

# COMPARATIVE CHROMATOGRAPHIC STUDY OF SILICA GEL-G & UREA FORMALDEHYDE AS AN ADSORBENTS FOR THE SEPARATION OF SOME TOXIC METAL CATIONS BY THIN LAYER CHROMATOGRAPHY

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## Abstract :

*Different stationary phases were used to obtained effective separation of  $Cr^{6+}$ ,  $Fe^{3+}$ ,  $Cu^{2+}$ ,  $U^{6+}$ ,  $V^{5+}$ ,  $Ni^{2+}$ ,  $Co^{2+}$  and  $Ag^+$  from their three and four component mixtures by thin layer chromatography. The separations were carried out on Silica gel G, Urea Formaldehyde polymer, mixture of Urea Formaldehyde & Silica gel-G using aqueous solutions of cationic and anionic surfactants as mobile phase. Of all above adsorbent, silica gel-G and urea formaldehyde were found to be most suitable adsorbents. The effect of concentration and pH of mobile phase on the  $R_f$  values of individual metal ions were studied and optimum conditions for separation of metal ions from their mixture were established. Up to mixtures of four components could be effectively separated by anionic surfactant( Sodium Dodecyl Sulphate) as mobile phase while only ternary mixtures could be separated by cationic surfactants (Benz Alkonium Chloride) as a mobile phase.*

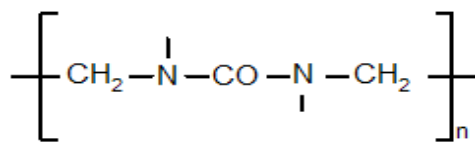
**Keywords :** SDS, BAC, Thin Layer Chromatography.

## Introduction :

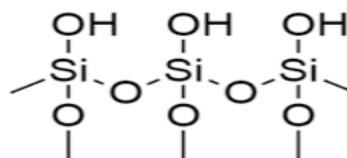
Due to industrialization, there is an increased usage of metallic substances. Industrial waste is the major source of different kinds of metal pollution in aquatic systems. The major sources of pollution of toxic metals are electroplating stainless steel industries, metal finishing industrial effluents, sewage and waste-water treatment plants discharge and chromates from cooling water [1]. A lot of organic and inorganic solvents have been used by different researchers. Even amino acid like Valine, L-IsoLeucine etc have been used for separation of toxic metal ions[1,2]. Some workers have tried to study difference in the migration of ions using impregnated and non impregnated silica gel-G using formic acid [4]. Aqueous solution of Oxalic acid as a solvent was used with silica gel G to develop rapid method of separation [3]. Ammonia solutions and amino acids was successfully used to separate heavy toxic metal cation[5] & [7], Carbamid-Formaldehyde polymer used as thin



layer for chromatographic separation[6]. There was need to study the effect on mobility on different adsorbent like urea formaldehyde and starch to enhance the method to separate mixture of toxic metal ions. The present study involves use of silica gel G, laboratory prepared urea formaldehyde polymer, starch, mixture of silica gel G and urea formaldehyde polymer and mixture of urea formaldehyde polymer and starch as an adsorbent to observe their effect on mobility variation on separation of metal cations comparatively.



Urea formaldehyde



Silica Gel

### Experimental :

#### Apparatus :

Glass plates , Glass jars with lid for the development of glass plates, Glass sprayer for spraying reagents, pH meter.

#### Reagents :

Urea , formaldehyde solution, silica gel-G, starch, SDS(Sodium Dodecyl Sulfate) and BAC(Benzalkonium Chloride) were obtained from Merck. Dimethylglyoxime, dithiozone, potassium ferrocynide, silver nitrate and ethanol were obtained from SD Fine (India). All other chemicals were of analytical grade.

#### Stock solutions :

TLC was performed using standard 1% aqueous solution of chloride, nitrate, potassium or sulphates of metal ions listed.

#### Detection :

Fe<sup>3+</sup>, Cu<sup>2+</sup>,U<sup>6+</sup>,V<sup>5+</sup> were detected using 1% aqueous potassium ferrocynide, Co<sup>2+</sup> & Ni<sup>2+</sup> were detected using a 1% solution of alcoholic dimethyloxime , Ag<sup>+</sup> was detected by 0.5% of dithizone in carbon tetrachloride and Cr<sup>6+</sup> was detected by using saturated solution of alcoholic silver nitrate.

#### Procedure :

##### 1. Preparation of plates :

Since different coating materials were to be used, the plates were prepared in laboratory. Slurry of Silica gel G, laboratory prepared urea formaldehyde polymer and mixture of silica gel G and urea formaldehyde 1:1 ratio were prepared using double distilled water . 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 SDS and BAC in different concentration were adjusted at require pH using sodium hydroxide and Hydrochloric acid. The plates were run for 2, 4 and 5 minuet 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  $R_f$  values were measured in triplicate for each measurement.

## **Result and discussion :**

The various experiments were carried out to study the change in  $R_f$  value of metal cation using anionic surfactant (SDS) and cationic surfactants(BAC). To decide upon the best suitable stationary phase and experimental conditions the different concentrations of both surfactants were prepared from 1% solution to 5% solution . Plates with stationary phase such as Silica gel G, Urea Formaldehyde, 1:1 mixture of Silica gel and Urea Formaldehyde were used. Also to fix the time the chromatogram was run from 2 min to 5 min.

## **Choice of Proper Adsorbents :**

The chromatogram of individual metal ions using different adsorbents i.e. Silica gel G, Urea formaldehyde , Starch ,1:1 mixture of Silica gel and Urea Formaldehyde and 1:1 mixture of Urea Formaldehyde were run. The conditions for running these chromatograms were arbitrarily fixed. The results obtained are tabulated in table no.1 and 2. Table 1 and 2 revels that most of the metal ions can be separated effectively on silica gel G, urea formaldehyde and mixture of urea formaldehyde and silica gel-G using surfactants. With urea formaldehyde as an adsorbent separation of metal ions in ternary and quaternary mixtures can be achieved in 5 min. When mixed adsorbents were used it was observed that most of the metal ions travel near solvent front. Using mixture of urea formaldehyde with silica gel-G as an adsorbent many ternary and quaternary mixtures can be separated using both the surfactants. From the results obtained from Table 1 and 2 it was concluded that urea formaldehyde is an alternative adsorbent in place of silica gel-G for the separation of metal ions in aqueous solution of surfactant media because many ternary and quaternary mixtures can be separated on urea formaldehyde polymer and the spots obtained were quite compact and sharp.



**Table 1**

<b>Variation in R<sub>f</sub> values with adsorbents</b>			
<b>Solvent: 3% SDS</b>		<b>pH=3</b>	
<b>Time= 3 Minutes</b>		<b>Development</b>	
<b>Metal ions</b>	<b>Adsorbents</b>		
	<b>R<sub>f</sub> values</b>		
	<b>Silica gel-G</b>	<b>Urea Formaldehyde</b>	<b>1:1 Mixture of Silica gel-G and Urea Formaldehyde</b>
Fe <sup>3+</sup>	0.88	0.10	0.15
Cu <sup>2+</sup>	0.65	0.46	0.50
U <sup>6+</sup>	0.59	0.28	0.30
V <sup>5+</sup>	0.89	0.78	0.80
Co <sup>2+</sup>	0.80	0.72	0.76
Ni <sup>2+</sup>	0.87	0.90	0.93
Ag <sup>+</sup>	0.90 <sup>b</sup>	0.090 <sup>b</sup>	0.093 <sup>b</sup>
Cr <sup>6+</sup>	0.56 <sup>c</sup>	0.59 <sup>c</sup>	0.62 <sup>c</sup>

**Table 2**

<b>Variation in R<sub>f</sub> values with adsorbents</b>			
<b>Solvent: 3% BAC</b>		<b>pH=3</b>	
		<b>Development Time= 3 Minuets</b>	
<b>Metal ions</b>	<b>Adsorbents</b>		
	<b>R<sub>f</sub> values</b>		
	<b>Silica gel-G</b>	<b>Urea Formaldehyde</b>	<b>1:1 Mixture of Silica gel-G and Urea Formaldehyde</b>
Fe <sup>3+</sup>	0.86	0.16	0.13
Cu <sup>2+</sup>	0.59	0.76	0.71
U <sup>6+</sup>	0.48	0.22	0.20
V <sup>5+</sup>	0.90	0.96	0.75
Co <sup>2+</sup>	0.88	0.82	0.76
Ni <sup>2+</sup>	0.88	0.90	0.89
Ag <sup>+</sup>	0.90 <sup>b</sup>	0.92 <sup>b</sup>	0.65
Cr <sup>6+</sup>	0.50 <sup>c</sup>	0.56 <sup>c</sup>	0.62 <sup>c</sup>

<sup>a</sup> Detection clarity is poor.

<sup>b</sup> Tailed spot.

<sup>c</sup> Double spot.

**Effect of development time :**

To study the effect of development time on R<sub>f</sub> values number of experiments performed and the results obtained are given in table 3 and 4. The R<sub>f</sub> values were measured at



pH=3 and concentration 3%. When migration time was restricted to 3 min in case of both surfactants only binary mixtures could be effectively separated. As the time was increased to 5 min up to quaternary mixtures could be separated. Further increase in time did not show much effect on  $R_f$  values. Hence migration time was fixed at 5 min.

**Table 3**

Variation in $R_f$ values with adsorbents				
Solvent: 3% SDS		pH=3		Adsorbent: Urea Formaldehyde Polymer
Metal ions	Development Time in Minuet			
	$R_f$ values			
	3	5	10	15
Fe <sup>3+</sup>	0.10	0.11	0.11	0.10
Cu <sup>2+</sup>	0.46	0.48	0.45	0.46
U <sup>6+</sup>	0.28	0.30	0.30	0.28
V <sup>5+</sup>	0.78	0.78	0.78	0.78
Co <sup>2+</sup>	0.72	0.75	0.76	0.76
Ni <sup>2+</sup>	0.90	0.90	0.91	0.90
Ag <sup>+</sup>	0.090 <sup>b</sup>	0.093	0.090	0.090
Cr <sup>6+</sup>	0.59 <sup>c</sup>	0.54	0.55	0.55

**Table 4**

Variation in $R_f$ values with adsorbents				
Solvent: 3% BAC		pH=3		Adsorbent: Urea Formaldehyde Polymer
Metal ions	Development Time in Minuet			
	$R_f$ values			
	3	5	10	15
Fe <sup>3+</sup>	0.16	0.15	0.15	0.16
Cu <sup>2+</sup>	0.76	0.72	0.73	0.72
U <sup>6+</sup>	0.22	0.20	0.20	0.20
V <sup>5+</sup>	0.96	0.90	0.90	0.91
Co <sup>2+</sup>	0.82	0.82	0.83	0.82
Ni <sup>2+</sup>	0.90	0.92	0.90	0.90
Ag <sup>+</sup>	0.92 <sup>b</sup>	0.90	0.91	0.88
Cr <sup>6+</sup>	0.56 <sup>c</sup>	0.52	0.52	0.53

<sup>a</sup> Detection clarity is poor.

<sup>b</sup> Tailed spot.

<sup>c</sup> Double spot.

**Effect of pH of aqueous solutions of cationic and anionic surfactants on migration of ions :**

Chromatograms were run using aqueous solutions of cationic and anionic surfactants of different pH. The graphs were plotted to clearly show the effect of pH of solvent on



migration of metal ions.

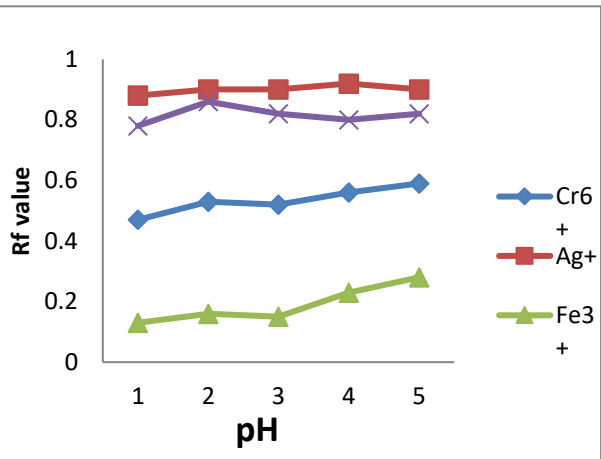
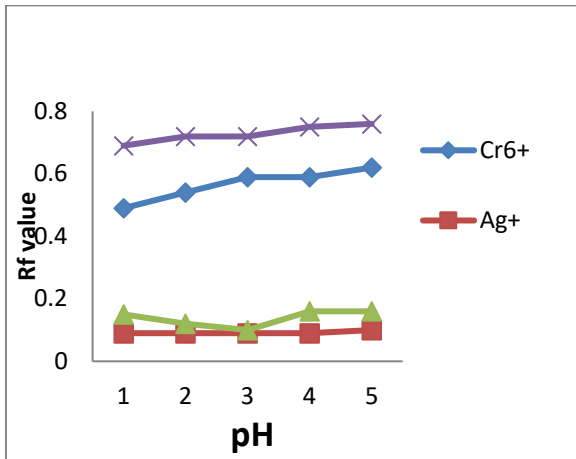


Figure 2: Separation of metal ions with 3% SDS

Figure 3: Separation of metal ions with 3% SDS

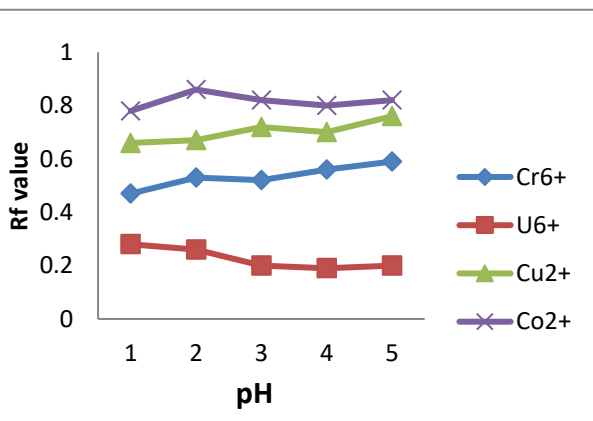
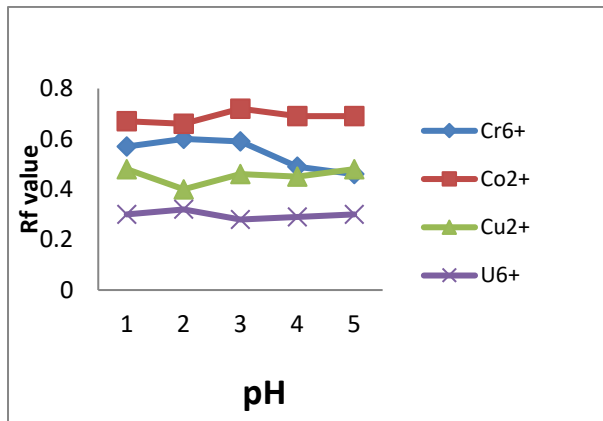


Figure 4: Separation of metal ions with 3% BAC

Figure 5 : Separation of metal ions with 3% BAC

The above graphs clearly indicate when individual metal ions were developed using urea formaldehyde polymer as an adsorbent and development time was maintained as 5 min. At pH=3 with aqueous solutions of SDS -and BAC ternary and quaternary mixtures can be separated.

**Conclusion :**

The entire data represented above clearly indicate that even though SDS and BAC are surfactants behaves entirely different. Of all stationary phases used silica gel-G and urea formaldehyde were found to be most suitable. The efforts were taken to minimize the development time and 5 min development time was found to be sufficient to develop chromatogram. Finally urea formaldehyde and silica gel – G shows better separations than rest of the adsorbents. Few representative mixtures are that were separated are shown below.

Mixtures that were separated with 3% SDS (Anionic surfactants) at pH=3 and development time 5 min with urea formaldehyde as stationary phase.



1.  $\text{Cr}^{6+}(0.58)$ ;  $\text{Ni}^{2+}(0.89)$ ;  $\text{Fe}^{3+}(0.11)$ ;  $\text{V}^{5+}(0.76)$ .
2.  $\text{Cr}^{6+}(0.54)$ ;  $\text{Ag}^{+}(0.091)$ ;  $\text{Cu}^{2+}(0.47)$ ;  $\text{Co}^{2+}(0.76)$ .
3.  $\text{Cr}^{6+}(0.54)$ ;  $\text{U}^{6+}(0.29)$ ;  $\text{Fe}^{3+}(0.10)$ ;  $\text{Co}^{2+}(0.74)$ .
4.  $\text{Co}^{2+}(0.72)$ ;  $\text{U}^{6+}(0.22)$ ;  $\text{Ag}^{+}(0.096)$ ;  $\text{Fe}^{3+}(0.12)$ .
5.  $\text{Cr}^{6+}(0.52)$ ;  $\text{Co}^{2+}(0.70)$ ;  $\text{Cu}^{2+}(0.46)$ ;  $\text{V}^{5+}(0.79)$ .

Mixtures that were separated with 3% BAC (Cationic surfactants) at pH=3 and development time 5 min with urea formaldehyde as stationary phase.

1.  $\text{Ag}^{+}(0.92)$ ;  $\text{U}^{6+}(0.26)$ ;  $\text{Cu}^{2+}(0.72)$ ;  $\text{Fe}^{3+}(0.18)$ .
2.  $\text{Cr}^{6+}(0.59)$ ;  $\text{Ni}^{2+}(0.88)$ ;  $\text{Co}^{2+}(0.80)$ ;  $\text{V}^{5+}(0.96)$ .
3.  $\text{Cr}^{6+}(0.62)$ ;  $\text{Ag}^{+}(0.94)$ ;  $\text{U}^{6+}(0.22)$ ;  $\text{Co}^{2+}(0.81)$ .
4.  $\text{Ni}^{2+}(0.90)$ ;  $\text{Fe}^{3+}(0.13)$ ;  $\text{Cr}^{6+}(0.58)$ ;  $\text{Cu}^{2+}(0.75)$ .

Many more combination of mixtures can be separated.

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