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Controlled Synthesis and Study Characterization of Pt Nanoparticles

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Abstract
In this paper, Palladium (Pt) nanoparticales (NPs) at different construction (150 and 500) mg capped with poly (vinylpyrrolidone) (PVP) was Synthesize by a polyol reduction method in an ethylene glycol solution at temperature of 45C˚. The structural and optical properties of Pt NPs have been investigated, all thin films were tested using X-ray diffraction (XRD), all XRD peaks can be indexed as face centered cubic (FCC) Structure, with strong crystalline orientation at (111) plane. The morphology properties of the prepared films were study by Atomic Force Microscopy (AFM) the results indicated that films have nanoscale grain size (29.18, 46.91) nm. The size of the particles increased with increasing of concentrations.

Keywords: Polyol Reduction, Pt Nanoparticles, Poly(vinylpyrrolidone), XRD, SEM, AFM, UV. Visible

1. Introduction

Photonics researches require materials with large nonlinear susceptibilities and fast response time. These two important properties are available in noble metal nanoparticles like gold, silver, and palladium. Such materials have close-lying bands and their conduction bands lye near Fermi level. The electrons in conduction band move nearly free with oscillation motion led to rise resonance of the surface plasmon [1].

Among many noble metal nanoparticles, palladium occupies a large importance in hydrogen storage, sensing, and other applications which require catalytic materials [2]. In addition to catalysis and sensing, metal nanoparticles are used in optics, electronics, biomedicine cancer therapy and fuel cells that work at low temperature [3]. Noble metal nanoparticles (NPs) work as homogeneous and heterogeneous catalyst in chemical process industry, therefore; they are considered as an enhancement factor in these processes. Such properties have been of interest for centuries.

Over the last few years, efforts have been focused on manufacturing noble metal NPs with controlled size, shape, and size distribution of the nanoparticles [4]. Accordingly different methods have been employing for this purpose. One of the preferred methods over the mentioned period is polyol's synthesis. It is suitable for synthesizing noble metal nanocrystals with well-defined shapes due to the ability of polyols such as ethylene glycol (EG) to dissolve many metal salts. In addition to the dependence of the reducing power of the reaction on the temperature [5]. In this process, the EG works as reducing agent to reduce a metallic cation to metallic collids [6]. The reduction reaction occurs at high temperature with the assistance of polymeric stabilizer usually polyvinylpyrroidone (PVP).

K. Patel *et al.* [7] synthesized Pt, Pd, Pt – Ag, and Pd – Ag nanoparticles using microwave – polyol method. In their work they studied the effect of PVP on complexing and stabilizing nanoparticles. T. Nishi *et al.* [8] prepared Pd nanoparticles using laser ablation in heavy and light water and tested the magnetic properties of the product.

In this paper, we report our results on preparation well shaped Pd nanoparticles using polyol system. The effect of variation of precursor concentration on the particle size is shown. Also, the experimental conditions were fixed.

2. Experimental

Palladium nanoparticles (Pt NPs) prepared using Palladium nitrate dehydrate $Pt(NO_3)_2.2H_2O$ and Polyvinylpyroiodine (PVP) as a stabilizer agent. There are dissolved by ethylene glycol ($C_2H_6O_2$).

The samples were prepared at temperature (45 ºC) ± 2 ºC for reaction time (60) min. Palladium nanoparticles (Pt NPs) were prepared by adding (500, 150) mg $Pt(NO_3)_2.2H_2O$ in 5 ml of ethylene glycol. Also 100 mg PVP as a stabilizing agent was dissolved in 25 ml of ethylene glycol. On the other hand, 45 ml of ethylene glycol was heated by magnetic stirrer at (45ºC) ± 2ºC for reaction time (60) minute, the temperature was monitored using a thermometer. The inject of the two solutions in the

heated ethylene glycol with a burette and a syringe pump and fed to reaction mixture for 15 minutes after dosing the precursor/stabilizing agent solution. A light brown coloration was observed indicating the formation of Pt nanoparticles. After the dosing was completed, the resulting mixture was heated in the same temperature of the E.G for the same reaction time. Finally, the solution was cooled to room temperature.

The excess PVP was removed by repeating suspension (3-5 times) of the particles in ethanol and acetone at 6000 rpm for 5 minutes each time by centrifuge. The final product was then dispersed in ethanol. Palladium thin films were prepared by dip coating technique. Glass substrates were dipped in the prepared solution and withdrawn at a rate of 1cm sec^{-1}. The substrate stayed in the solution for 60 sec and was subsequently dried for 5 mints at 100 C^o.

An optical interferometer method was used to determine the films thickness. In this method, the samples were exposed to He:Ne laser beam. An interference pattern formed due to the interference between the light beam reflected from the film surface with that reflected from the substrate. Then the sample thickness was found using the following equation [9].

$$d = \frac{\lambda}{2}\frac{\Delta x}{x} \quad (1)$$

where λ is the wavelength of the laser, x is the width of the bright fringes, and Δx is of the dark fringes.

3. Results and discussion

3.1 Structure measurement using

a) (XRD) Analysis

X-ray diffraction has been employed for identification and understanding the crystalline growth nature. The resultant diffraction patterns of the palladium nanoparticles are shown in fig (1) and (2) at different concentrations (150) and (500) respectively. These nanoparticles which prepared at 45C^o show two different located peaks (111) and (200) for Pt (150) and three peaks from (111), (200), and (220) plans for Pt at (500) respectively. From the figures, all XRD peaks can be indexed as faced centered cubic (FCC) with strong peak at (111) direction for both concentrations. The absence of peaks of any other plans gives an indication that the product is at high purity. Further from the figure, it can be seen that the peaks' intensities increased with increased concentrations.

The grain size of the produced Pt nanoparticles can be calculated from the peak broadening using Scherer equation [10, 11].

$$D = \frac{0.4\lambda}{\beta_{FWHM}\cos\theta_d} \quad (2)$$

Where D: The mean diameter of the Pt nanoparticles, λ is the wave length of the X-ray source diffracotmeter, and β is the full-width-half-maximum (FWHM) of the XRD peak corresponding to the Bragg angle $2\theta_d$. Also, the diffraction patterns have been used to calculate the lattice constant and the results with the grain size are listed in tables 1 and 2 for experimental and standard values. These results show a good agreement with literature [12, 13].

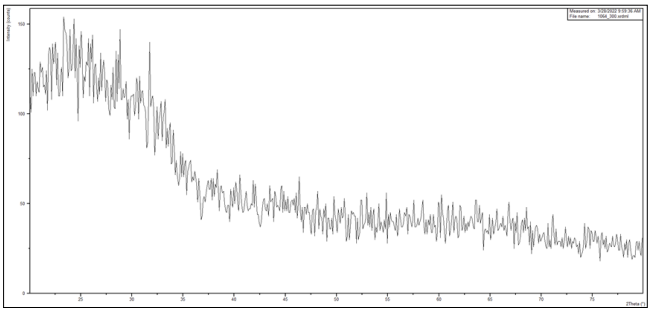

Fig 1: The XRD pattern of Pt NPs at concentration (150)

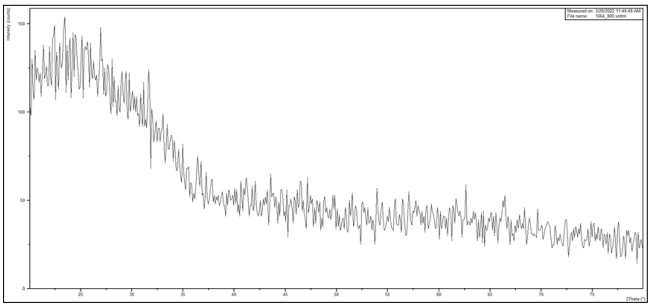

Fig 2: The XRD pattern of Pt NPs at concentration (500)

3.2 Structure measurement using

a) (AFM) Analysis

The surface morphology of the Pt NPs was investigated using (AFM) analysis. The value of roughness and grain size were calculated from the height values in AFM image using the commercial software.

Fig 3 (A and B) and4 (A and B) shows two and three-dimensional of (AFM) images of Pt NPs deposited at various construction (150, 500) respectively. The average grain size of the particles is in nanoscale and the values of average grain size and surface roughness were listed in Table (3).

Table 3: Average Grain size and surface roughness of Pt NPs

Pt at different construction	Average grain size (nm)	Roughness (nm)
150	29.18	2.27
500	46.91	2.98

A grain is a single crystalline or polycrystalline. It refers to agglomerate in bulk or thin film. The grain size is estimated by XRD pattern or Scherer equation but the latter is only an approximation. One particle consists of several grains and its size is larger. The particle size can be measured either by AFM, SEM, or TEM. This explanation justifies why there is a different between nanosizes measured by AFM and that of XRD pattern.

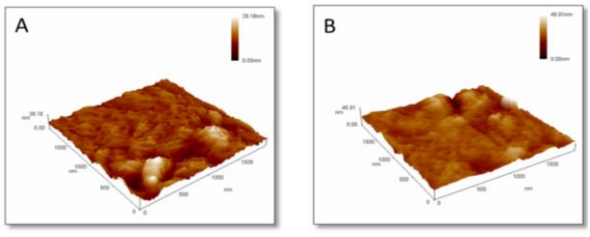

Fig 3: Two-dimensional AFM of images of Pt NPs (A 150) and (B 500) deposited on galas plate.

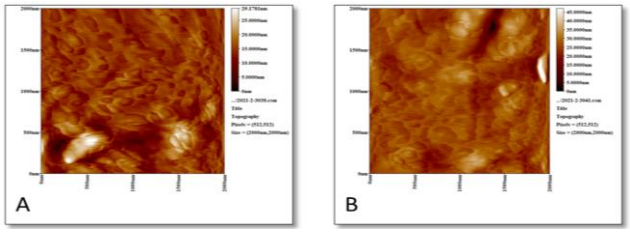

Fig 4: Three-dimensional AFM of images of Pt NPs (A 150) and (B 500) deposited on galas plate

3.3 Optical measurement

a) Absorbance spectrum

UV – VIS spectrophotometer was used to record the optical absorption spectra of the Pt nanoparticles thin films. The absorption spectra for the different precursor concentration and within the wavelength range of (200 – 1100 nm) are shown in fig 5 (A and B). It can be observed that when the concentration increases; the absorption value is also increases. This occurs due to the participation of more atoms in the absorption process. At visible light region, the strong photo-absorption is presented in the wavelength 330 nm at each pulse. The spectra in fig 5 (A and B) agree with references [7, 14, 15, 16, 17].

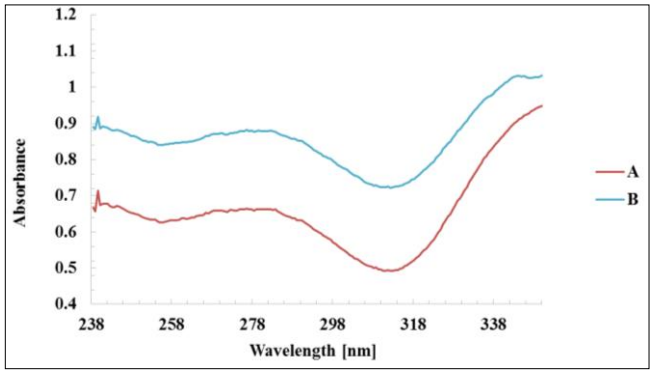

Fig 5: Absorption spectrum of Pt NPs at construction 150 (A) and 500(B)

3. Conclusion

Pt nanoparticls with uniform shape were prepared by polyol method at 45˚C. The current results show that polyol method is an easy and efficient method for synthesizing nanoparticles and modifying the kinetics of atoms to assembly, growth, and generate particle shapes. The size of the Pt nanoparticles could be manipulated by varying the metal precursor concentration. Further, using ethylene glycol as reducing agent leads to formation of chemical residues that can be easily removed from the reaction mixture.

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