Studii şi Cercetări Ştiinţifice
Chimie şi Inginerie Chimică, Biotehnologii, Industrie Alimentară
2022, **23** (4), pp. 333 – 343

Scientific Study & Research
Chemistry & Chemical Engineering, Biotechnology, Food Industry
ISSN 1582-540X

ORIGINAL RESEARCH PAPER

FACTORIAL DESIGN OPTIMIZATION OF URANIUM (VI) CLOUD POINT EXTRACTION WITH TRITON X-100/TWEEN-40/D2EHPA/ $BMIMMESO_4$

Nour Eddine Djebbari, Afaf Amara, Amel Didi, Mohamed A. Didi*

Tlemcen University, Faculty of Sciences, Department of Chemistry, Laboratory of Separation and Purification Technologies, Box 119, Algeria

*Corresponding author: madidi13@yahoo.fr

Received: July, 27, 2022
Accepted: December, 16, 2022

Abstract: This work reports the Uranyl (UO_2^{2+}) extraction from water by the Triton X-100 / Tween-40 / D2EHPA / $BMIMMeSO_4$ by two aqueous phases or cloud point extraction (CPE). The procedure has been developed to extract uranium (VI) using a mixture of non-ionic surfactants: Triton X-100 and Tween-40 in different contexts, and a mixture of lipophilic chelating extracting agent D2EHPA/ $BMIMMeSO_4$. Several parameters such as the concentrations of Triton X-100, Tween-40, and uranium (VI) metal ions, temperature, solution *p*H have been studied by the 3^3 factorial design method, in order to find the optimum conditions for uranium (VI) extraction. The optimal extraction of uranium (VI) by micelle-mediated extraction (CPE) procedure was obtained for *p*H= 3.0, Na_2SO_4 (% w/w) = 9.0 and $[UO_2^{2+}]$ = 5.50 mM. This system has research value and application in wastewater treatment.

Keywords: *cloud point extraction, design of experiments (DOE), D2EHPA, Triton X-100, Tween 40, Uranyl ion*

INTRODUCTION

Uranium, a weekly radioactive and toxic metal, is widespread in the environment, found at low levels in soils, waters and rocks. Its disposal in wastewater is of great importance. In the nuclear industry and mining, uranium and its compounds are potentially toxic [1 – 3]. This toxicity can be caused by breathing air containing uranium dust or by eating substances contaminated by uranium [4, 5]. The uranium separation from its associated components becomes necessary in view of its increased demand. Effective methods are increasingly being developed to separate uranium from the various flux encountered at the various stages of cycle of the nuclear fuel; although solvent extraction methods are the backbone of the nuclear reprocessing industry. The use of green and sustainable technology requires alternative processes with a lower organic solvent use.

To study the extraction of uranium (VI) using different techniques such as solvent extraction which uses large quantities of organic solvents, several investigations have been carried out [6, 7]: ion exchange [8], adsorption [9 – 11], electrochemical membrane separation [12], and other techniques [13, 14]. For technical and economic reasons, these processes do not respect environmental regulations.

Alternatively, cloud point extraction (CPE) was generally observed in non-ionic surfactant micellar solutions when the temperature of the surfactant solution is raised to a certain value [15 – 18]. As it is an aqueous two-phase extraction process, it does not require the presence of organic diluents. High values of concentration factors can be achieved compared to organic solvent extraction. Extraction technology using benign phases for the environment replaces the volatile organic solvents used in conventional solvent extraction technologies. CPE is based on the phase separation phenomenon, exhibited by micellar solutions of non-ionic surfactants [19].

Factorial design optimization has proven its usefulness to obtain empirical quadratic model relating process response to process factors [20].

The aim of this work is to study the effect by factorial designs of the operating parameters such as the uranyl ions concentration, Na_2SO_4 salt, and the *p*H, in order to find the optimum conditions for uranium (VI) extraction. The dependencies of the concentrations of Triton X-100 and Tween-40 on the cloud point behavior were also investigated.

MATERIALS AND METHODS

Reagents

Uranyl acetate dihydrate ($C_4H_6O_6U.2H_2O$, 424.15 g·mol^{-1}), NaCl 99 %, Na_2SO_4 99 %, CH_3COONa 99 %, $Na_2S_2O_3$ 99 %, KBr 99 %, KNO_3 99 %. were supplied from Merck, Germany. The nonionic surfactants used in these studies were p-octylpolyethylene glycol phenyl ether (Triton X-100, as shown in Figure 1) having an HLB value of 13.5 and a critical micelle concentration CMC equal to 3.0×10^{-4} M at 25 °C and, polyoxyethylene sorbitan monopalmitate (Tween-40, as shown in Figure 1) having an HLB value of 15.6 and a critical micelle concentration CMC equal to 2.7×10^{-2} M at 25 °C provided from Biochem Chemopharma and Fluka, respectively. Di-(2-ethylhexyl) phosphoric acid (D2EHPA, as shown in Figure 1) (322.43 g·mol^{-1}) from Fluka and 1-butyl-3-methylimidazolium-methyl sulfate (BMIMMeSO$_4$, 98 %, as shown in Figure 1) (250.32 g·mol^{-1}) were obtained from Sigma-Aldrich. 2,2'-(1,8-dihydro-3,6-

disulfanonaphthylene-2,7-biazo) bisbenzenearsonic acid (Arsenazo III, as shown in Figure 1) (776.36 g·mol^{-1}). Buffer solution at *p*H equal to 2.07 prepared by ammonium acetate and hydrochloride acid (37 %), were supplied by Merck. The preparation of ultra-pure water was carried out by the YOUNG LIN distiller, Resistivity (25 °C): 16MΩ·cm, Conductance: < 0.1µS·cm^{-1}).

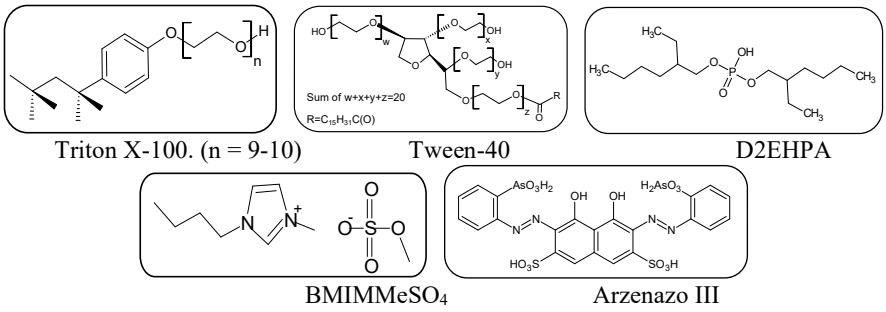


Figure 1. *Structures*

Instruments

The UV-Vis absorbance of solutions was measured using Specord 210 Plus Analytic Jena, UV–Visible spectrophotometer. *p*H-ORP-TEMP Bench Meter AD1030 was used for *p*H measurements. Thermostatic water bath (Thermo-Circulator) preserved at the distinct temperature, were applied for CPE. The weighing was made with an electronic analytical balance type Carat Series OHAUS Item: PAJ1003. The preparation of ultra-pure water was carried out by the YOUNG LIN distiller. The volumes were collected using micropipettes (SCI LOGEX 100 - 1000 µL).

Extraction procedure of uranyl ions

The CPE extraction operation was based on the following steps, as shown in Figure 2 [21]:

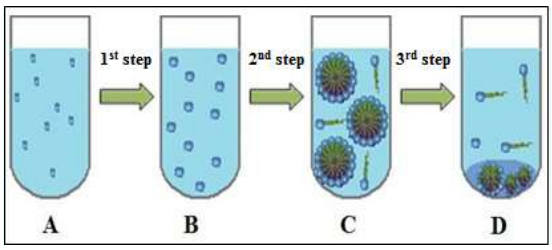


Figure 2*. Principle of cloud point extraction*
(A): Solute solution; (B): Formation of complexes after addition of complexing agent; (C): Trapping of complexes inside micelles;
(D): phase separation following the temperature rise

The balance established between the two obtained phases depends on certain parameters such as the nature and the concentration of the surfactant, those of the chelating agent, ionic strength, temperature, etc.

CPE is carried out in graduated tubes; the Triton X-100 (8 %) and Tween-40 (2 %) were mixed as non-ionic surfactants. A mixture of D2EHPA (0.02 g) and BMIMMeSO4 (0.02 g), salt (Na_2SO_4) 8 %, and then top up 10 mL with a uranium (VI) solution after *p*H adjustment. After stirring, the mixture was allowed to stand at 25 °C for 24 hours. The coacervate phase was distinguished from the dilute phase. The latter is measured by UV-visible spectrophotometer.

The UO_2^{2+} sample was analyzed by a mixture of 100 µL of Arsenazo III and 100 µL of UO_2^{2+} in a medium whose *p*H was equal to 2.0. The interaction product of Arsenazo III with UO_2^{2+} was determined at λ_{max}= 653 nm [22 – 24].

The percentage of extracted uranyl ions was determined, as observed at equation (1):

$$Yield(\%) = \frac{C_i - C_e}{C_i}.100 \quad (1)$$

Where C_i and C_e were the initial, and equilibrium UO_2^{2+} concentrations ($mol \cdot L^{-1}$), respectively.

RESULTS AND DISCUSSION

Factorial design study

The regression equation of matrix was represented by the Equation (2):

$$E(\%)= a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3 + a_{11}X_1^2 + a_{22}X_2^2 + a_{33}X_3^2 \quad (2)$$

The model calculations were achieved using non-dimensional or reduced values of these variables, each of them being varied on three levels. The following mathematical model shows the coefficients values (a_i) of the model, supposed to describe the individual effects of parameters, along with their possible interactions. The individual effects and interactions of the parameters were discussed on the basis of the sign and the absolute value of each coefficient. In our investigations, a series of 27 tests were conducted using a 3^3 factorial experience plan, varying three key variables: *p*H value (X_1), ion strength (X_2), and initial uranyl concentration (X_3). Three levels of variation for each parameter were considered summarized in Table 1 [27].

As a result, 27 experiments with all possible combinations of variables were conducted at room temperature to which three center points were added to estimate the error. The results of the uranyl extraction process were expressed in terms of extraction efficiency, considered to be the response function in the process studied. These results are summarized in Table 2. Preliminary observations show that extraction yields according to the parameters of the experiment reach values of 72.2 to 99.4 % under certain operating conditions.

From Table 2, it is clear that the lowest *p*H value, an average beginning uranyl concentration, and an average value for Na_2SO_4 (w/w %) were used to provide the maximum extraction yield value (99.4 %).

Table 1. *Factor levels used in the 3^3 factorial experiment designs at ambient temperature*

Factors	Symbol of coded variables	Low level (-1)	Medium level (0)	High level (+1)
*p*H	X_1	3	4.5	6
Na_2SO_4 [w/w %]	X_2	8	9	10
$[UO_2^{2+}]\cdot10^4$ [mol·L^{-1}]	X_3	1	5.5	10

Xj = 1 to 3: reduced variable which takes two values: –1 (low level) and + 1 (high level); low level = 2 (low value – mean)/range; high level = 2 (high value –mean)/range; mean = (high value + low value)/2; range = (high value–low value).

Table 2. *Experimental data*

Experiment Nº	Factor levels				Reduced values			Response function Extraction yield [%]
	*p*Hi	Na_2SO_4 [w/w%]	$[UO_2^{2+}]\cdot10^4$ [mol·L^{-1}]		X_1	X_2	X_3	
1	3	8	1		-1	-1	-1	95.4
2	3	8	5.5		-1	-1	0	94.3
3	3	8	10		-1	-1	+1	85.9
4	3	9	1		-1	0	-1	76.5
5	3	9	5.5		-1	0	0	99.4
6	3	9	10		-1	0	+1	84.0
7	3	10	1		-1	+1	-1	91.1
8	3	10	5.5		-1	+1	0	89.9
9	3	10	10		-1	+1	+1	79.8
10	4.5	8	1		0	-1	-1	93.1
11	4.5	8	5.5		0	-1	0	94.8
12	4.5	8	10		0	-1	+1	85.4
13	4.5	9	1		0	0	-1	82.1
14	4.5	9	5.5		0	0	0	89.3
15	4.5	9	10		0	0	+1	86.8
16	4.5	10	1		0	+1	-1	80.3
17	4.5	10	5.5		0	+1	0	91.8
18	4.5	10	10		0	+1	+1	89.1
19	6	8	1		+1	-1	-1	97.3
20	6	8	5.5		+1	-1	0	95.7
21	6	8	10		+1	-1	+1	93.9
22	6	9	1		+1	0	-1	72.2
23	6	9	5.5		+1	0	0	96.3
24	6	9	10		+1	0	+1	83.8
25	6	10	1		+1	+1	-1	80.4
26	6	10	5.5		+1	+1	0	92.8
27	6	10	10		+1	+1	+1	84.4
(28,29,30)[a]	4.5	9	5.5		0	0	0	86.5/89.3/89.0

[a]Three additional tests at the central point (0,0,0) for the calculation of the student's and Fisher's tests, using the normal rule of variance.

Model calculation and refinement

The uranyl extraction model was performed based on 27 measured values, using the second-order Taylor polynomial [28]. The model calculations were performed using non-dimensional or reduced values of these variables, each of which varied over three levels.

The following mathematical model shows the values of coefficients of the model, supposed to describe the individual effects of parameters, with their possible interactions.

$$E(\%) = 90.76 + 0.031X_1 - 3.12X_2 + 0.263X_3 - 1.21X_1X_2 + 2.11X_1X_3 + 1.83X_2X_3 + 1.146X_1X_2X_3 + 0.46X_1^2 + 4.13X_2^2 - 8.179X_3^2 \quad (3)$$

The Student's t significance test was carried out on coefficients of Equation (3) by analyzing the repeated values shown in Table 2.

With a view to reproducibility, it is necessary to verify whether this model describes with precision the studied process by determining which coefficients could be neglected, using Student's *t*-test and Fisher's test [29]. The suitability of the model strongly depends on the precision of the experiment. In the current experience, the main errors come from volume and weight measurements.

For this purpose, three additional tries of the central point (0,0,0) are demanded to estimate the average error in the value of every coefficient, based on the random variance. The calculations made are summed up in Table 3.

So, with a 95 % trust (i.e., α= 0.05), and for two variances (i.e., for three experiments at the central point), one assessed the value of $t_{v,1-\alpha/2}$ as being equal to 4.3.

As a result, in it $(1 - \alpha)$ level, the range of trust for all estimated coefficients to be using 27 runs (N = 27), will be $\Delta a_i = \pm$ 1.2673 at 95 % trust. According to Student's *t*-tests, it results in that $|\Delta a_i| < |a_i|$ for a_1 a_3, a_{12}, a_{11}, and a_{123}. Consequently, these coefficients should be removed from the mathematical model because they show a significant effect on the response function, being shaded by their mean error. Therefore, the final form of the polynomial model that describes the extraction of uranyl ions was in the following Equation (4).

$$E(\%) = 90.76 - 3.12X_2 + 2.11X_1X_3 + 1.83X_2X_3 + 4.132X_2^2 - 8.179X_3^2 \quad (4)$$

This model was supposed to accurately fit the extraction process of uranyl investigated herein.

Thus, near the expected optimal values of the parameters, it seems that only the individual effect of *p*H and the amount of Na_2SO_4 positively influences the extraction.

It can also be said that the quadratic effect of Uranyl concentration negatively influences the extraction.

The individual effects and interactions of the parameters were discussed based on the sign and the absolute value of each coefficient. These coefficient characteristics will define the strength of the corresponding effect involved and how it acts when extracting the yield (favorable or detrimental), respectively (see Table 4).

Interpretation

The effect of individual variables and interactional effects can be estimated from the above Equation (4). According to the equation of the model, it is clear that individual operating variables concentration of Na_2SO_4 (w/w %) and UO_2^{2+} concentration has a net negative effect on uranium extraction, whereas the interaction between *p*H of solution and concentration of UO_2^{2+} and the interaction between Na_2SO_4 (w/w %) and UO_2^{2+} concentration have a net positive effect. A positive afterwards negative value for the concentration of Na_2SO_4 (w/w %) indicate that the measured value of adsorbed metal amount increased and decreased as the factor was changed from its first level to its second level respectively.

Table 3. *Model adequacy tests and variance analysis*

Characteristics	Symbol/equations	Values
Parameter number	P	3
Level number	L	3
Number of experimental attempts	N	27
Number of tests at (0, 0, 0) point	n	3
Model variance	ν	2
Average yield at (0,0,0) point	$Y_0 = \sum Y_{oi}/3$	88.3
Random variance	$S^2 = \Sigma(Y_{oi} - Y_0)^2/v$	2.34
Square root of variance	S	1.53
Risk factor (chosen arbitrary)	α	0.05 (95%)[a]
Student's *t*-test factor	t_v	4.3[b]
Average error on the coefficient value	$\Delta a_i = \pm t_{v,\alpha/2} S/N^{0.5}$	±1.26
Number of remaining coefficients	R	6 [c]
Model response at (0,0,0)	$a_0(y_{000})$	90.76
Discrepancy on average yield Error on average yield discrepancy	$d = y_0 - y(0,0,0) = y_0 - a_0$ $\Delta d = \pm t_{v,\alpha/2} S(1/N + 1/n)^{0.5}$ with N=27& n=3	2.50 4.00
Average yield for the 27 attempts	$y_m = \Sigma\, y_i/27$	88.4
Residual variance	$S_r^2 = \sum (y_i - y_m)^2/(N - R)$	59.83
Degrees of freedom	ν_1	2
Residual degrees of freedom	ν_2	5
Observed Fisher's test	$F_{obs} = S_r^2/S^2$	25.51
Fisher-Snedecor law	F_{α,ν_1,ν_2}	5.78[d]

a. α = 5 % was arbitrary chosen. In this case, one regarded that a 95% confidence may be satisfactory;
b. Student tables with two degrees of freedom at a 95 % confidence, tcrit (2; 0,05);
c. After removing the less significant coefficients; d. See Fisher-Snedecor tables.

The interaction between *p*H and the concentration of uranium (VI) (X_1X_3) and the interaction between the concentration of Na_2SO_4 (w/w %) and concentration of uranium (VI) (X_2X_3) are plotted in Figures 4 and 5. As can be seen in Figure 4, for the interaction (X_1X_3) at the (+1) and (-1) for each factor at the same time, show a high extraction of uranium 84.70 %. Also, the Figure 5 show a higher extraction of (X_2X_3) 91.6 % at the (-1) and (-1) for each factor.

Table 4. *Coefficients and their corresponding effects upon yield extraction of* UO_2^{2+}

Variable	Model		Expected effect on the yield extraction
	Coefficient	Value	
X_0=1	a_0	90.76	High average extracting capacity of uranium (VI)
X_2	a_2	-3.12	Weak detrimental individual effect of X_2
X_1 X_3	a_{13}	+2.11	(++) Favorable binary interaction of X_1 and X_3
X_2 X_3	a_{23}	+1.83	(++) Favorable binary interaction of X_2 and X_3
X_2^2	a_{22}	+4.13	(++) Favorable quadratic interaction of X_2
X_3^2	a_{33}	-8.17	(- -) Slight and flat maximum with respect to X_3

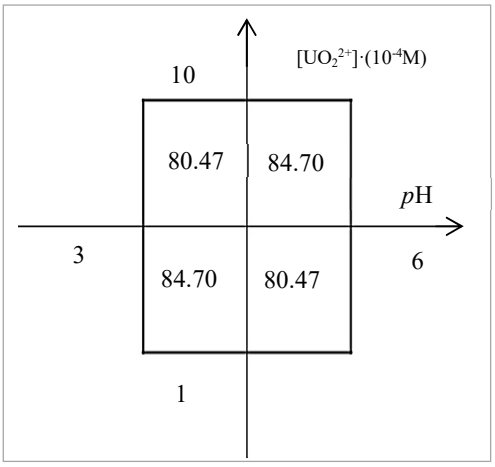


Figure 4. *Factorial interaction between pH and uranyl concentration* $[UO_2^{2+}](X_1X_3)$

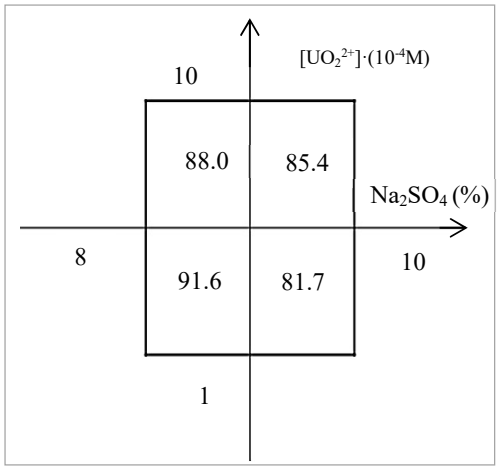


Figure 5. *Factorial interaction between ionic strength and uranyl concentration* $[UO_2^{2+}](X_1X_3)$

Figure 6 shows the relationship between expected and the current values of uranium (VI) removal from solutions using non-ionic surfactants and a mixture of lipophilic chelating extracting agent D2EHPA/ $BMIMMeSO_4$. The current data are the original measure of uranyl concentration in the solution that was estimated experimentally using Equation (1).

The expected values were generated using Equation (3). The fairly moderate value of the correlation coefficient R^2 (0.9800) was obtained between the experimental and expected response (Figure 6). It could be due to cover a wide range of process variables in a limited number of experiences and / or contribution of non-significant terms in the Equation (3). The shape of the response surface was plotted three times by fixing successively the three parameters at the central values. The vicinity around these central values is supposed to include the optimum, and the resulting 3-D representations of the response function, as illustrated by Figure 7.

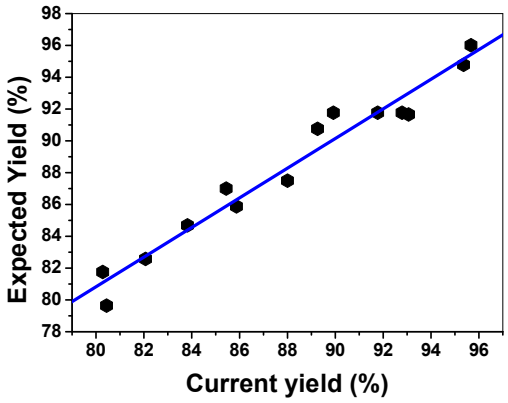
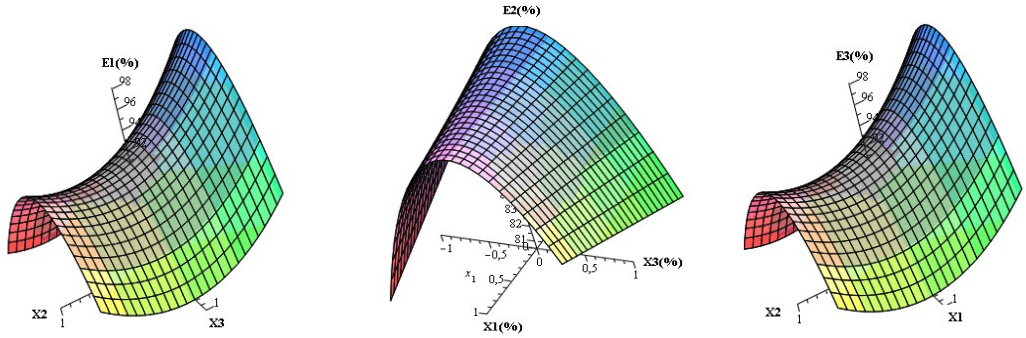


Figure 6*. The expected and current response plot of uranium (VI) extraction*



(A) (B) (C)

***Figure** 7. 3D representation of the yield extraction (%) of uranium (VI): at fixed pH (A), at fixed Na_2SO_4 (w/w %) (B) & at fixed UO_2^{2+} concentration (C)*

The comparison with other methods already used for the extraction of UO_2^{2+} ions show that CPE extraction is efficient and environmentally friendly [26 - 29].

CONCLUSIONS

Extraction of UO_2^{2+} ions in aqueous solution was investigated using cloud point extraction by a mixture of using a mixture of non-ionic surfactants Triton x-100 and Tween-40, and a mixture of extracting agent D2EHPA/ $BMIMMESO_4$ from aqueous solution. The use of ionic liquids $BMIMMESO_4$, characterized by this wide range, has various advantages such as environmental respect.

3^3 factorial design was employed for determinate the factors that would influence the extraction of uranium (VI). The most significant effect for uranium (VI) uptake is ascribed to interaction between *p*H of solution and concentration of uranium (VI) then the interaction between Na_2SO_4 (w/w %) and uranium (VI) concentration. This optimization showed that the best conditions were obtained for *p*H = 3.0, Na_2SO_4 (% w/w) = 9.0 and $[UO_2^{2+}]$ = 5.50 mM with extraction yield of 99.4 % in one step.

The results show that uranium (VI) was significantly extracted in a single-stage CPE under optimal conditions, and the polynomial models developed here can provide a valuable basis for industrial-scale wastewater treatment applications.

ACKNOWLEDGEMENT

We gratefully acknowledge the DGRSDT Algeria for the financial support.

REFERENCES

1. Sekkal, A.R., Didi, M.A., Belkhouche, N., Canselier, J.P.: Removal of chromium (III) by two-aqueous phases extraction, *Journal of Hazardous Materials*, **2009**, **167** (1-3), 896-903;

2. Kadous, A., Didi, M.A., Villemin, D.: A new sorbent for uranium extraction: ethylenediaminotris (methylenephosphonic) acid grafted on polystyrene resin, *Journal of Radioanalytical and Nuclear Chemistry*, **2010**, **284** (2), 431-438;
3. Didi, M.A., Sekkal, A.R., Villemin, D.: Cloud-point extraction of bismuth (III) with nonionic surfactants in aqueous solutions, *Colloids Surf A: Physicochem Eng. Aspects*, **2011**, **375**, 169-177;
4. Katsoyiannis, I.A., Zouboulis, A.I.: Removal of uranium from contaminated drinking water: a mini review of available treatment methods, *Desalination and Water Treatment*, **2013**, **51** (13-15), 2915-2925;
5. Meeravali, N.N., Manjusha, R., Madhavi, K., Jai Kumar, S.: Green technology for monitoring the quality of water: determination of toxic trace analyte concentrations using multi-elemental mixed micelle cloud point extraction, *Desalination and Water Treatment*, **2016**, **57** (55), 26880-26885;
6. Kadous, A., Didi, M.A., Villemin, D.: Removal of uranium (VI) from acetate medium using Lewatit TP 260 resin, *Journal of Radioanalytical and Nuclear Chemistry*, **2011**, **288 (2)**, 553-561;
7. Ferrah, N., Abderrahim, O., Didi, M.A., Villemin, D.: Sorption efficiency of a new sorbent towards uranyl: Phosphonic Acid Grafted Merrifield Resin, *Journal of Radioanalytical and Nuclear Chemistry*, **2011**, **289**,721-730;
8. Bouhoun, A.M., Badjah Hadj Ahmed, A.Y., Attou, M., Elias, A., Didi, M.A.: Liquid-Liquid extraction of uranium (VI) from aqueous solution using 1-hydroxyalkylidene-1,1-diphosphonic Acids, *Solvent Extraction and Ion Exchange*, **2012**, **30** (5), 469-479;
9. Bendiaf, H., Abderrahim, O., Villemin, D., Didi, M.A.: Studies on the feasibility of using a novel phosphonate resin for the separation of U(VI), La(III) and Pr(III) from aqueous solutions, *Journal of Radioanalytical and Nuclear Chemistry*, **2017, 312 (3)**, 587-597;
10. Medjahed, B., Didi, M.A.: Spectroscopic study of complexes from UO_2(II), Th(IV) and Sm(III) based on the effects of the characteristic TBP, TOPO and D2EHPA bands in various organic solvents, *Journal of Radioanalytical and Nuclear Chemistry*, **2018, 318** (2), 1427-1439;
11. Semmoud, R., Didi, M.A.: An efficient cloud point extraction of mixed organic-inorganic pollutants using an ionic liquid as extractant: separation of the red Bemacid dye from nickel (II) in saline medium and optimization through factorial design methodology, *Desalination and Water Treatment*, **2019**, **152**, 393-400;
12. Jashni, E., Hosseini, S.M.: Promoting the electrochemical and separation properties of heterogeneous cation exchange membrane by embedding 8-hydroxyquinoline ligand: Chromium ions removal, *Journal Pre-proofs*, **2019**, **234**, 116118;
13. Lu, Z., Hu, Z., Luo, H., Bai, Z., Zhang, B.: Application of a combined fibrous coalescence technique in deep purification separation of composite ionic liquid alkylation reaction products, *Journal of Cleaner Production*, **2020**, **261**, 121290;
14. Xie, S., Pau, M.C., Li, C.F., Xiao, D., Choi, M.M.: Separation and preconcentration of persistent organic pollutants by cloud point extraction, *Journal of Chromatography A*, **2010**, **1217** (16), 2306-2317;
15. Mortada, W.I., Kenawy, I.M.M., Abdel-Rhman, M.H., El-Gamal, G.G., Moalla, S.M.N.: A new thiourea derivative [2-(3-ethylthioureido) benzoic acid] for cloud point extraction of some trace metals in water, biological and food samples, *Journal of Trace Elements in Medicine and Biology*, **2017**, **44**, 266-273;
16. Sato, N., Mori, M., Itabashi, H.: Cloud point extraction of Cu(II) using a mixture of tritonX-100 and dithizone with a salting-out efect and its application to visual determination, *Talanta*, **2013**, **117**, 376-381;
17. Noorashikin, M.S., Mohamad, S., Abas, M.R.: Determination of parabens in water samples by cloud point extraction and aqueous two-phase extraction using high-performance liquid chromatography, *Desalination and Water Treatment*, **2016**, **57** (47), 22353-22361;
18. Swadesh, M., Susanta, L.: A review on extraction, preconcentration and speciation of metal ions by sustainable cloud point extraction, *Microchemical Journal*, **2022**, **175**, 107150;
19. Didi, M.A., Makhoukhi, B., Azzouz, A., Villemin, D.: Colza oil bleaching through optimized acid activation of bentonite. A comparative study, *Applied Clay Science*, **2009**, **42** (3-4), 36-344;
20. Cheng, C.S.: Theory of Factorial Design. Chapman and Hall/CRC, **2016**;
21. Naït-Tahar, S., Didi, M.A.: Cloud Point Extraction of Uranyl ions using TX-100 with N-butyl N'-triethoxy methyl imidazolium/D2EHPA-H ionic liquid in acetate media, *Current Physics*, **2017**, **7** (1) 57-62;

22. Oukebdane, K., Semmoud, R., Didi, M.A.: Cloud Point Extraction of Telon Orange Anionic Azo-dye from aqueous solutions by Tween 40 as non-ionic surfactant and A336 as ionic liquid and optimization through factorial design methodology, *Desalination and Water Treatment*, **2022**, 1-9;
23. Naït-Tahar, S., Didi, M.A.: Uranium micelle-mediated extraction in acetate medium: factorial design optimization, *Journal of Radioanalytical and Nuclear Chemistry*, **2012**, **293**, 789-795;
24. Fang, K.T.: Theory and application of uniform experimental designs, Springer, **2018**;
25. Hegazy, K., Abdel-Ghani, N.T., El-Chaghaby, G.A.: Factorial design for optimizing the removal of aluminium from aqueous solutions by adsorption on Typha domingensis phytomass, *Desalination and Water Treatment*, **2011**, **36** (1-3), 392-399;
26. Zaoui, F., Didi, M.A., Villemin, D.: Investigation of 7-((dioctylamino) methyl) quinoline -8-ol for Uptake and Removal of Uranyl ions, *Journal of Radioanalytical and Nuclear Chemistry*, **2013**, **295** (1), 419-424;
27. Zaoui, F., Villemin, D., Bar, N., Didi, M.A.: New complexone derivatives of 8-hydroxy-quinoline and theirs application in UO_2^{2+} extraction, *European Chemical Bulletin*, **2014**, **3** (8), 783-787;
28. Benmansour, Y., Didi M.A.: Uranyl (II) Removal by Lewatit TP 214 Resin, *International Journal of Scientific Research & Engineering Technology (IJSET)*, **2021**, **16**, 1-6;
29. Benmansour, Y., Didi, M.A., Abderarhim, O.: Amberlite IRA-93 and modified Amberlite IRA-93 resins for the extraction of uranyl ions: optimization through factorial design methodology, *Desalination and Water Treatment. Science and Engineering*, **2022**, **249**, 281-296.