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**M Yashini**  
Department of Planning and  
Monitoring Cell, National  
Institute of Food Technology  
and Entrepreneurship  
Management-Thanjavur  
Formerly IIFPT,  
Thanjavur, Tamil Nadu, India

**S Shanmugasundaram**  
Department of Planning and  
Monitoring Cell, National  
Institute of Food Technology  
and Entrepreneurship  
Management-Thanjavur  
Formerly IIFPT,  
Thanjavur, Tamil Nadu, India

## Optimization of parameters influencing the electro-deposition of Gold nanoparticles on electrode using Taguchi method

**M Yashini and S Shanmugasundaram**

### Abstract

Electro-deposition of metal nanoparticles is an emerging method that acts as a rapid and inexpensive process for the development of metal-based nanosensors. Metal nanoparticles such as gold nanoparticles (AuNPs) have gained great attention for the sensitive detection of analyte in the sensing field. In this study, Taguchi's design of experiment was employed in designing and optimization of AuNPs electro-deposition process on the electrode, in addition, to examine significant factors influencing the process, as an attempt to achieve maximum peak current from a minimum number of experimental runs. Cyclic voltammetry was applied to assess the influence of parameters on peak current. The scan rate, AuNPs concentration, potential range, and cycles were selected as parameters. A larger Signal-to-Noise ratio was applied to maximize the current response. An optimal combination was scan rate (0.05 V/s), potential range (+0 to +1), cycles (20), and AuNPs concentration (4 $\mu$ M). A significant difference ( $p < 0.05$ ) was observed in parameters such as potential range, scan rate, and cycles in electro-deposition process. A linear regression model was developed and validated successfully. The electrode surface was characterized. The results suggest that the Taguchi method could be used for the optimization of the sensor development process to enhance performance.

**Keywords:** Electro-deposition, gold nanoparticles, taguchi method, cyclic voltammetry

### 1. Introduction

In recent years, the combination of nanotechnology and electrochemistry has made great progress in the area of electrochemical sensors due to the rising interest in the design of novel nano-sensors by developing new electrochemical methods, nanomaterial's and different electrode modification approach for enhancement of sensor performance in terms of sensitivity. A nanomaterial performs a crucial role in sensing through its distinctive optical, magnetic, and electrical properties. Some examples of smart nanomaterials are carbon nanoparticles, metallic nanoparticles, metal oxide nanoparticles, and nanocomposites (Alafeef *et al.*, 2020; Agasti *et al.*, 2010) [3, 2]. Metal nanoparticles are more expedient in electro analytical techniques owing to distinctive properties such as improved electron transfer rate and electro catalytic ability, enhanced mass transport, higher surface area, and better signal-to-noise ratio (Campbell *et al.*, 2010) [5]. Among the metal nanoparticles, gold nanoparticles (AuNPs) have excellent biocompatibility, high conductivity, high surface-to-volume ratio, flexibility in synthesis and functionalization, low toxicity and are easier to detect (Sperling *et al.*, 2008; Tiwari *et al.*, 2011) [26, 28]. AuNPs modified-electrodes have been widely reported in sensing applications due to improved sensitivity, stability and selectivity of the sensor as novel sensing strategies (Saha *et al.*, 2012) [24]. It signifies excellent nanoplatfroms in the fabrication of sensors and biosensors for a variety of sensing applications, including chemical and biological samples. It mainly accelerates the electron transfer by increasing current and decreasing resistance, thereby, improves the sensitivity of a sensor without any enzyme (Sophia & Muralidharan, 2015) [25]. The major challenges of AuNPs modified sensor in the successful real-time application are reproducibility, reliability, scalability in the manufacturing process and stability (Zeng *et al.*, 2011) [32]. Recently, AuNPs are regarded as Nano Zymes as a result of mimicking the ability of horseradish peroxidase enzyme to oxidize peroxidase substrates in the presence of H<sub>2</sub>O<sub>2</sub> to form a colored product. It has promoted the wide application of AuNPs in the bio-sensing field (Lin *et al.*, 2020) [17].

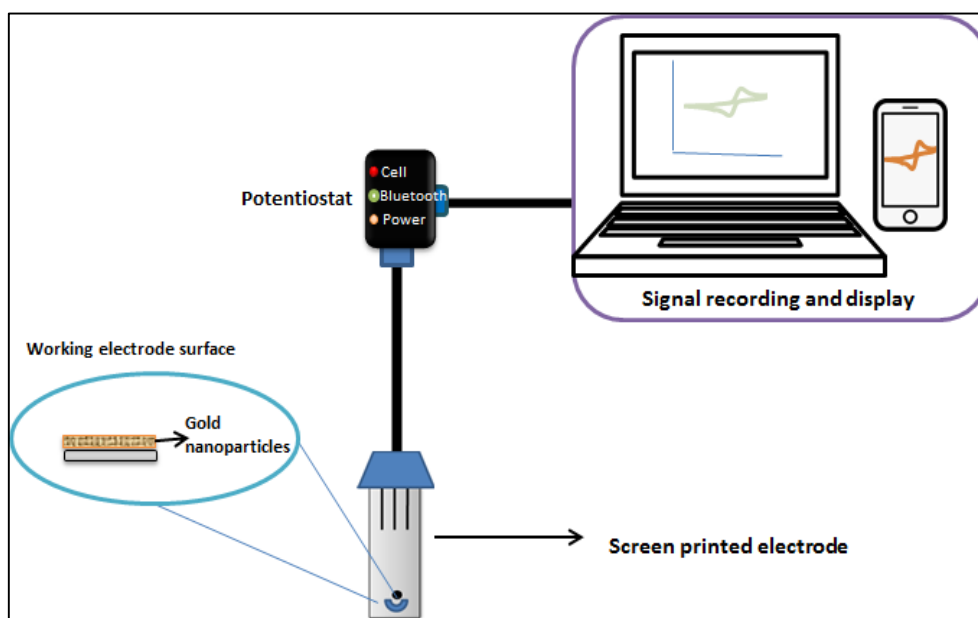
**Corresponding Author:**  
**S Shanmugasundaram**  
Department of Planning and  
Monitoring Cell, National  
Institute of Food Technology  
and Entrepreneurship  
Management-Thanjavur  
Formerly IIFPT,  
Thanjavur, Tamil Nadu, India

AuNPs are a colloidal suspension of nanometer-sized gold particles. It can be prepared by a chemical such as reduction of chloroauric acid in the presence of a stabilizing agent or physical methods such as radiolytic and photolytic methods (Turkevitch *et al.*, 1951; Saha *et al.*, 2012; Abidi *et al.*, 2010; Wang *et al.*, 2009) [29, 24, 1]. Among them, electro-deposition offers an easy, rapid and inexpensive alternate. The noteworthy use of electrochemical deposition is the potential to control the size and distribute nanoparticles by varying the potential, time, or solution concentration (Day *et al.*, 2007; Mohanty *et al.*, 2011) [8, 18]. The mechanism of AuNPs electro-deposition is where free Au (III) ions from solution bind to the electrode surface via electrostatic at first, and then the applied voltage to the electrode supports the reduction of Au (III) ions successively (Chiang *et al.*, 2019) [7]. Cyclic Voltammetry (CV) was selected to electrodeposit AuNPs onto glassy carbon electrode owing to its ability to distinguish between the relative relevance of mass transfer events and heterogeneous electron transport, to identify the presence of intermediates in the Au (III) reduction pathway, to indicate the occurrence of adsorption phenomena and to determine optimal conditions for AuNPs electro-deposition (Hezard *et al.*, 2012) [12]. Moreover, cyclic voltammograms' form is indicative of nucleation and growth activities.

The effect of parameters such as potential range, cycle number, nanoparticles concentration, and scan rate on the electro-deposition process influences the performance of the sensor (Day *et al.*, 2007) [8]. The applied parameters have an impact on the deposition time in the electro-deposition process (Etesami *et al.*, 2011) [10]. Therefore, these factors are essential to be optimized using multivariate analysis because of an individual factor affected by another factor. The design of experiment is best for optimizing the parameters with a

minimum number of experimental runs. In the past few years, the Taguchi-based multivariate (TM) optimization technique has been extensively employed for optimization and along with the comparative effect of the design parameters on the response (Vyas *et al.*, 2019; Mughal *et al.*, 2014) [30, 20]. The TM is an effective tool for examining control parameters to produce the desired results in the process. The signal-to-noise ratio (S/N) is defined as the “logarithmic function for the response of selected parameters” to form the best design. The S/N ratio integrates with the TM and predicts outcomes to attain enhanced performance in the process. It is an ideal way to study the impact of electro-deposition parameters on the response of the process (Mughal *et al.*, 2014) [20]. The features of TM include low cost and time, minimizes the number of runs, investigates individual factors, determines the contribution of a factor, predicts outcomes under optimal conditions, defines the contribution of errors, and finds the optimal conditions for multiple responses at the same time (Koorand *et al.*, 2018) [15].

There was no literature reported on optimization of the electro-deposition process of gold nanoparticles on glassy carbon electrode using Taguchi method. Therefore, this study was designed to examine the effect of selected parameters on electro-deposition of AuNPs using Taguchi model with minimum experimental runs. In this study, four parameters such as potential range (V), gold nanoparticles concentration ( $\mu\text{M}$  in PBS), scan rate (mV/s), and cycles, mixed-level design of one with two and three others with three levels were investigated. Furthermore, the characterization of AuNPs modified- electrode was studied using a Scanning electron microscope (SEM), contact angle and cyclic voltammetry methods.



## 2. Materials and Methods

### 2.1 Materials and apparatus

Gold nanoparticles (suspension in 0.1 mM phosphate buffer solution (PBS), diameter 100 nm), potassium ferricyanide and potassium chloride were procured from Sigma Aldrich. Electrochemical measurements were performed using electrochemical analyzer Sensit BT: SNS configuration (Palmsens 4) using a screen-printed electrode consisting of glassy carbon electrode as the working electrode, platinum

(Pt) wire as the auxiliary electrode and Ag/AgCl as a reference electrode.

### 2.2 Electrode position of AuNPs

Prior to electrode position of AuNPs, the electrode was polished with 0.05 $\mu\text{m}$  and 0.5 $\mu\text{m}$  alumina powders and then washed with ethanol. It was followed by the pre-treatment of an electrode with 0.1 M  $\text{H}_2\text{SO}_4$  in the potential between  $-0.5$  to  $+1$  V with a scan rate of 0.1 v/s for 20 cycles (Elewi *et al.*,

2020<sup>[9]</sup>, with slight modification). Cyclic voltammetry method was utilized in the AuNPs electro-deposition on glassy carbon electrode for a different combination of parameters in Table 1.

**Table 1:** Taguchi orthogonal array L18 (2<sup>1</sup> 3<sup>3</sup>) design of experiments

Run	Variables			
	Scan rate [A] (V/s)	Potential range [B] (V)	AuNPs concentration [C] (μM in PBS)	Cycles number [D]
1	0.05	-5 to 1	2	10
2	0.05	-5 to 1	4	20
3	0.05	-5 to 1	6	30
4	0.05	0 to 1	2	10
5	0.05	0 to 1	4	20
6	0.05	0 to 1	6	30
7	0.05	-1 to 1	2	20
8	0.05	-1 to 1	4	30
9	0.05	-1 to 1	6	10
10	0.1	-5 to 1	2	30
11	0.1	-5 to 1	4	10
12	0.1	-5 to 1	6	20
13	0.1	0 to 1	2	20
14	0.1	0 to 1	4	30
15	0.1	0 to 1	6	10
16	0.1	-1 to 1	2	30
17	0.1	-1 to 1	4	10
18	0.1	-1 to 1	6	20

### 2.3 Design of experiment (D.O.E)

Different potential ranges, scan rates, cycles, and AuNPs concentrations were selected for optimization of the electro-deposition process. Experimental runs were designed via TM orthogonal array of L18 (2<sup>1</sup> 3<sup>3</sup>) using MiniTab 17.0 software. Parameters and levels of the present experiment are shown in Table 1. Then, analysis of variance (ANOVA) was employed in an experiment to determine the significance of parameters on the electro-deposition process. S/N ratio was calculated at each parameter level to identify the optimal level for every parameter to eliminate the inappropriate response caused by the noise factors in the optimization process. This experiment aims to maximize the current response to study the parameter effects. Therefore, the larger S/N is better was selected and was determined using the following equation (Ghiasi *et al.*, 2021)<sup>[11]</sup>:

$$S/N = -10 \log \left[ \frac{1}{n} \sum \left( \frac{1}{y_i^2} \right) \right] \quad [1]$$

Where y = observed data & n = number of observations.

### 2.4 Characterization of electrode

The morphological characteristics of bare and modified electrodes surface were studied using a scanning electron microscope (SEM, Vega3 Tescan) at an accelerating voltage of 10 kV. The AuNPs modified electrode surface was air-dried after electro-deposition process prior to imaging.

Contact angle measurements were carried out by placing 15 μl of a distilled water droplet on the electrode surface and a side view image of the water drop was taken by a GigE vision area scan camera (C1600, Genie color series, DALSA). ImageJ software was used with plugin Drop analysis - Low-Bond Axisymmetric Drop Shape Analysis to measure the

water contact angle of a bare electrode and AuNPs modified electrode. The contact angle measurement was determined at room temperature.

### 2.5 Electrochemical characterization

Cyclic voltammetry method was employed to characterize the bare and AuNPs modified glassy carbon electrode using a three-electrode system in 5mM ferricyanide containing 0.1M KCl from -1.2 to 1.2 V potential at a scan rate of 0.5 V/s. Randles-Sevick equation was used in the determination of the electro-active surface area (Bard *et al.*, 1980):

$$I_p = 2.69 \times 10^5 A n^{3/2} D_R^{1/2} C^{1/2} \quad [2]$$

where  $I_p$ , A, C, n,  $D_R$ ,  $v$  represents peak current (μA), electrode surface area (cm<sup>2</sup>), concentration of ferricyanide solution (mM), number of electrons, diffusion coefficient (cm<sup>2</sup>/s), and scan rate (V/s) respectively.

## 3. Results and Discussion

### 3.1 Design of experiment analysis

The optimum levels of parameters such as potential range, scan rate, cycles, and AuNPs concentrations for AuNPs electro-deposition were investigated through the DOE method. A mixed level design of one parameter with two and three other parameters with three levels based on the L18 (2<sup>1</sup> 3<sup>3</sup>) in Minitab software, were conducted according to Table 1. The main effects and interaction plots of electro-deposition parameters are depicted in Figure 1. As seen, the peak current increases from 71.5 μA at -0.5 to +1V potential to 77.9 μA at +0 to +1V. This might be due to the increase of thickness of AuNPs film on GCE at the application of positive potential compared to negative potential in PBS containing AuNPs. A similar trend was observed in one factor at a time optimization method of AuNPs electro-deposition on the screen-printed electrode for detection of Hydrogen peroxide (Elewi *et al.*, 2020)<sup>[9]</sup>. The peak current was decreased for an increase in scan rate (A) parameter i.e., from 0.05 V/s to 0.1 V/s. For cycles (D) parameter, the maximum mean value of current is obtained for 20 cycles. The maximum current peak was seen when the scan rate was decreased with an increased number of cycles, which could be related to a thicker deposited layer. From the above results, the optimized combination is B (+0 to +1), A (0.05 V/s), D (20), and C (4 μM in PBS). Similar observations of optimal parameters such as potential scan rate and cycles were reported in previous studies (Hong *et al.*, 2013; Tian *et al.*, 2005)<sup>[13, 27]</sup>. A significant parameter is indicated by the highest inclination line in the main plot. The potential (B) parameter exhibited the most significant effect compared to AuNPs concentrations (C).

If the lines in the interaction plots are nonparallel, it indicates the interaction between parameters whereas, if the lines cross, it shows a strong parameter interaction (Kirati *et al.*, 2019)<sup>[14]</sup>. Figure 1 explains the strong interaction between parameters B and D, C and D and weak interaction between parameters A and D; and A and B. The C × D interaction had a significant effect and the C (4 μM) and D (20) combination exhibited the maximum value of peak current. Moreover, the B × D interaction was significant and B (+0 to +1) and D (20) combination offered a good current response. P × S interaction showed the least significant effect.

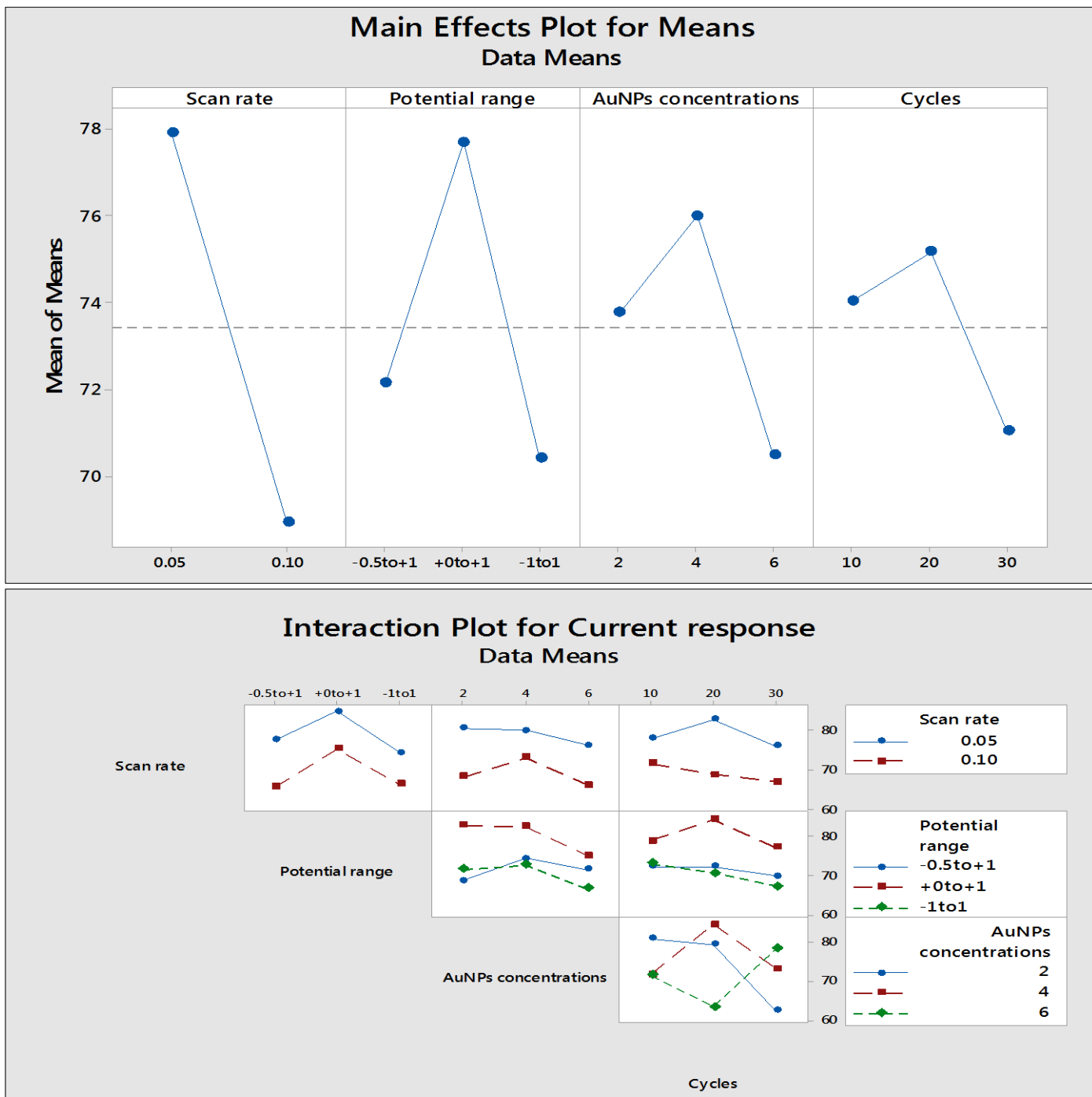


Fig 1: Main effects plot and interaction plot

Table 2 provides the response table, which compares the relative degree of impacts by ranking them according to delta values. The highest and lowest mean difference for each parameter is determined as delta (Nia *et al.*, 2019) [22]. The cycles (D), potential range (B) and scan rate (A) appeared to be the most significant parameters that determine the peak current response of the electrodeposited AuNPs. The current response was least affected by AuNPs concentration (C).

Table 2: Response Table for S/N

Level	A	B	C	D
1	37.62	37.03	37.21	37.17
2	36.92	37.74	37.40	37.69
3		37.04	37.21	36.96
Delta	0.70	0.71	0.19	0.73
Rank	3	2	4	1

**3.2 Analysis of variance**

In Taguchi analysis, the ANOVA test is commonly applied to describe the effective parameters on the average response and S/N ratio. Table 3 shows the ANOVA table based on S/N data to determine significance for each parameter. The ANOVA demonstrates the relative relevance of each parameter using the sequential (Seq) and adjusted (Adj) sum of squares, with the parameter with the highest SS having the main effect. SS values showed that the scan rate (A) parameter had the highest SS value, followed by potential range (B), cycles (D) and AuNPs concentration (C) in that order. Furthermore, the scan rate (A) had the lowest p-value (0.002), followed by the potential range (B), cycles (D), and AuNPs concentration (C), all of which had larger p-values of 0.009, 0.016, and 0.584, respectively. The parameters such as scan rate (A), potential (B) and cycles (D) were found to be significant with  $p < 0.05$

and whereas, the AuNPs concentration (C) with  $p > 0.05$  was not significant. Therefore, scan rate had the greatest impact on charge transfer i.e., peak current value, while concentration had the least impact. These parameters are considered key factors since even the smallest variations in any of them might result in substantial changes in response

**Table 3:** Analysis of Variance (S/N)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	1	2.185	2.185	2.185	16.8	0.002
B	2	2.007	2.007	1.003	7.72	0.009
C	2	0.147	0.147	0.073	0.57	0.584
D	2	1.687	1.687	0.843	6.49	0.016
Residual Error	10	1.300	1.300	0.130		
Total	17	7.328				

**3.3 Linear regression model**

The model equation was developed for the maximum current response. “Larger is the better” S/N was applied for the current response using Equation 1. Equation 3 & 4 denotes the regression equation of current mean and S/N ratio.

$$\text{Current} = 73.4277 + 4.5031A_1 + 1.2557B_1 + 4.2820B_2 + 0.3482C_1 + 2.5931C_2 + 0.631D_1 + 1.7530D_2 \quad [3]$$

$$\text{S/N ratio} = 37.2310 + 0.2495A_1 - 0.2016B_1 + 0.5119B_2 - 0.2864C_1 + 0.6435C_2 + 0.0231D_1 + 0.0636D_2 \quad [4]$$

Where A, B, C and D are the input parameters.

Multiple correlation coefficient ( $R^2$ ) was used to represent model descriptive quality. The  $R^2$  value and adjusted  $R^2$  value of the model were 91.5% and 88%, respectively. Because of the higher  $R^2$  value, it could be ascertained that the experimental and predicted values are very close indicating that the model is significant.

**3.4 Validation and residuals analysis**

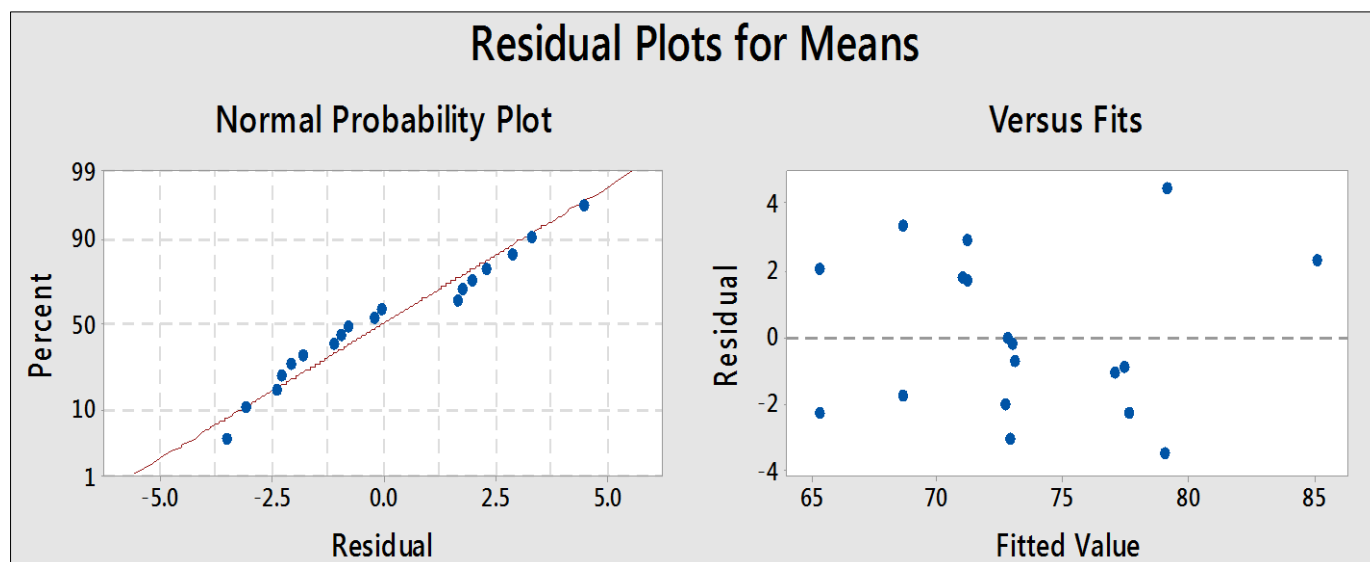
The model was validated with experimental values for the given parameters shown in Table 4. The relative error of current response and S/N ratio calculated for the given conditions was -0.59 and -0.107 percent respectively. The negative sign of relative error showed a good agreement predicted model (Vyas *et al.*, 2019) [30].

**Table 4:** Validation of the model

	Parameters				Experimental	Predicted	Relative Error (%)
	A	B	C	D			
Current	0.05	-5 to 1	2	10	77.200	77.654	-0.59
S/N	0.05	-5 to 1	2	10	37.746	37.786	-0.107

Figure 2 illustrates the residual analysis employed to find out if the model fits the statistical assumptions, i.e., “the residuals are random and normally distributed with a mean of zero and constant standard deviations”, and that was done with a 95% level of confidence (Montgomery *et al.*, 2012). The normal probability plot revealed that they resemble a straight line, indicating that the errors are normally distributed and the

normality hypothesis is satisfied (Figure 2 a). The difference of residuals as a function of fitted values is shown in Figure 2 b. The standardized residuals yielded a randomly distributed scattered point distribution within the range of  $\pm 4$ . The random scattering of residuals around the surface indicated that the model was appropriate, and the independence and constant variance assumptions were not infringed.



**Fig 2:** Residual plots

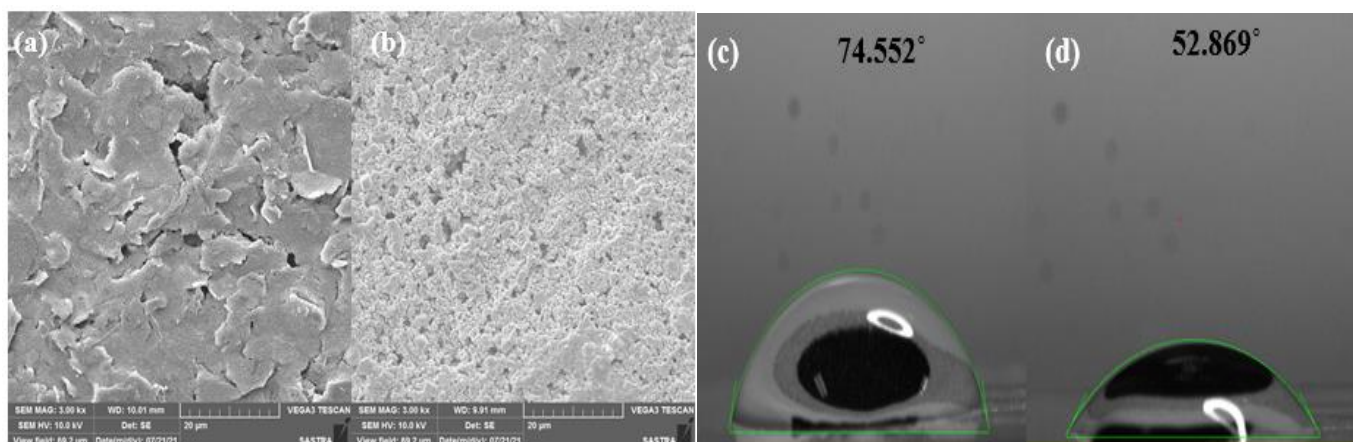
**3.5 Characterization of electrode:**

SEM analysis was performed to characterize the morphology of the electrode surface. The SEM images of a bare electrode and AuNPs modified electrode were shown in Figure 3 (a, b). The differences in SEM images at 3000x magnification were observed between both the electrodes. In the case of bare

electrode, the surface was observed to be rough. The AuNPs electrodeposited electrode surface revealed the presence of AuNPs as a uniform layer onto glassy carbon electrode. A similar observation was reported by Roushani *et al.* (2020) [23]. The hydrophilicity of the sensing electrode surface was studied using contact angle measurement. Figure 3 (c, d)

shows the contact angle of bare electrode and AuNPs modified electrode and their contact angles were  $74.552^\circ$  and  $52.869^\circ$  respectively. Results revealed that the largest contact angle of bare electrode is the expression of hydrophobic property. Consequently, the presence of AuNPs onto the electrode surface showed the hydrophilic property. Lee *et al.*

(2018) [16] observed similar results and stated that the hydrophilic property of AuNPs modified electrode is due to the oxidation and reduction cycles of AuNPs during the electro-deposition process. Overall, the results indicated the presence of the AuNPs after electro-deposition process onto the glassy carbon electrode.



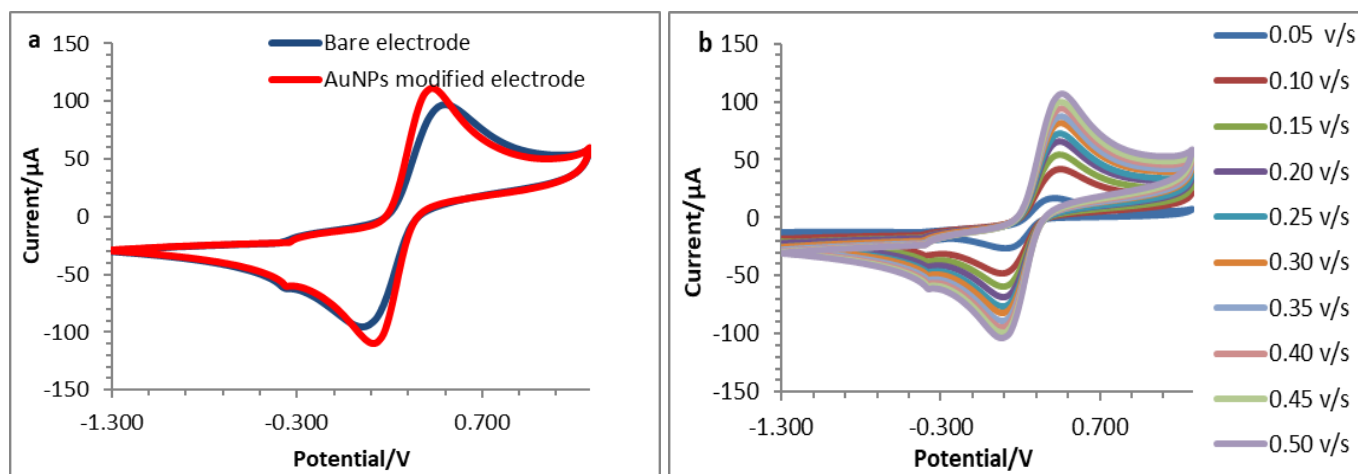
**Fig 3:** SEM images and contact angle images of bare electrode (a,c) and AuNPs modified electrode (b,d) respectively

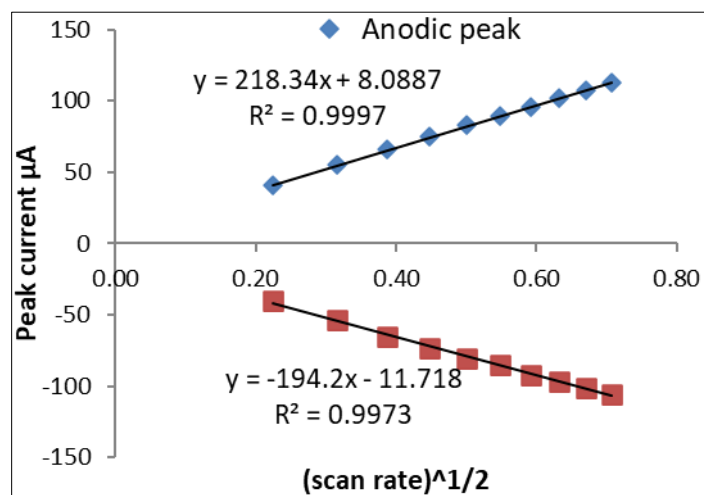
### 3.6 Electrochemical characterization

The electrochemical characteristics of bare and AuNPs modified electrodes were examined using CV responses in potential range from -1.2 to 1.2 V potential range at a scan rate of 0.05 V/s in 5mM ferricyanide containing 0.1 M KCl (Figure 4 a). The redox peaks were observed in this range. Electrochemical response for ferricyanide was found at the bare electrode, with a smaller redox peak current and the largest peak potential separation ( $E_p$ ) of 0.23 V as the slow electron transmission between the electrode surface and the electrolyte solution. An increased peak current and a significant drop in  $E_p$  (0.19 V) of ferricyanide were shown with the AuNPs modified electrode. The rate of electron transport is inversely related to the peak-to-peak separation (Chen *et al.*, 2009) [6]. In this electrochemical reaction, the

electro-deposition of AuNPs increased the electron rate transport as compared to the bare electrode (Elewi *et al.*, 2020) [9].

The slope of the peak current versus (scan rate)<sup>1/2</sup> plot was used to analyze the electrode active surface area. For the bare electrode and the AuNPs modified electrode, the active surface area was  $0.043 \text{ cm}^2$  and  $0.065 \text{ cm}^2$ , respectively. Figure 4 b displays the cyclic voltammograms of AuNPs modified electrode at the scan rate ranged from 0.050 to 0.50 V/s. The square root of the scan rate was shown to be linearly proportional to both cathodic and anodic current peaks. Linear regression equations  $i_{pa} (\text{A}) = 218.34x + 8.0887$ , and  $i_{pc} (\text{A}) = -194.2x - 11.718$  were obtained with correlation coefficients of 0.9997 and 0.9973, confirming the reaction as a surface-confined process. (Figure 4 c)





**Fig 4:** CV of bare electrode and AuNPs modified electrode (a), CVs of AuNPs modified electrode at different scan rates (b) and linear plot between the peak currents versus (scan rate)<sup>1/2</sup> (c)

#### 4. Conclusion

The present study demonstrated the use of the Taguchi method for optimization of AuNPs electro-deposition process to identify the optimal levels of parameters for attaining maximum current response. Potential range, AuNPs concentration, scan rate, and cycles, as well as parameter interactions, have been discovered to show a substantial influence in AuNPs electro-deposition. The optimal levels were obtained as the potential range of +0 to +1V, the scan rate of 0.05 V/s, the cycles of 20, and the AuNPs concentrations of 4 (μm in PBS). Remarkably, scan rate, cycles and the potential were found to be the most significant factors among the others. The model was developed with R<sup>2</sup> of 91.2% and validated by experimental values, resulted in a relative error of -0.58% current and -1.07% S/N. The electrodes were successfully characterized using SEM, contact angle and cyclic voltammetry methods. This study has shown the potential of Taguchi design as a rapid and effective optimization method for electro-deposition of AuNPs on glassy carbon electrodes by simultaneously inferring significant and non-significant parameters.

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