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INVESTIGATION OF THE ELEMENTAL COMPOSITION, MORPHOLOGICAL AND OPTICAL PROPERTIES OF LEAD OXIDE

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ABSTRACT

Lead oxide nanoparticle was investigated in terms of the elemental composition, morphological and optical properties using X-ray fluorescent (XRF), transmission electron microscope (TEM) and UV visible spectroscopy. It was also shown that the percentage of lead oxide in the sample as revealed by the XRF measurement is 73.98 wt %. There are other compounds present in the lead oxide sample with small percentages. The impurity compound with the highest concentration was found to be silicon oxide. The TEM analysis revealed that the particles are spherical in shape and are evenly distributed with average particle size was of about 18. 4 nm. The absorbance was found to decrease with increase in wavelength while the transmittance increases with an increase in wavelength. The absorption coefficient also decreases with increases in wavelength. The optical band gap obtained by extrapolating the graph of the square of the product of absorption coefficient and photon energy verse photon energy was observed to be 3.1 eV. This material could used as an absorber layer in the fabrication of solar cells due to it good morphological and high optical properties.

KEYWORDS: Elemental, morphological, semiconductor, absorbance, transmittance extrapolation and band gap.

1.0 INTRODUCTION

Semiconducting nano-materials have gained tremendous attention in recent time due to their unique and excellent properties that can be used for different applications [1,9,16]. Lead (II) oxide, which is also called lead monoxide, is an inorganic material that has the molecular formula PbO. This material occurs in two forms: the litharge having a tetragonal crystal structure, and the massicot having an orthorhombic crystal structure. Modern applications for lead oxide are mostly in lead-based industrial glass and industrial ceramics, including computer components [7].

Lead compounds are used in different industries world- wide due to their chemical and physical characteristics [14]**.** Its reactions with acids and bases as well as with air, which are well-known as oxidation is one of the most important characteristics. Compounds like "lead (II) oxide, lead (IV) oxide, sulfate, lead carbonate, lead nitrate as well as alkaline lead acetate" have been produced from this kind of reactions. Some are the end product of a desired process and most of them are undesired byproducts which are known as disturb compounds.

Due to their various optical and electronic properties, lead oxide have been used for a wide variety of microelectronic and optoelectronic applications, such as electroluminescent device, magnetic memory, dielectric layer and good material for warming applications in home, temperature regions, and in agriculture [10]. Lead oxide (PbO) is one of the metal oxides that has the most significant use in depot batteries, glass business, and dyes [6].

PbO is toxic and may be dangerous when swallowed or inhaled. It is irritating to the skin, eyes, and the respiratory tract. It damaged the gum tissue, central nervous system, kidneys, blood, and the reproductive system. It can also accumulate in plants and in mammals [10]

Lead oxide is well known as an important industrial material, which has been widely utilized in batteries, gas sensors, pigments and paints, ceramics, glass industry and as a catalyst in synthetic organic chemistry.

The morphologies of PbO semiconductor play significant roles in its properties. Lead oxide may appear in a various forms. The tetragonal crystalline structure is red in color with α-PbO form which is known as litharge with a band gap of ranging from 1.9 – 2.2 eV, which has proven to be stable at room temperature, while the orthorhombic crystalline structure is yellowish in color with the formula β-PbO which is known as massicot with a band gap ranging from 2.7 eV, which seems to be stable at high temperatures, above 488°C [4].

PbO nanoparticles can be produced successfully in rapid and easy chemical route techniques. XRD, SEM, characterizations techniques confirm the results for their potential applications in deferent fields [14]. Litharge has been developed which results in the formation of the experimental scheme produces lead oxide nanoparticles with no detectable amounts of yellow massicot form [2]. Since PbO does not contain impurities, one can reuse in the industry. Lead sulfate can be converted to PbO in industry which is an economical procedure [15]. The Morphological characteristics of β-irradiated lead oxide nano-sized particles was investigated and it was found out that the PbO sample's roughness increases after a affluence increase of the irradiation caused by time duration [3]. Paucity of data was observed from the AFM studies in lead oxides. As per the AFM studies, it is possible to control the size and the shape parameters of the nanoparticle samples by using beta ray irradiation. In particular, original lead nanostructures have been recently synthesized by plasma in liquid nitrogen. These nanostructures, which can be used for various applications. This method initially produces hexagonal nanosheets. However, the synthesis of lead nanosheets is limited by a specific erosion process that produces lead sticks beyond about 2000 discharges rather than hexagonal nanosheets [11].

An investigation on the Crystal structure, morphology and formation mechanism of a novel polymorph of lead dioxide, γ-PbO_2 was carried out and it was observed that the synthesis of the novel γ-PbO_2 nanosheets originates from spark discharge, in the liquid nitrogen, followed by oxidation in air [12].

2.0 METHODS

The materials used in this research were used as received without further purification. The elemental composition was studied using X-ray florescent in which the wet percentages of various constituent elements were observed. The morphological characteristic was investigated using transmission electron microscope (TEM). The optical properties of PbO semiconductor nanoparticles was studied using double beam UV-Vis spectrophotometer of the semiconductor wavelength range of 190 – 900nm with 1000 mm quartz cell. This resolution of the UV-Vis spectrophotometer was 1 nm. The UV-Vis spectra of resulting solution were recorded. The spectrum is plotted for absorbance on the Y-axis against wavelength on the X-axis. Other optical property such as transmittance, absorption coefficient was obtained by calculation and the optical band gap was obtained by extrapolation.

3.0 RESULTS AND DISCUSSIONS

3.1 X-ray fluorescent analysis

The result of X-ray fluorescent characterization is presented in table 1, the weight percentage of lead is 73.977 wt. %. While that of zinc oxide present in the sample is 0.126 wt. %. There are other elements found in lead oxide with low percentage. The Variation of weight percentage with different compound is shown in Figure 1.

The impurity element with the highest percentage is silicon with 9.649 wt. % and the one with the least percentage is tungsten trioxide (wo_3) with 0.003 wt. %.

The elements that constitute most of the impurities in lead oxide is silicon, aluminum and chlorine. The good crystallinity of lead oxide as seen in the transmission electron microscope images is a prove to their excellent opto-electronic applications.

The high presence of silicon in lead oxide makes it a suitable material for solar cell absorption layer. although, its toxic nature limits its applications, but it usefulness cannot be overlook as it has been established that perovskite solar cells fabricated by lead show high power conversion efficiency compare to those made from other materials. The toxic effect of lead could be reduced by the addition non-toxic element such ion.

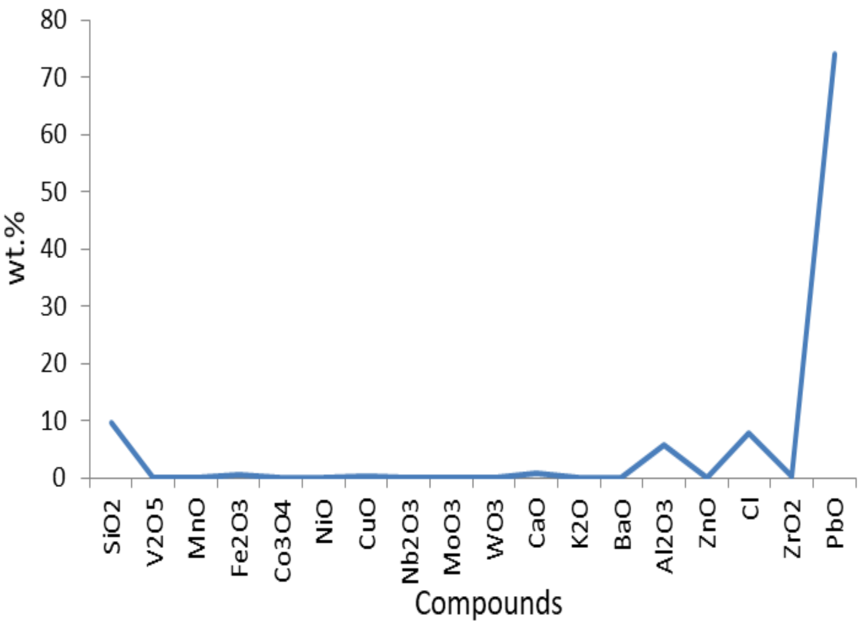


Figure 1: Weight Percentage against Compound.

Table 1: Weight Percentage of Some Impurity Compounds Found in Lead Oxide.

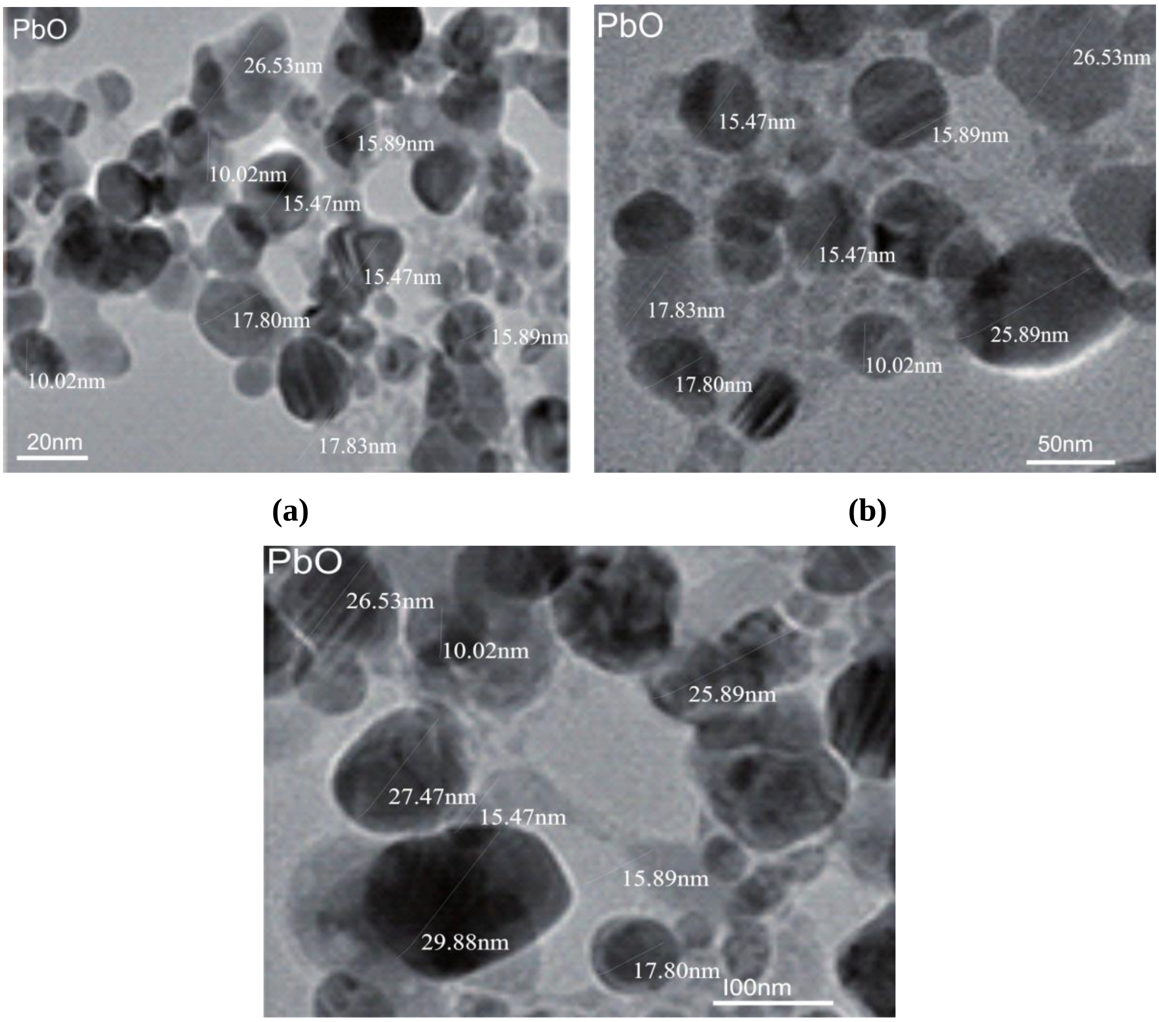
Compounds	Weight Percentage
SiO2	9.65 wt.%
V2O5	0.00 wt.%
MnO	0.13 wt.%
Fe2O3	0.57 wt.%
Co3O4	0.02 wt.%
NiO	0.12 wt.%
CuO	0.38 wt.%
Nb2O3	0.02 wt.%
MoO3	0.01 wt.%
WO3	0.01 wt.%
CaO	0.81 wt.%
K2O	0.14 wt.%
BaO	0.07 wt.%
Al2O3	5.76 wt.%
ZnO	0.13 wt.%
Cl	7.77 wt.%
ZrO2	0.44 wt.%
PbO	73.98 wt.%

3.2 Morphological Properties

Transmission electron microscope (TEM) Analysis

The morphological property of lead oxide was studied using transmission electron microscope. The TEM images revealed the shapes and distributions of the lead oxide Nanoparticles. Lead oxide has a spherical shape and the molecules are evenly distribution. In Figure 2a, when the wavelength is 20 nm, the particle sizes ranges between 10.02 nm to 26.53 nm, with the biggest particle on center top and the least particle on bottom left. As the wavelength was increased to 50 nm, the particle sizes ranges between 10.02 nm to 26.53 nm, with the biggest particle on the right top and the least particle on the center bottom (Fig. 2b). When the wavelength was increased to 100 nm, the particle sizes ranges between 10.02 nm to 29.88 nm (Figure. 2c).

The average particle size was found to be 18.4 nm.



(c)

Figure 2: (a) Transmission Electron Microscope Image of Lead Oxide at 20 nm, (b) at 50 nm, (c) at 100 nm.

3.3 Optical Analysis (UV –Vis Spectroscopy)

The optical properties of lead oxide semiconductor nanoparticles were studied using double beam UV-Vis spectrophotometer of the semiconductor wavelength range of 190 – 900nm with 1000 mm quartz cell. This resolution of the UV-Vis spectrophotometer was 1 nm. The UV-Vis spectra of resulting solution were recorded. The spectrum is plotted for absorbance on the Y-axis against wavelength on the X-axis. Other optical property such as transmittance and absorption coefficient were obtained by calculations and the optical band gap was obtained by extrapolation.

3.3.1 Absorbance and transmittance of lead oxide

The transmittance was obtained from absorbance using equation 1 [13].

$$T = 10^{-A} \qquad (1)$$

For lead oxide, the absorbance was observed to be high at 380 nm (Figure 3a). The value of the absorbance continues to decrease gradually as the wavelength increases up to 900 nm.

The transmittance was observed to have the lowest value at the wavelength of 380 nm (Figure 3b). Its value increases sharply to about 0.46. It was observed that the transmittance of lead oxide increases to a high value as the wavelength increases from 380 nm up to 900 nm.

The absorption coefficient is obtained from the transmittance using the relation

$$\alpha = \frac{1}{d} \log \frac{1}{T} \qquad (2)$$

Where, α is absorption coefficient, d particle thickness and T is the transmittance.
The band gap was obtained by extrapolation from the plot of the square of absorption coefficient multiplied by photon energy against photon energy (Figure 3c). The band gap obtained from tauc plot was observed to be 3.1 eV. The band gap could also be calculated using Tauc's relation.[5,17]

$$(\alpha\, hv)^n = A\,(hv - E_g) \qquad (3)$$

Where A is band edge parameter and value of n determines the nature of optical transition (n = ½ indicates direct transition and n = 2 indicates indirect transition).

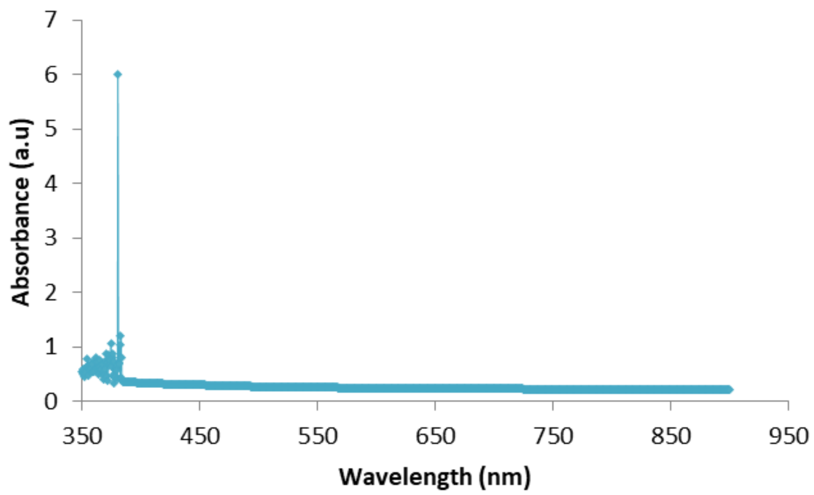


Figure 3a: A plot of absorbance against wavelength for lead oxide.

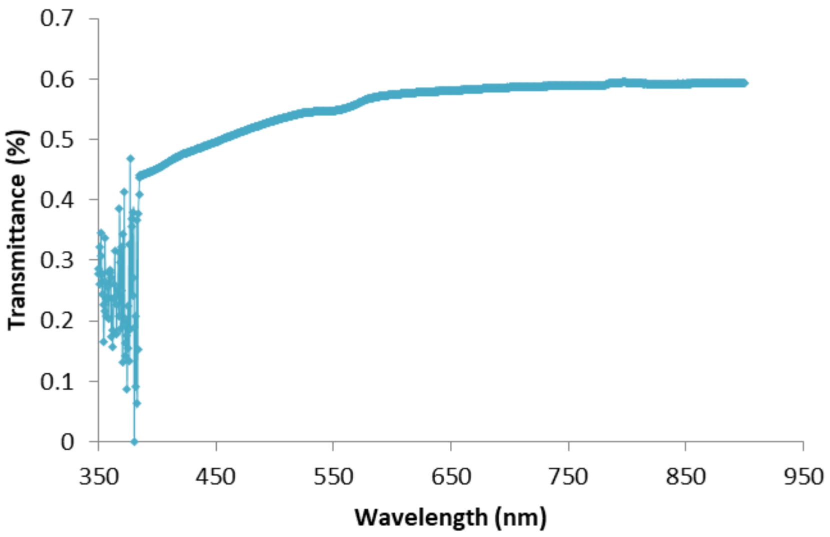


Figure 3b: A plot of Transmittance against wavelength for lead oxide.

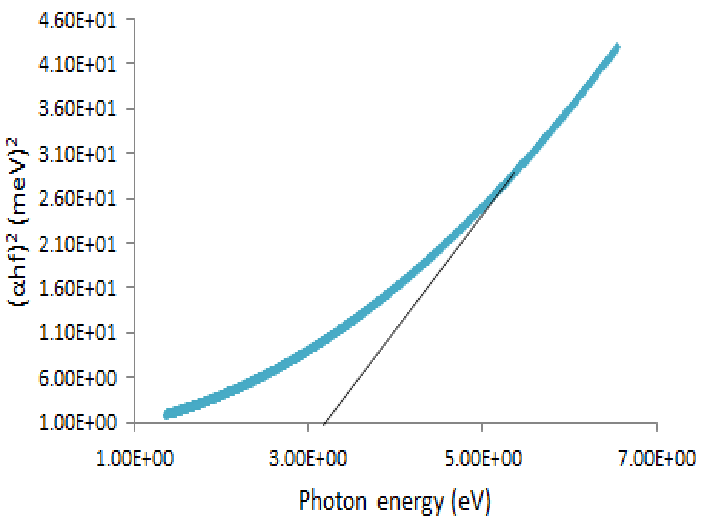


Figure 3c: A plot of $(\alpha hf)^2$ $(meV)^2$against Photon Energy (eV) for lead oxide.

4 CONCLUSIONS

The elemental composition, morphological, and optical investigation of lead oxide was carried out using x-ray fluorescent, transmission electron microscope and UV visible spectrophotometer and the following conclusions were made.

The results of x-ray fluorescent revealed that there is 73.98 wt. % of lead in lead oxide and 0.13 wt. % of zinc in lead oxide. There are other impurity elements found in lead oxide other than zinc oxide. Silicon was found to be the impurity element with the highest concentration in lead oxide.

The transmission electron microscope (TEM) results revealed that lead oxide has spherical shape; the wavelength has an influence on the particle sizes of lead oxide. The average particle size was found to be 18.4 nm.

The absorbance was found to be decreasing with increase in wavelength while the transmittance was found to increase with increase in wavelength.

The band gap obtained from tauc plot was observed to be 3.1 eV. Which an acceptable band gap for a noncrystalline lead oxide semiconductor.

Due to the excellent morphological and high optical properties of this material, it could be used as a potential absorption layer for solar cells fabrication.

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