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Effecting of pH Parameter on Simulated Wastewater Treatment Using Electrocoagulation Method

Wadood T. Mohammed	ForatYasirAlJaberi
College of Engineering-University of Baghdad	College of Engineering-University of Baghdad
wadood.taher@gmail.com	furat_yasir@yahoo.com

ABSTRACT

The aim of the present research is to investigate the effecting of pH parameter on the feasibility of lead removal from simulated wastewater using an electrochemical system. Electrocoagulation method is one of electrochemical technology which is used widely to treat industrial wastewater. Parameters affecting this operation, such as initial metal concentration, applied current, stirrer speed, and contact time of electroprocessing were taken as 155ppm, 1.5 Ampere, 150 rpm, 60 minutes respectively. While pH of the simulated wastewater was in the range of 2 to 12 in the experiments. It was found from the results that pH is an important parameter affecting lead removal operation. The best value of pH parameter is approximately (9.50) that gave higher removal efficiency larger than (99%).

Keywords: heavy metals, wastewater treatment, electrocoagulation, pH parameter.

تأثير الدالة الحامضية على معالجة مياه الصرف المحاكاة باستخدام طربقة التخثير الكهربائي

فرات ياسر الجابري قسم الهندسة الكيمياوية-كلية الهندسة- جامعة بغداد **أ.د. ودود طاهر محمد** قسم الهندسة الكيمياوية-كلية الهندسة- جامعة بغداد

الخلاصة

الهدف من الدراسة هو لمعرفة مدى تأثير الدالة الحامضية pH على امكانية از الة ايونات الرصاص من مياه الصرف المحاكاة باستخدام التقنية الالكتروكيميائية. تعتبر طريقة التخثير الكهربائي من التقنيات الالكتروكيميائية والتي تستخدم بشكل واسع لمعالجة المياه الصناعية. قيم المؤثرات الاخرى للعملية كالتركيز الاولي للمعدن والتيار الكهربائي وسرعة الخلط وزمن التلامس لتلك العملية تم اعتمادها كقيم ثابتة وهي على التوالي 155 ملغم لكل لتر و 1.5 امبير و150 دورة بالدقيقة و 60 دقيقة. بينما مدى الدالة الحمضية قيد الدراسة في التجارب قد كان 2 الى 12. وقد تبين من خلال التجارب ان القيمة الافضاللدالة الحامضية لاعطاء كفاءة اعلىلاز الة الرصاص هي (9.50)وبكفاءة از الة اكثر من (99%).

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1. INTRODUCTION

The continuous development of industries caused to dischargethe heavy metals directly or indirectly discharged into the environment. In general, organic contaminants are biodegradable, whereas the heavy metals are not. Moreover, heavy metals tend to accumulate in living organisms and its ions are known to be toxic or carcinogenic, Srivastava, and Majumder, 2008, such as cadmium, lead, and chromium that must be treated to protect the environment, Fenglian, and Wang, 2011. Practically, there are several techniques that can be used to remove heavy metals from wastewater such as adsorption, chemical precipitation, membrane filtration, coagulation, and electrochemical methods , Yang, 2003. Electrocoagulation is one of electrochemical types, which uses an electricity through the electrodes to remove pollutants from solution such as suspended solids, dissolved metals, and dyes. Electrocoagulation system is more efficient and cost-effectiveness rather than chemical coagulation technique which is not efficient, Fekete, 2016,. The contaminants present in wastewater such as lead ions are maintained in solution by electrical charges, Issabayeva, and Kheireddine, 2006. Electrocoagulation process has the ability to eliminate the drawbacks of the classical treatment techniques such as the large amount of sludge and the chance of causing secondary contamination of waterto achieve a sustainable and economic treatment of polluted industrial wastewater, Bazrafshan, et al., 2015.

Several parameters affect the efficiency of electrocoagulation process such as initial concentration of the metal, contact time, pH, current density or current, cell voltage, anode-cathode materials, batch or continuous, flow rate, inter-electrodes distance, conductivity of solutions, energy consumption, and the electrodes configuration. The value of pH of the simulated wastewater is an important parameter used to control the distribution of charge on the adsorbent, **Bayar**, **2013**, and the consumption of the used electrodes and the energy in the electrocoagulation cell.

In Electrocoagulation technique, water is electrolyzed in a parallel reaction when a potential is applied from a DC-Power supply, **Bayar**, **2013**. Oxidation and reduction operations occur on anode and cathode electrodes respectively as explained below:

• At the anode electrode with metal **M**:

$$M_{(S)} \rightarrow M^{+n}_{(aq)} + ne^{-}$$
(1)

 $2H_2O \rightarrow O_2 + 4H^+ + 4e^{-}$
(2)

• At the cathode electrode:

$$\mathbf{M}^{+n}_{(\mathrm{aq})} + \mathbf{n}\mathbf{e}^{-} \not\rightarrow \mathbf{M}_{(\mathrm{S})} \tag{3}$$

$$2H_2O + 2e^{-} \rightarrow H_{2(g)} + 2OH^{-}_{(aq)}$$

$$\tag{4}$$

In the present study, concentric aluminum electrodes were used, as explaining its configuration in **Table 1** with an active area equal to 283 cm^2 , to examine the effects of pH parameter under specified parameters on the lead removal efficiency, energy consumption, and electrodes consumption.



(5)

2. EXPERIMENTAL WORK

2.1 Apparatus

The schematic of electrocoagulation cell shown in **Fig.1** and **Fig.2**, which consists of three concentric aluminum tubes with different diameters and thicknesses as given in **Table1** with an active area of approximately 285 cm². Other tools as follow:

- 1- Digital DC- power supply (SYADGONG company-305D); 0-30 volt and 0-5Ampere.
- 2- Digital balance (500g x 0.01g) (PROF company).
- 3- Magnetic Stirrer (ALFA company: HS-860); 0-1000 rpm.
- 4- pH meter (ATC company).
- 5- Digital timer (SEWAN company).
- 6- Concentric Aluminum tubes with different diameters and thicknesses.

The batch electrocoagulator is made of plexiglass with a volume of 1000 ml. The parameters selected in the present experiments were described as follows in **Table 2**.

2.2 Materials

Simulated wastewater samples with an initial concentration of lead were prepared by dissolving leadnitrate $Pb(NO_3)_2$ in distillate water. Where the required mass of this salt could be measured according to the following equation, **Mohammed**, **2010**:

$$W = V \times C \times (M / m_A)$$

Where:

W: Weight of salt (grams); *V*: Volume of solution (liter); *C*: Initial concentration of lead ions in solution (ppm); *M*: M.wt of the lead nitrate; m_A : Atomic weight of lead.

In order to prevent the formation of an oxide layer on the anode electrode and to increase the conductivity of the simulated solution, an amount of sodium chloride was added.

Hydrochloric acid (0.1 N) and sodium hydroxide (0.1 N) was used to adjust the value of pH to be the solution neutral.

The simulated wastewatersof the experiments were prepared by dissolving $Pb(NO_3)_2$ having 99.99 of purity (*B.D.H- England*) in 500mL of distilled water. Electrical conductivity and decreasing passivation enhance removal efficiency by using 0.5 g/L of NaCl.

When the electrodes of the concentric tubes immersed in the synthesis wastewater, DC- current switched on to supply 1.5 Ampere (5.3 mA/cm^2) to the cell. Samples are collected from the treated simulated wastewater each 10 minutes and filtered by the cellulose Glass-Microfibre discs (Grade: MGC; pore diameter is 0.47 micrometer- MUNKTELL) before the analysis by the Atomic Absorption Spectroscopy (AAS- Type-SHIMADZUAA-7000*F*) for measuring quantities of lead ions present in the samples. At the end of each experiment, electrodes were washed one time with 0.1 N HCl and more than one time with water to ensure it was cleaned well. The same procedure was repeated for the next experiment according to the planned schedule.

3. RESULTS AND DISCUSSION: 3.1 Removal efficiency

Fig. 3. shows the increasing of removal efficiency with pH value until reach pH larger than approximately 9 then started to minimize. The general correlations and their coefficient of determination \mathbf{R}^2 , that relateremoval efficiency with timewhen other independent variables arefixed as shown in Table 3.

Curves in **Fig.3**. explain the different behavior of the process with time according to the value of pH of the solution. As seen, pH (2, 4, 6, and approximately 8) have the same behavior, their curves increase gradually then be stable. While other curves pH (10, and 12) increasing then decreasing at a different time for each one.

This ununiform behavior of curves refers to the different amount of Hydrogen releasing from the electrode which causes to change the value of acidity of the solution with time. If the value of pH of the solution is neutral or less then it will be increased and if it equal to 10 or more then it will be decreased. These changes in pH values will affect the efficiency of the preferred value of lead removal.

3.2 Energy consumption

It is an effective parameter for this kind of wastewater treatment technique which is determined according to the following equation:

$$E = (U. I. t)/(1000 .V)$$
(6)

where: **E**: energy consumption (kWh/m³),**U**: the applied voltage (volt), **I**: applied current (Amps.), *t*: contact time (h or min.), and **V** is the volume of the simulated wastewater (m³).

In the present study, energy consumption is directly proportional with all of pH values as shown in **Fig. 4** and their general correlations that relate this response with time are explained in **Table 4**. The designed value of energy consumption is determined belong to the required percentage of lead removal.

3.3 Electrode consumption

In this technique, some weight of both electrodes was consumed and its values varied with the value of pH. Where the consumption of electrodes is determined according to the quantity of lead removal which it equals to amount of aluminium hydroxyl adsorbent formed. In addition, weights of both electrodes are measured before and after each experiment.

3.3.1 Anode consumption

Fig. 5 and **Table 5** explain the consumption of anode electrode caused by the applied current supplied to the electrocoagulation cell via the DC supplier. It is value increases as pH value increased.

3.3.2 Cathode consumption

Similar to the anode electrode, cathode also consumed and minimized in its weight because of electricity supplied to the cell. Fig. 6 and Table 6, explain its behavior and their correlations



related to pH values. The value of consumption increases when the value of alkalinity rising as explained in eq.(10)Interpretation.

Figs. 7 to 10, explain the relation of the responses (i.e. the dependent variables) with the initial pH and also with the final pH that measured at the end of each experiment. Moreover, **Table 7** explains the summary of correlations that relate each of responses with the initial and final pH.All experiments are repeated three times and their results are pointed in these figures and drawn using Statistica-10 program.

The value of acidity and alkalinity (i.e. pH) of the simulated wastewater is an important parameter used to control the distribution of charge on the adsorbent surface between the adsorbate ion. Where the increase of pH value to a specified limit can increase the adsorption rate but, the further increment will minimize this rate (i.e. adsorbent ion tends to be unaffected) **Sulaymon, et al., 2013** and **Aji, et al., 2012**.

The initial value of pH was varying between 2 and 12 to investigate how far its effect on the removal efficiency. It is found that pH value is not fixed during reaction time for all its intervals investigated. It increases when its initial value was less than 10, whereas it decreased when its initial value is 10 or more, it is attributed, as suggested by many investigators, Salim, et al., 2015, to the dissolution reactions of aluminum and its hydroxide as $Al(OH)_4^-$ which are alkalinity consumers. In electrocoagulation, heavy metal removes either by precipitation following the cathode reduction or by adsorption on the aluminum hydroxide or its polymers ,Lekhlif, et al., 2014 and Bayar, 2013. When the pH value is favorable, the precipitation of $Al(OH)_3$ occurs according to the following reactions:

1- Reduction of water in the cathode electrode:

$$6H_2O + 6e^- \rightarrow 3H_2 + 6OH^-$$
(7)

2- Releasing of (Al^{+3}) cations in the electrodes:

$$2AI \rightarrow 2AI^{+3} + 6e^{-1}$$
(8)

3- For acidic medium, an amphoteric behavior of Al(OH)₃ :

$$Al^{+3} + 3OH^{-} \leftrightarrow Al(OH)_{3}$$
(9)

4- For alkaline medium, monomeric anions will release:

$$2Al_{(s)} + 6H_2O + 2OH_{(aq)} \rightarrow 2Al(OH)_{4(aq)} + 3H_{2(g)}$$

$$(10)$$

Different hydroxo-complexes including monomers and polymers will be formed, as shown in **Figs. 11-12**, when the value of pH increases such as $Al(OH)^{+2}$, $Al(OH)_{2^{+}}$, $Al_{2}(OH)_{2^{+4}}$, $Al_{6}(OH)_{15^{+3}}$, $Al_{7}(OH)_{17^{+4}}$, $Al_{8}(OH)_{20^{+7}}$, $Al_{13}O_{4}(OH)_{24^{+7}}$, and $Al_{13}(OH)_{34^{+5}}$.

The neutral form of Al(OH)₃is polymerized, as follows, to form flocs which have the high-flocking capacity **,Lekhlif, et al., 2014, Sulaymon, et al., 2013** and **Bayar, 2013**, to eliminate the pollutants from wastewater by adsorption process via electrocoagulation cell:



 $n \operatorname{Al}(OH)_3 \rightarrow \operatorname{Al}_n(OH)_{3n}$

and gives place to some aluminates in the basic medium as shown below which is considered as the precursor of Al_{13} polymer, **Lekhlif**, et al., 2014.

$$Al(OH)_3 + OH \rightarrow Al(OH)_4$$

Amorphous Al(OH)₃ formed by electrocoagulation process are much larger than chemical flocs and more stable that can be separated faster by filtration **,Ghanim, and Ajjam, 2013**, and it has the minimum solubility within the favorable range of pH. Therefore, the highest removal efficiency can be achieved in the medium and minimum solubility of the aluminum hydrolysis products in the specified range of pH.

Therefore, the adsorption behavior of lead that depends upon the quantity of adsorbent (aluminum hydroxide) generated, which is related to the time, applied current, and pH that has been investigated in the range from 2 to 12. In electrocoagulation process, pH values restricted according to the following reactions **,Salim, et al., 2015**:

$$2H_2O + 2e^{-} \rightarrow H_{2(g)} + 2OH_{(aq)}^{-}$$
(13)

$$2H_2O + O_2(g) + 4e^{-} \rightarrow 4OH_{(aq)}$$

At low pH, Pb^{+2} ions had to compete with H^+ ions for adsorption sites on the adsorbent surface. As *pH* increased beyond to the reactions(12) and (13), this competition weakens and more Pb⁺² ions were able to replace H^+ ions bound to the adsorbent surface **,Vasudevan, et al., 2011**,. Moreover, the low value of pH could not reach the desired levels for the formation of amorphous Al(OH)₃ with high-flocking capacity then the electrocoagulation process will not be efficient. So, initial pH is an important parameter and plays a significant role in the electrochemical removal of heavy metals from the simulated wastewater.

The highest removal efficiency obtained when the initial pH value approximately equals 9.5 and other parameters are fixed. Other responses values at this value of pH are listed in **Table 8**.

Fig. 13shows the relation between the initial and final values of pH which explains when the final pH minimized or maximized depending on the initial pH. The correlation of both variables is:

Final pH = -0.0443 (initial pH value)² + 0.9981(initial pH value) + 3.8651 (15)

4. CONCLUSIONS:

The following results are obtained from the investigation of pH behavior in the electrocoagulation cell:

- 1- The value of acidity and alkalinity (i.e. *pH*) of the simulated wastewater is an important parameter used to control the distribution of charge on the adsorbent.
- 2- initial pH is an important parameter and plays a significant role in the electrochemical removal of heavy metals from the simulated wastewater.
- 3- It is found that pH value is not fixed during reaction time for all its intervals investigated.Practical correlation relates initial and final pH had been found.

(11)

(12)

(14)



- 4- the adsorption behavior of lead that depends upon the quantity of adsorbent (aluminum hydroxide) generated, which is related to the time, applied current, and pH that has been investigated in the range from 2 to 12. It is found that the best value of pH to obtain maximum removal efficiency is approximately 9.5at fixed other parameters.
- 5- The values of other responses at pH equals 9.5 show that the energy and electrodes consumption wasaltered in the final pH curves. The designed value of energy and electrodes consumption are belong to the required value of lead removal efficiency.

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NOMENCULTURE

W=weight of salt,g.

V=volume of the simulated wastewater, liter or m³.

C=initial concentration of lead nitrate in solution, ppm.

M=M.wt of the lead nitrate, g/mole.

m_A=atomic weight of lead, g/mole.

E= energy consumption, kWh/m^3 .

U= applied voltage, volt.

I= applied current, Ampere.

t= contact time, h or minute.





Figure 1. Concentric electrodes.





Figure 2. The schematic of electrocoagulation reactor system.

Table 1. Electrodes configuration in the	e electrocoagulation cell.
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Configuration type	Ele	ctrodes
	Anode electrode(s)	Cathode electrode(s)
one cathode – two anodes	Outer and inner	Mid only

Table 2. Experimental parameters.

Parameters	Range or fixed value
Initial lead concentration (ppm)	155
pH	2-12
Current or current density (A or mA/cm ²)	1.5 or 5.3
Stirring speed (RPM)	150
Contact time (min)	60





Figure 3. Removal Efficiency .vs. time with several constant pH at fixed other parameters.

Table 3. Correlations of removal efficiency vs. time with several constant p	pH at fixed other
parameters.	

pН	Removal efficiency correlations	\mathbb{R}^2
2	$y = -0.0225x^2 + 2.8289x + 3.6328$	1.000
4	$y = -0.0225x^2 + 2.4435x + 30.192$	1.000
6	$y = -0.0225x^2 + 2.0581x + 52.023$	1.000
8	$y = -0.0225x^2 + 1.6727x + 69.126$	1.000
10	$y = -0.0225x^2 + 1.2873x + 81.501$	1.000
12	$y = -0.0225x^2 + 0.9019x + 89.148$	1.000



Figure 4. Energy consumption.vs. time with several constant pH at fixed other parameters.



Fable 4. Correlations of Energy consumption vs. time	with several constant pH at fixed other
parameters.	

pН	Energy consumptioncorrelations	\mathbb{R}^2
2	$y = -0.001x^2 + 0.3728x - 1.7291$	1.000
4	$y = -0.001x^2 + 0.3673x - 0.7885$	1.000
6	$y = -0.001x^2 + 0.3618x - 0.3079$	1.000
8	$y = -0.001x^2 + 0.3563x - 0.2873$	1.000
10	$y = -0.001x^2 + 0.3508x - 0.7267$	1.000
12	$y = -0.001x^2 + 0.3453x - 1.6260$	1.000



Figure 5. Actual anodes consumption .vs. time with several constant pH at fixed other parameters.

Table 5. Correlations of Actual anodes consumption vs. time with several constant pH at fixed other parameters.

pН	Actual anodes consumption correlations	\mathbb{R}^2
2	$y = 1E - 04x^2 - 0.0064x + 0.2219$	1.000
4	$y = 9E - 05x^2 - 0.0024x + 0.2004$	1.000
6	$y = 9E - 05x^2 + 0.0016x + 0.1389$	1.000
8	$y = 9E - 05x^2 + 0.0057x + 0.0377$	1.000
10	$y = 0.0001x^2 + 0.0071x - 0.0575$	0.9982
12	$y = 0.0003x^2 + 0.0003x - 0.0270$	0.9936



Figure 6. Cathode consumption vs. time with several constant pH at fixedother parameters.

Table 6. Correlations of cathode consum	ption vs. time	with several	constant pH a	at fixedother
	parameters.			

pH	Cathode consumption correlations	\mathbb{R}^2
2	$y = 7E - 05x^2 - 0.0003x + 0.0581$	1.000
4	$y = 8E - 05x^2 + 0.0004x + 0.0453$	1.000
6	$y = 7E - 05x^2 + 0.0011x + 0.0283$	1.000
8	$y = 7E - 05x^2 + 0.0018x + 0.0073$	1.000
10	$y = 7E - 05x^2 + 0.0025x - 0.0178$	1.000
12	$y = 7E - 05x^2 + 0.0032x - 0.0470$	1.000



Figure 7. Removal efficiency .vs. the initial and final pH at fixed other parameters.



Figure 8. Energy consumption .vs. the initial and final pH at fixed other parameters.



Figure 9. Actual anodes consumptionvs. the initial and final pH at fixed other parameters.



Figure 10. Cathode consumption vs. the initial and final pH at fixed other parameters.

Responses	рН Туре	Correlations
Removal	Initial pH	$y_1 = -0.543 x^2 + 10.374 x + 49.381$
efficiency%	Final pH	$y_2 = -2.198 x^2 + 43.983 x - 118.430$
Energy	Initial pH	$y_1 = 0.124 x^2 + 0.751 x - 1.528$
Consumption	Final pH	$y_2 = 0.658 x^2 - 6.582 x + 17.838$
Actual anode	Initial pH	$y_1 = 0.004 x^2 + 0.022 x - 0.059$
consumption	Final pH	$y_2 = 0.025 \ x^2 - 0.269 \ x + 0.784$
Cathode	Initial pH	$y_1 = 0.002 \ x^2 + 0.009 \ x - 0.005$
consumption	Final pH	$y_2 = 0.012 x^2 - 0.125 x + 0.369$

 Table 7. Summary of responses correlations.



Figure 11. Electrocoagulation cell shows monomer phases formed by Al⁺³ as a function of the pH, Lekhlif, et al., 2014.



Figure 12. Aluminum hydrolysis species distribution as a function of pH, Lekhlif, et al., 2014.

Table 8. Summary of responses values at pH equals 9.5.

Responses	рН Туре	Value
Energy	Initial pH	17.2 kWh/m^3
Consumption	Final pH	15.1 kWh/m ³
Actual anode	Initial pH	0.53 g
consumption	Final pH	0.47 g
Cathode	Initial pH	0.28 g
consumption	Final pH	0.24 g





Figure 13. Final pH vs. initial pHat fixedother parameters.