

## Production and Characterization of Gold Nanoparticles from Itagunmodi Gold Deposit

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### Abstract

*This paper has reported the production and characterization of gold nanoparticles from Itagunmodi gold deposit in Atakumosa West LGA in the State of Osun Nigeria. The gold ore was weighed and physically processed by hand picking the gangues and other physical impurities. The production of gold nanoparticles was carried out using the top down technique through mechanical attrition (ball milling) and thin film deposition. The gold nanoparticles obtained were subsequently characterized using Scanning Electron Microscopy (SEM) with Energy dispersive-X ray (EDX) attachment and X-Ray Diffraction (XRD). The morphology of the ball milled nanoparticles of gold from Itagunmodi deposit revealed oval and trihedral shapes. The distribution of the particle size in the ball milled gold nanoparticles revealed that they are inhomogeneous and that more of the particles size falls within 30 nm. The average particle size was evaluated statistically to be 32 nm. The particle size distribution for the gold thin film falls within 6 nm. The average particle size of the gold thin film was evaluated statistically to be 14 nm. The particle size distribution of the thermally deposited gold film revealed a better homogeneity.*

**Keywords:** Gold nanoparticles, gold, characterization, morphology

### 1. INTRODUCTION

Gold nanoparticles are the most stable metal nanoparticles; during the past decades colloidal gold nanoparticles have received significant research attention, both due to their unique physical and chemical properties, and promising applications (Tréguer-Delapierre *et al*, 2008; Lopez- Sanchez *et al*, 2011; Sadowsk, 2010). Synthesis of gold nanoparticles and using

as nanostructure materials become an exciting area of interdisciplinary research various techniques have been developed to synthesis colloidal gold nanoparticles. The recent interest of gold nanoparticles is propelled by both the advancement in our scientific understanding of their synthesis and physical properties as well as their possibilities of potential applications in the field of chemical and biological sensing (Kim *et al*, 2001; Kurniawan, 2008; Sobczak-Kupiec,

2011), cancer treatment (Narayanan, 2010), catalysts (Cai *et al.*, 2008; Cortie and Lingen, 2002; Kisailuset *et al.*, 2005), drug delivery (Fu *et al.*, 2005), electronics and optoelectronic devices (Huang *et al.*, 2003), colloidal dispersion of gold particles with small diameter appear ruby red color while with larger diameter appear bluish, this difference in color is due to larger particle size and when light interact with matter its optical properties change i.e. the size dependence color of colloidal gold is simply a consequence of how light interact with matter (Xiaohuahuanget *al.*, 2007). Optical properties and applications of these nanoparticles have been highlighted in many reviews and other research works (Marie-Christine and Didier, 2004; Colleen *et al.*, 2006; Nguyen *et al.*, 2009; Allen *et al.*, 2000). Colloidal gold nanoparticles are generally produced by the reduction of gold salt such as auric tetrachloride (HAuCl<sub>4</sub>) in an appropriate solvent, usually stabilizing agent is also added to prevent the particles from agglomeration, most frequently thiols modified ligands are used as a stabilizing agent for the formation of gold nanoparticles (AuNPs) which bind to the surface and form Au-sulfur bond (Ma *et al.*, 2004).

This work intends to produce and characterize gold nanoparticles from gold ore mined from Itaganmodi gold deposit. Due to negligible quantity of sieve fractions 106  $\mu\text{m}$  and 75  $\mu\text{m}$  obtained, the sieve fraction of 125  $\mu\text{m}$  was chosen as a working fraction for this research.

## 1. METHOD

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 production of gold nanoparticles was achieved using the top down technique through mechanical attrition (ball milling) of the gold ore. The gold ore was continuously pulverized for

12 hours in a table top ball mill in order to reduce the size of the gold to nanoscale. In another method, the processed gold ore was thermally deposited on a substrate as a thin film at the nanoscale level using vacuum thermal depositor.

An X-ray diffractometer (XRD) (X'Pert Pro X-ray diffraction system Panalytical) was used to investigate the crystal structure of the gold nanoparticles obtained through mechanical attrition.

The sample was ground and pressed into the sample holder to get a smooth plane surface, and the diffraction pattern was recorded over a  $2\theta$  range of  $30^\circ$ - $120^\circ$ . The diffractogram obtained was compared to the standard database of the International Centre for Diffraction Data (ICDD). The morphological features of the gold nanoparticles were studied with a scanning electron microscope (SEM) JEOL Model JSM-7600F.

## 2. RESULTS AND DISCUSSION

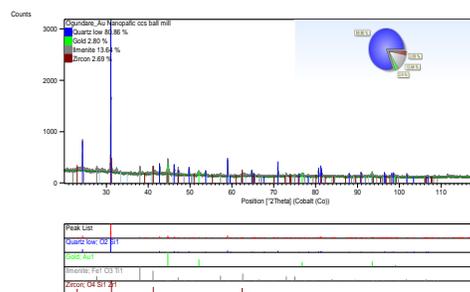


Figure 1: XRD pattern of gold nanoparticles produced by mechanical attrition (ball milling)

The XRD of the gold nanoparticles showed a lot of diffraction peaks with numerous background noise. This could be due to presence of numerous mineral oxides in the gold ore (Figure 1). It also indicates the amorphous nature of the associated minerals. The relative phase amounts (weight %) estimated using the Rietveld method showed the content of the gold

nanoparticles to be made up of quartz (80.86 %), gold (2.8 %), ilmenite (13.64 %) and zircon (2.69 %). The small quantity of gold present in the sample is now being confirmed by the XRD and was below detection by the EDX attached to the SEM. Hence, the peaks for the gold nanoparticles are almost invisible. However, a sharp peak for the crystalline phase of the gold was observed at  $2\theta = 32^\circ$ .

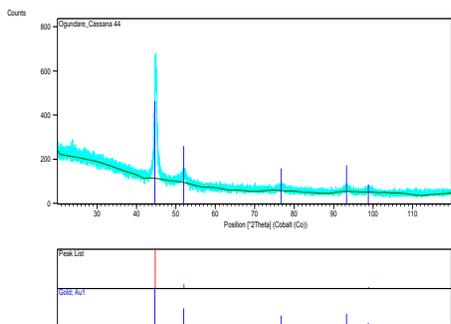


Figure 2: XRD pattern of gold nanoparticles produced by thin film deposition

Figure 2 above shows the typical XRD pattern of gold nanoparticles produced by thin film deposition. The XRD showed a broad peak at  $2\theta = 45^\circ$ . Analysis of the patterns revealed that the only crystalline phase present is the gold phase which is of small crystalline size. The thickness of the film deposition, as shown by the profilometry scan in Figure 6, could be responsible for the seeming presence of the background noise found on the diffractograms.

The SEM images of gold nanoparticles obtained from ball milling (mechanical attrition) of the gold ore at different magnifications are shown in Plate 1 (a-e) above. The morphology of the nanoparticles of gold revealed oval and trihedral shapes. The distribution of the particle size in the gold nanoparticles revealed that they are inhomogeneous and that more of the particles size falls within 30 nm (figure 2) at x100

magnification. The average particle size was evaluated statistically to be 32 nm.

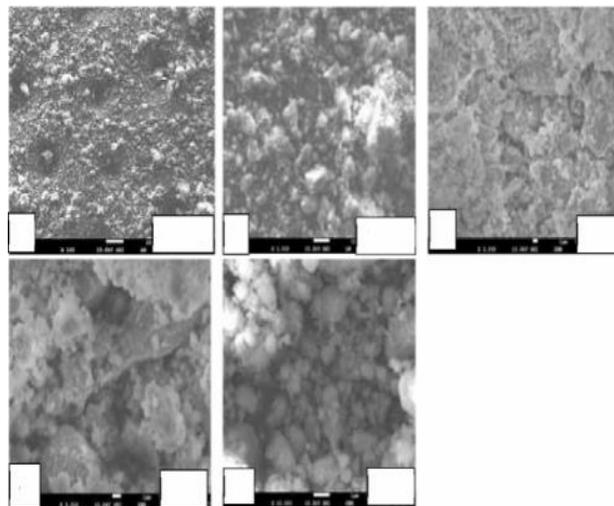


Plate 1: SEM images of gold nanoparticles obtained by Mechanical Attrition (a) (x100), (b) (x1, 000), (c) (x3, 000), (d) (x5, 000) and (e) (x10, 000)

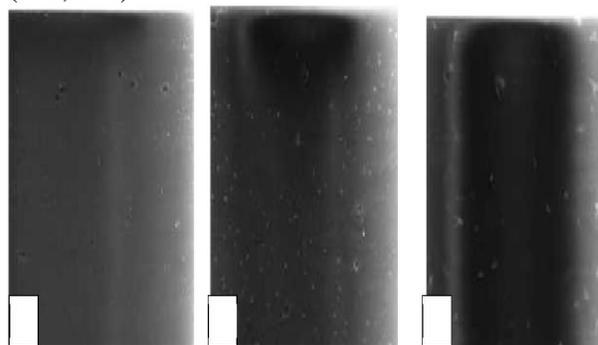


Plate 2: SEM images of gold nanoparticles obtained by thin film deposition (a) 500x, (b) 1000x, (c) 2500x

The SEM images of gold nanoparticles obtained from thin film deposition of the processed gold ore at different magnifications are shown in Plate 2 (a-c) above. The distribution of the particle size in the gold ore revealed that they are inhomogeneous and that more of the particles size falls within 30  $\mu\text{m}$  (figure 3).

The morphology of the gold thin film revealed oval shapes. The distribution of the particle size in the gold thin film revealed that they are homogeneous and that more of the particles size falls within 6 nm (see figure 4). The average particle size was evaluated statistically to be 14 nm. This result is in consonance with works of Yamamoto and Nakamoto, (2003); Henglein and Meisel, (1998); Dawson and Kamat, (2000); Mallick et al., 2001; Sau et al., 2001 that all reported narrow particle size

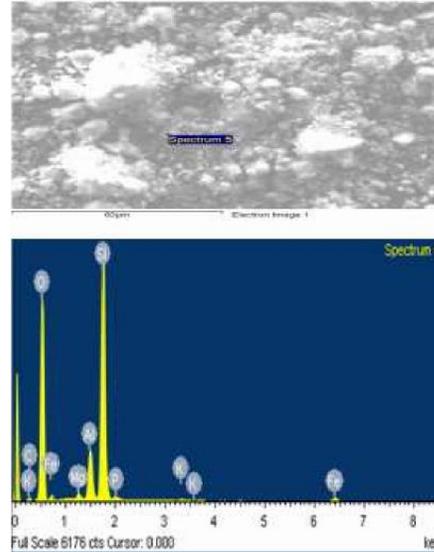


Figure 4: Particle Size Distribution of gold nanoparticles obtained by gold thin film deposition

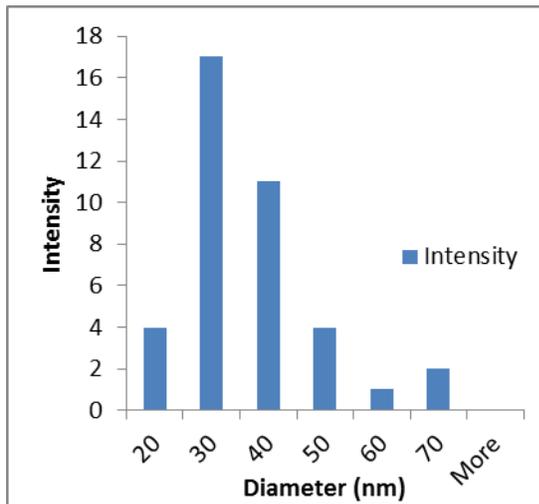
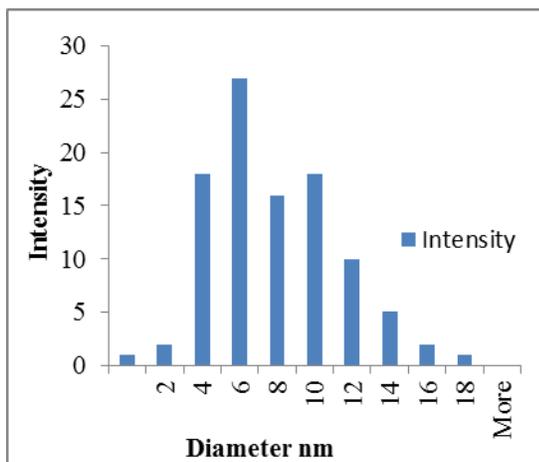


Figure 3: Particle Size Distribution of gold nanoparticles obtained by mechanical attrition



distribution of gold nanoparticles in the gold ore. The amount of gold present was negligible to be detected by the instrument. This observation is not surprising because Itagunmodi gold deposit has been classified among the low-grade ore since it is of alluvial placer origin (USEPA, 1994). The accompanying spectrum confirmed the presence of silicious and oxide minerals in the gold ore.

Element	Weight% Atomic%	
C K	3.94	6.32
O K	55.67	67.07
Mg K	0.66	0.52
Al K	5.22	3.73
Si K	30.14	20.68
P K	0.36	0.22
K K	0.43	0.21
Fe K	3.59	1.24
Totals	100.00	

### 3. CONCLUSION

- Gold nanoparticles have been successfully produced by mechanical attrition and thermal deposition of gold particles extracted from Itaganmodi deposit.
- The morphology of the nanoparticles of gold from Itaganmodi deposit revealed oval and trihedral shapes.
- The distribution of the particle size in the gold nanoparticles revealed that they are inhomogeneous and that more of the particles size falls within 30 nm at x100 magnification. The average particle size was evaluated statistically to be 32 nm.
- The particle size distribution for the gold thin film falls within 6 nm. The average particle size of the gold thin film was evaluated statistically to be 14 nm.
- The particle size distribution of the thermally deposited gold film produced a better homogeneity than the ball milled approach.

### 4. ACKNOWLEDGEMENTS

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### 5. REFERENCES

Tréguer-Delapierre V, Majimel J, Mornet S, Duguet E, Ravaine S. (2008), "Synthesis of non-spherical gold nanoparticles", Gold Bulletin, Pessac, France.

Lopez-Sanchez, Jose A, Dimitratos, Nikolaos, Hammond, Ceri, Brett, Gemma L, LokeshKesavan, White Saul, Miedziak Peter, TiruvalamRamchandra, Jenkins Robert L., CarleyAlbert F., Knight David, Kiely Christopher J. & Hutchings Graham J.,( 2011), "Facile removal of stabilizer ligands from supported gold nanoparticles", Nature Chemistry 3, 551–556.

SadowskiZygmunt,(2010), "Biosynthesis and application of silver and gold nanoparticles",Wroclaw University of Technology, Poland In Tech.

Kim Youngjin, Johnson Robert C, and Hupp Joseph T, (2001), "Gold Nanoparticle-Based Sensing of "Spectroscopically Silent" Heavy Metal Ions", American Chemical Society, Vol. 1.

KurniawanFredy, "New Analytical Applications of Gold Nanoparticles",(2008) PhD thesis, Faculty of Chemistry and Pharmacy, University of Regensburg, Germany.

Sobczak-KupiecAgnieszka, MalinaDagmara, ZimowskaMalgorzata, WzorekZbigniew

(2011), “Characterization of Gold Nanoparticles for Various Medical Application”, Digest Journal of Nanomaterials and Biostructures, Vol. 6, No 2, p. 803 – 808.

Narayanan Radha (2010), “Recent Advances in Noble Metal Nanocatalysts for Suzuki and Heck Cross-Coupling Reactions”, Molecules, ISSN 1420–3049.

Cai Weibo, Gao Ting, Hong Hao, Sun Jiangtao (2008), “Applications of gold nanoparticles in cancer nanotechnology”, Nanotechnology, Science and Applications.

Cortie M.B. and Lingen E. van der (2002), “Catalytic Gold Nano-Particles”, Materials Forum Vol. 26, pp.1-14.

Kisailus David, Najarina Mark, Weaver James C. and Morse Daniel E,(2005) ‘Functional Gold Nanoparticles Mimics Catalyst Activity of a Polysiloxane Synthesizing Enzyme’, Advanced Materials, Vol. 17 pp.34-1239.

Fu Wei, Shenoy Dinesh, Li Jane, Crasto Curtis, Jones Graham, Dimarzio Charles, Sridhar Srinivas, and Amiji Mansoor, (2005) “Biomedical Applications of Gold Nanoparticles Functionalized Using Hetero-Bifunctional Poly (ethylene glycol) Spacer”, Materials Research Society, Vol. 845.

Huang Daniel, Liao Frank, Molesa Steven, Redinger David, Subramanian band Vivek (2003), “Plastic-Compatible Low Resistance Printable Gold Nanoparticles Conductors for Flexible Electronics”, Journal of The Electrochemical Society, G412-G417.

Sharma Vivek, Kyoungweon Park, Srinivasarao Mohan,(2009) “Colloidal dispersion of gold nanorods: Historical background, optical

properties, seed-mediated synthesis, shape separation and self-assembly”, Elsevier.

Xiaohu Huang, Prashant k jain, Ivan H El-Sayed and Mostafa A El Sayed (2007), “Gold nanoparticles: interesting optical properties and recent applications in cancer diagnostics and therapy”, nanomedicine, Vol. 2 No 5 pp. 681 - 693.

Marie-Christine Daniel and Didier Astruc, (2004) “Gold Nanoparticles: Assembly, Supramolecular Chemistry, Quantum-Size-Related Properties, and Applications toward Biology, Catalysis and Nanotechnology”, American Chemical Society, Chemical Revolution, Vol. 104 pp. 293-346.

Colleen L. Nehl, Hongwei Liao. and Jason H. Hafner, (2006) “Optical Properties of Star-Shaped Gold Nanoparticles”, American Chemical Society, Nano Letter, Vol. 6 No 4, pp. 683–688.

Nguyen Ngoc Long, Le Van Vu, Chu Dinh Kiem, Sai Cong Doanh, Cao ThiNguyet, Pham ThiHang, Nguyen DuyThien, LuuManhQuynh (2009), “Synthesis and optical properties of colloidal gold nanoparticles”, Journal of Physics: Conference Series 187.

Allen C. Templeton, W. Peter Wuelfing, and Royce W. Murray,(2000) “Monolayer-Protected Cluster Molecules”, American Chemical Society, Vol. 33, No. 1.

Ma H, Yin B, Wang S, Jiao Y, Pan W, Huang S, Chen S, Meng F.(2004), “Synthesis of silver and gold nanoparticles by a novel electrochemical method”, Chemphyschemistry. Vol. 5 No 1 pp: 68-75.

