

Study of Effect of Adsorbants on Separation of Toxic Metal Ions by Thin Layer Chromatography

Gayatri Barabde

Department of chemistry Institute of Science Mumbai (India)

Abstract: Different stationary phases were used to obtain effective separation of Cr (VI), Cr(III), W(VI), V(V), and Mo(VI) from their three, four component mixtures by Thin layer Chromatography. The separations were carried out on Silica gel G, Kieselger G, cellulose, Mixture of Silica gel G and cellulose using aqueous solution of sodium acetate as Mobile phase. Of all above adsorbants, silica gel G was found to be most suitable adsorbant. The effect of concentration and pH of mobile phase on the Rf values of individual metal ions were studied and the optimum conditions for separation of metal ions from their mixture were established. Upto mixtures with four components could be effectively separated on silica gel G as adsorbent with sodium acetate as mobile phase.

Key words: Sodium acetate, Thin layer chromatography, Kieselger G, cellulose

I. Introduction:

The market for imitation materials has grown rapidly in last decade. To make them look attractive chrome plating became essential. Chrome plating is also done to prevent metal from rusting. Western suburbs of Mumbai are host for number of chrome plating industry. These small industries use chromium in the form of Cr(VI) for plating. After plating the effluent is thrown in gutters by qualitatively treating it with reducing agents. There are chances that Cr (VI) is not completely converted into Cr (III). To separate these two states of chromium was done using Flame atomic absorption spectrophotometer using flow injection on-line preconcentration with selective adsorption on activated alumina from water sample.¹ Later many researchers worked on separation of these two ions from water sample by FAAS.^{2,3} Ion Exchange Resin Can also effectively used for separation and preconcentration of these two ions ⁴. A study has been done to ascertain the analytical capabilities of combined HPLC-ICP-AES with ultrasonic nebulization or HPLC-ICP-MS systems to perform on-line separation of Cr(III)-Cr(IV) and on-line preconcentration of Cr(VI)⁵. Some workers used activated carbon prepared from coconut tree sawdust for removal Cr(VI)⁶. The separation of Cr (VI) from Cr(III) is successfully done by many hyphaneted techniques and also simple techniques like FAAS and ion exchange but there is still need to develop a rapid and economical method for separation. Thin layer chromatography is the rapid method also can be performed with minimum apparatus.

II. Materials and Methods

Apparatus

Glass plates of 4 x 20 cm size. Glass jars with lid for the development of glass plates.
Glass sprayer for spraying reagents
pH meter.

Chemicals And Reagents:

Silica Gel G, Kieselger G, Cellulose, Hydrochloric acid, Sodium hydroxide, Fumaric acid and Maleic acid (AR grade)

Stock Solutions:

The stock solutions were prepared of following salts.

- 1) Potassium salt of Cr(VI)
- 2) Chloride of Cr(III)
- 3) Oxides of V (V)
- 4) W (VI)
- 5) Ammonium Molybdate

Detection Reagents:

To detect the above cations following reagents were used

- 1) 1% Aq. Potassium thiocyanate
- 2) 1% tannic acid in Acetic acid

- 3) 0.02% dithiozone in carbon tetrachloride
- 4) 30% Hydrogen peroxide

Procedure:

1) Preparation of plates:

Since different coating materials were to be used, the plates were prepared in laboratory. Slurry of Silica gel G, Kieselger G, Cellulose and 1:1 mixture of Silica gel and cellulose were prepared using double distilled water. Each slurry was kept in Sonicator for smooth and uniform slurry formation. It was then immediately applied to the glass plates by the dipping method and dried overnight at room temperature..

2) Application of Sample and Running the plate:

The test solutions were spotted on overnight dried plates using fine capillary. The spots were dried using a drier. The Sodium Acetate was adjusted at require pH using sodium hydroxide and Hydrochloric acid. The plates were run for 5,10,15 and 20 min to conclude the best plate development time.

3) Development of plate:

The plates were removed from jar dried and spots were located by using different spraying reagents. All experiments were carried out at room temperature. The Rf values were measured in triplicate for each measurement.

III. Result And Discussion:

The various experiments were carried out in triplicate to study the change in Rf value using Sodium Acetate as Mobile phase.. To decide upon the best suitable stationary phase and experimental conditions the different concentrations of Sodium acetate were prepared from 0.01M to 0.2 M. Plates with stationary phase such as Silica gel G, Kieselger G, Cellulose and 1:1 mixture of Silica gel and cellulose were used. Also to fix the time the chromatogram was run from 5 min to 20 min.

Effect Of Ph On Migration Of Ions:

A Proper adsorbent is a one which provides different Rf values for different metal ions. So to make a proper selection, the chromatograms were run with individual metal ions using different adsorbents such as Silica gel G, Kieselger G, cellulose, Mixture of Silica gel G and cellulose. Sodium acetate was used as mobile phase of concentration 0.1 M. Fig 1 shows migration of ions on cellulose and silica gel G mixture as an adsorbant. It was observed that Cr(VI) and V(V) travel together but rest of the ions showed different Rf values at PH 2. While in Fig 2 where pure silica gel was used as adsorbent Cr(VI) and Mo(VI) showed similar Rf values.but others showed different Rf values at PH 2. In case of Silica Gel G the spots were compact. Kieselger G showed separation of Cr (VI) and Cr(III) at PH 2 but Mo(VI) and V(V) tailed at that PH. While in Cellulose as adsorbant W (VI) was never gave compact spot and Cr (VI) and Mo(VI) showed same Rf values but Cr(VI) and Cr(III) can be separated successfully.

Fig no.1

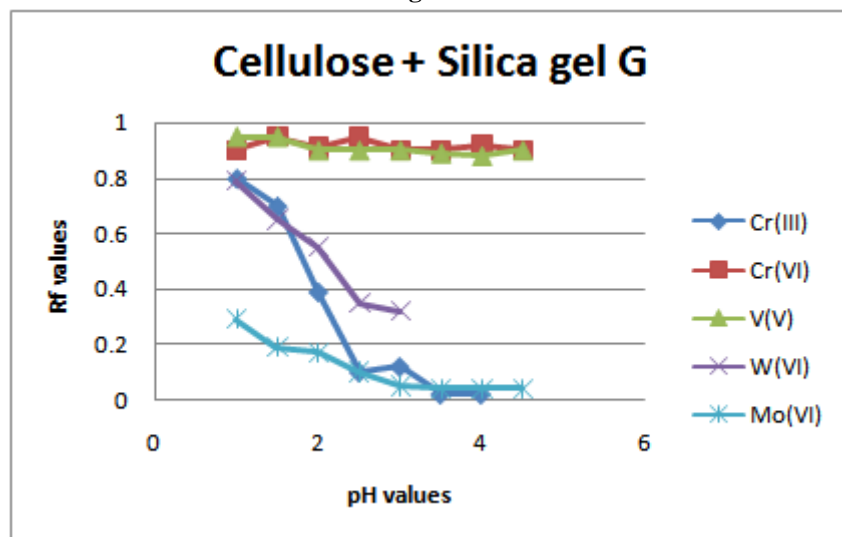
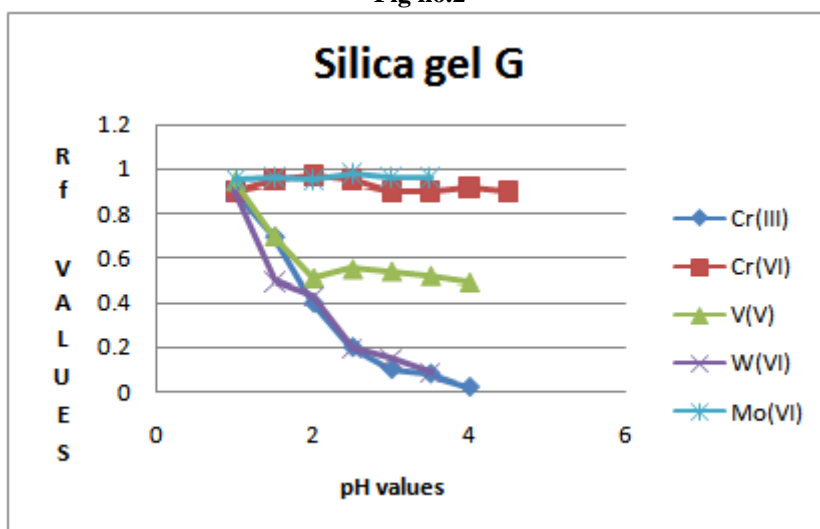


Fig no.2



Choice Of Proper Adsorbant:

The study of effect of PH on migration of ions showed that PH 2 was a recommended PH for separation as Cr (VI) and Cr (III) separated at that PH on all stationary phases. Keeping that PH constant and fixing development time at 10min chromatogram were run. Table no.1 shows results obtained. When mixture of silica gel G and Cellulose were used as adsorbants ternary to even quaternary mixture can be separated. While in case of silica gel G only ternary mixtures can be perfectly separated. While in case of Kiselger G and Cellulose with sodium acetate as mobile phase even though Cr (VI) and Cr (III) can be separated successfully for rest of ions these two adsorbants cannot be used.

Table no.1

Variation in Rf values with different adsorbents				
SOLVENT : Sodium Acetate		pH : 2		Development time : 10 min
METAL IONS	ADSORBANTS			
	Rf values			
	Silica gel G	Kiselger G	Cellulose	Silica gel and cellulose
Cr(VI)	0.97	0.92	0.91	0.95
Cr(III)	0.36	0.36	0.39	0.34
Mo(VI)	0.88	0.95(T)	0.72	0.12
W(VI)	0.40	0.39	-	0.55
V(VI)	0.42	0.6(T)	0.90	0.96

Effect of Development time on migration of ions

After initial study Silica gel G was excepted as the best adsorbant for separation as far as this study was concerned. So keeping Silica gel G constant all the metal ions were run using sodium acetate as mobile phase with different development time. Table no.3 depicts the results obtained. This task was taken up to decide on minimum time required for development. When development time was raised from 5 to 10 min the ions showed little change in Rf values but spots were compact and well defined but as the time increases Mo(VI) and V(V) showed tailing hence development time was fixed at 10 min.

Table no: 2

Variation in Rf values with development time				
SOLVENT : sodium acetate		pH : 2		Adsorbant : Silics gel G
METAL IONS	Development time in min			
	Rf values			
	5	10	15	20
)	0.96	0.97	0.98	0.96
Cr(III)	0.34	0.36	0.32	0.41
Mo(VI)	0.87	0.88	0.73T	0.74T
W(VI)	0.36	0.40	0.31	0.40
V(VI)	0.44	0.42	0.58T	0.63T

IV. Conclusion

After extensive study to identify best adsorbant to separate Cr(VI) and Cr(III) in a mixture along with other ions found in chrome plating industry effluent was silica gel G. From all the adsorbants used in the study showed separation of Cr(VI) and Cr(III) in a mixture but separation of these two ions from other ion found in effluent was not that effective. When the mixture of silica gel G and Cellulose was used as adsorbant quaternary mixtures could be separated.

Few representative mixtures that were separated are shown below

Mixtures that were separated with 0.1 M sodium acetate at pH 2 and development time 10 min with silica gel G as stationary Phase.

- 1) Cr⁺⁶(0.97) Cr⁺³(0.37)V⁺⁵(0.51)
- 2) Mo⁺⁶(0.90) Cr⁺³(0.41)V⁺⁵(0.51)
- 3) Cr⁺⁶(0.92) W⁺⁶(0.47)
- 4) Cr⁺⁶(0.92) Cr⁺³(0.37)
- 5) Mo⁺⁶(0.99) Cr⁺³(0.37)
- 6) Mo⁺⁶(0.99) W⁺⁶(0.47)

Mixtures that were separated with 0.1 M sodium acetate at pH 2 and development time 10 min with mixture of silica gel G and Cellulose as stationary Phase.

- 1) Cr⁺⁶(0.98) Cr⁺³(0.41) Mo⁺⁶(0.12)
- 2) V⁺⁵(0.89) Cr⁺³(0.40) Mo⁺⁶(0.12)
- 3) Cr⁺⁶(0.98) W⁺⁶(0.47) Mo⁺⁶(0.99)
- 4) W⁺⁶(0.47) Mo⁺⁶(0.99) V⁺⁵(0.89)

Many more combination of mixtures can be separated.

Acknowledgement

Dr.R.B.Kharat Former Director Institute of Science Nagpur for his guidance and support.

References

- [1]. "Determination of Cr(III) and Cr(VI) in water using flow injection on – line preconcentration with selective adsorption on activated Alumina and Flame absorption spectrometric detection." Michel Sberling, Shukun Xu and Bernhard Wela, Anal Chem 1992, 64(24),PP3101-3108
- [2]. Separation of Cr(III) and Cr(VI) with Saccharomy Cerevisiae immobilized in water by FAAS, Elsevier, vol 51, issue 5, 28 april 2000, Pages 896-902
- [3]. Separation of Cr(III) and Cr(VI) using melamine-formaldehyde resin and determination of both species in water by FAAS.I Tor,H. Filik,H.Afsar, Fresenius Journal of Analytical Chemistry, Nov.1996, Vol 356, issue 6, PP 375-377.
- [4]. Rapid ion-exchange technique for the separation and preconcentration of chromium(VI) and chromium(III) in fresh waters, [C.Annette Johnson](#), Analytica Chimica Acta, Elsevier, Vol 238, 1990, PP 273-278.
- [5]. On-line separation and preconcentration of chromium species in seawater, J. Posta, A.Alimoth, F. Petrucci, S.Coroli, Analytica Chimica Acta, Vol 325, issue 3, 30 May 96 PP 185-193
- [6]. Removal of Cr(VI) from aqueous solution by adsorption onto activated carbon, K.Selvi, S Pattabhi, K.Kadivelu, Biosource technology ,Elsevier, Vol 80, issue 1, Oct 2001, PP 87-89
- [7]. Determination of Cr(III) and Cr(VI) using suppressed ion chromatography inductively coupled plasma mass spectrometry., Journal of Analytical Atomic Spectrometry 2001,16, PP 926-930