*Macrocyclic Carriers for Separation of ReO⁻₄ in Bulk Liquid Membrane .

MoayyedG. Jalhoom   Jameel Muosa Dhababe   Yussra Oumran Mussa

1-Ibn Sina State Company   .2-Dep. Of Chemistry/Collage of Science/AL-Mustansiriya university. 3-Dep. Of Chemistry/Collage of Education /AL-Qadisyia university.

*Consltant in Ministry of Industrial and Minerales

Abstract:

Transport experiments a cross bulk liquid membranes (BLM$_s$) are   presented . The competitive transport of ReO⁻$_4$ ions through a BLM with dibenzo-18-crown-6(DB18c6) , di cyclohexyl- 18- crown-6 (DCH18C6, diaza-18-crown-6(DA18C6) and kryptofix 2.2.2 (Cryp$_{2.2.2}$)were investigated .

The effect of stirring speed , type of carrier and type of organic solvent on the transport of ReO⁻$_4$ ions from source phase consist of 0.001 M from KReO$_4$ in 10 ml of double distilled deionized water to receiving phase consisted of 10 ml of double distilled deionized water at PH = 3.2-3.7 a cross organic phase consist of 0.01 M of Crown ether in 40 ml of organic solvent . The permeability and the rate transport of ReO⁻$_4$ ions were determined,they are founed to be favrouble and more acceptable with the compound dicyclohexyle 18-Crown-6 as Carrier,1,2-dichloro ethane as organic solvent and 100 rpm rat speed in this model.

1-Introduction :

The selectivity and removeal of toxic metal cations from water has frequently been addressed in membrane separation systems . There are specific reasons why an ion receptors [1]first is the large radii of many anion , which necessitates the design of hosts with large cavities , second is the small binding affinities to hosts because of the weak basic character of the axoanion oxygen atoms and the broad distribution of charge over the anionic structure third anions often have large solvation energies , which must be over come as part of complexation process . Because of these difficulties , few selective membrane separation systems have been developed for anions[2].

Interest in the coordination chemistry of anionic substrates continues to attract of anions [3,4] in biological system can scarcely be underestimated anion have a great relevance from a biological point of view since over 70% of all cofactor and substrates involved in biological are of anion nature[5] . Another important target of metal anion separation is technetium . Nuclear wastes contain radioactive [99] Tc , which is often present in solution as pertechnetate[6]. Also , radioactive ^{99}Tc and [188]Re are generated in isotonic solutions as pertechnetate or perrhenate for use in medical diagnostic and therapeutics[7]. Another separation of radioactive using solvent extraction for excellent separation for technetium as H⁺ crown TcO⁻$_4$. nH$_2$SO$_4$ [8] and

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basic solution from irradiated uranium samples [9] . Also , the separation of ReO$^-_4$ anion done using solvent extraction technique from aqueous solutions of alkali metals hydroxides by swelled PDB18C6 and swelled PC221 can be represented by following equation[10] .

$$M_{aq}^{+} + + \mathrm{Re}\,O^-_{4\,aq} + 1L_{or} \xrightarrow{\quad Kexl \quad} [M + [\mathrm{Re}\,O^-_4]].......1$$

$$[M^+ L\,\mathrm{Re}\,O^-_4] \xleftarrow{\quad Kaiss \quad} \mathrm{Re}\,O^-_4 + [M^+L]_o...........2$$

$$[M^+L]_o + OH^-_{aq} \xrightarrow{\quad Kexz \quad} [M^+LOH^-]............3$$
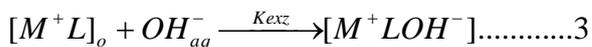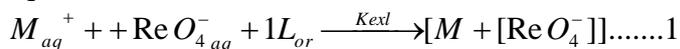
Where
 M$^+$ = Li, Na , K , Rb , Cs
 L = PDB18C6 and PC221

Another separation for ReO$^-_4$ ion using solvent extraction from aqueous acidic, neutral and basic solution of alkali metals hydroxides and chlorides with 12-crow-4 (12C4) ,15-Crown-5(15C5),18-Crown-6 (18C6) m dibenzo 18-crown-6 (DB18C6) , crypt 2.2.1 (C221) and Crypt 2.2.2 (C222) . as represented in the below equation[11] :

$$M^{n+}_{aq} + 1L_{org} + nA- \xleftarrow{\quad Kex \quad} [M^{n+}L, A^-_n]_{org}$$
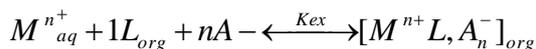
$$Where\ M^{n+} = Ba^{2+}, Sr^{2+}, ca^{2+}, Mg^{2+}, Cs^+, Rb^+, K^+, Na^+, Li^+, H^+$$

$$A^- = \mathrm{Re}\,O^-_4$$

2-Expermintal:
2.1-Reagents :
Inorganic chemicals such as Rhenium heptoxide ampoule (Re_2O_7) , potassium hydroxide ,1,2-dichloroethane($ClCH_2CH_2Cl$)were of analytical grade (98%) and were purchased from BDH company organic reagents , such as Dibenzo18-crown-6 (DB18C6) dicyclohexyl18 -Crown -6(DCH18C6) , diazal18-Crown -6 (DA18C6) and kryptofix222 were also of analytical grade (99%) and were purchased from SIGMA – Al DRICH and MERIC company. Aqueous solutions were prepared with double distilled water with a conductivity of ≥ 0.1 μS cm^{-1} .

2.2-Transport Studies :
Transport experiment were carried out in permeation shape tube setup as described in fig (1) . The BLM was contained in the bottom of the U- tube , The membrane consist of 0.001M of carrier dissolved in 40 ml of organic solvent and covered by two layer of source phase which is consist of 9x10^{-3} M of Re_2O_7 + 0.01 M KOH) in 10 ml of double distilled deionized water and receiving phase which is consist of 10 ml of doubled distilled deionized water at PH = 3.2-3.7 taken from feeding and receiving phase every 30 minuts along 3hr . Crown ethers which are used in the liquid membrane were recovered by washing the extraction liquid membrane phase five times using doubled distilled deionized water and the organic solvent then distilled using simple distillation setup to obtain the dried compound,the purity was checked by measuring the melting points .

Fig 1: U-shape setup.

2.3-Carrier and transport mechanism :

Ions are transported through hydrophobic membranes from the aqueous source phase (containing the ions to be separated) to the aqueous receiving phase . As carrier in the membrane binds cations from the source phase , anions must be Co-transported to maintain charge neutrality through the membrane as shown in fig(2). Ion can then be released into the receiving phase we used equation1&2 to calculate permeability (P) m/s and flux, C the concentration in receiving or source phase (M) , C_o represents the initial concentration in source phase (M) , The volume of source phase (L) , A the membrane surface area (m^2) , and t is the time (s) [12]and J_M cation flux :

$$P = \frac{(C \, lC_o)v/A}{t} \ldots\ldots\ldots\ldots\ldots\ldots(1)$$

$$JM = \frac{Cxv}{Axt} \ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$
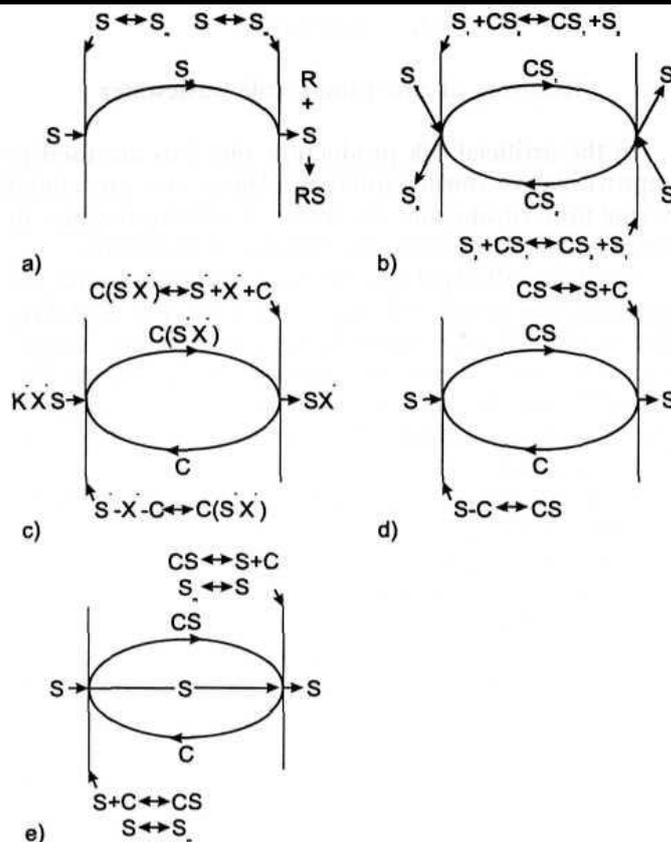
Fig.(2):Mechanisms of transport in LM,
C:Carrier,S:Substrate,x:Counter ion.

a)Simple nass transfer.

b)Combined Co-current mass transfer with a carrier.

c)Combined Countercurrent mass transfer with a carrier.

d)Complex mass transfer with a carrier.

e) Complex mass transfer with a carrier.

3-Result and Disscution:

3.1-Effect of stirring speed :-

Data obtained for the kinetic parameters , has reflectes that a stronger mixing would attributes an impact on the thickness of the diffusion layer ,accordingly an increase the kinetic reliance on the interfaced chemical reaction [13,14,15] might be obtained results obtained that optimum stirring rate should be controlled as high stirring speeds are not appropriate for removal of perrhenate ions form aqueous water solution which are associated with results as shown in table (1,2) and fig(3,4,5)

Table 1: Effect of stirring speed on flux and permeability of ReO_4^- in the source phase

Stirring speed	80 rpm	100 rpm	150 rpm
J/mo/m².sec	1.422×10^{-7}	3.920×10^{-7}	3.293×10^{-7}
Per/mol/sec	1.422×10^{-1}	3.920×10^{-2}	3.293×10^{-2}
K_{ex}	1.0×10^{-3}	1.7×10^{-3}	1.3×10^{-3}

Table 2: Effect of stirring speed on flux and permeability of ReO_4^- ion in the receiving phase

Stirring speed	80 rpm	100 rpm	150 rpm
J/mo/m².sec	5.64×10^{-10}	8.46×10^{-9}	1.693×10^{-9}
Per/mol/sec	5.64×10^{-4}	8.46×10^{-3}	1.693×10^{-3}
$K_{b\ ex.}$	1.08×10^{-4}	1.18×10^{-4}	1.27×10^{-4}
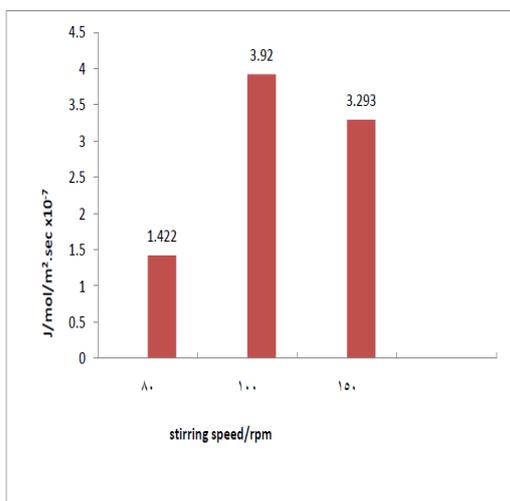
Fig 3: Effect of stirring Speed on flux of ReO_4^- ion
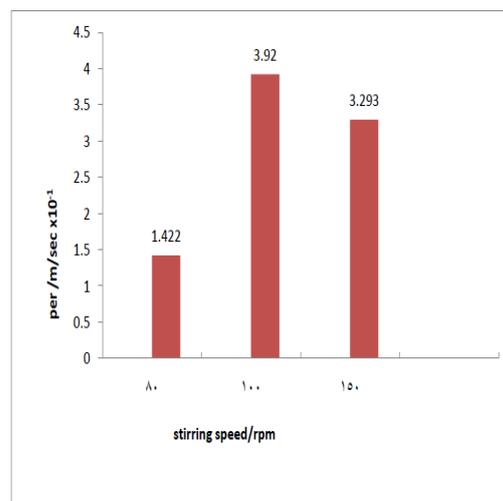
Fig 4 : Effect of stirring Speed on permeability of ReO_4^- ion
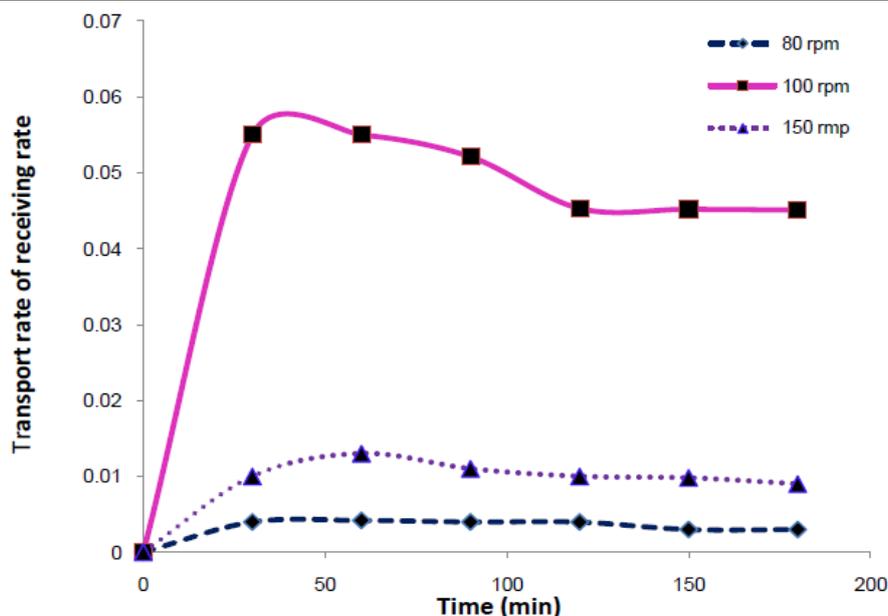
Fig5: Effect of Stirring Speed on the transport rate of receiving phase ReO₄⁻ ion

3.2- Effect of carrier structure :

The two – dimensional monocyclic structures (18-Crown-6) can be developed into three dimensional bi- and tricycle structures [2.2.1] cry ptaned,[2.2.2] cryptaned which are stronger complexation ability and higher selectivity towards metal ion[16,17,18],understanding of the relationship between structure and complexation property should explaine the effect of host topology on the complextion and selectivity since the host must undergo conformational adjustment to provide a proper binding environment during the host – guest interaction .

The bicyclic and tricycles macrocycles - encapsulate potassium cation guest inside their cavity and that nitrogen atoms are located at the bridge head positions in his structure will causes a high extracting power in the membrane phase and low striping for receiving phase with the time but in comparision withthe monocyclic structure (18- Crown-6) with its cavity (2.6–3.2) A° and according to the size-fit principle (matchining of size) as shown in fig (6).A host binds a guest most strongly if the diameter of the guest is close to , but no larger than the ring or cavity size of the host of the alkali metals , as expected K^+ has the highest binding affinity for the 18-Crown -6 cavity . We predict there is different effects of hosts which are substituted by di-benzo groups less fitness and weaker cavity interactions than dicyclo group to delocalize effect by double bonds causing decreasing the basisti of the oxygen atoms while, cyclohexyl group have flexibility enhancing conformation of cavity host to contain potassium cation guest.On the other hand diaza18-Crown-6 which was found to react with the solvent DCE to form K_{22C2} compound as in fig (7) and table(3,4).

$$M^{n+} + L \rightarrow ML_n \rightarrow M^{n+} + L$$

L:ligand

M^{n+} : Metal cation
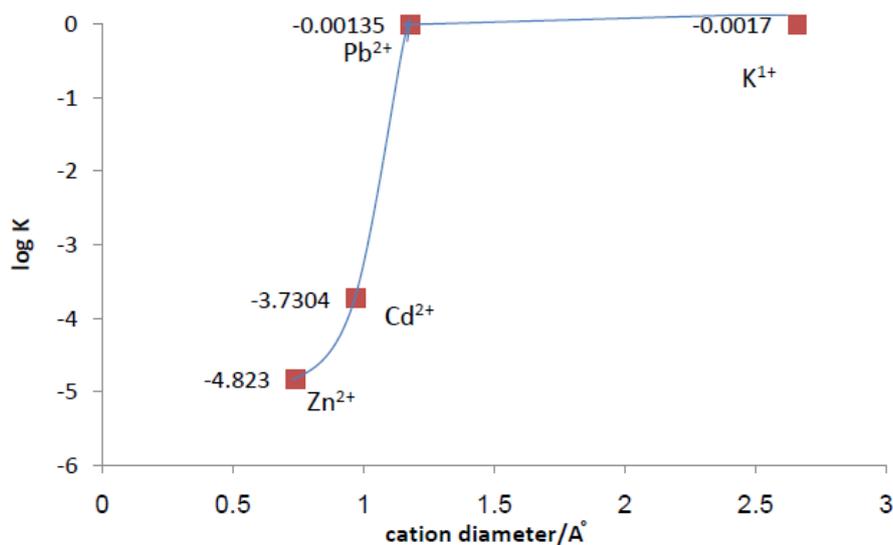
Fig. 6: Effect of ion diameter on Log K.

Table   3:Effect of carrier structure on flux and permeability of ReO$_4^-$ ion in the source phase

Carrier	DB18C6	DC18C6	DA18C6	Cry[2.2.2]
J/mo/m^2.sec	2.883x10^{-7}	9.889 x10^{-8}	9.576 x10^{-8}	3.920 x10^{-8}
Per/mol/sec	2.883 x10^{-1}	9.889 x10^{-2}	9.576 x10^{-2}	3.9201 x10^{-2}
K $_{ex.}$	1.5 x10^{-3}	1.7 x10^{-3}	1.3 x10^{-3}	0.91 x10^{-3}

Table 4:Effect of carrier structure on flux and permeability of ReO$_4^-$ ion in the receiving phase

Carrier	DB18C6	DC18C6	DA18C6	Cry[2.2.2]
J/mo/m^2.sec	1.436x10^{-9}	8.46 x10^{-9}	4.098 x10^{-9}	1.33 x10^{-8}
Per/mol/sec	1.436 x10^{-3}	8.46 x10^{-3}	4.098 x10^{-3}	1.33 x10^{-2}
K $_{b ex.}$	1.040 x10^{-4}	1.18 x10^{-4}	10. 98 x10^{-4}	10.84x10^{-4}
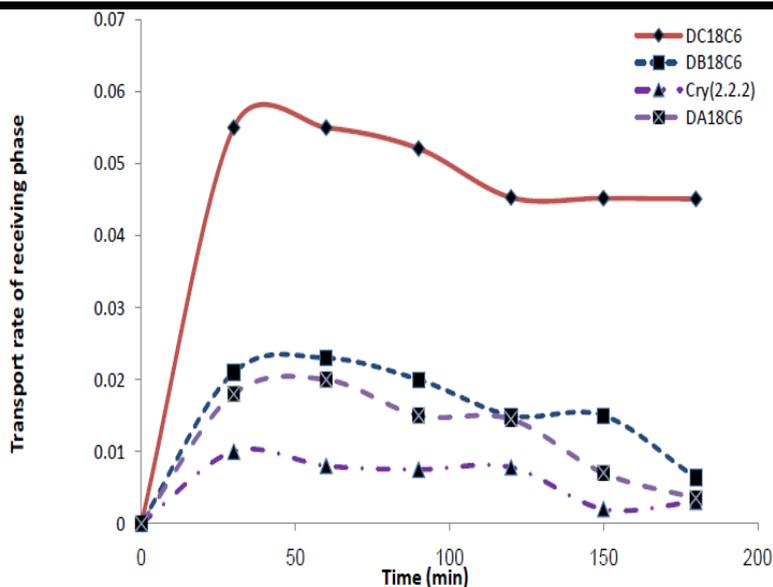
Fig 7 : Effect of carrier structure on the transport rate of receiving phase ReO$_4^-$ ion

3. 3-Effect of Solvent on perrhenate transport

Electrostatic model of anion transfers proofs to be useful in understanding ion solvation . The anion radius and charge are fundamental properties upon which selectivity can be based .The free energy of transfer of the anion from water to the non – polar solvent $\Delta G_{t\,r}$ is calculated by the method of Abraham and Liszi [19,20,21] . The solvation energy of anion is divided into an electrostatic term (ΔG_{el}) and an neutral term (ΔG_h) . The free enegry of transfer of the ion from water to the organic solvent is thus

$\Delta G_{t\,r} = \Delta G_{el} + \Delta G_n - \Delta G_h^{\circ}$

Where

ΔGh = hydration free energy of the ion

ΔG_n = neutral free energy of the ion.

ΔG_{el} = electrostatic free energy.

free energy of salvation can be expressed by the modified Born ؛s , equation as shown below [22] :

$$\Delta G\,sol = \frac{Ne^2}{2(1-\,1/\varepsilon)} \times \frac{1}{(r+ + R+)} + \frac{1}{(r- + R-)}$$

Where ε= is the dielectric constants

e= is the charge of ion

r_+ and r_-= are effective increments to a account for dielectric saturation, ion-solvent interaction ,etc.

According to the ionic radio of various anions together with selected M-0 bond distances d_{M-O} and free- energies of hydration[23] ,ΔG_h (M^+)since the concept the strength of anion hydration decreases as anion size increase as aresult , the removal of perrhanate was higher than that of sulfate and nitrate anions in spite of the high excess

of NO_3^- and SO_4^{2-} anions $ReO_4^- > NO_3^- > SO_4^{2-}$

For these transport conditions the selectivity coefficient for hydration energy of SO_4^{2-}, NO_3^- and ReO_4^- anions were -1103 KJ.mol^{-1}, -314KJ.mol^{-1} and -240 KJ. mol^{-1}, respectively [24, 25].

According to table(5,6) and fig(8,9,10) the permeability ,flux and transport rate increase in the dichloroethane thane chloroform and carbon tetrachloride

Table 5 :Effect of solvent   on flux and permeability of ReO_4^- ion in the source phase

Type of solvent	$ClCH_2CH_2Cl$	$CHCl_3$	CCl_4
J/mo/m².sec	3.92×10^{-7}	3.327×10^{-7}	2.105×10^{-7}
Per/mol/sec	3.92×10^{-2}	3.327×10^{-2}	2.105×10^{-1}
K $_{ex}$	1.700×10^{-3}	1.00×10^{-3}	0.98×10^{-3}

Table 6 : Effect of solvent on flux and permeability of ReO_4^- ion in the receiving phase

Type of solvent	$Cl-CH_2CH_2-Cl$	$CHCl_3$	CCl_4
J/mo/m².sec	2.423×10^{-9}	8.46×10^{-9}	7.527×10^{-10}
Per/mol/sec	2.423×10^{-3}	8.46×10^{-3}	7.527×10^{-4}
K $_{b\,ex.}$	1.18×10^{-4}	2.05×10^{-4}	2.15×10^{-4}
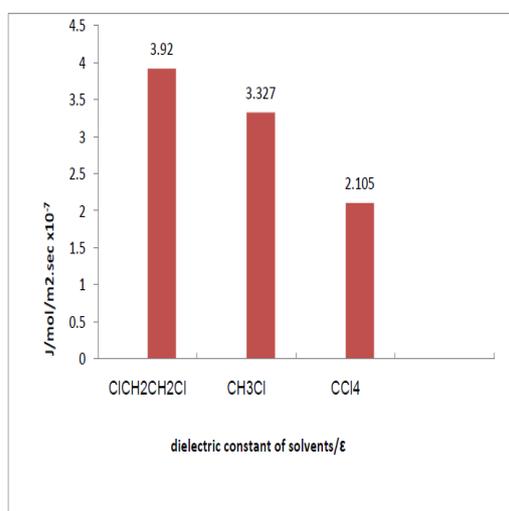
Fig 8 :Effect of Solvent on the flux of Re O $_4^-$ ion
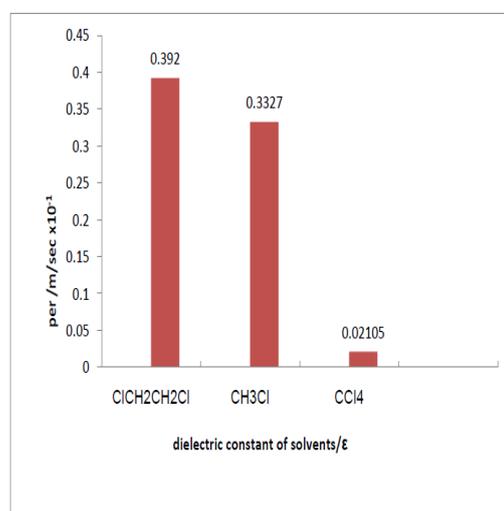
Fig 9 :Effect of Solvent on the permeabilit of Re O $_4^-$ ion
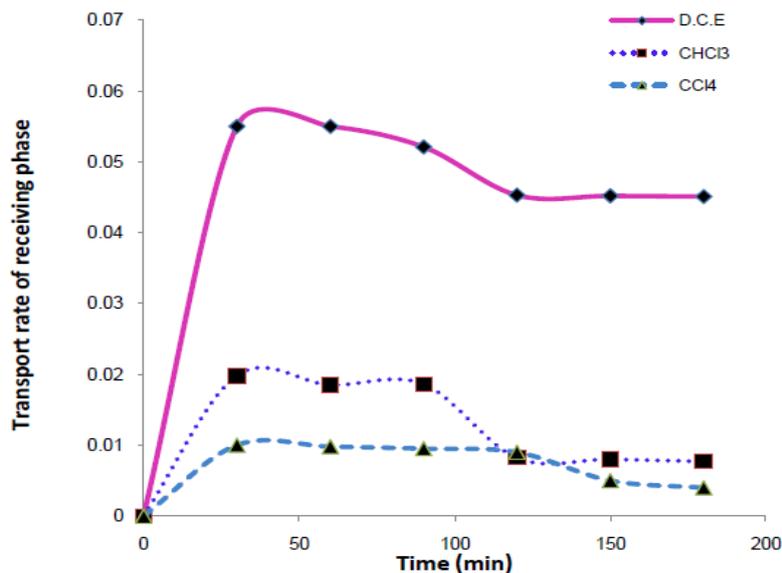
Fig 10 : Effect of Solvent on the transport rate of receiving phase ReO_4^- ion

3.1.4-Effect of temperature:

Transport behavior of ReO_4^- anion was studied at varying temperatures. The transport process suggests that with increasing temperature the diffusion is facilitated by the decreasing viscosity of the membrane phase which favors the mobility of the complex and carrier molecules ,according to permeability and flux parameters[26] in the table (7,8).The activation energy was given by plot of log K vs 1000/T[27] ,as shown in fig(14)

$$Log\ K = Log\ A - \frac{E_a}{R\,T}$$

Where K= rate constant
A= pre-exponential factor or frequency factor
E_a= Activation energy
R=gas constant
T= Temperature /K
The activation energy of ReO_4^- anion in the liquid membrane was(1.0584 Kcal/mol).
From fig (11),the calculated activation energy for ReO_4^- anion shows that the transport of the ReO_4^- anion is a diffusion-controlled process[28] which is assist in the fig(11,12,13).

Table 7 : Effect of temperature on flux and permeability of ReO_4^- in the source phase

Temperature/K	298	303	313
J/mo/m^2.sec	1.022×10^{-7}	9.889×10^{-8}	8.243×10^{-8}
Per/mol/sec	1.022×10^{-1}	9.889×10^{-2}	8.243×10^{-2}
K_{ex}	1.300×10^{-3}	1.700×10^{-3}	0.821×10^{-3}

Table 8 :Effect of temperature on flux and permeability of ReO_4^- ion in the receiving phase

Temperature/K	298	303	313
J/mo/m^2.sec	5.1302×10^{-9}	8.46×10^{-9}	6.37×10^{-10}
Per/mol/sec	5.1302×10^{-9}	8.46×10^{-3}	6.37×10^{-10}
$K_{b\,ex}$	1.10×10^{-4}	1.18×10^{-4}	1.02×10^{-4}
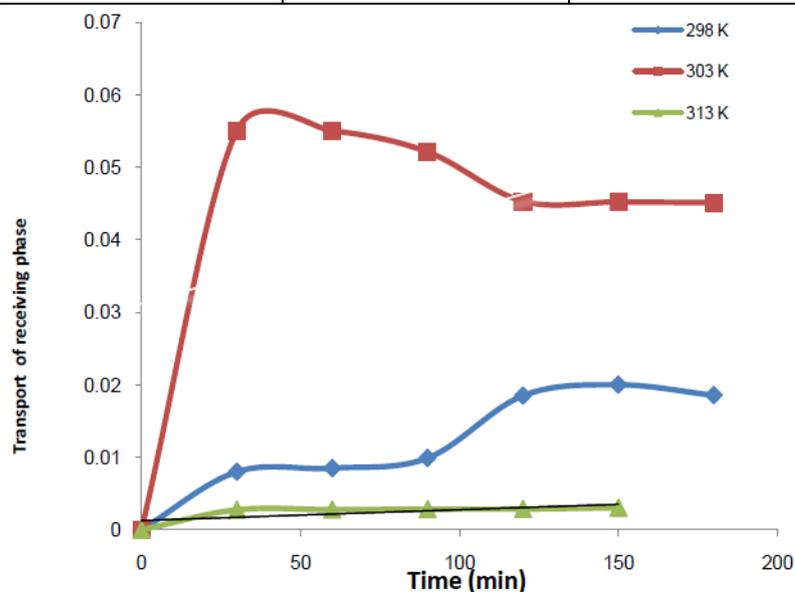
Fig 11: Effect of Temperature on the transport rate of receiving phase ReO_4^- ion
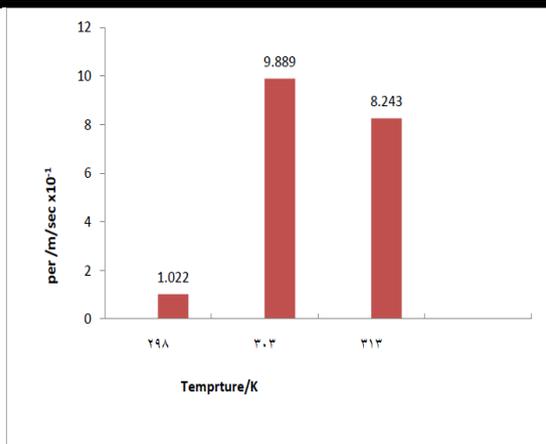
Fig 12: Effect of temperature on permeability of Re O$_4^-$
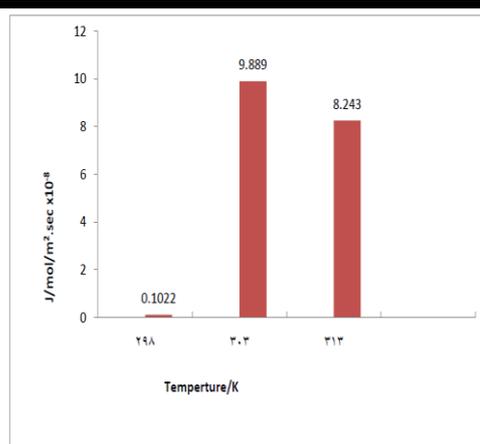
Fig 13: Effect of temperature on flux of Re O $_4^-$
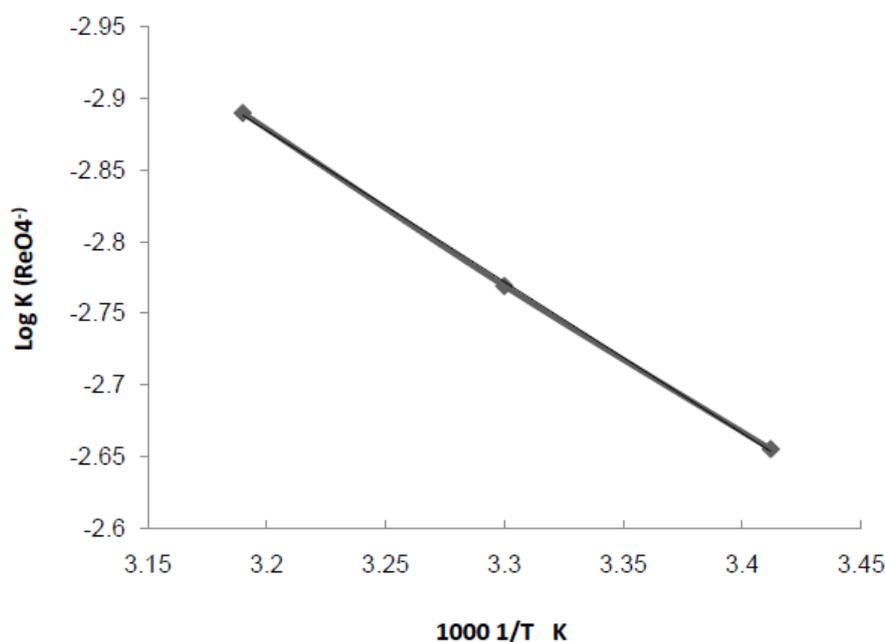
Fig 14: Effect of Temperature on Log K on of ReO$_4^-$ ion

3.2-Conclusion

The Separation of chemical species from a complex mixture is often an important process in industry. Liquid membrane separations system has been applied for the separation and removing of toxic and radioactive ions in which the extraction and stripping operations are combined in a single process**.** The stirring speed could not exceed 150 rpm because at higher stirring speeds the interface ripped up and hydrodynamic instabilities prevailed also, the phases are dispread the interfaces between donor and acceptor phases are increased.

1. DC18C6 bind to cations with faster kinetics and the resulting complex is thermodynamically less stable du to more flexibility exist, accordingly on the molecular conformation to bind cations by way of incontrast,the cryptand [2.2.2] bind to cations with slower kinetics, but the resulting complex is

thermodynamically more stable. The polarity of solvent influence the transport rate of salts through bulk liquid membrane containing crown ethers as carriers. Medium polar solvents are preferable to ensure the necessary supply of sufficient solvation energy .

The solubilities of inorganic salts also depened on type of anion involved . The solubility increases with an increasing diameter of anion. Generally , the inorganic salts having hard anion such as F^- and SO_4^- are barely solubilize, while the salt having soft anions such as I^- , SCN^-, NO_3^- , ReO_4^- and picrate are readly soluble in the organic solvents .

The temperature will effect on the transport and diffusion process by increase the rate constants of reactions and diffusion coefficient at the same decrease the extraction process by decreasing the K_{ex}.

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Abbreviation :
BLM : Bluk Liquid Membrane .
DB18C6 : Dibenzo-18-Crown-6
DCH18C6 : Dicyclohexyl-18-Crown-6 .
DA18C6 : Diaza-18-crown-6 .
Cry(2-2-2) : Kryptofix 2.2.2
K_{ex} : Extraction constant
$K_{b\,ex}$: Back extraction constant
rpm : run per minute .
Per. : Permeability
J   : Flux

*فصل ال4 ReO^- في الغشاء السائل بالمركبات العيانية

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مؤيد كاصد جلهوم   جميل موسى ضباب   يسرى عمران موسى•

1. شركة أبن سينا العامة
2. قسم الكيمياء /كلية العلوم/الجامعة المستنصرية
3. قسم الكيمياء/كلية التربية/جامعة القادسية

حاليا أستشاري في وزارة الصناعة والمعادن

الخلاصة

تم دراسة تجارب النقل عبر الغشاء السائل الضخم ، حيث تم تعين النقل التنافسي لايونات ReO_4^- خلال الغشاء السائل الضخم معثلاثة مركبات تاجية ثنائية بنزو-18-كراون-6 ، ثنائي سايكلو-18-كراون- 6 ، ثنائي أزا-18-كراون-6 ، كربتوفكس 2.2.2 , وقد درس تأثير سرعة التحريك، نوع الناقل، نوع المذيب على أنتقال أيونات ReO_4^- من الطور المصدر والمتضمن (0.001 مولاري من ReO_4^-+0.01 مولاري +0.01 مولاري من KOH) والمذاب في 10مل من الماء المقطر اللاأيوني الى الطور المستقبل والمتضمن 10مل من الماء المقطر اللاأيوني عند PH =3.2-3.7 عبر الطور العضوي والمتضمن 0.01 مولاري من الناقل المذاب في 40 مل من المذيب العضوي .لوحظ من النتائج أن قيم السماحية والجريان وسعة الانتقال تصبح أكثر قبولا مع ثنائي ـسايكلوهكسان18-كراون-6 كناقل و 2,1-ثنائي كلورو أيثان كمذيب عضوي و 100 دورة بالثانية سرعة تحريك في هذا النموذج.