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REMOVAL OF CADMIUM FROM PHOSPHORIC ACID BY THE LIQUID-LIQUID EXTRACTION PROCESS USING SYNTHETIC AGENT $C_{11}H_{18}N_2O$

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ABSTRACT

The removal of cadmium from phosphoric acid has been the subject of several studies while using different commercial and synthetic extracting agents. In this work we were interested in the liquid-liquid extraction of cadmium from the phosphoric acid solution by a synthesized extracting agent $C_{11}H_{18}N_2O$ *diluted in benzene. Subsequently, the influence of the following parameters was studied: the concentration of phosphoric acid, the concentration of the extracting agent, the stirring time, the organic/aqueous phase ratio and the equilibrium pH. The results obtained ensure that our synthesized product is promising. The cadmium extraction percentage of phosphoric acid reached 98.6% while using a concentration of 2.5M phosphoric acid, a concentration of* 0.5×10^{-2} *M of the synthesized extracting agent, a stirring time of 90min and with a phase ratio of 1/1.This extraction percentage is reduced when the pH value is increased.*

Key words: liquid-liquid extraction process, Phosphoric acid, Cadmium, synthesized extracting agent

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1. INTRODUCTION

In recent decades, there has been a growing awareness of the importance of ecological balances in natural environments. A balance threatened by pollution although the sources of soil and water pollution are numerous, a large part comes from phosphate products, which respond massively to soils. One such product is phosphoric acid. It is an important and necessary intermediate chemical compound in the fields of fertilizers, food, detergents, etc. Phosphoric acid can be synthesized in two ways, by a thermal and a wet process. In the thermal process, Phosphoric acid is produced first by reducing the phosphate rock, followed by oxidation and hydration[1]. But 70% of phosphoric acid production is done by the wet process, where phosphates are solubilized by mineral acids, which often results in a product contaminated by several heavy metals [2].

Cadmium is one of these harmful metals; its presence in the environment can mitigate negative impacts on the environment and affect human and aquatic life [3]. The Joint FAO/WHO Expert Committee on Food Additives (JECFA) of the Food and Agriculture Organization of the United Nations (FAO) has established a provisional tolerable monthly intake for cadmium of 25 µg/kg body weight [4] and the majority of environmental regulations require that cadmium must not exceed 20ppm of fertilizer[5]. Especially as the content of natural cadmium in sedimentary and igneous phosphate rocks varies from 3 to 51 mg/kg according to the different deposits in Morocco [2]. The elimination of cadmium is one of the major problems facing manufacturers of phosphoric acid and/or fertilizers.

Several methods for the purification of phosphoric acid from cadmium have been evaluated, including membrane technologies such as electrodialysis (ED), reverse osmosis and nanofiltration [6], by adsorption on activated carbon [7], and by liquid-liquid extraction [8]–[15].

The last-mentioned process has used several types of commercial extracting agents, notably for the extraction of cadmium from phosphoric acid, which we mention here: Almela et al. [10] whose used bis(2,4,4-trimethylpentyl) thiophosphinic acid (cyanex 302) diluted in kerosene; Elyahyaoui and Bouhlassa [15] verified that di-2-ethyl hexyl phosphoric acid (D2EHPA) remains the best cadmium extractant from phosphoric acid, comparing it to either tributylphosphate (TBP), trioctyl phosphine oxide (TOPO), triphenyl phosphine oxide (TPPO), or diphenylamine used individually or in a mixture; This same extractant (D2HEPA) was used diluted in kerosene by Mellah and Benachour [12]. In our previous work, we were interested in the synthesis of an oxygenated product ($C_{11}H_{18}N_2O$) that we used as an extracting agent[16]

This article deals the liquid-liquid extraction of cadmium from phosphoric acid using $C_{11}H_{18}N_2O$ as the extracting agent and benzene (C_6H_6) as the solvent, by taking into account different parameters: phosphoric acid concentration, concentration of extracting agent, pH, organic phase/aqueous phase ratio, stirring time, which act on the cadmium extraction yield of phosphoric acid.

2. MATERIALS & EXPERIMENTAL PROCEDURES

2.1. Reagents & Solutions

For all experiments, the aqueous phase consists of phosphoric acid solution of different concentration (VWR CHEMICALS, (85%)) which contains 10^{-3} mol/l of cadmium. The organic phase is prepared by dissolving three quantities of extracting agent $C_{11}H_{18}N_2O$: 0.5×10^{-2}M, 10^{-2} M, 1.5×10^{-2} M in benzene (C_6H_6) (Riedel-dehën). All reagents were of analytical quality and used without further purification. The pH of the solutions is adjusted to the desired value by adding a small amount of NaOH. The pH was measured by using a pH meter (model JENWAY 3520 pH Meter).

2.2. Extraction Procedure

The various extraction experiments were carried out in a batch system (comprising 5 ml of the aqueous phase and 5 ml of the organic phase), magnetically stirred at room temperature 20°C (model VWR incubating Mini Shaker). Each aqueous phase is tested for the three prepared organic phases. Each mixture undergoes vibrational stirring and a stirring time that varies from 30 min, 60 min to 90 min.

After stirring the aqueous and organic phases, this mixture underwent gravity settling and then separation of the two phases. Cadmium concentrations were determined in the aqueous phase by inductively coupled plasma spectrometry method (ICP-OES PerkinElmer Optima 8000). Cadmium concentrations in the organic phase were calculated from the difference between cadmium concentrations in the aqueous phase before and after extraction. The results are expressed as a percentage of the cadmium extraction .

3. RESULTS AND DISCUSSION

3.1. Effect of Extractant Concentration

The effect of the concentration of extracting agent has been studied for different concentrations of phosphoric acid (ranging from 1M to 5.5M), each concentration of which contains 10^{-3}M of cadmium. Each concentration of phosphoric acid is tested with the three organic phases (0.5×10^{-2} M, 10^{-2} M, 1.5×10^{-2} M).

According to the results found in our previous work [16], the highest percentages (98.58%) of cadmium extraction are obtained by the first organic phase (0.5×10^{-2} M) of extracting agent ($C_{11}H_{18}N_2O$) (figure1).

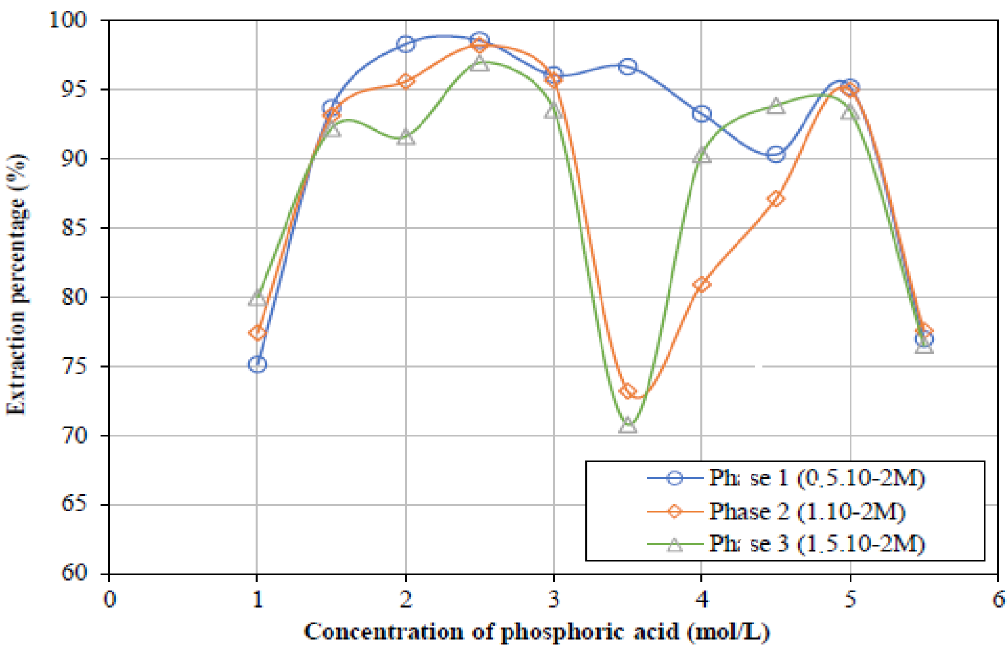

Figure 1 Influence of the concentration of extracting agent on the percentage extraction of cadmium from phosphoric acid (Operating condition: [cd^{+2}] = 10^{-3}M, T = 20 °C and stirring time = 90 min)

3.2. Effect of Various Parameters on Cadmium Extraction

The effect of phosphoric acid (H3PO4) concentration, stirring time, O/A phase ratio, and equilibrium pH on cadmium extraction was studied. The results are presented in Figures 2, 3, 4 and 5.

3.2.1. Effect of Phosphoric Acid Concentration

Using the optimum concentration of extractant (0.5×10^{-2} M), the effect of phosphoric acid concentration on cadmium extraction was tested. According to the shape of the curve obtained, the percentage extraction undergoes an increase in the range of phosphoric acid concentration from 1M to 2.5M (Fig.2), this increase went from 75.12% to 98.58%, thereafter the percentage of cadmium extraction decreased to 76.95% at the 5.5M phosphoric acid concentration.

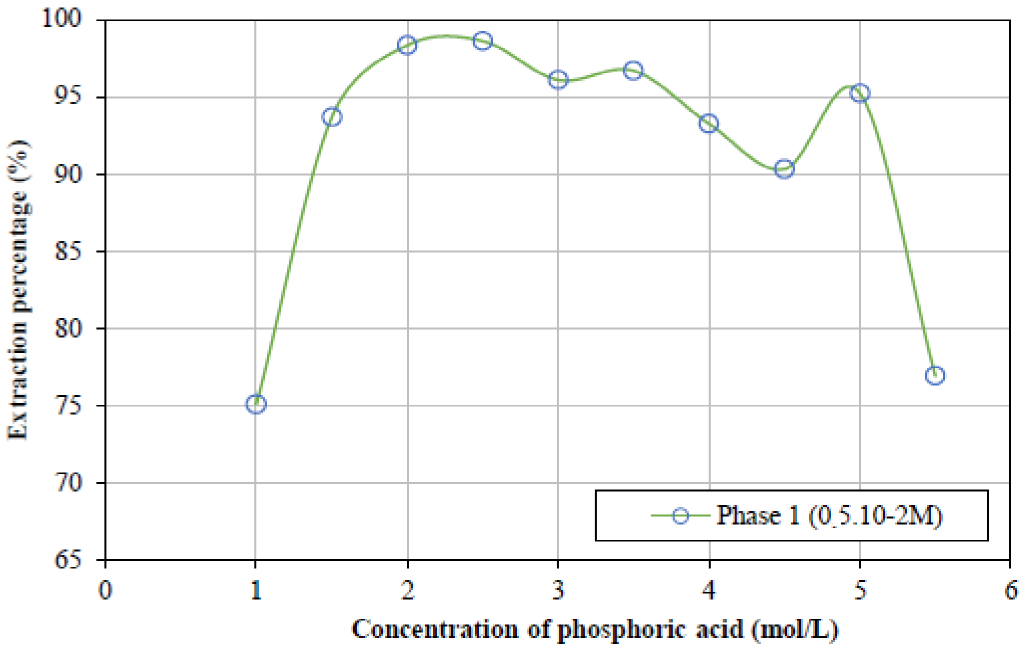

Figure 2 Influence of phosphoric acid concentration on extraction percentage (Operating condition: $C_{11}H_{18}N_2O$] = 0.5×10^{-2}M, T = 20 °C and stirring time = 90 min)

3.2.2. Effect of Stirring Time

In order to study the effect of stirring time, the optimal concentration of extractant (0.5×10^{-2} M), the concentration of phosphoric acid which allowed us to have a better extraction of 2.5M cadmium with a phase ratio of O/A=1 and an equilibrium pH = 1.8 ±0.2, was used. We varied the agitation time between 30 min, 60 min and 90 min.

According to the results (fig.3), it can be seen that the more the contact time between the two phases increases, the more the percentage of cadmium extraction is important, from 82.06% for 30min, to 94.62% for 60min, up to 98.58% for 90min.

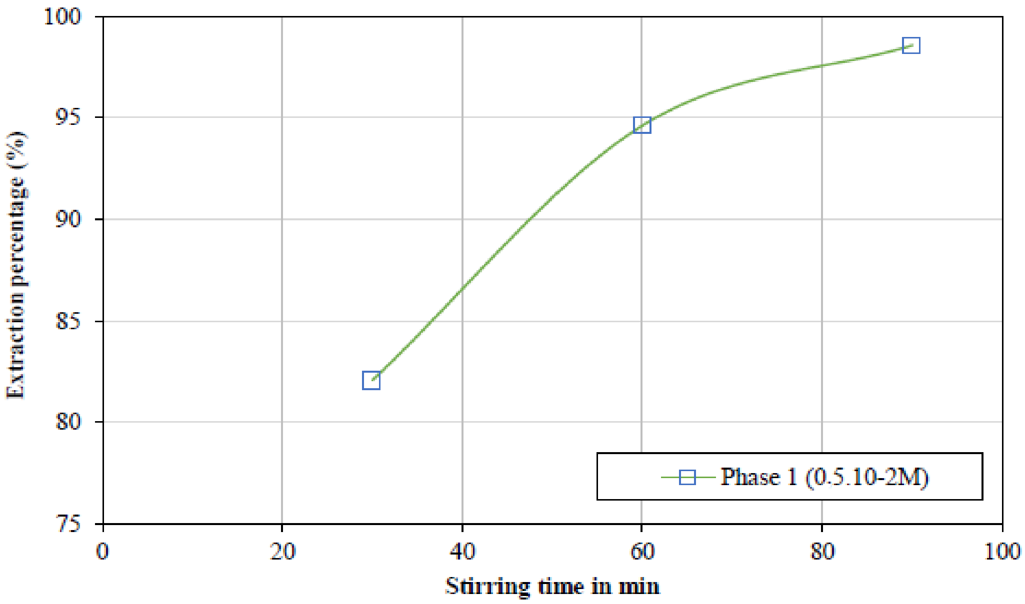

Figure 3 Influence of agitation time on the extraction percentage (Operating condition : $[H_3PO_4]$= 2.5M, equilibrium pH = 1.8 ±0.2, $[C_{11}H_{18}N_2O]$= 0.5×10^{-2}M, $[cd^{+2}]$ = 10^{-3}M and T = 20 °C)

3.2.3. Effect of the O/A Phase Ratio

In order to study the O/A phase ratio effect, these operating conditions were ensured: $[H_3PO_4]$= 2.5M, equilibrium pH = 1.8 ±0.2, $[C_{11}H_{18}N_2O]$= 0.5×10^{-2}M, $[cd^{+2}]$ = 10^{-3}M and T = 20 °C. The O/A ratio ranged from 1/5 to 5/1.

According to the results (fig.4), the percentage of camium extracted increases with the increase of the O/A phase ratio. The variation of the phase ratio from 1/5 to 1/1 results in an increase of the extraction percentage from 68.97% to 98.6% respectively. Beyond the 1/1 ratio, the phase ratio had no effect on the cadmim extraction percentage.

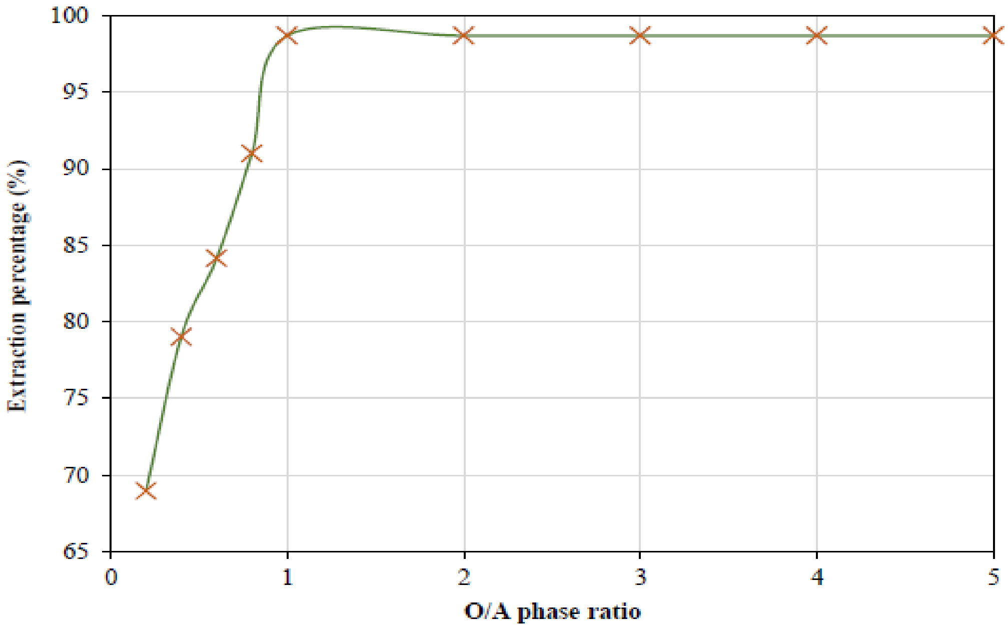

Figure 4 Influence of the O/A ration on cadmium extraction (Operating condition : $[H_3PO_4]$= 2.5M, equilibrium pH = 1.8 ±0.2, $[C_{11}H_{18}N_2O]$= 0.5×10^{-2}M, $[cd^{+2}]$ = 10^{-3} M and T = 20 °C)

3.2.4. Effect of pH Equilibrium

The extraction of cadmium from the phosphoric acid was carried out using, the extracting agent ($C_{11}H_{18}N_2O$) (0.5×10^{-2} M) and a phosphoric acid concentration of 2.5M.

The pH of the solution was adjusted using NaOH to allow us to work in a range from 1 to 5. According to the result (fig.5), there is a linear regression of the extraction curve justifying that the percentage of cadmium extraction is inversely proportional to the pH, it goes from 98.58% at pH=1 to 89.59% at pH=5.

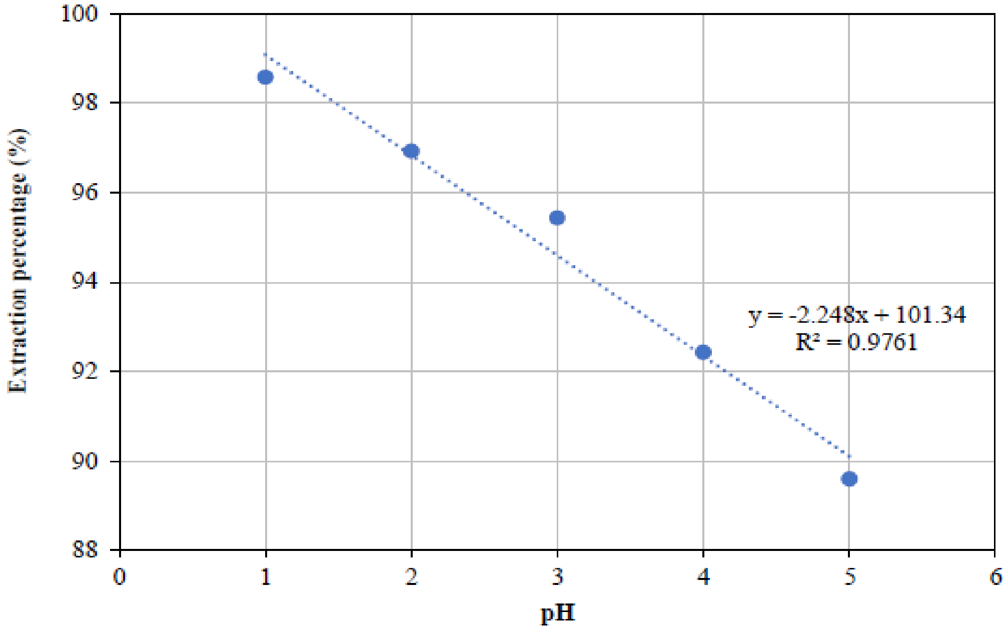

Figure 5 Influence of pH on cadmium extraction (Operating condition : $[H_3PO_4]$= 2.5M, equilibrium pH = 1.8 ±0.2, $[C_{11}H_{18}N_2O]$= 0.5×10^{-2}M, $[cd^{+2}]$ = 10^{-3} M and T = 20 °C)

4. CONCLUSIONS

The removal of cadmium from phosphoric acid was carried out by liquid-liquid extraction using $C_{11}H_{18}N_2O$ as an extracting agent diluted with C_6H_6 benzene. The results can be summarized as follows:

- The optimal concentration of the extracting agent $C_{11}H_{18}N_2O$ that allowed us to have the highest percentages of cadmium extraction was of the order of 0.5×10^{-2}M.
- The extraction percentage of cadmium depends on the concentration of phosphoric acid; over the concentration range of 1 to 2.5M, the extraction percentage has varied from 75.12% up to 98.58%.
- The more the contact between the aqueous and organic phase is promoted, the more the cadmium extraction percentage increases. The equilibrium of this extraction is established after 90min.
- The variation of the phase ratio from 1/5 to 5/1 results in an increase of the extraction percentage from 68.97% to 98.6%.
- The percentage extraction of cadmium is reduced by increasing the equilibrium pH of the solution.

A reaction mechanism will be predicted and the modelling and simulation of this extraction process will be the subject of future work.

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