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SYNTHESIS, CHARACTERIZATION AND APPLICATION OF NANO-SIO_2 AND NANO-PBO IN DYE DEGRADATION

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ABSTRACT

The precursor solutions were made by mixing Silicon Tetrachloride and Lead chloride respectively for SiO_2 and PbO nanoparticles. The detailed Microscopic and Spectroscopic characterizations of the surface morphology for both nanoparticles were conducted by using Ultraviolet Spectroscopy (UV), Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD). To study the effect of these synthesized nanoparticles, the photo catalytic reduction of harmful azo dye – methylene blue has been investigated using SiO_2 and PbO photo catalyst in aqueous solution under irradiation. Experiments were conducted further to check phytotoxicity. These observations indicate that all the two dyes could be degraded completely with similar time intervals. Hence, it may be a viable technique for the safe disposal of textile wastewater into the water streams.

KEY WORDS : Nanomaterial, Silicon dioxide, Lead oxide, Azo textile dyes, Characterization, Phytotoxicity

INTRODUCTION

Nanoparticles have a high specific surface area, interfacial dominated properties, and electrical/ optical properties that can be distinguished with respect to corresponding bulk materials and the one hand and individual molecules on the other. Therefore nanoparticles find application in diverse fields such as battery technology, fuel cell, wastewater treatment, electronics and sensors, etc. Because of the simplicity of design, low cost of manufacturing, reliability and relative safety there is great use of nanomaterial in various fields. Dyes used in textile industry are the vast source of coloured organic pollutants that increased the environmental danger. During the manufacturing and production huge quantity of wastewater containing these toxic dyes can be introduced into the aquatic systems. Due to the stability of these modern textile dyes, convenient methods are ineffective for their decolouration and toxicity reduction. Recent literatures have revealed that photocatalysis can be the viable way to mitigate this issue using photocatalyst in irradiation (Manoj Singh *et al*., 2011; Anastas and Williamson, 1998; Aileen *et al.,* ?????? ; Mohammed Mehdi *et al*., 2011).

The purpose of this study was to find the degradation of harmful methylene blue dye with the photocatalytic system by using different type of catalyst with different loading, concentration of dye, and the role of these catalysts on the degradation efficiency with its phytotoxic effect of the recycled wastewater (Manoj Singh *et al*., 2011; Rajamohan and Karthikeyan, 2006).

The present work deals with the synthesis of nano-SiO_2 and nano-PbO particles by using hydrothermal method at lower temperature. The obtained powder was characterized by using SEM, XRD, UV and FTIR. Also, the photocatalytic activity checked using Congo red dye and methylene blue dye. Further, we have also studied the phytotoxicity, recyclability and photocatalytic activities of SiO_2 and PbO Nano powders.

Chemicals

Silicon tetrachloride ($SiCl_4$) and lead chloride

($PbCl_2$) were used as a source of silicon and lead in the fabrication of nanoparticle. Sodium hydroxide flakes (NaOH), TRITON X100 LR (t-octypenoxypolyethoxyethanol) was used as received from Sigma Aldrich Corporation. For the dye preparation, methylene blue dye was used in various amounts as received with distilled water.

Procedure

A. Process for synthesis of nanoparticles

Take 4 g of NaOH flakes and 50 mL distilled water were mixed together in a beaker. Add 3.4 mL of SiCl4 (silicon tetrachloride). Add 3.4 ml of Triton (t-octylphenoxypolyethoxyethanol). Shake well & add 50 ml+10ml of distilled water. Take in Teflon bottle in auto clave reactor. Shake well; Keep the mixture in an oven for 24hr at 120 °C. After 24 hours remove the sample s from the oven and filter with the help of silica grouch crucible by water jet vacuum pump. Dry the samples in hot air oven at 120 °C for 5 hours. Take the dry samples in alumina crucible. The dried samples are kept in muffle furnace for calcinations purpose at 600 °C for 3 hours and then the samples are grinded. After grinding products are stored in vials. Similar procedure has followed for the synthesis of PbO nanomaterial.

B. Process for preparation of dyes

Take 25 mg of Congo red dye in distilled water and make up solution of 500 ml. similar process has done for the preparation of methylene blue dye.

Characterization

A. SEM

Table 1. EDX analysis of SiO_2 nanoparticles

Element	Weight%	Atomic%
O K	46.18	60.10
Si M	53.82	39.90
Total	100	100

Smaller rods structures have seen at the lower magnification. The Rod of flecks may be the effect of possible agglomeration. The average particle size is 353.83 nm. Fig. 1 (d, e, f) represents SEM pictures of the synthesized SiO_2. The shapes of the particles are similar to each other and likely become spherical in general. In the SEM images it is also seen that the synthesized SiO_2 powder particles are agglomerates. However, the size distribution of the powder varies

Table 2. EDX analysis of PbO nanoparticles

Element	Weight%	Atomic%
O K	18.21	74.25
Si M	81.79	25.75
Total	100	100

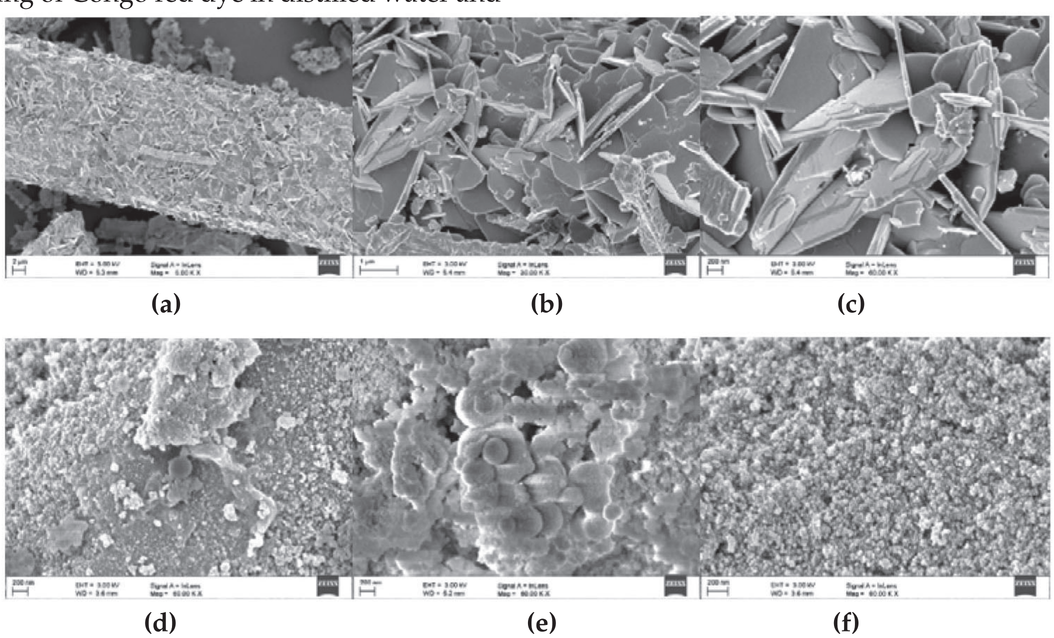


Fig. 1. Scanning Electron Microscopy (SEM) of Nanoparticles
a,b,c : SEM of Lead nanoparticles(PbO); **d,e,f :** SEM of Silica nanoparticles (SiO_2)
Fig 1(a, b, c) shows SEM images of PbO material is showing the flakes structure.

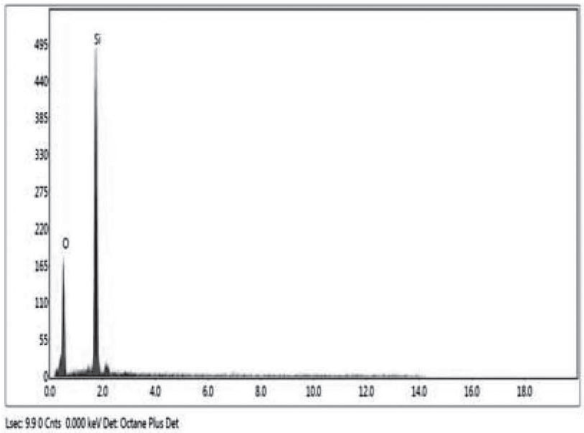


Fig. 2A EDX of SiO_2 nanoparticles

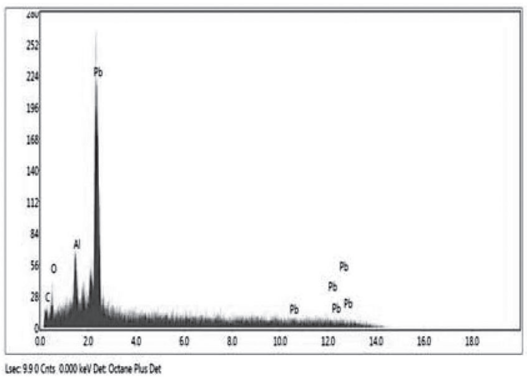


Fig. 2B EDX of PbO nanoparticles

in the range for 76.3 nm to 728.34 nm. The agglomeration may result from the chemical treatment conditions.

For elemental analysis of the given samples, energy dispersive x-ray spectroscopy was used. The above Fig. 2A represents EDX of SiO_2 nanoparticles and Fig. 2B represents the EDX of PbO nanoparticles. The elemental composition is clearly states that, there is formation of SiO_2 and PbO compositions. The other peaks in the graphs of PbO nanoparticles may arise due to impurities during the process.

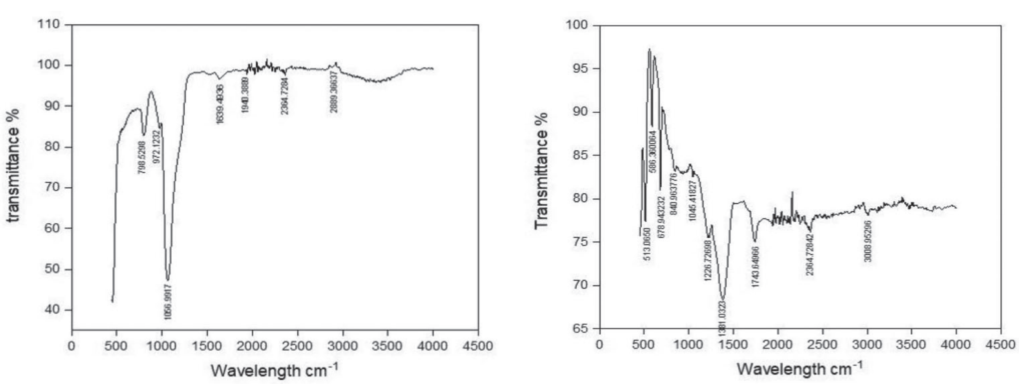
Table 3. Adsorption Vs Time study for methylene blue dye (30 ppm)

Time	Adsorption in SiO_2 for 30 ppm	Adsorptionin PbO for 30 ppm
0	1.987	1.987
30	1.531	1.726
60	1.164	1.495
90	0.821	0.856
120	0.596	0.367
150	0.203	0.126
180	0.03	0.043

B.FTIR

FTIR provides the sample information on the basis of physical state (solid, liquid, gas) and the chemical composition. The FT-IR spectrum of synthesized SiO_2, dried at 1200C and that of calcined at 6000C is shown in Fig. 3(a). The IR band at 2889.36 cm^{-1} could be assigned to the stretching vibrations of Si–OH groups in the structure of amorphous SiO_2. Correspondingly, the IR band at 1639.38 cm^{-1} is due to the bending vibration of H2O molecules. The very strong and broad IR band at 1066.99 cm^{-1} is usually assigned to Si–O–Si asymmetric stretching vibrations. The IR band at 798.52 cm^{-1} can be assigned to Si–O–Si symmetric stretching vibrations.

The FT-IR spectrum of synthesized PbO, dried at 120°C and that of claimed at 500 °C is clearly represented in Fig. 3(b). The peak of 513.06 cm^{-1} indicates the formation of PbO material. The IR peak at 586.36 cm^{-1} indicates the presence of oxides. Peak value 678.94 cm^{-1} represents Pb-O-Pb starching



(a) **(b)**

Fig. 3. FTIR diffraction of (a) SiO_2 nanoparticles & (b) PbO nanoparticle

vibration. These two peaks are very sharp. It is confirmed that the final product is the presence of lead and oxide. The peak value 1381.03 cm^{-1} assigned for the presence of OH group.

C. XRD

Fig. 4(a) shows a broad peak located approximately at 2θ = 21.24°, that suggests an Amorphous characteristic of the sample and agrees with the reported JCPDS data (card No. 01-086-1561). Fig. 4(b) shows the XRD pattern of Lead Oxide nanoparticles. The phase purity of the prepared tetragonal n-PbO can be clearly seen and all diffraction peaks are perfectly indexed to the tetragonal PbO structure. No characteristics peaks of impurities were detected. The broadening of the peaks indicated that the particles of nanometer scale.

The XRD pattern of the PbO reveals crystallographic structure of the synthesized PbO powders. The three strongest peaks of scattering angle of 29.24, 31.16, 31.54, 35.68, 44.98 corresponds to the reflections from the 111, 020, 200, 201 and 022 crystal planes respectively according to the literature (JCPDS Card No. 38- 1477) (Fang and Huang, ???????? ; Theivasanthi and Alagar, 2010; Kikuo Okuyama *et al*., 2002; Schraml Marth *et al*., 1992

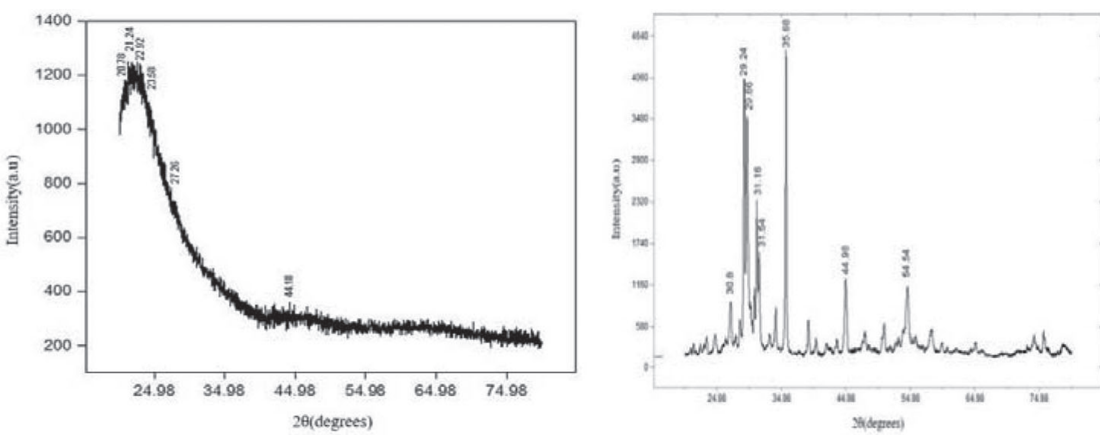
UV

1. The photo reactivity of nanostructured SiO_2 is greatly improved by the high efficiency and wide range of light absorption. The sharp peak at 303 nm is due to near band edge emission in the case of PbO (Das Chayan and Kapgate Bharat, 2012; Rajkumar *et al*., ????? ; Ramesh Kumar *et al.,* 2004).

RESULTS AND DISCUSSION

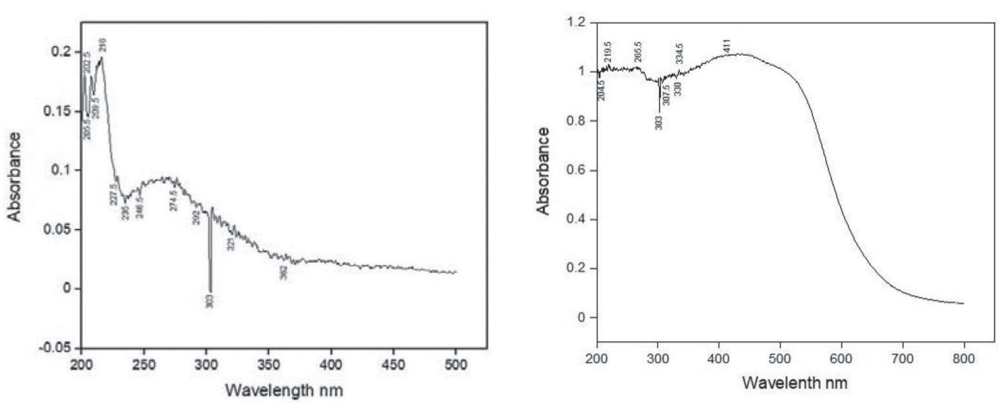
Dye Degradation

Methylene blue using $SiO_2$2. Methylene blue using PbO



(a) **(b)**

Fig. 4. X-ray diffraction of (a) SiO2 nanoparticles & (b) PbO nanoparticle



(a) **(b)**

Fig. 5(a) UV-Vis of SiO_2 nanoparticles, &(b) UV-Vis of PbO nanoparticles

Fig. 6 (a). Degradation of methylene blue by using SiO_2 Nanoparticles and (b) Degradation of methylene blue by using PbO Nanoparticles

Phytotoxicity Check

Toxic effect on the growth of the plant may define as phytotoxicity. This kind of damage may cause by the several variety of compounds like pesticides, phytotoxins, etc. The plant shows the negative growth during the effect of phytotoxins. In some instances, plants react to substances like humans with food allergies react to certain foods. The substance can be applied, and within a short period, it will become evident that the plants are intolerant to it. The plants may be able to tolerate a substance in smaller doses, but a grower may consciously or unconsciously over-saturate the plants only to find that it poisons them. Here, the last solution of the methylene dyes (after 180 minutes) used to check the phytotoxicity. The plant seed sowed in the pots and the effects of the degraded samples checked. It had seen that the plant growth is normal in degraded samples. Hence, the process described in the paper may be used to treat the industrial dyes solutions.

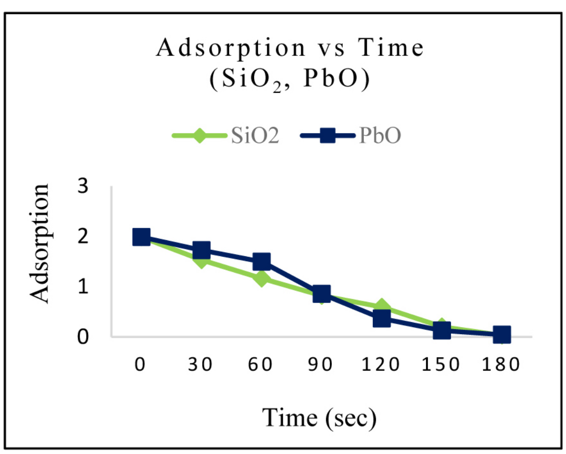


Fig. 7. Adsorption Vs Time study for methylene blue dye (30ppm)

The adsorption of methylene blue dye of (intensity =30 ppm) through nano-SiO_2 and nano-PbO catalyst performed in UV photo chemical reactor separately. It is evident from Table that within 180 minutes the colour of dye is completely adsorbed. But the result shows that during initial 30 minutes the dye couldn't show any significant change in color in the case of PbO nanoparticles and afterwards it is adsorbed by the catalyst.

CONCLUSION

The obtained result describes a simple sol-gel method for the synthesis of SiO_2 and PbO as a catalyst at lower temperature with high yields. This nanoparticles catalyst provides a new way for continuous processes. Both the nanoparticles characterized by UV-Vis, FTIR, XRD, SEM. This catalyst would be helpful to understand the advantages combination of the properties of homogenous catalyst and the development of new catalytic systems. Further the same Catalyst can be used for a number of cycles which will reduce the cost of operation.

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