

Silica Thin-Layer Chromatographic Studies of Surfactants with Mixed Aqueous-Organic Eluents Containing Thiourea: Simultaneous Separation of Co-existing Cetyltrimethylammonium Bromide, Dodecyltrimethylammonium Bromide, and Polyoxyethylene (20) Sorbitan Monolaurate

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Abstract

Silica thin-layer chromatography of three surfactants using various solvent systems is described. The mutual separation of coexisting cetyltrimethylammonium bromide (CTAB), dodecyltrimethylammonium bromide (DTAB), and polyoxyethylene (20) sorbitan monolaurate (Tween 20) is achieved on silica layer using 5% aqueous thiourea–acetone–methanol (60:20:20, v/v/v) as the mobile phase. The effect of the carbon chain length of alcohols (methanol, ethanol, *n*-propanol, and *n*-butanol) on the mobility of these surfactants is examined on silica layers. The comparative study is performed with sulfur- (thiourea) and oxygen- (urea) containing compounds in the eluent on the mobility as well as on the separation of co-existing CTAB, DTAB, and Tween 20. The interference on the resolution of the mixture of CTAB, DTAB, and Tween 20, due to presence of metal cations as impurities, is also examined. The limits of detection of CTAB, DTAB, and Tween 20 are estimated.

Introduction

Separation by thin-layer chromatography (TLC) is mainly controlled by a mutual action of the stationary and mobile phases on the analyte. As a general practice, the composition of mobile phases is usually altered to obtain a desired separation on a particular adsorbent. According to the literature on the TLC analyses of surfactants, several mixed organic or aqueous-organic mobile phases are currently in use, including

methanol in combination with dichloromethane, 0.1M aqueous glutamic acid, chloroform, water, acetone, 2N NH₃, 0.1N sulfuric acid, 3.84% ammonium acetate, etc. (1–8); ethanol in combination with 20% sodiumtetrphenylborate, methylethylketone, benzene, water, etc. (9–12); propanol in combination with 4-methyl-2-pentanone, acetic acid, etc. (10); butanol in combination with acetic acid, water, EDTA, etc. (13); acetone in combination with benzene, water, methylethylketone, ethylacetate, etc. (14–18); acetic acid in combination with 4-methyl-2-pentanone, 1-propanol, acetonitrile, chloroform, water, butanol, EDTA, ethylacetate, isooctane, etc. (10,13,19,20); chloroform in combination with methanol, water, 0.1N sulfuric acid, etc. (3–5); carbon-tetrachloride in combination with acetonitrile (21); and benzene in combination with acetone, water, etc. (10,15,17). Alcohols (methanol, ethanol, and 1-propanol) have generally been used as organic modifiers of the aqueous mobile phases (22).

From what has been previously mentioned, it is clear that most of the mobile phase systems comprised of pyridine, benzene, chloroform, or carbon tetrachloride as one of the components are not especially useful due to their strong toxic nature. Therefore, any attempt to develop new mobile phases for TLC analyses of the surfactants seems to be of interest for chromatographers. With this perspective, we have identified a new TLC system comprised of silica gel G as stationary phase and aqueous thiourea (5%)–acetone–methanol (60:20:20, v/v/v) as mobile phase for the detection and identification of coexisting cetyltrimethylammonium bromide (CTAB), dodecyltrimethylammonium bromide (DTAB), and polyoxyethylene (20) sorbitan monolaurate (Tween-20) surfactants with preliminary TLC separation. Thiourea was used, as it has been reported that it forms inclusion complexes with

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branched-chain and cyclic aliphatic compounds and with the straight-chain compounds containing more than fourteen carbon atoms (23). The suggested TLC system does not involve the use of toxic solvents. Furthermore, the proposed method is capable of identifying surfactants present in different aqueous systems.

The analysis of the mixture of cetyltrimethylammonium bromide (CTAB), dodecyltrimethyl-ammonium bromide (DTAB), and polyoxyethylene (20) sorbitan monolaurate (Tween 20) is important because of their wide applicability in many benign environmental systems. Tween-20 has found applications in enzyme-linked immuno sorbent assay for the detection of levels of antibodies in serum samples of patients. CTAB as the mobile phase in micellar TLC and as impregnant for silica in ion-pair TLC has proven to be an excellent TLC medium for achieving analytically important separations of heavy metal cations and organic dyes, respectively (24).

Experimental

All experiments were performed at $25 \pm 2^\circ\text{C}$.

Apparatus

A TLC applicator was used for coating silica gel on 20×3.2 cm glass plates; the chromatography was performed in 24×6 cm glass jars. A glass sprayer was used to spray reagent on the plate to locate the position of the spot of analyte.

Chemicals and reagents

Silica gel G (Merck, Mumbai, India), thiourea and urea (Merck), ethanol (CDH, New Delhi, India), acetone, methanol, *n*-propanol, and *n*-butanol (Qualigens, Mumbai, India) were used. All the reagents used were of analytical grade.

Surfactants studied

DTAB was purchased from Sigma-Aldrich (Steinheim, Germany), and both CTAB and Tween-20 were obtained from CDH. All surfactants were used as received.

Test solutions

Solutions of the surfactants were prepared in double distilled water to give concentration of 0.5% (w/v). The stationary phase was silica gel G. The mobile phase was, in addition to ternary mobile phases listed in Table I, solvent systems 1–5% aqueous thiourea solutions (M_1 – M_3), acetone (M_4), methanol (M_5), and binary mobile phases (M_6 – M_{14}) containing acetone and 5% aqueous thiourea in 10:90, 20:80, 30:70, 40:60, 50:50, 60:40, 70:30, 80:20, and 90:10 ratio, v/v were also used as solvent systems.

Detection reagent

The surfactants were detected using modified Dragendorff reagent (2).

Preparation of TLC plates

TLC plates were prepared by mixing silica gel G with double distilled water in a 1:3 ratio. The resultant slurry was mechanically shaken for 5 min and then coated onto glass plates with the help of a TLC applicator to give a layer of 0.25 mm thickness. The plates were first air dried at room temperature and then activated by heating at 100°C for 1 h. After activation, the plates were kept in an airtight chamber until used.

Procedure

The surfactant solutions (0.01 mL) were spotted onto TLC plates (50 $\mu\text{g}/\text{spot}$) with a micropipette at approximately 2 cm above the lower edge of the TLC plate. The spots were dried at room temperature ($25 \pm 2^\circ\text{C}$). The glass jars containing the mobile phase were covered with lids and left for 10 min for saturation before introducing the plates for development. The plates were developed with the chosen solvent system to a distance of 10 cm from the origin in all cases. After development, the plates were detected using Dragendorff reagent and all the studied surfactants visualized as orange colored spots. The development time was 35–40 min for 10 cm ascent.

For the separation of surfactant mixtures, equal volumes of surfactants were

Table I. Mobile Phases

Nature	Symbol	Composition
Mixed aqueous-organic three-component systems	M_{15}	5% aqueous thiourea + acetone + methanol (60:40:10, v/v/v)
	M_{16}	5% aqueous thiourea + acetone + methanol (60:40:20, v/v/v)
	M_{17}	5% aqueous thiourea + acetone + methanol (60:40:30, v/v/v)
	M_{18}	5% aqueous thiourea + acetone + methanol (60:10:20, v/v/v)
	M_{19}	5% aqueous thiourea + acetone + methanol (60:20:20, v/v/v)
	M_{20}	5% aqueous thiourea + acetone + methanol (60:30:20, v/v/v)
	M_{21}	5% aqueous thiourea + acetone + ethanol (60:20:20, v/v/v)
	M_{22}	5% aqueous thiourea + acetone + <i>n</i> -propanol (60:20:20, v/v/v)
	M_{23}	5% aqueous thiourea + acetone + <i>n</i> -butanol (60:20:20, v/v/v)
	M_{24}	5% aqueous urea + acetone + methanol (60:20:20, v/v/v)

mixed and 0.01 mL of the resultant mixture was applied onto TLC plates. The plate was developed with M_{19} , the spots were detected, and R_F values of the separated spots of surfactants were calculated.

In order to examine the effect of alcohol on the mobility of surfactants, the methanol in M_{19} was substituted with ethanol, *n*-propanol, *n*-butanol, and resultant mobile phase systems M_{21} [(5% aqueous thiourea + acetone + ethanol), (60:20:20, v/v/v)], M_{22} [(5% aqueous thiourea + acetone + *n*-propanol) (60:20:20, v/v/v)], and M_{23} [(5% aqueous thiourea + acetone + *n*-butanol), (60:20:20, v/v/v)] were used as eluents.

For the study of comparison of sulfur- and oxygen-containing compounds (viz. thiourea and urea) on the mobility and separation of CTAB, DTAB, and Tween-20, thiourea was substituted in M_{19} by urea and the resulting mobile phase system M_{24} [(5% aqueous urea + acetone + methanol), (60:20:20, v/v/v)] was used as eluent. The results obtained with M_{19} and M_{24} were compared.

For investigating the interference of presence of metal cations as impurities on the resolution of CTAB, DTAB, and Tween-20 from their mixtures, 0.01 mL of standard test mixtures of surfactant solution were spotted onto the plate followed by spotting of 0.01 mL of the cations being considered as impurities. The plates were developed with M_{19} , detected, and R_F values of the separated surfactants were calculated.

The limits of detection of surfactants were determined by spotting different amounts of solutions of the CTAB, DTAB, and Tween-20 on the plates. The plates were then developed and the corresponding spots were detected. The method was repeated with successive lowering of the amounts of CTAB, DTAB, and

Tween-20 until no spot was detected. The minimum amount of surfactants that could be detected was taken as the limit of detection. For accuracy, this was carried out in six replicate measurements.

The robustness, which is one of the validation parameters, was studied for resolution of the mixture (CTAB, DTAB, and Tween-20) in six replicate R_F value measurements at a concentration of 50 µg/spot of the mixture.

Results and Discussion

The results of the present study are summarized in Tables II–V and Figures 1–5. The mobility pattern of surfactants under study was examined on silica gel layer using one component (1–5% aqueous thiourea, acetone, and methanol), two component (5% aqueous thiourea + acetone), and three component (5% aqueous thiourea + acetone + methanol) mobile phase systems. The results obtained during this study are presented in Table II. With aqueous thiourea (1–5%), Tween-20 (R_F 0.21, tailed spot), and DTAB (R_F 0.11) show enhanced mobility on silica layer with increased concentration of thiourea (up to 5%), but CTAB remains at the point of application. This 5% concentration of thiourea was used for further study. In acetone, Tween-20 (R_F 0.17 T) produces badly tailed spots while other surfactants form compact spots with little mobility. In the case of methanol, Tween-20 (R_F 0.45 T) shows enhanced mobility with badly tailed spots while other surfactants show little mobility. The spots with $R_L - R_T \geq 0.3$ have been termed as tailed in this paper (25).

Table II. R_F Values of Different Surfactants with Different Mobile Phases Using Silica Gel as a Stationary Phase

Surfactants	1%	3%	5%	acetone methanol	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	acetone +5% tu	
	tu*	tu	tu		10:90, v/v	20:80, v/v	30:70, v/v	40:60, v/v	50:50, v/v	60:40, v/v	70:30, v/v	80:20, v/v	90:10, v/v	
Tween-20	0.17T [†]	0.21T	0.21T	0.17T	0.45T	0.22T	0.58	0.72	0.82	0.85	0.88	0.86	0.85T	0.50T
CTAB	0.0	0.0	0.0	0.05	0.06	0.0	0.02	0.06	0.08	0.15	0.18	0.19	0.22	0.25T
DTAB	0.09	0.11	0.12	0.05	0.06	0.20	0.22	0.25	0.27	0.30	0.25	0.19	0.17	0.14

* tu = thiourea.
† T = tailed spot, $R_L - R_T \geq 0.3$.

Table III. R_F Values of Different Surfactants in 5% Aqueous Thiourea with Changing Ratios of Acetone and Methanol on Silica Gel as a Stationary Phase

Surfactants	5% aq.* thiourea + acetone + methanol	5% aq. thiourea + acetone + methanol	5% aq. thiourea + acetone + methanol	5% aq. thiourea + acetone + methanol	5% aq. thiourea + acetone + methanol	5% aq. thiourea + acetone + methanol
	60:40:10, v/v/v	60:40:20, v/v/v	60:40:30, v/v/v	60:10:20, v/v/v	60:20:20, v/v/v	60:30:20, v/v/v
Tween 20	0.78	0.80	0.82	0.60	0.75	0.78
CTAB	0.08	0.10	0.13	0.03	0.03	0.05
DTAB	0.22	0.20	0.16	0.28	0.27	0.22

* aq = aqueous.

In the case of two-component solvent systems (5% aqueous thiourea plus acetone in different volume ratio), better separation possibilities were realized. At a lower volume ratio of acetone (i.e., in M_6 [acetone + thiourea, 10:90, v/v]), Tween-20 (R_F 0.22T) forms tailed spots. Conversely, at a much higher concentration of acetone (i.e., in M_{14} [acetone + thiourea, 90:10, v/v]), Tween-20 and CTAB produce tailed spots. For obtaining compact spots with an increased separation possibility of surfactants, the optimum concentration range of acetone is 30–70 V% (i.e., in solvent systems comprising of acetone plus 5% aqueous thiourea). The most preferred mobile phase from a separation point of view was M_9 (acetone: 5%

aqueous thiourea, 40:60, v/v). The increase in the volume ratio of acetone up to M_{11} (acetone + 5% aqueous thiourea, 60:40, v/v) leads to an increase in the R_F value of Tween-20. Similarly, the mobility of CTAB, which remains at the point of application in M_6 (acetone + 5% aqueous thiourea, 10:90, v/v), increases as the volume ratio of acetone increases in the mobile phase systems (M_7 – M_{14}). In the same fashion, the mobility of DTAB increases with an increase in acetone concentration from 20 V% (M_7) to 50 V% (M_{10}) in the mobile phase.

From data listed in Table II, it is evident that one-component mobile phases (M_1 – M_5) and two-component mobile phases (M_6 acetone: 5% aqueous thiourea, 10:90, v/v) are not even capable of separating nonionic surfactants from cationic surfactants. However, the addition of 20–70 V% acetone to 5% aqueous thiourea (mobile phase, M_7 – M_{12}) makes it possible to separate a nonionic surfactant (Tween-20) from all cationic surfactants being studied (i.e., CTAB and DTAB), but these mobile phase systems failed to resolve a mixture of cationic surfactants to discriminate among cationic surfactants. An organic modifier co-solvent (methanol) was added into the mobile phase M_9 and the resultant mobile phases (M_{15} – M_{20}) were used for the chromatography of the surfactants. The results obtained are listed in Table III. From the results of Table

III, it is clear that methanol modifies the R_F values (or mobility) of surfactants and provides an opportunity to separate intra- (nonionic from cationic) and inter- (cationic from cationic) group surfactants. As a result, a good separation of coexisting CTAB, DTAB, and Tween-20 can be achieved with M_{15} and M_{16} (Figure 1). However, a good separation of these surfactants could be obtained with M_{18} – M_{20} (Figure 2), M_{19} being the best mobile phase for producing highly compact and well-resolved spots of CTAB, DTAB, and Tween-20 from their mixtures. This favorable situation for the mutual separation of nonionic (Tween-20)–cationic (CTAB)–cationic (DTAB) with methanol-

Table IV. Comparison of Thiourea- and Urea-Containing Compounds on the Mobility of CTAB, DTAB, and Tween 20

Surfactants	R_F values	
	5%aqueous thiourea + acetone + methanol (60:20:20, v/v/v) M_{19}	5%aqueous urea + acetone + methanol (60:20:20, v/v/v) M_{20}
	CTAB	0.03
DTAB	0.27	0.19
Tween-20	0.75	0.72

Table V. Identification of CTAB, DTAB, and Tween 20 in Spiked Water Samples on Silica Gel Layers with 5% Thiourea + Acetone + Methanol (60:20:20, v/v/v)

Surfactants	R_F values	
	Tap Water	River Water
CTAB	0.0	0.0
DTAB	0.17	0.14
Tween-20	0.80	0.81

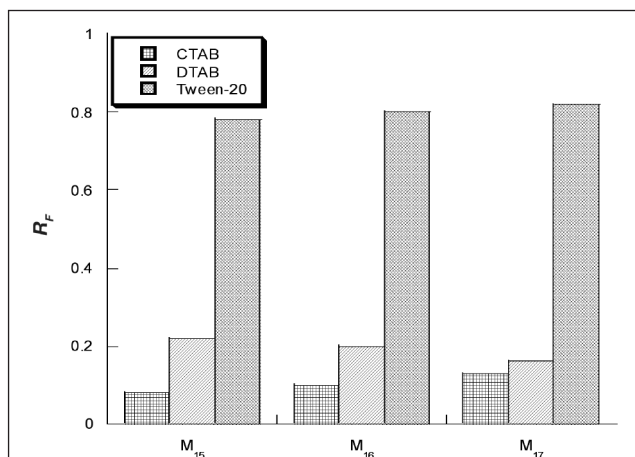


Figure 1. Effect of an increased volume ratio of methanol on the mobility of CTAB, DTAB, and Tween-20. M_{15} : 5% aq. thiourea–acetone–methanol (60:40:10, v/v/v); M_{16} : 5% aq. thiourea–acetone–methanol (60:40:20, v/v/v); M_{17} : 5% aq. thiourea–acetone–methanol (60:40:30, v/v/v).

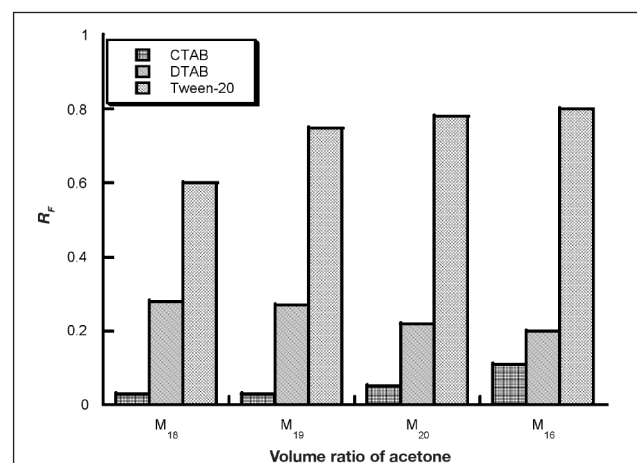


Figure 2. Effect of an increased volume ratio of acetone on the mobility of CTAB, DTAB, and Tween-20. M_{18} : 5% aq. thiourea–acetone–methanol (60:10:20, v/v/v); M_{19} : 5% aq. thiourea–acetone–methanol (60:20:20, v/v/v); M_{20} : 5% aq. thiourea–acetone–methanol (60:30:20, v/v/v); M_{16} : 5% aq. thiourea–acetone–methanol (60:40:20, v/v/v).

containing mobile phase systems is achieved due to the reduction in the R_F value of CTAB. It has been reported (26) that methanol, being the most hydrophilic alcohol, modifies the hydrophobic interactions between water and the surfactant molecules in a CTAB–H₂O–CH₃OH system. Amongst methanol-containing mobile phase systems, M₁₉ was found most efficient for the mutual separation of surfactants, and hence it was selected for further study.

To examine the influence of the nature of alcohol on surfactants, the methanol was substituted by different alcohols (viz. ethanol, *n*-propanol, and *n*-butanol in M₁₉). The results, shown in Figure 3, clearly indicate that with an increase in the length of the alkyl group or an increase in the bulkiness of the alkyl group, the mobility of the surfactants increases. In an *n*-butanol-containing mobile phase, all the surfactants show the formation of badly tailed spots.

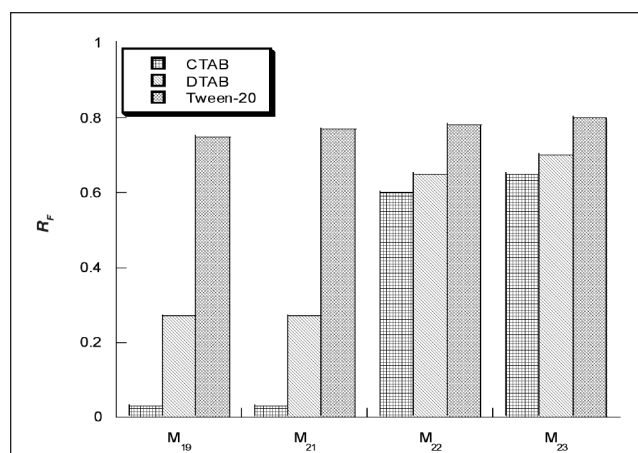


Figure 3. Effect of the nature of alcohols on the mobility of CTAB, DTAB, and Tween-20 on silica gel layers. M₁₉: 5% aq. thiourea–acetone–methanol (60:20:20, v/v/v); M₂₁: 5% aq. thiourea–acetone–ethanol (60:20:20, v/v/v); M₂₂: 5% aq. thiourea–acetone–*n*-propanol (60:20:20, v/v/v); M₂₃: 5% aq. thiourea–acetone–*n*-butanol (60:20:20, v/v/v).

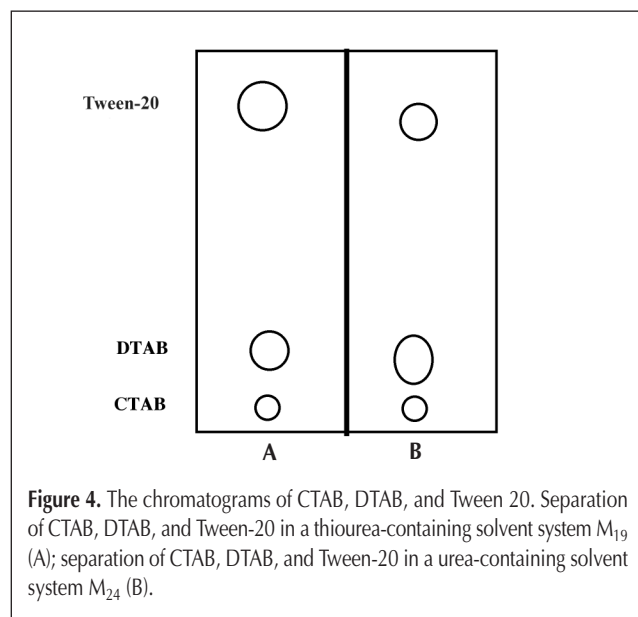


Figure 4. The chromatograms of CTAB, DTAB, and Tween-20. Separation of CTAB, DTAB, and Tween-20 in a thiourea-containing solvent system M₁₉ (A); separation of CTAB, DTAB, and Tween-20 in a urea-containing solvent system M₂₄ (B).

The results, listed in Table IV for the separation of surfactants and Figure 4, clearly indicate the effect of –CS– and –CO– groups and the R_F value of DTAB and Tween-20. DTAB has a slightly higher R_F value in a thiourea-containing mobile phase [(5% aqueous thiourea + acetone + methanol), (60:20:20, v/v/v)] as compared to its mobility in a urea-containing mobile phase [(5% aqueous urea + acetone + methanol), (60:20:20, v/v/v)]. This may be due to the large size of “S” (sulphur) as compared to the “O” (oxygen), which results in the formation of efficient inclusion compounds with these surfactants as compared to urea. Hence, better resolution of surfactants was achieved with a thiourea-containing mobile phase.

The results, showing the effect of various metal cations as impurities on the mutual separation of CTAB, DTAB, and Tween-20, are presented in Figure 5. It is clear from Figure 5 that separation is hampered by the presence of Al³⁺, Cr⁶⁺, Pb²⁺, Tl⁺, and UO₂²⁺. All the three surfactants (CTAB, DTAB, and Tween-20) co-migrate in the presence of Al³⁺, Cr⁶⁺; whereas, in the presence of Pb²⁺, Tl⁺, CTAB, and DTAB and in the presence of UO₂²⁺, DTAB and Tween-20 co-migrate. In the presence of other metal cations (Ni²⁺, Cu²⁺, Co²⁺, Hg²⁺, Zn²⁺), mutual separation of CTAB, DTAB, and Tween-20 is always possible. Thus, certain metallic impurities in the surfactants have a detrimental effect on their chromatographic performance as a result of ionic interactions.

The lowest possible detectable amount of CTAB, DTAB, and Tween-20 on silica gel plates developed with M₁₉ was 1.45, 1.47, and 1.50 µg/spot, respectively. This indicates that the proposed TLC method is highly sensitive for the detection of surfactants.

The low values of standard deviation (S.D.) for CTAB (S.D. = 0.008), DTAB (S.D. = 0.014), and Tween-20 (S.D. = 0.014), obtained after resolving the mixture (CTAB, DTAB, and Tween-20) in six replicate R_F value measurements indicated the robustness of the TLC method developed.

Application

To widen the applicability of the proposed method, water

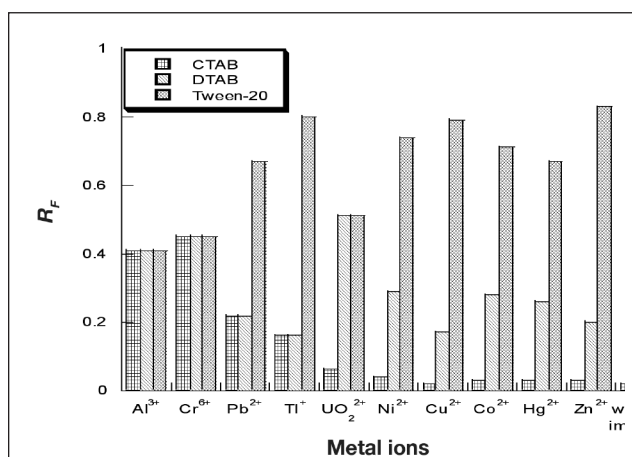


Figure 5. Effect of metal cations as impurities on the mutual separation of CTAB, DTAB, and Tween-20 on silica gel layers developed with 5% aqueous thiourea + acetone + methanol (60:20:20, v/v/v).

samples were spiked with CTAB, DTAB, and Tween-20. The results, presented in Table V, show that CTAB, DTAB, and Tween-20 can be easily identified in tap water and river water samples on silica gel plates developed with M₁₉.

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