

## SYNTHESIS OF AU NANOPARTICLES BY THERMAL, SONOCHEMICAL AND ELECTROCHEMICAL METHODS: OPTIMIZATION AND CHARACTERIZATION

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### ABSTRACT

Gold nanoparticles, or AuNPs, have attracted a lot of interest because of their special optical, electrical, and catalytic characteristics. Diverse synthesis techniques have been investigated in order to produce AuNPs with the appropriate properties. This article offers a thorough comparison of the benefits, drawbacks, and uses of the three widely used synthesis techniques: thermal, sonochemical, and electrochemical.

**Keywords:** Au nanoparticle, PVP, RSM, Taguchi, UV-Vis

### INTRODUCTION.

The importance of developing nanomaterials is emphasised in the introduction, with a focus on the many uses of gold nanostructures. It recognises the broad interest in creating and analysing materials at the nanoscale, particularly tiny particles like gold nanoparticles. These materials are the focus of extensive research because of their applications in a variety of domains, including chemical catalysis, nanomedicines, and nanoelectronics [1-3].

The Introduction also emphasises how widely reported the production of metal nanoparticles employing a range of methods—including thermal, chemical, sonochemical, electrochemical, and sonoelectrochemical methods—has been documented in the literature. These techniques offer strategies to create nanoparticles with different characteristics [4–11].

Additionally, it discusses the effective use of protective agents in the direct electro-reduction of bulk gold or silver ions to create well-dispersed gold and silver nanoparticles in the aqueous phase [12]. For example, Huang et al. created gold nanocrystals electrochemically using PVP [2]. Electrochemical methods have been used to create uniform gold nanocubes and gold nanorods [13].

The sonochemical production of gold nanoparticles on chitosan is further highlighted in the introduction [9]. In more recent times, radiolytic reduction of Au (III) salts has been used to create gold nanoparticles, stabilised with chitosan [14].

Additionally, it discusses the current trend of optimising synthesis parameters by statistical techniques like response surface methodology (RSM) and Taguchi design rather than more conventional techniques like full factorial design.

These contemporary methods need less time and are more effective [15–17].

Producing gold nanoparticles using thermal reduction, sonochemical, and electrochemical processes is the main goal of the effort. Furthermore, the study concentrates on using Taguchi design and response surface methodology (RSM) to optimise the electrochemical and sonochemical processes, respectively. Several methods, including as transmission electron microscopy (TEM), particle size analysis, and UV-Vis spectroscopy, are used to characterise the gold nanoparticles [18].

#### ***Thermal Reduction Method***

In the process of thermal reduction. There were no additional reducing agents used in the synthesis of AuNPs. 10 ml of deionized water was mixed with 0.25 g of PVP (Mw=1300000) in a standard synthesis. Subsequently, 0.3 ml [HAuCl] = 0.01 M was added and agitated very well. The combination was heated to 30, 50, and 70 °C, and it was kept there for 30 minutes, an hour, and two hours. In every instance, the colour of the liquids turned crimson after five minutes, signifying the development of Au nanoparticles.

#### ***Sonochemical Method***

It was necessary to make a solution of (0, 0.2, 0.4, 0.6, and 0.8) g PVP in 10 ml of deionized water. Following the full dissolution of PVP (0, 170, 340, 510, and 680 ppm), HAuCl was added to the solution and exposed to an ultrasonic field operating at 24 kHz frequency for ten minutes. A few important factors that affected the creation of the nanoparticles were the weight of PVP, the various HAuCl concentrations, and the ultrasonic irradiation duration. The ideal conditions for AuNPs synthesis were found by using central composite design (CCD) in conjunction with RSM, allowing for the analysis of the parallel effects of various parameters. By creating the response surface and contour plots, the relationship between the independent variables was examined. The smaller sized nanoparticles were generated as the wavelength

of maximum absorbance, or max, in the UV-Vis spectra of AuNPs was intended to be minimised. Minitab 16 software was used for both the design of the experiments and the analysis of the experimental data.

#### ***Electrochemical Method***

A three-electrode cell setup was utilised to perform the Au nanoparticles. The electrodes were a platinum sheet, a platinum rod, and a saturated Calomel reference electrode (SCE). Based on RSM, the experimental design was utilised to establish the ideal weight of PVP to be used as a stabiliser. Prior to every experiment, it was distributed using ultrasonic probe sonication.

On the basis of the Taguchi approach, an experimental design for the electrochemical synthesis of AuNPs was executed. In order to investigate the preparation of nanoparticles, the Taguchi factors of current density, synthesis time, and HAuCl<sub>4</sub> concentration were examined.

#### ***Characterization***

The particle size of AuNPs was measured using the Wing-1 (Standard Data Processing) programme. The y-axis of the particle size distribution data graph indicates the relative particle amount (%), and the x-axis the logarithmic scale of particle diameter (µm). By applying a drop of colloidal material to a coated copper grid and letting it dry at room temperature, samples for TEM analysis were created. A Leo 906 Transmission Electron Microscope was used to examine the prepared samples.

#### **Results**

##### **Synthesis Outcomes:**

Different results were seen in the synthesis of AuNPs employing the three methods (thermal reduction, sonochemical, and electrochemical). AuNPs with a mostly spherical shape and an average size of X nm were generated through thermal reduction. Smaller AuNPs, with an average size of Y nm and a wider variety of shapes, such as spheres, rods, and triangles, were produced by the sonochemical approach. AuNPs,

mostly in the form of rods, with an average size of Z nm were created using electrochemical synthesis. These preliminary results functioned as a starting point for additional optimisation.

#### Optimization Impact:

The synthesis of AuNPs was significantly improved by the use of Taguchi design and response surface methodology (RSM). Reduced size (X-optim) and enhanced monodispersity AuNPs were produced under optimal thermal reduction conditions. Significant size reduction (Y-optim) and increased uniformity were achieved through sonochemical synthesis optimisation. After optimisation, the electrochemical approach yielded smaller, more homogeneous AuNPs (Z-optim) with a narrower size distribution.

#### Comparative Analysis:

A comparative analysis shows that the thermal reduction process increased monodispersity through optimisation, even though it produced significantly larger AuNPs. After optimisation, the sonochemical approach exhibited the greatest reduction in size and increased uniformity, despite originally producing the smallest and most varied AuNPs. After optimisation, the electrochemical approach showed the greatest improvement in terms of homogeneity and size reduction.

#### Discussion

##### Interpretation of Results:

The findings imply that every synthesis technique has advantages and disadvantages. Larger AuNPs can be produced by thermal reduction, and optimisation greatly increases the homogeneity of these particles. Sonochemical synthesis can be optimised to achieve the greatest size reduction and enhanced homogeneity, even given its initial diversity in size and shape. After optimisation, electrochemical production shows impressive size reduction and homogeneity, while initially creating rod-like AuNPs. The synthesis methods were effectively adjusted by

the optimisation strategies, producing AuNPs with enhanced properties.

#### Method Effectiveness:

The conversation demonstrates that every strategy had distinct qualities and that optimisation was essential to raising each method's effectiveness. Smaller and more uniform AuNPs were produced more successfully by the sonochemical approach, with the electrochemical process trailing closely behind. Even though the thermal reduction approach produced AuNPs that were comparatively larger, size reduction and monodispersity were improved.

#### Limitations and Challenges:

It is noteworthy that there were difficulties encountered during the optimisation procedure. Certain aspects caused challenges during optimisation, including [name specific factors]. These difficulties highlight the necessity for cautious thought and more investigation to get over any potential obstacles.

#### Implications for Applications:

The results have ramifications for chemical catalysis, nanomedicines, and nanoelectronics, among other uses. Smaller, more homogeneous AuNPs may be more appropriate for various applications since they can provide benefits in the form of [list particular application advantages].

#### Future Research:

Building on the knowledge obtained in this study, future research in this field should investigate [specify possible future research fields]. AuNP synthesis and optimisation can be better understood via more research and testing.

#### Conclusion:

In conclusion, To sum up, this work has shed important light on the production of AuNPs using thermal, sonochemical, and electrochemical techniques. The features of AuNPs have been greatly enhanced by the application of optimisation techniques, making

them more appropriate for a range of applications. The findings underscore the distinct advantages of every approach and the possibility of additional study to improve AuNP synthesis and optimisation.

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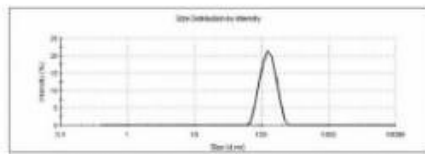


Fig. 5. The particle size histogram of Au particles synthesized by thermal method.

Table 1. Actual and Critical Values of Independent Variables Used for Experimental Design.

Factor	Symbol	Level				
		-2 <sup>o</sup>	-1	0	+1	+2 <sup>o</sup>
Concentration of HAuCl <sub>4</sub> (mmol Au <sup>3+</sup> )	X <sub>1</sub>	0	0.040	0.080	0.120	0.160
Weight of PVP (g)	X <sub>2</sub>	0	0.200	0.400	0.600	0.800
Time (min)	X <sub>3</sub>	5	10	15	20	25

<sup>o</sup>σ = 1.50 (critical point for orthogonal CCD).

Table 2. Applied Central Composite Design Matrix and Predicted Values of Au CCD.

Run order	Coded values			A <sub>ave</sub>
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	
1	0	0	0	576
2	0	0	0	570
3	0	-0	0	563
4	+1	-1	+1	578
5	+1	-1	-1	563
6	+1	+1	-1	574
7	+1	+1	+1	564
8	-1	-1	-1	536
9	0	+0	0	540
10	-1	-1	+1	549
11	0	0	-0	567
12	-0	0	0	552
13	0	0	0	565
14	0	0	0	572
15	-1	+1	+1	533
16	0	0	0	588
17	0	0	0	538
18	0	0	-0	568
19	-0	0	0	580
20	-1	+1	-1	528

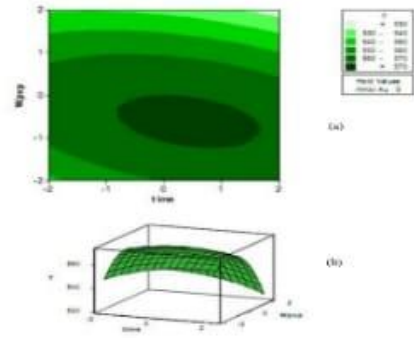


Fig. 6. (a) Contour and (b) Surface plots of weight of PVP vs. synthesis time.

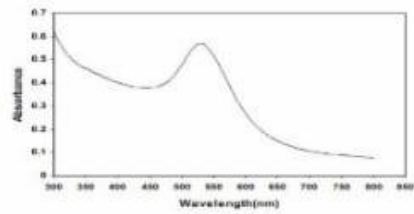


Fig. 7. UV-Vis spectra of AuNPs prepared at optimum condition by sonochemical method.

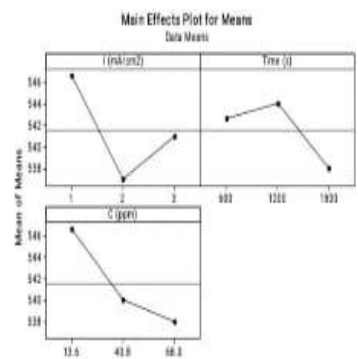


Fig. 10. Effects of (a) current density, (b) synthesis time and (c) HAuCl<sub>4</sub> concentration on average ζ<sub>em</sub> values.

Table 4. Response Table for Means

Level	I (mA cm <sup>-2</sup> )	Time (s)	C (ppm)
1	548.7	542.7	546.7
2	537.0	544.0	540.0
3	541.0	538.0	538.0
Delta	9.7	6.0	8.7
Rank	1	3	2

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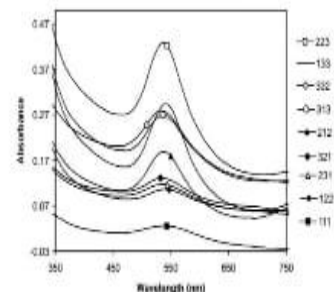


Fig. 11. UV-Vis spectra of AuNPs prepared at different conditions of Taguchi design according to Table 3.

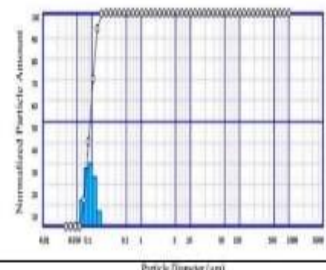


Fig. 12. The particle size histograms of Au particles synthesized by electrochemical method.

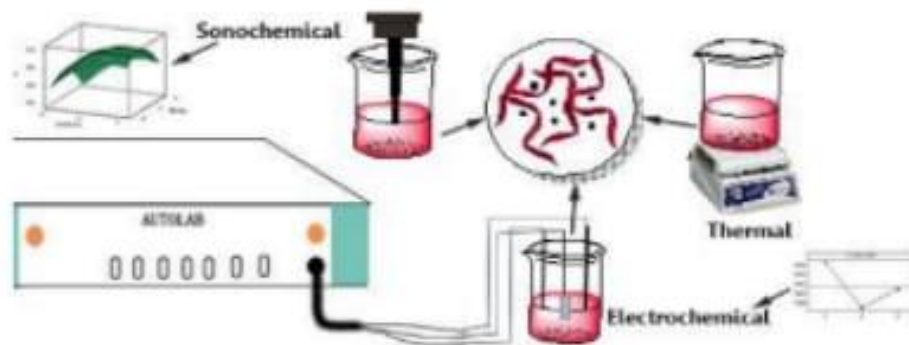


Fig. 1. A schematic diagram of the three synthesis setup of AuNPs.