Synthesis of the platinum particle with the pH variation for the particle size control

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Abstract. Platinum is a precious metal widely used in the jewelry industry due to its property and intrinsic value. The different particle sizes of platinum can be applied in various applications, especially for jewelry production. In the present article, submicron, and nano-sized platinum particle sizes were synthesized through simple chemical reduction methods and the effect of pH variation was revealed. The scanning electron microscope (SEM) images showed that the pH variations give rise to significant changes of the obtained particle size. The size of platinum particle was decreased from submicron to nanoscale while the pH was increased. The UV-Vis spectra indicated the maximum absorption at 220 nm confirming the spherical shape of the platinum particle. The FT-IR spectroscopy was used to analyze the residuals from the synthesis. The result showed that there is no indication of residual in the synthesized particle. Additionally, this synthesis can provide stability in terms of size and shape, as well as high production yield.

1. Introduction

Platinum is a noble metal largely applied in various applications i.e., a catalytic converter, dentistry equipment, platinum resistance thermometers, laboratory equipment, and jewelry. In jewelry manufacture, platinum is a problematic element in the production phase due to its high sintering and melting temperature. Nowadays, nanotechnology is a burgeoning field widely applied for the synthesis of platinum nanoparticles (Pt NPs) [1-2]. According to the size of a particle, the nanoparticle has a lower sintering and melting temperature than those of the microparticle. Moreover, both chemical and physical properties of Pt NPs were found to be productive and, in many cases, fascinating in the nanosized range. It can possess a wide range of properties that can be used for many practical applications, including the jewelry aspect. In recent years, articles on the synthesis of Pt NPs have been published and different features such as electrocatalyst, polymer membranes, cancer therapy, and coating have been discussed as well as the effect of synthesis parameter [1-2]. The size and shape of the synthesized particles have been affected by several processing parameters. Therefore, the purpose of this research is to synthesize platinum particle with the simplest chemical reduction and to control the obtained particle size by pH variation as well as to use it in further jewelry application. Moreover, this research concerns on developing this synthesis method for laboratory-scale to pilot-scale and to near the commercial scale. Percentage yield (%) was calculated to measure the efficiency of the chemical synthesis. The UV-Vis measurement was employed for investigating platinum nanoparticle in as-synthesized platinum solution. The particle sizes and surface morphology of the precipitated

platinum particles were determined via scanning electron microscope (SEM). In addition, Fourier transform infrared (FTIR) spectroscopy was used to identify the residuals that could be occurred from chemical contamination.

2. Experiment

2.1. Materials

Simple chemical synthesis was performed to synthesize the platinum particle. To reduce the cost of the synthesis, in this research, platinum metal plates were dissolved with aqua regia for using as the platinum reactant. The aqua regia solution was prepared by mixing of concentrated hydrochloric acid (12M, Merck) and concentrated nitric acid (15.8M, Merck) in the ratio 3:1, respectively. L-ascorbic acid, of 99% purity Merck, was used as reducing agent. Sodium hydroxide, of 97% purity Merck, was performed to adjust the pH. All chemicals were used without further purification and deionized water was used throughout the reactions.

2.2. Synthesis of platinum particles

For the simple synthesis of the platinum particles, a certain volume of 0.1 M ascorbic solution was slowly added to the 0.5%w/v platinum solution and then continuously stirred with a stirring speed of 400 rpm and a stirring time of 15 min. Then, the solution was adjusted to various pH (pH 1, pH 4, pH 7, pH 10 and pH 13) by adding 0.1 M sodium hydroxide, and the solution was continuous stirred for 8 hrs. With increasing the pH values, the color of the solution changed from yellow to brown which indicated the formation of the Pt NPs. After 8 hrs, the result revealed colloidal platinum mixing with some precipitated platinum particles. The amount of platinum precipitates depended on a variation of pH value. The precipitates were removed from the solution and repeatedly washed with distilled water three times. The products were dried at 80 ^{o}C for 4 hrs and the percentage yield was calculated afterward. The platinum powder was stored before the SEM and FTIR analysis, while the as-obtained colloidal solution was separated for determining with UV-Vis measurement.

2.3. Instrumentation and data acquisition

After completing of the synthesis, the as-obtained colloidal solution was monitored by Cary 60 Agilent UV–Vis spectrophotometer to determine the remaining platinum particle in the sample solution. The particle sizes and morphology of the precipitated platinum powder under different pH values were obtained using LEO 1450 VP scanning electron microscope. FTIR spectra were recorded on an Alpha II Bruker FTIR spectrometer to analyze the residual from the synthesis method. For the FTIR measurements of capped platinum particles, a small amount of dried platinum was mixed with potassium bromide (KBr) to form a round disk suitable for transmission sampling technique. The samples were investigated in the wavenumber of 500 to 4000 cm^{-1} and reference against air.

3. Results and discussion

3.1. Percentage yield

Since the purpose of this article is not only to study the pH effect on the particle size of platinum but also to attempt developing the synthesis method from laboratory-scale to pilot-scale, the percentage yield is a parameter that should be concerned. The relationship between the pH value and the yields of platinum powder precipitated from the different pH conditions is shown in figure 1. As seen in figure 1, the results showed that pH plays a very important role in the yield of platinum particles. When increase pH, the percentage yield tends to be increased.

3.2. SEM analysis

The average particle sizes obtained from every pH condition are shown in figure 2. The results revealed that the average sizes of platinum particle decreased when the pH value was increased. The SEM images of the as-prepared platinum powder precipitated by pH variation are shown in figure 3. Considering at the pH 1, the sample consists of large agglomerates of spherical particles in the range

of micron, average size about 2.15 microns. At the other pH conditions, the SEM images reveal submicron-sized to nano-sized platinum particle. The average sizes of the obtained particles at pH 4, pH 7, pH 10 and pH 13 were 490 nm, 466 nm, 372 nm, and 110 nm, respectively.

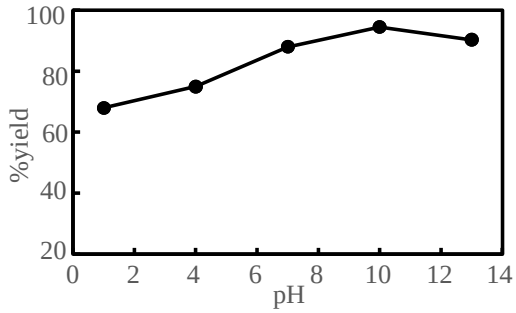

Figure 1. Relationship between increasing pH and percentage yield of precipitated platinum particle.

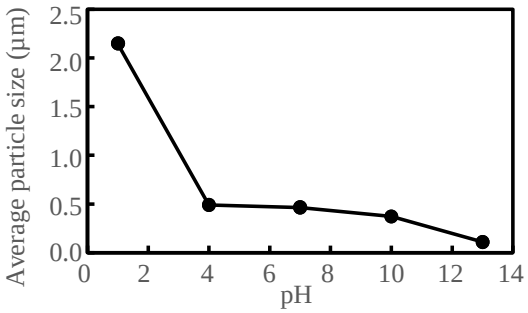

Figure 2. Relationship between increasing pH and particle size of precipitated platinum particles.

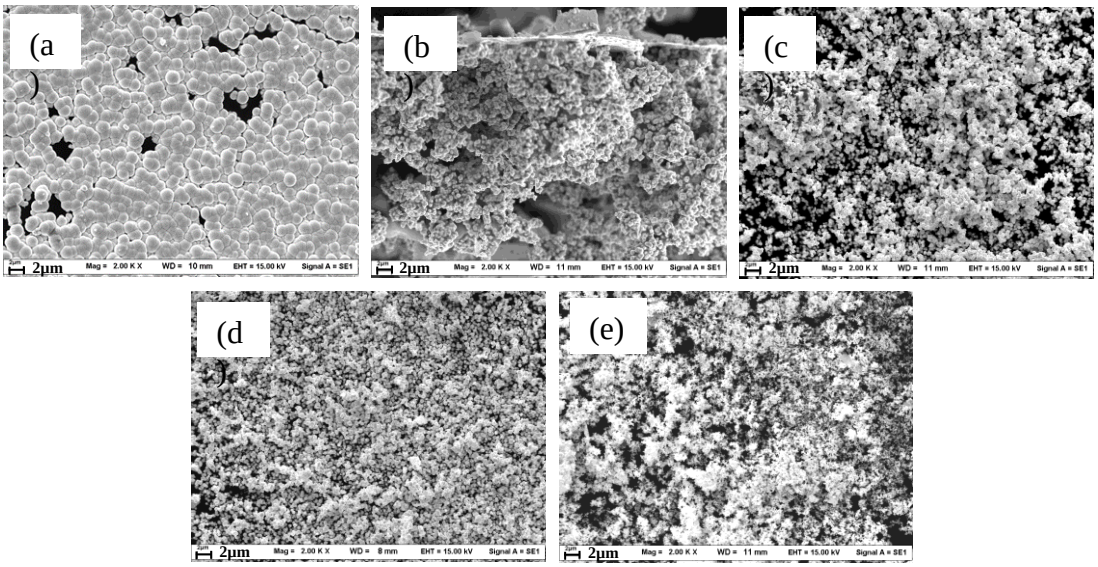

Figure 3. SEM images of precipitated platinum obtained from different pH values; (a) pH 1, (b) pH 4, (c) pH 7, (d) pH 10, and (e) pH 13.

3.3. UV-Vis spectra analysis

The UV-Vis spectra of as-obtained platinum colloids from different pH are presented in figure 4. Considering the spectrum of pH 1, the spectrum shows the absorption bands at 240 and 267 nm corresponding to $PtCl_4^{2-}$ and $PtCl_6^{2-}$ that remaining in solution [3-4]. Therefore, the results indicated that the reaction rate is very slow and the formation rate of Pt^0 is low. For this reason, a low percentage yield of platinum precipitation was achieved. Moreover, the size of platinum particles obtained from this condition is micro-sized particle which can be confirmed from the figure 3 (a). When the pH was increased, the band at 267 nm disappeared and the color of the colloidal solution changed. It can be assumed that the $PtCl_6^{2-}$ was reduced to $PtCl_4^{2-}$, and the formation of Pt^0 tend to completely be performed. In addition, the surface plasmon resonance peak shifts toward the short wavelength region as well as becomes narrower. This blue shift indicates a reduction in the mean diameter of the particles [5-6]. It can be implied that the particle size of the precipitated platinum particle is reduced to a

smaller one as a nano-sized platinum particle and the percentage yield of high pH tends to be increased as well. Additionally, the UV-Vis spectra indicate the maximum absorption at 220 nm confirming the spherical shape of the platinum particle [3-5].

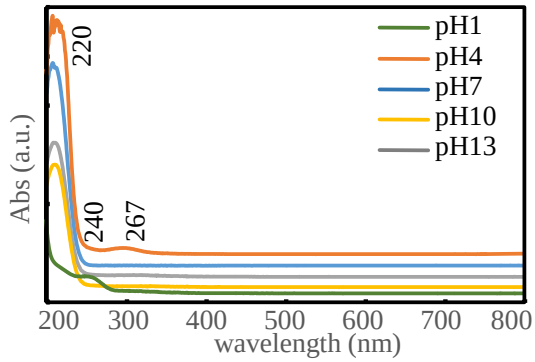

Figure 4. UV-Vis spectra of synthesized platinum obtained from different pH values.

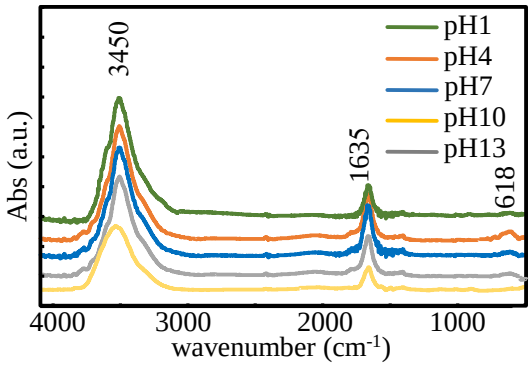

Figure 5. FTIR spectra of precipitated platinum obtained from different pH values.

3.4. FTIR analysis

Figure 5 displays the FTIR spectra of the dried platinum powder according to variation of pH values. All spectra present absorption bands at 3450, 1635, and 618 cm^{-1}. The bands relate to OH stretching, OH bending, and OH out of plane bending of absorbed water interference in KBr. The obtained spectra do not reveal any absorption bands corresponding to the other contaminants from the synthesis. It can be assumed that there is no residual in synthesized platinum powder.

4. Conclusion

In this research, the impact of pH variation on the simple chemical synthesis of the platinum particle was investigated. The results affirmed that the different pH affecting to the percentage yield and the size of platinum particle. By increasing pH value, the percentage yield of precipitated platinum was increased, and the particle size was decreased from submicron to the nano-sized, as seen in the SEM images. The UV-Vis spectra of prepared samples showed characteristic properties of Pt NPs and platinum ion remaining in colloid solution. The FTIR spectra confirmed that there is no indication of residual contamination in the synthesized particles. Therefore, this simple synthesis attributing to pH control can produce the purity platinum powder with high production yield. It can be applied for further jewelry application and other industries.

Acknowledgments

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