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Separation and identification study of different metal ions by thin layer chromatography using mixture of urea – formaldehyde polymer and silica gel-G as thin layer and surfactants, organic solvents as mobile phase.

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ABSTRACT

Different metal ions were separated on thin layer of laboratory prepared urea-formaldehyde polymer using cationic and anionic surfactants, organic, acidic and neutral mobile phases. Using different organic solvents in different ratios of water as a mobile phase different metal ions and their binary and tertiary mixture of metal ions were separated. By using ethanol and surfactants in different ratios as a mobile phase different metal ions and their binary mixture of Cu^{2+} - V^{5+} , Fe^{3+} - Cu^{2+} , U^{6+} - Fe^{3+} , U^{6+} - V^{5+} and tertiary mixture of U^{6+} - Fe^{3+} - Cu^{2+} were successfully separated.

Key Words: Thin-layer chromatography Urea-formaldehyde polymer Silica gel-G Separation of metal ions.

INTRODUCTION

Thin layer chromatography (TLC) is a very convenient and rapid method for the separation and identification of organic ions. Therefore, any attempt to improve TLC for this application seems to be the part of interest for Chromatographers. TLC is described as a method for chromatographic analysis on thin layer of adsorbents. Some workers refer this technique as open column chromatography, spread layer chromatography and surface chromatography. TLC has a number of basic advantages over other chromatographic techniques. While a methodology is being developed for a specific separation; it uses less solvent and polarity of the solvent or the type of a solvent mixture can be changed in a matter of minutes. Thus, because of short development time and easy change of mobile phase it is the easiest method for separation and identification of metal ions. It has been successfully utilized in the analysis of waste water for heavy metal contents^[1], characterization of hazardous waste^[2], estimation of toxic metal ions in industrial sewage^[3], separation of heavy metal cations^[4], separation of metal ion in tube well water sample^[5], TLC of metal ions on a new carbamide – formaldehyde polymer^[7]. Number of metal ions were systematically chromatographed on thin layer of carbamide-formaldehyde polymer (aminoplast) which are already used for the separation of amino acids^[16] and water soluble vitamins^[17], utilizing neutral and acidic eluents. The detection, identification and separation of certain heavy metal ions on silica gel-G have been studied^[8]. Recently^[9-12] chromatographic separation of inorganic species on silica gel and alumina layer, with surfactants mediated mobile phase have been reported in literature. Separation studies of Transition metal ions with cationic micellar eluents in normal phase TLC^[13], recovery of metal ions from micellar solution^[14], identification

and separation of cationic and anionic surfactants by reversed phase TLC^[15] are some of the recently reported separation in TLC.

In continuation of our earlier work on TLC^[5-6], the present communication deals with the systematic study of separation and selectivity of mobile phase for different metal ions by TLC using mixture of urea-formaldehyde and silica gel-G as thin layers.

MATERIALS AND METHODS

Apparatus:

Ordinary glass plates 4 x10 cm size were used for coating with mixture urea formaldehyde and silica gel-G. Elico pH meter model was used for adjusting the pH of the solution. The plates were developed in 18 x13 cm glass jar.

Chemicals and Reagents:

Urea, formalin solution, silica gel-G, dimethyl glyoxime, potassium ferrocyanide, dithiozone, acetone, ethanol, methanol, starch, monochloroacetic acid, cationic surfactant(Benz alkonium chloride) and anionic surfactant(sodium dodecyl sulphate).

Metal ions studied: Cu²⁺, U⁶⁺, Zr⁴⁺, V⁵⁺, Sb³⁺, Pb²⁺, Ag⁺, Zn²⁺, Fe³⁺ and Ni²⁺.

Test solutions:

Chromatography was performed using standard aqueous solution of chlorides, nitrates or sulphates of above metal ions.

Detection:

Cu²⁺, U⁶⁺, Zr⁴⁺, V⁵⁺ and Fe³⁺ were detected with aqueous solution of potassium ferrocyanide. Sb³⁺, Pb²⁺, Ag⁺ and Zn²⁺ were detected with solution of dithiozone in carbon tetrachloride. Ni²⁺ was detected with alcoholic solution of dimethyl glyoxime.

Stationary phase:

Mixture of urea formaldehyde and silica gel-G in 1:1(wt/wt) ratio.

Mobile phase:

Different organic, neutral and acidic eluents in different ratios with varying pH were used in the mobile phase. Similarly, chromatographic study of different metal ions was examined in cationic and anionic surfactants with different concentrations and pH of mobile phase.

List of different solvent system used is given in Table 1:

Table 1. List of solvent systems used as mobile phases.

Sr.no.	Symbol	Composition(v/v)
1	Solution of water and ethanol in different ratios	
	S1	Ethanol + water in 3:2 ratio
	S2	Ethanol + water in 2:2 ratio
2	Solution of water and acetone in different ratios	
	S3	Ethanol + water in 1:3 ratio
	S4	Acetone + water in 3:2 ratio
3	S5	Acetone + water in 2:2 ratio
	S6	Acetone + water in 1:2 ratio
	S7	Acetone + water in 1:3ratio
	Surfactants and ethanol in different ratios	
3	S8	3% Cationic surfactant + ethanol in 3:1 ratio
	S9	3% Cationic surfactant + ethanol in 3:1 ratio at pH=3.5
	S10	3% Cationic surfactant+ ethanol in 3:1 ratio at pH=2.5

CHROMATOGRAPHY:**Preparation of plates:**

Urea formaldehyde was prepared in the laboratory using urea and formalin solution. Mixture of urea formaldehyde polymer and silica gel-G was taken in 1:1(wt/wt) ratio. The TLC plates were prepared by mixing mixture of silica gel-G and urea formaldehyde polymer with demineralised water in 1:2 ratio by weight with constant stirring to obtain homogeneous slurry. It was then immediately applied on the glass plates by dipping method. The plates were allowed to dry over night at room temperature and were used next day for TLC.

Procedure:

The test solutions of metal ions were spotted on the urea formaldehyde silica gel-G plates with glass capillaries. The spots were dried with hot air from air blower. The plates were developed in the glass jar containing 50 ml of selected mobile phase. Various spot reagents such as 1% alcoholic solution of dimethyl glyoxime, 0.02% dithizone solution in carbon tetra chloride and 3% aqueous solution of potassium ferrocyanide solution were sprayed for the detection of various metal ions.

RESULTS AND DISCUSSION

The results of this study have been summarized in Tables 2-7. The mobility of 10 metal ions was examined on mixture of urea formaldehyde and silica gel-G layers using different mobile phases as per given in table no.1. Mobility of metal ions was examined under the effect of:

- 1) Solution of water and ethanol in different ratios.
- 2) Solution of water and acetone in different ratios.
- 3) Surfactants and ethanol in different ratios.

1) Effect of composition of water and ethanol on R_f values of metal ions.

Table 2. Effect of solution of water and ethanol in different ratios of R_f values of metal ions.

Metal ion	R_f values		
	S1	S2	S3
Cu^{2+}	0.41T	0.39	0.32T
Zn^{2+}	0.27T	0.37	0.29T
Zr^{4+}	0.88	0.92	0.83
Sb^{3+}	0	0.72T	0.87T
Pb^{2+}	0	0.51	0.51
Fe^{3+}	0.25	0.31	0.27
V^{5+}	0.85	0.93	0.84
U^{6+}	0.38T	0.33	0.3T
Ag^+	0.2T	0.29T	0.44T
Ni^{2+}	0.2	0.24	0.49

T=Tailed spot

To understand the effect of the composition of mobile phase solution of water and ethanol in different ratios on the mobilities of metal ions chromatography was performed on mixture of urea formaldehyde and silica gel-G layers. The R_f values of metal ions obtained in solution of water and ethanol in different ratios (S1-S3) are listed in Table 2.

Following conclusions may be drawn from the data given in Table 2 and Figure 1:

- 1) The mixture of water and ethanol at 3:2 ratio(S1), metal ions like Zr^{4+} and V^{5+} show higher mobilities where as Cu^{2+} and U^{6+} show mid mobilities. Metal ions like Zn^{2+} , Fe^{2+} , Ag^+ and Ni^{2+} show little mobilities. Similar mobilities of metal ions were obtained in case of S2 (ethanol and water in 2:2 ratio) and S3 ethanol and water in 1:3 ratio).
- 2) Metal ions Ag^+ and Ni^{2+} show increase in the mobilities with decrease in the concentration of ethanol. On the contrary U^{6+} show decrease in the mobility with the decrease in concentration of ethanol.
- 3) Zr^{4+} and V^{5+} show higher mobilities in all cases. Where as Sb^{3+} shows higher mobilities in S2 ($R_f=0.72$) and S3 ($R_f=0.87$).
- 4) The difference between the mobilities of metal ions facilitates the opportunities for its selective separation from multi component mixture of metal ions.

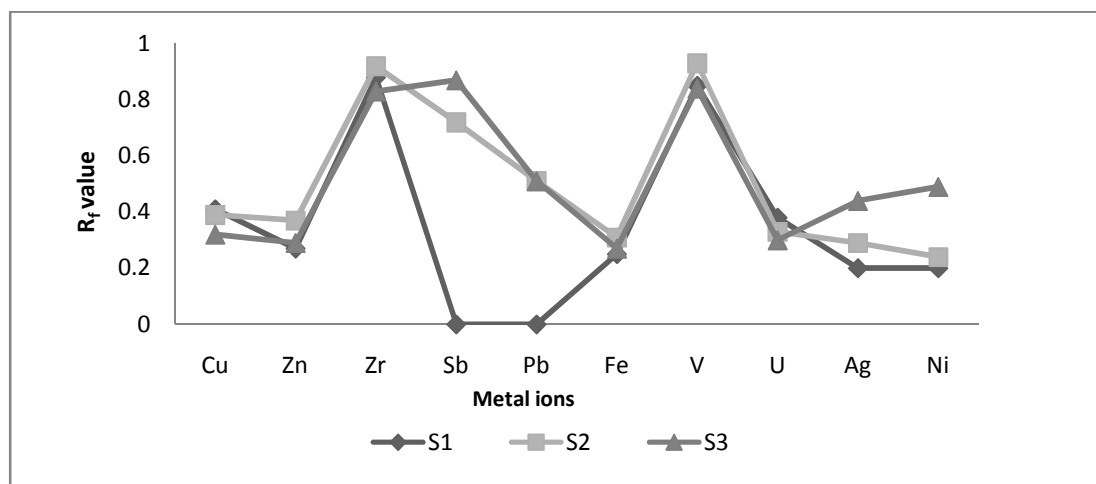


Figure 1 Chromatogram of some metal ions on mixture of urea formaldehyde and silica gel –G layers with solvent S1, S2, S3.

2) Effect of composition of water and acetone on R_f values of metal ions.

Table 3. Effect of composition of water and acetone in different ratios on R_f values of metal ions.

Metal ion	R _f values			
	S4	S5	S6	S7
Cu ²⁺	0.46T	0.28	0.27	0.15
Zn ²⁺	0.46T	0.35T	0.29T	0.52T
Zr ⁴⁺	0.86	0.86	0.84	0.89
Sb ³⁺	0.91T	0.87T	0.83T	0.83T
Pb ²⁺	0.58	0.4	0.26	0.25
Fe ³⁺	0.3	0.26	0.17	0.18
V ⁵⁺	0.85	0.85	0.88	0.88
U ⁶⁺	0.37	0.39T	0.36T	0.24
Ag ⁺	0.17T	0.15T	0.33T	0.56T
Ni ²⁺	0	0.59	0.55	0.60

T= Tailed spot.

Thin layer chromatography of metal ions was also performed using different composition of water and acetone in different ratios as mobile phase. Acetone and water was taken in different ratios and mobilities of metal ions were studied. Acetone and water was taken in 3:2, 2:2, 1:2, and 1:3 ratios. Difference in mobilities was observed as the concentration of acetone was varied. Different R_f values were obtained at different ratios of acetone and water and are given in Table 3:

Following conclusions may be drawn from the data given in Table 3 and Figure 2:

1) Metal ions like Zr⁴⁺, Sb³⁺ and V⁵⁺ show higher mobilities in mixture of acetone and water (S4-S7). Where as U⁶⁺, Pb²⁺, Ni²⁺ and Zn²⁺ show mid mobilities in the mixture of acetone and water (S4-S7). Fe³⁺ shows lower mobility in the same mobile phase.

2) With the decrease in the concentration of acetone mobility of Cu²⁺, Pb²⁺ and Sb²⁺ was decreased. But Sb³⁺ shows slight decrease in the mobility with decrease in the concentration of acetone. On the contrary V⁵⁺ shows slight increase in the mobilities with decrease in the concentration of acetone.

3) Zn²⁺ shows mid mobility in the mixture of acetone and water (S4-S7). But it was observed that Zn²⁺ shows decrease in mobility in (S4-S6). But there was sudden increase in mobility in S7 (R_f= 0.52). Ag⁺ shows lower mobility in S4 (R_f= 0.17) but it shows increase in the mobility at lower concentration of acetone S7 (R_f=0.56). 4) At 3:2 ratio of acetone and water S4 Pb²⁺ Shows high mobility (R_f= 0.58) but at the lowest concentration of acetone and water (acetone and water in 1:3 ratio) S7, it shows decrease in the mobility (R_f=0.25). Zr⁴⁺ Shows near about constant mobility in all concentrations of acetone. It shows high mobility in all concentration. U⁶⁺ shows constant mobility in (S4-S6) But their was sudden decrease in mobility at the lowest concentration of acetone S7 (R_f=0.24).

5) Ag⁺, Zn²⁺ and Sb³⁺ depict elongated tailed spot in all concentration of acetone and water (S4-S7).

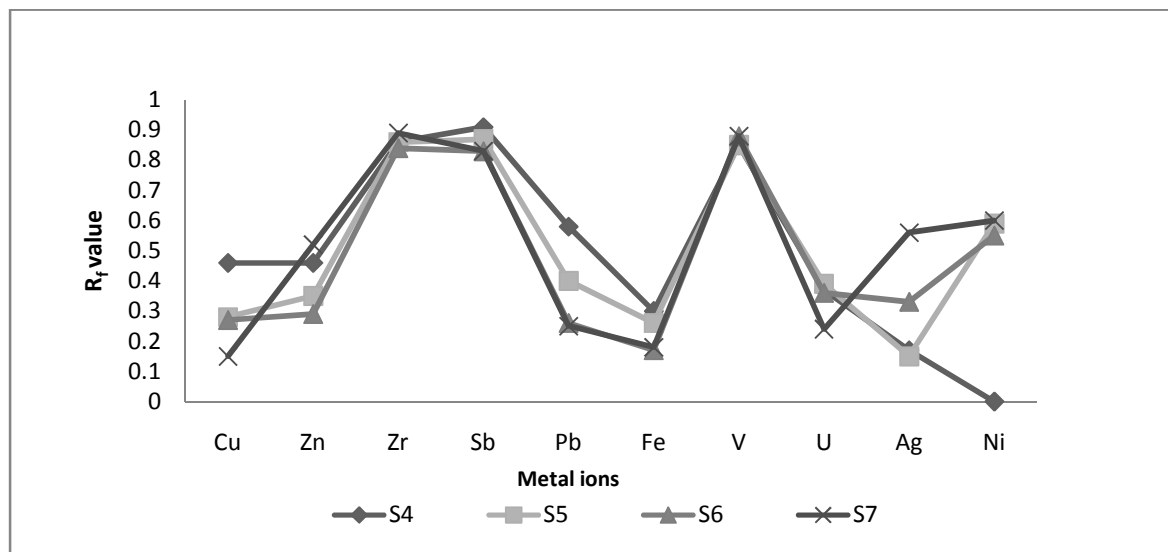


Figure 2 Chromatogram of some metal ions on mixture of urea formaldehyde and silica gel –G layers with solvent S4, S5, S6, S7.

3) Effect of composition of surfactant and ethanol in different ratios.

Table 4. Effect of composition of surfactant and ethanol in different ratios on R_f values of metal ions.

Metal ion	R _f values		
	S8	S9	S10
Cu ²⁺	0.63T	0.69T	0.70
Zn ²⁺	0.61T	0.66	0.72
Zr ⁴⁺	0.90	0.84	0.90
Sb ³⁺	0.90T	0.95T	0.93T
Pb ²⁺	0.35	0.47T	0.47
Fe ³⁺	0.22	0.11T	0.10
V ⁵⁺	0.87	0.89	0.89
U ⁶⁺	0.36T	0.31T	0.20
Ag ⁺	0.18	0.17	0.17
Ni ²⁺	0.93	0.0	0.96

T=Tailed spot

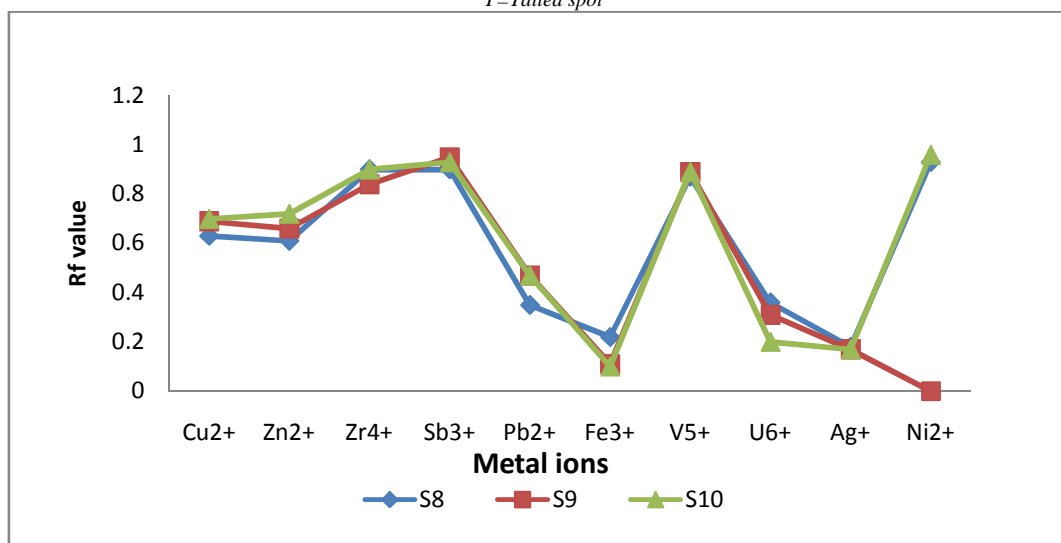


Figure 3 Chromatogram of some metal ions on mixture of urea formaldehyde and silica gel –G layers with solvent S8, S9, S10.

Cationic (BAC) and anionic (SDS) surfactants were mixed with ethanol at specific pH to examine the mobility of ions. We got good results of mobility of metal ions in 3% of cationic surfactant (S14), so 3% cationic surfactant were mixed with ethanol in 3:1 ratio and keeping this ratio constant, we changed the pH of this solution (S8-S10) to study the mobility of ions at specific polarity, R_f values obtained at normal pH of 3% cationic surfactant and ethanol (S8), at pH3.5 (S9) and pH=2.5(S10) is listed in Table 6.

By observing above R_f values in Table 4 and Figure 3 following conclusions can be drawn :

- 1) At S8 metal ions such as Zr^{4+} , Sb^{3+} , V^{5+} and Ni^{2+} show high mobilities, similar mobilities they show in S9 and S10. Not much difference in R_f values was observed in comparison with different pH.
- 2) Cu^{2+} , Pb^{2+} and Zn^{2+} show increase in the mobilities with the decrease in the pH. On the contrary U^{6+} and Fe^{3+} show decrease in the mobilities with the decrease in the pH.
- 3) Cu^{2+} and Zn^{2+} show the mid mobilities whereas Pb^{2+} , Fe^{3+} , U^{6+} and Ag^+ show lower mobilities.
- 4) Lowest mobility was shown by Ag^+ in S8-S10. There was not much difference in the mobility of Ag^+ and V^{5+} in S8-S10. It shows that mobility of Ag^+ and V^{5+} was unaffected with pH of S8-S10.
- 5) There was observable change found in the mobility of Pb^{2+} , it was increased with the decrease in the pH of S9 ($R_f=0.47$) and further it remained constant with decrease in pH of S10 ($R_f=0.47$).
- 6) There was continuous decrease in the mobility of U^{6+} and Fe^{3+} was observed with the decrease in the pH of mobile phase.
- 7) We found considerable difference in the R_f values of the different metal ions in S8-S10. This facilitates the opportunity to separate the individual metal ions from binary and ternary mixture components.

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