Production and Characterization of Porous Gold

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Abstract

This work has presented an attempt at the production and characterization of porous gold by different procedures. The porousity was created as a result of mechanical attrition on the gold ore particles followed by acid leaching, carbon–in-leach gold extraction and carbon–in- leach gold extraction followed by double acid leaching and refining at 650°C. The porous gold samples obtained were characterized using Scanning Electron Microscope (SEM) and X-Ray Diffractometer (XRD).

The result showed that the carbon–in- leach gold extraction procedure followed by double acid leaching and refining at 650°C was the most promising.

Keywords: *Gold, porous gold, carbon-in- leach, mechanical attrition.*

Introduction

Specific synthesis of nanoparticles and nanostructured materials are attracting attention in recent research because of their valuable properties which make them useful for catalysis [Narayanan and ElSayed,, 2004], sensor technology [Gomez-Romero, 2001], biological labeling [Shankar et al., 2003], optoelectronics recording media and optics [Qiu et al. 2004]. The size, shape and surface morphology play pivotal roles in controlling the physical, chemical, optical and electronic properties of these nanoscopic materials [Gracias et al., 2002; Kamat, 2002]. This is particularly important for noble metals such as gold (Au) and silver (Ag) which have strong surface plasmon resonance (SPR) oscillations. The shape-selective metal nanoparticles such as rods, tubes, wires, triangles, prisms, hexagons and cubes can be regularly synthesized by chemical, biological and physical methods. [El-Sayed, 2001; Lim et al., 2008]

Many colloidal methods of synthesis have been approached to obtain metallic nanoparticles for this purpose, such as homogeneous reduction in aqueous solution [Shankar et al., 2004], or phase transfer reactions [Liz-Marzan & Philipse, 1995], with sodium citrate, hydrazine, $NaBH_4$, and lithium triethylborohydride ($LiBEt_3H$) as reducing agents, each of them yielding products with different physicochemical and structural characteristics [Han et al., 1998]. Among these, the polyol method has been reported to produce small nanoparticles as the final product, easily changing composition and surface modifiers. This technique does not require an additional reducing agent since the solvent by itself reduces the metallic species.

However, besides the stoichiometry and order of addition of reagents in the synthesis process, one of the most important parameters in the preparation is the temperature. Modifications in temperature influence the reaction by changing the stabilization of the nanoparticles formed and the surface modifiers, e.g., PVP, and the nucleation rate of the reduced metallic atoms [Schmid, 1994].

Gold (Au) and silver (Ag) nanoparticles have a diversity of interesting properties between which they emphasize the electrical ones, optical, catalytic and the applications in biomedicine like antibacterial and antiviral, same that depend on their morphology and size.

Characterization of these systems has been a difficult process where researchers have employed indirect measurements to identify the localization of the elements within the nanoparticles. A novel approach to study this kind of particles is based on the use of a High Angle Annular Dark Field (HAADF) Technique, in a Transmission Electron Microscope (TEM), which allows the observation of the elements due to atomic number, densities, or the presence of strain fields due to differences in lattice parameters, structure, the presence of surfactants or any other surface modifier besides the size of the particle and also by Near-Field Scanning Optical Microscopy (NSOM), we determine the size of the particles [Henglein, 2000; Turkevich et al., 1951].

This research has investigated the production of porous gold by different procedures and subsequent characterization.

Experimental

The production of gold nanoparticles was achieved using the top down technique through mechanical attrition (ball milling) of the physically processed gold ore. The gold ore was weighed and physically processed by hand picking the gangues and other physical impurities. It was then fractionated into different sizes with the aid of sieves of sizes ranging from 0.1mm and 3.5 mm. The physically processed gold ore was pulverized in a table top ball mill for 12 hours continuously in order to reduce the size of the gold to nanoscale. The sample was added to 10 ml HNO_3 so as to remove more of the impurities. The acid was decanted and 100 ml de-ionized water was added to the mixture twice and then decanted in order to wash it to neutrality. The residue was dried in the oven for one hour at 150°C. The sample was then characterized.

In the second beaker, the physically processed gold ore was finely ground and slurried with cyanide solution from analar grade sodium cyanide in a single tank so that leaching and recovery can be taking place concurrently (carbon – in – leach). The resulting gold cyanide complex was adsorbed onto the activated carbon. The samples was divided into two, one part was used for the third approach below. To the second part, zinc granules were added to the pregnant activated carbon in order to recover the gold by precipitation. The precipitated gold was washed to neutrality and subsequently characterized.

In the third beaker, the carbon –in- leach was then washed with concentrated HCI so as to remove iron which may co-precipitates with gold. Washing with HCI is an effective method for the removal of iron associated with gold [Bello, 1997]. Iron does dissolve and complex with cyanide solution along with gold. Further refining was carried out by adding 10 ml HF to the mixture and left for one hour in a fume cupboard at 50°C. The acid was decanted and 100 ml de-ionised water was added to the mixture

twice and then decanted in order to wash it to neutrality. The residue was dried in the muffle furnace for one hour at 650°C. The recovered gold was characterized.

An X-ray diffractometer (XRD) (X'Pert Pro X-ray diffraction system Panalytical) was used to investigate the crystal structure of the samples. The samples were ground and pressed into the sample holder to get a smooth plane surface, and the diffraction pattern was recorded over a 2θ range of 30°-120°. The diffractogram obtained was compared to the standard database of the International Centre for Diffraction Data (ICDD).

The morphological features of the samples were studied with a scanning electron microscope (SEM) JEOL Model JSM-7600F.

Result and Discussion

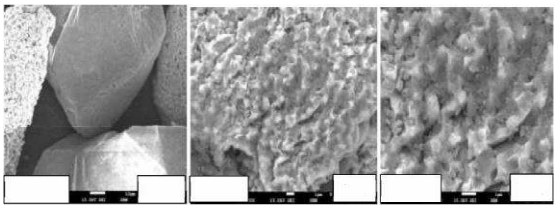

Figure 1: SEM images of porous gold obtained from mechanical attrition on the physically processed gold ore particles followed by acid leaching.

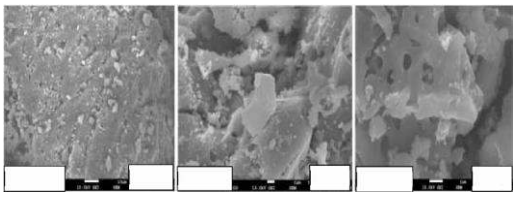

Figure 2: SEM images of porous gold obtained from Carbon–in-leach Gold Extraction (Gold ore slurry and activated carbon)

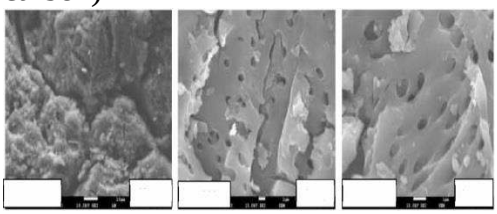

Figure 3: SEM images of porous gold obtained from Carbon–in-leach Gold Extraction followed by double acid leaching and refining at 650°C.

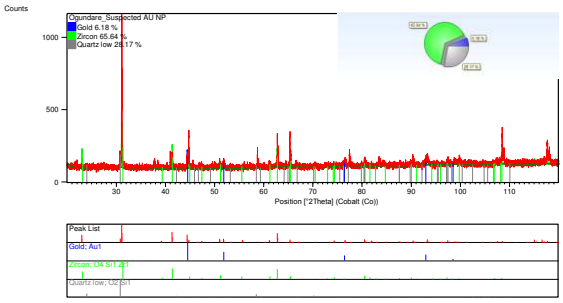

Figure 4: XRD of porous gold obtained by Mechanical Attrition followed by acid leaching

Figure 1 shows the SEM images obtained from mechanical attrition on the physically processed gold ore particles followed by acid leaching. The physically processed gold ore was pulverized in a table top ball mill for 1 2hours continuously in order to reduce the size of the gold to nanoscale. It can be observed from the SEM images that the pores created on each gold grain were more conspicuous as the magnification increased.

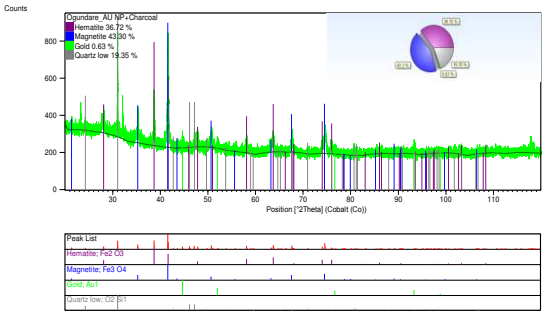

Figure 5: XRD of porous gold obtained from Carbon–in-leach Gold Extraction

The pore formation could be as a result of the chemical leaching effect on the gold nanoparticles. The characteristic pattern produced on the morphology of the gold nanoparticle was unique. Figure 2 represents the SEM images obtained from carbon–in-leach gold extraction procedure. It can be observed that as the magnification increased, the number of pores formed became more distinct and increased. This porousity could be due to the complex interaction between the gold ore and the activated carbon leading to physisorption. This phenomenon could bring about pore formation on the morphology of the gold grains.

Figure 3 shows the SEM images obtained from carbon–in-leach gold extraction followed by double acid leaching and refining at 650°C. The shape and size of the pores formed can be observed to be the most pronounced out of all the procedures used. The number of pores on each gold grain is observed to be the greatest. This could be as a result of the synergetic combination of double leaching of the gold ore and firing at an elevated temperature.

Figure 4 shows the XRD of nanoporous gold obtained by mechanical attrition followed by acid leaching. The diffraction pattern revealed that the only crystalline phases identifiable using Rietveld method are the gold nanoparticles, zircon and quartz. The quantitative analysis revealed that gold was about 6%, zircon was about 65% and quartz was about 29%. The diffraction peak for gold was shown at $2\theta = 45°$.

Figure 5 represents the XRD of porous gold obtained from carbon–in-leach gold extraction. The diffraction pattern showed that the only crystalline phases present using Rietveld method are the heamatite, magnetite, gold particles and quartz. The quantitative analysis revealed that heamatite was about 37%, magnetite was about 43%, gold was less 1% and quartz was about 19%. The undesirable presence of iron (in form of heamatite and magnetite) associated in the gold ore has been confirmed by the quantitative analysis. Iron has been known to co-precipitate with gold during carbon – in – leach procedure. Washing with HCI is an effective method for the removal of iron associated with gold [Bello, 1997].

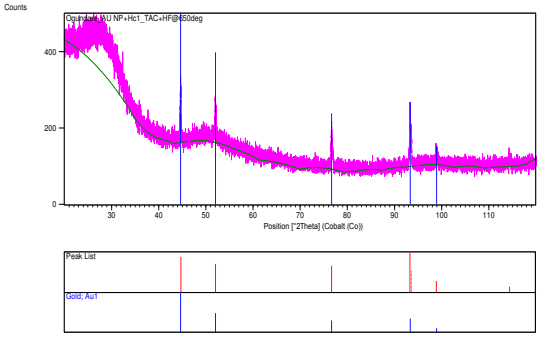

Figure 6: XRD of porous gold obtained from Carbon–in-leach Gold Extraction followed by double acid leaching and refining at 650°C.

Figure 6 reports the XRD of the porous gold obtained from carbon–in-leach gold extraction followed by double acid leaching and refining at 650°C. The diffraction pattern revealed that the only crystalline phase identifiable using Rietveld method is the gold. Any form of iron co-precipitated has been taken care of by the double acid leaching procedure. Further refining was also achieved by firing at 650°C as a result of oxidation and vapourization.

Conclusion

1. The carbon–in- leach gold extraction procedure followed by double acid leaching and refining at 650°C was the most promising for applications

in catalysis, sensor technology, biological labeling, optoelectronics recording media and optics.

2. There was in-situ diffusion zone created that facilitated the pores formation. This porousity could be due to the complex interaction between the gold ore and the activated carbon leading to physisorption.

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References

Narayanan, R.; EI-Sayed, M.A. Nano Letters 2004, 4, pp. 1343–1348

Gomez-Romero, P., Advanced Materials, 2001, 13, pp. 163–174.

Shankar, S. S.; Ahmad, A.; Pasricha, R.; Sastry, M.; Journal of Materials Chemistry 2003, 13, pp. 1822–1826.

Qiu, H.; Rieger, B; Gilbert, R.; Jerome, C.; Chemical Materials 2004, 16, pp. 850–856.

Gracias, D.H.; Tien, J.; Breen, T.; Hsu, C.; Whitesides, G. M.; Science, 2002, 289, pp. 1170–1172.

Kamat, P.V.; Journal of Physical Chemistry B 2002, 106, pp.7729–7744.

EI-Sayed, M.A.; Acc Chemical Resource, 2001, 34, pp. 257–264

Lim, J.K.; Kim, Y.; Lee, S.Y.; Joo, S.W.; 2008, Spectrochim Acta A, 69, pp. 286–289.

Shankar, S. S.; Rai, A.; Ankamwar, B.; Singh, A.; Ahmad, A.; Sastry, M.; 2004, Nature Materials 3, pp. 482–488 Liz-Marzan, L. M.; Philipse, A. P. Journal of Physical Chemistry 1995, 99, pp. 15120. Schmid, G. Clusters and Colloids, From theory to Applications; VCH Publishers: Weinheim, Germany, 1994.

Henglein, A. Journal of Physical Chemistry B 2000, 104, pp. 2201

Turkevich, J.; Stevenson, P.; Hillier, J. Discuss. Faraday Society 1951, 11, pp. 55. Bello, A. A. Extraction of gold from Ilesa gold ore deposit. M.Sc. Thesis (Unpublished). Department of Chemistry, University of I1orin 1997.