International Standard Serial Number (ISSN): 2319-8141
International Journal of Universal Pharmacy and Bio Sciences 7(1): January-February- 2018

INTERNATIONAL JOURNAL OF UNIVERSAL PHARMACY AND BIO SCIENCES

*IMPACT FACTOR 4.018****
*ICV 6.16****

Pharmaceutical Sciences

RESEARCH ARTICLE .....!!!

GREEN WAY APPROACH OF PbO NANO PHOTOCATALYTIC DEGRADATION OF AN ORGANIC POLLUTANT AS METHYLENE BLUE

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KEYWORDS:

Green synthesis, photodegradation, Uv-Vis, IR, SEM & XRD.

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ABSTRACT

Organic dyes are extensively used in textiles, leather, printing, food and cosmetic industries. The direct release of unreacted dye effluents into the water bodies (rivers and lakes) is one of the important sources of environmental pollution.These dyes contaminants are chemically stable, produce deep color and bad smell to the water and also highly carcinogenic in nature even at low concentration. Metal oxide play a very important role in many in many area of chemistry physics and material science. Metal oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of cornor or edge surface. Metal oxide nano particle are important because of their small size. Lead oxides have wide range applications such as it is used in glass materials, pigments, gas sensors, paints, storages batteries. Because of it has low cost of manufacturing, reliability. *Solanum Seaforthianum* is an important plant of Indian Ayurvedic system which is used for several health problems. The resultant nanoparticle was characterized using UV-vis, IR, SEM and XRD respectively.

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INTRODUCTION:

In our world today, there is an increasing need in many countries to access clean and safe water both for domestic use and other purposes. The demand for clean water surpasses its supply. This means that water emitted out of industries should be recycled, purified and later used for other purposes pollution that accompanies the dye industry is of great significance and importance. This is particularly because of the non-biodegradable nature of the dyes as well as due to the presence of Acid/Alkali toxic trace metals/carcinogenic aromatic amines in the effluent. In addition to effluent, gaseous emissions such as sulphur dioxide, nitrogenous gases, ammonia/hydrochloric and solid wastes inthe forms of iron sludge, gypsum and sludge from treatment facilities are generated. More than 15% of the total world production of dyes is lost during the dyeing process and is released as a source of textile pollution [1]. Organic dyes are extensively used in textiles, leather, printing, food and cosmetic industries. The direct release of unreacted dye effluents into the water bodies is one of important sources of environmental pollution. These dyes containers are chemically stable, produce deep colour and bad smell to the water and also highly carcinogenic in nature even at low concentration [2].

Methylene blue is water soluble,distinctly used in textile industries. Of the recent technologies, heterogenous photo catalysis using PbO nanoparticles are thee inexpensive, clean and promising method for the degradation of organic pollutents in water. However, the photochemical process of PbO can only be initiated by UV light owing to its wide band gap which confines its application under visible light. Furthermore, the photocatalytic activity of PbO is hindered by the recombination of the photo generated electron hole pairs[3].PbOwas synthesized using *Solanum seaforthianum*. PbO nanopowder were characterised by UV, FTIR,XRDand SEM.

MATERIALS AND METHODS

Materials

- Lead acteate
- Copper sulphate
- Solanum seaforthianum,
- Mentha longifolia
- Spirit

- De-ionized water

Preparation of *Solanum seaforthianum* leaf extract

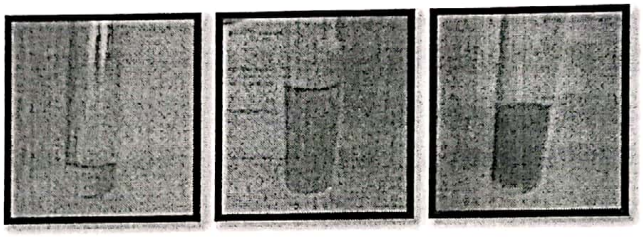
Solanum seaforthianumleaves were collected fromhome and was thoroughly washed with distilled water in order to remove the dust particle. All glass wares were washed with de-ionized water. 5gms of Solanum seaforthianumleaves weighed and boiled for30 minutes, and filtered. The filtrate used as reducing agent.

Preparation of stock solutions

A stock solution of 0.1N lead acetatewas prepared by dissolving 3.79g/100ml using de-ionized water respectively.

Green Synthesis of lead oxide nanoparticles

For the synthesis of the PbO nanoparticles, 4 ml of the *solanum seaforthianum*extract was added to the 2ml of lead acetatesolution . The solution was stirred for 30 minutes. Nano particles were followed by the color change of the solution from green to golden yellow. After 2hours the PbO powder which is in brownish yellow.



Green Synthesis of PbO Nanoparticles

Preparation of dye solution

10^{-3} dye solution was prepared by weighing about 3.274 gof methyene blue made upto 100ml using de-ionized water respectively.

From this 10 ml of methylene blue was taken in a test tube and add 0.1 g of PbO nano catalystthen kept in sunlightrespectively. The dye has been degraded bychanging the colour.

RESULTS AND DISCUSSION

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

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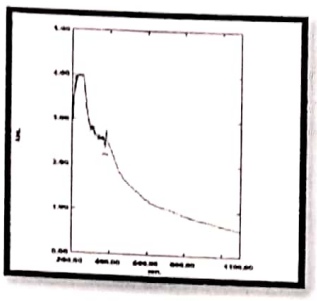
UV-Visible Spectrum

In these spectra λ_{max} for PbO are observed at 260 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 for PbO is at 200-380nm.

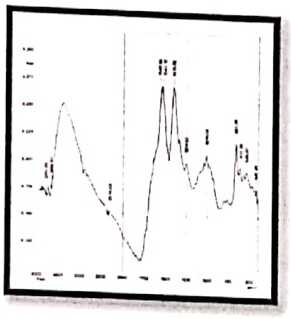


UV Visible spectrum of PbO

FT-IR Analysis

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

The stretching frequency of PbO is 657.73cm^{-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 oxide was PbO.



FT-IR Spectrum of PbO

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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 samples. The sizes of the synthesized CuO nanoparticleswere calculated from powder XRD pattern using Scherrer's formula.

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

Where

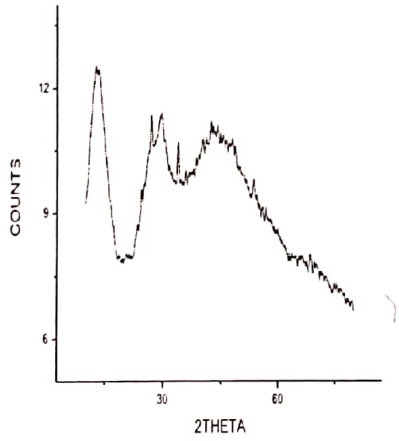
The constant k is the shape factor ≈ 0.94

λ is the wavelength of incident X-Ray (1.5406Å)

β is the full width for half maximum

θ is the Bragg's angle for the peak.

β can be calculated using the equation.



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XRD spectrum of PbO Nanoparticle

No.	Peak no.	2 Theta (deg)	D (A)	FWHM (deg)
1	1	26.822	3.2940	0.245
2	2	33.662	2.6342	0.294

CALCULATION

From XRD Data

$2\theta=33.662$

$\theta=16.831$

$\beta=0.294$

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

Apply the values in this equation

The crystalline size (D) =5.14 nm

Fig. 15 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 5.14nm.

Scanning Electron Microscopy (SEM)

The image obtained by SEM of the samples for PbO show sphere [4] 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 sample of PbO synthesized is having particle size in the Nano scale.

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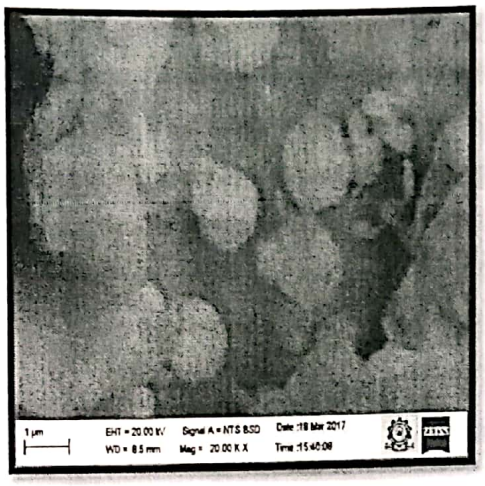


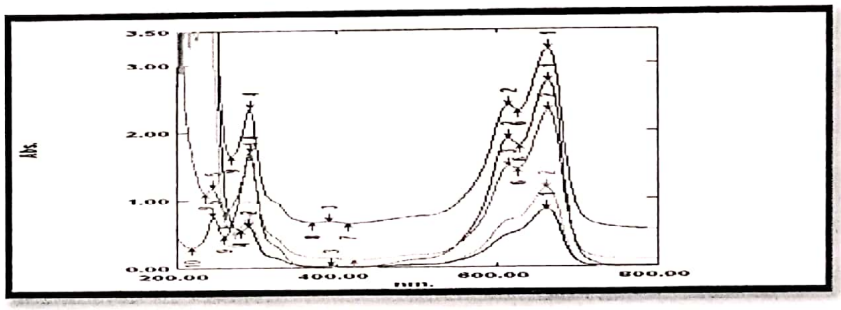
SEM image of PbO Nanoparticle

PHOTO CATALYTIC DEGRADATION STUDIES

Effect of variation of initial concentration of Methylene blue dye

The mixture containing the dye solution and the nanoparticles is exposed to sunlight and its effect on rate of bleaching was studied. The extent of degradation of the dye in solution is studied at a definite intervals of time [30 minutes] using UV-visible spectrum. The variations in absorbance are represented below. If more concentration of dye is taken, it imparts a darker colour to the solution and it may act as filter to the incident light reaching the semiconductor surface. As a consequence, the rate of Photo catalytic bleaching of Methylene blue dye decreases.

Study of Photodegradation of Methylene blue using UV spectrum



When PbO irradiated with light, PbOis excited by photons led to the formation of electrons and holes in the conduction and valance band of PbO. The electrons react with surface adsorbed O_2 to produced $O_2^{\bullet -}$ and holes react with H_2O to create $^{\bullet}OH$.

DETERMINATION OF PERCENTAGE REMOVAL OF DYE

The extent of removal of the dye in terms of the value of percentage removal of dye has been calculated using the following relationships.

Percentage removed =100(C_o–C) /C_o

Where

C_0 =Initial concentration of dye (ppm)

C =Final concentration of dye (ppm)

CALCULATION

Methyene Blue with PbO

Percentage removed = 100(C_0-C)/ C_0

Where

C_0 = 0.373M ; C = 0.0568M

Percentage removed = 100 (0.373-0.0568)/0.373

Percentage removed = 84.7%

CONCLUSION

The green synthesismethod has been used for the synthesis of PbO nanoparticles using solanum seaforthinum. The two samples obtained by the green synthesis were characterized by XRD, FT-IR, UV and SEM instrumental methods. The IR analysis of the spectra shows broad band between 601-648cm^{-1} with shoulder shape, characteristic of PbO band. The images obtained by SEM of samplesPbO showspherelike nanoparticles. From the XRD results the size of PbO nanoparticles were calculated to be 5.14 nm. The PbO nanoparticles have been distributed well within the range of ≈100 nm which is the favourable property to exhibit better photo catalytic activity. The photo catalytic degradation of the dyes was carried out using UV radiation. So we can conclude that, photo catalytic degradation of the dyes were carried out with UV radiation and the photodegradation for methylene

blue with PbO was found to be 85% From this project, the green synthesized nano catalysts have good photocatalytic properties for the degradation of methylene blue dye.

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