

Removal Efficiency of Polycyclic Aromatic Hydrocarbons From Synthetic Water Samples By Electrocoagulation Processes

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ABSTRACT

In the present work, batch electrocoagulation experiments were carried out to evaluate the removal of polycyclic aromatic hydrocarbon (PAHs) from water using aluminum electrodes. The effects of initial pH, current density, electrolysis time, initial concentration of PAHs, electrolyte type, and electrolyte concentration were investigated to achieve the optimal removal efficiency. The results indicated that the electrocoagulation utilizing the aluminum, as anode and cathode, was an efficient tool in the reduction of these contaminants. The treatment process was found to be largely affected by the current density and the initial composition of water. The removal rate was significantly increased using NaCl as an electrolyte where indirect oxidation by hypochlorite forming later during the treatment was occurred. The results demonstrated that the technical feasibility of the electrocoagulation as a possible and reliable technique for the treatment of PAHs contaminants in water.

Keywords: Polycyclic aromatic hydrocarbons (PAHs), Water, Treatment, Electrocoagulation, Aluminum electrode, HPLC.

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PAH

(PAHs)

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1. Introduction

The family of polycyclic aromatic hydrocarbon (PAH) pollutants has been found in different water bodies all over the world, and these compounds pose, even at very low concentrations, a great threat to ecological and human health due to their benzene structures [1]. PAHs are listed as US-EPA and EU priority pollutants, and their concentrations therefore need to be controlled, but only 16 are monitored by US-EPA and the Environmental Commission of European Community.

PAHs are by-products of incomplete combustion or hydrolysis of organic materials with recalcitrant and strong mutagenic/carcinogenic properties [1-2]. PAHs are hydrophobic compounds, once they enter into the water systems; it is difficult to be removed by conventional methods, as PAHs are persistent, and non-reactive in water [1-2] mainly due to their low water solubility [1,3].

The electrocoagulation technique involves the generation of coagulants by electro oxidation of an appropriate sacrifice anode [4-5]. In recent years, electrocoagulation was evaluated and applied in the treatment and purification of water and wastewater [4,6-7]. Electrocoagulation is efficient in removing suspended solids as well as oil and greases [4,8]. It removes the pollutants from aqueous media by introducing highly charged polymeric metal hydroxide species. These species neutralize the electrostatic charges on suspended solids and oil droplets to facilitate agglomeration or coagulation [4,9]. The treatment prompts the precipitation of certain metals and salts.

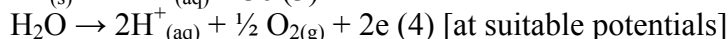
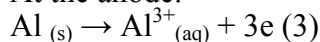
The reaction occurring in an electrochemical cell using aluminum electrodes are as follows [4,9]:

At the cathode:

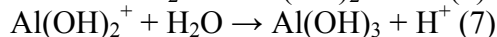
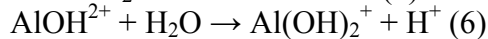


$\text{H}_2\text{O} + 1/2 \text{O}_2 + 2\text{e} \rightarrow 2\text{OH}^-_{(\text{aq})}$ (2) [when oxygen exists in treated solutions]

At the anode:



In the solution:



Since the electrocoagulation does not require any addition of chemicals, the quantity of coagulant released in solution depends on the applied cell potential (or current density). Some of the advantages of the electrocoagulation are the simple equipment required and the easy automation of the process [4-5]. Other advantages include the enhanced of the flocculation process, caused by the turbulence generated by the hydrogen and the oxygen evolution, according to equations 1 and 4 respectively, which produces a soft mix, and helps the destabilized particles generate bigger particles. Furthermore, the formed hydrogen and oxygen bubbles increase the efficiency of the separation process through electrofloatation [4,10].

The aim of this work, therefore, is to evaluate the removal of PAHs from water using aluminum electrodes at different operating conditions. Several parameters, namely initial pH, current density, PAH concentrations, electrolyte type and concentration, and electrolysis time, were investigated for their effects on the removal efficiency.

2. Experimental

2.1. Materials and Chemicals

Acetonitrile, methylene chloride, and water were of HPLC grade, purchased from Acros organics (Geel, Belgium). A certified standard mixture solution (EPA 610 PAH Mix), used for calibration and spiking, was purchased from Supelco Analytical (Bellefonte, USA) and containing 2000 mg L⁻¹ of acenaphthylene, 1000 mg L⁻¹ of each of acenaphthene, and naphthalene, 200 mg L⁻¹ of each of benzo (b) fluorene, benzo (g,h,i) perylene, dibenzo (a,h) anthracene, fluorene, fluorene, and 100 mgL⁻¹ of each of anthracene, benzo (a) anthracene, benzo (a) pyrene, benzo (k) fluoranthene, chrysene, indeno (1,2,3-cd) pyrene, phenanthrene, and pyrene. Fig.1 illustrates the structure of 16 the PAHs on the EPA priority pollutant list.

2.2. Instruments

HPLC analysis was done using a smartline HPLC system manufactured by Knauer GmbH (Berlin, Germany) consisted of degasser, gradient pump, manual injector with 20 µL loop, column oven, photo diode array and fluorescence detectors.

Chromatographic separation was performed on 250 x 4.6mm, Supelcosil LC-PAH column (Supelco Analytical, Bellefonte, USA). A PC interfaced to the HPLC using ClarityChrom software (Knauer GmbH) was used for data acquisition and processing.

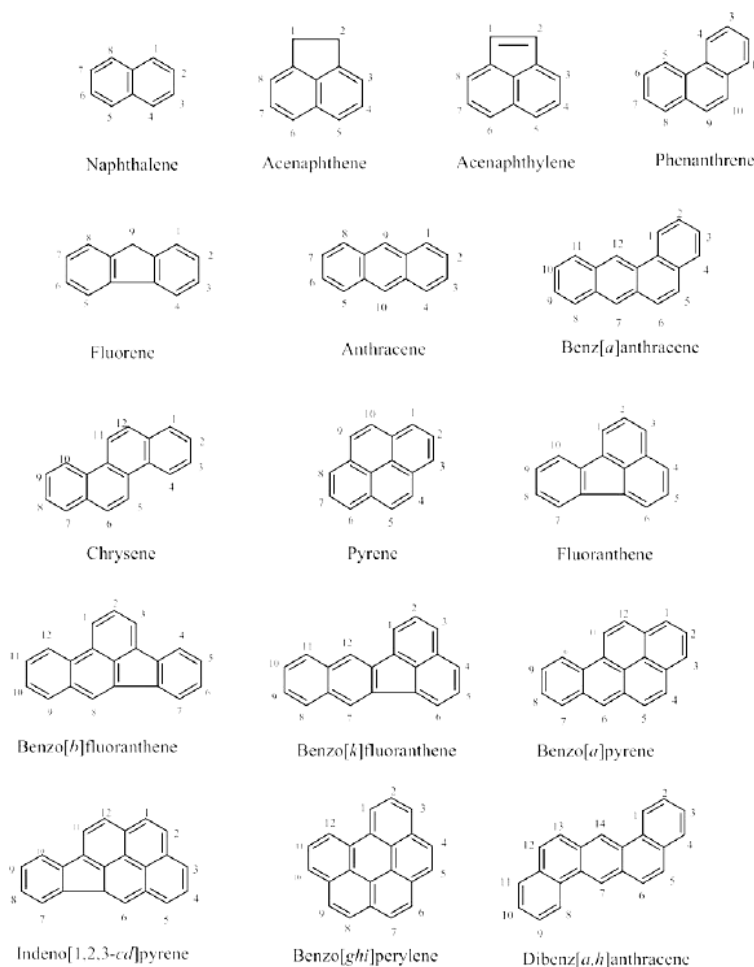


Fig. 1 structure of the 16 PAHs on the EPA priority pollutant list

Extraction steps were done as the following: Liquid-liquid extraction was performed using 1 L separatory funnel, extract concentration was done by rotary evaporator model Rotavapor R-210 (BÜCHI Labortechnik AG, Flawil 1, Switzerland).

pH measurements were done using a calibrated pocket pH meter model HI-98107 pHep produced by Hanna instruments (Mauritius).

Electrocoagulation experiments were carried out in a batch electrochemical glass beaker with a total volume of 250 mL; a digital hot plate magnetic stirrer model AREC (VELP Scientifica srl, Milano,

Italy) was used to provide sufficient mixing inside the reactor and control its temperature. In each experimental run, a total amount of 200 mL of the spiked water was treated in the reactor; the aluminum electrodes were dipped into the sample and connected to an ammeter, and a DC power source was used to provide the needed current. The current density was maintained constant by using a voltage regulator, and the aluminum electrodes were made of rectangular sheets with dimensions of 30 x 60 mm. A schematic diagram of the electrochemical reactor is shown in Fig. 2.

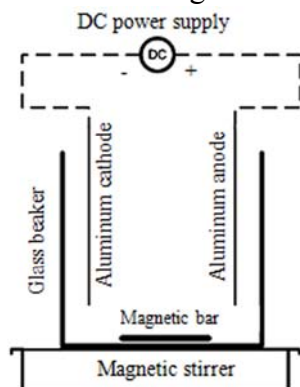


Fig. 2 Schematic diagram of the electrochemical apparatus

2.3. Synthetic spiked water samples

Water samples were made by spiking water of HPLC grade (200 mL) with the necessary volumes of PAHs standard certified solution (EPA 610 PAH Mix) to make three different initial concentrations of total PAHs ($100 \mu\text{g L}^{-1}$, $10 \mu\text{g L}^{-1}$, and $5 \mu\text{g L}^{-1}$). These concentrations were chosen according to the data of oil and gas produced water derived from several international reports, which mentioned that PAH concentrations in produced water are ranged from 0.8 to $22.4 \mu\text{g L}^{-1}$ and could be down below $1 \mu\text{g L}^{-1}$ in surface and ground water [21].

2.4. Electrocoagulation experiments

The spiked water samples were treated in the electrochemical reactor which is described in section 2.2 with stirring at constant speed on a rotating velocity of 150 rpm and at constant temperature of $25 \text{ }^\circ\text{C}$ and by adding the proper electrolyte. The total effective area of aluminum electrode was 33.0 cm^2 (only part of the total area of electrode was dipped in solution) and the distance between both electrodes was 50 mm. After EC process, flocculation was enhanced under low stirring speed with a rotating velocity of 30 rpm for 10 min.

The water after EC treatment was placed in sedimentation beaker. The total PAH removal efficiencies were calculated after 30 min settling time based on the sum of areas of the 16 peaks as follows:

$$\% \text{ Removal} = (C_i - C_f)/C_i \times 100 \quad (6)$$

where C_i is the initial total PAHs concentration ($\mu\text{g L}^{-1}$) and C_f is the final PAHs concentration ($\mu\text{g L}^{-1}$).

Before each run, organic impurities on electrode surfaces were removed by washing with acetone and HCl solution (10 wt%) respectively and then washed with distilled water prior to use.

2.5. Liquid-liquid extraction method

The following procedure was based on the extraction steps mentioned in the US-EPA method 550.1 [19]. The total volume of treated water was placed in separatory funnel and 25 mL of methylene chloride were added, then, the sample was extracted by shaking the funnel for two minutes with periodic venting to release excess pressure. After permitting the organic layer to separate from the water phase for 10 minutes, the methylene chloride extract was collected in an Erlenmeyer flask. The extraction step should be repeated twice with combining the extracts in the same Erlenmeyer flask.

The concentration step was done by evaporating the methylene chloride to reach a volume of about 0.5 mL using the rotary evaporator with a water bath temperature of 50 °C and a rotating velocity of 60 rpm. The solvent was changed by adding 1 mL of acetonitrile and the remaining volume of methylene chloride (the upper phase) was evaporated by blowing a stream of nitrogen over it to reach a final volume of 1 mL.

2.6. Analysis Procedure

An aliquot of 20 μL of the extract was injected into the HPLC system using a flow rate of 1.0 mL min⁻¹, and a gradient elution of water and acetonitrile was used as mobile phases. The separation was accomplished using the mentioned column at 30 °C for 60 min. Table 1 shows the program of gradient elution which has been used. The detection was carried out using diode array and fluorescence detectors.

Table 1. Time table of the gradient elution used in the HPLC separation

Time (min)	Solvent A % (Water)	Solvent B % (Acetonitrile)	Flow rate (mL min ⁻¹)
0.00	50.00	50.00	1.0
5.00	50.00	50.00	1.0
30.00	1.00	99.00	1.0
52.00	1.00	99.00	1.0
55.00	50.00	50.00	1.0
60.00	50.00	50.00	1.0

Table 2 shows the parameters of each detector to detect the target analytes.

Table 2. HPLC detection parameters used in diode array and fluorescence detectors

Diode array detector			Fluorescence detector			
Signal	Wavelength (nm)	Bandwidth (nm)	Time (min)	Excitation (nm)	Emission (nm)	Gain
A	245	8	0.00	256	390	1
			22.50	275	420	4
			32.00	270	385	1
			38.00	290	430	1
			Spectrum scan(nm)	55.00	305	480
From 190 to 450			60.00	256	390	1

Fig. 3 displays the chromatogram of the spiked HPLC water sample acquired using the photo diode array detector while Fig. 4 refers to the chromatogram acquired by the fluorescence detector. The diode array detector is recommended for the determination of naphthalene, acenaphthylene, and acenaphthene, and the fluorescence detector is recommended for the remaining PAHs [19,20]. The evaluation of total PAHs concentration was estimated by calculating the sum of areas of the 16 peaks.

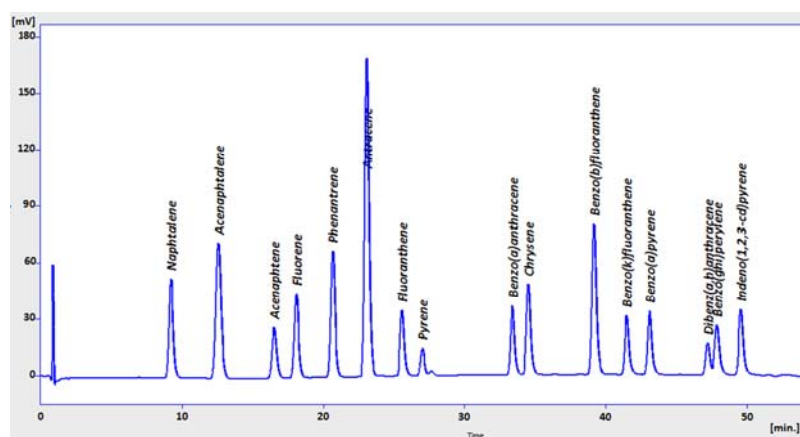


Fig. 3 Chromatogram of 16 PAHs resulted from diode array detector

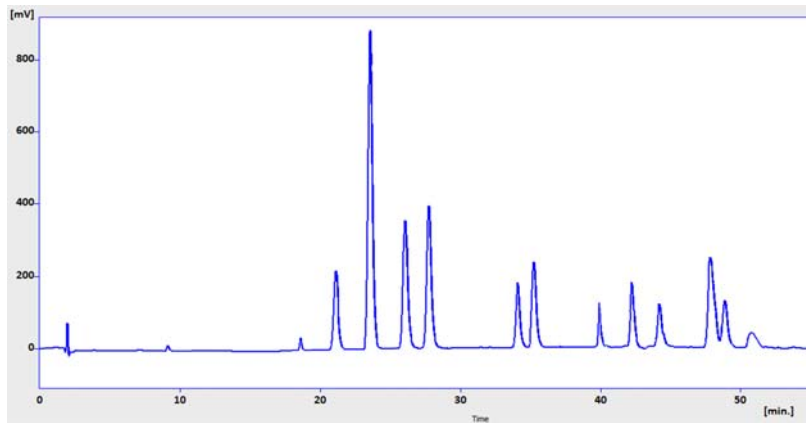


Fig. 4 Chromatogram of 16 PAHs resulted from fluorescence detector

3. Results and Discussion

3.1. Influence of initial pH

The effect of the initial pH on the removal of PAHs ($C_0 = 10 \mu\text{g L}^{-1}$) from water was evaluated at a current density of $20 \text{ mA}\cdot\text{cm}^{-2}$, NaCl 1 g L^{-1} and temperature of $25 \text{ }^\circ\text{C}$ for 100 min, by varying the initial pH from 5 to 10 using HCl or NaOH (0.1 M). The results presented in Fig. 5 show that the optimal pH for the reduction of these contaminants was found to be about 8, at which higher PAHs removal efficiency could be reached since the solid aluminum hydroxide $\text{Al}(\text{OH})_3$, which formed during the process, contributes to the removal of PAHs through adsorption and co-precipitation of these organic compounds, and considering that the solubility of aluminum hydroxide has been reported to be minimal at the pH range of 6–8 [4,11].

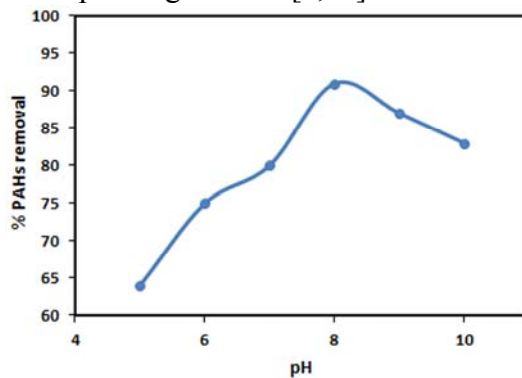


Fig. 5 The effect of pH on the PAHs removal efficiency (Current density: 20 mA cm^{-2} , $C_0 = 10 \mu\text{g L}^{-1}$, NaCl: 1 g L^{-1} , temp: $25 \text{ }^\circ\text{C}$ and time: 100 min)

The decrease in the removal efficiency at both, low and high pH values could be explained by the amphoteric character of aluminum hydroxide which does not precipitate at very low pH [12-13], while high pH leads to the formation of soluble $\text{Al}(\text{OH})_4^-$ [12,14].

3.2. Effect of electrolysis time and current density

Operating time experiments were carried out at pH 8 and initial PAHs concentration of $10 \mu\text{g L}^{-1}$ in the presence of 1 g L^{-1} NaCl. Batch electrocoagulation experiments were conducted for 100 min using different current densities. Fig. 6 depicts the effect of electrolysis time and the current density on electrocoagulation. As shown, the removal efficiency increases with increasing time at all the current densities which have been used as a result of the augmentation in aluminum ions generated in solution during electrolysis time. After 100 min the maximum removal efficiency of PAHs were about 72, 91, 96 and 98% for current densities of 10, 20, 25 and 30 $\text{mA}\cdot\text{cm}^{-2}$ respectively.

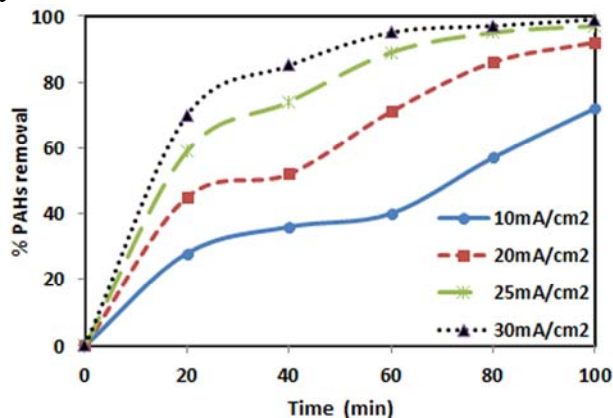


Fig. 6 The effect of electrolysis time and current density on PAHs removal (C_0 : $10 \mu\text{g L}^{-1}$, NaCl: 1 g L^{-1} , pH: 8, and temp: $25 \text{ }^\circ\text{C}$)

This is ascribed to the fact that at high current densities the dissolution of aluminum anode increases according to Faraday's law, enhancing the formation of aluminum hydroxides which produce more sludge with a consequent removal of PAHs due to sweep coagulation [12,15]. In addition, more bubbles will be generated at higher current density improving the degree of mixing of $\text{Al}(\text{OH})_3$ and PAHs which enhances floatation ability and increases in the PAHs removal efficiency [12,16]. As the treatment by electrocoagulation involves

passing of electrical current in the solution, this will affect the temperature of the solution being treated. The experiments showed that as the electrolysis time or the current density increases, the temperature of the solution increases [4,17].

The results in Fig. 6 show that we could reach more than 95% removal if we use a current density of 25 mA cm^{-2} , where the total PAHs concentration does not change significantly after 80 min of operation. This indicates that the optimal electrolysis time for the current density (25 mA cm^{-2}) is 80 min and that any operation beyond this time will be unjustified.

3.3. Influence of initial PAHs concentration

In order to evaluate the effect of water composition on the electrocoagulation performance, batch electrocoagulation experiments were carried out to treat three spiked water samples, which differed in the initial total PAHs concentration ($100 \mu\text{g L}^{-1}$, $10 \mu\text{g L}^{-1}$ and $5 \mu\text{g L}^{-1}$). Each sample contained NaCl: 1 g L^{-1} and treated at pH: 8 for 100 min using a current density of 25 mA cm^{-2} at $25 \text{ }^\circ\text{C}$. Fig. 7 represents the relation between the percentage removal of PAHs and initial solution concentration. The percentage removal decreased from about 99 to 34% as the PAHs concentration increased from 5 to $100 \mu\text{g L}^{-1}$. These observations could be explained by the fact that at a constant current density the same amount of aluminum ions passes to the solution at different PAH concentrations, hence, the formed amount of complex aluminum hydroxide was insufficient to coagulate the greater number of PAH molecules at higher PAH concentrations [12,18].

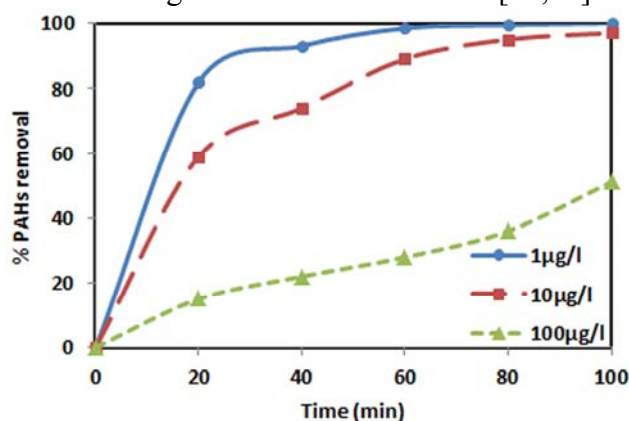


Fig. 7 The effect of initial PAHs concentration on the removal efficiency (Current density: 25 mA cm^{-2} , NaCl: 1 g L^{-1} , and temp: $25 \text{ }^\circ\text{C}$)

3.4. Effect of electrolyte type

The effect of electrolyte type on the removal of PAHs in water was evaluated by experiments on three types of electrolytes (NaCl, NaNO₃, Na₂SO₄) at some electrolyte concentration of 1 g L⁻¹, temperature of 25 °C, current density of 25 mA cm⁻², initial concentration of 10 µg L⁻¹ of PAHs, and electrolysis time of 100 min. The percentage removal of PAHs was strongly affected by the electrolyte composition as shown in Fig. 8. All the subjected PAHs were well removed. In inert electrolyte solutions of Na₂SO₄ and NaNO₃, PAHs pollutants were removed by electrocoagulation [1]. The removal efficiency was significantly increased using NaCl, as an electrolyte where indirect oxidation by hypochlorite, formed by the electrolysis of chloride, is involved, showing a clear positive effect [1]. The following reactions explain the formation of hypochlorite [1].

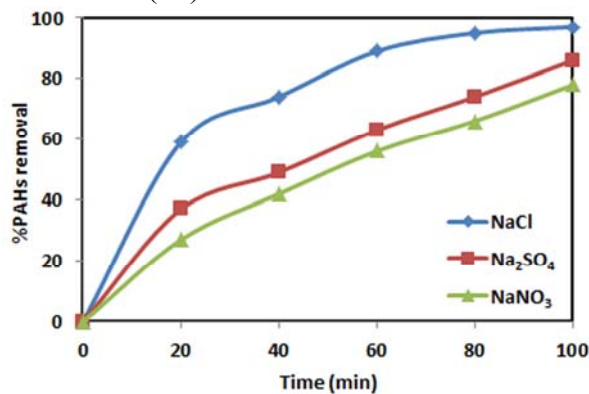
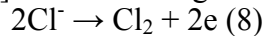


Fig. 8 The effect of electrolyte type on the removal efficiency (C_0 : 10 µg L⁻¹, Current density: 25 mA cm⁻², electrolyte concentration: 1 g L⁻¹, and temp: 25 °C)

We could conclude that when using NaCl as an electrolyte, the treatment of PAHs was due to both, electrocoagulation based on the formation of Al(OH)₃, and electrochemical degradation as a result of the indirect oxidation by hypochlorite.

3.5. Influence of electrolyte concentration

Several values of NaCl concentration in the range from 0.5 to 2 g L⁻¹ were used to increase the ionic conductivity of the solution. The augmentation of chloride ions removes the passive oxide layer form

on electrode surface; hence it increases the availability of aluminum hydroxide in the solution [12,16]. Furthermore, the indirect oxidation by hypochlorite also has to be increased. The above mentioned processes improve the efficiency of PAHs removal (Fig. 9).

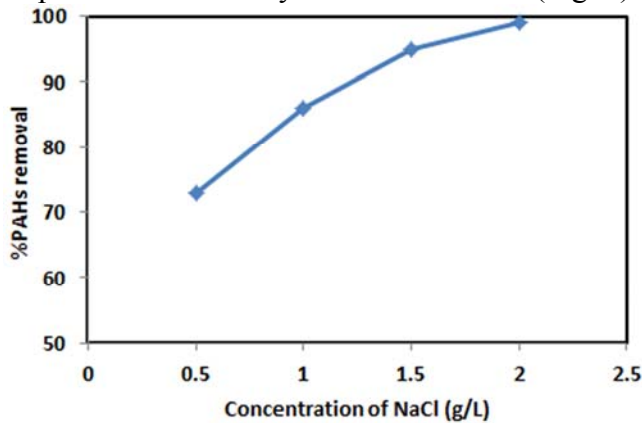


Fig. 9 The effect of electrolyte concentration on the removal efficiency (C_0 : $10 \mu\text{g L}^{-1}$, Current density: 25 mA cm^{-2} , electrolysis time: 60 min, and temp: $25 \text{ }^\circ\text{C}$)

In addition, increasing of chloride ions will minimize energy consumption, which decreased from 0.71 to 0.28 kWh g^{-1} total PAHs removed with increasing NaCl concentration from 0.5 to 2 g L^{-1} as shown in Fig. 10. This indicates the importance of the presence of NaCl in solution on the economy of electrocoagulation process.

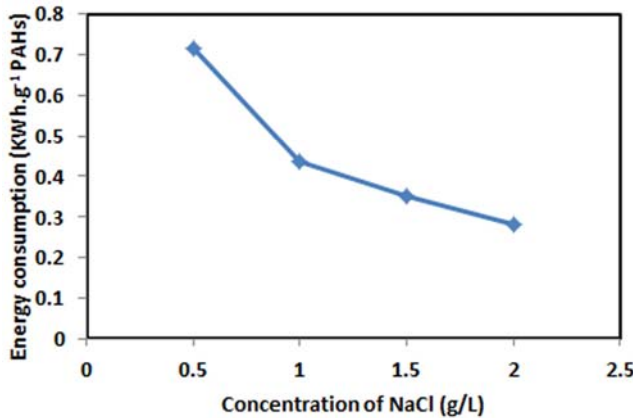


Fig. 10 The effect of electrolyte concentration on the energy consumption (C_0 : $10 \mu\text{g L}^{-1}$, Current density: 25 mA cm^{-2} , electrolysis time: 100 min, and temp: $25 \text{ }^\circ\text{C}$)

3.6. Treatment of contaminated water samples:

A sampling point was chosen in Deir Azzor area (North East Syria), which was from a surface water next to Al Jourzi petroleum well (34° 55' 26" N, 40°35' 57" E). Water samples were collected in 1000 ml glass containers which were completely filled to the top and stoppered with no headspace. Drops of HCl (1:1) were added to each sample at the time of collection to adjust the pH to below 2 to inhibit the activity of microorganisms. All samples were maintained at 4 °C, treated and analyzed in triplicates within 72 hours of sampling according to sections 2.5 and 2.6. Table 3 resumes the results obtained from the analysis of the environmental collected samples before and after treatment using optimum conditions (1.5 g L⁻¹ of NaCl, initial pH of 8, and current density of 25 mA cm⁻²). As shown in this table, electrocoagulation treatment reduces the concentration of pollutants to undetectable levels, but it can be seen that the efficiency of this treatment during the first 60 minutes was lower than that of the synthetic studied samples because it could contain other pollutants which can contribute to this treatment.

Table 3 The results of the treatment of environmental contaminated water samples

Time (min)	Before treatment	20	40	60	80	100
Total PAHs concentration (µg L ⁻¹)	7.05±0.11	3.07±0.08	2.29±0.05	1.31±0.03	0.42±0.02	Not detected

4. Conclusion

Electrocoagulation of PAHs using aluminum as a sacrificial anode seems to be a very efficient method for the treatment of water contaminated with traces of PAH pollutants as revealed by the present study. The experimental results showed that the treatment is based on coagulation and adsorption; furthermore, it is improved by indirect oxidation by hypochlorite. Using the optimum experimental conditions (1.5 g L⁻¹ of NaCl, initial pH of 8, and current density of 25 mA cm⁻² for 80 min) could help to remove up to 98.85 % of the total PAHs. It can be concluded from this study that electrocoagulation is a promising technique for the removal of PAHs from such contaminated water that contain concentrations up to 30 µg L⁻¹ of PAHs.

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