MANSOURA JOURNAL OF CHEMISTRY
Official Journal of Faculty of Science, Mansoura University, Egypt
E-mail: scimag@mans.edu.eg ISSN: 2974-4938

Cyclo voltammetric parameters for the interaction of uranyl zinc acetate (UZAc) with 4-Fluoro benzoic acid (FBA) at 291.95K using glassy carbon electrode

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Received:17/1/2023
Accepted: 27/3/2023

Abstract: The redox behavior of uranyl zinc acetate (UZAc) alone and in the presence of 4-Fluoro benzoic acid (FBA) was studied in 0.1 M HCl as supporting electrolyte. The cyclic voltammograms gave a reversible redox couple for the transfer of U (VI) to U (IV). Zn ion gave waves out of our discussion range. Complex is formed by the interaction of UZAc, with 4-Fluoro benzoic acid was formed. The thermodynamic Gibbs free energies and stability constant for the interaction of uranyl zinc acetate with 4-Flouro benzoic acid were estimated and their values were discussed.

keywords: Cyclic voltammetry, reversible processes, electron transfer rate constant, stability constant , Gibbs free energy of complexation.

I.Introduction

Uranium is the heaviest natural element in theworld,with 92 atomic number. Uranium is the source of concentrated big energy. A kilogram of natural uranium produces bigger energy than 20 tons of coal. Uranium is found in granite and sedimentary rocks. Unlike fossil fuels, uranium can combine with oxygen releasing heat. Uranium atoms can be made to study the redox behavior of uranyl ions in solutions help for obtaining many results about different valences of uranium in solutions fission releasing energy [1,2]. It is composed of two isotopes:

U-238 is 99.2% of the entire mass of uranium ore and has a half-life of 4.46 billion years. Fissile U-235 has a half-life of 713 million years and makes up only 0.7% of the total mass. A plentiful source of focused energy is uranium. Uranium atoms may be made to fission to release heat, unlike fossil fuels which release heat when they burn with oxygen. The heat output of one kilogram of natural uranium is equivalent to 20 tons of coal. This heat has the potential to produce steam and power. More than 190 minerals include uranium, which is mostly found in granite and sedimentary rocks. It is widespread throughout the crust of the earth and can be found in many different geological locations.

Uranium is predicted to be present in the earth's crust in amounts ranging from 1 to 4 parts per million (ppm). It is possible to extract uranium from the oceans. Seawater is reported to contain0.003 ppm of uranium on average.

Limited information is available on the probable long-term chemical toxicity [3–8] of uranium (U).

2. Materials and methods

Uranyl zinc acetate (UZAc), 4-Flouro benzoic acid, and HCl were supplied by sigma aldrich and el-gomheria pharmaceutical company, respectively. The electrochemical study was done using a DY2000 potentiostat in a cell containing 30 ml of 0.1 M HCl as the supporting electrode. One electrode was a platinum wire auxaliry electrode, followed by a glassy carbon working electrode (GCE) and an Ag/AgCl standard electrode inserted into a saturated KCl reference electrode. The cell used is Ag/AgCl (s), KCl (sat) /aqueous 0.1M HCl /GCE. Cleaning the working electrode using line emery paper doped with Al_2O_3 powder is crucial and required in the subsequent production of the working electrode was washed several times with double distilled water and dried.

3. Results and Discussion

I. The electrochemical behavior of ($ZnUO_2(CH_3COO)_4$) in HCl (0.1M) in absence of 4-Fluoro benzoic acid (FBA)

Electrochemical study of uranyl zinc acetate (UZAc) alone in 0.1 M HCl using window range from 1.4V to - 1.4V. Many small prewave were obtained for uranyl zinc acetate (UZAc) in the studying range in comparison to the blank 0.1 M HCl uranyl ion (UO_2^{+2}) giving two reversible redox waves for both reduction and oxidation, both at ~ -1.0V. These two waves one for reduction and another one for oxidation are the main study target here. Zinc ions have reduction waves above 2 volts which is out from our study. The reversible uranyl ion waves given in Fig.(1) & Fig.(2) are using two electrons for the reduction transfer of hexavalent uranium to tetravalent one at the glassy carbon electrode and then the reverse oxidation of the tetravalent uranium to hexavalent one [9,10] following equation (1).

$$UO_2^{2+} + 4\,H^+ + 2\,e^- \rightleftharpoons U^{4+} + 2\,H_2O \quad (1)$$

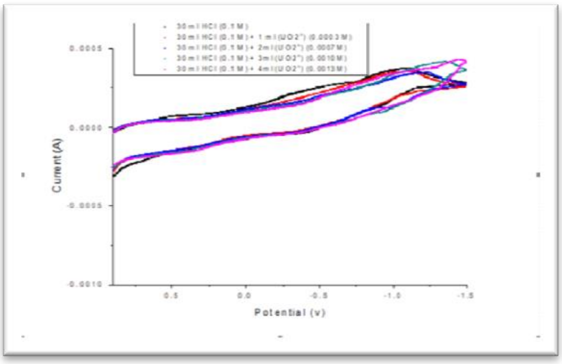

Fig.(1) Cyclic voltammogram of different concentrations of UO_2^{+2} (0.0003, 0.0007,0.001 and 0.0013) M at 291.95K.

The cyclic voltammograms for different uranyl zinc acetate concentrations are shown in Fig (1) with small increase in the reduction wave at ~ - 0.5 V and oxidation peaks at + 0.4V indicating diffusion-controlled reaction.

The various equations used for data analysis by cyclic voltammetry are listed below. [11-20]

$$i_p = 0.4463\, nFAC(nFD_C\, \nu/RT)^{1/2} \quad (2)$$

$$D^{\frac{3}{2}} = (\text{slope, Ip } \nu^{\frac{1}{2}}) * \frac{(RT)^{1/2}}{0.4463\, n^{3/2} F^{3/2}\, A\, C} \quad (3)$$

$$\Delta Ep = Ep{,}a - Ep{,}c = 2.303\,\frac{RT}{nF} \quad (4)$$

$$\psi = \frac{\gamma^{\alpha} * ks}{\sqrt{\pi \frac{nF}{RT} \nu\, Da}} \quad (5)$$

$$\gamma = \sqrt{\frac{Da}{Dc}} \quad (6)$$

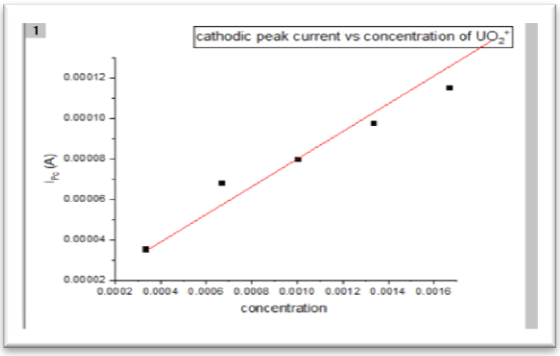

Fig. (2): Cathodic peak current against concentration of UO_2^{+2} in 0.1 M HCl (0.1 M) at 291.95K

where α is the charge transfer coefficient, Ψ is the charge transfer parameter, which is assumed to be one for better approximation[21], ν is the scan rate, while s is the standard rate constant for electron transmission, n is the number of electrons, Da is the anodic diffusion coefficient, F is the Faraday constant, R is the gas constant, T is the absolute temperature, and = 0.5 for computations that can be reversed. The surface area of the working electrode is A. The Ks, the heterogeneous electron rate constant can be evaluated by applying equation (7)

$$k_s = 2.18 * [D_C\, \alpha n_a\, F\nu/RT]^{1/2} * \exp[\alpha^2\, nF\, \Delta E_p/RT] \quad \mathbf{(7)}$$

The surface coverage of the redox species in ($mol.cm^{-2}$) was estimated by the use of

equation (8)[20--25]

$$\Gamma = i_p\, 4RT/n^2F^2A\nu \quad (8)$$

The quantity of electricity used for the reduction and oxidation reactions was evaluated by applying equation (9) [25]

$$Q = n\, FA\, \Gamma \quad (9)$$

All the above parameters following equations from 2 to 8 for the redox reaction of uranyl zinc acetate were evaluated and presented in Table (1).

Table (1) Cyclic voltammetry parameters of different concentrations of UO_2^{+2} in absence of 4-flouro benzoic acid (FBA) in (0.1 M) HCl at 291.95 K

[M]	**(-) EPa(V)**	**(-) Epc (V)**	**(-) ipa $*10^5$ (A)**	**ipc $*10^5$ (A)**	**Da (Cm ^{2}.S^{-1})**	**Dc $*10^2$ (Cm^2.S^{-1})**	**Ana**	**ks *10 (Cm.S^{-1})**	**Γc $*10^6$ (mol.cm^{-2})**	**Qc x10^{-3} (C)**	**Γa $*10^{-6}$ (Mol.cm^{-2})**	**Qa x10^{-3} (c)**
0.0003	0.9923	1.0884	4.44	3.55	4.4354	2.84	0.1681	26.8972	1.0247	1.33	1.2809	1.67
0.0007	1.0451	1.0811	3.89	6.83	0.9099	2.80	0.1717	26.5018	2.0577	2.62	1.1729	1.49
0.0010	1.0485	1.1164	2.66	7.99	0.1998	1.80	0.1749	21.0982	2.4959	3.12	0.83197	1.04
0.0013	1.0439	1.1381	7.10	9.76	0.8184	1.55	0.1762	19.5129	3.0987	3.84	2.2536	2.79
0.0017	1.0213	1.0489	5.32	11.5	0.3931	1.18	0.1672	17.3832	3.2964	4.31	1.5214	1.99
0.0020	1.0193	1.0200	6.21	12.2	0.3274	1.67	0.1647	6.5742	3.4093	1.96	1.7225	2.29

Ks, electron transfer rate constant is decreased by increasing in uranyl zinc acetate solution favoring more diffusion and solvation effect.

Increasing the concentration of uranyl zinc acetate (UZAc) from 3* to 2* in 0.1M

HCl causes an increase in the cathodic peak potential Epc, anodic peak potential

Epa, anodic current Ipa, cathodic current Ipc, anodic diffusion coefficient Da,

cathodic diffusion coefficient Dc, anodic surface coverage c, Qa anodic quantity of

electricity, and cathodic quantity of electricity, This backs up the reduction and

oxidation of uranyl zinc acetate using the diffusion process (UZAc). The

relationship between ip,c and (UZAc) concentration in Fig. 2 provided a positive,

straight line that also supported the diffusion process.

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HCl causes an increase in the cathodic peak potential Epc, anodic peak potential

Epa, anodic current Ipa, cathodic current Ipc, anodic diffusion coefficient Da, cathodic diffusion coefficient Dc, anodic surface coverage c, Qa anodic quantity of electricity, and cathodic quantity of electricity, This backs up the reduction and oxidation of uranyl zinc acetate using the diffusion process (UZAc). The relationship between ip,c and (UZAc) concentration in Fig. 2 provided a positive, straight line that also supported the diffusion process.

Increasing the concentration of uranyl zinc acetate (UZAc) from $3*10^{-4}$ to $2*10^{-3}$ in 0.1M HCl causes an increase in the cathodic peak potential Epc, anodic peak potential Epa, anodic current Ipa, cathodic current Ipc, anodic diffusion coefficient Da, cathodic diffusion coefficient Dc, anodic surface coverage c, Qa anodic quantity of electricity, and cathodic quantity of electricity, this backs the reduction and oxidation for uranyl zinc acetate using the diffusion process (UZAc).The relationship between ip,c, and (UZAc) concentration in Fig. (2) provided a positive straight line that also supported the diffusion process.

II.The electrochemical behaviour for uranyl zinc acetate (UAZc) in presence of 4-Flouro benzoic acid.

The electrochemical behaviour of the complex interaction between uranyl zinc acetate (UZAc) and P-Flouro benzoic acid) (FBA) in the range between 1.4-1.8V.

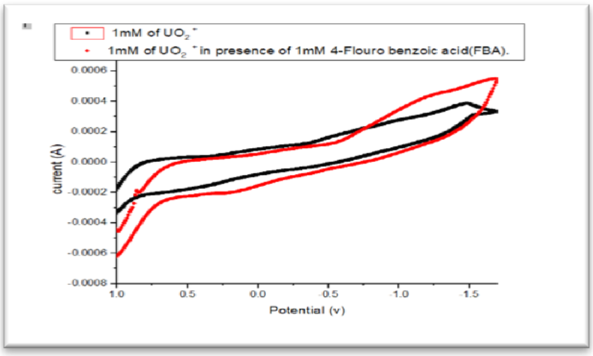

Fig (3). Cyclic voltammograms for interaction of 1mM of UO_2^{+2} in presence of 1mM (FBA).

4-fluoro benzoic acid (FBA) increase the peak currents for both the reduction process of uranyl zinc acetate and the oxidation one indicating slight complexation effect observed.

Different concentration of 4-Fluoro benzoic acid was used with $2*10^{-3}$M (UZAc) in the range of FBA ranging from $8*10^{-4}$ to $5*10^{-3}$M.The temperature at which the experiments done is 291.95K.

Fig.(4) and Table.2 explain the different effect of FBA concentration on used UZAc cyclic voltammogram.

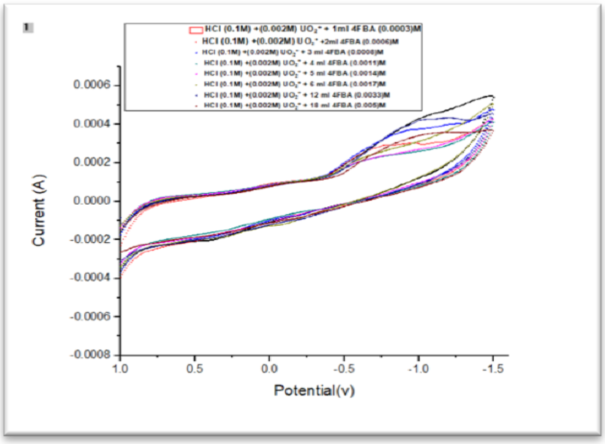

Fig (4). Cyclic voltammograms for interaction of 1mM of UO_2^{+2} in presence of 4-Flouro benzoic acid (FBA).

Fig (4) shows the appearance of reduction and oxidation uranyl peaks at ˜ - 1.0 v. Also, we observed two small peaks at ˜0.2v for the reduction and + 0.2 v at the oxidation site which prove small appearance of unstable U (v) waves which are disproportionation to uranium four and uranium six

ΔE increased by increase in 4-fluoro benzoic acid. Concentration followed by small increase in the free energy values indicating electrostatic attraction between them

All the cyclic voltammetric analysis data given in Table 3 & 4 for the effect of FBA on (UZAc) waves show smaller data than that in absence of FBA. This indicate that the interaction between FBA and UZAc is weak in the range of electrostatic attraction. The complexation stability constant (β) for (UZAc – FBA) complexes in 0.1M HCl is calculated by applying equation (10)

$$(Ep)_c - (Ep)_M = 2.303 \frac{RT}{nF} \text{ Log } \beta_c + 2.303 \frac{RT}{nF} \log C_L \quad (10)$$

Where β_c is the stability constant (Ep) M is peak potential for UZAc in absence of FBA, (Ep)c is the peak potential of the complex. Other symbols are explained before. The Gibbs free energies of interaction between (UZAc)and FBA [26] were calculated by the use of equation **(11)**

$$\Delta G = -2.303 \text{ RT Log } \beta_c \quad (11)$$

From the data given in Table 4 we conclude that the interaction between UZAc and FBA leads to the formation of a weak complex with small stability constant. This is also supported by the Gibbs free energies of interaction between FBA and UZAc.

Table (2) Effect of concentration for Ligand 4- Fuoro benzoic acid (FBA) on 1 mM UO_2^{+2} at 0.1 V scan rate at 291.95 K

[L]	(-) EPa(V)	(-) E pc(V)	(-) Ipa x10-5 (A)	Ipc x10-4 (A)	Da ($Cm2.S^{-1}$)	Dc ($Cm2.S^{-1}$)
0.0003	1.0384	1.0411	7.99	1.86	0.3985	2.1694
0.0006	0.9181	0.9736	1.06	1.86	0.5332	1.6330
0.0008	1.0340	1.0742	7.99	1.24	0.4152	1.0047
0.0011	1.0524	1.0600	8.87	7.10	0.5157	0.3300
0.0014	0.9704	0.9725	5.26	1.06	0.1407	0.5777
0.0017	0.8236	0.8570	5.32	1.42	0.0935	0.6648
0.0033	0.9145	0.9683	4.44	1.95	0.0913	1.7672
0.0050	0.9327	0.9900	7.99	2.31	0.3150	2.6286

Increasing of Ipa, Ipc, Da, Dc by adding 4-fluoro benzoic acid (FBA) to uranyl zinc acetate solution favoring complexation reaction

Table (3) Effect of concentration for Ligand 4-Flouro benzoic acid (FBA) 1 mM UO_2^{+2} at 0.1v scan rate.

[L]	Epc/2	αna	Ks *10 ($cm.S^{-1}$)	Γc $x10^6$ (mol.cm-2)	Qc $x10^3$ (C)	Γa $x10^6$ (mol.cm-2)	(-) Qa $x10^3$ (C)
0.0003	1.0450	0.1067	23.5193	5.373	6.99	2.3028	3.00
0.0006	0.9771	0.1173	21.1649	4.446	6.36	2.5407	3.63
0.0008	1.0782	0.1052	15.9239	3.682	4.72	2.3669	3.04
0.0011	1.0639	0.1050	9.1196	2.112	2.71	2.6402	3.38
0.0014	0.9761	0.1142	12.4570	2.680	3.73	1.3229	1.84
0.0017	0.8602	0.1320	14.1715	2.674	4.30	1.0027	1.61
0.0033	0.9718	0.1178	22.0585	4.614	6.63	1.0487	1.51
0.0050	0.9936	0.1154	26.6802	5.687	8.00	1.9686	2.77

The parameters Qc, Γa, Qa are increased by increase of 4-fluorobenzoic acid concentration added to uranyl zinc acetate favoring interaction of FBA with uranyl ions in 0.1M HCl solutions.

Table (4) Solvation parameter of the interaction among UO_2^{+2} and 4-Flouro benzoic acid (FBA) at 291.95K

[M]	[L]	E1/2 (M)	E1/2 (C	ΔE (mv)	log βj	βj	ΔG (KJ/mol)
0.002	0.0003	0.0004	0.0013	0.0009	0.4020	2.5236	-2.2473
0.002	0.0006	0.0004	0.0277	0.0274	1.3407	21.9110	-7.4943
0.002	0.0008	0.0004	0.0201	0.0197	1.0165	10.3883	-5.6825
0.002	0.0011	0.0004	0.0038	0.0034	0.3797	2.3972	-2.1226
0.002	0.0014	0.0004	0.0010	0.0007	0.2619	1.8278	-1.4642
0.002	0.0017	0.0004	0.0167	0.0163	0.8319	6.7908	-4.6504
0.002	0.0033	0.0004	0.0269	0.0265	1.1584	14.3999	-6.4752

Effect of change scan rate of complex

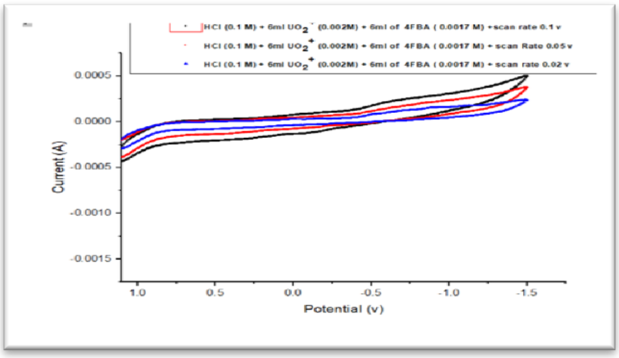

Fig (5) Cyclic voltammetry behaviors of 1mM UO_2^{+2} in presence of 4FBA at 0.1 M HCl at various scan rates.

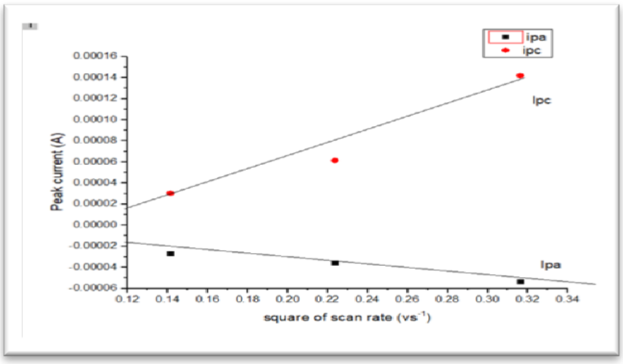

Fig (6) cathodic and anodic current density against square root of different sampling for 1mM UO_2^{2+} in presence of 4FBA at 0.1 M HCl at 291.95K

Table (5) Analysis data of 1:1 complex at different scan rates for complex UO_2^{+2} in presence of 4-fluorobenzoic acid.

	v^ (1/2)	(-) EPa(v)	(-) Epc (v)	(-) ipa x105(A)	Ipc(A)	Log βj	βj	ΔG(KJ/mol)
0.1	0.3162	1.0152	1.0348	5.325	0.00014	2.7751	595.7823	-15.5128
0.05	0.2236	1.0439	0.9018	3.550	0.00006	2.7773	598.7613	-15.5249
0.02	0.1414	1.0607	0.9151	2.662	0.00003	2.7780	599.8247	-15.5292

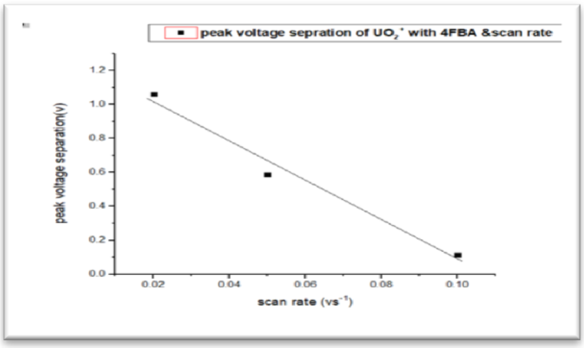

Fig (7) peak voltage separation against scan rate for cyclic voltammograms of 1 mM of UO_2^{+2} in presence of 4 FBA at various scan rate at 291.95K

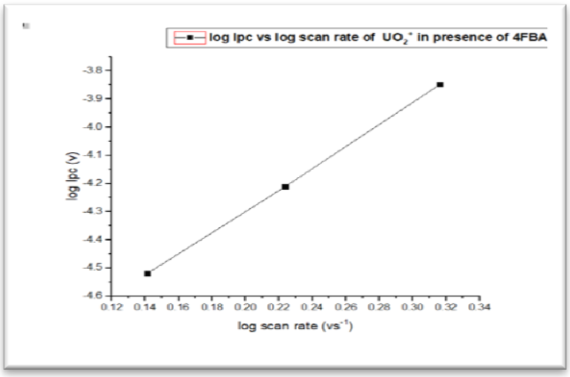

Fig (8) log Ipc and log ν for 0.003 M of UO_2^{+2} in presence of 4 FBA at various scan rates at 291.95K

Conclusions:

We explained the redox reaction for the uranyl zinc acetate (UZAc) from the cyclic voltammograms of different (UZAc) concentrations in 0.1M HCl and at 291. 95K.The different effect of p-fluoro benzoic acid on the redox peaks of (UZAc) was studied and their effect was discussed. The complex formation between UZAc and FBA was founded to happen and the Gibbs free strength complexation in the range association processes.

All curves of the scan rate effect are linear ones indicating that the reaction is diffusion controlled.

The cyclic voltammetry peaks for zinc acetate are above our scanning range as done and supported in literature [27].

Both the adsorption and desorption reactions as well as reduction/oxidation reactions of uranyl acetate on glassy carbon electrode were studied and found it is reversible from the little difference in anodic and cathodic peak potentials [28]. Uranyl hexavalent is first pre-adsorbed on the glassy carbon electrode and the washed during cyclic voltammetry scanning in 0.1M HCl is shifted to small values. The difference between adsorption-desorption depends on the type of electrode. The peak width is also dependent upon the type of working electrode [29]. Uranyl molecules exhibits a characteristic asymmetric stretching whose energy is dependent on the oxidation state of U(VI) and its reduction will result in downshift of the vibration value [30]. Also, it is known that the coordination of uranyl ions with functional group like carboxylic group leads to change in U(VI)/U(IV) redox potential [31]. Coordination of uranyl ion with carboxylic group is also in agreement with DFT theoretical calculations which show that

–COOH groups form much stronger surface complexes with U(VI) than other oxygen or hydroxides [32].

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