samples. Dakhel and El-Hilo (2010) found that the spectrum energies dispersion x-ray fluorescence (EDXRF) for Gd-doped ZnO powder showed Zn Kα (8.638 KeV), and Zn k $_{\beta}$ (9.572 KeV), Gd Lα (6.05 keV), Gd L $_{\beta 1}$(6.71keV), Gd L $_{\beta 2}$ (7.10 keV) lines, and signals from the Cu source of the x-ray tube. No other signals (including an aluminum signal) were detected. Peaks of ZnO samples obtained from Zn annealed at 1000ºC for 3 hours (Fig. 1) and from ZnS annealed at 1000ºC for 9 hours (Fig. 6) represented sharp and smooth peaks, while the other samples represented sharp and rough peaks. This may be attributed to proper conditions applied for these two samples and improper annealing temperature and time applied for the other samples. The presence of impurities plays an important role to affect the properties of produced samples (Lannoo and Bourgoin , 1981, 1999). Higher annealing temperature for along time is greatly improves the properties of ZnO samples obtained in this project. The longer annealing time provides more activation energy to atoms to grow larger grains, which is consistent with results of XRD. Also the grain boundaries were fewer and the grains grown much bigger with further increase of annealing time (Wang , 2002). Similar results obtained by (Lin , 2005) who described that high temperature can stimulate the migration of grain boundaries and cause the coalescence of more grains during the annealing processes. Another study conducted by (Fang , 2005) who indicated that at high temperature, more energy should be available for the atoms to acquire so that they may diffuse and occupy the correct site in the crystal lattice and grains with lower surface

energy will grow larger at high temperature. The major grain growth also grow yield an increase in the surface roughness (Liu , 2006; Virt , 2010).

Generally samples annealed at 1000^{o}C for 9 hours recorded the highest intensity values comparing with the other samples. These intensity values were 8.1x103a.u for ZnO obtained from Zn, 4.8X10^{3}a.u for ZnO obtained from ZnSO4 and 4.1x10^{3}a.u for the sample obtained from ZnS. With respect to the samples annealed at 1000^{o}C for 6 hours, ZnO sample obtained from Zn showed a higher intensity value (7.8x10^{3}a.u) than the samples obtained from ZnSO4 (6.8X10^{3}au), followed by ZnS and commercial ZnO (4.8X10^{3}a.u). All samples annealed at 1000^{o}C for 3 hours showed the lowest intensity values (4.4X10^{3}-4.8X10^{3}a.u) except ZnO sample obtained from ZnSO4 recorded the highest intensity value (9.9x10^{3}a.u) among them and all samples. However, results similar to the present were obtained by Chen , (2009) who found that the intensity of ZnO nanopillar (002 diffraction peak) increased with the increase of annealing temperature claming that the crystallization of ZnO thin film was improved by an adequate supply of energy. Chauhan , (2011) found that the intensity value of undoped ZnO was higher (9.0X10^{2} a.u) comparing with doped ZnO $_{0.5}$ Ni $_{0.5}$ (8.5x10^{2} a.u) at the doping concentration of 5% and the sintered temperature at 500^{O}C. Sridevi and Rajendraan (2009) found that the strong emission peak of ZnO nanomaterials synthesized in aqueous solution was centered at 397nm and the intensity value was 9.5X10^{6} a.u. As seen from the results ZnO intensity was increased and improved at high temperature with the increase of time. The quality of ZnO was improved by increasing of annealing temperature (Chen , 2009). High annealing temperature has been found to improve ZnO film characteristics of piezoelectric application (Sharma, 2002).

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