

*Full Paper*

## **Cadmium Removal from Phosphoric Acid by a Process Combining Ion Exchange on Resin and Electrodialysis**

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**Abstract-** The cadmium removal from phosphoric acid using a hybrid process combining ion exchange resin and electrodialysis called electropemutation was investigated. Continuous electropemutation has been shown to be more effective than conventional electrodialysis with a cadmium removal rate of 70.70% versus 13.70%. The study of the influence of some operating parameters on continuous electropemutation has revealed that the cadmium removal efficiency increases with the applied current density, and the flow rate of the feed solution. However, the efficiency decreases as the phosphoric acid and cadmium concentration increases. The cation exchange resin and the electroregeneration electrolyte nature also have an effect on the continuous electropemutation efficiency. The Duolite C20 resin and the HNO<sub>3</sub> electroregeneration electrolyte were found to be more effective in removing cadmium from phosphoric acid by continuous electropemutation.

**Keywords-** Phosphoric acid, Wet process, Cadmium, Electrodialysis, Continuous electropemutation

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### **1. INTRODUCTION**

Phosphoric acid is the most produced mineral acid after sulfuric acid. About 90% of the phosphoric acid is produced by the wet process of phosphoric acid (WPA) which is based on

the attack of phosphate ore by sulfuric acid [1]. WPA is characterized by its low cost and the presence of high content of cationic impurities (Cd, Fe, Mg, Al and Ca) which affect its properties and limit its use [2-5] is used in synthesis of phosphate fertilizers, and in development of pure products for the food and detergent industries. Therefore, cadmium present in industrial phosphoric acid can be found in these products, which induces soil pollution, water resources and its presence in the food chain. The presence of cadmium in water, even at very low concentrations, is extremely harmful to the aquatic environment and the human health [6,7]. Further cadmium as a contaminating element, which affects a wide variety of living organisms and accumulated in the human body. Cadmium is classified as a human carcinogen (Group 1) by the International Agency for Research on Cancer [8]. Cadmium is an unwanted and extremely toxic element so its removal from WPA is essential. Several WPA purification methods have been tested in many works. They include precipitation [9], ion-exchange [10], liquid-liquid extraction [11], adsorption on activated carbon [12] or on modified bentonite [13] and membrane separation processes such as liquid membrane process [14], electrodialysis (ED) [15], electro-ED [16], electrodeionization (EDI) [17], reverse osmosis and nanofiltration [18].

The use of these techniques was limited due to a number of drawbacks such as limited efficacy, high costs of organic solvents and resins (more often not regenerable), difficulty in recovering all the solvents from both the raffinate and the purified acid, environmental pollution by some by-products. Membrane separation technologies are well-known methods for metal removal or recovery [19]. ED is one of the methods dedicated to toxic metal removal from wastewater [20] and surface treatment effluents [21-26]. ED is a process that can be easily scaled and combined with other processes. Its combination with ion exchange (Continuous electropermutation, CEP) has been successfully applied to remove heavy metals from wastewater [27-30]. For example, in our recent work on cadmium removal [30], the pH of the solutions studied was between 5.9 and 6.0 and the operating pH range of the ion exchange resin used (Purolite C 100) is 6-14. These conditions do not cause difficulties for cadmium removal. However, when it comes to strongly acidic medium ( $\text{pH} < 1$ ), which we do, the challenge is greater because the high acidity makes some ion exchange resins unusable due to inadequate their operating pH range for such application. In addition, the high acidity provides a very high concentration of protons which are recognized as highly competitive species with metal cations in the ion exchange process. In the literature, we did not find any work focused on the removal of heavy metals from strongly acidic media by CEP.

The objective of this work is to study the possibility of effectively removing cadmium from phosphoric acid at 28% by CEP. In order to overcome the difficulties related to the high acidity of the phosphoric acid solution, the appropriate resin and optimum operating conditions must be sought.

## 2. EXPERIMENTAL

### 2.1. Materials and methods

#### 2.1.1. Solutions

The phosphoric acid solution used in this study was prepared from commercial phosphoric acid provided by Biochem Chemopharma (85% mass and 1.71 density). Its composition (28% P<sub>2</sub>O<sub>5</sub> and 20 mg L<sup>-1</sup> Cd) was chosen according to the composition of the phosphoric acid produced by WPA from phosphate ore from Jebel Onk (Algeria). The salt of cadmium nitrate tetrahydrate (Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, 99%), NH<sub>4</sub>Cl (99.5%), NaOH (98%), H<sub>2</sub>SO<sub>4</sub> (98%) and HNO<sub>3</sub> (69%) were purchased from Sigma-Aldrich. All chemicals were of analytical grade purity.

#### 2.1.2. Ion exchanger materials

The main characteristics of the ion exchange materials (IEM) used in this work are shown in Table 1. This is CMX and Nafion 117: cation exchange membranes (CEM); ADS: anion exchange membrane (AEM); Duolite C20 and Amberlite IR120: cation exchange resins (CER). Both Duolite C20 and Amberlite IR120 resins have been chosen especially at their very wide operating pH ranges 0-14 and 1-14 respectively, so they can be used in a strongly acidic medium.

**Table 1.** Characteristics of IEM

<b>Membrane</b>	<b>Manifactory</b>	<b>Functional groups</b>	<b>EC (meq.g<sup>-1</sup>)</b>	<b>WC (%)</b>	<b>R<sub>ms</sub> (Ω.cm<sup>2</sup>)</b>	<b>Thickness (μm)</b>
CMX	Asahi glass	-SO <sub>3</sub> <sup>-</sup>	1.5 - 1.8	25 - 30	2.5-3.5	170-190
ADS	Asahi glass	-NR <sub>3</sub> <sup>+</sup>	1.27	32	1,2	153
Nafion 117	DuPont	-SO <sub>3</sub> <sup>-</sup>	0.90	22	1.1	220
<b>Resin</b>	<b>Manifactory</b>	<b>Functional groups</b>	<b>EC (meq.g<sup>-1</sup>)</b>	<b>WC (%)</b>	<b>Ionic form</b>	<b>Particle size (mm)</b>
Duolite C20	Rhom & Haas	-SO <sub>3</sub> <sup>-</sup>	2.05	43 - 46	Na <sup>+</sup>	0.3 - 1.2
Amberlite IR120	Rhom & Haas	-SO <sub>3</sub> <sup>-</sup>	2.0	45 - 50	Na <sup>+</sup>	0.6-0.8

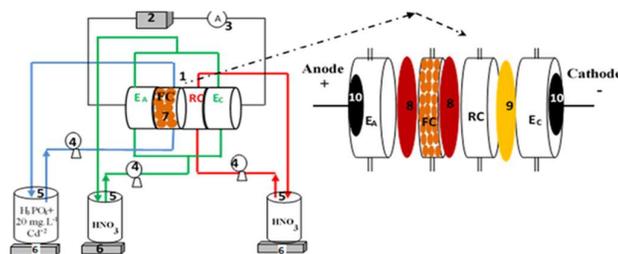
#### 2.1.3. Ion exchange material conditioning

When a CER is in the form of Na<sup>+</sup> and percolated with a solution containing Cd<sup>2+</sup> cations, the exchange between the Na<sup>+</sup> and Cd<sup>2+</sup> cations takes place until equilibrium is reached. In order to avoid a transitional step when studying the removal of Cd<sup>2+</sup> cations by CEP, the resin

was initially equilibrated with a 28%  $\text{H}_3\text{PO}_4$  solution containing cadmium at  $20 \text{ mg L}^{-1}$  for 24 h. Then, it is carefully rinsed with deionized water.

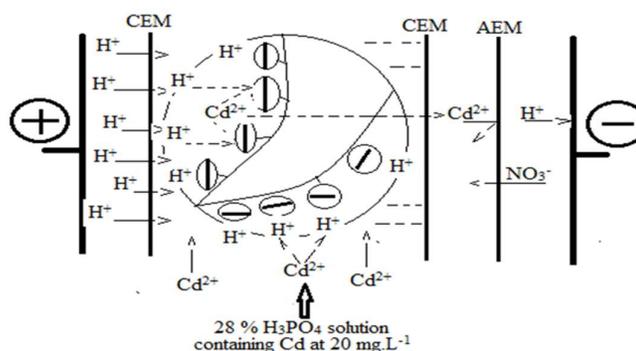
#### 2.1.4. Experimental set-up and principle of cadmium removal by EP process

The laboratory pilot (Figure 1) included a cylindrical ED cell composed of four compartments. These compartments are separated by two CEM and an AEM. Each separating membrane had an effective area of  $28.26 \text{ cm}^2$ . The feed compartment (FC) was filled with 20 g of CER clamped between two CEM. The thickness of this compartment is 2 cm. The two extreme compartments of the cell are the electrode rinsing compartments (anode ( $\text{E}_\text{A}$ ) and cathode ( $\text{E}_\text{C}$ )). The RC and  $\text{E}_\text{C}$  are separated by an AEM. In our case, the  $\text{E}_\text{A}$  also acts as a proton donor compartment to electro-regenerate the IER. Using peristaltic pumps, all solutions circulate between the tanks and compartments closed circuit. A well-defined current density is imposed between the electrodes using a power supply. In the circuit of  $\text{E}_\text{a}$  circulates the electrolyte solution of electro-regeneration of the resin.



**Fig. 1.** Laboratory pilot of continuous electropermutation. 1. Cell with four compartments; 2. Power supply; 3. Ammeter; 4. Peristaltic pump; 5. Solution tanks; 6. Magnetic Stirrers; 7. Ion-exchange resin; 8. CEM; 9. AEM; 10. Platinized titanium electrode

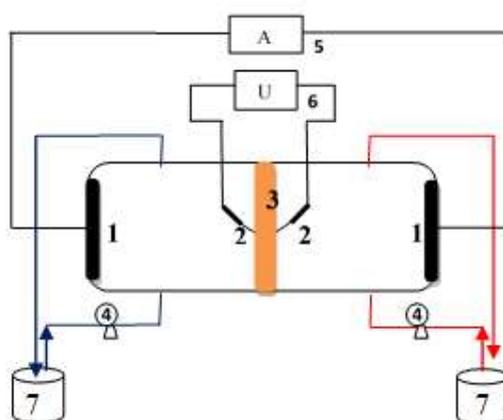
The principle of CEP has been detailed in some works [28-31] which we recall here briefly. Its description with respect to the solution studied composition is given in the Figure 2. The Figure 2 schematizes the ion transfer and ion exchange processes that take place during the CEP on a resin grain. In fact, during the percolation of the CER by the studied solution, the  $\text{Cd}^{2+}$  cations are first retained by the negatively charged functional sites of the REC. Then the protons of the  $\text{E}_\text{a}$  compartment cross the CEM and exchange with  $\text{Cd}^{2+}$  cations. The  $\text{Cd}^{2+}$  cations displaced from the functional sites, under the effect of the electric field, are transferred into the RC compartment.  $\text{Cd}^{2+}$  cations are prevented from passing to the EC compartment by the AEM and therefore they are confined in the RC compartment.



**Fig. 2.** Principle of electro-extraction of  $\text{Cd}^{2+}$  cations by CEP on a grain of CER

### 2.1.5. Determination of limiting current density

To reduce the duration of treatment by ED, it is recommended to work with high current density ( $i$ ). This cannot be increased indefinitely, since from a certain value of  $i$  the concentration polarization phenomenon occurs. Indeed, the transport of cations to the anode through a CEM leads to a concentration decrease of counter-ions in the laminar boundary layer at the membrane surface the dilute side and an increase at the concentrate side. When the cation concentration at the surface of the CEM dilute side will become zero, the current will approach to the maximum value in the process, which is defined as the limiting current (LC). When the LC in an electrodialysis desalination process is exceeded, the electrical resistance in the dilute solution increases considerably due to the depletion of ions in the laminar boundary layer at the membrane surface [32]. The depletion of ions on the surface of the membrane leads to an increase in the voltage drop. As a result, energy consumption increases significantly.



**Fig. 3.** Limit current measuring cell. 1. Platinum titanium electrodes; 2. Ag-AgCl electrode; 3. Cation- exchange membrane; 4. Peristaltic pump; 5. Power supply; 6. Voltmeter; 7. Solution tanks

To determine experimentally the value of the limiting current density of a phosphoric acid solution containing cadmium, we used the cell described in Figure 3. The evolution of the current intensity is measured; who crosses stacking according to the applied voltage. The plot of the curve Current-Voltage allows highlight the value of the current limit.

### 2.1.6. Analytical method and expression of results

During the EP experiments, solution samples from FC and RC compartments are taken at well-defined times for analysis. The concentration of cadmium was determined by atomic absorption spectrometry using a spectrometer SHIMADZU AA 6200.

The results of the cadmium removal from the phosphoric acid solution are expressed by the removal rate, which is calculated according to the following equation:

$$R = \frac{c_{FC}^i - c_{FC}^f}{c_{FC}^i} \times 100 \quad (1)$$

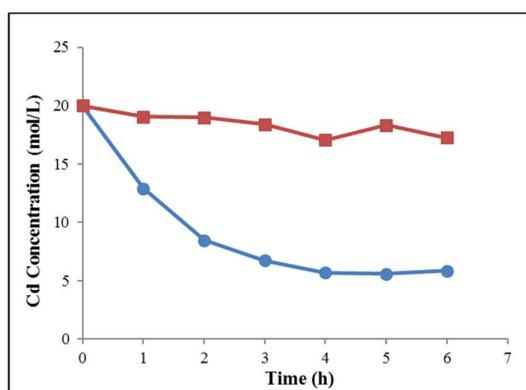
Where,

$c_{FC}^i$  and  $c_{FC}^f$  are initial and final concentrations (mol L<sup>-1</sup>) of cadmium in the feed compartment.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of the presence of resin on the ED

In order to show the performance of the combination of ED and ion exchange, we carried out experiments to remove cadmium from phosphoric acid by ED in the presence and absence of CER.



**Fig. 4.** Evolution of the cadmium concentration in the FC compartment during treatment with continuous EP and ED (in the presence of resin (●), in the absence of resin (■), CER: Duolite C20, FC solution: 28% H<sub>3</sub>PO<sub>4</sub>+20 mg L<sup>-1</sup> Cd, ERE: 0.1 M HNO<sub>3</sub> i: 10 mA cm<sup>-2</sup>, f: 4 mL min<sup>-1</sup>).

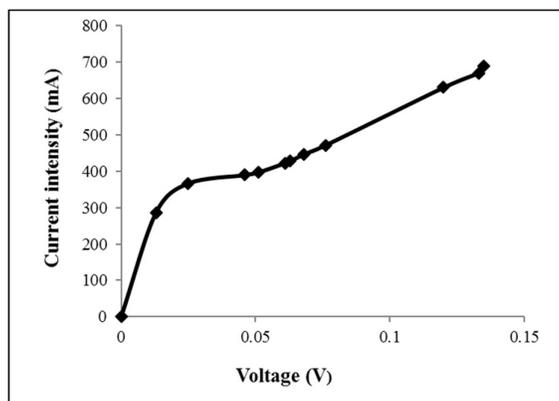
**Table 2.** Cadmium removal rates by ED in the presence and absence of CER (CER: Duolite C20, FC solution: 28% H<sub>3</sub>PO<sub>4</sub>+20 mg L<sup>-1</sup> Cd, ERE: 0.1 M HNO<sub>3</sub>, i: 10 mA cm<sup>-2</sup>, f: 4 mL min<sup>-1</sup>)

Process	ED in absence the CER	ED in presence the CER (CEP)
R (%)	13.70	70.70

The results obtained are compared in the Figure 4 and the Table 2. Figure 4 shows that the cadmium concentration in the studied solution decreases significantly during treatment with ED in the presence of CER. Indeed, it is observed that after three hours of treatment with ED in the presence of CER (CEP), the cadmium concentration decreased from 20 to 6.5 mg L<sup>-1</sup>. However, for the same duration of treatment with ED in the absence of CER, the cadmium concentration is reduced to only 18 mg L<sup>-1</sup>. After 6 h of treatment, the recorded removal rates are 70 and 13% (Table 2) for ED in the presence and absence of CER respectively. This result clearly shows that to remove cadmium from phosphoric acid, CEP is more effective than ED.

### 3.2. Effect of current density

With the help of the experimental setup (Figure 3) of the determination of the limit current, we have traced the variation of the current intensity as a function of the voltage on both sides of the membrane (Figure 5). The value of the limit current obtained is equal to 389.5 mA, which corresponds to a limit current density of 31.0 mA cm<sup>-2</sup>. The study of the current density effect will concern values lower than the limit current density.



**Fig. 5.** Current-potential curve of membrane in contact of a phosphoric acid solution

**Table 3.** Cadmium removal rate at different current densities (CER: Duolite C20, FC solution: 28% H<sub>3</sub>PO<sub>4</sub>+20 mg L<sup>-1</sup> Cd, ERE: 0.1 M HNO<sub>3</sub>, f: 1mL min<sup>-1</sup>, t: 6 h)

Current density (mA cm <sup>-2</sup> )	5	10	20	25
R (%)	23.65	46.05	48.54	46.63

To examine the effect of current density on cadmium removal efficiency from phosphoric acid solution by continuous EP, experiments were carried out at different current densities (5, 10, 20, 25 mA cm<sup>-2</sup>). The results obtained after six hours of treatment are presented in Table 3. The analysis of the results (Table 3) shows that when the current density is increased from 5 to 20 mA, the removal rate of cadmium increases from 23 to 48%. However, increasing the current density from 20 to 25 mA cm<sup>-2</sup> leads to a small regression of the removal rate from 48.54 to 46.63%. The low variation in the efficiency of cadmium removal with the current density in the range 10-20 mA cm<sup>-2</sup>, then its regression beyond 20 mA cm<sup>-2</sup> are probably due to proton competition with Cd<sup>2+</sup> cations. In fact, the increase in applied current density causes an increase in proton production in E<sub>a</sub> and proton transfer flux from E<sub>a</sub> to FC. This increasing transfer with current density intensifies proton competition with Cd<sup>2+</sup> cations on the occupancy of resin functional sites and on the transfer from the FC to the RC through the CEM. The increase in proton transfer in the RC is at the expense of that of Cd<sup>2+</sup> cations. The increase in the cadmium removal rate resulting from the doubling of the 10 mA cm<sup>-2</sup> value is much less than that obtained with the doubling of the 5 mA cm<sup>-2</sup> value. Thus, we consider that 10 mA cm<sup>-2</sup> is an optimal current density.

### 3.3. Influence of flow rate

Flow rate is another parameter that could influence the cadmium removal efficiency. For this reason, we performed CEP experiments at different flow rates of the feed solution. From Table 4, it is observed that the increase in the flow rate favors the cadmium removal efficiency.

**Table 4.** Effect of flow rate on cadmium removal efficiency (CER: Duolite C20, FC solution: 28% H<sub>3</sub>PO<sub>4</sub>+20 mg L<sup>-1</sup> Cd, ERE: 0.1 M HNO<sub>3</sub>, i:10 mA cm<sup>-2</sup>; t:6 h)

Flow rate (mL min <sup>-1</sup> )	1	4	8	16
R (%)	46.05	70.70	71.05	75.47

These results also allow us to notice that the increase in efficiency resulting from the increase in the flow rate of 1 to 4 mL min<sup>-1</sup> is relatively greater than that resulting from the

increase in the flow rate from 4 to 16 mL min<sup>-1</sup>. Based on the economic criterion we retained, for the rest of the study, a value of 4 mL min<sup>-1</sup> as optimal flow rate.

### 3.4. Effect of resin nature

In order to study the effect of the nature of the resin on the cadmium removal efficiency, CEP experiments were performed with different resins. Duolite C20 gave a slightly higher rate of cadmium removal than that of Amberlite IR120 (Table 5). This difference is probably due to the difference in the properties of the two resins. The size of the particles could be a factor responsible for this result. Indeed, the two resins Duolite C20 and Amberlite IR120 have different particle sizes, 0.3 to 1.2 and 0.6-0.8 mm (Table 1) respectively. In general, the smaller the resin grain, the greater the surface area will be.

**Table 5.** Effect of the nature of IER on the removal rate (FC solution: 28% H<sub>3</sub>PO<sub>4</sub>+20 mg L<sup>-1</sup> Cd, ERE: 0.1 M HNO<sub>3</sub>, i: 10 mA cm<sup>-2</sup>, f: 4 mL mn<sup>-1</sup>, t: 6 h)

Resin	Duolite C20	Amberlite IR120
R (%)	70.70	67.29

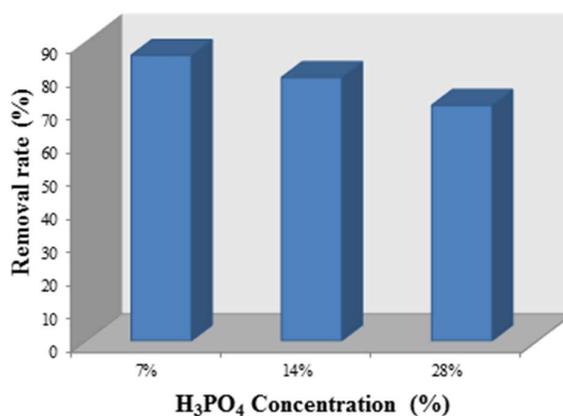
Another factor that could influence the ability of the resin to remove cadmium is the degree of crosslinking (DC). Jackson and Pilkington [33], in their work on "Effect of the Degree of Crosslinking on the Selectivity of Ion-exchange Resins" have shown that increasing the DC improves the selectivity of the resin for the divalent cation with respect to H<sup>+</sup>. In our case, the DC of the two resins are unavailable. However, it is known that for the same structure of the polymer the water content (WC) is directly related to the DC. The higher the DC, the lower the WC will be. Both of our resins have the same polymer structure: polystyrene, having sulfonic functional groups, crosslinked with divinylbenzene. Therefore, the value of the WC directly reflects the DC. The values of the WC (Table 1), 43-46% (Duolite C20) and 45-50% (Amberlite IR120) indicate that the DC of Duolite C20 is higher than that of Amberlite IR120. Consequently, from the values of WC it can be said that the Duolite C20 resin has a selectivity for Cd<sup>2+</sup> over H<sup>+</sup> higher than that of the Amberlite IR120.

### 3.5. Effect of phosphoric acid concentration

The performances of CEP under various concentrations of phosphoric acid were analyzed in terms of cadmium removal rate in the FC. The histogram presented in Figure 6 shows clearly that the removal rate decreases with the increase in the concentration of the phosphoric acid. This result can be explained by the competition between the cadmium ions and the protons to be fixed on the resin and then to cross the membrane. Increasing the phosphoric acid

concentration increases the concentration of protons, which intensifies proton competition with cadmium cations.

Figure 6 shows that the cadmium removal rate decreases with increasing phosphoric acid concentration. It is noted that the dilution of phosphoric acid from 28% to 7% makes it possible to improve the cadmium removal rate from 70 to 90%. Since the phosphoric acid concentration produced by the wet process is approximately 28%, the choice of the option to dilute the acid to 7% to obtain a higher cadmium removal rate depends on the cost of the operation of reconcentration of the acid from 7 to 28%.



**Fig. 6.** Effect of acid phosphoric concentration on cadmium removal rate (CER: Duolite C20, [Cd]: 20 mg L<sup>-1</sup>, ERE: 0.1 M HNO<sub>3</sub>, i: 10 mA cm<sup>2</sup>, f: 4 mL min<sup>-1</sup>, t: 6 h)

### 3.6. Effect of the cadmium concentration

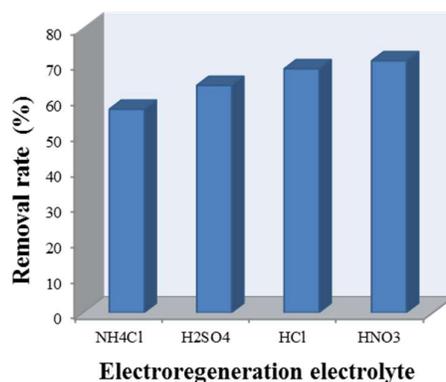
To study the effect of the cadmium concentration in the phosphoric acid solution on the cadmium removal efficiency by CEP we tested three concentrations: 20, 75 and 100 mg L<sup>-1</sup>. The results obtained (Table 6) show a decrease in the elimination rate with the increase in the increase of the cadmium concentration. It should be noted that the decrease in the removal rate is not significant, which means that CEP remains effective even at relatively high concentrations.

**Table 6.** Effect of cadmium initial concentration on cadmium removal rate (CER: Duolite C20, [H<sub>3</sub>PO<sub>4</sub>]: 28%, ERE: 0.1 M HNO<sub>3</sub>, i: 10 mA cm<sup>-2</sup>, f: 4 mL min<sup>-1</sup>, t: 6 h)

[Cd] (mg L <sup>-1</sup> )	20	75	100
R (%)	70.70	69.10	67.29

### 3.7. Effect of the electroregeneration electrolyte nature

The purpose of this study is to see if the nature of the electroregeneration electrolyte (ERE) affects the removal efficiency of cadmium by CEP. The solution of ERE flowing in the compartment  $E_a$  provides the cation, which passes through the CEM and then performs the electroregeneration of the CER. We studied four different electrolytes (HCl,  $H_2SO_4$ ,  $HNO_3$  and  $NH_4Cl$ ). The presented results in histogram form (Figure 7) show that all three acid electrolytes (HCl,  $H_2SO_4$  and  $HNO_3$ ) are more effective than the  $NH_4Cl$  salt.



**Fig. 7.** Effect of ERE nature on cadmium removal rate (CER: Duolite C20, FC solution: 28%  $H_3PO_4$ +20 mg  $L^{-1}$  Cd,  $i$ : 10 mA  $cm^{-2}$ ,  $f$ : 4 mL  $min^{-1}$ ,  $t$ : 6 h)

This means that the  $H^+$  proton is more effective than  $NH_4^+$ . We also note that the HCl and  $HNO_3$  monoacids, especially the  $HNO_3$  acid, are more effective than the  $H_2SO_4$  diacid.

## 4. CONCLUSION

In this study, the cadmium removal from phosphoric acid using a hybrid process combining ion exchange resin and ED was investigated. It has been shown that the combination of ion exchange with ED improves the efficiency of the latter from 13.70 to 70.70%. The study of the some parameters influence showed that the cadmium removal efficiency increases with the increase of the current density from 5 to 20 mA  $cm^{-2}$  and the flow rate from 1 to 16 mL  $min^{-1}$ . The nature of the CER and ERE also has an effect on the CEP effectiveness. However, the cadmium removal efficiency decreases with increasing concentration of the phosphoric acid solution. Increasing the cadmium concentration from 20 to 100 mg  $L^{-1}$  results in a decrease in the cadmium removal rate of only 70.70 to 67%.

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