Synthesis and characterization of PdO nanoparticles by solution combustion method and kinetics Study of Indigocarmine dye

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Abstract

Palladium Oxide (PdO) nanoparticles were synthesized by solution combustion method by using palladium (II)nitrate. The PdO nanoparticles were characterized by UV-Visible spectra- Ray diffraction (XRD), Scanning Electron Microscope (SEM), The UV – Visible spectra revels that the band gap energy PdO nanoparticles is 2.45eV using Tauc plot. The XRD pattern shows that the synthesized PdO nanoparticles were pure and nano-sized, The SEM image revels that the PdO nanoparticles were spherical shape. The nanoparticles and the presence of Pd and O in the nanoparticle is confirmed from EDAX spectrum. The photocatalytic activity of the synthesized PdO nanoparticles was examined by the kinetics of degradation of indigo carmine dye. The photocatalytic decolourization of the dye follows first order kinetics.

Keywords: Palladium Oxide (PdO) nanoparticles, solution combustion method, Photodegradation Indigocarmine (IC).

I. Introduction

Due to the optimal combination of activity and selectivity, palladium is one of the most used transition metals for both homogeneous and heterogeneous catalysis of organic reactions. In general, heterogeneous catalytic processes are preferred with respect to homogeneous ones due to the higher yields and easier reusability of the catalytic material. Since heterogeneous catalysis is based on processes taking place at the surface, the use of finely dispersed catalysts is often adopted in order to reduce the amount of material and maximize its catalytic effect. For this purpose, palladium nanoparticles are widely used to catalyze organic reactions. Similar to palladium, palladium(II) oxide (PdO) has also found applications in many catalytic processes [1]. Metallic nanoparticles continue to receive attention of interdisciplinary researchers because of their unique physicochemical, electronic and magnetic properties. In order to realize the potential of these nano scale metal particles, the focus of research is on to developing new synthetic methodologies, improved characterization techniques and finding novel applications [2-4]. Great interest has been focused on the noble metal nanoparticles due to their interesting size- and shape dependent physical and chemical properties. They are technologically important in many fields such as catalysis. Major of Pd applications requires very small and fine Pd particles where both the size and shape are critical parameters that should be controlled in order to maximize their efficiencies. For example, the reactivity and selectivity of a nanocatalyst can be tailored by controlling the shape, as it will determine the crystallographic facets exposed on the surface of a nanocrystal and therefore the number of atoms located at the edges or corners [5-8]. Palladium nanophases such as Pd(0), PdO, and PdS have potential applications in catalysis, electronics, and hydrogen storage. Palladium nanoparticles are effective catalysts for Suzuki reactions in both aqueous and non-aqueous solvents [9-13].Recently, with relatively lower cost of production, palladium (Pd)-based electrocatalysts have attracted great attention aiming to replace the costly platinum group metals, including oxygen reduction reaction (ORR)[14].In the present work we are

synthesized PdO nanoparticles by solution combustion method and kinetic degradation of Indigo carmine has been studied by using PdO nanoparticles under the UV light.

II. Experimental Details

Synthesis of PdO nanoparticles by solution combustion method.

1g of palladium (II) nitrate was taken in a beaker to this 2g of glycine and 30ml of water was added then kept beaker on a hot plate upto completely charged .The solid was obtained. The obtained solid was transferred into crucible and kept it in combustion chamber maintained at 8000C for 3hour.The coloured PdO was obtained.

III. Results and discussions

3.1. X-ray Diffraction

The purity of synthesized PdO nanoparticles were identified using X-ray diffraction .The position of the peak 2teta is 38.70,44.2 .Rapidly indexed as (111) (200) .The crystal structure was found to be face-centered cubic crystal structure an using d average crystal size was calculated using Debye-scherrers formula $D = k\lambda/\beta\cos\theta$ and it was found to be 19.2nm from the XRD pattern [15].

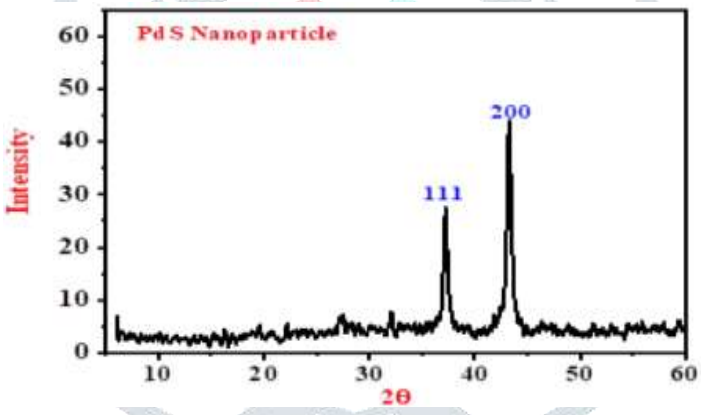

Figure. 1. X-ray diffraction spectra of PdO nanoparticles

3.2. UV-visible Spectra:

The synthesized PdO nanoparticles has showed that maximum intensity peak at 299.13nm.The band gap of PdO nanoparticles was calculated using Tauc plot it was found to be 2.45eV .

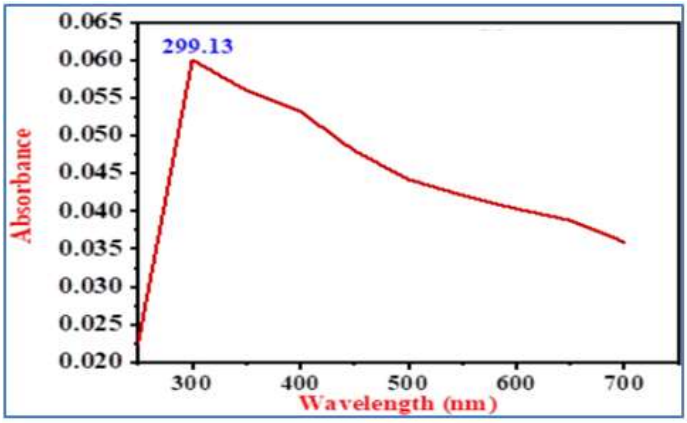

Figure. 2. UV-Visible spectra PdO nanoparticles

3.3. Scanning Electron Microscopy (SEM)

The SEM image of PdO nanoparticles consists of agglomerated particles and it was observed using SEM micrographs. The EdAX spectra show the presence of Pd and O in the nanoparticles.

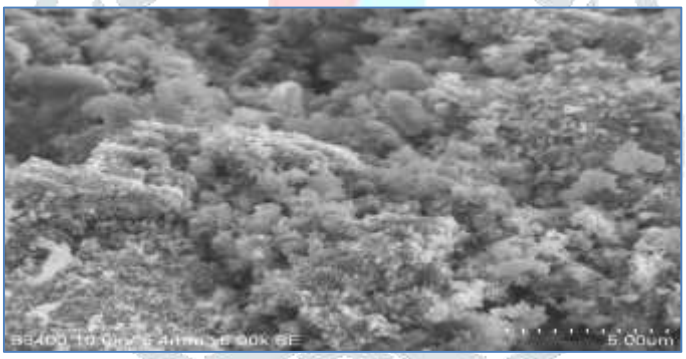

Figure 3. SEM images of electrochemically synthesized PdO Nanoparticles.

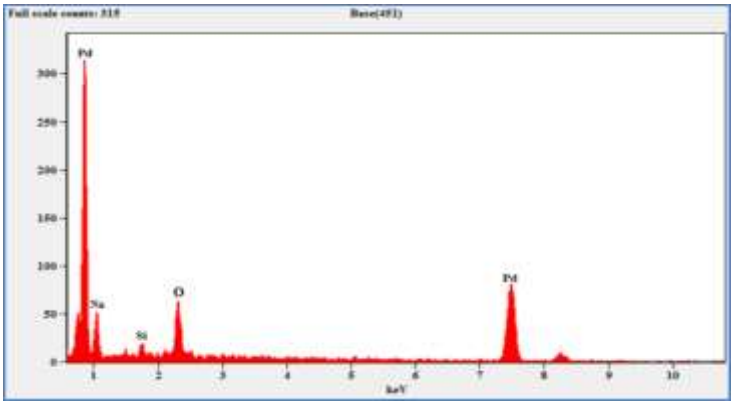

Figure 4. Energy dispersive X-ray analysis spectrum of PdO Nanoparticles.

IV. Photodegradation Kinetics

4.1. Effect of concentrataion and catalyst

The photodegradation experiment was carried out with different concentration of indigo carmine dye solution ($1x10^{-4}$,$2x10^{-4}$,$3x10^{-4}$ N) with constant weight of PdO nanoparticle. The change in concentration of the indigo carmine was reported by change in color using spectrophotometer. A plot of log Tt (percentage transmittance of light) versus time was linear up to 60 % of the reaction indicating the disappearance of indigo carmine follows first order kinetics (Figure 5). The rate constant values are given in table 1 and the reaction rate decreased with increase in indigo carmine. The reason beyond that is with increase in the dye concentration, the solution becomes more intense colored and the path length of the photons entering the solution is decreased and the few photons reached the catalyst surface. Hence the production of hydroxyl radicals is reduced. Therefore, the Photodegradation efficiency is reduced. The experiments were performed by taking different amount of catalyst varying from 10mg to 30mg keeping dye concentration constant in order to study the effect of catalyst loading. The study showed that increase in catalyst loading from 10mg to 30mg increased dye removal efficiency. Further increase in catalyst above 30mg decreased the photo activity of the catalyst, due to aggregation of PdO nanoparticles at higher concentration causing a decrease in the number of active sites on catalyst surface and increase in the light scattering of PdO nanoparticles at high concentration.

Table 1. Effect of Photodegradation at different concentration of Indigocarmine dye under UV light

Concentration of IC in 10^{-4} N	10^4 k Sec^{-1}	Effect of COD mg/l	
		Before degradation	After degradation
1.0	2.163	456	32
2.0	1.568	530	16
3.0	1.231	585	48

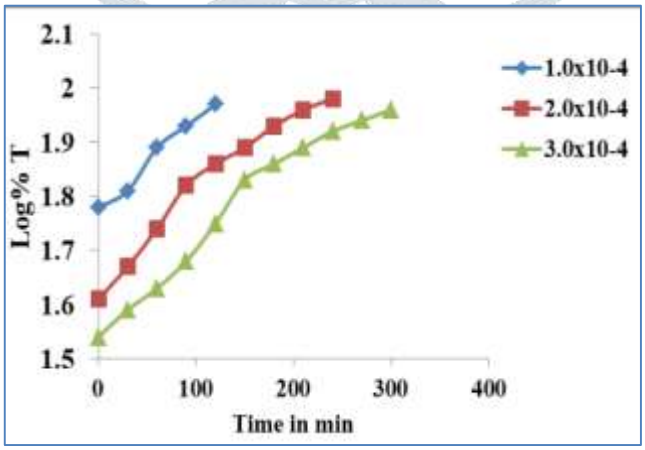

Figure 5. Effect of concentration of IC on the rate of degradation under UV light.

Catalyst PdO in mg	10^4 k Sec^{-1}	COD Values in mg/l	
		Before degradation	After degradation
10	1.325	530	48
20	1.568	530	16
30	2.236	530	16

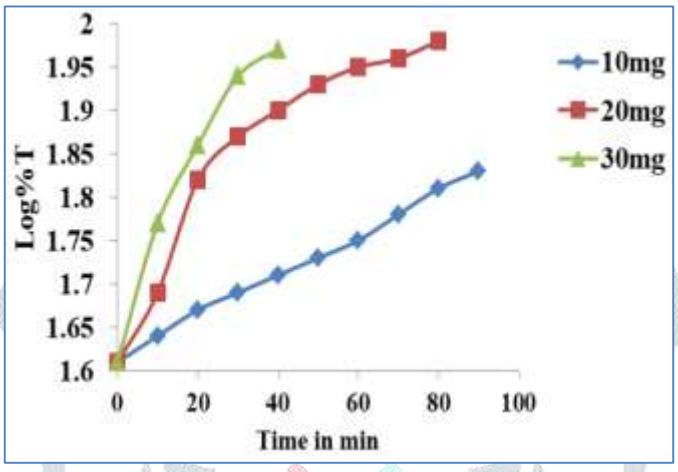

Figure 6. Effect of catalyst loading on the rate of degradation of Indigo Carmine under UV light

Reuse of catalyst:

The reuse of catalyst was investigated to see the degradation efficiency of the IC dye solution. After the degradation of dye, the dye sample was kept outside without expose the UV-light for 5 h and supernatant liquid sample was decanted. The catalyst was thoroughly washed with double distilled water and reuse for the photodegradation by taking new indigo carmine dye solution. The reuse of photocatalyst shown almost same degradation efficiency compared to the fresh sample. This can be recommended the photocatalyst can be regenerated and reused.

VI. CONCLUSION

In the present paper the PdO nanoparticles were synthesized by solution combustion method .The synthesized PdS nanoparticles were characterized by SEM, XRD, and UV analysis. The photodegradation by this semiconductor offers a green technology for removal of organic dyes present in waste water and industrial effluents. Kinetics of photodegradation of organic dye (IC) recommended that the dematerialize of IC follows 1st order kinetics. The photodegradation rate in UV light is high compared to sunlight hence the synthesized PdO nanoparticles acts as a very good photocatalyst and is active under UV light.

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