Electrodeposition of Platinum (Pt) Particles as a Catalyst of Hydrogenation of Acetone

Yusnita Rahayu1,2, Setia Budi3* and Vivi Fauzia1*

1Departemen Fisika Universitas Indonesia, Kampus UI Depok 16424, Indonesia
2Sekolah Tinggi Teknik-PLN, Jl. Lingkar Luar Barat, Jakarta Barat 11750, Indonesia
3Department of Chemistry, Universitas Negeri Jakarta, Rawamangun Muka, Jakarta 13220, Indonesia

*Corresponding author: vivi@sci.ui.ac.id

Abstract. Heterogeneous catalysts play an important role in accelerating chemical reactions in order to save energy and cost during the reaction process. Platinum (Pt) has received a great deal of attention for its application in many catalytic processes, but very few studies have evaluated it in the hydrogenation process of acetone. In this study, Pt particles were successfully grown on an indium tin oxide (ITO) coated glass substrate by means of a facile, one-step, and template-free electrodeposition method using a solution containing 2.0 mM H2PtCl6 and 0.5 M H2SO4 at room temperature. Potentiostatic and galvanostatic methods were employed to conduct the electrodeposition process under different potentials, current densities, and deposition times to observe their influence on the morphology of the electrodeposited Pt particles. It was found that the morphology of the particles could be facilely manipulated by adjusting the deposition current and potential by using the galvanostatic and potentiostatic methods, respectively. The potentiostatic method produced a spherical or bayberry-like shape whereas the galvanostatic method produced a flower-like shape. The sharp tips on the surface of the bayberry-like shape are electron-rich sites that can break the C=O bond in acetone, thereby forming C-OH bonds that can generate isopropanol molecules.

Keywords: electrodeposition, platinum, indium tin oxide, hydrogenation

1. Introduction

Platinum (Pt) is still the most promising and commonly used catalyst due to its high stability in the aggressive chemical environment of strongly oxidizing and highly acidic conditions and high electrochemical potentials and gradients [1]. Since Pt is expensive and rare, extensive efforts have been made to tailor the morphology of noble metals with nanoscale precision [2], [3]. Several techniques have been used for the synthesis of nanoparticles. These methods are classified as involving either physical or chemical techniques: (i) physical methods include sputtering, evaporation, pulsed laser deposition, and ion implantation; (ii) chemical methods include sol-gel, co-precipitation, impregnation, chemical vapor synthesis (CVS), hydrothermal and liquid phase depositions [4]–[6]. Compared with these methods, electrodeposition has proven to be the most effective, clean, and facile method for controlling the morphology and size of the electrodeposited nanoparticles through the choice of suitable electrolysis conditions [7], [8].

The preparation of shape-tunable metal nanoparticles remains a synthetic challenge. Moreover, Pt nanoparticles with a variety of shapes, such as nanowires, nanoflowers, nanothorn, nanorod, and
nanodendrites have been explored using electrodeposition [2], [3], [9]–[12]. The formation of porous, branched, or dendritic shapes is an interesting issue because it will increase the surface-area-to-volume ratio significantly such that the surface/facet and interface effects become the predominant factor and can even significantly modify the macroscopic properties of the nanomaterials to enhance their catalytic performance [13], [14].

Li et al. [15] analyzed Pt Nanocrystals enclosed by both low-index facets and high-index facets. Their study also revealed that the supporting electrolyte, growth potential, and concentration of the precursor are the key elements in tuning the shape and surface structure of Pt NCs. Pt particles with different surface morphologies were deposited onto an indium tin oxide (ITO) substrate by controlling the electrochemical deposition current density, and their electrocatalytic activities were reported [2], [3], [12]. Previous studies have demonstrated that two types of Pt electrodes prepared by direct and pulse current electrodeposition produced different morphologies: dendritic growth and nanocluster, respectively [9]. Zhang et al. [16] investigated the deposition potential of Pt nanoparticles and found that it largely influences the size and morphology of Pt crystalline orientation domains.

Catalytic acetone hydrogenation is known as a benign method to produce isopropanol, which is widely regarded as serving an essential function in fine chemical synthesis and in technological processes by acting as a solvent for many organic reactions or for potential fuel cell materials precursors [17]–[19]. Apart from its promising applications, the production of isopropanol is expensive, time-consuming, and produces a toxic by-product. The effort to produce isopropanol with an efficient but green method should continue [19]. Acetone hydrogenation is a cheap and environmentally safe alternative method for the production of isopropanol because of its easy reduction of the C=O bond into C-OH [20].

2. Experimental Methods

2.1. Pt deposited onto the ITO preparation

Electrochemical preparation of the Pt particles was conducted at room temperature in a standard three-electrode cell with a Pt plate counter electrode and Ag/AgCl in saturated potassium chloride. The eDAQ 163 potentiostat was used to control the electrical current flowing through the system. Before electrodeposition, the ITO coated glasses substrate (1.5 cm x 2.5 cm) were sequentially washed by sonication in acetone, isopropanol, and ultrapure water and subsequently dried in an oven for 1 h. Reduction of Pt$^{4+}$ ions in an aqueous solution consisting of 2.0 mM H$_2$PtCl$_6$ and 0.5 M H$_2$SO$_4$ resulted in the deposition of the Pt particles onto the ITO substrate.

Pt particles were electrodeposited by the galvanostatic method (at current: $I = 0.12$ mA and 1.00 mA, deposition time: $t = 400$ s) and the potentiostatic method (at voltage: $V = -0.15$ V and -0.25 V, deposition time: $t = 400$ s). The obtained samples were rinsed with plenty of ultrapure water and dried at room temperature.

![Figure 1](image_url)  
**Figure 1.** Scheme of the electrochemical cell used in the synthesis of Pt particles onto the ITO substrate.
2.2. Morphology characterization
The optical properties of the Pt particles deposited onto the ITO substrate were evaluated by measuring the absorbance using a Thermo Fisher Scientific GENESYS 10S UV-Vis spectrophotometer. The surface morphology and the elemental analysis of deposited Pt particles were identified by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) using the FEI Quanta 650.

2.3. Hydrogenation catalytic characterization
The catalytic properties of the particles were evaluated by using a hydrogenation reaction of acetone into isopropanol under microwave radiation. In the typical process, one Pt/ITO substrate 0.75 cm x 2.5 cm was immersed to 5 mL 0.1 M acetone solution and placed in a tightly closed Teflon container in a commercial microwave oven. The microwave power of 110 W was used due to its ability to produce the highest isopropanol yield [21]. The hydrogenation of acetone was observed based on the degradation of the absorbance peak characteristic of acetone at 278 nm over the course of 300 s.

3. Result and Discussion
Figure 2 shows SEM images of Pt particles electrodeposited at different currents on the ITO surface. The bright particles refer to the deposited Pt that was confirmed by energy dispersive X-ray spectrum (Figure 4).

Pt particles that were electrodeposited at a current of 0.25 mA (Figure 2(a1) and 2(a2)) appear cotton candy-like with a diameter range of 496 nm. When the current was set to 1.00 mA, the size of the Pt particles increased to 1.506 μm with a higher number of particles. As the depositing current was increased the average size of the Pt particles increased. However, the number density of Pt particles gradually increased as the deposition current was increased. This result indicates that a higher current tends to promote new nucleation instead of the growth of existing nuclei. It is worth noting that the deposition current has a significant influence on surface morphology.

Furthermore, it appears that the surface morphology of the Pt deposits is related to changes in the electrode potential. The electrodeposition process is controlled by the mass transfer of Pt ions from the
bulk solution through the diffusive layer toward the electrode (diffusion process) and the reduction reaction on the electrode (activation process) [22].

A substrate material seems to influence the growth mechanism of deposited Pt particles. The Pt deposits featured relatively isolated Pt particles on the ITO substrate, whereas Pt deposits tended to grow into a film on the glassy carbon substrate. This variation may be attributed to the different surface energy between the ITO and other substrates [12]. Li et al. reported the influence of ion electromigration on the growth of Pt NCs [15]. The conductivity of a solution with a supporting electrolyte has a greater value than that of a solution without a supporting electrolyte. \( \text{H}_2\text{PtCl}_6 \) plays a role in electromigration and migrates towards the anode in a solution without a supporting electrolyte. Low conductivity in supporting electrolyte-free solutions blocks the electromigration of ions, thus only a limited amount of Pt precursors can reach the cathode.

From the SEM images, we observed that the galvanostatic method tended to produce larger particles than those obtained by using the potentiostatic method (Figure 3). Figure 3 shows the SEM images of the surface morphologies of the Pt particles depositing using potentiostatic method at -0.25 V and -0.15 V for 300 s. The size of the Pt particles prepared by controlling the electrode potentials at -0.25 V and -0.15 V increased from 396.9 nm to 732.9 nm. When the depositing potential was increased, different morphologies of the deposited Pt were observed (Figures 3(a2) and 3(b2)). A gradual morphological transition of the particles occurred when the applied potential exceeded -0.25 V. The spherical with little petal tip particles were initially formed at an earlier stage (Figure 3(a1) and 3(a2)). They acted as the nuclei for the subsequently produced Pt particles with a bayberry-like shape. At a relatively higher growth potential, sharp branches began to grow on the Pt particle surface, resulting in the bayberry-like shape at -0.15 V. The as-prepared Pt bayberry-like shape was formed mainly due to the spherical-petal shape growing anisotropically, thus they are naturally porous [10].

**Figure 3.** SEM images of the surface morphologies of Pt particles on the ITO substrate by the potentiostatic method at voltage (a) -0.25 V and (b) -0.15 V with deposition time 300 s; (a2) and (b2) are the magnified views of (a1) and (b1), respectively.
Figure 4. EDS spectrum of Pt particles deposited on the ITO substrate using the electrodeposition method.

Based on the SEM micrograph information of the as-prepared Pt particles, the possible growth mechanism of the various deposit morphologies can be proposed. When the Pt deposit is subjected to the anodic one with relatively low potential (e.g. $E < -0.15$ V), the metal particles cease to grow [3]. At this point, the Pt atoms on the region with high surface energy are inclined to diffuse or migrate into the regions with lower surface energy, thereby forming a more thermodynamically stable morphology, such as a spherical shape [23]. By increasing the anodic potential, the Pt surface is oxidized with the following typical reactions [24]:

\[
\begin{align*}
\text{Pt} + \text{H}_2\text{O} & \rightarrow \text{PtOH}_{\text{ad}} + \text{H}^+ + 2e^- \\
\text{PtOH}_{\text{ad}} & \rightarrow \text{PtO}_{\text{ad}} + \text{H}^+ + e^- \\
\text{PtO}_{\text{ad}} + 2\text{H}^+ & \rightarrow \text{Pt}^{+2} + \text{H}_2\text{O} \\
\text{Pt} & \rightarrow \text{Pt}^{+2} + 2e^- \\
\text{PtO}_{\text{ad}} + \text{H}_2\text{O} & \rightarrow \text{PtO}_2 + 2\text{H}^+ 
\end{align*}
\]  

Figure 5. Schematic illustration of Pt deposition by means of four-electron electroreduction of $[\text{PtCl}_6]^{2-}$ to Pt.
The Pt surface is, therefore, covered by oxygen molecules, including OH_{ad} and O_{ad}. The Pt particle finally grows into a bayberry-like shape because the edge and corners of the crystal nucleus grow quicker than other parts do. In addition, the formation of Pt particles with sharp-tips is related to the effect driven by sulfuric acid anions. Since sulfuric acid anions, such as HSO_4^- and SO_4^{2-}, in the solution have a preference in selective adsorption on specific surface planes, further growth of these specific crystal planes are disturbed or inhibited, thereby promoting anisotropic growth of the material [8], [12], [22]. Figure 5 demonstrates how the Pt deposition occurs by means of a disproportionation reaction of \([\text{PtCl}_6]^{3-}\) to Pt. It shows that the surface morphology of the deposited Pt particles on the ITO substrate is strongly dependent on electrocatalytic conditions (electrodeposition methods, time, concentration of metal salt solution and electrolytes, etc.) [7].

Figure 6 shows the optical absorbance spectra of as-prepared Pt/ITO with different electrodeposition methods. The spectrum shows absorption bands with two peaks: one at ~370 nm and one at ~600 nm, which may be due to their surface plasmon resonance properties. Pt particles electrodeposited by the galvanostatic method at current 0.25 mA have shifted the optical absorption peak to 375 nm and 630 nm (Figure 6(a)), and this shift may be due to the bulk size of the particle. The optical absorption and scattering of nanoparticles are strongly dependent on their size, shape, and composition. The optical properties, such as absorption, scattering, and extinction cross section of a particle’s arbitrary shape have been extensively studied by using Mie theory and the discrete dipole approximation [25], [26]. Because of the bulk size of the Pt particles, along with the absorption of electromagnetic radiation, scattering is also expected. Figure 6(b) indicates that the absorbance intensity of the Pt particles that were prepared by the potentiostatic method at -0.15 V has decreased, which may be due to the decrease in the number density of Pt particles.

![Figure 6](image-url)  
**Figure 6.** UV-visible absorbance spectrum of Pt/ITO electrodeposited by (a) the galvanostatic method with different currents and (b) the potentiostatic method with different voltages.

The heterogeneous catalytic properties of Pt particles were examined in the heterogeneous catalytic hydrogenation of acetone to isopropanol under microwave irradiation. Balouch reported that Pt exhibits excellent catalytic properties in the acetone hydrogenation reaction to produce isopropanol under microwave irradiation at 110 W, which provides a high-yield conversion [27], [28].

The catalytic activity of the Pt particles in the hydrogenation process of acetone into isopropanol was performed by observing the characteristic absorbance peak of acetone at a wavelength of 265 nm. The hydrogenation process of acetone under microwave radiation (110 W) for 150 s appears to decrease the absorbance peak intensity of acetone. To determine the degradation rate, we can use the absorbance intensity data using the following equation [21]:

\[
\eta = \frac{A_0 - A_1}{A_0} \times 100\%
\]
$A_0$ is the absorbance peak intensity of the original acetone solution at $t = 0$ whereas $A_1$ is the absorbance peak intensity of acetone solution after $t$ seconds. The optical absorbance dynamic of acetone during the catalytic hydrogenation is shown in Figure 6. The acetone was effectively hydrogenated to form isopropanol as shown as the decrease of acetone characteristic absorbance peak (265 nm) as the reaction time increases. The graph of the degradation rate in the absence of a catalyst is ±20.6% whereas the final yield of degradation rate of acetone in presence of catalyst Pt particles deposited by the galvanostatic method at 0.2 mA and 1.0 mA reached ±48.3% and ±71%, respectively (Figure 7(a)). This low catalytic activity may be due to the bulk structure and low density of the Pt particles deposited at 0.2 mA.

This study did not use a chemical as a hydrogen source because water itself is often used as a hydrogen source to perform the hydrogenation of acetone to isopropanol [27]. The catalytic acetone hydrogenation follows the Langmuir-Hinshelwood mechanism in which both hydrogen and acetone molecules are chemisorbed onto the metal particle surface [29]. Electropositive hydrogen molecules coordinate with the oxygen atom in the carbonyl group (C=O) such that polarization and the weakening of the C=O bond occur. Thus, the C=O bond of acetone breaks up, causing the formation of C-OH alcohol group, and isopropanol molecules are subsequently formed on the surface of the catalyst. This process is followed by the breaking of the metal-carbon bond, thereby releasing the alcohol product from the metal surface to the solution, which then creates an empty space on the catalyst surface for the adsorption of new acetone molecules [21] (Figure 8).

Figure 7. Degradation rate of acetone during the hydrogenation reaction in the absence and presence of catalyst Pt/ITO prepared by (a) the galvanostatic method and (b) the potentiostatic method.

Figure 8. Schematic illustration of the mechanism of catalytic activity for the hydrogenation of acetone.
Figure 7(b) shows the degradation rate of acetone after the immersion of Pt particles deposited by the potentiostatic method. The degradation of acetone in the absence of a catalyst was ±20.6%, whereas the degradation of acetone in the presence of Pt particles deposited by the potentiostatic method at -0.25 V and -0.15 V was 73.4% and 72.4%, respectively. Both electrodeposition conditions produced almost similar catalytic activity despite the Pt particles having different morphologies. The sharp tips on the surface of the bayberry-like shape serve as electron-rich sites that induce superior surface chemistry and catalytic reactivity properties. Moreover, the Pt particles provide an electron-rich surface to break the C=O bond in acetone, thereby forming C-OH bonds that can generate isopropanol molecules.

4. Conclusion
Pt particles with different morphologies and particles sizes were electrodeposited onto an ITO glass substrate. We found that the morphology of the particles could be facilely manipulated by adjusting the deposition current and potential by using the galvanostatic method and the potentiostatic method, respectively. With the potentiostatic method, the spherical shape and the bayberry-like shape were produced using a voltage of -0.25 V and -0.15 V, respectively. With the galvanostatic method, the bulk shape and the flower-like shape were produced using an electrical current of 0.2 mA and 1.0 mA, respectively. The morphology and size of the particles prepared by the potentiostatic method at a potential of -0.15 V show excellent catalytic performance in acetone hydrogenation even though the sample has a lower density than the one prepared at -0.25 V, approximately 72% in 150 s of reaction time. In addition to this promising practical application, our study provides a fundamental understanding of the heterogeneous catalytic hydrogenation of ketones, which could assist in the rational design of a catalyst that can be used in the hydrogenation of other carbonyl compounds.

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