SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Applied Mechanics

Journal homepage:
https://semarakilmu.com.my/journals/index.php/appl_mech/index
ISSN: 2829-7895

Optimization of Electroless Plating Solution Parameter for Coating PETG Electrode with Copper using Design of Experiment (DOE)

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ARTICLE INFO

Article history:
Received 16 July 2024
Received in revised form 18 August 2024
Accepted 25 August 2024
Available online 30 September 2024

Keywords:
EDM; PETG; DOE; electroplating; composite

ABSTRACT

In the study, an attempt was made to electrolessly plate copper onto PETG plastics that aren't conductors. Three manipulating factors, i.e. volume of copper sulphate and sulphuric acid (A), volume of sulphuric acid (B), and mass of copper sulphate (C), were selected and carried out using Taguchi method with three levels to examine their interactions and effects on the responses, including plating thickness, and electrical resistance. The Design Expert 13 software creates a total of nine runs with a single centre point. Three days were spent submerging every electroplated sample part in a different bath solution concentration. The resistance of the metalized Polyethylene Terephthalate Glycol (PETG) component was measured in the meantime using a digital multi-meter. The Cu-deposited PETG was analysed and measured using scanning electron microscopy (SEM). The ideal situation was identified as having 10 possibilities for achieving the goal, based on interaction effects. According to the results of the experiment, run number 3 provides the ideal solution parameter for copper deposition metallization. For the lowest electrical resistance of 0.64 ohm and the highest plating thickness value of 211.49 m, these runs yield the best ideal results. Because each factor reacts to a response individually, the Analysis of Variance (ANOVA) demonstrates that there are little interactions between the factors and responses. The lowest electrical resistance and highest plating thickness values are obtained with the best chemical composition parameter choices.

1. Introduction

Electrical discharge machining (EDM), a common production technique in the mould and die industry, is used to create intricate, deep voids in a variety of materials during roughing and finishing procedures. EDM is a sophisticated machining process that uses electrical sparks to melt and remove conductive materials, primarily hard metals and alloys. Because the tool (electrode) does not come

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https://doi.org/10.37934/aram.125.1.113

into direct contact with the work piece, EDM has a number of advantages in terms of tool wear and surface finish [1].

EDM is well recognised for cutting hard and brittle conductive materials because it can melt any electrically conductive substance, regardless of how hard it is. EDM equipment can drill holes through tool steels, titanium, silver, copper, aluminium, carbide, iron, chromium, cobalt, manganese, and tungsten, as well as any other conductive substance [2].

The workpieces that are machined by EDM are influenced by the materials' melting points, electrical resistance, and heat conductivity. A dielectric fluid, such as kerosene, deionized water, or any other appropriate fluid, is used to submerge both the tool and the workpiece [3]. Because they have a high melting point and good electrical and thermal conductivity, copper, graphite, and copper alloys are employed as electrode materials in EDM. However, producing electrodes is expensive because they must withstand high temperatures and pressures. There is techniquie called metallization through which coating of non-conductive material with conductive material is possible. Through metallization, the physical and mechanical properties of plastics, such as their reflectivity, heat resistance, and strength, can be enhanced or changed. The likelihood of integrating metal and plastic properties in one component material has increased as a result [4,5].

Metallization of the non-conductive electrode core is mandatory to satisfy an electrode's EDM requirements. Metallization is a surface treatment process in which a thin metal layer is coated onto a substrate, component, or part [6]. Its purpose is to improve the structural strength and electrical conductivity of the RT electrode. Metallization can be subdivided into primary and secondary processes. Primary metallization converts the non-conductive surface of an RT electrode core into conductive by inducing a fine layer of a metallic coating onto the substrate's surface. The techniques for primary metallization are electroless plating, spraying, brushing, dipping into metallic paint, and others. Secondary metallization produces a fully functional RT electrode by thickening the initial coating layer. Its metallization technique also depends on the shape of the electrode core. If the electrode core shape is positive, electroplating or spraying is conducted [7].

There are a lot of researcher around the globe working and investigating the an alternative way of replacing the current electrode that is available in the market for EDM. FefarSavan and Karajagikar have attempted the initial study on the sinker EDM machining performance between the metalized FDM and solid copper electrodes on En-19 alloy steel. Their study used acrylonitrile butadiene styrene (ABS) as the electrode core material. 700 μm of the copper is coated onto the cylindrical-shaped FDM electrode core by electroless plating. The Box-Behnken design used EDM parameter settings: peak current, gap voltage, and pulse-on time. The machining time of each electrode is 35 minutes. The experiments showed that peak current significantly affects the MRR, TWR, and SR. They concluded that the machining performance is quite similar between the two electrodes. Lastly, they recommended that complex electrode geometry and broader input parameters be considered in future research [8].

Pawar *et al.,* conducted research similarly to Fefar Savan and Karajagikar. However, their study only tested the ABS-FDM electrode. 1.5 mm of the copper coating was electroplated onto the ABS-FDM electrode with electroless copper plating by Metaffin Industries, Mumbai. Taguchi L9 orthogonal array was used as DOE with input EDM parameter settings of peak current, pulse-on time, and gap voltage. Each electrode was machined on a mild steel workpiece until a 1 mm cut depth was achieved. Peak current was the significant factor affecting the MRR, TWR, and SR [9]. Saxena and Metkar performed identical work as the previous research. 2 mm of the copper coating was electroplated on the square-shaped ABS-FDM electrode by electroless copper plating. The main difference compared to previous research was the Taguchi method applied for sinker EDM parameter settings: current, pulse-on time, and duty factor. They also concluded that the current significantly

affects the MRR, TWR and SR. Lastly, they agreed that this method was feasible to replace the conventional electrode because both electrodes perform similarly to the machining performance of sinker EDM [10].

As for this studies, Polyethylene Terephthalate Glycol (PETG) are chosen as the core material for the electrode. PETG is one of many plastics that can be coated with metals such as copper (Cu), aluminium (Al), gold (Au), and silver (Ag). Due to its excellent electrical conductivity and low cost, copper (Cu) has been researched extensively for metallization and has been plated on a number of polymers. The most popular technique for metallizing plastic is electroless plating since it is straightforward and inexpensive [11]. Using the fused deposition modelling (FDM) approach of the rapid prototyping (RP) method, the effects of electroless copper (Cu) metallization on PETG (Polyethylene Terephthalate Glycol) parts were examined. This metallization technique does not require pricey palladium (Pd) for activation or etching [11].

Therefore the aim of this research is to produce an alternative and working PETG coated copper electrode for EDM using metallization techniques. This electrode expected to perform like the common copper electrode during the EDM machining process.

2. Methodology

2.1 Polyethylene terephthalate glycol (PETG) filament

The 3D drawing of the part were drawn using Solid Work 2019 and based on ASTM B 604–91. Thermoplastic materials such as Polyethylene terephthalate glycol (PETG) are used to print the electrode sample. The FDM 3D printer, 3D V2 Plus machine was utilized to print the electrode samples.

2.2 Aluminium Powder

In this research, Aluminium Metal Powder extra fine were used as the main element for the paste. The details for the Aluminium Metal Powder specification as as follows; Formula = Al 2. CAS No = 7429-90-5 3. M = 26.98 g/mol 4. Apperance: A bright silver gray metals powder 5. Assay (Min.): 99.60% 6. Mesh Size (Passing through 200 mesh) - Min. 93.6%.

3. Experiment Details

The electroless plating on PETG experiment are carried out using a suitable range of parameter settings. The plating thickness and electrical resistance are determined based on the results. The data then sent to DOE software for analysis. The importance of each factors are analyzed by the results of the Analysis of Variance (ANOVA). Next, a confirmation run is performed to validate the experiment's results. Figure 1 shows the flowchart of the experimental work for this research.

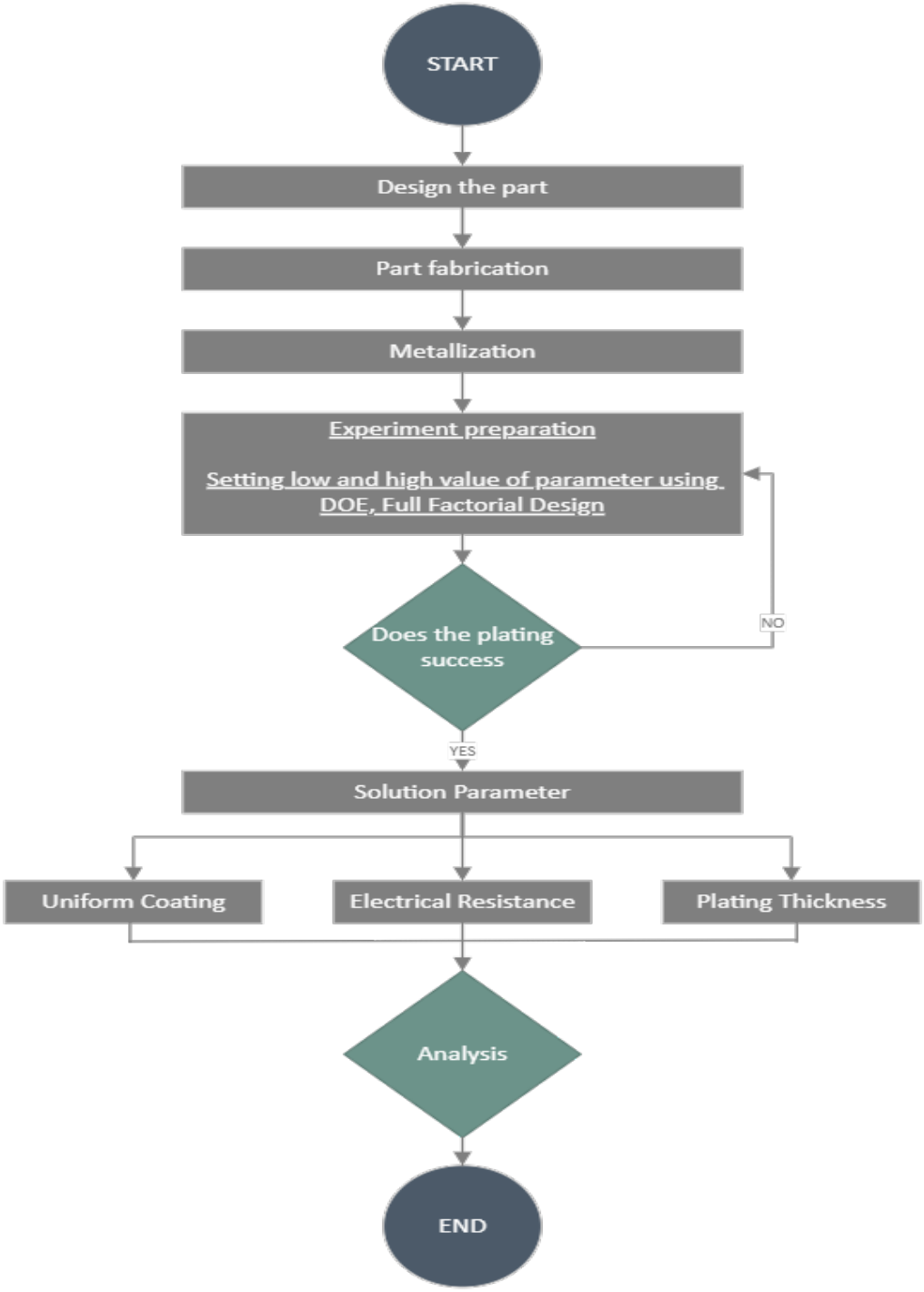

Fig. 1. The experiment flowchart

3.1 Part Fabrication

The parts are fully printed using 3D rapid prototyping from aRaise 3D V2 PlusFDM machine. The material that has been considered is Polyethylene Terephthalate Glycol (PETG). Former stated material is utilized as FDM filament 1.75 mm in diameter. Figure 2 (a) shows the FDM machine used to fabricate the parts and 2(b) electrode fabricated from PETG.

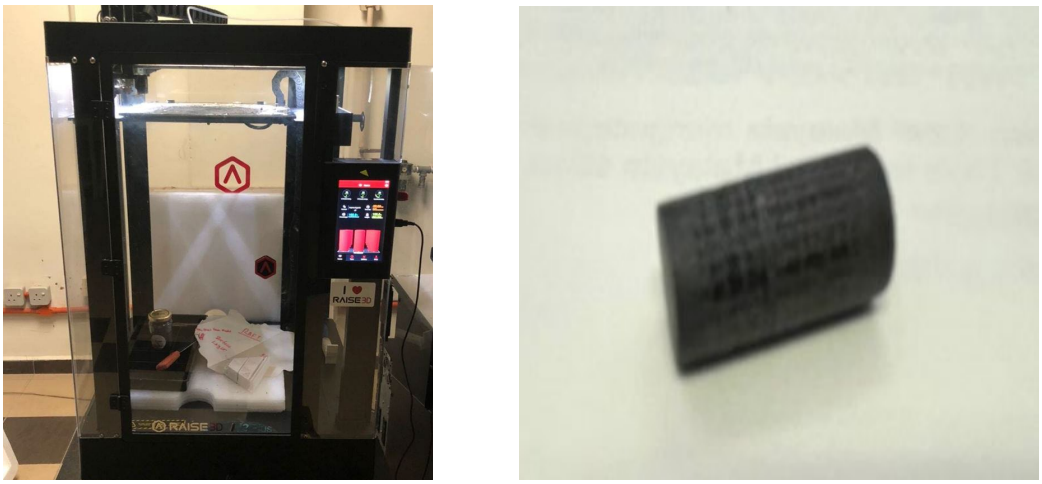

Fig. 2. (a) Raise 3D V2 PlusFDM machine (b) PETG electrode

3.2 Al-C Paste Preparation and Its Application

The various components, such as calcium carbonate, aluminium powder, distilled water, and enamel, are initially considered independently in the following weight ratios: 40:36:21:3. The ingredients are then combined in a 400 ml beaker. A stirrer having glass rod is used to vigorously stir the mixture for about 50 minutes, or until it resembles a paste. Further, it was hand-applied with caution on the PETG pieces. The pasted PETG pieces are all given a full night (12 hours) at room temperature to dry entirely without any surface moisture. To ensure that the outside surfaces of the dried specimens are completely covered in aluminium, they are subsequently sanded with 330-grit sandpaper. The pieces are then carefully washed with water and dried for 45 minutes in an oven that has been prepared to 45 °C. This scouring is comparable to the necessary etching process in conventional electroless techniques.

3.3 Electroless Copper Deposition

Copper sulphate (CuSO4), sulphuric acid, and other electroless baths were combined for copper metallization (H2SO4). The pre-treated PETG components were subsequently submerged for 72 hours (3 days) at room temperature in these electroless copper deposition baths. The Cu-deposited PETG components underwent a water wash before drying for 45 minutes in an oven set to 45 °C. The copper deposited PETG samples were removed, washed with water, and baked to dry them. With the aid of a linear precision saw machine, they have been cut into half pieces.

3.4 Electrical Resistance Measurement

A digital multi-meter (VC830L) is considered for testing the opposition of the metalized PETG part at various locations on its face for various electroless baths in various deposition baths. The average resistance per unit area of the PETG surface was calculated. All of the above deposition techniques are compared to see which one gives the lowest standard deviation and average resistance value.

4. Results

4.1 Experimental Results for Electrical Resistance by Using 3 Level Factorial Design

The experiment involved three factors and consisted of three levels of each factor; a single replication plus one centre point. So, there are eight two-level factorials and one centre point in this experiment, for a total of nine runs. Design expert software version 13 was employed to plan and design experimental runs and analyse the responses from the experiments. The experimentresults for electrical resistance for 9 runs are summarised in Table 1. Five readings were taken to get the average.

Table 1
Experimental results for electrical resistance

Run	Total Volume of Copper Sulphate and Sulphuric Acid (ml)	Volume of Sulphuric Acid (ml)	Mass of Copper Sulphate (g)	Average Electrical Resistance (ohm)
1	50	7.5	17.5	0.62
2	40	5	20	0.58
3	60	5	15	0.64
4	60	10	20	1.24
5	60	5	20	1.50
6	40	5	15	1.60
7	40	10	20	1.62
8	60	10	15	1.66
9	40	10	15	1.74

The conductivity of each sample can be used to comprehend the expansion and allocation of the copper on the samples. The amount of copper that deposits on the samples increases with the length of time that they are submerged in the acidic solution. The average electrical resistance rose with each run, as can be shown. The electrical resistance measured at run number 1 was 0.62 ohm, whereas run number 9 measured 1.74 ohm.

In order to replace the conductive plastic shell of PETG parts with a non-conductive one, copper sulphate and sulfuric acid baths were effective due to the plentiful supply of hydrogen. Copper ions are discovered to be particularly stable in strong sulfuric acid due to the rich accessibility of hydrogen [12]. Additionally, the production of copper ions in sulphuric acid is facilitated by the rapid dissociation of the solution, and the barrier of the electrolytic solution to copper ion transfer to the plastic substrate decreases as more and more ions are produced. Electrical performance measurements revealed that copper crystal size and conductivity varied at various locations on the sample surface and improved as deposition time was maximised [13].

4.2 Analysis of Variance (ANOVA) for Electrical Resistance

The ANOVA analysis for the electrical resistance response is displayed in Table 2. The F-ratio with the necessary degrees of freedom at a predetermined significance level is compared to the ratio of the regression sum of squares to the error sum of squares in order to determine the significance of the regression model. At a 95% confidence level, models in the ANOVA table with values of "Prob > F" equal to or less than 0.05 are considered significant.

Table 2
ANOVA analysis for the electrical resistance

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1.38	3	0.4588	24.22	0.0005	Significant
B-Volume of Sulphuric Acid	0.4704	1	0.4704	24.84	0.0016	
AC	0.3121	1	0.3121	16.47	0.0048	
ABC	0.5941	1	0.5941	31.36	0.0008	
Curvature	1.32	1	1.32	69.47	< 0.0001	
Residual	0.1326	7	0.0189			
Lack of Fit	0.1326	4	0.0332			
Pure Error	0.0000	3	0.0000			
Cor Total	2.83	11				

Electrical resistance's ANOVA and regression summary are shown in Table 3. Predicted R-squared values are 0.9121, 0.8745, and 0.6485, respectively, while adjusted R-squared values are 0.8745 and 0.6485. As one might typically anticipate, the discrepancy between the projected R2 of 0.6485 and the modified R2 of 0.8745 is greater than 0.2. This might point to a substantial block effect or suggest a potential issue with the model and/or data. Model reduction, response transformation, outliers, etc. are things to think about. Confirmation runs should be used to test all empirical models. With sufficient accuracy, the signal-to-noise ratio is measured. The ideal ratio is at least 4. The ratio of 11.5936 indicates a strong enough signal. Use this model to navigate the design space.

Table 3
Electrical resistance's ANOVA and regression summary

Response Model	Quadratic Model
R-Squared	0.9121
Adjusted R-squared	0.8745
Predicted R-squared	0.6485
Adequate Precision	11.5936

4.2.1 Main effect plots

Figure 3 shows the main effect plot for electrical resistance. During this process, smaller is better has been adopted for the analysis. Based on the selection, best outcome comes with A1B3C1 i.e. 60 mL coper sulphate and sulphuric acid, 5.0 mL of sulphuric acid and 20.0 g of copper sulphate.

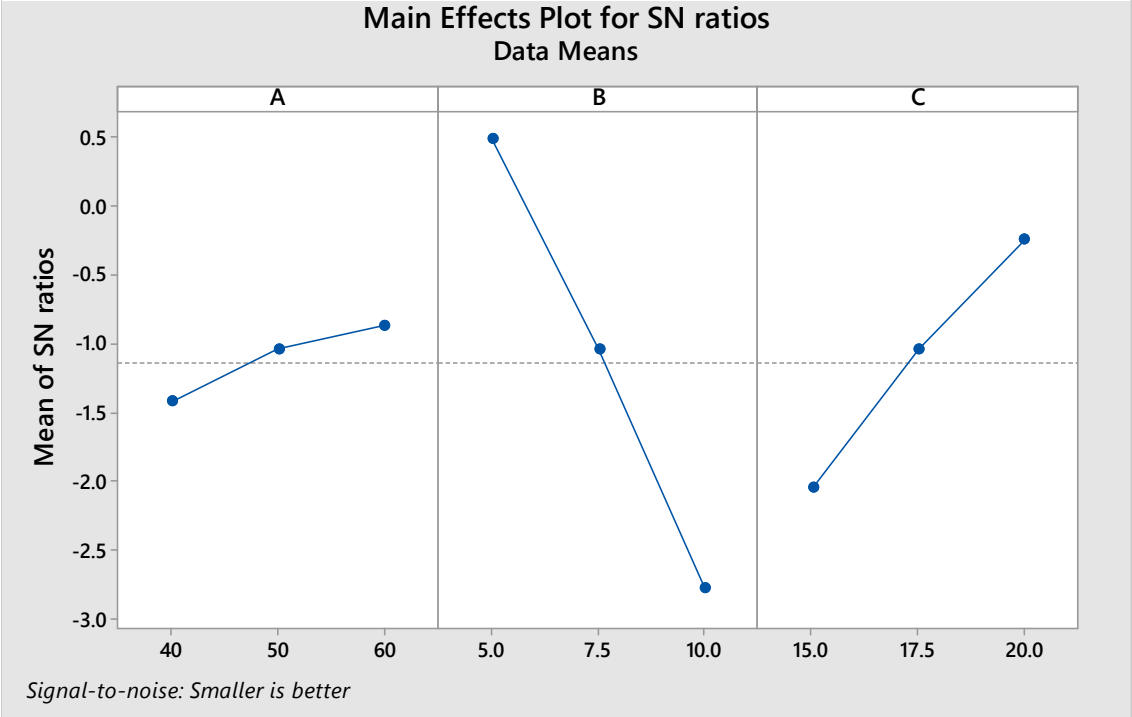

Fig. 3. Main effect plot for electrical resistance.

4.3 Experimental Results for Plating Thickness by using 3 Level Factorial Design

It has been decided to investigate the response regarding plating thickness. The nine readings were taken using the microscopic examination to get the average. We noticed that the plating thickness along the covered PETG was inconsistent. The response result for average plating thickness is shown in Table 4.

Table 4
Experimental results for plating thickness

Run	Total Volume of Copper Sulphate and Sulphuric Acid (ml)	Volume of Sulphuric Acid (ml)	Mass of Copper Sulphate (g)	Average Plating Thickness (µm)
1	50	7.5	17.5	199
2	40	5	20	219
3	60	5	15	211
4	60	10	20	227
5	60	5	20	258
6	40	5	15	224
7	40	10	20	181
8	60	10	15	199
9	40	10	15	170

Table 4 shows that run number 7 resulted in the lowest plating thickness, which is 181 µm. While waiting, run number 5 recorded the greatest plating thickness at 258 µm. The biggest volume of copper sulphate and sulphuric acid, as well as the highest mass of copper sulphate, contributed to the thickest plating, it can be seen. On the other hand, a thinner plating layer is produced by using less copper sulphate and sulfuric acid in both volume and mass. This happens because the process slows down when the acidic bath's volume decreases. The concentration of the acidic bath is decreasing as a result. The blue tint of the copper sulphate and sulfuric acid solution disappeared as

a result. The sluggish deposition of copper is due to the fact that the copper ions in the solution decrease as the concentration of the acidic bath rises [14].

Copper sulphate solution, which is used for copper deposition, is extremely soluble in sulfuric acid solution. As a result, when the time deposition increased, the copper deposition also rose. The size of Cu crystals continues to expand as the deposition time increasingly lengthens. When the deposition time was greater, massive, homogeneous Cu crystals with metallic sheen were seen to develop on the samples [15,16].

4.4 Analysis of Variance (ANOVA) for Plating Thickness

Table 5 displays the ANOVA table for plating thickness responses. The significance of the regression model is evaluated by calculating the ratio between the regression sum of squares and the error sum of squares and comparing the result to the F-ratio with the required degrees of freedom at a predetermined significance level. Models in the ANOVA table with "Prob > F" values equal to or less than 0.05 are regarded as significant at a 95% level of confidence.

Table 5
ANOVA analysis for the plate thickness

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	4948.06	4	1237.01	17.08	0.0020	Significant
A- Total Volume of Sulphuric Acid and Copper Sulphate	1280.18	1	1280.18	17.68	0.0057	
B-Volume of Sulphuric Acid	2261.95	1	2261.95	31.24	0.0014	
C-Mass of Copper Sulphate	824.18	1	824.18	11.38	0.0150	
AC	581.75	1	581.75	8.03	0.0298	
Curvature	378.42	1	378.42	5.23	0.0623	
Residual	434.49	6	72.42			
Lack of Fit	434.49	3	144.83			
Pure Error	0.0000	3	0.0000			
Cor Total	5760.97	11				

The ANOVA and a summary of the electrical resistance's regression are shown in Table 6. Predicted R-squared values are 0.4260, 0.8655, and 0.9193, respectively, while adjusted R-squared values are 0.8655 and 0.9193. As one might typically anticipate, the inconsistency among the expected R2 of 0.4260 and the modified R2 of 0.8655 is greater than 0.2. This might point to a substantial block effect or suggest a potential issue with the model and/or data. Model reduction, response transformation, outliers, etc. are things to think about. Confirmation runs should be used to test all empirical models. With sufficient accuracy, the signal-to-noise ratio is measured. The ideal ratio is at least 4. The ratio of 13.1670 indicates a strong signal. Use this model to navigate the design space.

Table 6
Plate thickness ANOVA and regression summary

Response Model	Quadratic Model
R-Squared	0.9193
Adjusted R-squared	0.8655
Predicted R-squared	0.4260
Adequate Precision	13.1670

4.4.1 Main effect plot for plating thickness

Figure 4 shows the main effect plot for plating thickness. During this process, smaller is better has been adopted for the analysis. Based on the selection, best outcome comes with A3B1C3 i.e. 60 mL coper sulphate and sulphuric acid, 5 mL of sulphuric acid and 20 g of copper sulphate.

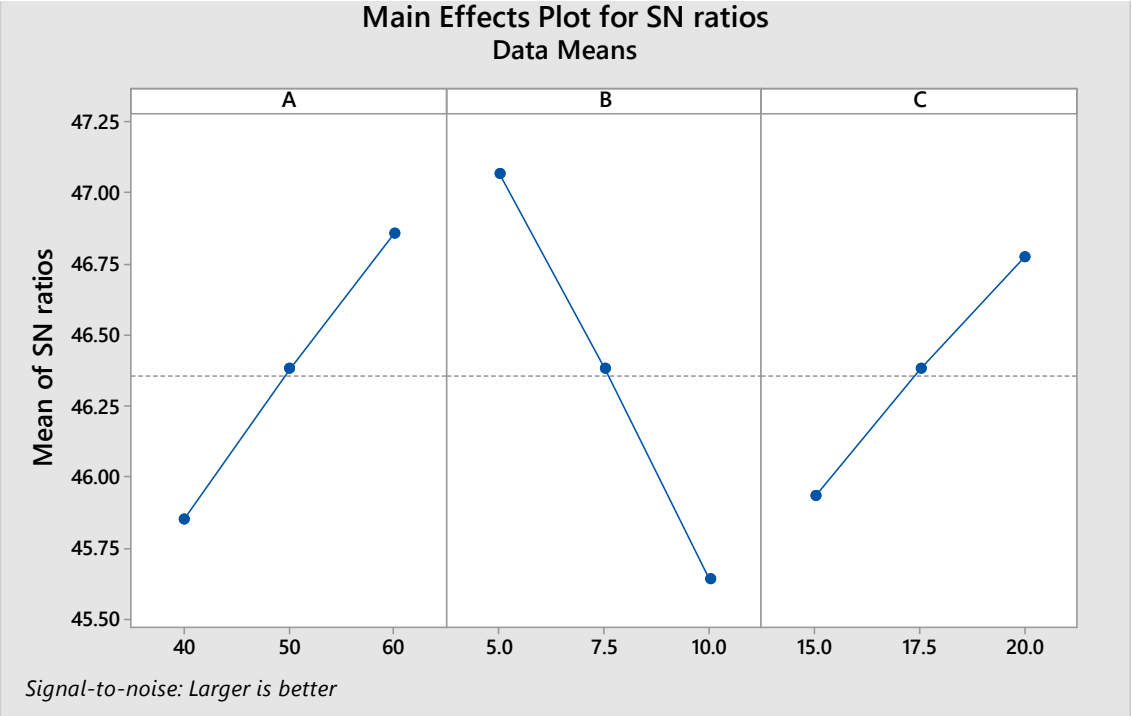

Fig. 4. Main effect plot for plating thickness

4.5 Copper Plating Thickness

Once all 9 samples are successfully plated with copper, the thickness achieved during the electroplating process is investigated under the microscope. The plating thickness was investigated under magnification of 200 µm. Figure 5 below shows the magnification of all the samples and their average thickness. It can be seen that the highest plating thickness achieved is 258µm from sample 5 meanwhile the lowest plating thickness recorded is 170 µm from sample 9. The higher the plating thickness is better due to the EDM process itself that erodes the electrode during the machining process. From this observation, it proves that the Volume of Copper Sulphate and Sulphuric Acid (A) and the Mass of Copper Sulphate (g) (C) that are set at the highest value contributed to the highest plating thickness. Inversely, by decreasing the Volume of Copper Sulphate and Sulphuric Acid (A) and the Mass of Copper Sulphate (g) (C), lead to a lower plating thickness.

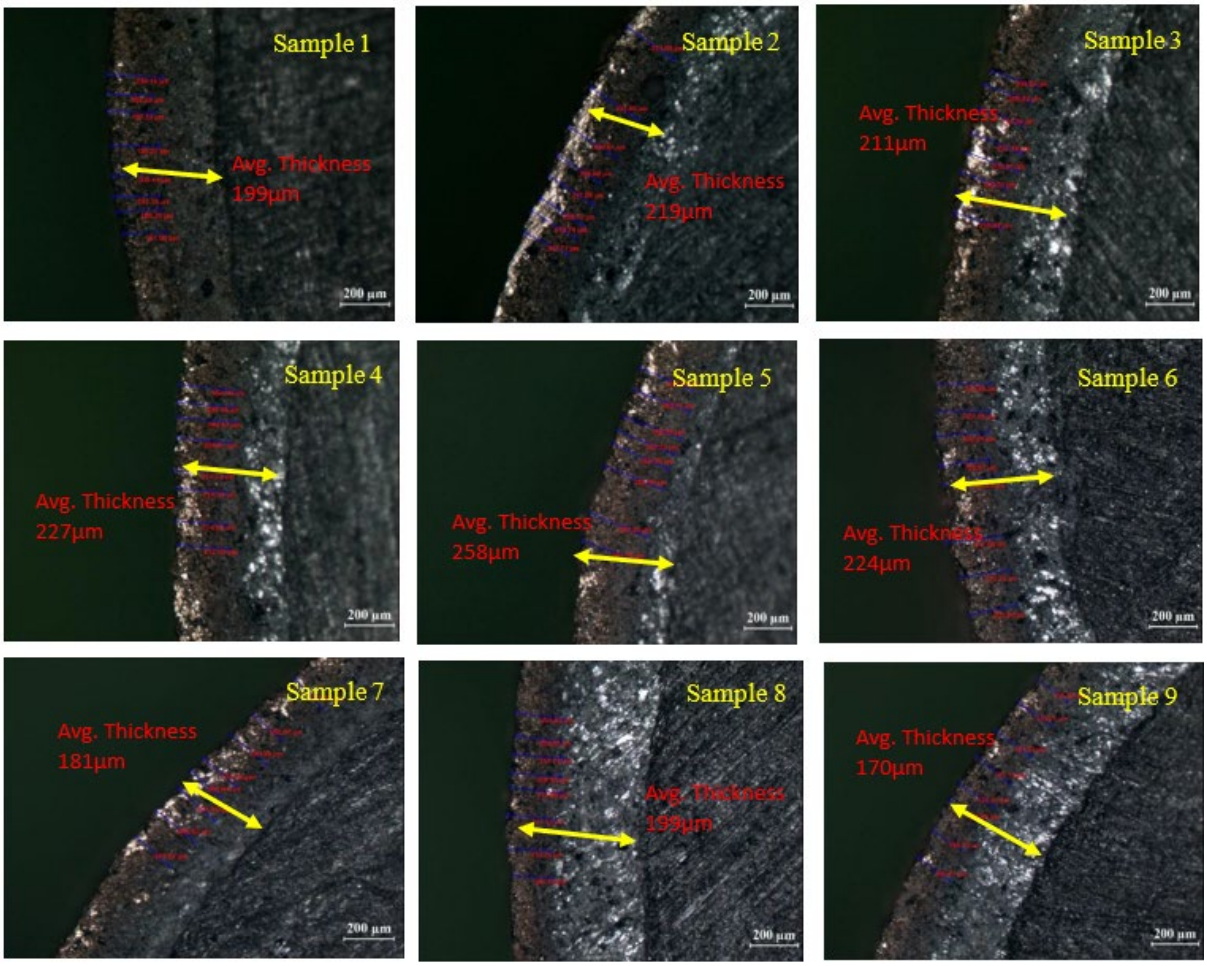

Fig. 5. Average plating thickness for all experiments

4.6 Optimization Condition

Ten parameter settings with various values are offered based on the findings. Due to the near proximity of all values to 1, the combination of all recommendations has a specific attractiveness value. Table 7 displays the recommended setup parameter for the lowest electrical resistance and maximum plating thickness value.

Table 7
Suggested parameter for lowest electrical resistance and maximum plating thickness value

Run	Total Volume of Copper Sulphate and Sulphuric Acid (ml)	Volume of Sulphuric Acid (ml)	Mass of Copper Sulphate (g)	Plating Thickness (µm)	Electrical Resistance (ohm)	Desirability
1	60	5	15	221.950	0.610	0.759
2	60	5	15.019	222.095	0.614	0.759
3	60	5	15.042	222.265	0.618	0.759
4	60	5	15.077	222.525	0.624	0.759
5	59.923	5	15	221.918	0.614	0.758
6	60	5	15.184	223.323	0.645	0,757
7	60	5.030	15	221.746	0.616	0.756
8	60	5	15.400	224.938	0.685	0.754
9	59.645	5	15	221.804	0.627	0.753
10	59.650	5	15.026	221.999	0.631	0.752

4.7 Confirmation Runs

Confirmation runs are performed to compare the results of electrical resistance and plating thickness between theoretical and experimental values. Ten runs are recommended from the optimization two-level factorial design to reach the goal. Run 1 was chosen for the confirmation run to determine the margin error value from the parameter range shown in Table 7. The outcome of the confirmation runs is shown in Table 8.

From the table it shows that the average error for both responses, Electrical Resistance and Plating Thickness, is less than 2%. The percentages are less than 15%, and the results of the conformation run were valid for the existence of an ideal point [17,18].

Table 8
Confirmation runs results

Run	Plating Thickness (µm)			Electrical Resistance (ohm)		
	Actual	Predicted	Error (%)	Actual	Predicted	Error (%)
1	220.105	221.950	1.845	0.550	0.610	0.06
2	221.895		0.055	0.575		0.035
3	220.240		1.71	0.602		0.008
4	221.065		0.885	0.501		0.109
5	221.255		0.695	0.628		0.018
	Average error (%)		1.038	Average error (%)		0.046

5. Conclusions

According to the study, the variables that had a significant impact on the electrical resistance response were the volume of sulphuric acid (B), the mass of copper sulphate, and the total volume of copper sulphate and sulphuric acid with volume of sulphuric acid with mass of copper sulphate (AC) (ABC). The mass of copper sulphate (C), the total volume of copper sulphate and sulphuric acid (A), and the total volume of copper sulphate and sulphuric acid combined with the mass of copper sulphate all have substantial impacts on plating thickness (AC). In terms of plating thickness and electrical resistance, the optimal chemical solution parameter for metallization is found, and run number 3 provides the best parameter settings for the metallization of copper deposition. This experiment results in the least electrical resistance and maximum plating thickness. The value of the inaccuracy from the confirmation run is below 15%, which is acceptable and confirms the electroless plating's ideal state. From this research, an alternative and working PETG coated copper electrode for EDM using metallization techniques successfully fabricated. It is expected that this electrode to perform like the common copper electrode during the EDM machining process.

Acknowledgment
The authors would also like to thank the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia and the Research Enhancement Graduate Grant (RE-GG), VotQ085 for its support. Communication of this research is also made possible through monetary assistance from Universiti Tun Hussein Onn Malaysia.

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