Synthesis and structural properties of gold nanoparticles by the chemical reduction method using sodium borohydride

Hadeel Salih Mahdi, Azra Parveen and Ameer Azam
Department of Applied Physics, Z.H. College of Engineering & Technology, Aligarh Muslim University, Aligarh-202002, India

E-mail: azrap2015@gmail.com

Abstract. Sodium borohydride has been utilized to synthesize Au-NPs as stabilizing agents. The characterization of Au-NPs have been done by different techniques. X-ray diffraction machine has been used to study the structural properties of Au-NPs, which shows that the Au-NPs were in spherical and cubic structure. The surface morphology and the elemental composition of Au-NPs were analyzed by SEM attached with the EDAX. UV-visible spectroscopy has been utilized to study the optical properties which shows the absorption peak of Au-NPs at 521nm. The crystallite size of Au-NPs was calculated via Debye-Scherrer and come out to be 12 nm.

Keyword: Sodium borohydride, XRD, Optical Properties, SEM.

1. Introduction
Nanotechnology is an emergent field that has seen an upsurge in advancement during the last few decades. It is going to playing a major role in the commercial use in our future society [1]. The Au-NPs is common stable metal nanoparticles with their fascinating features like their size-linked electronic, optical and magnetic properties, biocompatible, non-cytotoxic properties [2], and used in numerous fields such as medicine, biosensor, pharmacology and drug delivery [3-7]. The Au-NPs are being broadly utilized in a variation of biomedical uses because of their compatibility of synthesis and functionalization, less toxicity and facility exposure [8]. The Au-NPs can be accumulated in the tumor cells showing the optical scattering, therefore, producing a significant role in the microscopy study of cancer cells and chemotherapy [9, 10]. The synthesis of Au-NPs can be attained by a different methods such as chemical, physical, thermal and biological. In the current article, the chemical technique has been used to synthesize Au-NPs by using sodium borohydride as a stabilizing agent [11, 12].

2. Synthesis method
Au-NPs have been synthesized by a chemical process with sodium borohydride as a stabilizing agent. 0.5 mM of gold chloride was dissolved in 100 double distilled water in conical flask and kept stirred for 1 hr. After 1 hr 38.8 mM of sodium borohydride (NaBH₄) was dissolved in 30 ml ice double
distilled water and added dropwise to the solution of gold chloride and kept stirred for 30 min. The color of solution became red after 30 min indicating the structure of Au-NPs as expound in Fig. 1. the chemical reaction is exposed in eqn(1).

\[
\text{HAuCl}_4 + \text{NaBH}_4 \rightarrow \text{Au} + \text{BH}_4^- + \text{HCl} + \text{NaCl}_3
\]  

3. Characterization of Au-NPs

The Au-NPs has been analysed by different techniques. X-Ray diffraction (XRD) has been utilized to analyse the structure of crystalline of the prepared sample. The optical properties have been analysed by UV-visible spectroscopy (Perkin Elmer Lambda-35). The surface and the element structure of the Au-NPs were investigated via scanning electron microscopy (SEM) (JEOL, Japan) attached with (EDAX). The Fourier transforms infrared (FTIR) spectrometer (Perkin Elmer) has been used to study the existence of numerous bonding vibrational frequencies.

4. Results and Discussion

4.1. X-Ray Diffraction

The crystalline structure of the prepared Au-NPs by using (NaBH₄) were studied by XRD analysis, expound in Fig. 2. Typically the XRD plot of the synthesis sample was found via Bragg reflections at diffraction angle 2θ between 25°-85° indexed of (38°), (44°), (64°) and (77°) corresponding to (111), (200), (220) and (311) groups of lattice planes as shown which can be done on the assets of the fcc structure of Au-NPs. The diffraction peak obtained are similar to the standard of gold metal (JCPDS-card no.04.0784) [13].
The crystallite size of the prepared sample can be determined through usage the Deby-Scherrer equation (2) [14, 15].

\[ D = \frac{k\lambda}{\beta \cos \theta} \quad (2) \]

Where, \( D \) is the average crystallite size, \( k \) is constant and equal to (0.9), \( \lambda \) the wavelength (1.54 nm), \( \beta \) is the (FWHM), \( \theta \) is the Bragg angle. The crystallite size is found to be 12 nm.

4.2. **Scanning electron microscopy (SEM)**

The SEM machine was utilized to analyse the surface morphology of Au-NPs. The spherical and cubic shapes of Au-NPs were observed with different size and shown in Fig. 3a. The metallic gold was confirmed by EDAX analysis and shown in Fig. 3b. The elemental composition of the synthesized particles was analysed through EDAX which shown the presence of all Ca, Cl, Na, Mg, Si, C, O and Au ions.
4.3. FTIR Spectroscopy

FTIR analysis of Au-NPs are shown in Fig. 4, by using FTIR spectrophotometer (Perkin-Elmer) in the range of 350-4000 cm\(^{-1}\). The strong band revealed at 3447 cm\(^{-1}\) exhibits the presence of polyphenolic OH group and primary OH band [16]. The absorption peak at 2940 cm\(^{-1}\) may be lead to C-H stretching vibrations of alkanes groups. The narrow peak at 1640 and 1383 cm\(^{-1}\) may arise due to the presence of amide I and C-C stretching aromatic ring. The stretching group of C-O is shown at 1035 cm\(^{-1}\) [17]. The weak peak at 834 and 590 cm\(^{-1}\) is corresponding to S-O stretching of sulfonates and alkyl halides [18].

![Figure 4. FTIR spectra of Au-NPs](image)

4.4. UV-visible Spectroscopy

The optical properties of the Au-NPs were analysed via utilizing UV-visible spectroscopy in the scope of 350-800 nm and expound in Fig. 5. The absorption peak was revealed at 521 nm. The absorption commonly depend upon various factors like oxygen deficiency, impurity centres, surface roughness, etc. [19].

![Figure 5. Absorption spectra of Au-NPs](image)
5. Conclusions
In the current article, the Au-NPs were successfully prepared via chemical reduction method. The confirmation of Au-NPs structure was done by XRD data which shows all peaks. The cubic and spherical form with a different size of Au-NPs has been confirming by (SEM). Through (EDAX), the presence of Au ions and Ca, Cl, Na, Mg, Si, C, O have been confirmed. The optical properties of Au-NPs have been analysed by UV-visible spectroscopy. The functional groups for the formation of Au-NPs have been analysed by the FTIR spectra.

6. References
[1] A. De, R. Bose, A. Kumar, and S. Mozumdar, 2014 Springer Briefs in Molecular Science. Springer India, 35-36.
[2] M.-C. Daniel, and D. Astruc, 2004, Chemical reviews, 104 293-346.
[3] Sobczac-Kupiec A., Malina D., Zimwska M. and Wzorek Z., 2011 Dig. J. Nanomater Bios. 6(2) 803-808.
[4] Bhumakar D. R., Joshi, H. M., Sastry M. and Pokharkar V. B., 2007 Pharmaceut. Res. 24(8) 1415-1426.
[5] Boopathi S, Senthilkumar S. and Phani K. L., 2012 J. Anal. Methods Chem. 1-6.
[6] Chen P. C., Mwakwari S, C. and Oyelere A. K., 2008 Nanotechnol. Sci. Appl. 1 45–66.
[7] Saha, B., Bhattacharya, J., Mukherjee, A., Ghosh A. K., Santra C. R., Dasgupta A. K. and Karmakar, P.I., 2007 Nanoscale Res. Lett. 2 614–622.
[8] Tiwari PM, Vig K, Dennis VA, Singh SR. 2011 Nanomaterials. 1 31 – 63.
[9] Tomar A, Garg G. 2013. Global J Pharmacol. 7 34–38.
[10] Cai W, Chen X. 2007. Small. 3 1840–1854.
[11] Mandal S. 2014 J Radioanal Nucl Chem 299 1209–1212.
[12] Nakanishi M, Takatani H, Kobayashi Y, Hori F, Taniguchi R, Iwase A, and Oshima R 2005 Appl Surf Sci. 241 209–212.
[13] J. Anuradha, T. Abbasi, and S. A. Abbasi 2015 Journal of Advanced Research. 6 711–720.
[14] H. S. Mahdi, A. Parveen, S. Agrawal, and A. Azam 2018 AIP Conf. Proc. 1953 030013.
[15] H. S. Mahdi, A. Parveen, and A. Azam 2018 AIP Conf. Proc. 1953 30031.
[16] Devi, JS, BhimbA, BV, Ratnam, K: Devi, JS, BhimbA, BV, Ratnam, K. 2012 Int. J. Pharm. Pharm. Sci. 4 710–715.
[17] J. Sarkar, S.K. Roy, A. Laskar, D. Chattopadhayay, K. Acharyaa 2013 Mater. Lett. 92 313–316.
[18] S. Rajeshkumar, C. malarkodi, G. Gnanajobitha et al 201 J. nanostructure in chemistry. 3 44.
[19] A. Parveen, S. Agrawal, and A. Azam 2018 Opt. Mater. (Amst) 76 21–27.