

# Biosynthesis of lead oxide nanoparticles using *Ocimum lamiifolium* leaves extract

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## Research Article

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# Abstract

In this study we report the biosynthesis of lead oxide nanoparticles from *Ocimum Lamiifolium* aqueous extract and lead citrate precursor. Characterization techniques such as powder X-ray diffraction, scanning electron microscopy, Fourier transform infrared spectroscopy and UV-Visible spectroscopy were used to study the desired phase formation, crystal structure and morphology of the synthesized nanostructure. The powder X-ray diffraction analysis revealed that lead oxide nanoparticles with an average crystallite size of 39 nm have been synthesized. Scanning electron microscopy analysis exhibited the surface morphology. Fourier transform infrared spectroscopy study also confirmed also the successful synthesis of lead oxide nanoparticles. The UV-Visible spectrum absorbance peak is in the range of 200–250 nm and this corresponds to the characteristic of lead oxide nanoparticles.

## 1. Introduction

Over the past few years, synthesis of metal oxide nanoparticles is becoming the focus of material scientists because of their unique physicochemical characteristics and potential application in making nanoscale electronic and optoelectronic devices [1]. So far, various types of metal oxide nanostructures such as VO<sub>2</sub>, ZnO, WO<sub>3</sub>, NiO have been synthesized successfully.

Lead oxide exists, basically, in the form of PbO, Pb<sub>2</sub>O<sub>3</sub>, Pb<sub>3</sub>O<sub>4</sub>, and PbO<sub>2</sub> [2]. Among these, the lead (II) oxides (PbO) are attractive due to their wide range of application for lead storage batteries, ceramics and gas sensors [3–7]. Now a days PbO has received due attention because of its cost-effectiveness, air and water compatibility, good reactivity, and recyclability [8]. PbO is semiconductor and has two polymorphic forms: the tetragonal  $\alpha$ -PbO and the orthorhombic  $\beta$ -PbO [5]. The  $\alpha$ -PbO also known as litharge has a bandgap of 1.9-2.2eV and is stable at room temperature; whereas, the  $\beta$ -PbO form has a bandgap of 2.7 eV and is stable above a temperatures of about 490°C [3, 9].

A plethora of methods such as spray pyrolysis [4], ball milling [10], calcination [11], sol-gel [12, 13], hydrothermal [14–17], microwave irradiation [18], sonochemical [2], and biosynthesis [19–24] techniques have been employed to synthesize PbO nanostructures. Among these, the biosynthesis route is an environmentally sound, cost-effective, biocompatible, safe and allows large scale production of nanoparticles with free of additional impurities [19–21]. For instance, Tailor et. al attempted to synthesize PbO nanoparticles using *Eucalyptus globulus* Labill. (leaves) extract [23]. Noukelag et. al synthesized PbO nanoparticles using *Rosmarinus officinalis* aqueous leaves extract as an effective chelating agent [20]. Khalil et. al also successfully synthesized PbO nanoparticles biogenically synthesized via complete green process [21].

*Ocimum Lamiifolium* (family Lamiaceae) known as 'Damakesse' in Amharic is a versatile medicinal plant widely grown in most parts of Ethiopia is used to treat fever, pain, for the treatment of wound [25–27]. Experiments on the essential oils of various *Ocimum* species have indicated that the oil possess antimicrobial, antibacterial, antifungal properties [25]. In this work, the crude extract of *Ocimum*

*Lamiifolium* leaf is used as a reducing as well as stabilizing agent to synthesis lead (II) oxide nanoparticles.

## 2. Materials and methods

### 2.1 Materials

Lead citrate [ $\text{Pb}(\text{C}_6\text{H}_6\text{O}_7) \cdot \text{H}_2\text{O}$ ], absolute ethanol (99.9%), deionized water and detergents were supplied by Sigma Aldrich. Disease free and fresh leaves of *Ocimum Lamiifolium* were collected from Debre Berhan Town of Amhara Region, Ethiopia.

### 2.2 Methods

Following the taxonomical identification, the collected leaves of *Ocimum Lamiifolium* were thoroughly washed with tap water followed by distilled water for removing unwanted impurities such as dust and scum and kept in a clean place for a week to dry under room temperature.

Weighed 30 gm of crushed *Ocimum Lamiifolium* leaves were put in to 300 mL of deionized water ( $\text{DI-H}_2\text{O}$ ) and heated at a temperature of  $60^\circ\text{C}$  for 30 min. The mixture was cooled to room temperature and filtered using Whatmman No 1 filter paper. The filtered *Ocimum Lamiifolium* solution was stored in refrigerator at  $4^\circ\text{C}$  for next experiments. On the other hand, 0.5M lead citrate solution was prepared by dissolving 47 gm of lead citrate ( $\text{Pb}(\text{C}_6\text{H}_6\text{O}_7) \cdot \text{H}_2\text{O}$ ) in 250mL deionized water.

To synthesize PbO nanoparticles, 50mL of *Ocimum Lamiifolium* were slowly poured into 25mL of  $\text{Pb}(\text{C}_6\text{H}_6\text{O}_7) \cdot \text{H}_2\text{O}$ . The mixture was stirred continuously for 1 hour with magnetic stirrer until the yellowish solution is reached. Finally, the precipitate were oven dried at  $60^\circ\text{C}$  overnight and the dried yellow powder was annealed using (BK-5-12GJ) furnace at  $500^\circ\text{C}$  for 1 hour [21] and the resulting powder was kept in a sample holder for further characterization.

## 3. Results and discussion

### 3.1 X-Ray Diffraction (XRD)

The crystalline structure of the biosynthesized PbO nanoparticles were investigated using X-ray Diffractometer (Bruker D8 Advance Diffractometer). XRD spectrum was recorded from  $10^\circ - 80^\circ$  with  $2\theta$  angles using  $\text{CuK}\alpha$  radiation operated at 40kV and 30mA. The average crystallite size was estimated from the intensity peak of the diffracted beam and the corresponding Bragg angle ( $\theta$ ) using Scherer's formula:

$$D = \frac{0.9\lambda}{\beta \cos \theta},$$

where, 'D' is the averaged crystallite size, ' $\lambda$ ' is wavelength of incident beam (1.5406Å), ' $\beta$ ' is full width at half-maximum (FWHM) in radians and ' $\theta$ ' is scattering angle in degree.

Figure 1 shows the XRD pattern of the synthesized PbO nanoparticles. The diffraction peaks are observed at  $2\theta$  of 17.65, 28.67, 31.85, 48.61, 54.85, 59.95, and 78.51 correspond to the Miller indices (100), (101), (110), (002), (112), (211), (202), and (103) of wurtzite structure of lead oxide nanoparticles confirmed from JCPDS card no. 36-1451 with lattice parameters ( $a = 3.2493 \text{ \AA}$  and  $c = 5.2056 \text{ \AA}$ ).

## 3.2 Scanning Electron Microscopy (SEM) Analysis

The morphology of PbO nanoparticles were studied using SEM (JSM-6480 LV) microscopy. Figure 2, revealed that nearly spherical nanoparticles has been synthesized.

## 3.3 Fourier Transform Infrared (FT-IR) Spectroscopy

PerkinElmer, Spectrum 65 FTIR spectroscopy (in the region of  $4000 - 400 \text{ cm}^{-1}$ ) were employed for the identification of functional groups. As shown in Fig. 3, the broad peak around  $3370.7 \text{ cm}^{-1}$  corresponds to the stretching vibration of O-H group [28]. The intense peaks around  $1395.2 \text{ cm}^{-1}$  are due to bending vibration of OH [21]. The sharp peak observed at  $681.6$  is due to the bending vibration of Pb-O-Pb and the peak observed around  $437.5 \text{ cm}^{-1}$  indicate the Pb-O stretching [28].

## 3.4 UV-Visible Spectroscopy Analysis

The optical characterization of PbO nanoparticles was carried out using Perkin-Elmer lambda 950 UV/VIS/NIR/ spectrometer in the wave length region of 200 nm – 900 nm. The UV-Vis spectra of the synthesized PbO nanoparticles are shown in Fig. 4. The absorption peak of the prepared PbO sample was observed around 200 nm and its sharp absorption feature indicates that PbO nanoparticles are synthesized [12, 19].

## 4. Conclusion

The use of natural resources for production of nanoparticles is eco-friendly, inexpensive and free of chemical contaminants compared to the physicochemical methods. In the present study, both the  $\alpha$  – PbO and  $\beta$ -PbO phases were successfully synthesized using leaf extract of *Ocimum Lamiifolium*. The XRD study confirms the formation of pentagonal wurtzite structure PbO nanoparticles with an average crystallite size of 39.86 nm. The SEM image analyses revealed that aggregate structures of lead oxide nanoparticles were synthesized. The characteristic absorption peaks of UV-Spectroscopy result for the designed two samples observed around 200 nm also supports the formation of PbO nanoparticles. *Ocimum Lamiifolium* is, therefore, a good reducing and stabilizing agent for the synthesis of PbO nanoparticles.

## Declarations

**Author contributions:** Asratemedhin Bekele Habtemariam prepared the samples, analyzed the results and wrote the main manuscript text.

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**Conflict of interest:** no conflict of interest.

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## Figures

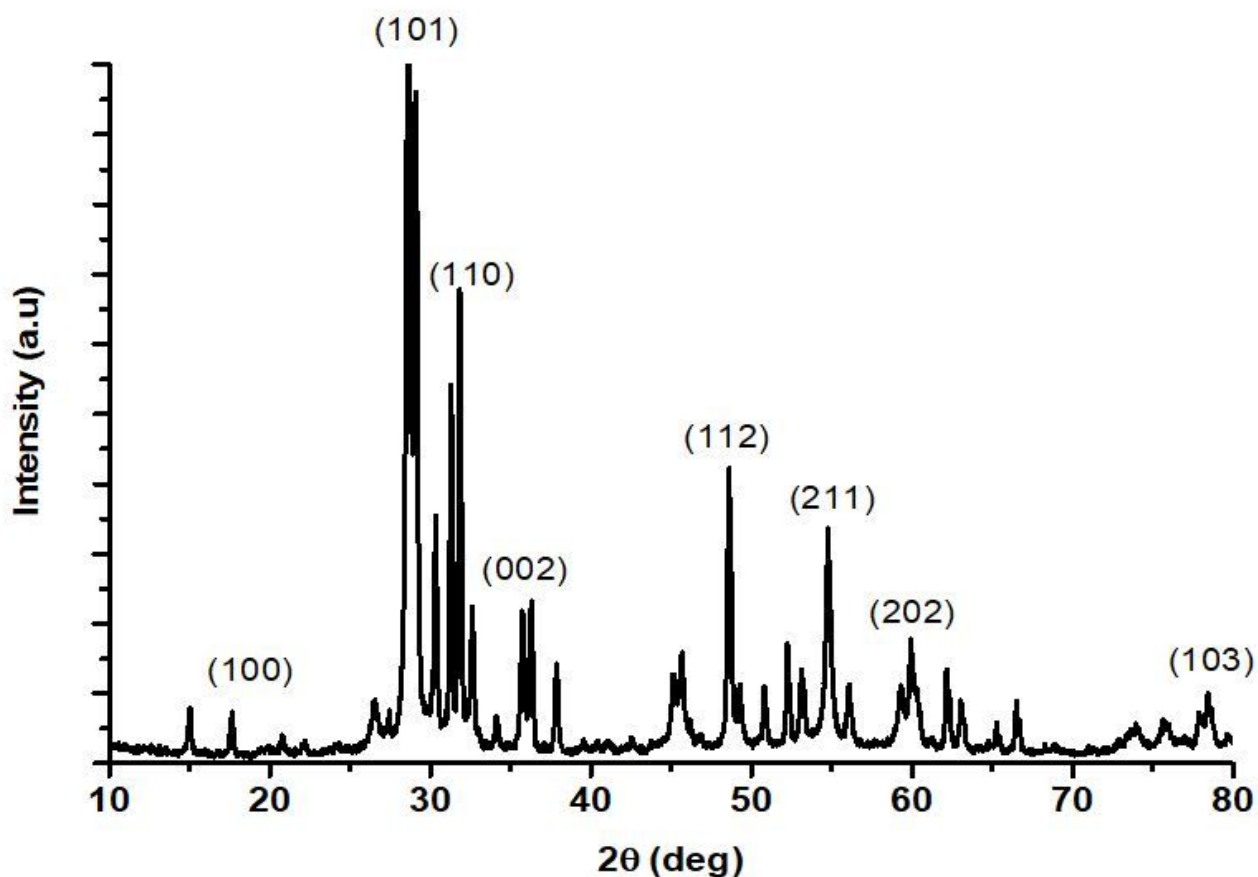


Figure 1

XRD Patterns for  $\alpha$ -PbO nanoparticles

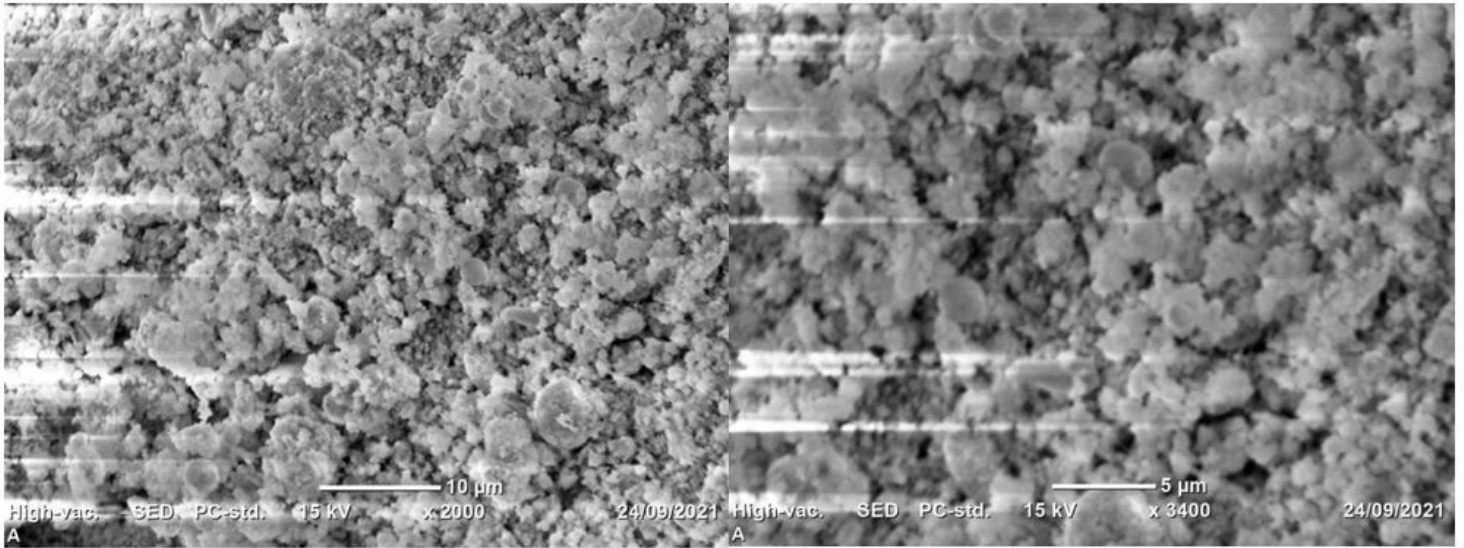


Figure 2

SEM image of PbO nanoparticles

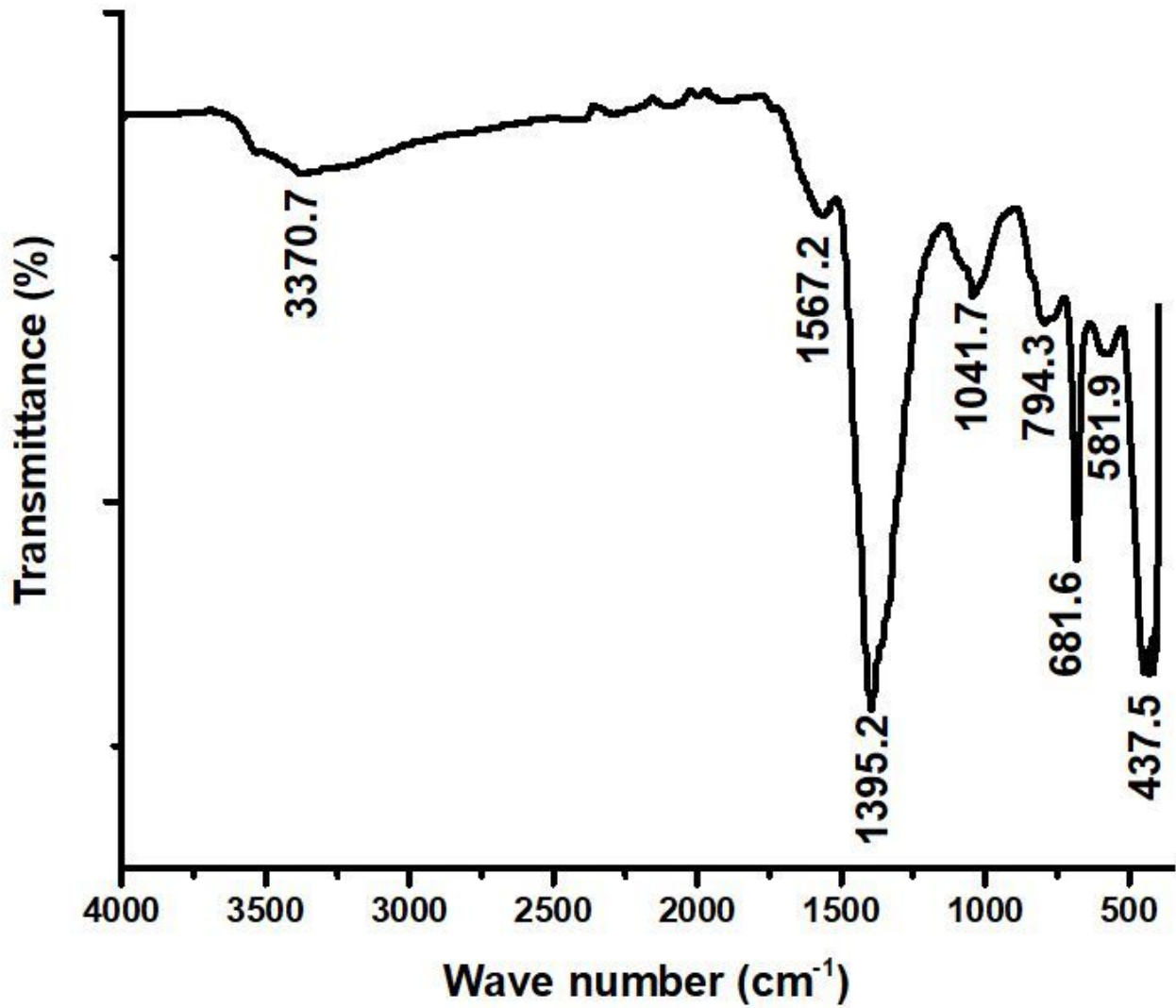




Figure 3

FTIR spectrums of biosynthesized PbO nanoparticles

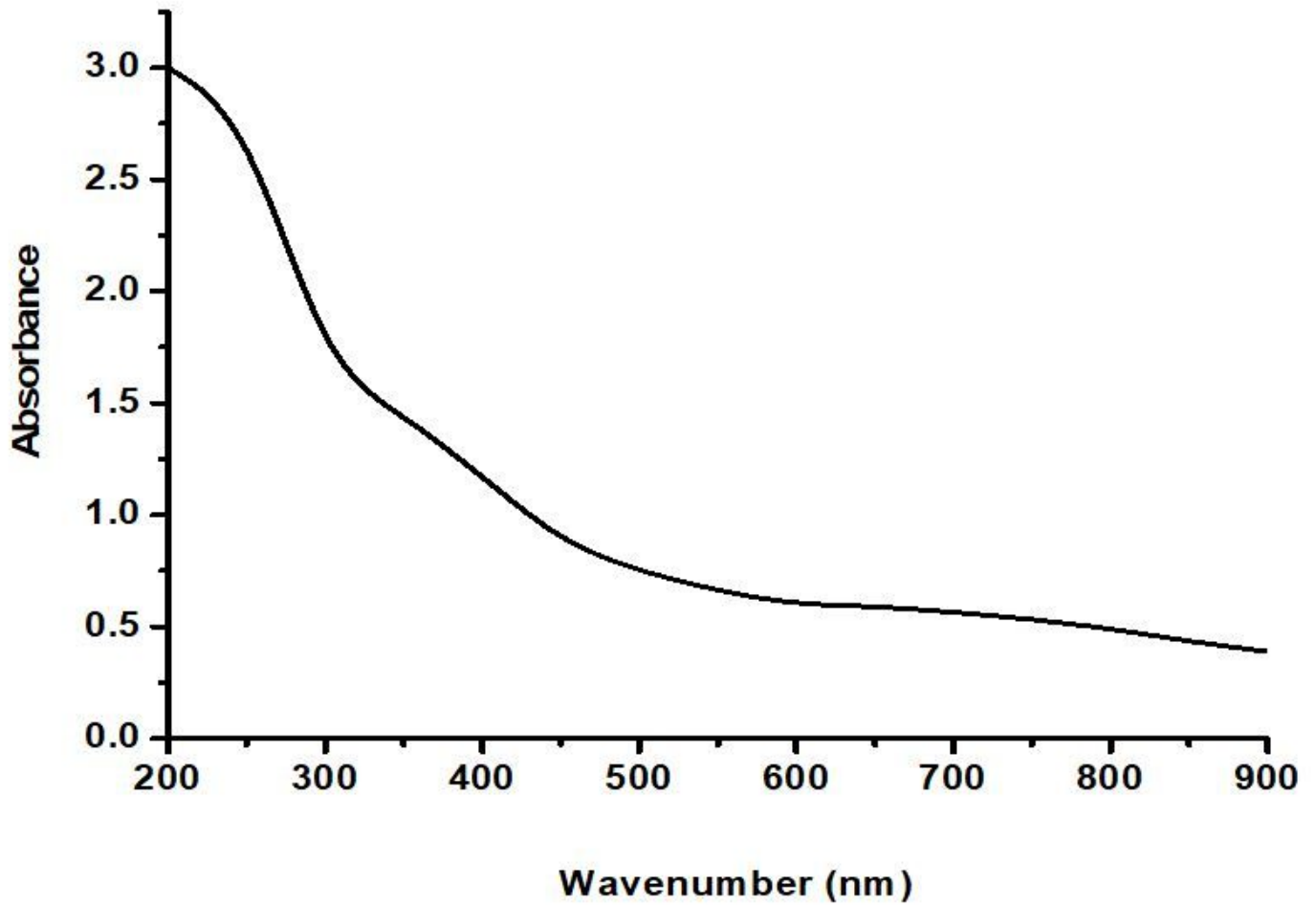


Figure 4

UV- Visible Spectra for PbO nanoparticles