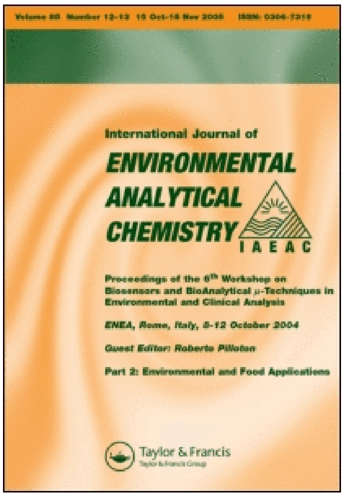

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L. Deshmukh[a]; R. B. Kharat[a]
[a] Department of Chemistry, Institute of Science, Nagpur, India

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SEPARATION OF METAL IONS IN TUBE WELL WATER SAMPLES BY THIN LAYER PARTITION CHROMATOGRAPHY

L. DESHMUKH and R. B. KHARAT

Department of Chemistry, Institute of Science, Nagpur 440001, India

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A TLC method has been developed for the separation of Fe(III), Zn(II), Cu(II), Pb(II), Mn(II) and Ni(II). Thin layers of silica gel G were used as an adsorbent. R_f values were determined using 0.01–0.2 M aqueous sodium acetate as mobile phase. The dependence of the R_f values on the pH, the width of the plates and the time required for migration of the ions was studied. Optimum conditions for possible 3- and 4-component separations have been determined. Ni(II), Zn(II), Fe(III), Cu(II), Mn(II) and Pb(II) having minimum concentration of 0.65, 0.5, 4.0, 0.70, 0.5 and 2.0 μg/ml, respectively have been separated. The present method was applied to the separation and detection of metal ions in tube well water samples from different villages of Orissa (India).

KEY WORDS: Metal ion separation, thin layer, partition chromatography, environmental samples, tube well water.

INTRODUCTION

Considerable attention has been devoted to the use of TLC for the separation of cations. A literature survey reveals the use of thin layers of silica gel G impregnated with high-molecular-weight amines, substituted quaternary ammonium salts, heterocyclic amines and tetrasubstituted pyrazole as stationary phases.[1–2] Qureshi *et al.*[3] reported the use of various solvent systems like 1 M HCl, 1 M HNO_3, 0.5 M H_2SO_4, 0.1 M oxalic acid, 0.1 M tartaric acid and 0.1 M citric acid as mobile phase for the separation of cations on thin layers of silica gel G impregnated with tri-n-butyl phosphate. 3-Component separations were reported by Fatima *et al.*[4–5] on impregnated and unimpregnated silica gel G layers using formic acid and sodium formate as mobile phase. Many interesting separations are reported by Leene *et al.*[6] on thin layers of silical gel impregnated with Alamine 336-S, Alamine 336-S oxide, tri-n-octyl phosphine oxide, tri-n-octylarsine oxide and non-impregnated silica gel using nitric acid as eluant.

This paper deals with the rapid separation of cations in 4-component mixture of non-impregnated silica gel G using aqueous solutions of sodium acetate as the mobile phase. Mixtures of cations from tube well water were separated and detected.

EXPERIMENTAL

Apparatus

Ordinary glass plates of 4 × 10 cm size were used for coating with silica gel G. An Elico pH meter model L 1 10 T (Elico Private Ltd, Hyderabad, India) was used for adjusting the pH of the solutions. The plates were developed in 18 × 13 cm glass jars.

Chemical and Reagents

The stock solutions—0.05 M of chlorides, nitrates and sulphates—were prepared in 0.1 M hydrochloric acid. All solutions were made in doubly distilled water. Silica gel G (E. Merck) was used for preparation of thin layers. Sodium acetate and other reagents used were of AnalaR grade.

Preparation of Thin Layers

A slurry was prepared by mixing silica gel G and doubly distilled water in the ratio of 1:3 with constant shaking for about 5 min. It was then immediately applied to the glass plates by the dipping method.[7] The plates were allowed to dry overnight at room temperature and were used next day for TLC.

Procedure

The test solutions were spotted on the silica gel G plates with glass capillaries and they were blow-dried with hot air. The sodium acetate solution of a particular concentration was adjusted to the desired pH using sodium hydroxide or hydrochloric acid solutions. The plates were developed for 15 min in the glass jar containing 20 ml of sodium acetate solution. The volume of mobile phase required was 2–3 ml per plate. Various spot test reagents[8] such as a 1% alcoholic solution of dimethyl-glyoxime (Ni(II)), 0.02% dithiozone in carbon tetrachloride (Pb(II) and Zn(II)) and 3% aqueous potassium ferrocyanide (Fe(III) and Cu(II)) were sprayed for the detection of cations. Mn(II) was detected by spraying with 0.1% aqueous solution of 4-(2-Pyridylazo)-resorcinol.

All experiments were carried out at room temperature. The R_f values were measured in triplicate for each set of determinations. Various experiments were carried out to study the effect of plate width (2 to 4.5 cm), acetate concentration in mobile phase (0.01–0.2 M), pH (1–10) and time (5–25 min) on the R_f values of the individual cations.

RESULTS AND DISCUSSIONS

The R_f values of different cations are presented graphically in Figures 1–3 and are

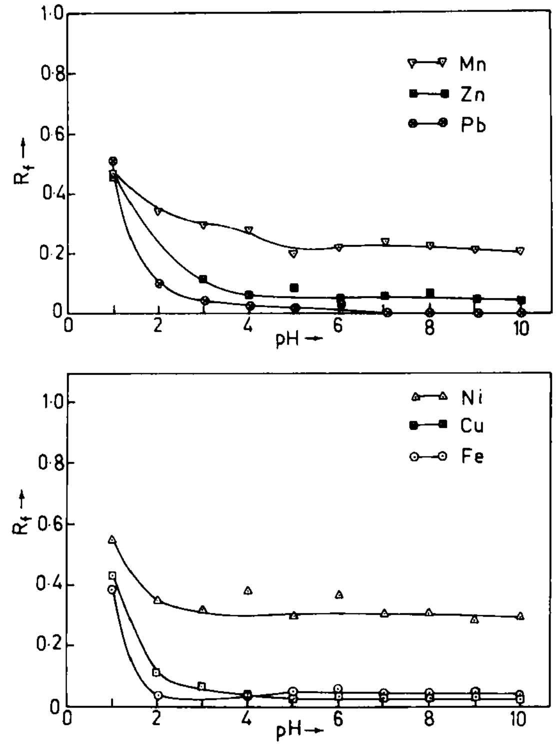

Figure 1 Variation in R_f value with pH at a 0.01 M acetate concentration in the mobile phase.

plotted against the acetate concentration in the mobile phase. The 4-component separations are shown in Figure 4.

Figure 1 shows the behaviour of cations for a 0.01 M acetate concentration at different pH values. The R_f values of Fe(III), Ni(II), Zn(II), Pb(II), Mn(II) and Cu(II) are between 0.39 and 0.55 at pH 1; Fe(III), Cu(II) and Pb(II) remain near the baseline for pH 5–10, while Pb(II) precipitates for pH > 7. As the pH increases from 3 to 10, it does not have any effect on the R_f values. In this pH range, only Ni(II) and Mn(II) show a tendency to migrate.

Figure 2 shows that if the acetate concentration is increased from 0.01 M to 0.05 M, the cations show little separation tendency until pH 4. From pH 5 onwards, Cu(II), Fe(III), Zn(II) and Pb(II) remain near the baseline. At a 0.1 M acetate concentration (Figure 3) all cations move rather close to the solvent front at low pH. Maximum differences in the R_f values of the cations are observed at pH 5, but some separation also occurs at pH 4. Cu(II), Zn(II) and Pb(II) show tailing for pH > 6.

Since the best separation is achieved with a 0.1 M acetate concentration as the mobile phase (Figure 3), this concentration was used for further studies. When the acetate concentration in the mobile phase was increased to 0.2 M there was no further appreciable change in the R_f values of any of the six cations. Hence the use of higher concentrations was avoided. As is to be expected, varying the plate

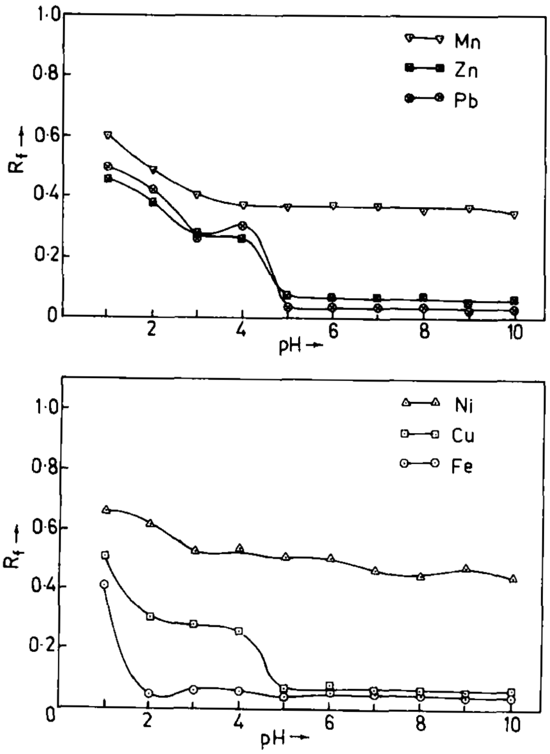

Figure 2 Variation in R_f value with pH at a 0.05 M acetate concentration in the mobile phase.

width had no effect on the R_f values of the cations. When the effect of the time of migration was studied, it was observed that 3-component and 4-component separations are possible within 5–7 min. Separation of the 4-component synthetic mixtures Ni(II)-Mn(II)-Cu(II)-Fe(III), Ni(II)-Mn(II)-Pb(II)-Fe(III), Ni(II)-Mn(II)-Zn(II)-Fe(III) (1–3) and a 3-component mixture in a tube well water sample (4) is shown in Figure 4. The R_f values for Ni(II), Mn(II), Cu(II), Pb(II), Zn(II) and Fe(III) were 0.80, 0.60, 0.34, 0.38, 0.33 and 0.11, respectively.

The metal ions could be separated and detected in the concentration range of 0.65–3.2 μg/ml Ni(II), 0.70–3.5 μg/ml Cu(II), 2–10 μg/ml Pb(II), 0.5–4.0 μg/ml Zn(II), 0.5–10.0 μg/ml Mn(II) and 4–20 μg/ml Fe(III). The proposed method was applicable to the separation and detection of Mn(II), Fe(III) and Zn(II) in tube well water samples collected from different villages in the Phulbani district of Orissa (India). The results are presented in Table 1.

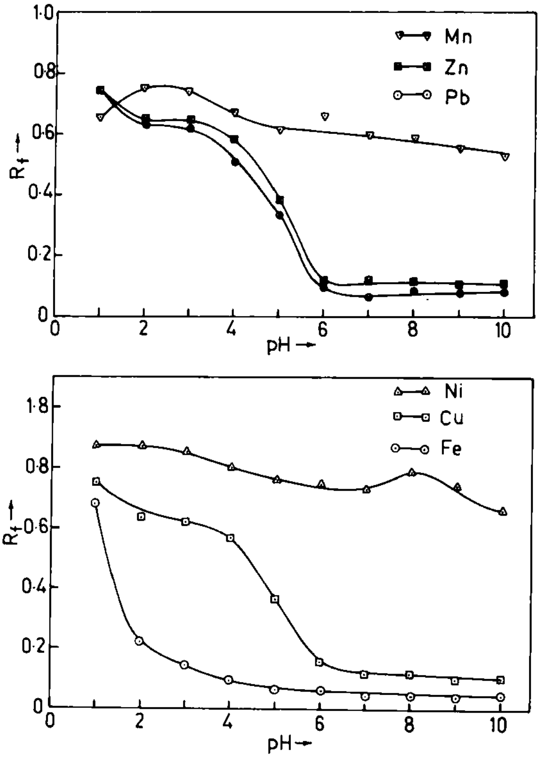

Figure 3 Variation in R_f values with pH at a 0.1 M acetate concentration in the mobile phase.

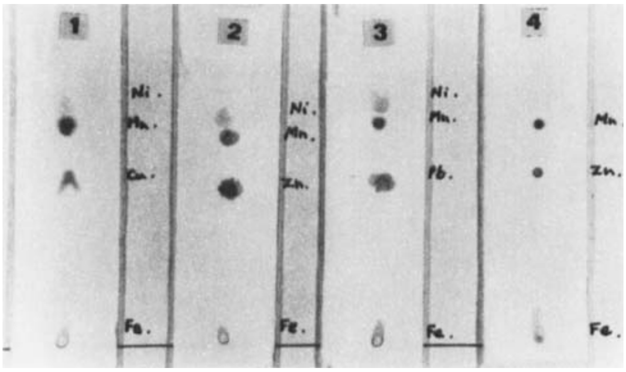

Figure 4 4-Component separations (1–3) in synthetic mixtures and a 3-Component separation (4) in a tube well water sample. Conditions: pH 5, migration time 15 min, 0.1 M aqueous acetate as mobile phase.

Table 1 Separation of metal ions in tube well water samples. Adsorbent: Silica gel G; mobile phase: 0.1 M Sodium acetate solution; plate width: 4 cm; pH 5; migration time 5 min

Sample	*Composition*	*Concentration μg/ml*	*R_f values*
1. Village: Dokedi Gram Panchayat: Ratingia, Block G. Udayagiri	Fe(III)	16.1	0.11
	Mn(II)	0.6	0.59
	Zn(II)	3.6	0.33
2. Village: Gatamaha Gram Panchyat: Lingagate, Block G. Udayagiri	Fe(III)	16.7	0.11
	Mn(II)	0.6	0.60
	Zn(II)	0.6	0.30
3. Village: Sandhakupa Gram Panchayat: Gumamha, Block: Raikia	Fe(III)	15.2	0.10
	Mn(II)	0.6	0.59
	Zn(II)	3.0	0.33

References

1. U. A. Th. Brinkman, J. H. Taphroorn and G. De Vries, *J. Chromatogr.* **84**, 407 (1973).
2. G. J. De Jong, W. Th. Kok and U. A. Th. Brinkman, *J. Chromatogr.* **135**, 249 (1977).
3. M. Qureshi, B. M. Sethi and S. D. Sharma, *J. Liq. Chromatogr.* **6**(1), 165 (1983).
4. N. Fatima and A. Mohammad, *Sep. Sci. and Technol.*, **19**(6 & 7), 429 (1984).
5. M. Qureshi, A. Mohammad and N. Fatima, *J. Liq. Chromatogr.*, **8**(7), 1279 (1985).
6. H. R. Leene, G. De Vries and U. A. Th. Brinkman, *J. Chromatogr.* **80**, 221 (1973).
7. J. J. Peifer, *Mickrochim. Acta.*, **529** (1962).
8. F. Feigl, *Spot Test Inorganic Applications* (Elsevier Pub. Co., Amsterdam, 1954), 4th ed., Chap. 1.