Faculty Development Grant Final Report

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Title of Project:

Optimization of Nanosilver Synthesis For The Efficient Catalysis of Bacterial and Organic Pollutants

Awardee Information:

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Outcome:

- Faith Riley presented a poster at the LU Science Fair (Attachment)
- A manuscript is ready for the formal journal submission (Attachment)

Statistical Validation and Parametric Optimization of *Eucommia ulmoides* Mediated Gold Nanoparticle Synthesis Using a Taguchi L_9 Orthogonal Array

Abstract

This research presents a systematic optimization of gold nanoparticle (AuNP) synthesis using an aqueous extract of *Eucommia ulmoides* (EU) barks. The study employed a Taguchi L_9 orthogonal array to evaluate the influence of four primary parameters: EU extract volume, system pH, incubation temperature, and reaction time. The surface plasmon resonance (λ_{max}) intensity served as the response variable to quantify the efficiency of the biogenic reduction process. Statistical analysis via ANOVA revealed that system pH and reaction time were the most critical determinants, contributing 46.64% and 40.81% to the total variance, respectively. The optimal synthesis conditions were identified as the A3B2C1D2 configuration. A predictive regression model demonstrated high statistical fidelity with an R^2 of 95.2%. Residual diagnostics confirmed that the experimental data adhered to Gaussian distribution and homoscedasticity assumptions. These results establish a robust, mathematically validated framework for the sustainable production of AuNPs using *Eucommia ulmoides* as a potent bio-reductant.

Keywords: *Eucommia ulmoides*, Gold nanoparticles, Taguchi method, Green synthesis, ANOVA, L_9 orthogonal array, Process optimization, Predictive modeling, Signal-to-Noise Ratio.

1. Introduction

Gold nanoparticles (AuNPs) have attracted significant attention in materials science due to their exceptional localized surface plasmon resonance (LSPR) and size-dependent physical properties. These characteristics make them essential components in fields such as heterogeneous catalysis, chemical sensing, and optoelectronics [1, 2]. Conventional fabrication techniques typically involve chemical reduction using reagents like sodium borohydride or hydrazine [3]. While effective, these methods often necessitate the use of toxic stabilizers and involve complex purification steps to remove hazardous byproducts [4, 5]. The growing emphasis on sustainable chemistry has led to the development of biogenic synthesis, which utilizes plant-derived phytochemicals to act as both reducing and capping agents. This approach is not only environmentally benign but also cost-effective and scalable for laboratory and industrial applications [6-9].

Eucommia ulmoides, often referred to as Du-zhong, is a deciduous tree that is highly regarded for its diverse secondary metabolites. The leaves of this species contain a complex mixture of iridoids, flavonoids, and lignans. Key compounds such as aucubin, geniposidic acid, and various polyphenolic acids are particularly relevant for nanoparticle synthesis [10-12]. These molecules possess abundant hydroxyl groups that facilitate the reduction of Au^{3+} ions to metallic Au^0 through electron transfer mechanisms. Furthermore, the large molecular structure of these phytochemicals provides steric stabilization, preventing the nanoparticles from undergoing unwanted aggregation [13, 14]. Despite the well-documented antioxidant properties of *Eucommia ulmoides*, its potential as a biological factory for the precision synthesis of AuNPs remains insufficiently explored in the existing literature.

The quality and yield of biogenic AuNPs are highly sensitive to the experimental environment [15]. The synthesis is governed by a set of interconnected kinetic and thermodynamic variables, including the concentration of the plant extract, the pH of the precursor solution, the incubation temperature, and the duration of the reaction [16]. Among these, pH is particularly influential as it dictates the ionization state of the phenolic groups, thereby modulating their reducing potential and the resulting nucleation rate [17, 18]. Similarly, temperature and reaction time control the growth and stabilization phases of particle formation [19]. Understanding the individual and synergistic effects of these parameters is essential for achieving a reproducible and high-yield synthesis process [20].

Traditional experimental optimization often relies on the one-factor-at-a-time (OFAT) approach. This method is inherently limited because it requires a large number of experimental runs and cannot account for the interactions between different variables [21-23]. To overcome these challenges, the Taguchi method has been adopted as a more efficient strategy for experimental design. The Taguchi framework utilizes orthogonal arrays to study the entire parameter space with a significantly reduced number of trials [24]. By calculating the signal-to-noise (S/N) ratio, this method identifies the optimal settings that minimize the impact of experimental noise, ensuring that the process is robust and reproducible [22, 25].

In this study, we utilize a Taguchi L_9 design to optimize the synthesis of AuNPs mediated by *Eucommia ulmoides* bark extract. We focus on maximizing the λ_{max} absorbance intensity, which serves as a direct indicator of nanoparticle concentration and synthesis efficiency. The study goes beyond simple observation by employing rigorous statistical tools, including analysis of variance (ANOVA) and residual mapping, to validate the accuracy of the optimization model. By quantifying the percentage contribution of each factor, we aim to identify the dominant drivers of the synthesis process. This research provides a mathematically rigorous foundation for the biogenic production of AuNPs, demonstrating how *Eucommia ulmoides* can be leveraged as a sustainable resource for high-quality nanomaterial fabrication. The findings contribute to the broader goal of integrating green chemistry principles into nanotechnology while providing insights into the parametric control required for successful biogenic synthesis.

2. Materials and Methods

Chloroauric acid ($HAuCl_4$) was obtained from Sinopharm Chemical Reagent Co. Ltd. (China). All other reagents, including sodium hydroxide (NaOH) for pH adjustment, were of analytical grade. High-purity water (18.30 MΩ cm) was utilized for all preparations. *Eucommia ulmoides* bark was sourced locally, cleaned, and ground into a fine powder.

2.1 Taguchi Design of Experiments (DoE)

The four primary controllable parameters investigated in this work, which included EU volume (A), pH (B), temperature (C), and time (D), are summarized in **Table 1**. Each of these factors was evaluated across three separate levels. To refine the experimental process, a Taguchi L_9 orthogonal array (OA) was constructed with the aid of Minitab® 17 statistical software (Minitab Inc., Coventry, UK).

Table 1: Experimental Factors and Levels for Taguchi L_9 Array.

Process parameters			**Level**		
Factor	***Description***	***Unit***	***1***	***2***	***3***
A	EU Volume	ml	2	3	4
B	pH	−	5	7	9
C	Temperature	°C	40	50	60
D	Time	minute	20	30	60

The Taguchi method employs orthogonal arrays (OAs) to systematically determine optimal processing parameters while simultaneously minimizing experimental variability. Performance evaluation within this framework is conducted using the signal-to-noise (S/N) ratio, expressed in decibels (dB), which functions as a quantitative quality index. Defined as a logarithmic objective function, the S/N ratio reflects the relationship between the mean response (signal) and its associated standard deviation (noise) [26]. Consequently, it serves as a robust indicator for identifying process conditions that suppress variability arising from uncontrollable factors [27].

Three standard formulations of the S/N ratio are commonly applied: smaller-the-better, larger-the-better, and nominal-the-best. Regardless of the formulation, higher S/N values correspond to improved performance and enhanced product quality. In the present study, optimization focused on maximizing the S/N ratio to attenuate the influence of random noise on system performance [28]. Because λ_{max} increases monotonically with the desired response, the larger-the-better criterion was selected [29]. The corresponding mathematical formulation is presented in Equation (1).

$$S/N = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \qquad \text{(Eq. 1)}$$

Herein, S/N represents the signal-to-noise ratio, n is the number of observations, and y denotes the measured nanofiber diameter.

2.3 Analytical Characterization Tools

UV-Vis spectroscopy was performed using a Tu-1901 spectrophotometer from 300 to 800 nm. HRTEM images and EDX spectra were recorded on a JEOL-JEM-2100F to determine morphology and composition. XRD patterns were recorded using a Bruker D8 Advance Diffractometer with Cu Kα radiation. FTIR measurements (VERTEX 70) identified capping functional groups. Zeta potential and DLS analysis were performed using a Malvern ZEN 3600 Zetasizer.

2.4 Analysis of variance

Analysis of variance (ANOVA) is a statistical framework employed to evaluate the influence of selected process factors on an output response through systematic partitioning of the total variability. This approach isolates the relative contributions of individual parameters to response fluctuations and quantitatively assesses their effects [30]. The percentage contribution, derived from each factor's sum of squares relative to the total sum of squares, serves as a direct measure of its significance in governing performance characteristics. Statistical significance is evaluated using the F-test, wherein larger F-values indicate a more pronounced influence of the corresponding factor [31]. Consequently, the ANOVA table provides critical insights for identifying dominant input parameters, thereby guiding rational process optimization via targeted parameter control. Unlike direct data comparison, ANOVA emphasizes variance decomposition to elucidate underlying factor–response relationships [32].

3. Results and Discussion

3.1 Determination of S/N ratio

Within the Taguchi optimization framework, the signal-to-noise (S/N) ratio is employed as a quantitative indicator of process performance by describing deviations of the experimental response from its target value. In this context, the signal represents the desired mean response, whereas noise reflects the associated standard deviation. Using an L_9 orthogonal design, the individual effects of key synthesis parameters on gold nanoparticle (AuNP) formation were systematically evaluated. The UV–vis absorption maximum (λ_{max}) was selected as the response variable and analyzed through the S/N ratio.

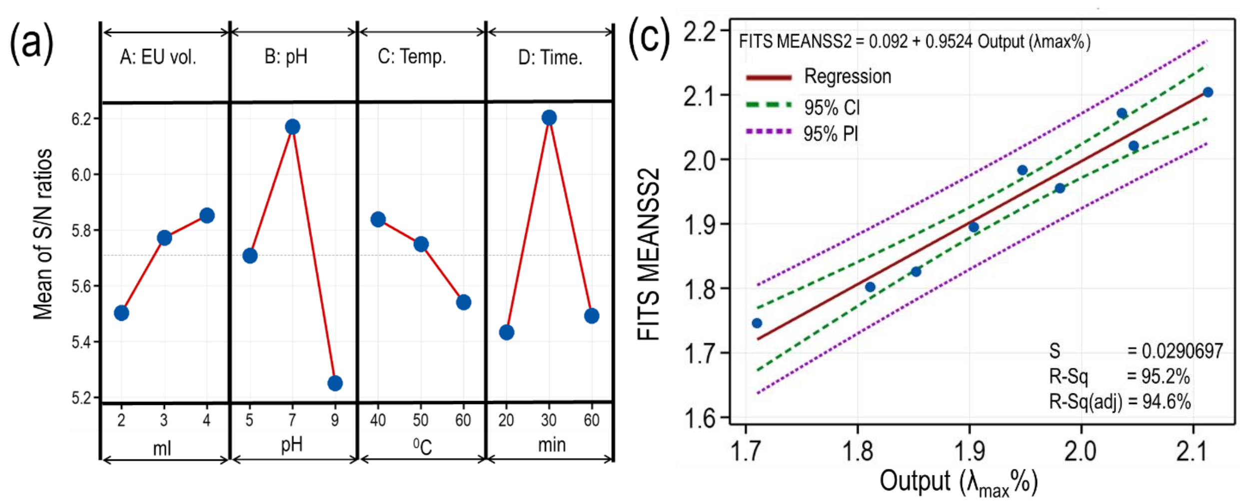
Table *3* summarizes the calculated S/N ratios and corresponding factor rankings for EU volume, system pH, incubation temperature, and reaction time, enabling direct comparison of their relative influences. In addition, **Table 2** reports the cumulative S/N ratios obtained for all synthesized samples. The main effects plot shown in **Figure 1:a** illustrates the variation in the S/N ratio as a function of each control parameter based on λ_{max} values. Factor significance was assessed using the delta statistic, defined as the difference between the maximum and minimum mean S/N ratios across the investigated levels of each parameter. As indicated in **Table *3***, system pH exhibited the highest delta value of 0.918 and was therefore identified as the dominant factor governing AuNP synthesis, followed by reaction time and EU volume, whereas incubation temperature exerted the least influence. This hierarchy was established using the larger-the-better optimization criterion.

Table 2: Taguchi L_9 Orthogonal Array with Experimental Output and S/N Ratios.

Run	A (EU mL)	B (pH)	C (Temp °C)	D (Time min)	Output (λ_{max} %)	S/N Ratio (dB)
1	2	5	40	20	1.852	5.352
2	2	7	50	30	2.113	6.497
3	2	9	60	60	1.710	4.659
4	3	5	50	60	1.904	5.593
5	3	7	60	20	1.947	5.787
6	3	9	40	30	1.981	5.937
7	4	5	60	30	2.036	6.175
8	4	7	40	60	2.047	6.222
9	4	9	50	20	1.811	5.158

Among the experimental trials in **Table 2**, Runs 2 (EU volume = 2 mL, pH 7, 50 °C, 30 min) and 8 (EU volume = 4 mL, pH 7, 40 °C, 60 min) yielded the highest S/N ratios, indicating more robust and favorable AuNP formation under these conditions. To further verify the relative contributions and statistical significance of each control factor, the Taguchi analysis was complemented by an analysis of variance (ANOVA), as discussed in the subsequent section.

The primary influence of synthesis parameters on the signal-to-noise (S/N) ratio for EU-mediated AuNP fabrication is illustrated in **Figure 1:b**. Within this Taguchi framework, the magnitude of the slope relative to the reference line serves as a proxy for parametric sensitivity. Herein, steep gradients denote substantial influence, and near-parallel trajectories signify negligible impact [27]. Statistical analysis revealed that system pH (Factor B) and reaction time (Factor D) exerted the most pivotal effects on synthesis optimization. The influence of pH on AuNP formation is highly contingent upon thermal conditions, necessitating precise simultaneous control of both variables. Conversely, incubation temperature (Factor C) and EU volume (Factor A) manifested marginal influences on the response. This dualistic behavior underscores the necessity of managing specific synergistic pairings, particularly pH–time, to govern AuNP synthesis kinetics effectively. Under these optimized trajectories, the parameter constellation A3B2C1D2 was identified as the ideal configuration, maximizing the λ_{max} value, a critical metric directly correlated with enhanced dyeing performance.



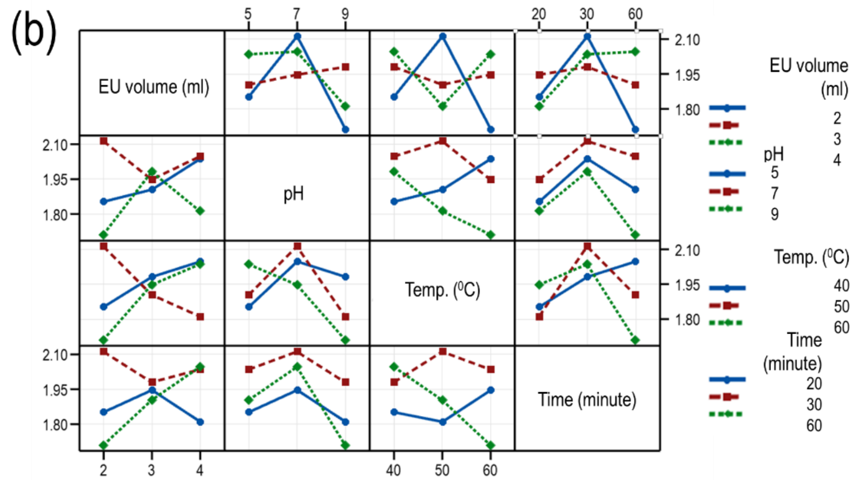


Figure 1: (a) Main effect plots for the investigated synthesis parameters, (b) interaction plots illustrating combined factor effects, and (c) fitted line plots validating experimental and model-predicted fixation rates for Taguchi L_9-optimized EU-mediated AuNP.

Table 3: Response Table for Signal-to-Noise (S/N) Ratios.

Level	A (EU Volume)	B (pH)	C (Temperature)	D (Time)
1	5.503	5.707	5.837	5.432
2	5.772	6.169	5.749	6.203
3	5.852	5.251	5.549	5.491
Delta	0.349	0.918	0.297	0.771
Rank	3	1	4	2

Finally, a robust mathematical relationship was established via a predictive regression model. The parity plot (**Figure 1:c**) demonstrates exceptional congruence between experimental observations and model predictions [33]. The model exhibited high statistical fidelity, with a coefficient of determination (R^2) of 95.2% and an adjusted R^2 of 94.6%, confirming its ability to account for nearly all observed variance. Pearson correlation analysis corroborated this predictive accuracy ($p < 0.001$), validating the model's reliability for forecasting synthesis outcomes and enabling comparative analysis across diverse experimental landscapes.

To quantify the statistical variance partitioning and identify the primary determinants of the fabrication process, analysis of variance (ANOVA) was performed (**Table *4***) [34]. Statistical results indicate that system pH represents the most pivotal factor, exhibiting an F-value of 9.05 ($p = 0.099$) and accounting for a substantial 46.64% of the total variance. Reaction time emerged as the second most influential variable ($F = 7.92$, $p = 0.112$), contributing 40.81% to the synthesis outcome. Conversely, the marginal contributions of EU extract volume (7.40%) and incubation temperature (3.10%) suggest that the AuNP synthesis kinetics are relatively robust and insensitive to fluctuations within these specific parametric ranges [35]. To verify the statistical integrity and adequacy of the experimental design, we evaluated the model's reliability through residual analysis.

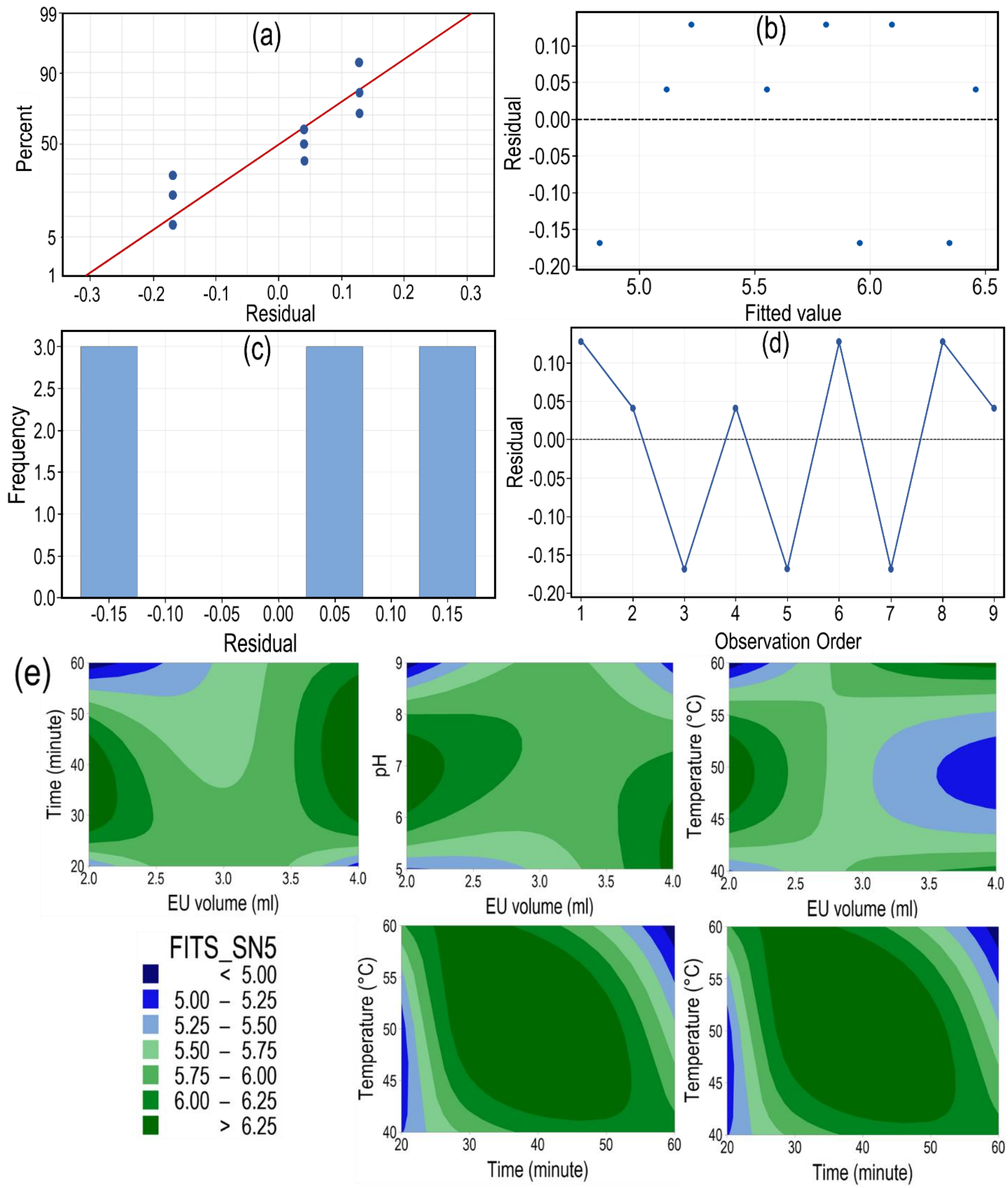


Figure 2: Diagnostic residual plots for fixation rate S/N ratios including (a) normal probability plot, (b) fitted value plot, (c) residual histogram, and (d) observation order plot. (e) Contour plots illustrating the effects of EU volume, reaction time, temperature, and pH on the S/N ratio.

The statistical integrity and predictive reliability of the developed regression model were rigorously validated through diagnostic residual analysis (**Figure *2***). The normal probability plot (**Figure *2*:a**) illustrates that the residuals are positioned in close proximity to the linear reference, confirming that the experimental errors follow a Gaussian distribution [36]. This finding is further corroborated by the histogram of residuals (**Figure *2*:c**), which manifests a symmetric, bell-shaped profile centered around zero [37]. To assess the assumption of homoscedasticity, we evaluated the relationship between residuals and fitted values (**Figure *2*:b**). The stochastic and uniform distribution of data points across the midline indicates a constant variance, suggesting that the model effectively captures the underlying process variability without systematic bias [38]. Furthermore, the residuals-versus-order plot (**Figure *2*:d**) demonstrates that the error terms are randomly distributed without discernible temporal trends or autocorrelation, thereby confirming the independence of the observations [39]. Collectively, these diagnostic metrics substantiate the robustness of the Taguchi-based experimental design, ensuring that the optimized parameters are derived from a statistically sound foundation. Beyond error validation, contour plots (**Figure *2*:e**) were generated to elucidate the complex interaction effects and synergistic landscapes of the synthesis variables on the λ_{max} response, facilitating a visual mapping of the optimal fabrication regime [40].

Table 4: Analysis of variance (ANOVA) for the S/N ratio of EU mediated AuNP synthesis.

Source of Variation	**DF**	**Sum of Squares (SS)**	**Mean Square (MS)**	**F-value**	**p-value**	**Contribution (%)**
EU volume	2	0.2002	0.1001	1.44	0.410	7.40
System pH	2	1.2620	0.6310	9.05	0.099*	46.64
Temperature	2	0.1393	0.0496			3.10
Reaction time	2	1.1045	0.5523	7.92	0.112	40.81
Residual Error		0.2694	0.0697			2.05
Total	**8**	**2.9754**				**100.00**

5. Conclusion

This research demonstrates the successful optimization of gold nanoparticle synthesis using *Eucommia ulmoides* bark extract through a Taguchi L_9 experimental design. Statistical analysis confirms that system pH and reaction time are the primary factors influencing the synthesis, with a collective contribution of 87.45%. The optimal configuration was established as A3B2C1D2, which maximized the LSPR response and ensured efficient nanoparticle formation. The regression model exhibited high predictive reliability, supported by an R^2 of 95.2% and a p-value < 0.001. Comprehensive residual diagnostics further validated the model's robustness, showing that the errors are normally distributed and independent. Future investigations should focus on evaluating the catalytic efficiency of these biogenic nanoparticles in environmental remediation and exploring their synergistic bioactivity in targeted nanomedicine. This study provides a validated framework for scaling this green protocol through continuous flow microfluidic reactors to facilitate broad industrial implementation.

References:

[1] H.S. Mahdi, A. Parveen, J. Bahadur, S. Singh, A. Azam, A facile biosynthesis approach for development of eco-friendly stable gold nanoparticles, Materials Letters 406 (2026) 139859.

[2] N.M. El-Deeb, S.M. Khattab, M.A. Abu-Youssef, A.M.A. Badr, Green synthesis of novel stable biogenic gold nanoparticles for breast cancer therapeutics via the induction of extrinsic and intrinsic pathways, Scientific Reports 12(1) (2022) 11518.

[3] S. Pedireddy, H.K. Lee, W.W. Tjiu, I.Y. Phang, H.R. Tan, S.Q. Chua, C. Troadec, X.Y. Ling, One-step synthesis of zero-dimensional hollow nanoporous gold nanoparticles with enhanced methanol electrooxidation performance, Nature Communications 5(1) (2014) 4947.

[4] E. Naderi-Samani, R.S. Razavi, K. Nekouee, H. Naderi-Samani, Synthesis of silver nanoparticles for use in conductive inks by chemical reduction method, Heliyon 9(10) (2023) e20548.

[5] M.A. Awad, N.E. Eisa, P. Virk, A.A. Hendi, K.M.O.O. Ortashi, A.S.A. Mahgoub, M.A. Elobeid, F.Z. Eissa, Green synthesis of gold nanoparticles: Preparation, characterization, cytotoxicity, and anti-bacterial activities, Materials Letters 256 (2019) 126608.
[6] M. Sathishkumar, S. Pavagadhi, A. Mahadevan, R. Balasubramanian, Biosynthesis of gold nanoparticles and related cytotoxicity evaluation using A549 cells, Ecotoxicology and Environmental Safety 114 (2015) 232-240.
[7] G.I. Edo, A.N. Mafe, A.B.M. Ali, P.O. Akpoghelie, E. Yousif, E.F. Isoje, U.A. Igbuku, K. Zainulabdeen, J.O. Owheruo, A.E.A. Essaghah, H. Umar, D.S. Ahmed, A.A. Alamiery, Eco-friendly nanoparticle phytosynthesis via plant extracts: Mechanistic insights, recent advances, and multifaceted uses, Nano TransMed 4 (2025) 100080.
[8] A.D. Tiwari, A.K. Mishra, S.B. Mishra, O.A. Arotiba, B.B. Mamba, Green synthesis and stabilization of gold nanoparticles in chemically modified chitosan matrices, International Journal of Biological Macromolecules 48(4) (2011) 682-687.
[9] S.K. Begum, D. Shabnam, N. Haque, M.J. Alam, J. Ferdous, U.J.B. Nur, K. Fatema, R.J. Shabiha, R.J. Clarke, P. Chowdhury, M.N. Uddin, Green synthesis of magnetite (Fe3O4) and hematite (Fe2O3) nanoparticles using Moringa oleifera and Psidium guajava leaf extracts for sustainable applications, Scientific Reports 15(1) (2025) 36465.
[10] X. Dai, Q. Huang, B. Zhou, Z. Gong, Z. Liu, S. Shi, Preparative isolation and purification of seven main antioxidants from Eucommia ulmoides Oliv. (Du-zhong) leaves using HSCCC guided by DPPH-HPLC experiment, Food Chemistry 139(1) (2013) 563-570.
[11] X. Ran, Y. Liu, D. Zhao, The relationship between EuSRPP1 gene expression and rubber biosynthesis in Eucommia ulmoides Oliver (Du-zhong), Industrial Crops and Products 175 (2022) 114246.
[12] Y. Wang, L. Liu, Z. Tan, Polarity-controlled deep eutectic solvents-based biphasic system for the selective separation of geniposidic acid and aucubin from Eucommia ulmoides male flowers, Journal of Molecular Liquids 358 (2022) 119200.
[13] H. Wan, S. Habib, H. Liu, S. Mahmud, Catalytic degradation of diverse azo dyes using Eucommia ulmoides bark-mediated gold nanocatalyst, Environmental Chemistry and Ecotoxicology 6 (2024) 171-179.

[14] S. Guo, J. Tong, Y. Liu, D. Qin, J. Yan, H. Peng, L. Sun, X. Jing, X. Wu, B. Li, Synthesis of Eucommia ulmoides-derived carbon dots for anti-inflammatory and accelerated wound healing, International Immunopharmacology 143 (2024) 113606.
[15] M. Suliman, E.H. Ibrahim, M.Y. Alshahrani, M. Kilany, R.N. Alsulami, Z. Khired, H.M. Shubaily, A.F. El-kott, A.E. Ahmed, S.A. Salama, Biogenic synthesis of gold nanoparticles using Coleus forskohlii leaf extract enhances anticancer activity against HT-29 cells and modulates the p53 regulated immune microenvironment in colon adenocarcinoma, Scientific Reports 16(1) (2026) 2374.
[16] M. Reindl, V. Zach, G. Cvirn, S.P. Schwaminger, Influence of coprecipitation synthesis parameters on the physicochemical properties and biological effects of iron oxide nanoparticles, Nanoscale Advances 7(22) (2025) 7395-7407.
[17] E.B. Ferreira, J.F. Gomes, G. Tremiliosi-Filho, L.H.S. Gasparotto, One-pot eco-friendly synthesis of gold nanoparticles by glycerol in alkaline medium: Role of synthesis parameters on the nanoparticles characteristics, Materials Research Bulletin 55 (2014) 131-136.
[18] L. Lin, M.J. Alam, H. Liu, M. Islam, S. Mahmud, Green synthesis of redroot medicated nanosilver for acid azo dyes catalysis, AIP Conference Proceedings 3342(1) (2025).
[19] T. Ahmad, M. Irfan, M.A. Bustam, S. Bhattacharjee, Effect of Reaction Time on Green Synthesis of Gold Nanoparticles by Using Aqueous Extract of Elaise Guineensis (Oil Palm Leaves), Procedia Engineering 148 (2016) 467-472.
[20] J. Chen, Y. Liu, Y. Xiong, D. Wei, J. Peng, S. Mahmud, H. Liu, Konjac glucomannan reduced-stabilized silver nanoparticles for mono-azo and di-azo contained wastewater treatment, Inorganica Chimica Acta 515 (2021) 120058.
[21] G. Zhang, M.J. Alam, M. Islam, H. Liu, S. Mahmud, Biosynthesis and Stabilization of Nanosilver Using Houttuynia Extract for Degradation of Azo Acid and Mordant Dyes, MATEC Web Conf. 405 (2024) 01001.
[22] E.M. Williamson, Z. Sun, L. Mora-Tamez, R.L. Brutchey, Design of Experiments for Nanocrystal Syntheses: A How-To Guide for Proper Implementation, Chemistry of Materials 34(22) (2022) 9823-9835.

[23] D. Wang, B. Xue, L. Wang, Y. Zhang, L. Liu, Y. Zhou, Fungus-mediated green synthesis of nano-silver using Aspergillus sydowii and its antifungal/antiproliferative activities, Scientific Reports 11(1) (2021) 10356.
[24] Y.-D. Chiang, H.-Y. Lian, S.-Y. Leo, S.-G. Wang, Y. Yamauchi, K.C.W. Wu, Controlling Particle Size and Structural Properties of Mesoporous Silica Nanoparticles Using the Taguchi Method, The Journal of Physical Chemistry C 115(27) (2011) 13158-13165.
[25] B. MacQueen, E. Barrow, G. Rivera Castro, Y. Pagan-Torres, A. Heyden, J. Lauterbach, Optimum Reaction Conditions for 1,4-Anhydroerythritol and Xylitol Hydrodeoxygenation over a ReOx–Pd/CeO2 Catalyst via Design of Experiments, Industrial & Engineering Chemistry Research 58(20) (2019) 8681-8689.
[26] K.-L. Tsui, AN OVERVIEW OF TAGUCHI METHOD AND NEWLY DEVELOPED STATISTICAL METHODS FOR ROBUST DESIGN, IIE Transactions 24(5) (1992) 44-57.
[27] M.N. Pervez, F. Shafiq, Z. Sarwar, M.M. Jilani, Y. Cai, Multi-Response Optimization of Resin Finishing by Using a Taguchi-Based Grey Relational Analysis, Materials 11(3) (2018) 426.
[28] S. Mavruz, R.T. Oğulata, Taguchi Approach for the Optimisation of the Bursting Strength of Knitted Fabrics, Fibres & Textiles in Eastern Europe (2010).
[29] G.R.R. Jagannath, Basawaraj, C.K. Naik Narayana, M. Hulikere Mallaradhya, A. Majdi, M.Q. Alkahtani, S. Islam, Enhancing Wear Resistance of UHMWPE Composites with Micro MoS2 and Nano Graphite: A Taguchi-DOE Approach, ACS Omega 9(14) (2024) 16743-16758.
[30] R. Singh, R. Bhateria, Optimization and Experimental Design of the Pb2+ Adsorption Process on a Nano-Fe3O4-Based Adsorbent Using the Response Surface Methodology, ACS Omega 5(43) (2020) 28305-28318.
[31] M. Outokesh, M. Hosseinpour, S.J. Ahmadi, T. Mousavand, S. Sadjadi, W. Soltanian, Hydrothermal Synthesis of CuO Nanoparticles: Study on Effects of Operational Conditions on Yield, Purity, and Size of the Nanoparticles, Industrial & Engineering Chemistry Research 50(6) (2011) 3540-3554.
[32] S.J. Ahmadi, M. Hosseinpour, F. Javadi, R. Tayebee, Optimization Study on Formation and Decomposition of Zinc Hydroxynitrates to Pure Zinc Oxide Nanoparticles in Supercritical Water, Industrial & Engineering Chemistry Research 52(4) (2013) 1448-1454.

[33] S. Dutta, M. Prabha, R.R.S. Kiran, H. Ashoka, Machine learning guided Box–Behnken design optimized green synthesis of carbon nitride nanoparticles with antioxidant activity and low SH-SY5Y cytotoxicity, Scientific Reports (2026).
[34] J. Moghaddam, S. Kolahgar-Azari, S. Karimi, Determination of Optimum Conditions for Nano-Silver Preparation from AgCl Based on the Taguchi Design by the Use of Optical Properties of Silver, Industrial & Engineering Chemistry Research 51(8) (2012) 3224-3228.
[35] M. Rahimnejad, G. Najafpour, M. Jahanshahi, Evaluation of effective parameters on fabrication of BSA nanoparticles, Nature Precedings (2009).
[36] H.-K. Hwang, S.-J. Kim, Machine learning based optimization of titanium electropolishing using artificial neural networks and Taguchi design in eco-friendly electrolytes, Scientific Reports 15(1) (2025) 28561.
[37] P. Thejasree, N. Manikandan, N. Rajesh, R. Lokanadham, P.C. Krishnamachary, K. Shubham, R.Y. Sembeta, Multi-response optimization of PETG FDM parameters using taguchi–grey relational analysis and perdition by regression modeling, Scientific Reports 15(1) (2025) 41831.
[38] Rohan, A. Sharma, A. Ahmad, A.K. Yadav, Parametric optimization of FSAM-Fabricated Al7075/Graphene/B4C hybrid composites using a Taguchi-ensemble machine learning framework, Scientific Reports (2026).
[39] M.N. Morshed, M.N. Pervez, N. Behary, N. Bouazizi, J. Guan, V.A. Nierstrasz, Statistical modeling and optimization of heterogeneous Fenton-like removal of organic pollutant using fibrous catalysts: a full factorial design, Scientific Reports 10(1) (2020) 16133.
[40] H.F.M. Zaid, M.F. Majid, N.A.A.M. Amin, S.M.M. Ghani, Rapid and deep diesel desulfurization using bimetallic Cu-Fe/TiO2 nano-photocatalysts optimized by response surface methodology, Next Nanotechnology 9 (2026) 100375.

Optimizing the Green Synthesis of Gold Nanoparticles

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Introduction

Gold nanoparticles are used in biomedical and ecological applications. Green synthesis has gained popularity as an eco-friendly altemative to the traditional chemical methods. Green synthesis implores the use of plant extracts; plant extracts contain phytochemicals and other bioactive compounds that are used to reduce metal compounds and stabilize particle size, making this method biocompatible and cost-effective. Green synthesis are their own is ineffective in controlling nanoparticle uniformity and introduces too much variability.

This approach is paired with the Taguchi Design of Experiments (DoE) to optimize parameters while also minimizing experimental runs. This method also aims to increase parameter interactions. The study focuses on four parameters- pH, temperature, reaction time, and Eu concentration. These parameter interactions are evaluated, and nanoparticle formation is measured based on a given set of parameters. This is measured by calculating the absorbance maximum. This method aids in overcoming the shortcomings of the green synthesis model by itself, making the study reproducible and producing gold nanoparticles more efficiently.

Hypothesis

The Taguchi L9 array will optimize the parametes of pH, time, tempature and concentration., effectivly producing stable gold nanoparticles.

Methods

- A Taguchi L9 orthogonal array was used to evaluate four factors at three different levels each.
- The parameters included are pH, EU concentration, temperature (° C), and reaction time (min).
- UV–Vis absorbance (λmax) measured nanoparticle formation.
- The signal-to-noise (S/N) ratio was calculated using the "larger-the-better" criterion.
- Statistical analysis, main effects, interaction plots, regression, residual, and contour models, was performed using Minitab Version 22.

Table 1. Experimental Factors and Levels

Factor	Description	Level 1	Level 2	Level 3
A	EU Concentration (mL)	2	3	4
B	pH	5	7	9
C	Temperature (°C)	40	50	60
D	Time (min)	20	30	60

Methods

Table 2. Taguchi Orthogonal Array

Run	A (EU)	B (pH)	C (Temp °C)	D (Time min)	Output (λmax %)
		5	40	20	1.852
2	2	7	50	30	2.113
3	2	9	60	60	1.710
4	3	5	50	60	1.904
5	3	7	60	20	1.947
6	3	9	40	30	1.981
7	4	5	60	30	2.036
8	4	7	40	60	2.047
9	4	9	50	20	1.811

Table 2. This table shows the four factors- Concentration(A), pH(B), Temperature (C), and time (D), and how they affect the Output(λmax%.)

Results

Figure 3.1. Main effects plots S/ N ratios

Figure 3.1. Main effects plots S/ N ratiosFigure 3.1. Shows how each factor influences the signal-to-noise ratio. The model indicated that pH. The most optimal conditions observed are pH of 7, time of 30 minutes, a concentration of 3mL, and a temperature of 50°C.

Results

Figure 3.2. Interaction Plot for S/N Ratio

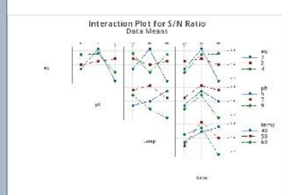

Figure 3.2. Observes how each factor interacts with the other. There is a high level of interdependence among the factors, most notably temperature and time. This model supports the idea that optimal synthesis relies on how the factors work together rather than individually.

Results

Figure 3.3. Fitted Line Plot

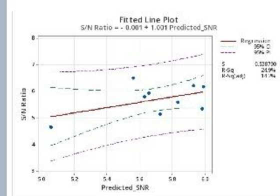

Figure 3.3. Compares experimental and predicted S/N values. An R^2 value of 24.9% was determined, indicating some correlation.

Results

Figure 3.5. Contour Plots

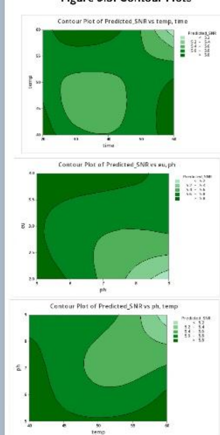

Figure 3.5. Show how one factor interacts with another while keeping other factors constant. These plots help identify critical factors and optimize nanoparticle synthesis.

Discussion and Conclusion

- The most optimal conditions were pH 7, 30+ minutes reaction time, 50° C temperature, and 3 mL concentration, producing the most consistent data.
- pH proved to be the most influential parameter followed by time, concentration, and temperature.
- Interaction plots showed a high level of interdependence among factors, supporting that optimal synthesis depends on factor combinations rather than individual variables.
- The results are consistent with previous studies, pH continues to play a huge role in controlling nanoparticle formation and surface stabilization through bioactive compounds found in plant-based reducing agents.
- This study further validates the use of Taguchi (DoE) as an effective way to optimize nanoparticle synthesis while being replicable and time and cost-efficient.

Future Directions

- Expand Taguchi optmization by adding more parameters to improve predictive stregnth of the model.
- Futher analyzise the most optimal paramers (pH 7, 30+ minutes reaction time, 50° C temperature, and 3 mL concentration) to confitm valadity and reproducbility.
- Apply the gold sythesis technique to produce other types of nanoparticles for comparsion.

References

- 1 Lee, X., Rukayadi, Y., & Lee, M. H. (2016). Green synthesis of gold nanoparticles using aqueous extract of *Garcinia mangostana* fruit peel and their antimicrobial activity. *Journal of Nanomaterials, 2016*, 1–9. https://doi.org/10.1155/2016/8489094
- 2 Mahmud, D. S. M., Shuvo, M. M. R., Rahman, A., & Shuvo, M. F. H. (2025). Statistical optimization of green synthesis of AgNPs and AuNPs using plant extract via Taguchi DoE approach. *Proceedings of the International Conference on Chemical Engineering and Applied Sciences (ICCEAS) 2025*, 1–8.
- 3 Parveen, S., Misra, R., & Sahoo, S. K. (2018). Nanoparticles: a boon to drug delivery, therapeutics, diagnostics and imaging. *Nanomedicine: Nanotechnology, Biology and Medicine, 14*(1), 53–70. https://doi.org/10.1016/j.nano.2017.09.001
1. 4 Patra, A. R., Pattnaik, A., & Ghosh, P. (2025). *The latest breakthroughs in green and hybrid nanoparticle synthesis for multifaceted environmental applications. Journal of the Taiwan Institute of Chemical Engineers.* Advance online publication. https://doi.org/10.1016/j.jtice.2025.106157

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