A COMPARATIVE STUDY OF IRON PLATE ELECTRODES AND ALUMINIUM PLATE ELECTRODES FOR PHOSPHATES REMOVAL BY ELECTROCOAGULATION PROCESS AND IT'S OPTIMIZATION USING BOX–BEHNKEN DESIGN.

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Abstract

The present study describes an electrocoagulation procedure that uses iron and aluminium plates both as cathode and anodes to treat phosphate-contaminated water. Three isotherm models were used to assess the different parameters, including the effects of NaCl concentration, pH, initial concentration of phosphate, voltage, temperature, and the adsorption capacity. The monolayer coverage of adsorbed molecules is implied by the phosphate adsorption, which ideally fits the Langmuir adsorption isotherm. The findings indicated that iron plate electrode provided the highest removal effectiveness of 93.94%. Adsorption is governed by second-order kinetics. Optimization of phosphates by electrocoagulation process is performed with above parameters and observed for iron plate electrode and aluminium plate electrode to be contact time (25 &35min) , Voltage (10 & 20V), pH (7 & 6), NaCl Concentration (2.0 & 2.5 g/L) and initial concentration (20 mg/L). From these optimum conditions the highest removal of phosphate obtained is 93.94% for iron plate electrode which is higher than removal obtained by aluminium plate electrode (91.9%). Kinetic, isothermal and thermodynamic studies has been studied and obtained best results. SEM and FTIR analysis has been done for the samples obtained. Using the experimental data, the predicted data is been analyzed by Box-Behnken design, where, It showed good fit with experimental data.

Keywords:- Electrocoagulation; Kinetics; Phosphates; FTIR; SEM; Box–Behnken design.

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1. Introduction

The importance of protecting water bodies has increased recently as a result of increased industrialization and urbanization, but the expenses of aerobic wastewater treatment have remained expensive.[1]. Eutrophication is one of the major issues currently faced in the monitoring of environmental water sources in industrialized countries, as is well known. The excessive phosphate concentration in the effluents from municipal or industrial plants that are released into the ecosystem is what causes this phenomenon, which is to blame for the dramatic development of algae in drinking water.[2].

Numerous algae blooms, excessive development of invasive aquatic plants, unfavorable visual effects, deoxygenation, and issues with water purification for drinkable use are all issues related to eutrophication.[3]. Due to their adaptability, which enables the handling of liquids, gases, and solids, and environmental compliance, electrochemical technologies have garnered a lot of attention. These procedures include electrodialysis, electrooxidation, electro flocculation, and electrocoagulation. This study focuses on the electrocoagulation remediation of synthetic effluent that contains phosphates. There are many research on the elimination of phosphorus from effluent.[4]. These methods have some benefits over chemical treatment methods, such as the need for fewer coagulant ions, the lack of chemical addition, the relative lack of area demand, the cheap cost of investment, and the small amount of sludge generated when compared to those in a traditional chemical process.[5].

The electrocoagulation technique is receiving a lot of focus these days for treating wastewater. This technique has been used to effectively eliminate various types of pollutants, including phosphorus. [6].

Electro coagulation, which has been used for wastewater treatment, is the electrochemical generation of destabilization agents that results in charge neutralization for pollutant elimination. These electrodes can hydrolyze electrochemically produced metallic ions to create a succession of activated intermediates that can destabilize the finely distributed particles in the water or refuse to be treated. Following destabilization, the particles gather to create groups. High efficiency, ambient working conditions, compact apparatus, little sludge production, quick start-up, and the potential for total automation are all benefits of electrochemical treatment. Furthermore, the Electro coagulation method can quickly and efficiently remove phosphorus under high pressure and temperature conditions[7].

This article examined the effectiveness of phosphate elimination and energy usage of aluminum and iron electrodes. According to the findings, Electro coagulation procedures using iron electrodes removed phosphate at a faster rate and used less energy than those using aluminum electrodes.

2. Materials and Methods

2.1. Materials Required:

Sodium diphosphate (AR) is used to prepare stock solution and Ammonium molybate, Potassium antimony tartrate, ascorbic acid, NaCl, NaOH, HCl chemicals are used as reagents for determination of phosphate are purchased from Lotus enterprises private ltd., Visakhapatnam, India.. Iron plate electrodes and aluminium plate electrodes are purchased from local iron mart, Visakhapatnam, India.

2.2. Preparation of Aqueous Solution:

A Standard solution of 1.49 g of sodium diphosphate in 1000ml of distilled water is prepared for varies concentration (mg/L) as stock solution for phosphates. pH is maintained by adding 0.1 N HCL/NaOH solution. NaCl is added as a support for electrocoagulation.

2.3 Experimental setup:

An aqueous solution of one-liter volume was utilised in the experimental apparatus for the electrocoagulation research. A magnetic stirrer was utilised to accomplish effective mixing. Consistent cell currents were provided using a DC power source with a range of 0–35V and 0–3A. The four iron electrode plates, which serve as two anodes and two cathodes, are arranged horizontally in the tank and have the following dimensions: 10 cm 6 cm 0.05 cm, spacing of 2.5 cm between electrodes.

2.4 Experimental procedure :

One litre of phosphate solution was combined with a known quantity of sodium chloride, which served as a conductor, for each run. The electrolytic cell was filled with the solutions. By adding HCl/ NaOH solutions, the pH can be changed. During the electrolysis run period, direct current from the D.C power source was transferred through the solution via the four electrodes.

2.4 Experimental Parameters:

In the current investigation, iron plate electrodes and aluminium plate electrodes used an electrocoagulation technique to remove phosphate. At regular intervals, samples were taken. A UV-Spectrophotometer was used to measure the amount of phosphates in the supernatant. It investigated how to optimize process variables such as electrolysis time, NaCl concentration, pH, voltage, and initial concentration.

3.Results and Discussions:

3.1. Effect of Contact time:

Electrolysis time in the range of 0 to 35 minutes, initial concentration (20 mg/L), pH (5), (10) volts and NaCl concentration (1 g/L) for iron plate and (1.5g/L) for aluminium plate electrode. The concentration was estimated for every 5 min. There was no change in % removal of phosphates with increase in time after 25 min and 35 min. As shown in Figure-1(a) most of phosphate removal was reached under some optimum values of electrolysis time (25min) for iron and (35 min) for aluminium plate electrode. The percentage removal of phosphate obtained was 79.25% for iron plate electrode and 75.8% for aluminium plate electrode.

3.2 Effect of NaCl concentration:

The concentration of phosphates was estimated for every 0.5g/L increase in NaCl concentration. There was no change in % removal of phosphates with increase in NaCl concentration after 2.0 g/L for iron plate electrode and 2.5 g/L for aluminium plate electrode. As shown in the Figure-1(b), most of phosphate removal was reached under some optimum values of NaCl concentration (2.0 g/L) and (2.5 g/L). The percentage removal of phosphate is increased from 76.85 % to 84.67 % as NaCl concentration is increased for iron plate electrode and for aluminium plate electrode,the percentage removal is increased from 73.54 % to 78.62 % as the NaCl concentration is increased from 0.5 g/L to 3.0 g/L.

3.4 Effect of pH:

The concentration of phosphates was estimated for every pH number increase. The percentage removal is increased from 82.42 % to 92.64 % as pH is increased from 4 to 7. The percentage removal is decreased from 92.64 % to 84.54 % as pH increases from 7 to 11 for iron plate electrode. Similarly, the percentage removal is increased from 75.8 % to 80.38 % as pH is increased from 4 to 6. The percentage removal is decreased from 80.38 % to 76.38 % as pH increases from 6 to 9 for iron plate electrode.is shown in Figure-1(c).

3.3 Effect of Voltage:

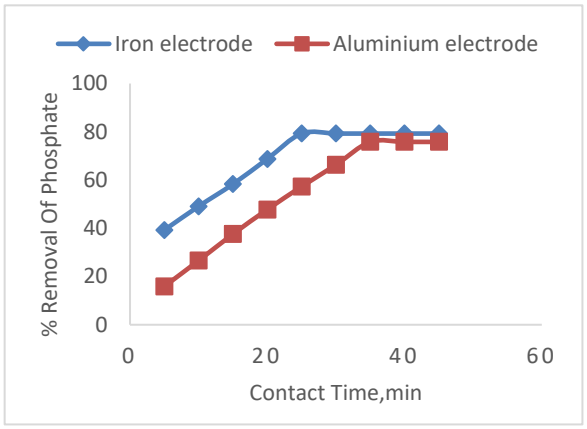
Voltage is in the range of 5 to 30 V, the concentration of phosphates was estimated for every 5V increase in Voltage. There was no change in % removal of phosphates with increase in voltage after 10V for iron plate and 20 V for aluminium plate electrode. As shown in Figure-1(d),the % removal is increased from 89.62 % to 90.38 % as voltage is increased from 5 to 30 volts for iron plate electrode. Similarly, the % removal is increased from 78.8 % to 82.36 % as voltage is increased from 5 to 30 volts for aluminium plate electrode..

3.5 Effect of Initial concentration of Phosphates:

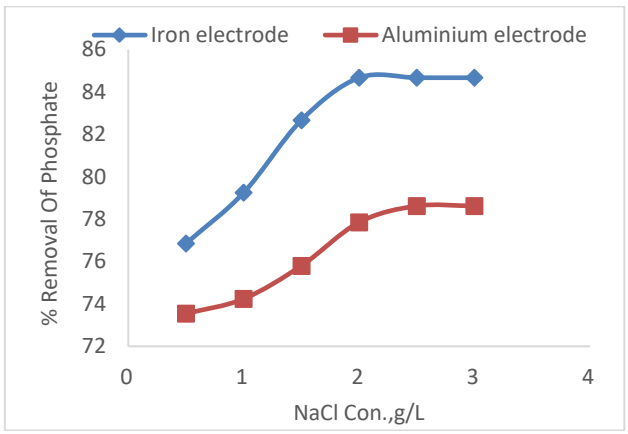
Initial Concentration of phosphates is in the range of 10 to 100 mg/L, NaCl concentration 2.0g/L & 2.5 g/L, pH 7 &6, 10 volts & 20 Volts and time 25 mins & 35 min. The concentration of phosphates was estimated for all Concentration of phosphates respectively. There % removal of phosphates decreased with increase in Initial Concentration of phosphates. As shown in the Figure-1(e), The percentage removal of phosphate obtained was 93.9% and 91.9% for iron and aluminium plate electrode.

3.6 Effect of Temperature:

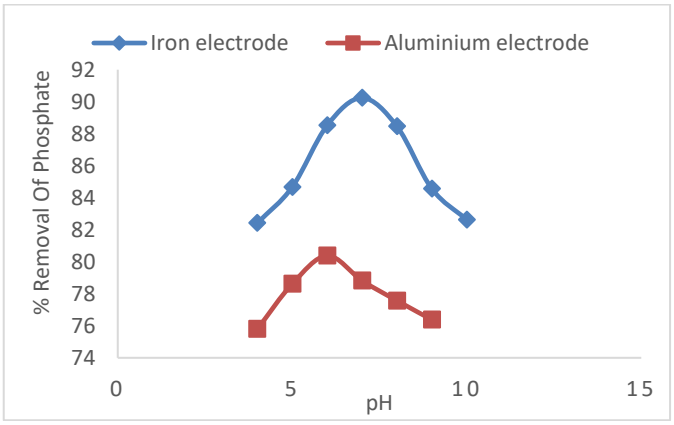
The effect of temperature was investigated from batch experiments carried out at four constant temperatures: 303, 308, 313 and 318 K shown in Figure-6. With an increase in temperature, the % removal was increased from 93.94 % to 96.27 % for iron plate electrode and 91.9 % to94.85 % for aluminium plate electrode respectively, for the initial concentration of 10 mg/L and other parameters are kept at optimum values.


Iron electrode
Aluminium electrode
% Removal Of Phosphate
100
80
60
40
20
0
0
20
40
60
Contact Time,min

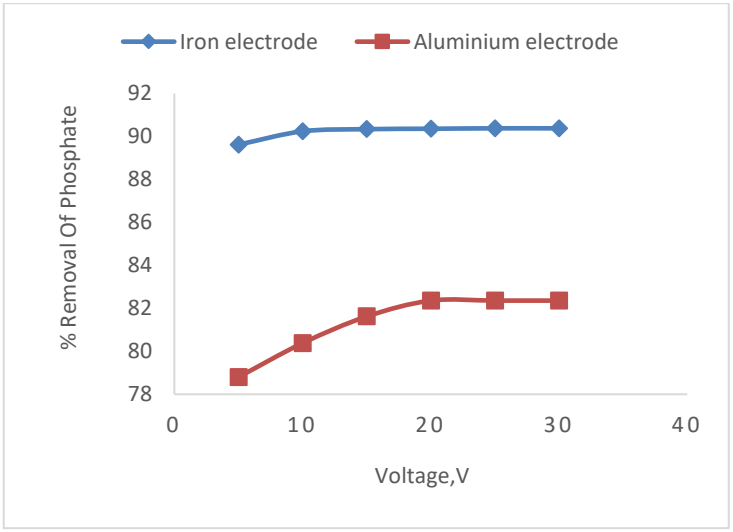
(a)


Iron electrode
Aluminium electrode
% Removal Of Phosphate
86
84
82
80
78
76
74
72
0
1
2
3
4
NaCl Con.,g/L

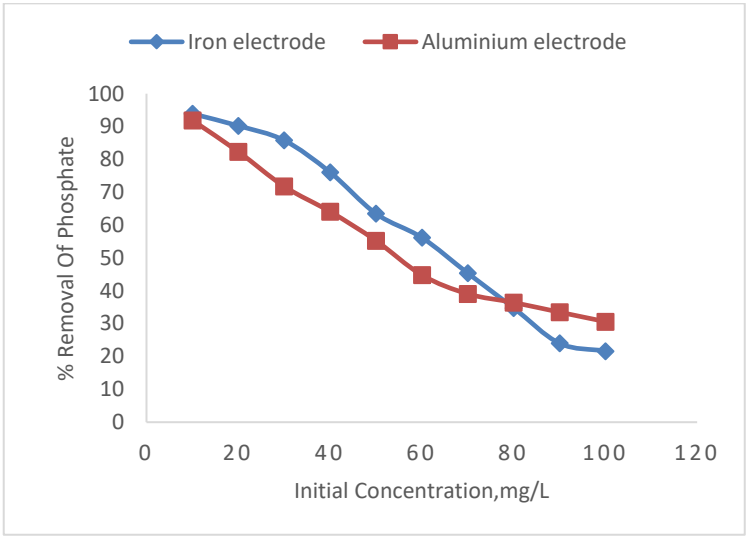
(b)


Iron electrode
Aluminium electrode
% Removal Of Phosphate
92
90
88
86
84
82
80
78
76
74
0
5
10
15
pH

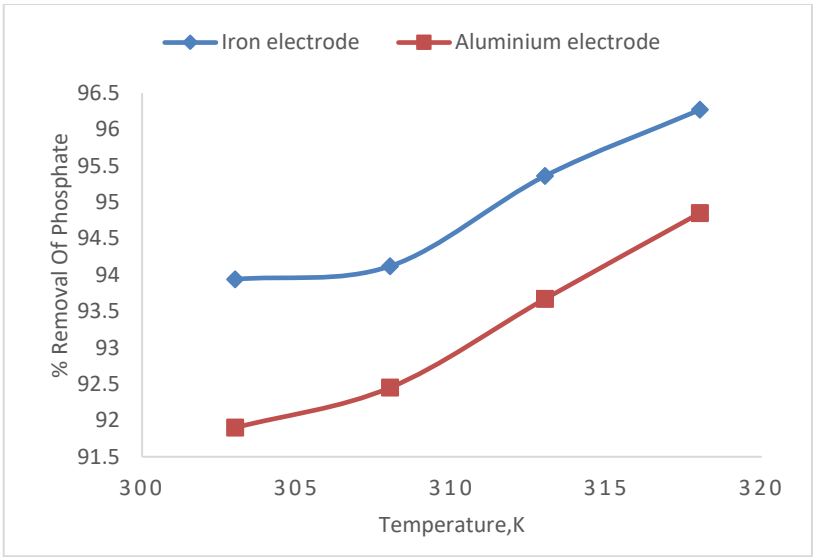
(C)


Iron electrode
Aluminium electrode
% Removal Of Phosphate
92
90
88
86
84
82
80
78
0
10
20
30
40
Voltage,V

(d)



(e)

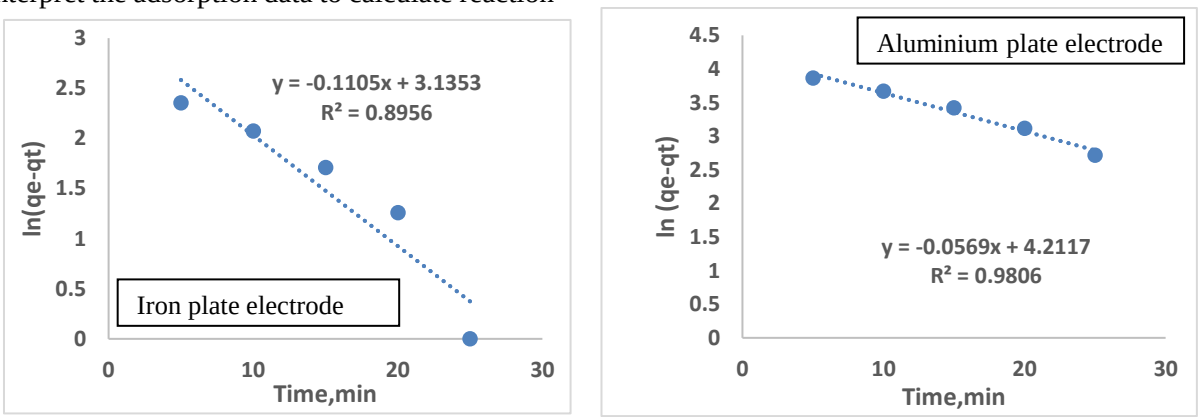


(f)

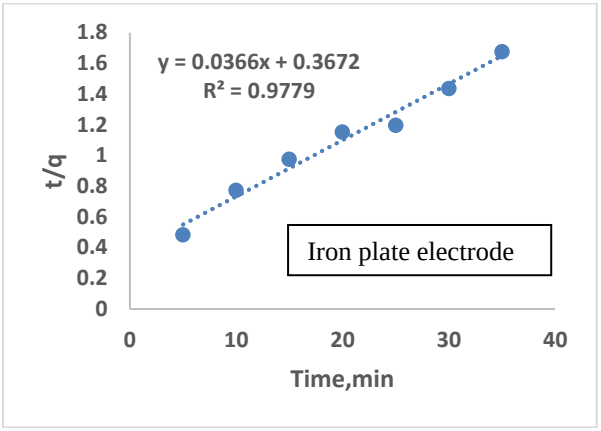
Figure – 1 Effect of (a) Contact time ,(b)NaCl Conc.,(c) pH,(d)Voltage,(e) Initial concentration and (f) temperature on % removal of phosphate using iron plate and aluminium plate electrode.

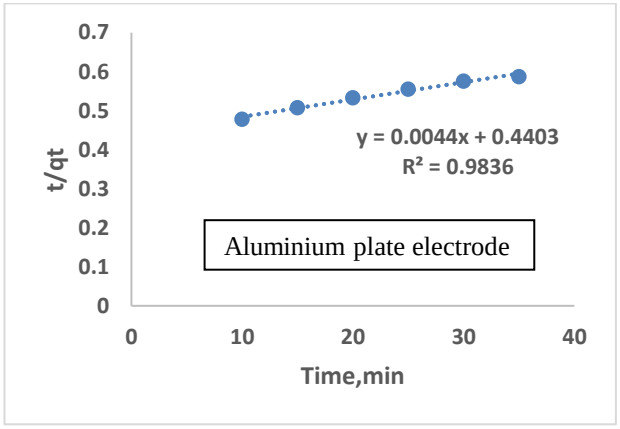
4. Kinetics, Adsorption Isotherms and Thermodynamic studies:

Figures. 2 (a) , (b) and (c) show the plots of the three different kinetic models that were employed to interpret the adsorption data to calculate reaction rate shown in Table-1. Pseudo-second order models (R^2 = 0.9779) and (R^2 = 0.9836) fit the experimental data better than pseudo-first order models and elovich model.[8]

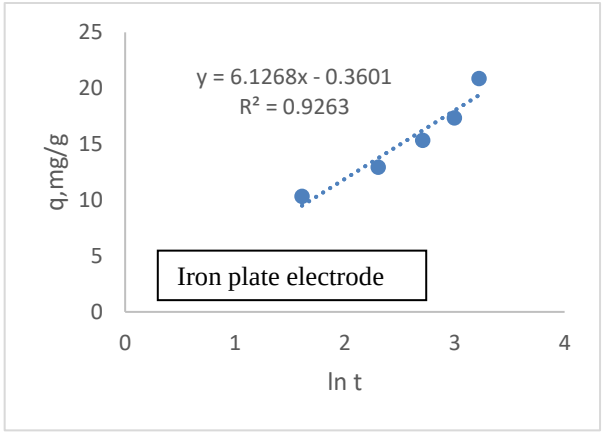


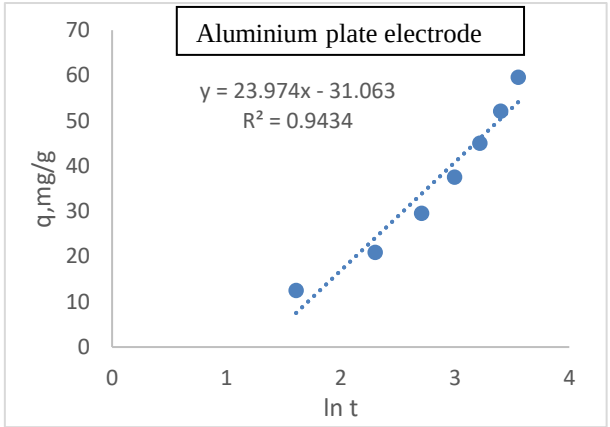
(a)





(b)





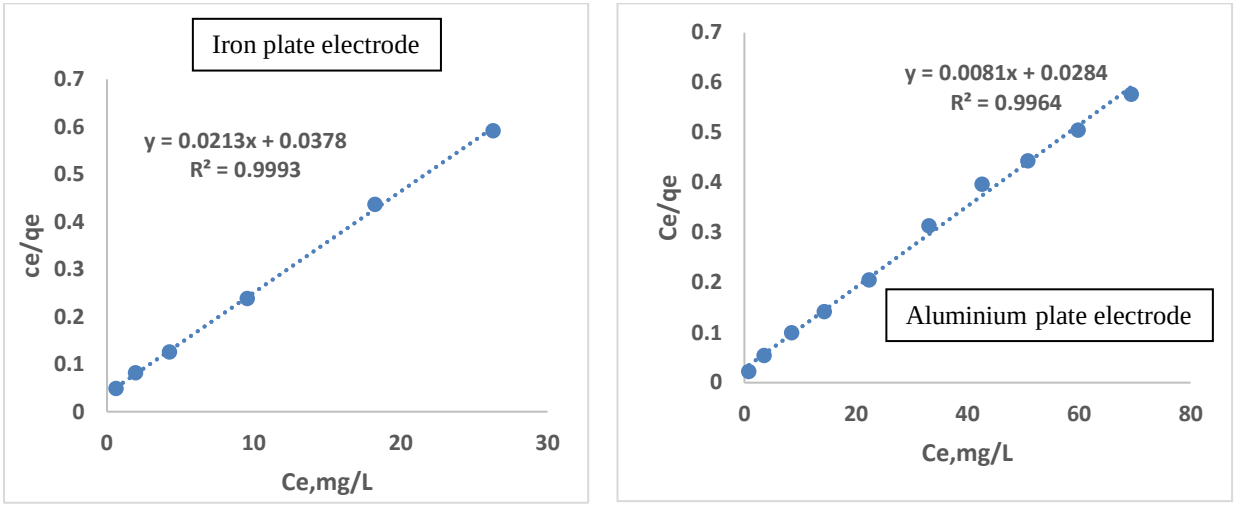
(C)

Figure-2 Shows the kinetic studies (a) Pseudo-first order for iron and aluminium plate electrode (b) Pseudo-second order for iron and aluminium plate electrode and (c) Elovich model for iron and aluminium plate electrode.

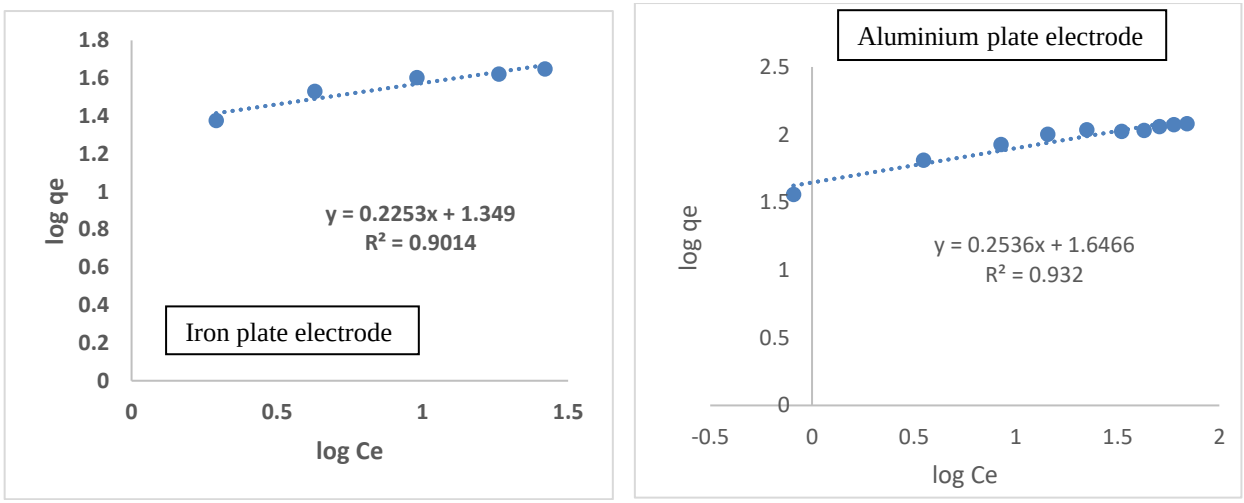
Table – 1:- Equations and rate constants

Order	constants	Iron plate electrode	Aluminium plate electrode
Pseudo-first order	K_1(1/min)	0.1105	0.0569
	q_e (mg/g)	22.99	67.47
	R^2	0.8956	0.9806
Pseudo-second Order	K_2 (g/mg.min)	3.6x10-3	4.39x10-5
	$q_{e,cal}$(mg/g)	27.32	227.27
	R^2	0.9779	0.9836
Elovich	γ (g/mg)	0.1632	0.0417
	α (mg/g.min)	6.498	87.614
	R^2	0.9263	0.9434

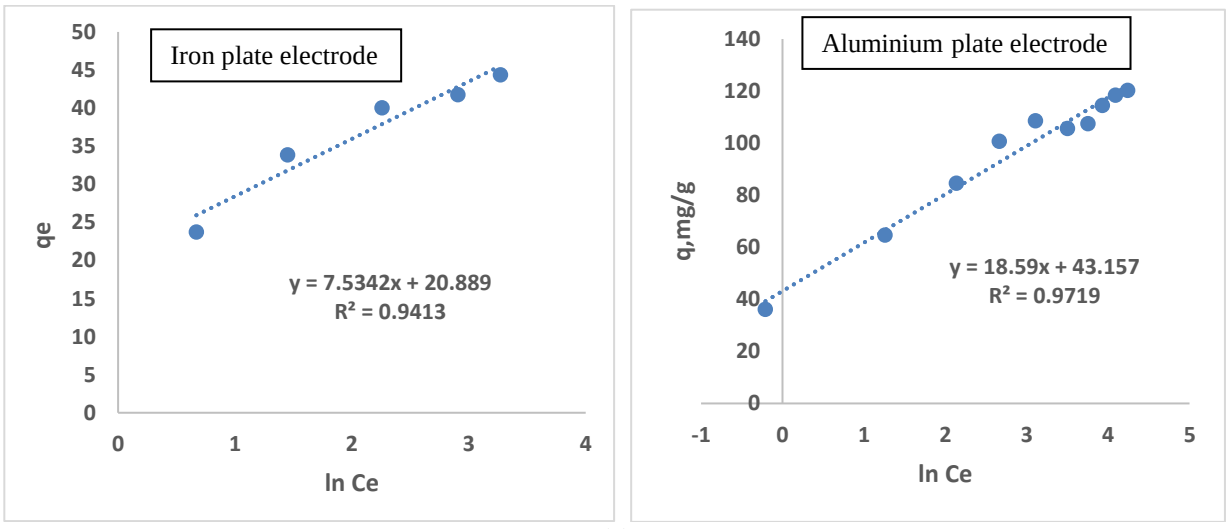
Three isotherm models, the Langmuir , Freundlich and Temkin equations, describe the adsorption equilibrium. The adsorption data are better explained by the Langmuir model, which has a higher correlation coefficient (R^2 = 0.9993) & (R^2 = 0.9964) than the Freundlich model (R^2 = 0.9014) & (R^2 = 0.932) and Temkin model (R^2 = 0.9413) & (R^2 = 0.9719) shown in figure- 3 (a),(b) & (c) and Table -2.[16]



(a)



(b)



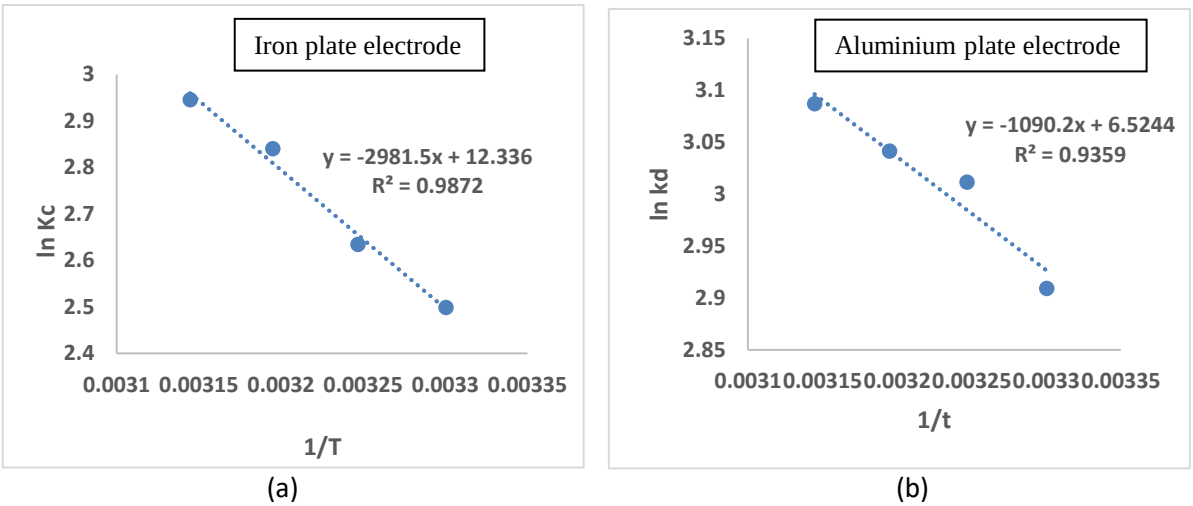
(c)

Figure-3: Shows Isotherm studies (a)Langumir isotherm for iron plate and aluminium plate electrode,(b) Frendulich isotherm for iron plate and aluminium plate electrode and (C) Temkin studies for iron plate and aluminium plate electrode.

Table – 2:- Isotherm constants

Isotherm Model	constants	Iron plate electrode	Aluminium plate electrode
Langmuir	K_L(L/mg)	0.5634	0.2852
	q_m (mg/g)	46.94	123.45
	R^2	0.9993	0.9964
	R_L	0.7442	0.812
Freundlich	K_f (mg/g)	22.33	44.32
	n	0.2253	0.2536
	R^2	0.9014	0.932
Temkin	A_T	15.99	10.19
	B	7.5342	18.59
	R^2	0.9413	0.9719

Capacity and final concentration can be used to determine the Gibbs free energy (ΔG) as shown in Figure-4 (a) & (b) and Table-3.[17].It is found to be enothermic in nature.



(a) (b)

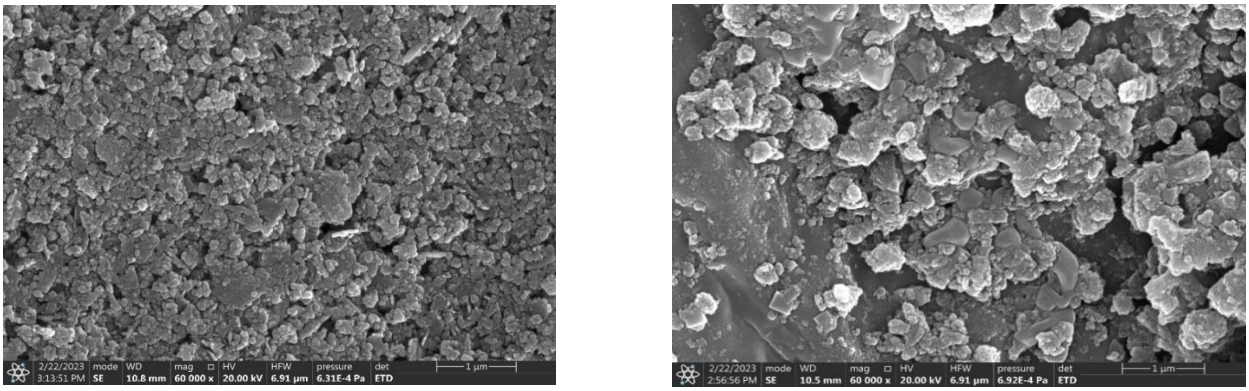
Figure -4 Thermodynamic plot for (a) Iron plate electrode and (b) Aluminium plate electrode

Table – 3:- Thermodynamic Parameters

Temperature, K	Iron plate electrode			Aluminium plate electrode		
	ΔG^0 (kJ/mol)	ΔH^0 (kJ/mol)	ΔS^0 (J/mol.K)	ΔG^0 (kJ/mol)	ΔH^0 (kJ/mol)	ΔS^0 (J/mol.K)
303	-6295.69	24.788	102.56	-7328.16	9.06	54.243
308	-6746.54			-7711.54		
313	-7391.37			-7914.18		
318	-7788.97			-8161.73		

5.SEM for Iron electrode sludge and Aluminium electrode sludge:

The impact of the electrochemical phosphate removal procedure on the morphology of the sludge was investigated using SEM technology. Figure-5(a) & (b) images clearly show that after multiple electrolysing runs, the iron plate electrode sludge gives globular and irregular shapes.Similarly for aluminium plate electrode sludge shows the phosphates are entrapped with the aluminium ions.They are irregular in shape and nonuniform in size.[9,13]



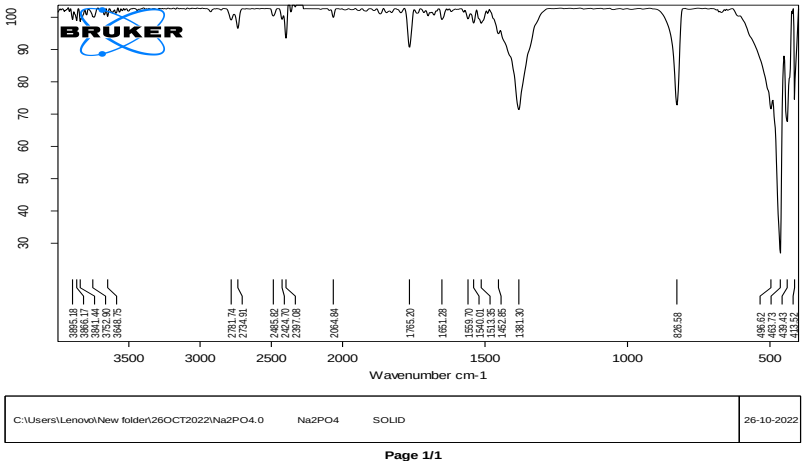
(a) **(b)**

Figure 5- (a) SEM images of the Iron plate electrode and (b) Aluminium plate electrode .

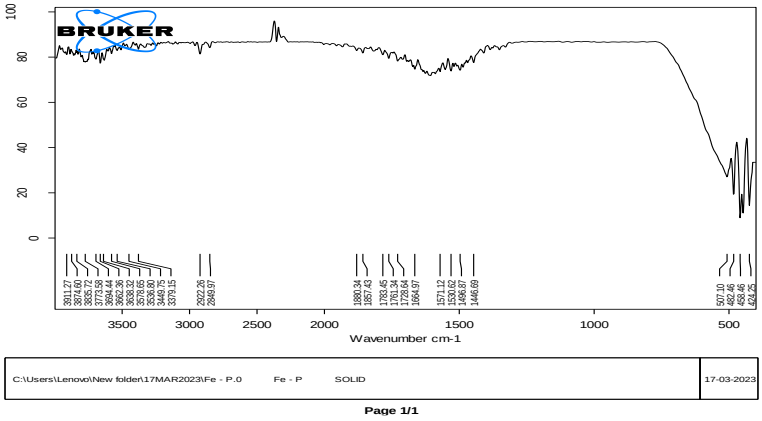
6. FTIR spectrum:

The result showed that numbers of peaks were detected, informing the complex structure material. The FTIR

spectrum of before and after analysis (sludge) is presented in Figure.11 (a),(b) & (c).The peaks obtained for Sodium diphosphate in Figure.6 (a) consist of Phosphine group, carboxyl group, Aldehydes and amine groups.The peaks obtained for Iron electrode sludge Figure.6 (b) shows that the groups of Aromatic compounds and P-OR groups are not found and aluminium electrode sludge shown in Figure.6(c) shows the same but they are high peaks obtained for other compounds.[11,12,13]



(a)



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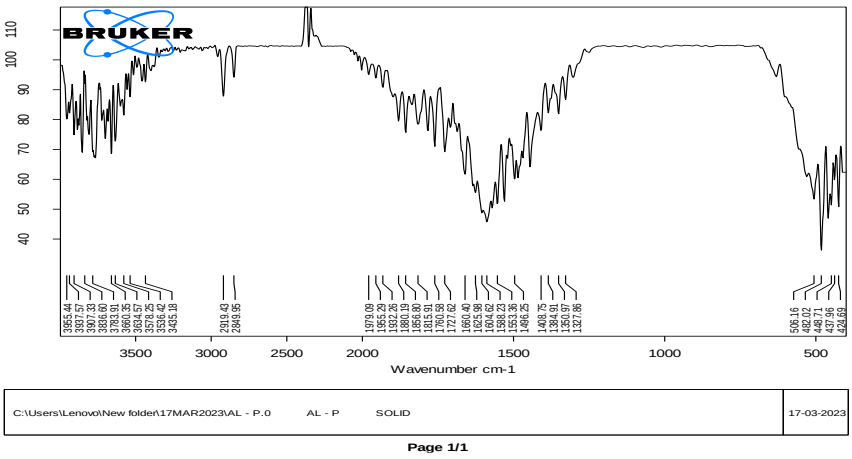


(c)

Figure-6. FTIR spectrum peaks for (a) Sodium diphosphate ,(b) Iron plate electrode and (c) Aluminium plate electrode.

7.Response Surface Methodology:

The investigation of statistically developed combinations meant for maximising the desired response, the estimation of coefficients by fitting experimental data to response functions, the prediction of response using the created regression model, and the evaluation of the model's suitability are all included in the optimization of process parameters using BBD. To increase the effectiveness of phosphate removal, three independent variables solution pH, voltage, and electrolysis time, were chosen for the current investigation as shown in Table-4. On the basis of the BBD matrix, 17 experimental runs total of 4 axial, 8 factorial, and 5 times repetition of the centre points were created at three distinct levels (-1, 0, 1) of independent parameters. The phosphate removal efficiency (% Y) is the desired outcome (dependent variable).The software produce the regression equations, coefficients, and expected responses.[14,15]

Table–4:- Levels of different process variables in coded and un-coded form for % removal of phosphate using iron plate electrode and Aluminium plate electrode

Variable	Name	Range and Levels Iron plate electrode			Range and Levels Aluminium plate electrode		
		-1	0	1	-1	0	1
X_1	pH of aqueous solution	6	7	8	5	6	7
X_2	Voltage, V	5	10	15	15	20	25
X_3	Electrolysis Time, T ,Min	20	25	30	30	35	40

F-value and p-value are significant parameters that indicate the models' sufficiency and relevance, as shown in Table 5.

Regression equation for the optimization of adsorption is:

% removal of phosphate (Y) is function of pH of aqueous solution (X_1), Voltage (X_2), and Contact time (X_3).

The multiple regression analysis of the experimental data has yield the following equation for iron plate electrode:

$$\mathbf{Y = 502.03 + 0.3003\ X_1 + 0.1624\ X_2 + 3.39\ X_3 + 0.3721 X_1X_2 + 1.58 X_1X_3 + 0.2025 X_2X_3 + 230.24\ X_1^2 + 122.43\ X_2^2 + 93.60\ X_3^2.}$$

Table-5:- Estimated regression coefficients for removal of phosphate using iron plate electrode

Source	Regression coefficient	Mean Square	F-value	p-value	
Model	502.03	55.78	62.55	< 0.0001	Significant
A-pH	0.3003	0.3003	0.3368	0.5799	
B-voltage	0.1624	0.1624	0.1822	0.6823	
C-Time	3.39	3.39	3.80	0.0921	
AB	0.3721	0.3721	0.4173	0.5389	
AC	1.58	1.58	1.77	0.2255	

BC	0.2025	0.2025	0.2271	0.6482	
A^2	230.24	230.24	258.19	< 0.0001	
B^2	122.43	122.43	137.29	< 0.0001	
C^2	93.60	93.60	104.96	< 0.0001	
Residual	6.24	0.8918			
Lack of Fit	6.11	2.04	63.05	0.0008	Significant
Pure Error	0.1293	0.0323			
Cor Total	508.27				

F-value and p-value are significant parameters that indicate the models' sufficiency and relevance, as shown in Table 6.

Regression equation for the optimization of adsorption is:

% removal of phosphate (Y) is function of pH of aqueous solution (X_1), Voltage (X_2), and Contact time (X_3).

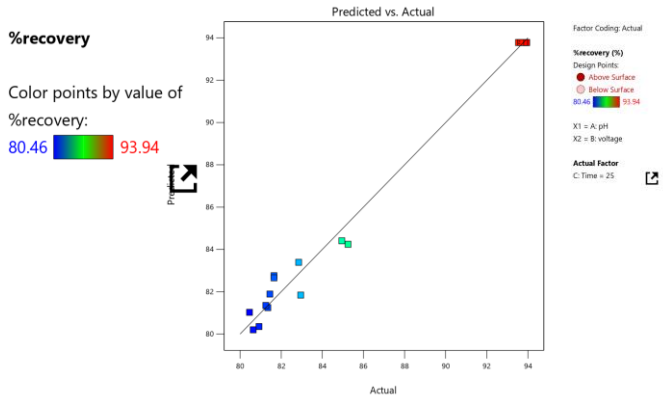
The multiple regression analysis of the experimental data has yield the following equation for aluminium plate electrode:

$$\mathbf{Y = 181.04 + 0.7503\ X_1 + 3.60\ X_2 + 0.0098\ X_3 + 0.0552\ X_1X_2 + 0.1521\ X_1X_3 + 2.37\ X_2X_3 + 141.88\ X_1^2 + 48.39\ X_2^2 + 39.23\ X_3^2.}$$

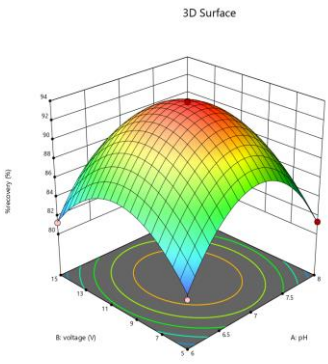
Table-6:- Estimated regression coefficients for removal of phosphate using Aluminium plate electrode

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	181.04	9	20.12	101.82	0.0002	Significant
A-pH	0.7503	1	0.7503	3.80	0.1231	
B-Voltage	3.60	1	3.60	18.25	0.0129	
C-Time	0.0098	1	0.0098	0.0496	0.8347	
AB	0.0552	1	0.0552	0.2795	0.6250	
AC	0.1521	1	0.1521	0.7699	0.4298	
BC	2.37	1	2.37	12.00	0.0257	
A^2	141.88	1	141.88	718.20	< 0.0001	
B^2	48.39	1	48.39	244.95	< 0.0001	
C^2	39.23	1	39.23	198.57	0.0001	
Residual	0.7902	4	0.1976			
Lack of Fit	0.7900	3	0.2633	1316.71	0.0203	Significant
Pure Error	0.0002	1	0.0002			
Cor Total	181.83	13				

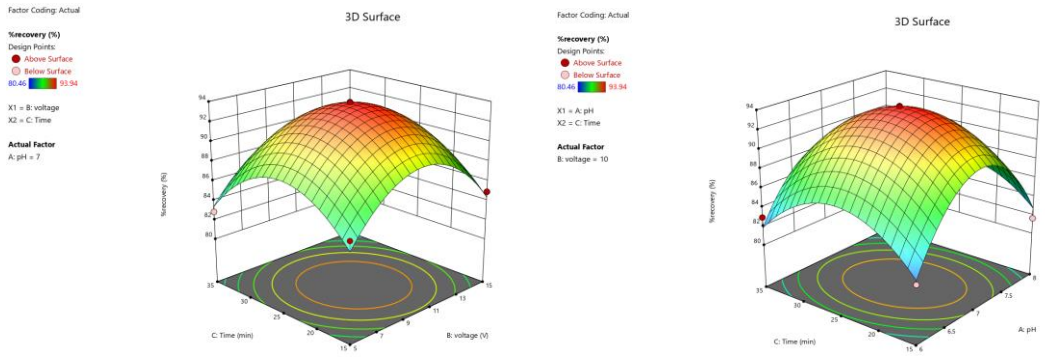
The results for iron plate electrode are supported by the fact that the values predicted by the model agree with the experimental values, as shown in Figure.7(a) & Figure.7(b,c,d) displays the 3D-graphs for three different variable combinations.



(a)



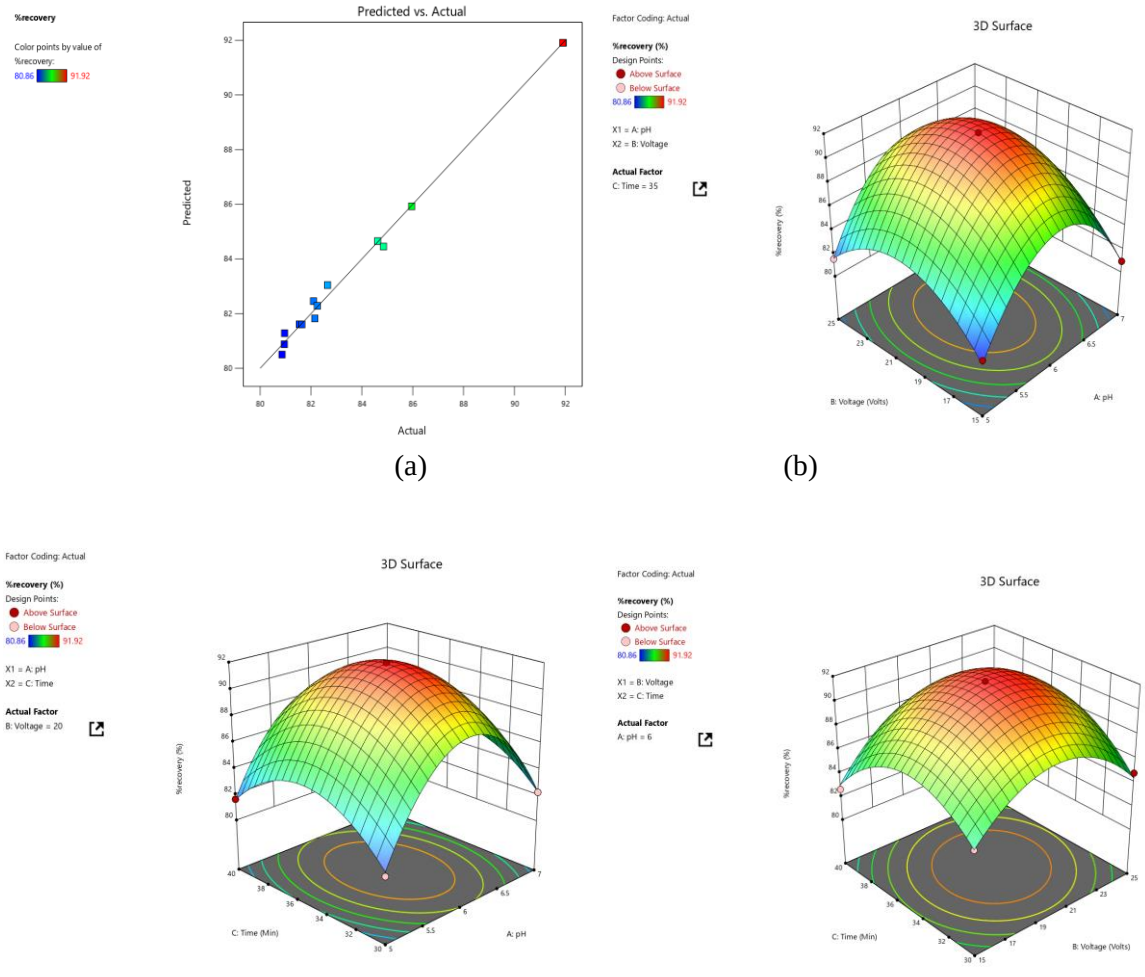
(b)



(c) **(d)**

Figure.7. Shows (a) predicted vs actual graph;3D response surface graphs for the interactive effects of (b) Voltage and pH ,(c) contact time and voltage, (d) pH and time.

The results for iron plate electrode are supported by the fact that the values predicted by the model agree with the experimental values, as shown in Figure.8(a) & Figure.8(b,c,d) displays the 3D-graphs for three different variable combinations.



(a) (b)

(c) (d)

Figure.8. Shows (a) predicted vs actual graph;3D response surface graphs for the interactive effects of (b) Voltage and pH ,(c) contact time and voltage, (d) pH and time.

8.CONCLUSION:

The present study investigated the removal of phosphates by Electrocoagulation process using iron and aluminium plate electrodes. It was found that high percentage of phosphate is been removed by iron plate electrode (93.94%) when compared with aluminium plate electrode (91.9%).SEM and FTIR is used to characterise Iron plate electrodes and aluminium plate electrode sludge obtained by electrocoagulation process.From experimental data, it is observed iron plate electrode is effective for the removal of phosphates. We can conclude that BBD is an effective statistical method for forecasting and maximising phosphate removal by EC and these findings are consistent with expectations. We may conclude that modelling can assist us in achieving such good outcomes. This study aims to assess the predictive value of quadratic models for real effluents.

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