



A CO-PRECIPIATION METHOD FOR THE SYNTHESIS AND CHARACTERIZATION OF LEAD OXIDE AND SILVER OXIDE NANOPARTICLES

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Abstract

In the current studies, the creation of nanoparticles from silver oxide and lead oxide was carried out. Using the co-precipitation approach the PbO (Lead Oxide) and AgO (Silver Oxide) nanoparticles of the current work were successfully prepared. After calcination at 600° C, X-ray diffraction (XRD), ultraviolet (UV) spectroscopy, Fourier transform infrared (FTIR), and scanning electron microscopy (SEM). The produced nanoparticles' size and particle distribution were determined using X-ray diffraction analysis. The procedure is easy to perform and reducing chemicals can be used to control the size. The results show the discovery of AgO and PbO nanoparticles with crystal sizes of 29 nm and 35 nm respectively.

Keywords: Lead oxide, FTIR, Silver oxide, SEM, UV and XRD

I. Introduction

There is currently a lot of interest in investigating chemical species at the nano scale because of the relevance that has been demonstrated in chemical research disciplines due to their various features [1]. Because of their small size and metallic makeup, as well as their biological, optical, and catalytic properties, among others, they are even more intriguing for their practical application. The mass vibration of the free conduction electrons as a result of their interaction with the electromagnetic field is the main focus of the optical characteristics of metal nanoparticles. The nanoparticles develop

a dipole as a result of the electric field produced by radiation to counteract this action, giving it its therapeutic capabilities. Research is being done on the qualities and many potential uses concurrently. Although silver has many applications, its usage as an antibacterial disinfectant is probably one of the most fascinating [2]. This page makes an effort to describe a few of the techniques, classify them and pay close attention to how efficient they are as antibacterial agents. Numerous physical, chemical and biological procedures have been devised to produce silver nanoparticles as a result of the great growth in nanotechnology.

The silver nanoparticles are the most appealing due to the high surface area to volume ratio [3]. The surface area of the nanoparticles is also important and should be controlled as a change in surface area can cause a change in the physical and chemical properties of the nanoparticles [4]. Nanoparticles are of great scientific value because they were developed to bridge the gap between bulk materials and the atomic/molecular structures. But what factors affect these nanoparticles' properties. What factors can we influence to change their characteristics? It is clear that the properties are directly correlated with size when the particles reach a size between 1 and 100 nm. By modifying the size and shape of the particles, one can control properties such as temperature, redox potential, color, conductivity, chemical stability, electrical properties, optics, etc [5]. Numerous studies have demonstrated that the experimental conditions of their synthesis, the kinetics of the reaction, the interaction of the

ions with the reducing agents, and the absorption processes of the stabilizing agent used all have a significant impact on the size, morphology, stability, and properties specifically of silver nanoparticles. As a result, the synthesis technique used will determine how precisely you can control the shape, size, and dispersion of the desired silver nanoparticles. The majority of chemical synthesis is based on reduction reactions of metallic silver salts; however, the desired form of the nanoparticles must first be chosen, from spherical to triangular to cubic to pyramidal to rod-shaped to cylindrical [6]. Once the form is understood, the procedure that best suits the nanoparticle's shape is selected since the nanoparticle's shape is determined by the rate of reaction and how it interacts with the stabilizers. It is important to understand how nanoparticles are created [7]. As was already noted, careful control over size and form during synthesis is necessary to produce a collection of particles with a certain attribute. The following elements are present in a synthesis of the general and must be understood in order to be used and manipulated metallic precursor, reducing agent (solvent), and stabilizing agent [8]. In addition, two crucial formation processes are taken into account: growth and nucleation [9]. In the former, a high activation energy is necessary for the agglomeration of the atoms, whereas in the latter, a low activation energy is available for the formation of particles [10]. At the synthesis points, shape and size are solely dependent on concentration, temperature, reducing power, and pH-7. The stabilizing agent, on the other hand, is crucial to the synthesis [11].

II. Materials and methods

Without additional purification, lead nitrate, silver nitrate, sodium hydroxide, and ethanol were purchased and used. Coprecipitation techniques were used to create lead oxide and silver oxide nanoparticles utilizing lead nitrate, silver nitrate, and sodium hydroxide as precursors. In this experiment, a 0.8M aqueous solution of sodium hydroxide (NaOH) was prepared in the same manner while stirring for 1 hour, along with a 0.1M aqueous solution of lead nitrate and silver nitrate that was kept under constant stirring with a magnetic stirrer to completely dissolve the lead nitrate and silver nitrate. After the lead nitrate had completely dissolved, 0.8 M NaOH

aqueous solution was added dropwise and stirred continuously at high speed while touching the vessel walls for 45 minutes. After finishing with the sodium hydroxide addition, the reaction was let to continue for two hours. Under these circumstances, the beaker was sealed for 4 hours. After the reaction was finished, the solution was let to settle for an entire night before the supernatant was carefully removed. X-ray diffraction, UV spectroscopy, Fourier transform infrared spectroscopy, and scanning electron microscopy analyses are used to describe the nanoparticles.

III. Results and discussion

a. XRD Analysis

This study used XRD to determine the crystal size and purity of the prepared nanomaterials. Figures 1 show the XRD patterns of AgO and PbO nanoparticles. AgO diffraction peaks were noticed at 2θ degrees = 31.46, 37.54, 34.16, 55.25 and 67.41 well-matched with (JCPDS Card No:75-0969). 2θ degrees = 23.65, 26.25, 34.04, 45.27, 50.20, 55.28 and 63.12 its corresponding to Lead oxide nanoparticles emerged simultaneously and were well-matched to (JCPDS Card No:76-1796).

The crystalline size was measured using the Debye-Scherer equation (1). The x-ray wavelength the line broadening at half maximum intensity (FWHM), the Bragg's angle and the crystalline form constant, denoted by $D = 0.9 \lambda / \beta \cos \theta$ are the four constants in this equation. According to this equation, the crystal sizes of AgO and PbO are 29 nm and 35 nm, respectively. The lack of any further peaks in the investigation proved the nanomaterial's purity.

$$D = 0.9 \lambda / \beta \cos \theta \quad \text{----- (1)}$$

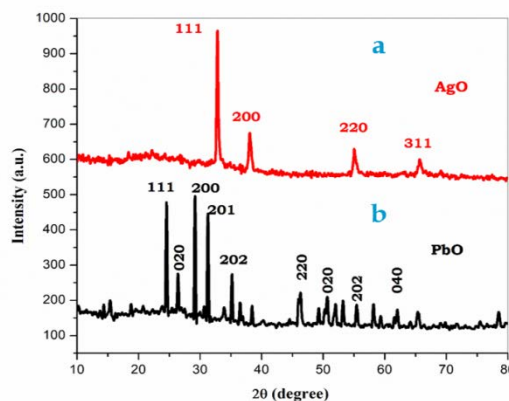


Fig.1 XRD Analysis of AgO and PbO nanoparticles

b.Functional group analysis

Functional group analysis was used to verify the existence of synthesised nanomaterials and to examine the different stretching and bending vibrations that developed between 4000 cm⁻¹ and 500 cm⁻¹ in this work. There was a prominent peak at 701 and 881cm⁻¹ in the AgO crystalline phase which demonstrates the stretching vibration of a bridging oxygen atoms between (O-Ag-O) and that proved the existence of peroxy groups, which can be seen in the wide AgO vibration at 3193cm⁻¹. The broadband 497 and 678 cm⁻¹ PbO nanomaterial peaks were generated. Here we see (Pb-O) vibration groups extending.

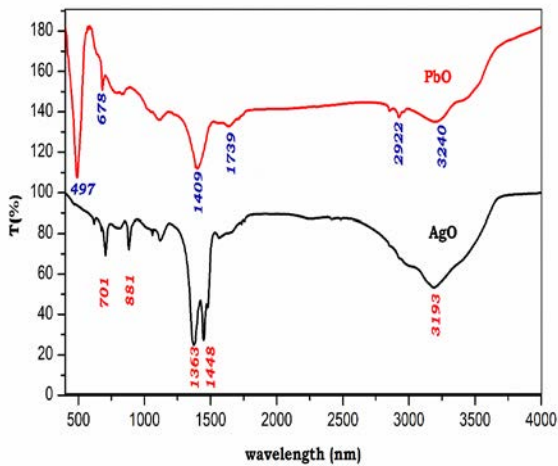


Fig.2 FT-IR Analysis of AgO and PbO nanoparticles

c.Morphology analysis

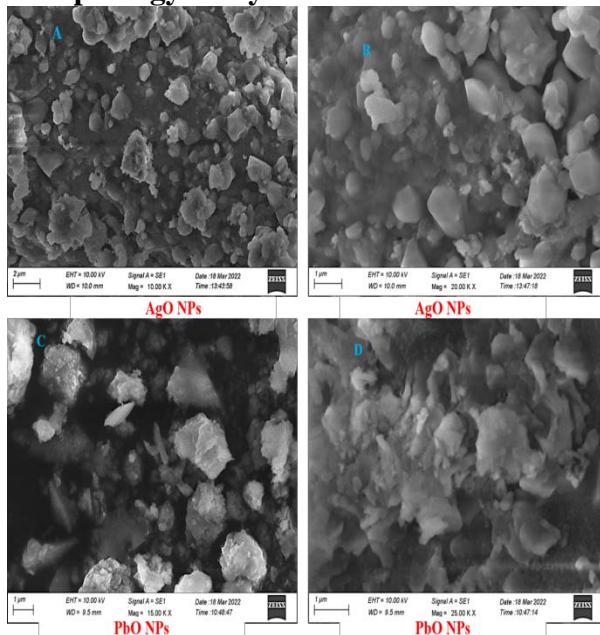


Fig.3 SEM Analysis of AgO and PbO nanoparticles

The SEM images of prepared Nanomaterials are shown in **Fig.3 a & b** Portrait the SEM micrographs of AgO appeared as Nanoparticles. From the **Fig c and d**, the prepared PbO were formed at small agglomeration structure.

d. UV – Vis spectral analysis

UV- spectra of monoclinic AgO and PbO are shown in fig.4. The prepared nanomaterials manifested the high-optical quality in the UV region at the wavelength range of 300nm to 800nm. The spectrum of PbO had slightly shifted due to the addition of the host matrix, Manganese ions. Since AgO has a larger energy gap than PbO due to direct transitions between the nanomaterials, the crystalline size of AgO is higher than PbO.

Equation 2: $(\alpha \cdot hv)^{1/y} = B(hv - E_g)$

Where h is the Planck constant, V is the frequency of the photon, E_g is the energy of the band gap, and B is the constant. Y, the variable that changes depending on the type of electron transition, is equal to 1/2 or 2, depending on whether the transition is direct or indirect. Approximately, Calculated Band gap values of AgO is 2.5 eV & PbO is 2.7eV

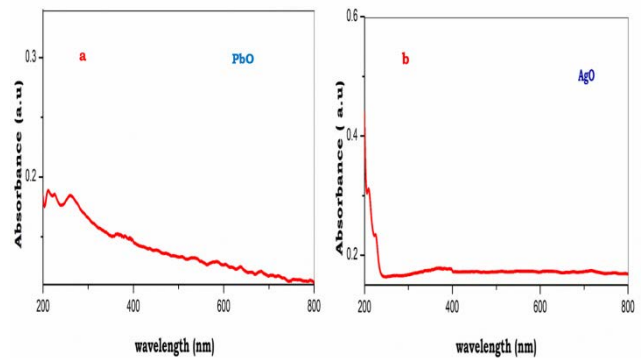


Fig.4 UV spectral analysis of AgO and PbO nanoparticles

IV. Conclusion

The PbO and AgO nanoparticles of the current work were successfully fabricated. FTIR analysis shows a prominent peak at 701 and 881 cm⁻¹ in the AgO crystal phase, showing the stretching vibration of a bridging oxygen atom between (O-Ag-O) and proving the existence of peroxy groups seen in width AgO vibration at 3193 cm⁻¹. The broadband peaks of 497 and 678 cm⁻¹ PbO nano material were generated. Here we see (Pb-O) vibrational groups expanding.

The SEM images of prepared nanomaterials are portraits, the SEM images of AgO appeared as nanoparticles. Since AgO has a larger energy gap than PbO due to direct transitions between the nanomaterials, the crystal size of AgO is larger than that of PbO. XRD to determine the crystal size and purity of the manufactured nanomaterials. It was found that AgO and PbO have a crystal size of 29 nm and 35 nm, respectively.

Author contribution

Methodology, Investigation, Resources N.Mathivanan Writing-Original draught preparation, Synthesis chemicals Conceptualization, Validation, Supervision, Writing-Reviewing and Editing are all responsibilities of K.Raja and K.Tharini.

Competing interests' declaration

According to the authors, there are no financial or personal conflicts of interest that could have skewed the study's findings.

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