Preparation of Silica-Gold Composite particles using impregnation method

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함침법을 이용한 실리카-골드 복합체의 제조

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요 약

Silica-gold composite particles were prepared by wet chemical route including impregnation method. Obtained composite particles were characterized using FE-SEM, and XPD.

1. Introduction

Recently, a great deal of attention has been devoted to design and preparation of composite particles with metallic layer because of their applications in various field such as surface-enhanced raman scattering (SERS) [1], photonic crystals [2, 3], catalysis [4] and biochemistry for potential uses as chemical sensors [5], and so on.

In addition, these hybrid materials have outstanding characteristics that distinguish them from conventional bulk and nanophase materials. One of the important problems in most applications of nanoparticles is the aggregation of individual nanoparticles. In powder state, nanoparticles form micron-sized secondary particles adhering to each other during preparation, storage and application. If this occurs, the properties of the materials may be determined by the size of the secondary particles, not by the individual particles. In colloid state, individual nanoparticles can be maintained well-dispersed state by steric hindrance effect or electrostatic repulsion via precise control of the chemical composition and physical properties of the solution. However, well-dispersed colloidal nanoparticles also can easily lose their stability by unexpected variation of solution state during storage and application. These problems can be greatly improved by immobilization of nanoparticles onto the support materials. Moreover, these nanoparticles are easily retrieved owing to the relatively large size of supports. As a result, they make it easy to handle metal nanoparticles, which can decrease the cost of process and prevent the potential environmental pollution caused by undesired spread of the metal nanoparticles.

Gold is famous for its anti-aging effect due to their promotion of blood circulation. In this reason, many cosmetic companies have tried to apply gold as an active ingredient for cosmetics. However, the high cost and is a serious limitation of enlargement of gold incorporated cosmetics. Silica-gold composite material can be a strong candidate to overcome these limitations due to their unique characteristic.

Recently, novel preparation routes for composite materials have been proposed to simplify the conventional complex reaction process or to obtain

dense and uniform metal layers: sono-chemical deposition [6], electroless plating [7], electrostatic attraction [8] techniques and so on. However these preparation routes have a quite complex process and resulting materials are too expensive to use as a cosmetic raw materials

In this study, we investigated simple and economic preparation route based on impregnation method to make silica-gold composite particles with high yield and economical price. The obtained Silica-gold composite particles were characterized through FE-SEM and XRD.

2. Experimental

2.1 Materials

Hydrogentetrachloroaurate(Ⅲ) hydrate($HAuCl_4 \cdot nH_2O$,n=3.7,Kojima Chemicals) and polyvinylpyrrolidone (PVP, K-15, Mw10,000, Junsei Chemical Company ,Japan) were purchased and used. Silica particles were suggested and provided by Quick&Safe. All materials were used as received.

The water used in this study was deionized by Milli-Q Plus system(Millipore, France), having18.2MΩ electrical resistivity.

2.2. Experimental Procedure

In typical process, support materials, silica particles, were added into reactants solutions. And then resulting silica particles were centrifuged or dried. Finally, dried silica particles were calcinated in furnace at 500 ℃ - 700 ℃ in 2hours. Experimental conditions are listed in table 1.

[Table 1] Experimental conditions

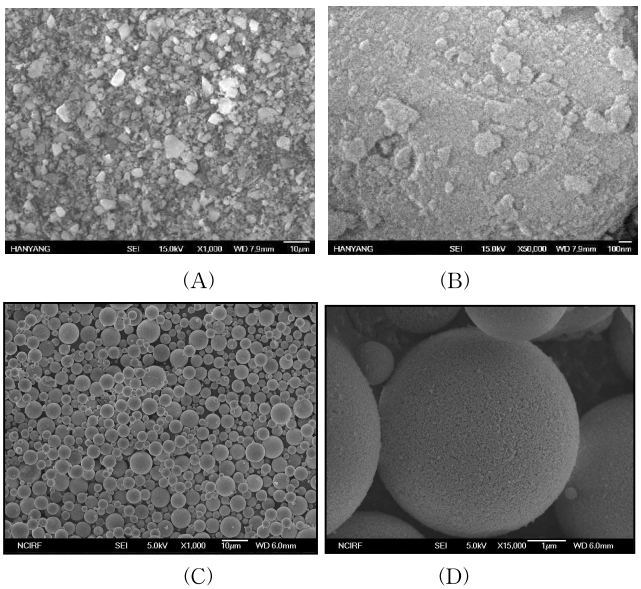
	Silica	Solvent	Precursor
No 1.	S type		
No 2.	B type	Water	Gold ions
No 3.	B type	Water	Gold ions, PVP

2.3. Characterization

To investigate formation and morphological properties of composite particles, field emission scanning electron microscopy (FE-SEM, JEOL JSM-6700F) was used. To verify the formation of gold, X-ray diffractometry measurement was performed with a Rigaku D/MAX-3C x-ray diffractometer. The incident wavelength was Cu Kα1 = 1.789Å, and the detector moved step by step (△2Θ = 0.05°)between10° and 90° 2Θ. The scan speed was 2°/minute.

3. Results and discussion

The morphology of the two kind of silica particles were investigated and compared using FE-SEM as shown in fig. 1.



(A) (B) (C) (D)

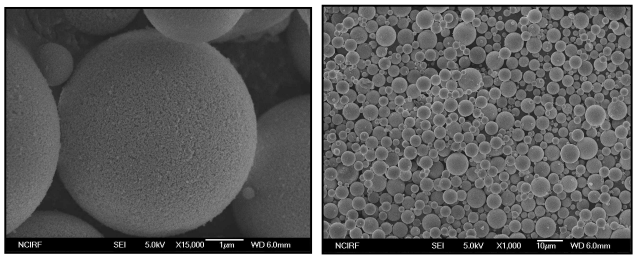
[Fig. 1] FE-SEM photographs: (A) S type silica particles (X1,000 magnification), (B) S type (X50,000 magnification), (C) B type silica particles (X1,000 magnification) and (D) B type silica particles (X15,000 magnification)

S type silica particles showed very rough and sharp edged shapes as shown in fig. 1(A) and (B). On the contrary, B type silica particles consisted of almost perfect sphere shaped individual particles. Average diameter of B type silica particles is determined about 6μm. B type particles are better than S type particles with respect to cosmetic applications because spherical particles with 6μm average diameter scatter light on the surface of its particles, i. e. which are transparent, but give natural coverage of skin and silky feeling.

To investigate the effect of precursor on the

formation of silica-gold composite particles, two different precursor conditions were examined: gold ions(No2 in table 1) and gold-PVP complexes(No3 in table 1). The concentration of gold in composite particles fixed to 1.0%.

Compare with hydrogentetrachloroaurate(Ⅲ) hydrate aqueous solution, PVP and hydrogentetrachloroaurate(Ⅲ) hydrate aqueous solution was used, obtained silica-gold composite particles showed better color reproducibility. Fig. 2. show the silica-gold composite particles obtained using PVP and hydrogentetrachloroaurate(Ⅲ) hydrate aqueous solution as a precursor.

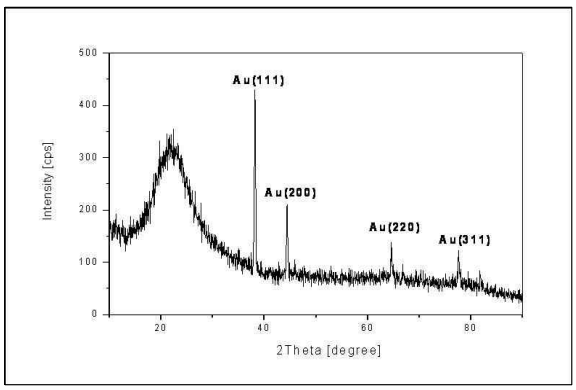


(A) (B)

[Fig. 2] FE-SEM photographs of silica-gold particles (A) X15,000 magnification and (B) X1,000 magnification

Despite of composite formation, silica particles maintained the spherical shape.

To verify the formation of silica-gold composite particles, resulting powder was examined using XRD as shown in fig. 3.



[Fig. 3] XPD patterns of silica-gold particles

Fig. 3 verifies the amorphous silica-gold metal composite particle formation.

4. Conclusions

Silica-gold composite particles were prepared by impregnation method. When spherical silica particles and PVP and hydrogentetrachloroaurate (Ⅲ) hydrate aqueous solution were used as support material and precursor solution, silica-gold composite particles with light pink color successfully obtained.

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