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UTILIZATION OF 2^K FACTORIAL EXPERIMENTS FOR THE DETERMINATION OF FACTORS THAT INFLUENCE ELECTROCHEMICAL PROCESS

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ABSTRACT

As a follow up of previous studies, in this paper a report on a two-level factorial experiment that provides information on the impact of selected operational variables on efficiency of electrochemical method using aluminium/carbon-resin electrodes in a synthetic wastewater was presented. Carbon –resin electrodes were developed from used dry cells using a non-heat treatment process. 2^4 factorial experiments were used to determine the influence of selected factors (volume of the wastewater, distance between the electrodes, contact surface area and current through the electrodes) on efficiency of an electrochemical treatment process. Factors with significant effects were optimized using a steepest ascent method. The results of the optimization were analyzed using least squares method and confirmed with further studies.

The study revealed that volume of the wastewater and clear distance between the electrodes had negative effects on efficiency of an electrochemical treatment process, while contact surface area and current through the electrodes had positive effects. The significant factors at 90.0 % significant level ($p<0.0005$) were distance between the electrodes ($F = 6.91; 12.28$), contact surface area ($F = 16.83; 30.08$) and current through the electrodes ($F = 13.00; 23.10$), while volume of the wastewater used was not a significant factor at the same level ($F = 1.62; 2.91$). Optimization values for current, separation distance, volume and contact surface area were 1.85 A, 0.50 cm, 0.60 L and 0.240 cm^2 respectively. Results of the verification showed that the mathematical model agreed with experimental responses and an optimum model had correlation coefficients (R^2) of 0.984 for first order model.

Keywords: *factorial experiments, electrochemical treatment process, efficiency, wastewater, factors, interactions, mathematical model*

1. INTRODUCTION

The stringent potable water standard and effluents quality limits by Environmental Regulatory Authorities have brought a greater challenge to Environmental and Water Engineers. Increasing energy cost and reports of outbreak of water-borne diseases such as schistosomiasis in many parts of Africa have caused Environmental and Water Engineers to look for effective means or methods of treating water and wastewaters rather than relying on conventional methods (Smith *et al.*, 2002; Zhou and Smith, 2002; Diaz *et al.*, 2008; Oturan and Pinson, 1995; Oturan *et al.*, 2000 and 2001; Pouet and Grasmick, 1995; Rajkumar *et al.*, 2006; Roa *et al.*, 2005; Sanroman *et al.*, 2005; Vlyssides *et al.*, 2004; Mondal , 2008). This indicates that there is a need to control water pollution effectively. Water pollution control can be done easily at the source using any of the wastewater treatment methods, but action is now on methods that can reduce harmful pollutants at the lowest cost such as the electrochemical method.

In the last two decades several studies have been conducted on the performance of electrochemical treatment process as a wastewater treatment method. The process has been successfully utilized for the treatment of various wastewaters generated by industries such as distillery (Manisankar *et al.*, 2004), textile (Naumczyk *et al.*, 1996, Oke, 2006), dye processing industry (Shen *et al.*, 2006) just to mention a few. Chen (2004) reviewed the development, design and applications of electrochemical technologies in water and wastewater treatment. A particular focus was given to electrodeposition, electrocoagulation (EC), electroflotation (EF) and electrooxidation. Over 300 related publications were reviewed with 221 cited or analyzed. It was reported that electrodeposition is effective in recovering heavy metals from wastewater streams (Chen, 2004). It is considered as an established technology with possible further development in the improvement of space-time yield. EC has been in use for water production or wastewater treatment. It is finding more applications using aluminium, iron or the hybrid Al/Fe electrodes. The separation of the flocculated sludge from the treated water can be accomplished by using EF. The EF technology is effective in removing colloidal particles, oil and grease, as well as organic pollutants. It is proven to perform better than dissolved air flotation, sedimentation and impeller flotation (IF). The newly developed stable and active electrodes for oxygen evolution would definitely boost the adoption of this technology. Electrooxidation is finding its application in wastewater treatment in combination with other technologies. It is effective in degrading the refractory pollutants on the surface of a few electrodes. Titanium-based boron-doped diamond film electrodes (Ti/BDD) show high activity and give reasonable stability. Its industrial application calls for the production of Ti/BDD anode in large size at reasonable cost and high durability (Oke, 2006; Chen, 2004). More data on electrochemical treatment processes can be obtained from literature (Chen *et al.*, 2000; Golder *et al.*, 2006; Holt *et al.*, 2002; Khemis *et al.*, 2006; Kobya *et al.*, 2003; Koparal and Ogutveren, 2002; Kobya *et al.*, 2006; Kumar *et al.*, 2004; Mollah *et al.*, 2001).

However, these previous studies describe the roles of many factors such as nature of electrodes (anode and cathode), voltage, current, pH and many others during the treatment process, but little has been said about the significance of each of these factors. The use of a statistical method to establish the significance of these factors and their interactions has not been well documented. Considering the importance of electrochemical treatment method in removing pollutants such as chloride, oil and grease, sulphate and heavy metal at an economical rate (Chen, 2004) there is a need to identify factors and interactions that have significant influence on the efficiency of electrochemical treatment process. The main purpose of this work was to utilize factorial experiments (2^4) to establish factors that influence the performance of electrochemical treatment process using simulated (synthetic) wastewater and to conduct further studies to verify results of the factorial experiments.

2. Materials And Method

Carbon resin electrodes were developed from used dry cells. Discarded dry cells (size D R20 UM-1) were collected from different locations in Nigeria. The cells were sectioned; graphite (carbon) were removed from them and pulverised. Powdered graphite was sieved into different particle sizes. A known mass of the powdered graphite was mixed with an organic binder, moulded into 2.5 cm diameter, 10-cm long electrode using locally-fabricated extruder and plunger and a compaction machine. Details and properties of the electrodes were presented in another paper (Oke *et al.*, 2007). An electrolysing equipment was developed from local materials (Oke and Ogedengbe, 2007). Synthetic (simulated) wastewaters were prepared using procedures and methods specified in Standard Methods for Water and Wastewater Examination (APHA, 1998). Analytical Sodium Chloride (24.6 grams) was dissolved in 1000ml of distilled water as a stock solution and working synthetic wastewaters were prepared from the stock. A standard 2^4 factorial matrix was developed (Tables 1 and 2) and 2^4 factorial experiments were utilized at random to determine influence of selected factors (separation distance between the electrodes, volume of the wastewater used, applied current and contact surface area of the electrode used) and interactions on efficiency of electrochemical process. The choice of the parameters to be studied was done on the basis of the theoretical data about several factors that determine the efficiency of an electrochemical method and the scarce knowledge concerning carbon-resin / aluminium electrodes. An electrochemical treatment plant on a laboratory scale was setup (Figures 1a and b). From the results of the factorial experiments, a mathematical model was developed using Yates' algorithms. The factors with significant effects (identified through the use of hypothesis tests) from the results of the factorial experiments were then optimised using steepest ascent method and analysed using least squares method; the optimised parameters were confirmed by further experimental studies. Efficiency of the process was based mainly on chloride removal (Y,%), which was computed using equation (1a). The choice of chloride removal for efficiency determination was done on the basis of literature (Holderness and Lambert, 1978), which stated that removal of chloride ion from aqueous solution electrochemically can be achieved by using carbon electrode as anode. Chloride determination in both raw and treated wastewaters was carried out using argenotometric method specified in APHA (1998). The choice of argenotometric method was based on accuracy, type of wastewater (clear aqueous solution) and availability of required reagents.

$$Y = 100\frac{(C_o - C_t)}{C_o} \qquad (1a)$$

Table 1: Summary of 2^4 Fractional Factorial Design matrix

Experiments	Name and Code of the factors with the levels				Natural factors				Combinations
	Current through the electrodes (A_a)	Separation distance between the electrodes (X)	Volume of the wastewater used (V_o)	Contact surface area (A_c)	A_a (A)	X(mm)	V_o(ml)	A_c($x 10^{-3} m^2$)	
1	-	-	-	-	1	40	750.0	1.20	Mean
2	+	-	-	-	2	40	750.0	1.20	A_a
3	-	+	-	-	1	80	750.0	1.20	X
4	+	+	-	-	2	80	750.0	1.20	X A_a
5	-	-	+	-	1	40	1000.0	1.20	V_o
6	+	-	+	-	2	40	1000.0	1.20	A_a V_o
7	-	+	+	-	1	80	1000.0	1.20	X V_o
8	+	+	+	-	2	80	1000.0	1.20	A_a X V_o
9	-	-	-	+	1	40	750.0	3.20	A_c
10	+	-	-	+	2	40	750.0	3.20	A_a A_c
11	-	+	-	+	1	80	750.0	3.20	X A_c
12	+	+	-	+	2	80	750.0	3.20	A_a X A_c
13	-	-	+	+	1	40	1000.0	3.20	V_o A_c
14	+	-	+	+	2	40	1000.0	3.20	A_a V_o A_c
15	-	+	+	+	1	80	1000.0	3.20	X V_o A_c
16	+	+	+	+	2	80	1000.0	3.20	A_a X V_o A_c

Table 2: Parameters used in the establishment of the factors, interactions and mathematical model

Code	Parameters	Levels	
		Low (-1)	High (+1)
A_a (A)	Current through the electrodes	1.0	2.0
X(mm)	Separation distance between the electrodes	40	80
V_o(ml)	Volume of the wastewater used	750	1000
A_c($x 10^{-3}$ m^2)	Contact surface area	1.20	3.20

Specifically, 10 ml each of the samples was diluted to 100ml with distilled water in duplicates. The pH was adjusted to the range between 7.0 and 10.0 with 0.1 M NaOH. Then 1 ml of K_2CrO_4 indicator was added and the resulting solution was titrated with standard Silver Nitrate to a pinkish yellow end point. The procedure was repeated for blank (as a control). Chloride concentration was calculated using equation (1b)

$$Cl^-(in\ \ mg/l)\ \ 35450\frac{(A\ \ -B)NP}{volume\ \ of\ \ the\, sample} \qquad (1b)$$

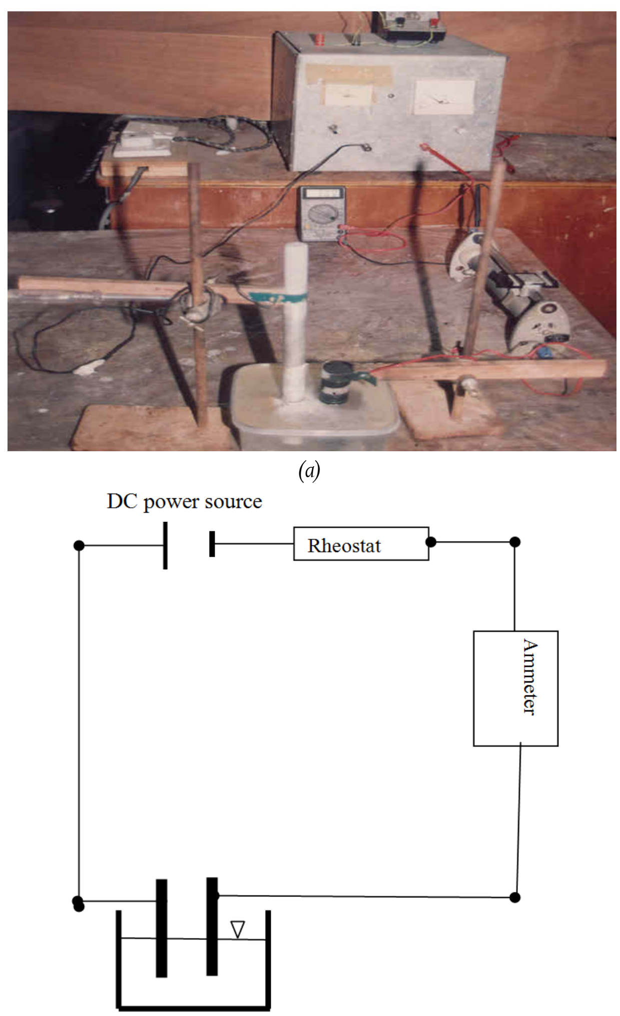


Figure 1 Laboratory Setup of the treatment process (a) Setup of the instruments (b) Schematic diagram

3. Results And Discussion

The results of the 2^4 factorial experiments, Yates' algorithms and significance analysis are presented in Table 3. Table 3 shows the response of each of the 2^4 factorial experiments. Efficiency of the process ranges from 2.3 to 52.4 % (averages). In all cases the least response occurred when separation distance and volume of wastewater were at the high levels, current through the electrodes and contact surface area were at the low levels (Table 3), that is, the interaction of separation distance and volume of wastewater produces the least chloride removal. This result indicates that these two factors (separation distance and volume of wastewater) are negative factors, which decreased efficiency of the process. Hence, the higher the values of these factors, the lower the efficacy of the treatment method.

From the table it can be seen that separation distance between the electrodes is a factor with negative effect on the efficiency of the process. This can be attributed to various properties of the electrolyte (wastewater) such as reduction in the conductivity and increase in internal resistance and reduction in electromotive force due to low conductivity. This observation is similar to observations made in relation to this factor by some researcher such as Golder *et al* (2006); Holt *et al* (2002); Khemis *et al* (2006); Kobya *et al* (2003); Koparal and Ogutveren (2002); Kobya *et al* (2006); Kumar *et al* (2004) and Mollah *et al* (2001) that higher separation distance between electrodes should be avoided to attain higher efficiency of the process.

The results reveal (Table 3) that volume of the wastewater used is a factor that can reduce efficiency of the process (negative factor, 7.53 % < 11.03%). This phenomenon can be attributed to increase in the number of pollutants to be removed and increase in the internal resistance due to an increase in the pollutant's concentration. Literature (Sanroman et al., 2005; Vlyssides *et al.*, 2004; Mondal , 2008) reported that for high flow rate of wastewater more retention time will be required to attain higher efficacy. This indicates that the result obtained using this factorial experiment agrees with literature.

Also, from the Table, the results show influences of some interactions [A_a and V_o] on the efficiency of the process. These two interactions influenced the process positively and can be attributed to overriding influence of current and contact surface area. Although there is no information in literature on interactions of these factors but data on these individual factors show that combinations of these two factors are important in electrochemical treatment of wastewaters (Diaz *et al.*, 2008; Mondal, 2008).

Also, in all cases the maximum efficiency occurred when A_c and A_a are at the high levels with low level of volume of wastewater and separation distance. This indicates that these two factors and their interactions are positive factors, which improve efficiency of the process.

Yates' algorithms were obtained using procedures stated in Guttman *et al* (1971) and Devore and Farnum (1999). Divisors (equation 2), sum of squares , effects, means of squares and F- values were obtained using methods stated in literatures (Gardiner and Gettinby, 1998; Devore, 2000; Oke, 2008). Table 3 shows standard matrix, total response, sum of squares, degree of freedom, mean of sum of squares and F values of the four selected factors (X,, V_o ; A_c , A_a) and their interactions.

Effects of factors and interactions shown in Table 4 can be grouped into two, namely:

i. Factors and interactions with negative coefficients, and
ii. Factors and interactions with positive coefficients.

The factors and interactions with negative coefficients are the factors and interactions that have negative influence on the efficiency of the process. This indicates that higher values of these

Table 3: Summary of 2^4 factorial experimental results and statistical analysis

Experiments	Factors				Total chloride removed (%)	Chloride removed		Effect (eff)	Statistical Analysis		
	Current through the electrodes (A_a)	Separation distance between the electrodes (X)	Volume of the wastewater used (V_o)	Contact surface area (A_c)		Mean	Standard deviation		Sum Square (SSQ)	Mean of Sum of Squares (MSS)	F-ratios
1	-	-	-	-	44.10	11.03	0.750	19.14	5863.73	-	-
2	+	-	-	-	89.10	22.28	1.209	13.32	2838.23	946.08	13.00
3	-	+	-	-	14.60	3.65	0.370	-9.71	1509.32	503.11	6.91
4	+	+	-	-	44.90	11.23	0.562	-3.98	253.61	84.54	1.16
5	-	-	+	-	30.30	7.58	1.081	-4.70	353.44	117.81	1.62
6	+	-	+	-	72.10	18.03	1.970	0.64	6.63	2.21	0.03
7	-	+	+	-	8.10	2.03	1.100	3.96	251.22	83.74	1.15
8	+	+	+	-	66.90	16.73	0.974	1.36	29.43	9.81	0.13
9	-	-	-	+	110.50	27.63	0.597	15.16	3675.39	1225.13	16.83
10	+	-	-	+	209.60	52.40	0.416	2.33	86.49	28.83	0.40
11	-	+	-	+	73.90	18.48	0.250	-3.39	184.28	61.43	0.84
12	+	+	-	+	101.70	25.43	1.401	-4.13	272.25	90.75	1.25
13	-	-	+	+	60.70	15.18	0.911	-3.74	224.25	74.75	1.03
14	+	-	+	+	151.60	37.90	1.283	-0.94	14.06	4.69	0.06
15	-	+	+	+	57.90	14.48	1.044	1.07	18.28	6.09	0.08
16	+	+	+	+	89.80	22.45	1.271	-0.62	6.25	2.08	0.03

factors and interactions reduce efficiency of the process. The reverse is the case for factors and interactions with positive coefficients.

Similarly, the F- values presented in Table 4a and b can be categorized into three as follows:

i. Those interactions with F- values less than unity,
ii. Those factors and interactions with F- values greater than one but less than the critical F value at 90.0% confidence level, and
iii. Those factors with F- values greater than the critical F-value at 90.0 % confidence level.

The grouping indicates that there are interactions with negligible effects ($F<1$), factors and interaction with little effects ($1<F < F_c$) and factors with significant effects ($F>F_c$). From the coefficients and F- values the experimental parameters such as A_a, A_c and X have significant effects on the efficiency of the process, volume of the wastewater used has no significant effect (F = 1.62; 2.91) on the efficiency of the process. It can be seen that the critical value for the F-ratios (statistical test comparison) in Table 3 are 2.25 respectively. Based on this we can conclude that factors A_a, , A_c and X are significant at 99.5% significance level indicating that they have significant influence on efficiency of electrochemical treatment method. The significance of these three factors can be attributed to the fact that in electrochemical treatment processes a lot of reactions take place depending on the type of electrodes used. For example, when active electrodes such as iron and aluminium are used, the main reactions are electro-coagulation and electro-oxidation simultaneously and when passive electrodes such as carbon, gold and titanium are used electro-adsorption, electro precipitation and electro-oxidation are the main reactions.

The influence of these factors on overall efficacy of the process is as presented in Figure 2a and b. The response prediction tested with an actual efficiency of the process and the correlation coefficient (R^2) is 0.9994 (Fig 2c). Though, this value indicates that the model agreed with the actual efficiency of the process, a slight difference in the values of actual and experimental efficiencies can be attributed to losses in the energy supplied due to eddy, heat and other forms.

Results from the further studies are presented in Figures 3 to 6. Figure 3 shows effects of current through the electrodes on the efficiency of the process. From the figure it can be seen that as current increases at a fixed time, the amount of chloride removed increases. Similarly, when the time decreases at a fixed current through the electrodes, the chloride removed decreases. When both the current through the electrodes and the time increase it can be seen that chloride removed increases exponentially (Fig.3). This result indicates that the higher the current the higher the efficiency and confirms the results from factorial experiments and optimization.

Figure 4 presents influences of volume of wastewater on the efficiency of the process. Like figure 3, it can be seen from the figure that as volume increases the amount of chloride removed decreases (a negative factor). When the volume of the wastewater decreases, it can be seen that chloride removed increases dramatically (Fig.4).This result shows that the higher the volume, the lower the efficiency and confirms the results from factorial experiments and optimization. It can be explained further that efficiency of the process is a function of discharge and retention time (as volume can be expressed as equation 2). This indicates that efficiency of the process decreases with increasing flow rate and increases with increasing retention time. In addition to all these, it means that Faraday's first law (removal of dissolved substance is proportional to quantity of electricity passed) can be written mathematically as equation 3). Equations (4 and 5) show that the higher the volume the higher the time and current required for effective treatment (higher efficiency).

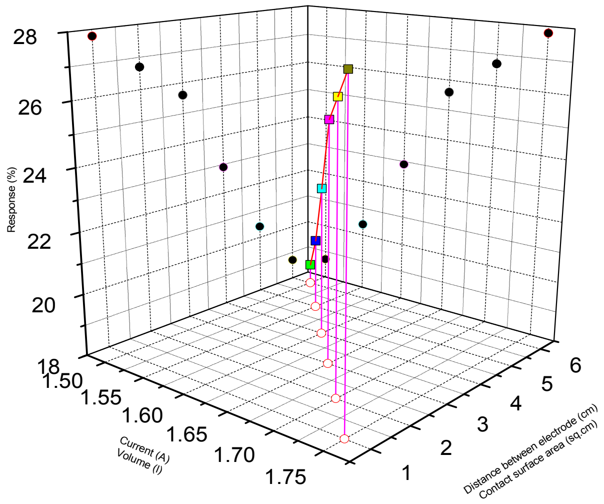
$$V = Qt \tag{2}$$

$$M = kIt = Qt\rho \tag{3}$$

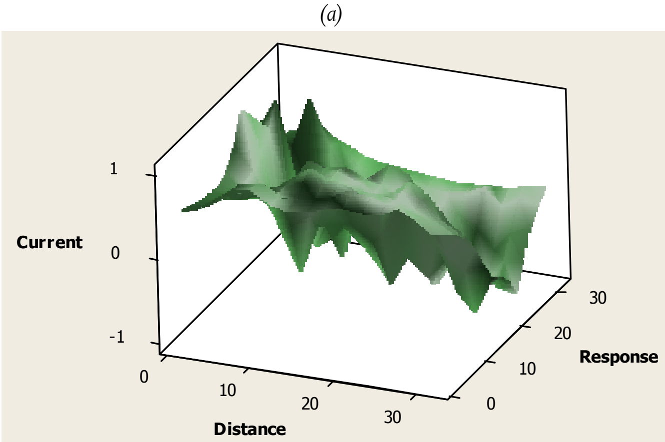
From equation (3) the volume of wastewater treated at a particular time can be expressed as follows:

$$M = kIt = V\rho \tag{4}$$

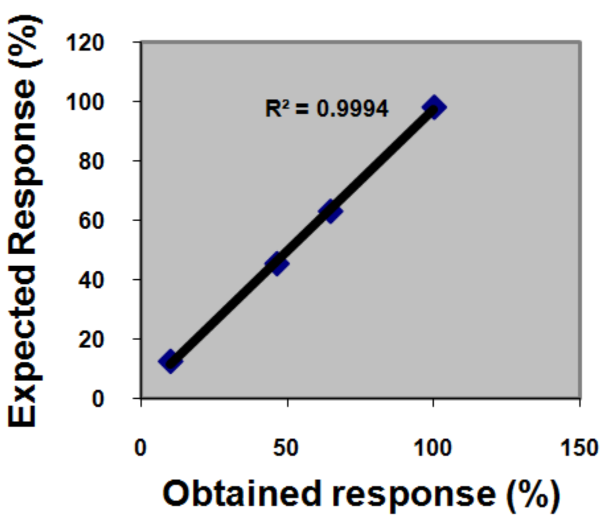
$$V = \frac{k}{\rho} It \tag{5}$$



(a)



(b)



(c)

Fig. 2 Relationship between factors and response (a) In 3D with trajectory line (b) Surface plot of current against response (c) experimental response and expected response

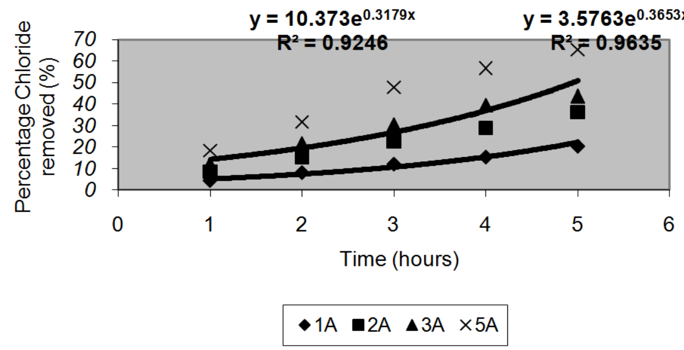


Fig. 3: Influence of current on chloride removal

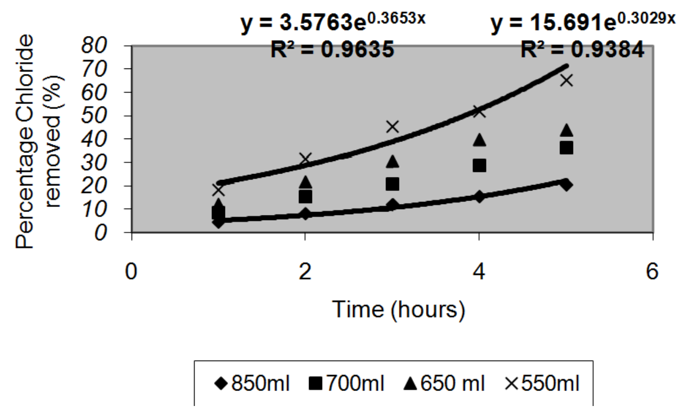


Fig. 4: Influence of volume of wastewater used on chloride removal

Figure 5 shows the influence of contact surface area of the electrodes on the efficiency of the process, which indicates that the higher the contact surface area the higher the efficiency. This also agrees with the results from factorial experiments and optimization. This indicates that efficiency of the process is a function of depth and width of the electrodes, i.e. higher depth or /and width of the electrodes will increase efficiency of the process.

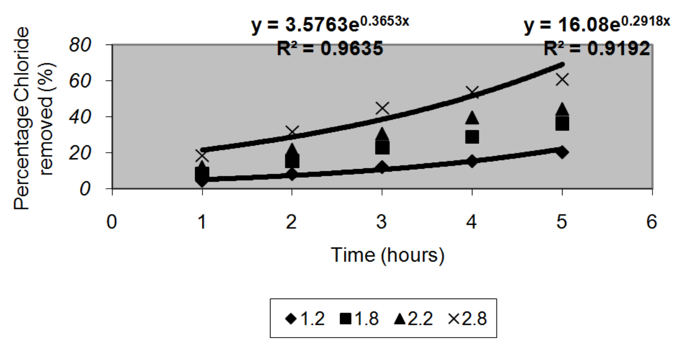


Fig. 5: Influence of electrode's surface contact area on chloride removal

Figure 6 represents influence of separation distance between the electrodes on the efficiency of the process, which indicates that the higher the separation distances between the electrodes the lower the efficiency. This confirms the results from factorial experiments and optimization. This can be explained further that the higher the distance apart the lower the efficiency, as more energy will be required to overcome resistance of the wastewater.

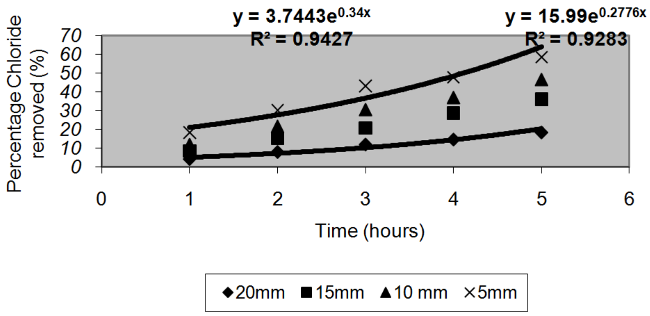


Fig. 6: Influence of separation distances between the electrodes on chloride removal

4. CONCLUSIONS

It can be concluded based on the study that:

i. The factorial experiments used in this study enable the determination of the significant factors that influence the efficiency of an electrochemical treatment process.
ii. Using a two-level factorial design matrix taking into account all interaction effects appeared to be an efficient means of obtaining a mathematical model in conformity with criteria validity.
iii. The predictions given by the factorial experiments agreed with the actual experimental results.
iv. Among factors that influence efficiency of electrochemical treatment process significantly are current, contact surface area and separation distance.

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Abbreviations

A	ml of the titrate used for the sample (ml).
B	ml of the titrate used for the blank (ml).
N	normality of Silver Nitrate
P	dilution factor
C_o	initial chloride concentration in the synthetic wastewater (mg/l).
C_t	final chloride concentration in the synthetic wastewate (mg/l)
Y	chloride removed (%)
K	number of the factors examined (4)
SSQ	sum of squares
MSS	mean of sum of squares
V	volume of the liquid (m^3)
Q	flow rate (discharge in m^3/d)
I, t , k and ρ	current, retention time, proportionality constant and density (A, s, kg/C and Kg/m3) respectively.
Ac	Contact surface area
Vo	Volume of the wastewater used
X	Separation distance between the electrodes
Aa	Current through the electrodes