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Performance evaluation of a simple electrochemical treatment model for saline wastewaters: Part B

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ABSTRACT

This paper investigated the performance of the electrochemical treatment technique in removing chloride from saline wastewater (brine) with the critical objective of purifying the wastewater, evaluated the efficacies of selected mathematical models and particular attention to selected polynomial regression models as a follow-up to previous studies. The saline wastewaters were prepared and subjected to electrochemical treatment using developed carbon–resin (anode) and aluminium (cathode) electrodes. Electrochemical treatment of the synthesised saline wastewaters (between 10 x 10^3 mg/l and 40 x 10^3 mg/l of chloride) was conducted on a laboratory scale. The influences of selected or picked-out operational factors on the functioning or efficacy of the electrochemical purification process of the wastewater were monitored using fractional factorial experiments. Three mathematical models were formulated using Microsoft Excel Solver and evaluated statistically. The study revealed that the current, the time and the interval distance between the electrodes were significant and vital factors that impacted on the performance of the electrochemical purification treatment of brine. The factors with negative special effects on the performance of the treatment process of brine were separation distance between the electrodes, pH, the depth of the electrode, the initial and primary concentration of the chloride and the flow and discharge rate of the wastewater. The performances or efficacy of the polynomial regression models in predicting the performance of the treatment technique were with average errors of 2.99%, 2.97% and 2.94% and accuracy of 97.01%, 97.03% and 97.06% for Models A, B and C, respectively. It was concluded that the electrochemical treatment of brine with carbon-resin electrodes is efficient in removing chloride from brine and the selected models predicted the performance of the treatment technique well.

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INTRODUCTION

The world is facing problems with different solutions in relation to the provision of potable water through water treatment techniques, which are meant at producing safe water and protecting the environment [1]. Presently, a vital research focus is on the next-generation wastewater and water treatment techniques to solve global potable water shortage and pollution issues [2]. In the case of conventional water treatment techniques, their performances in

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removing emerging pollutants from aqueous are not dependable. These conventional water and wastewater treatment techniques are categorised as physical (screening, filtration, sedimentation, floatation, evaporation, distillation and aeration), chemical (coagulation, flocculation, precipitation, adsorption, chlorination), biological and advanced. In the present-day water treatment techniques for saline water and wastewater are membrane-based techniques, irradiation, electric-current-based techniques and a combination of two or more of these conventional techniques. Among these present-day water and wastewater treatment techniques, membrane and electric-current-based (electrochemical) techniques play a prevailing role in the worldwide market and engineering applications. In line with the comparative advantages of electric-current-based and membrane-based techniques of higher and dependable efficiency, higher energy utilization efficiency and lower footprint [2]. Among these two techniques (electric-current-based techniques and electric-current-based techniques), electric-current-based techniques have attracted wider attention [1] than membrane-based techniques, based on some limitations and disadvantages of membrane techniques [3]. In the last three decades, the electrochemical technique of treating water and wastewater has been suggested as a substitute process for the removal of contaminants in wastewater or effluents discharge into the environment. These electrochemical treatment techniques have shown reliable performance results in several matrices of polluted and contaminated wastewater such as herbicides and pesticides, textile dyes, dairy, pulp and paper, heavy metals, landfill leachate, aquaculture, pharmaceutical residues, and other industrial effluents or wastewaters [1]. In addition, a wide range and varieties of electrode substances and materials have been utilised and suggested in electrochemical treatment techniques. Some of the utilised and suggested electrodes(active, non-active or passive) are noble metals (silver, platinum, gold and graphite), , dimensionally stable anodes, PbO_2 based, carbon or graphite-based anodes, and Boron Doped Diamond (BDD). These utilised and suggested electrodes have attained different removal and reduction efficacies of organic matter [1]. These non-active anodes, which include BDD, are beneficial for direct electrooxidation of organic material through hydroxyl radicals. The Dimensionally Stable Anode (DSA) another non-active electrode, which includes Ti and IrO_2 with Ta_2O_5, are active in enhancing hypochlorite-mediated chemistry in the presence of chloride ions. The anodic electrooxidation of brine created by petroleum exploration of the Petrobras plant in Brazil utilises an electrochemical reactor with electrode made of a Ti and RuO_2 with TiO_2 and SnO_2 was recently evaluated [1]. The evaluation was under the current density of 89 mA cm^{-2} (galvanostatic conditions and situations). The study confirmed that the degradation of the organic pollutants at different discharge and flow rates (1.3, 0.8, 0.5 and 0.25 dm^{-3} h) attained the removal efficacies of 84%, 95%, 97% and 98%, respectively. Da Silva et al. [1] reported that the electrooxidation (electrochemical oxidation) of brine in galvanostatic situations and conditions utilising platinum supported on titanium (Ti and Pt) and BDD anodes, in a batch reactor. The results showed that complete Chemical Oxygen Demand (COD) removal was achieved using BDD electrode. The study stated that the production of high amounts of hydroxyl radicals ($OH^{\cdot}$) and oxidizing species (Cl_2, HClO, ClO^{-}) aided the performances. Utilization of these electrooxidation techniques and materials to create very strong oxidant materials and species (such as chlorine) has been anticipated due to the electrocatalytic characteristics of these treatment techniques and materials. Several studies have reported the treatment and remediation of petrochemicals and brine effluents in the literature. The other treatment techniques for the treatment of brine can be summarized as follows:

a) Biological treatment techniques as indicated in Akyon et al. [4]; Baptista et al. [5]; Beneduce et al. [6]; Kargi and Dincer [7]; Zhang et al.[8] and Ziemkiewicz et al. [9],

b) Electrochemical, electrocoagulation and other electric current techniques as documented in Soni et al. [10]; Madrona et al. [11]; Al-Raad et al. [12]; Al-Raad et al. [13] and Ayadi et al. [14]

c) Photocatalysis as highlighted by Andreozzi et al. [15]; Feroz [16] and Ye [17];

d) Soil remediation as indicated in Ekama et al. [18]; Estabragh et al. [19]; and Jiang et al. [20];

e) Electromagnetication techniques as documented in Hachicha et al. [21];

f) Chemical as highlighted by Jin et al. [22]; Kaith et al. [23]; Pfennig et al. [24]; and Shrivastava [25]; and

g) The membrane as indicated in Zhang et al. [26]; Xu et al. [27] and Yue et al. [28].

More information and data on the treatment of brine wastewater and water are established in the literature and can be summarized as follows:

a) Treatment using osmotic agent in water flux enhancement during osmotic membrane distillation (OMD) for treatment of highly saline brines osmotic agent in water flux enhancement during osmotic membrane distillation (OMD) for treatment of highly saline brines and a microbial desalination cell for sustainable wastewater treatment and saline water desalination, in Zhang et al. [8], and Zhang et al. [26];

b) Treatment using Carbide coated tools in Yusof et al. [29]; Adsorption techniques in Aber and Sheydari [30];

c) Treatment of the Brine Generated from Reverse Osmosis Advanced Membrane Wastewater Treatment Plant Using Epuvalisation System. Qurie et al. [31];

d) Electrochemical Catalytic Oxidation Treatment of Coking Wastewater RO Brine in Wang et al. [32];

e) Innovative Application of Water Quality and Flow Modeling to Design a Softening, UF/RO and Brine Handling System for Copper and Gold Mining Wastewater Treatment in the Peruvian Andes in Burbano et al. [33];

treatment of meat industry wastewater using electrochemical treatment method in Thirugnansanhanghan et al. [34];

f) Bioelectrochemical treatment of table olive brine processing wastewater for biogas production and phenolic compounds removal in Marone et al. [35];

g) Treatment of a hypersaline brine, extracted from a potential CO_2 sequestration site, and an industrial wastewater by membrane distillation and forward osmosis in Salih et al. [36];

h) Treatment of brine wastewater through a flow-through technology integrating desalination and photocatalysis in Ye et al. [37];

i) Assessment of three brine recycle humidification-dehumidification desalination systems applicable for industrial wastewater treatment in Ghofrani and Moosavi [38];

j) Membrane distillation treatment of municipal wastewater desalination brine and its mitigation by foam fractionationin Rajwade et al. [39];

k) Sonophotocatalytic treatment of AB113 dye and real textile wastewater using ZnO/persulfate: Modeling by response surface methodology and artificial neural network in Asgari et al. [40];

l) Removal from aqueous solutions using ionic liquid-modified magnetic activated carbon in Bazrafshan et al. [41]; and

m) Techno-economic assessment of minimal liquid discharge (MLD) treatment systems for saline wastewater (brine) management and treatment in Panagopoulos [42].

With reference to the importance of these electrochemical treatment techniques in removing both conventional and emerging pollutants from aqueous solutions the performance of the techniques in removing chloride from bine wastewater and polynomial models that relate and simulate operational factors to their removal efficacies are limited in the literature. The main objectives of the current study are to evaluate the performance electrochemical process (using carbon–resin and aluminium electrodes) in removing chloride from saline water, to establish mathematical (polynomial regression form) models that relate the performance of the system to selected operational factors and to evaluate accuracy of these models statistically.

MATERIALS AND METHODS

Carbon-resin or graphite-resin electrodes (CRE) were produced from wasted dry cells. The discarded and spent dry cells were collected and picked from several dumpsites located in Nigeria. These collected dry cells were segmented and carbon (graphites) were separated from these cells and crushed. Powdered carbon was separated into different British standard particle sizes. A fixed mass of the powdered carbon was mixed with resin (organic binder), and moulded into 25- a millimetre diameter, 100-millimetre-long electrode utilising a plunger and extruder, with a compaction or compressive device. Details of the development and characteristics of the electrodes were presented in previous and other studies as follows:

a) Development, stability and properties of carbon-resin electrode Oke et al. [43];

b) Orthogonal experiment in the development of carbon-resin electrode Oke [44];

c) Establishment of factors that influence stability and properties of carbon-resin electrode Oke et al. [45];

d) Properties of doped carbon-resin electrode in Oke et al. [46]; aluminium and Calcium oxide doped Oke et al. [47];

e) Utilization of Weibull distribution in the development of carbon-resin electrode in Oke et al [48];

f) Effects of carbonization on stability and electrical conductivity of carbon-resin electrode in Oke [49];

g) Development and optimization of carbon-resin electrode Oke et al. [50] and

h) Thermal properties of carbon-resin electrode in Oke et al. [51]

Microstructures of the electrode were monitored to ascertain the composition of the electrode utilising a scanning electron microscope (of model Carl Zeiss Smart Evo 10 available at the Department of Materials Science and Engineering, Obafemi Awolowo University, Ile-Ife, Nigeria). Electrolysing equipment was developed from local materials. The development and performance of the device are as presented in previous publications such as Oke and Ogedengbe [52]; and Fehintola et al. [53]. The synthesised chloride wastewaters (salty water, brine) were prepared and calibrated by utilising procedures, techniques and methods stated and specified in the Standard Methods for Water and Wastewater Examination such as APHA [54], and van Loosdrecht et al. [55]. The analytical Sodium Chloride (60.0 grams) salt was dissolved in 1000 ml of distilled water as a stock solution, and secondary solutions for calibration and working salty wastewater were prepared from the stock. Selected calibration solutions of 0.0 mg/l, 250 mg/l, 500 mg/l, 1000 mg/l; 5000 mg/l; 10000 m/l and 15000 mg/l of chloride were used to calibrate the equipment used in the determination of chloride concentrations. Salty wastewaters were subjected to electrochemical treatment utilising developed carbon–resin or graphite-resin (anode) and aluminium (cathode) electrodes. Figure 1a, b present the laboratory setup of the electrochemical treatment of the simulated wastewater.

The impacts of selected operational factors (volume of the wastewater used, separation distance between the electrodes, flow and discharge rate, pH, applied current, initial concentration of the chloride, contact surface area of the electrode used and depth of the electrode) on the performance of electrochemical purification process were monitored using fractional factorial experiment and optimised using Microsoft Excel Solver. Table 1 presents the standard fractional factorial experiments and the factors. Sta-

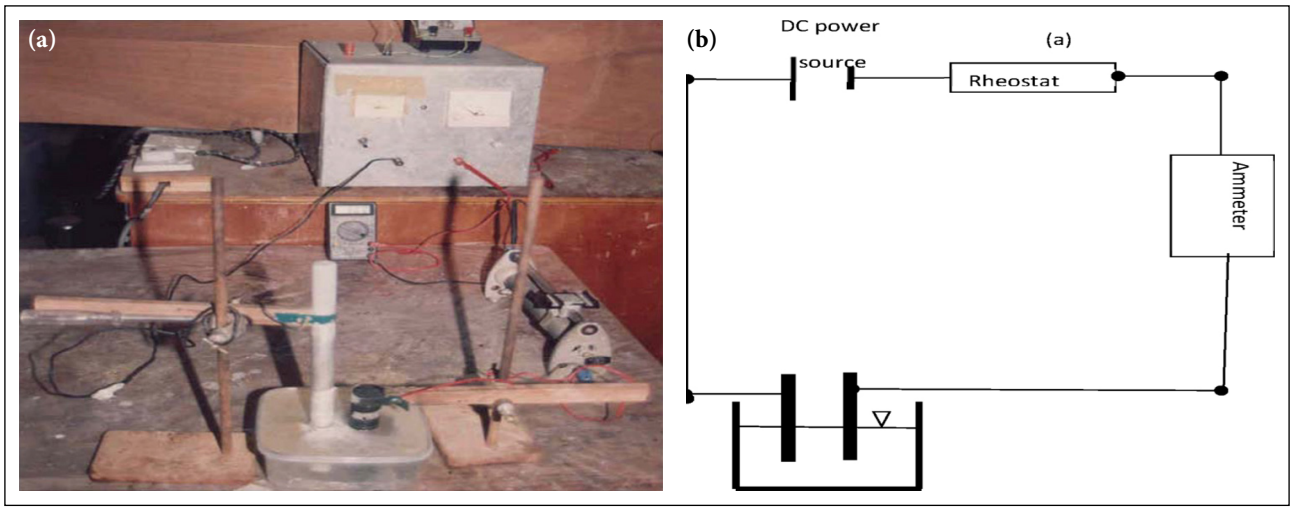

Figure 1. **(a)** Laboratory setup of the electrochemical treatment process. **(b)** Schematic diagram laboratory setup of the electrochemical treatment process.

Table 1. Standard fractional experiment (2^{k-p}) and factors used

Experiment	Initial concentration of chloride (g/l)	Current (A)	Separation distance (cm)	Time (hr)	Flow (l/hr)	Depth (cm)	Area (cm^2)	pH
1	10.0	1.0	2.0	1.0	1.0	0.4	4.91	3.0
2	40.0	1.0	2.0	1.0	2.5	0.4	19.64	10.0
3	10.0	10.0	2.0	1.0	2.5	1.0	4.91	10.0
4	40.0	10.0	2.0	1.0	1.0	1.0	19.64	3.0
5	10.0	1.0	10.0	1.0	2.5	1.0	19.64	3.0
6	40.0	1.0	10.0	1.0	1.0	1.0	4.91	10.0
7	10.0	10.0	10.0	1.0	1.0	0.4	19.64	10.0
8	40.0	10.0	10.0	1.0	2.5	0.4	4.91	3.0
9	10.0	1.0	2.0	4.0	1.0	1.0	19.64	10.0
10	40.0	1.0	2.0	4.0	2.5	1.0	4.91	3.0
11	10.0	10.0	2.0	4.0	2.5	0.4	19.64	3.0
12	40.0	10.0	2.0	4.0	1.0	0.4	4.91	10.0
13	10.0	1.0	10.0	4.0	2.5	0.4	4.91	10.0
14	40.0	1.0	10.0	4.0	1.0	0.4	19.64	3.0
15	10.0	10.0	10.0	4.0	1.0	1.0	4.91	3.0
16	40.0	10.0	10.0	4.0	2.5	1.0	19.64	10.0

tistical parameters (average, median, standard deviation, Skewness, coefficient of variation) of the performances of the treatment technique were determined using standard techniques. The choice of the Microsoft Excel Solver for the computation was grounded on the accessibility of the software at no extra cost (established in all Microsoft Excel packages). The models were modified to accommodate interactions between the selected factors (in this case the interactions were considered to be significant factors). The modified and qualified model equations were solved Microsoft Excel Solver (MiES) technique. These results from modified models were evaluated using statistical methods. The modified and qualified model equations are expressed as follows Asgari et al. [40], and Bazrafshan et al. [41]:

$$\text{Model A:} \quad Y = \beta_0 + \beta_i \sum_{i=1}^{k} X_i + \xi \tag{1}$$

$$\text{Model B:} \quad Y = \beta_0 + \beta_i \sum_{i=1}^{k} X_i + \beta_{ii} \sum_{ii=1}^{k} X_{ii}^2 + \xi \tag{2}$$

$$\text{Model C} \quad Y = \beta_0 + \beta_i \sum_{i=1}^{k} X_i + \beta_{ij} \sum_{j=1}^{k} \sum_{i=1}^{k} X_i X_j + \xi \tag{3}$$

The experimental data was fitted to polynomial models. The coefficients of polynomial models were determined using Microsoft Excel Solver. The Solver is an Add-in software for the Microsoft Excel packages which is typically or originally not enabled during the initial or primary installation of Microsoft Office (which includes Excel). The procedures

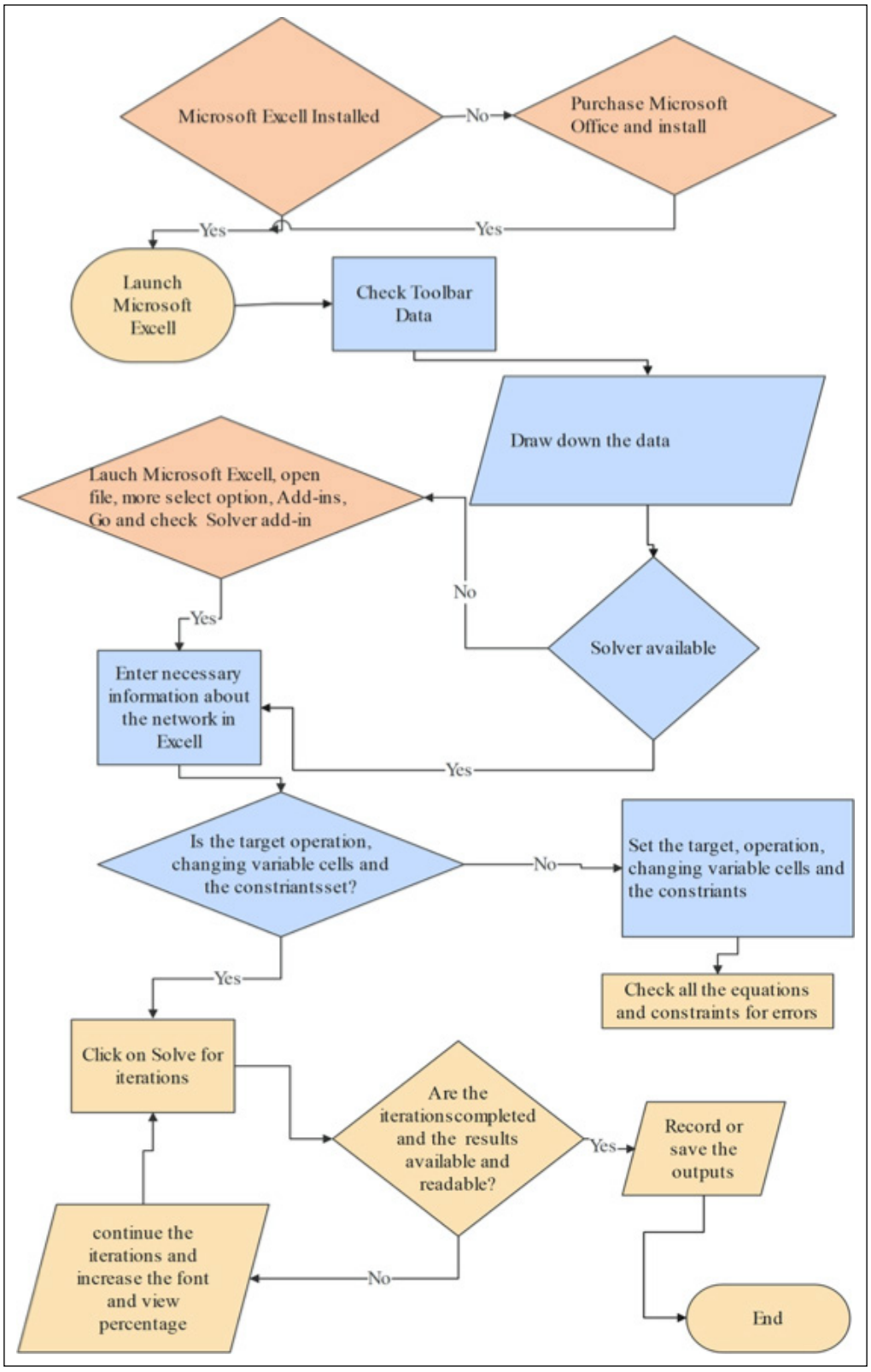

Figure 2. Procedure and flow chart for utilising Microsoft Excel Solver in the calculation of the unknown coefficients.

required in using Microsoft Excel Solver can be summarized as indicated in Figure 2. Figure 2 presents the flow chart of the procedures. The selection of these factors to be researched or studied was based on the theoretical information and data about numerous factors that establish the performance of the electrochemical treatment process and the knowledge regarding graphite-resin or carbon-resin and aluminium electrodes. Chloride determinations in both raw and treated salty wastewater were carried out using the argenotometric technique or method specified in APHA [54]. Chloride concentration was calculated using equation (5) as follows:

$$Cl^{-}(mg/l) = 35450 \times C_f \left(\frac{(A-B) \times N_0 \times P_1}{V_s} \right) \quad (4)$$

Where; N_0 is the normality of Silver Nitrate used, P_1 is the dilution factor; V_s is the volume of effluent or sample used (ml), A is the volume of the titrate used for the sample (ml), C_f is the calibration factor and B is the volume of the titrate used for the blank (ml).

Efficiencies of the process were based mainly on pollutant (chloride) removal (Y,%), which was computed using equation (5). The choices of the argenotometric and instrumentation methods [54] were based on accuracy, type of wastewater (clear aqueous solution) and availability of required reagents.

$$Y = 100 \times \left(\frac{(Z_0 - Z_t)}{Z_0} \right) \quad (5)$$

Where: Z_o is the initial concentration of the chloride in the synthetic wastewater (mg/l). Z_t is the final concentration of chloride in the synthetic wastewater (mg/l) and Y is the chloride ions removed (%).

Statistical evaluations were conducted utilising Analysis of Variance (ANOVA), Coefficient of Determination (CD), Model of Selection Criterion (MSC), average relative error (A_{re}), accuracy (E^r) and total error. Average Relative Error (A_{re}), Accuracy (E_r) and Total error (Err^2) were computed as follows:

$$A_{re} = \frac{1}{N} \sum_{i=1}^{N} \left(\frac{(X_i - X_{ci})}{X_i} \right) \quad (6)$$

Where; X_i is the observed concentration and X_{ci} is the calculated concentration.

$$E_r = 100 \times \left(1 - \frac{(X_i - X_{ci})}{X_i} \right) \quad (7)$$

$$Err^2 = \sum_{i=1}^{N} (X_i - X_{ci})^2 \quad (8)$$

The coefficient of Determination can be shown and computed as follows:

$$CD = \frac{\sum_{i=1}^{N} (X_i - \overline{X_{ci}})^2 - \sum_{i=1}^{N} (X_i - X_{ci})^2}{\sum_{i=1}^{N} (X_i - \overline{X_{ci}})^2} \quad (9)$$

Where; $\overline{X}_i$ is the average or statistical means of experimental concentration and $\overline{X}_{ci}$ is the average or statistical means of calculated concentration. The model of Selection Criterion can be calculated utilizing equation (9) indicated as:

$$MSC = \ln \left(\frac{\sum_{i=1}^{N} (X_i - \overline{X_i})^2}{\sum_{i=1}^{N} (X_i - X_{ci})^2} \right) - \frac{2p}{N} \quad (10)$$

Where; p is the number of variables or parameters and N is the number of experimental and data points

Skewness is a quantity of symmetry, proportion or exact of the data. The data set or information is symmetric when it looks the same or similar to the left and right from the centre point. Skewness was computed as follows (Equation 11):

$$\gamma = \frac{\sum_{i=1}^{N} (Y - \overline{Y})^3}{N\delta^3} \quad (11)$$

Where; γ is the skewness, δ is the standard deviation, Y is the performance of the treatment process in removing chloride; $\overline{Y}$ is the mean of of the performance of the treatment process in removing chloride and N is the number of samples.

Kurtosis is an important ingredient in statistical measurement and engineering design of treatment facility was computed as follows (equation 12):

$$\beta = \frac{\sum_{i=1}^{N} (Y - \overline{Y})^4}{N\delta^4} \quad (12)$$

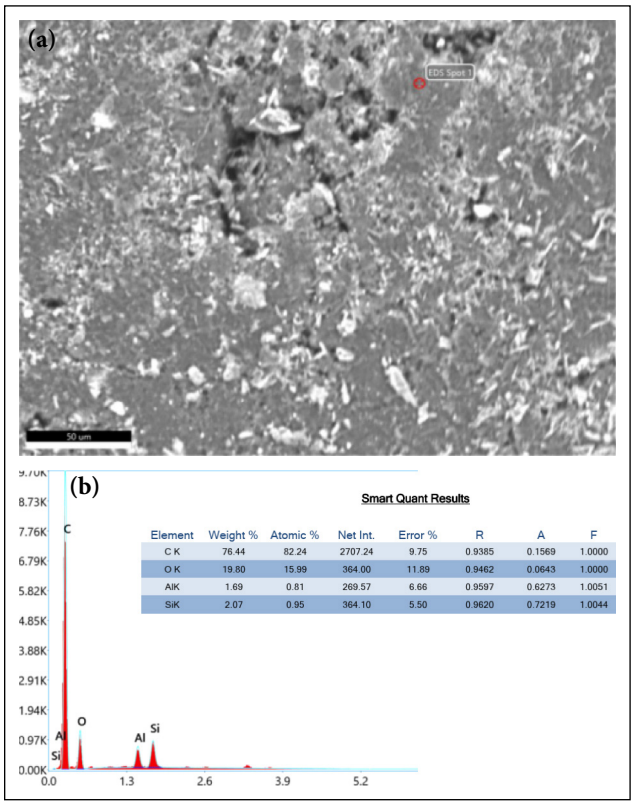

Figure 3. **(a)** The Spot for major composition of the carbon resin electrode. **(b)** The major composition of the Carbon resin electrode.

RESULTS AND DISCUSSION

Figure 3a, b shows the major compositions of the Carbon resin electrode. The figure revealed that the major components of the electrode are Carbon (76.44%) Oxygen (19.80%), Si (2.07%) and Al (1.69%). The result indicated the presence of Carbon and Oxygen at the spot, which had the highest portion of the electrode, which can be attributed to the binder used and the size of the powdered graphite [3]. This result of the composition established and discovered that the removal and reduction of chloride may

be attributed to adsorption by the pores and conversion of some of the components to silicon, aluminium, chloride and oxygen products such as $Al(OCl)_3$, $SiCl_4$ and $AlCl_3$. The $SiCl_4$ reacts with water to give silicon dioxide and acidic end products (equation 13).

$$SiCl_4 + 2H_2O \rightarrow SiO_2 + 4HCl \quad (13)$$

This reaction of $SiCl_4$ can be attributed to the bigger size of the Silicon atom, which provides more room and space around the atom to enable the water molecule to attach. In addition, the silicon atom has empty 3d orbitals available to accept a lone pair from the water molecule. This reaction indicated that the oxygen atom can bond to the silicon atom before the need to break the silicon-chlorine bond and support the whole process energetically. Aluminium chloride ($AlCl_3$) is an influential Lewis acid, industrial catalyst, non-explosive, non-flammable but corrosive solid and reacts violently with water or bases. $AlCl_3$ is believed to be a hygroscopic salt. Usually, this salt fumes in the moist - air. The reaction creates a heckling sound as it comes in connection with water. As these reactions take place or occur the Cl^- ions are displaced and replaced with water molecules and form hexahydrate $(Al(H_2O)_6)Cl_3$. At an anhydrous state, $AlCl_3$ is lost and as the heat is utilised HCl is dissipated and aluminium hydroxide is the final product that is attained.

$$Al(H_2O)_6 Cl_3 \rightarrow Al(OH)_3 + HCl + 3H_2O \quad (14)$$

As the temperature is increased further to a level of 400 °C, aluminium oxide is transformed from the hydroxide.

$$2Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O \quad (15)$$

One distinct characteristic of $AlCl_3$ in aqueous solutions is that the solutions are ionic in character. With reference to this reason, these solutions are good conductors of electricity. The solutions are also acidic, which can result in the partial hydrolysis in Al^{3+} ion. The overall reaction can be expressed as follows:

$$\left[Al(H_2O)_6\right]^{3+}_{aq} \qquad \left[Al(OH)(H_2O)_3\right]^{2+}_{aq} + H^+_{aq} \quad (16)$$

Aluminium compounds and salts that is made up of hydrated Al3+ ions are similar in reaction to the behaviour of aqueous solutions of aluminium chloride. These solutions behave in the same way or similar by giving a profuse precipitate of Al(OH)3 in reaction with a dilute basic oxide such as sodium hydroxide.

$$AlCl_3 + 3NaOH \rightarrow Al(OH)_3 + 3NaCl \quad (17)$$

Figure 4a–c shows the Scanned electromagnetic (SEM) structures of the electrode. From the figure, it is clearly revealed that the powdered particles of carbon electrode were closely parked and the porosity is very low. This lower porosity can be attributed to a lower concentration of binder, higher compressive pressure and nano-particle sizes used in the development of the electrode [53]. Table 2 presents the arrangement of the fractional factorial experiment, the performance of electrochemical treatment in removing chloride from aqueous solutions and the statistical summary of the performance of electrochemical treatment.

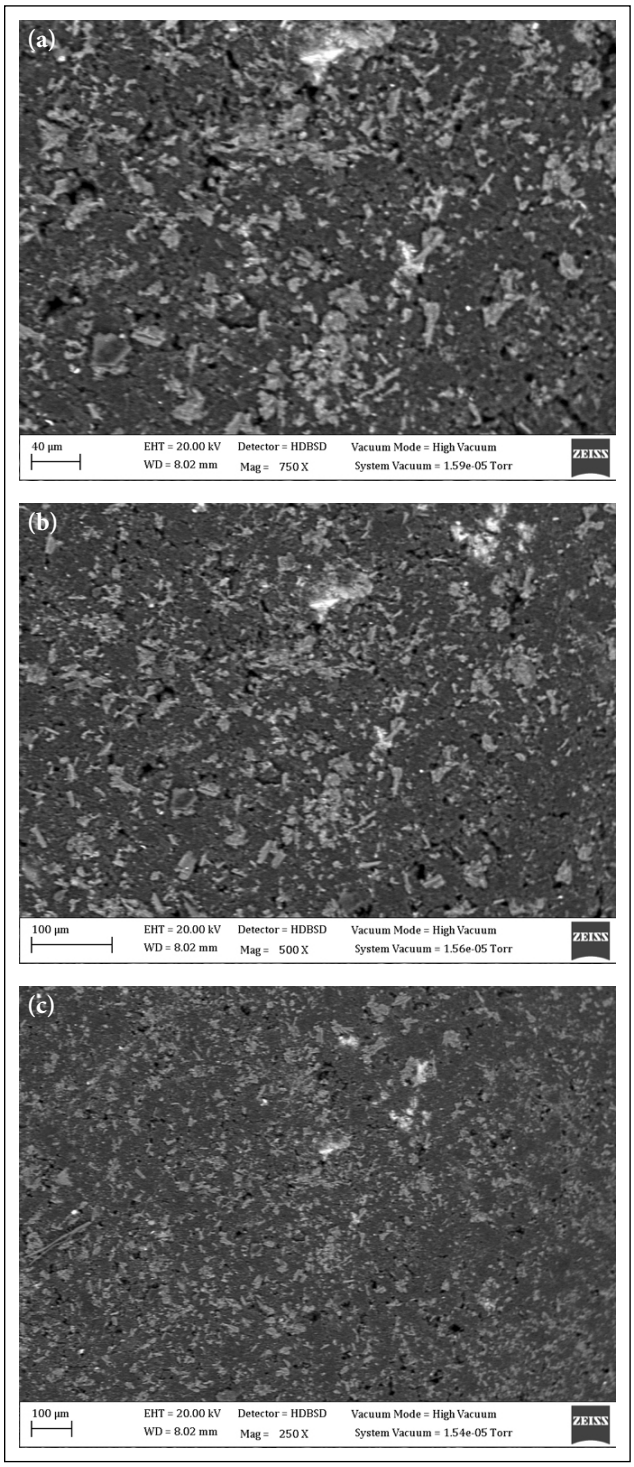

Figure 4. **(a)** Scanned Electro Magnetic (SEM) structures of the electrode at 40 µm. **(b)** Scanned Electro Magnetic (SEM) structures of the electrode at 100 µm. **(c)** Another Scanned Electro Magnetic (SEM) structures of the electrode at 100 µm.

The Table revealed that the maximum values the performance occurred at experiment number 11 with 94.82% removal of chloride concentration when the surface area (19.64 cm^2), the flow (2.0l/hr), the treatment time (4.0 hr) and the current (10.0 A) were at their higher levels, which indicated that these selected and mentioned factors had positive influences on the performance of electrochemical

Table 2. Fractional factorial experiment, and the statistical summary of the performance of electrochemical treatment in removing chloride from aqueous solution

Exp.	Response				Total	Ave.	Ske.	Max.	Min.	SD	Med.	CV	Kur.
1	89.55	89.25	87.06	83.79	349.65	87.41	-1.12	89.55	83.79	2.66	88.16	3.04	0.160
2	85.77	85.67	83.78	80.5	335.72	83.93	-1.29	85.77	80.50	2.46	84.73	2.93	0.903
3	91.94	91.74	89.85	86.17	359.7	89.93	-1.35	91.94	86.17	2.67	90.80	2.97	1.232
4	88.46	88.46	86.47	82.98	346.37	86.59	-1.32	88.46	82.98	2.58	87.47	2.98	1.039
5	71.54	71.64	69.85	67.06	280.09	70.02	-1.25	71.64	67.06	2.14	70.70	3.05	0.701
6	70.5	69.95	68.36	65.27	274.08	68.52	-1.22	70.50	65.27	2.35	69.16	3.43	0.855
7	85.5	85.37	83.48	80.2	334.55	83.64	-1.28	85.50	80.20	2.47	84.43	2.95	0.875
8	83.5	83.38	81.59	78.31	326.78	81.70	-1.32	83.50	78.31	2.42	82.49	2.96	1.080
9	96.1	96.52	94.23	90.45	377.3	94.33	-1.32	96.52	90.45	2.77	95.17	2.94	1.174
10	88.9	87.66	86.76	83.28	346.6	86.65	-1.23	88.90	83.28	2.41	87.21	2.78	1.832
11	96.9	96.81	94.62	90.94	379.27	94.82	-1.27	96.90	90.94	2.79	95.72	2.94	0.802
12	87.9	87.76	85.87	82.49	344.02	86.01	-1.30	87.90	82.49	2.52	86.82	2.93	0.978
13	83.3	83.56	81.29	78.11	326.26	81.57	-1.18	83.56	78.11	2.52	82.30	3.09	0.407
14	81.09	81.09	79.2	90.94	332.32	83.08	1.83	90.94	79.20	5.32	81.09	6.40	3.518
15	91.14	90.84	89.05	85.47	356.5	89.13	-1.34	91.14	85.47	2.61	89.95	2.92	1.226
16	87.04	86.96	85.07	81.59	340.66	85.17	-1.34	87.04	81.59	2.55	86.02	3.00	1.140

Exp: Experiment; Ave: Average; Ske: Skewness; Max: Maximum; Min: Minimum; SD: Standard deviation; Med: Median; Kur: Kurtosis.

Table 3. ANOVA of the performance

Source of variation	Sum of squares	Degree of freedom	Mean Sum of square	F-value	P-value
Within experiment	3034.023	15	202.2682	55.41323	$6.19x10^{-24}$
Between runs	209.3126	3	69.77087	19.11437	$3.87x10^{-08}$
Error	164.2581	45	3.650179		
Total	3407.594	63			

during the treatment process. The lowest value of the performance of the process occurred in experiment numbered 6 with 68. 52% removal of the chloride by the treatment process [27, 28, 32, 45]. This level of performance occurred when the initial concentrations of chloride (40 x 10^3 mg /l), the separation distances between electrodes (10.0 cm), the depth of the electrode (1.0 cm) and pH (10.0) were at higher factorial factor levels, which meant that these latter mentioned selected factors contributed negatively to the performance of the treatment process. From Table 2, the Skewness of the performance of electrochemical treatment in removing chloride from an aqueous solution was between -1.12 and 1.83. With the exception of experiment number 14, which has positive Skewness, all the other experiments were of negative Skewness, which indicated that most values are concentrated on the right of the mean, with extreme values to the left. Kurtosis (β) is a degree of data peakedness or horizontalness relative to a normal distribution pattern. The data sets or information with higher values of kurtosis tend to have a distinct peak near the mean, either decline and reduce rapidly or have heavier tails than necessary. From Table 2, the Kurtosis of the performance of electrochemical treatment in removing chloride from aqueous solution was between 0.160 and 3.518. The Kurtosis values of the performance of electrochemical treatment in removing chloride from an aqueous solution were positive, which indicated the data sets have a distinct peak near the mean, decline rather rapidly, and have heavy tails. With the exception of experiment number 14, which has Kurtosis greater than 3, all the other experiments had Kurtosis less

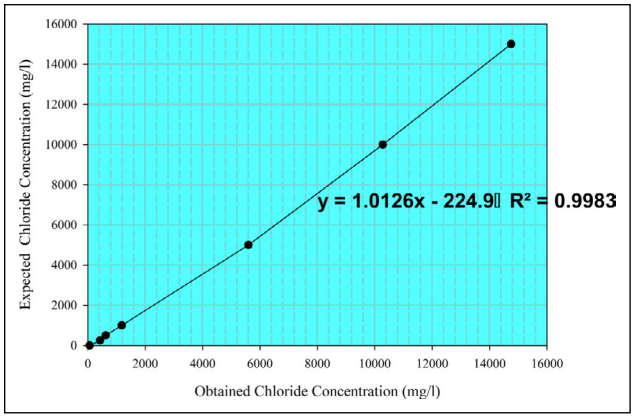

Figure 5. Calibration curve (relationship between obtained and expected chlorideconcentrations).

Table 4. Result of ANOVA for the calibration

Source of variation	Sum of squares	Degree of freedom	Mean Sum of square	F-value	P-value
Between groups	96114.29	1	96114.28571	0.002819033	0.95853
Within groups	4.09E+08	12	34094771.43		
Total	4.09E+08	13			

Table 5. The coefficient for the mathematical polynomial regression models

Experiment	Average	Model A's coefficients		Model B's coefficients		Model C's coefficients			
1	87.41	Constant	87.629	Constant	86.671	Constant	86.930	Cubic Initial Con	-6.5E-07
2	83.93	Initial Con	-0.122	Initial Con	-0.110	Initial Con	-0.108	Cubic Current	0.002882
3	89.93	Current	0.576	Current	0.675	Current	0.362	Cubic Sep D	-0.00377
4	86.59	Sep D	-1.044	Sep D	-0.980	Sep D	-0.515	Cubic Time	0.051667
5	70.02	Time	2.042	Time	2.193	Time	1.113	Cubic Flow (l/hr)	-0.01613
6	68.52	Flow (l/hr)	-0.411	Flow (l/hr)	-0.292	Flow (l/hr)	-0.157	Cubic Depth(cm)	-0.00316
7	83.64	Depth(cm)	-2.462	Depth(cm)	-1.440	Depth(cm)	-1.432	Cubic Area	-7E-06
8	81.70	Area	0.091	Area	0.113	Area	0.116	Cubic pH	-6.2E-05
9	94.33	pH	-0.113	pH	-0.078	pH	-0.070	Cubic Initial Con	-6.5E-07
10	86.65			Sq. Initial Con	-0.00024	Sq. Initial Con	-0.00024		
11	94.82			Sq. Current	-0.00902	Sq. Current	-0.00964		
12	86.01			Sq. Sep D	-0.0054	Sq. Sep D	-0.00523		
13	81.57			Sq. Time	-0.03026	Sq. Time	-0.03129		
14	83.08			Sq. Flow (l/hr)	-0.03373	Sq. Flow (l/hr)	-0.02743		
15	89.13			Sq. Depth(cm)	-0.72951	Sq. Depth(cm)	-0.72654		
16	85.17			Sq. Area	-0.00089	Sq. Area	-0.00089		
				Sq. pH	-0.00272	Sq. pH	-0.0026		

Table 6. The statistical evaluation of the mathematical model equation

	Average	Accuracy	MSC	CD	R	AIC	SC
Model A	2.99	97.01	0.630	0.804	0.897	96.023	96.658
Model B	2.97	97.03	0.690	0.874	0.935	95.945	96.602
Model C	2.94	97.06	0.705	0.893	0.945	95.945	96.602

than 3, which indicated that experiment 14 is a leptokurtic distribution, sharper than a normal distribution, with values concentrated around the mean and thicker tails.

Table 3 presents the ANOVA of the performance of the treatment process. The table revealed that there were significant differences between the performances and the runs (experiments) at a 95% confidence level. Table 4 presents the results of ANOVA for the calibration conducted on the equipment used in the determination of the chloride concentrations. The table revealed that there was no significant difference between obtained and expected chloride concentrations at a 95% confidence level ($F_{1,12}$=0.0028; p=0.95853). Figure 5 presents the relationship between obtained and expected chloride concentrations. The Figure established that there is a good relationship between the obtained and expected chloride concentrations (R^2=0.9983). Table 5 shows the analysis of the fractional factorial experiment, the effects and the coefficient of each of the selected factors for model A. The table revealed that these selected factors can be classified into two groups as positive and negative factors. The current through the wastewater, the treatment time and the surface area of the electrodes were the positive factors. The above-mentioned factors are the factors with increment in magnitude that improved the performance of the treatment process. The initial concentration of the salt, the distance between the electrodes, the flow rate of the wastewater, the depth of the electrodes and the pH value of the wastewater were the negative factors, that reduced the performance of the treatment process. These negative factors were the factors with a decrement in the magnitude of the performance of the treatment process. From the table, the significant factors (at a 90% confidence level) on

Table 7. Effects of selected factors on performance of electrochemical treatment in removing chloride from aqueous solution, optimum and statistical evaluation (Model A)

	Initial concentration of chloride	Current	Separation distance between electrodes	Treatment time	Flow rate	Depth of electrode	Surface area of electrodes	pH	Error	Total
Sum of squares	0.06	1.33	4.36	16.67	0.67	24.24	0.03	0.05	3.15	758.51
Degree of freedom	1	1	1	1	1	1	1	1	7.00	15.00
Means Sum of squares	0.06	1.33	4.36	16.67	0.67	24.24	0.03	0.05	0.45	50.57
F-value	0.13	2.95	9.70	37.05	1.50	53.86	0.07	0.11		

F-critical values at 90 %, 95 %, 97.5 %, 99 % and 99.5 % are 3.59, 5.59, 8.07, 12.25 and 16.24, respectively.

Table 8. Effects of selected factors on the performance of electrochemical treatment in removing chloride from aqueous solution, optimum and statistical evaluation (Model B)

	Initial concentration of chloride	Current	Separation distance between electrodes	Treatment time	Flow rate	Depth of electrode	Surface area of electrodes	pH	Error	Total
Sum of Squares	0.05	1.82	3.84	19.24	0.34	8.30	0.05	0.02	16.91	758.51
Degree of Freedom	1	1	1	1	1	1	1	1	7.00	15.00
Means Sum of Squares	0.05	1.82	3.84	19.24	0.34	8.30	0.05	0.02	2.42	50.57
F-value	0.11	4.05	8.53	42.74	0.76	18.44	0.11	0.05		

Table 9. Effects of Selected Factors on the performance of electrochemical treatment in removing chloride from an aqueous solution, optimum and statistical evaluation (Model C)

	Initial concentration of chloride	Current	Separation distance between electrodes	Treatment time	Flow rate	Depth of electrode	Surface area of electrodes	pH	Error	Total
Sum of Squares	0.05	0.52	1.06	4.96	0.10	8.20	0.05	0.02	35.61	758.51
Degree of Freedom	1	1	1	1	1	1	1	1	7.00	15.00
Means Sum of Squares	0.05	0.52	1.06	4.96	0.10	8.20	0.05	0.02	5.09	50.57
F-value	0.10	1.16	2.35	11.01	0.22	18.21	0.12	0.04		

electrochemical performance toward chloride removal are current, separation distance between electrodes and time. Table 5 revealed the coefficient for the mathematical polynomial regression models as follows (equations [18–20]):

a) For Model A.

$$Y(\%) = 87.629 - 0.122I_c + 0.576C_u - 1.0424D_s + 2.042T_t - 0.411Q_f - 0.2.462D_p + 0.091A_s - 0.113pH \quad (18)$$

b) For Model B.

$$Y(\%) = 87.629 - 0.122I_c + 0.576C_u - 1.0424D_s + 2.042T_t - 0.411Q_f - 0.2.462D_p + 0.091A_s - 0.113pH \quad (19)$$

For Model C.

$$Y(\%) = 87.629 - 0.122I_c + 0.576C_u - 1.0424D_s + 2.042T_t - 0.411Q_f - 0.2.462D_p + 0.091A_s - 0.113pH \quad (20)$$

Table 6 shows the statistical evaluation of the mathematical model equations. The table revealed that average error, accuracy, MSC, CD, and R of these model equations (Models A, B and C) were 2.99%, 97.01 0.41183, 0.804, and 0.897; 2.97%, 97.03%, 0.690, 0.874, 0.935; 2.94%, 97.06%, 0.705, 0.893 and 0.945, respectively. This result indicated that these mathematical models are reliable with 97.01%, 97.03% and 97.06% accurate, which indicates that Model A can predict 97.01% of the experimental data, Model B predicts 97.03% and Model C provide information on 97.06% of the experimental data. Tables 7 to 9 present the results of ANOVA on the effects of the selected factors in each of the model equations. Table 7 presents the effects of selected factors on the performance of electrochemical treatment in removing chloride from aqueous solution, optimum and statistical evaluation (Model A). Table 8 shows the effects of Selected Factors on the performance of electrochemical treatment in removing chloride from aqueous solution, optimum and statistical evaluation (Model B) and Table 9 is for the effects of selected factors on the performance of electrochemical treatment in removing chloride from an aqueous solution, optimum and

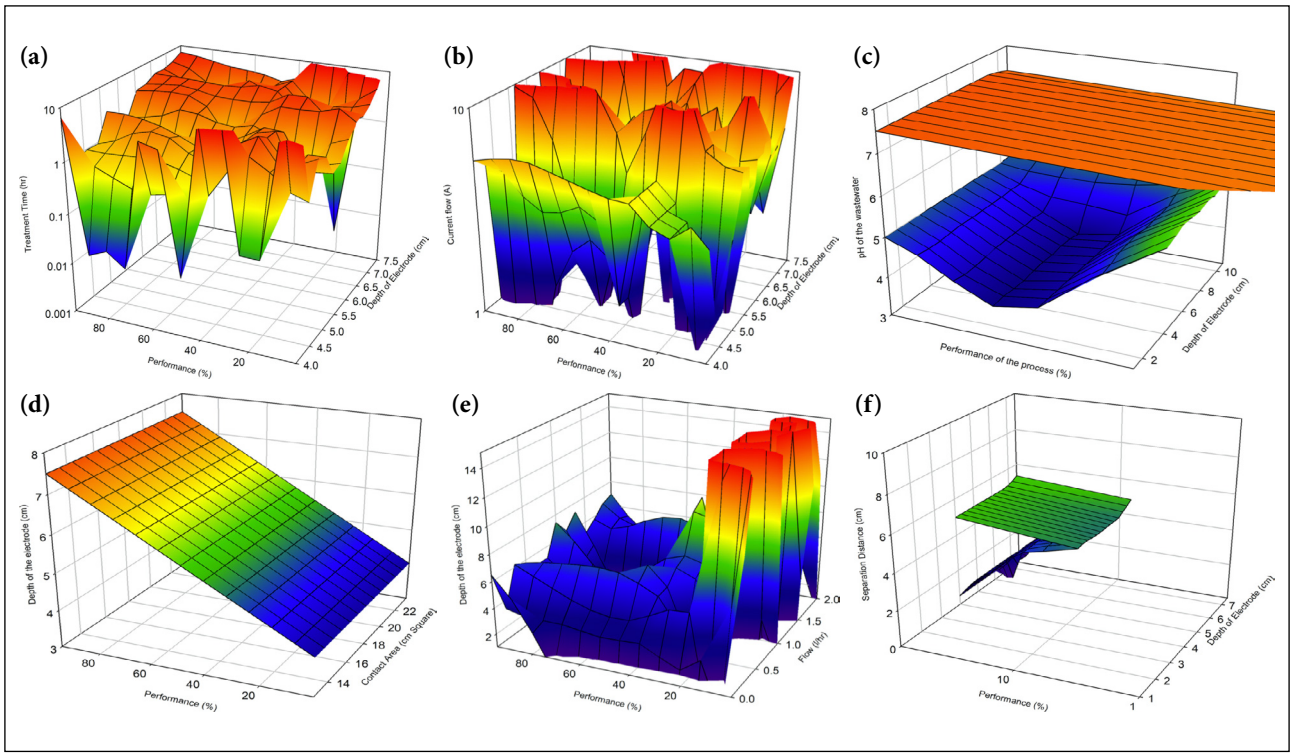

Figure 6. **(a)** Relationship between treatment time, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. **(b)** Relationship between the current, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. **(c)** Relationship between the pH, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. **(d)** Relationship between Contact Area, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. **(e)** Relationship between the flow rate, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. **(f)** Relationship between the separation distance between the electrodes, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution.

statistical evaluation (Model C). From these Tables (Table 7–9) the factors can be grouped into two factors with effects but not significant and factors with effects and factors are significant. The factors with effects and significance are separation distance between electrodes, treatment time, current and depth of electrodes. Figure 6 presents the relationship between the performance of the treatment process and selected operational factors. Figure 6a shows the relationship between treatment time, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution.

Figure 6b presents the relationship between the current, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. Figure 6c shows the relationship between the pH, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. Figure 6d is for the relationship between Contact Area, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. Figure 6e is for the relationship between the flow rate, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. Finally, Figure 6f presents the relationship between the flow rate, depth of electrodes and performance of electrochemical treatment in removing chloride from an aqueous solution. All these figures revealed that there were polynomial relationships between the selected operational factors and the performance of the treatment process. These revelations indicated that optimizations of these factors are necessary and will be helpful in operational techniques.

It was observed that these relationships were similar to the surface response of the influence of some the operational variables and parameters on the arsenic reduction and removal by electrocoagulation utilising iron electrodes and other techniques in Oke et al. [56], Vijaya Bhaskar et al [57], Can et al. [58], Darvishi et al. [59], and Majumder and Gupta [60], the response surface technique and methodological examination or evaluation of the adsorption of textile dye onto biosilica or alginate nano-biocomposite: kinetic, and isotherm studies and thermodynamic behaviour in Darvishi et al. [59], the removal and elimination of methylene blue dye from aqueous solutions by zeolite composite from shrimp waste and new chitosan in Gilhotra et al. [61], Yao et al. [62], Gadkari et al [63], and Elimelech and Phillip [64], and electrocoagulation technology for high strength arsenic wastewater: process optimization and mechanistic study in Can et al. [58].

CONCLUSIONS

It can be concluded based on the study that:

a. Electrochemical treatment with carbon electrodes is efficient in removing chloride from salty wastewater. The system was able to reduce chloride concentration from 15000 mg/l to 5% of the initial concentration at a current flow of 9.68 A and retention time of 5 hours.

b. The operational factors with negative effects (increasing these operational factors decreases the removal performance) on the performance of the treatment process were separation distance between the electrodes, pH, depth of the electrode, initial concentration of the chloride and flow rate.

c. These operational factors applied current, treatment time and contact surface area of the electrode used are positive factors (increasing these operational factors increases the removal performance) that influence the electrochemical treatment of salty wastewaters.

d. The operational factors that had significant effects on the performance of the treatment process are current, time and separation distance between the electrodes.

e. In the case of models and their evaluations Model C performed better than Model B and Model A based on average errors of 2.99%, 2.97% and 2.94%, respectively and R values of 0.945, 0.935 and 0.897 for Models C, B and A, respectively, which supported conclusions on nonlinear models in Mahmoud [65] and Amin et al. [66].

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

USE OF AI FOR WRITING ASSISTANCE

The authors confirmed that AI was not use in the writing or as a writing assistant in the manuscript.

ETHICS

There are no ethical issues with the publication of this manuscript.

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