

Separating a mixture of Re (VII), Mo (VI), V (V) and W (VI) ions using the thin layer electro-chromatography method on a thin layer of perlite

Yasin Moalla, Ibrahim Ibrahim*

Biomedical Engineering Faculty, AL-Andalus University, AL-Kadmous, Tartous, Syria

Email address:

i.ibrahim77@gmail.com (Ibrahim Ibrahim)

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Abstract: This paper presents a new method for separating Rhenium, Molybdenum, Vanadium and Tungsten ions on a thin layer of Perlite using the thin layer -electro-chromatography method. This method is based on the combination between chromatographic forces and electric forces by applying different voltages on the separation plate for different times using several electrolytes as different mobile phase concentrations, at different applying field durations on the process. This method also shows the possibility of using Perlite as a thin layer in this kind of separation process, which can apply in the separation of other types of biochemistry and inorganic ions This relatively simple method is cost effective.

Keywords: Thin layer Chromatography, Perlite, Thin layer Electro-Chromatography, Electrophoresis

1. Introduction

In recent years, Rhenium, Molybdenum, Vanadium, Tungsten and other rare elements have become more important as the constituent components of materials and products for the new technology. The growing interest in these elements is due to the presence of multiple important properties of both the elements and their compounds. The method of thin layer-electro-chromatography for physical and analytical chemistry is used more in attesting organic materials than inorganic materials. Increased interest in Rhenium, Molybdenum, Vanadium and Tungsten has spawned the idea to supplement the chemistry of these elements in the new research, especially in the field of thin layer chromatography (TLC), and thin layer electro-chromatography (TLEC).

1.1. Perlite

Perlite rocks expand in size during heat treatment of hydrous volcanic glasses the water content as well as the gases CO₂, N₂, H₂ are important for the swelling process. The quantitative correlation between gas and water for young volcanic glasses ranges between 1:1 to 1:3.[1].

In order for the gradual transition among the species of Perlite rocks to take place, it is advisable to establish the boundaries between them by volume weight. Perlite rocks have

a bulk density ranging between 1500 - 2200 kg / m³: the heavier refers to Obsidian, the lighter to Pumice.

These rocks are located in the following order from bottom to top: obsidian, perlite, pumice, and their natural porosity increases in the same sequence.

The average content of components of Perlite rocks is ordered in the following Percentages:

SiO₂ -72,9 %; Al₂O₃ -14,91 %; Fe₂O₃ -1,24 %; FeO -0,5 %;
TiO₂ -0,06 %; CaO -0,39% ;

MgO -0, 36 %; Na₂O +K₂O -7, 67%; MnO -0, 05%; P₂O₅ -0, 02%; others -1, 9%;

The slight difference in the various compositions of Perlite rocks corresponds to their genesis. Therefore, obsidian is characterized by high silica content - an average of 74.99%, slightly less dense perlite - 72.92 % and pumice (porous Perlite) - by even lower silica content - 71.29 %. In the geological section, Obsidian rocks are naturally on the bottom. Therefore, they are dense, durable, and can hardly absorb water.

The Perlite used in this research was brought from Aragats site in Armenia ready to use. When using electro-chromatography on a thin layer carrier the greatest value for the separation is the choice of a suitable electrolyte and the nature of the sorbent-carrier .Experiments were carried out using: a particulate carrier powder (expanded).

Thermo-treatment Perlite (fraction < 0.1 mm) on electrochromatographic unit. Plexiglas dimensions 80mm × 200mm. Bulk composition used in this study is shown in Table 1.

Table 1. Bulk composition of the Aragats perlite.

Oxide	Contentment%
SiO ₂	72,20
Al ₂ O ₃	13,03
Fe ₂ O ₃	0,58
FeO	0,35
MgO	0,11
CaO	0,81
Na ₂ O	4,65
K ₂ O	4,10
SO ₃	Traces

1.2. Thin Layer Electro-Chromatography

The term thin layer chromatography (TLC) was introduced by Consden, Gordon and Martin in 1946 [2]. They separated a mixture of amino acids and peptides on the silica layer whose thickness is 1.4 mm. Smithies developed electrophoresis on starch layers [3]. This method is used in biological research and in inorganic analysis [4-6]. Thin layers made from different sorbents: cellulose, nitrocellulose, alumina, silica gel, perlite [7], etc. Effectively use combination of TLC and thin-layer electrophoresis. With this kind of separation, we get a regular thin layer Chromatogram.

1.3. Electro-Chromatography Technique

Chromatographic methods of studying the state of ions in solutions are used in many studies [8, 9].

The electrophoresis on paper is a more reliable method and widely used for separating organic compounds or inorganic ions, and explains their charges [10]. Paper electrophoresis technique is presented in the study [11].

1.4. Thin Layer Electro-Chromatography Technique

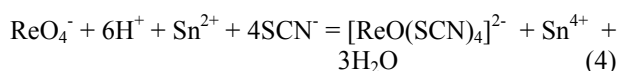
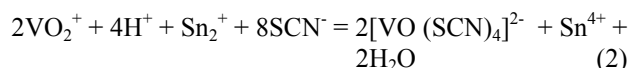
Two cells filled with specific concentration electrolytes are used where carbon electrodes are immersed. One end of chromatographic paper tape was dipped into a cell with a positively charged electrode; the other end was dipped into a negatively charged one. After that, 1-2 drops of the test solution have put at the middle (along the length) of tape saturated by electrolyte... The device is connected to a DC power source. Electrophoresis is performed within 1- 4 hours at a voltage of 50-600V.

The amperage in the tape depends on the size of the tape and concentration of the electrolyte. Upon completion of electrophoresis, the tape is left to dry at room temperature for 8 – 10 hours or in an oven at a temperature of 100-105 C⁰ for 10-20 minutes. The movement of ions becomes visible by naked eye using a certain developer.

1.5. Manifestation of Ions Reactions

Developing solution for Rhenium, Molybdenum, Vanadium and Tungsten is used as a saturated solution of SnCl₂ in 6M HCl. After spraying the plate with the developing solution,

further spraying with a saturated aqueous solution of ammonium thiocyanate. For these zones of the mentioned ions, and due to the formation of thiocyanate complexes compounds of Molybdenum, Vanadium, Tungsten, and Rhenium, are colored pink, yellow, yellow- green and orange respectively, according to the following reactions:



From the chromatogram, R_f (Retardation Factor) is calculated and evaluated in terms of the shape and the quantity of ions formed zones. To determine the concentration of the absorbed ions, the photometric method was applied.

2. Experimental

On glass plates of size 8, 0 cm × 18, 0 cm, a thin powder layer of Perlite 0.25 mm thickness was uniformly applied. To prepare a thin layer we mixed 4.0 g of the perlite powder with 10 ml heated distilled water and thoroughly stirred them to obtain a homogeneous substance. The plate is placed in the unit, and the edges of the carrier layer are immersed in the electrodes chambers. After the layer is saturated with electrolyte. The solutions ions under study were placed on the middle of the plate at the same distance of 1.5 cm from one another by a graded syringe. The device was covered with a transparent Perspex lid, and after that fed with a constant voltage to the electrodes. The applied solutions of the volume 4.0 μl contain 2.0 μg from ions: Rhenium (VII), Molybdenum (VI), Vanadium (V) and Tungsten (VI). For fixing the location of the analyzed ions after applying electro-chromatography, the plates are put at room temperature to dry.

To determine the optimal separation conditions for Rhenium (VII), Molybdenum (VI), Vanadium (V) and Tungsten (VI) ions, their electric chromatographic behavior was examined, i.e. the dependence of the mean free path of the ions on the nature and concentration of the electrolytes and duration of process. We measured the length of the path of the ions at a voltage of 300V, a layer thickness of 0.25 mm and duration of 120 min and more.

3. Results and Discussion

The data presented in Table 2, show that the amount of distance traversed by ions on the thin layer of perlite depends on the nature of the electrolyte. As electrolytes use solutions of hydrochloric acid, oxalic acids and potassium hydroxide with various concentrations.

Regardless of the nature and concentration of electrolytes, Rhenium (VII) is moved toward the anode, as in all cases, it is

in the form of anion as ReO_4^- , and in hydrochloric acid solutions with decreasing concentration of the acid, the traversed path for Rhenium (VII) ions is reduced.

In hydrochloric acid solutions with a concentration of 0.025 M, the ions of the studied elements are arranged in the following order in accordance with the increase of their traversed path: $V > Mo > Re > W$. the ion zones on electrochromatograms overlap and the possibility of their separation is limited.

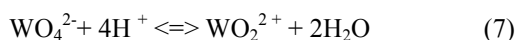
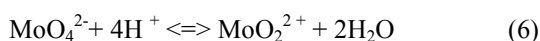
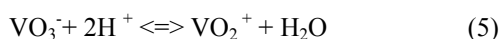
Table 2. Dependence of the traversed path of Rhenium, Molybdenum, Vanadium and Tungsten ions, of the nature and concentration of the electrolyte on a thin layer of Perlite 0.25 mm thickness.

Electrolytes	Concentration mole/l	Charges of ions and their traversed path, mm							
		Re(VII)	Mo(VI)	V(V)	W(VI)				
Hydrochloric Acid	0,025	-	3-27	-	10-37	-	20-45	-	5-22
	0,010	-	20-50	-	27-45	-	29-50	-	5-20
	0,005	-	0-25	+	5-20	+	20-47	+	3-17
	0,100	-	28-39	-	22-35	-	15-32	-	13-25
Oxalic acid	0.025	-	10-29	-	35-50	-	30-55	-	5-15
	00,01	-	40-60	-	30-45	-	28-43	-	0-15
	0,0100	-	25-42	-	20-35	-	25-45	-	0-15
Potassium Hydroxide	0,0050	-	45-55	-	45-55	-	50-65	-	48-64
	0,0025	-	35-45	-	30-42	-	25-35	-	0-10

Table 3. Dependence of the traversed path ions on the duration of the process.

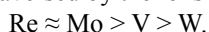
Electrolyte	concentration mole/l	Process duration, min	traversed ions path, mm			
			Re(VII)	Mo(VI)	V(V)	W(VI)
HCl	0,01	120	40-50	20-45	20-30	5-20
		180	40-60	25-40	28-43	10-25
		360	40-70	25-45	35-60	10-30
KOH	0,0025	120	35-45	30-42	25-35	0
		180	50-60	32-45	26-38	0
		240	50-70	45-57	30-40	0

Vanadium and Tungsten ions move to the cathode due to their transition from anionic to cationic forms (reactions 5-7):



In the oxalic acid solution with a concentration of 0.10 - 0.025 M Molybdenum (VI), Vanadium (V) and Tungsten (VI) ions form anionic complexes (12, 13, 14), and move toward the anode. The study of Complexes partly facilitates the separation of ions. As a result, when using a qualitative electrolyte potassium hydroxide solution with a concentration of 0.01 M, with respect to the path traversed by the ions, the value is placed on the chromatogram as follows: $V \approx Re > Mo > W$ (see table 2).

However, with a concentration of 0,005 M, value of the path traversed by the ions placed next follow: $V \approx W > Re \approx Mo$, and with a concentration of 0,0025 M, value of the path traversed by the ions placed next follow:



The data in table.2 show that the possibility of separating a mixture of ions is limited. Diluting the alkaline solution increases the partial path of the ions due to a decrease in their

activity. For more diluted solutions of hydrochloric acid (in particular, at a concentration of 0.01 M), the quantity of the traversed path by the ions of Rhenium, Molybdenum and Vanadium increase, and Tungsten remains unchanged, and the ions of all four elements are moved toward the anode. Under these conditions, only the Tungsten ions can be separated from the ions of other elements. With a further decrease in the concentration of hydrochloric acid ($\leq 0,005$ M), the running of all four ions decrease, and Molybdenum,

activity.

Table 3, shows the dependence of the path traversed by the ions of the duration of the process when using electrolytes of hydrochloric acid with concentration of 0,01 M and potassium hydroxide with concentration of 0,0025 M.

With increasing duration of the process for hydrochloric acid from 120-360 minutes, the value of the path traversed by the ions increase.

The obtained data allow the choice of the conditions of the separation of two, three and four component mixtures studied elements. The data presented in Table 3, show that in alkaline solutions of potassium hydroxide all elements are in the anionic state. According to the mobility degree, the elemental ions are arranged as follows:

$Re > Mo > V > W$. With an increasing duration of electrochromatography for potassium hydroxide from 120 - 240 minutes, the value of the traversed path by ions increase regularly except Tungsten ions, which do not move in all cases, which improves the conditions of their separation.

4. Conclusion

The study of electro-chromatographic behavior of rare elements ions, such as Rhenium (VII), Molybdenum (VI), Vanadium (V) and Tungsten (VI) on thin layers of Perlite has

been established. According to the nature and concentration of electrolytes and process duration, regardless of the nature and concentration of electrolytes, the ions in the electric field always move to the anode. Electro migration path decreases with dilution of electrolytes. In solutions of hydrochloric acid at a concentration of 0.005 M ions of Molybdenum, Vanadium and Tungsten transferred from anionic to cationic form and move to the cathode.

In solutions of oxalic acid, all of the oxalic acid ions form anionic complexes and migrate towards the anode. In alkaline solutions, the following locations according to ions mobility's $V(V) \approx Re(VII) > Mo(VI) > W(VI)$ are observed. It is clear by this method the effect of the concentration and type of used electrolytes on the efficiency of the separation process.

It is obvious that the thin layers of perlite can be successfully separated into two-, three - and four- component mixtures of ions.

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