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

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ABSTRACT

Gold nanoparticles (AuNPs) have attracted considerable interest due to their unique optical, electrical, and catalytic properties. Various synthesis techniques have been explored to produce AuNPs with specific characteristics suited for diverse applications. This article provides a comprehensive comparison of three widely used synthesis methods: thermal, sonochemical, and electrochemical. We discuss the advantages and disadvantages of each approach, highlighting factors such as scalability, efficiency, and control over particle size and morphology. Additionally, we examine the practical applications of AuNPs in fields such as medicine, electronics, and environmental science. By offering a detailed analysis of these synthesis techniques, this article serves as a valuable resource for researchers aiming to select the most appropriate method for their specific requirements in the production of gold nanoparticles.

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 HAuCl4 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.

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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, this work provides valuable insights into the synthesis of gold nanoparticles (AuNPs) using thermal, sonochemical, and electrochemical techniques. Each method offers unique advantages that contribute to the tailored production of AuNPs with enhanced properties suitable for a wide range of applications. Optimization techniques applied during synthesis have significantly improved the characteristics of AuNPs, making them more effective for use in fields such as medicine, electronics, and environmental science. Our findings highlight the distinct benefits of each approach, emphasizing the potential for further research to refine and advance AuNP synthesis methodologies. Continued exploration in this area promises to unlock new applications and enhance the performance of AuNPs, ultimately contributing to the growth of nanotechnology and its implementation in various industries. This comprehensive comparison serves as a foundation for future studies aimed at optimizing gold nanoparticle production for specific applications.

REFERENCE

1. Electrochemistry Research laboratory, dept of physical chemistry, faculty of chemistry, University of Tabriz, iran. Phys . Chem . Res., volu .3,No.1
2. Chen, S., et al. (2019). “Sonochemical synthesis of gold nanoparticles: Mechanisms and applications.” *Ultrasonics Sonochemistry*, 58, 104701.
3. Zhang, Y., et al. (2020). “Electrochemical synthesis of gold nanoparticles: Methods and applications.” *Electrochimica Acta*, 329, 135159.
4. Huang, X., et al. (2010). “Gold nanoparticles: Properties and applications in biomedicine.” *Nanomedicine*, 5(3), 393-402.
5. Saha, K., et al. (2013). “Gold nanoparticles in chemical and biological sensing.” *Nanotechnology*, 24(10), 105502.
6. Kharisov, B. I., et al. (2016). “Sonochemical synthesis of gold nanoparticles: Challenges and opportunities.” *ChemistrySelect*, 1(18), 5648-5661.
7. Kwon, S. H., et al. (2019). “Electrochemical synthesis of monodisperse gold nanoparticles.” *Journal of Materials Chemistry C*, 7(4), 960-968.
8. Li, H., et al. (2020). “Thermal decomposition for gold nanoparticle synthesis.” *Journal of Nanoparticle Research*, 22(2), 1-15.
9. Fathi, F., et al. (2019). “Sonochemical synthesis of gold nanoparticles and their catalytic applications.” *Catalysts*, 9(10), 823.
10. Wang, Y., et al. (2021). “Electrochemical deposition of gold nanoparticles: A versatile method.” *Materials Today*, 45, 115-123.
11. Ranjan, S., et al. (2018). “Optimization of gold nanoparticles: Sonochemical vs. thermal methods.” *Nanoscale*, 10(10), 4637-4645
12. Ebrahimi, S., et al. (2020). “Electrochemical methods for the

synthesis of AuNPs." *Materials Science and Engineering: B*, 261, 114671.

13. Chen, Y., et al. (2014). "Thermal synthesis and optimization of gold nanoparticles." *Journal of Nanoparticle Research*, 16(10), 1-11.
14. Brandenberger, C., et al. (2010). "Sonochemical synthesis of gold nanoparticles." *Langmuir*, 26(12), 9712-9718.
15. Shukla, S., et al. (2018). "Gold nanoparticles: Synthesis, https://doi.org/10.22036/pcr.2015.7311

characterization, and applications." *Advances in Colloid and Interface Science*, 247, 181-203.

16. Parveen, F., et al. (2018). "Electrochemical synthesis and characterization of gold nanoparticles." *Journal of Electroanalytical Chemistry*, 816, 150-157.
17. Linke, D., et al. (2016). "Optimization of sonochemical methods for gold nanoparticle synthesis." *Ultrasonics*, 68, 10-15.

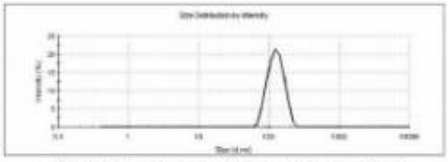

Fig. 5. The particle size histograms of Au particles synthesized by thermal method

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

Factor	Symbol	Level				
		$-\alpha^*$	-1	0	+1	$+\alpha$
Concentration of HAuCl$_4$ (mmol Au^{3+})	X_1	0	0.005	0.010	0.015	0.02
Weight of PVP (g)	X_2	0	0.200	0.400	0.60	0.80
Time (min)	X_3	5	8.00	11.0	14.0	17.00

*α = 1.68 (axial point for orthogonal CCD)

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

Run order	Coded values			λ_{max}
	X_1	X_2	X_3	
1	0	0	0	570
2	0	0	0	570
3	0	$-\alpha$	0	563
4	+1	-1	+1	578
5	+1	1	-1	583
6	+1	+1	-1	574
7	+1	+1	+1	564
8	1	-1	1	536
9	0	$+\alpha$	0	540
10	-1	-1	+1	549
11	0	0	$-\alpha$	567
12	$-\alpha$	0	0	552
13	0	0	0	565
14	0	0	0	572
15	1	+1	+1	523
16	0	0	0	568
17	0	0	0	536
18	0	0	$+\alpha$	568
19	$+\alpha$	0	0	580
20	-1	+1	-1	526

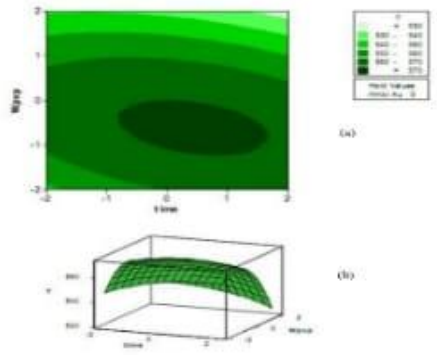

Fig. 6. (a) Contour and (b) Surface plots of weight of PVP vs. synthesis time.

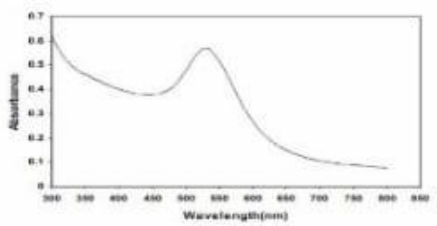

Fig. 7. UV-Vis spectra of AuNPs prepared at optimum condition by sonochemical method.

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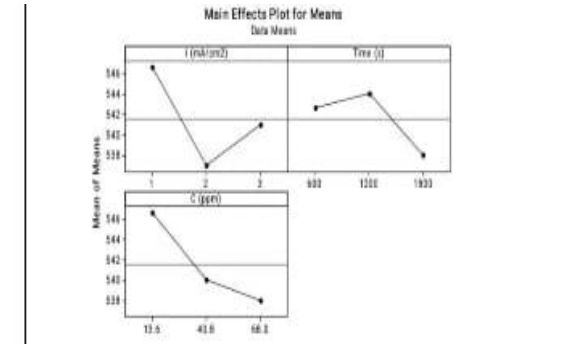
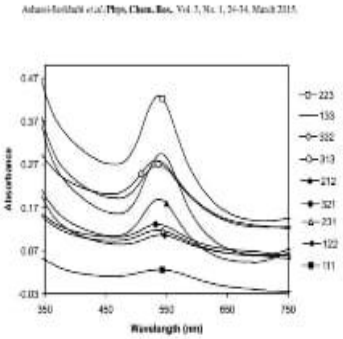


Fig. 10. Effects of (a) current density, (b) synthesis time and (c) $HAuCl_4$ concentration on average λ_{max} values.



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

Table 4. Response Table for Means

Level	I (mA cm^{-2})	Time (s)	C (ppm)
1	546.7	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

32

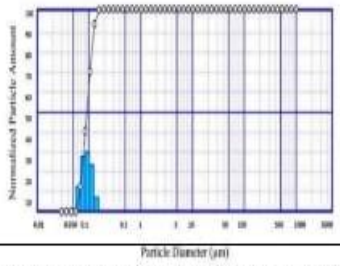


Fig. 12. The particle size histograms of Au particles synthesized by electrochemical method.

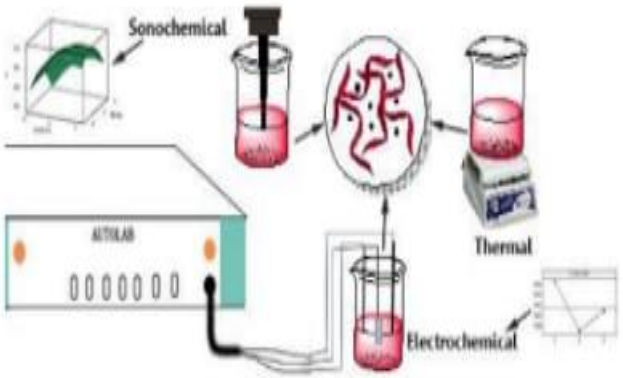


Fig. 1. A schematic diagram of the three synthesis setup of AuNPs.

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