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Pharmaceutical Sciences RESEARCH ARTICLE .....!!!

GREEN SYNTHESIS OF LEAD OXIDE NANOPARTICLES USING CASSIA AURICULATA

A.JERLI AUXILIA*, M.KAMALI, S.PRIYANKA, S.MARIA ESTHER

Jayaraj Annapackiam College for Women (Autonomous), Periyakulam-625 601, India.

KEYWORDS:

Cassia Auriculata, UV-Visible, FT-IR, SEM, XRD.

For Correspondence:

A.JERLI AUXILIA *

Address:

Jayaraj Annapackiam College for Women (Autonomous), Periyakulam-625 601, India.

ABSTRACT

Nanotechnology is mainly concerned with synthesis of nanoparticles of variable sizes, shapes, chemical compositions and controlled disparity and their potential use for human benefits. Lead oxide is an important industrial material due to its unique electronic, mechanical, optical properties and its potential applications in Nano devices. Chemicals used for nanoparticles synthesis and stabilization are toxic and lead to nano-ecofriendly byproducts. In recent years green synthesis of metal nanoparticle is an interesting issue of the Nanoscience and Nanobiotechnology. Plants provide a better platform for nanoparticles synthesis as they are free from toxic chemical as well as provide natural capping agents. *Cassia Auriculata* flowers which are used in traditional medicine for rheumatism, conjunctivitis and diabetes. It is used in the treatment of ulcer. In the present work lead oxide nanoparticles have been synthesized from Cassia Auriculata. The synthesized nanoparticles were characterized by UV-Visible, FTIR Spectroscopy, the size of the synthesized particles were determined by SEM and XRD.

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1. INTRODUCTION:

In recent years green synthesis of metal nanoparticles is an interesting issue of the Nanoscience and Nanobiotechnology. There is a growing attention to biosynthesis the metal nanoparticles using organisms. Among these organisms, plants seem to be the best candidate and they are suitable for large scale biosynthesis of nanoparticles [1]. Nano crystals have been of interest in research and technical applications, with respect to its size dependent optical and electronics properties [2-4]. Nanotechnology has gained massive applications in the fields of biology and pharmacology. The intense interest in nanotechnology is being driven by various interesting fields and is leading to a new industrial revolution [5-8]. Plants provide a better platform for nanoparticles synthesis as they are free from toxic chemical as well as provide natural capping agents. Moreover, use of plant extract also reduces the cost of micro organisms isolation and culture media enhancing the cost competitive feasibility over nanoparticles synthesis by micro organisms. Use of biology organisms such as microorganism, plant extract or plant biomass could be an alternative to chemical and physical methods for the production of nano particles in an eco-friendly manner. Cassia Auriculata commonly known as in Tamil language is a shrub belongs to the Caesalpinaceae family. The flowers which are used in the treatment of skin disorders, urinary disorders, female antifertility, leprosy, worm infestation diarrhea, and body odour.

Lead oxide is a general term and can be either lead monoxide or "litharge" (PbO); lead tetroxide or "red lead" (Pb_3O_4); or black or "gray" oxide which is a mixture of 70 percent lead monoxide and 30 percent metallic lead. Black lead is made for specific use in the manufacture of lead acid storage batteries. The major lead pigment is red lead (Pb_3O_4), which is used principally in ferrous metal protective paints. Other lead pigments include white lead and lead chromates. There are several commercial varieties of white lead including leaded zinc oxide, basic carbonate white lead, basic sulfate white lead, and basic lead silicates. Of these, the most important is leaded zinc oxide, which is used almost entirely as white pigment for exterior oil-based paints. [9].

2. Experimental Methods

2.1 Materials:

- *Cassia Auriculata*
- Lead acetate

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- Double Distilled water

2.2 Preparation of *Cassia Auriculata* flower extract:

Cassia Auriculata flowers were collected from periyakulam surroundings and it allowed to dry in dark room for long time till it should be completely dried without the help of sunlight. After that, the flowers are grained well to get powder form. The powder is weighed to 2 gm. The weighed substance is boiled with 100 ml of double distilled water for about 10 minutes. After 10 minutes the boiled solution is filtered. The filtrate show as yellow in color which used as reducing agent. The filtrate solution is called the Cassia Auricualta flower extract.

2.3 Preparation of lead acetate solution

The 0.01N of lead acetate solution is prepared by using 5.74 gm of lead acetate is weighed accurately and transferred in to 250 ml beaker and dissolved completely using 200ml distilled water.

2.4 Synthesis of lead oxide nanoparticles

For synthesis of lead nanoparticle by adding 50 ml of extract and 100 ml of prepared solution of lead acetate both are mixing in 1:2 ratio. The reduction process Pb^{+} to Pb^{0} nanoparticle was followed by the color change of the solution from yellow to light-yellow in colour.

3. Results and Discussion

Characterization

Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Ultraviolet (UV), Infrared (IR) was used to characterize the crystal structure, morphologies, impurities and properties of Pb Nanostructure.

3.1 UV-Visible Spectrum

The UV-Vis spectrum is recorded by Shimadzu 1800 UV Double beam spectrophotometer.

UV-Visible spectrum has been widely used to characterize the semiconductor nanoparticles. As the particles size decreases absorption wavelength will be shifted to shorter wave length and the band gap increases for the nano sized particles. This is Quantum confinement effect of semiconductor nanoparticles. The UV-Visible spectrum of Pb given in fig.1.

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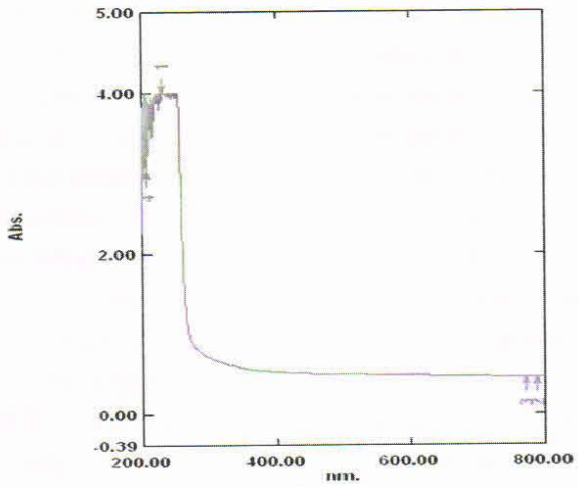

Fig.1 UV-Visible spectrum of PbO

Wavelength nm	Abs
231.00	4.00
788.50	0.45
771.50	0.45
207.50	3.03

In these spectra λ_{max} for PbO is observed at 231 nm. This indicates the absorption shift towards the shorter wavelength, because of the particle size reduction. From these spectra, it is evident that resultant nanoparticles were embedded in silica matrix and exhibited the significant blue shift. This is an indication of strong quantum confinement. The bulk value is at 200-380 nm.

3.2 FT-IR Analysis

Infrared spectroscopy (IR) is the spectroscopy that deals with the infrared region of the electromagnetic spectrum that is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on the absorption spectroscopy. As with all spectroscopic

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techniques, it can be used to identify and study chemicals. A common laboratory instrument that uses this technique is a Fourier Transform Infra red (FT-IR) spectrometer. FT-IR spectra for Pb show fig.2

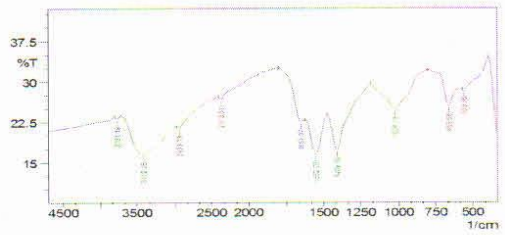

Fig.2 FT-IR spectrum of PbO

The FT-IR spectra of metal sample show specific stretching vibrations for the different structural forms of metal. The specific metal and their IR vibrational frequencies are given below.

The spectrum in the range 659.66 $cm^{-1}$was showing IR absorption due to the various vibrations involved. The stretching frequencies are observed 400-4000 at cm^{-1}. It is confirmed that the obtained nano metal was Pb.

3.3 XRD Pattern

The XRD spectrum is recorded by X-ray diffract meter with Cu kα radiation at 25^0c. the average particle size is determined using Debye-Scherrer's equation applied to major, peaks corresponding to maximum intensity in the XRD patterns of the samplefig.3.

The size of the synthesized PbO nanoparticle were calculated from powder XRD pattern using Scherrer's formula.

$$D=K\lambda/\beta \cos\theta$$

Where

The constant k is the shape factor ≈ 0.94

λ is the wavelength of incident X-Ray (1.540Å)

β is the full width for half maximum

θ is the Bragg's angle for the peak.

B can be calculated using the equation.

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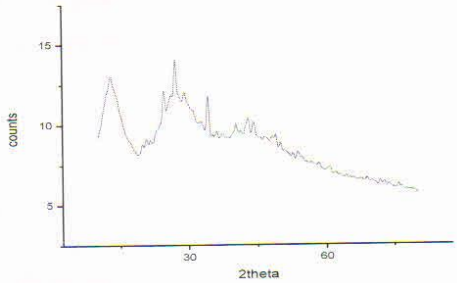

Fig.3 XRD spectrum of PbO Nano

3.4 XRD Data of PbO Nano particles

Group: Out- XRD-818

Data: PbO

Strongest 3 peaks

No	Peak no	2 Theta	D	FWHM
1	2	24.356	3.60886	0.243
2	3	26.842	3.28230	0.292
3	4	33.702	2.62720	0.338

CALCULATION

From XRD Data

$2\theta = 33.702$

$\theta = 16.851$

$\beta = 0.338$

$D = 0.94\lambda/\beta\cos\theta$

Apply the values in this equation

The crystalline size (D) = 4.1002nm

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This shows the XRD pattern of PbO. The observed "2θ" values come in good agreement with standard "2θ" values. This confirms that powder prepared was PbO. The size of the PbO nanoparticles thus estimated was found to be 4.1002 nm.

3.5 Scanning Electron Microscopy (SEM)

The SEM is recorded by JEOL Model 6390 computer-controlled microscope. The image obtained by SEM of the samples for PbO show quasi spherical like nanoparticles. The PbO Nano particles have been distributed well within the range of~ 100nm which is the favorable for some other purpose. We can conclude that the samples of PbO synthesized are having particle size in the Nano scale.

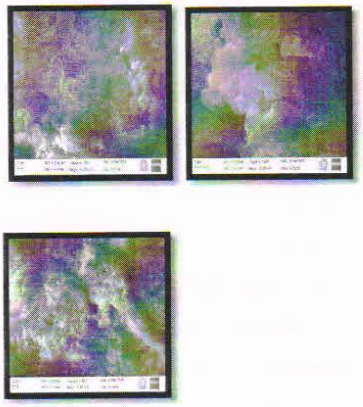

Fig.4 SEM images of PbO Nano

4. CONCLUSION:

The PbO nanoparticles were synthesized by green synthesis using Cassia Auriculta flower extract. The samples obtained by the green synthesis were characterized by UV, FT-IR, XRD, and SEM instrumental methods. The IR analysis of the spectra shows broad band between 400-4000 cm-1 with shoulder shape, characteristic of PbO band. The images obtained by SEM of sample PbO shows quasi spherical like nanoparticles. From the XRD results the size of PbO nanoparticles were calculated to be 4.1002nm. The PbO nanoparticles have been distributed well within the range of ≈100 nm, so it can be used to some other purpose.

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