ACID MODIFIED BABY POWDER BASED TALK – A SORBENT MATERIAL FOR THIN-LAYER CHROMATOGRAPHY

Soner Ergül
Ondokuz Mayıs University, Turkey

Abstract. Baby powder based talc (BP) is modified by keeping in 0.3 M HCl solution at room temperature for 24 hours. The product, acid modified baby powder based talc, is called "modified-BPT". Modified-BPT is investigated for their usefulness in the stationary phase of Thin-Liquid Chromatography (TLC). The separability of the components of a commercial ink has been tested on modified-BPT layers successfully. These results may be also said for the adaptability or validity on column chromatography.

Keywords: analytical chemistry, TLC, baby powder, modified baby powder talc, chromatography

Introduction

Chromatography is one of the most important analytical techniques used to separate components of mixtures. In chromatographic separations and teaching chromatography, the thin-layer chromatography (TLC) is often used as a quick, easy and separation method extensively used for organic species but rarely used for inorganic samples. While TLC is not common for inorganic cations, in the literature, some researchers have recently revealed that its utility is also valid for inorganic samples (Ergül, 2004; 2006; 2008; Mohammad et al., 2002; 2004; Mohammad & Jabeen, 2003; Mohammad & Agrawal, 2000; 2002; Mohammad, 1997; Mohammad & Khan, 1995; Ajwal et al., 1988; 1989a; 1989b; Mohammad & Fatima, 1987; Savaşcı & Akçay, 1999; Gürkan & Savaşcı, 2005).There are numerous applications of TLC for the qualitative and quantitative analysis of components of natural and artificial mixtures (Curtright et al., 1999; Baum & Shanks, 1975; Anwar, 1963; Goller, 1963; Rolins, 1963; Frodyma & Frei, 1969).

A number of minerals have previously been used as TLC adsorbents, including talc (Walsch, 1967), activated bentonite (Popov & Stefanov, 1968), kaolinite (Fayez et al., 1967), china clay (Sheen, 1971), activated bleaching earth (Hashimoto et al., 1965), modified perlite (Karakaş & Yüksel, 1998), acid modified diatomaceous earth (Ergül et al., 2005; Ergül & Savaşcı, 2008). Silica gel and alumina have been

used most frequently as adsorbents for the separation to components of the mixtures on TLC applications (Sthal, 1969).

Successful TLC separation depends on the properties of the sample, and also those of the mobile and stationary phases. Finding suitable resolution for a TLC application involves usually by changing the properties of mobile phase only. It does not involve by changing the properties of stationary phase although a possibility sorbent is present to change the properties of stationary phase (Ergül et al., 2005; Ergül & Savaşcı, 2008).

On TLC, adsorbent materials should be inexpensive and easily available. Besides, commercial baby powders are easily available products, and they are mixtures based on talc or corn starch as major components. Some commercial baby powders and their contents were given (Ergül, 2009). Talc as a major component of baby powder is a magnesium silicate mineral with the composition $Mg_3[Si_4O_{10}](OH)_2$ and a layered lattice structure. Its available surface for absorption markedly depends on particle size. Talc has been used as a TLC adsorbent to separate fatty acids, lanatosides, amino acids, the sugars and the flavonoids (Walsch, 1967; Sthal, 1969). In addition, the retention factors and separability of commercial ink components on thin layers prepared by acid modified diatomaceous earth (Ergül et al., 2005), baby powder based talc (Ergül, 2009) and silica gel $60GF_{254}$ (Ergül et al., 2005; Ergül, 2009) were examined and were discussed in the context of the variation of the stationary and mobile phases.

In practice, the modification of the baby powder based talc has not been investigated yet. Therefore, in this study, the baby powder (BP) based talc was modified by refluxing with acid processes. Modified BP (modified BPT) was used as stationary phase. The commercial ink samples were run on activated thin layers of acid modified BPT. The retention factors (R_F) and separabilty of commercial ink components were examined and discussed in the context of the variation of stationary and mobile phase properties and the adaptability or validity on column chromatography. In addition, this layers was compared with already known R_F, and separability of ink compenents on activated baby powder and Si-$60GF_{254}$ layers on cited literature.

Experimental

Chemicals, reagents, materials

Ammonia, n-butanol, acetic acid and ethanol were purchased from Merck (Darmstadt, Germany). Baby powder of Bebeten trademark was used to prepare modified BPT. Red and blue inks of Pelikan 4001 were used as the sample on TLC applications.

2 mol L^{-1} ammonia solution was prepared by adding distilled water to 150.4 mL of ammonia (d= 0.904 g/mL, 25%) on 1.0-L volumetric flask. Butanol-ethanol-2M ammonia (3:1:1, v/v) and butanol-acetic acid-water (12:3:5, v/v) mixtures were used as mobile phases. Modified BPT was used as stationary phases. The plates

were prepared using a Loughborough-Griffin&George, TLC Unikit (Leicestershire, England). All the chemicals were of analytical grade.

Modification of BP

200 mL of 0.3 mol L^{-1} HCl was poured into a beaker and BP (40 g <50 μm particle size) was added to it and then was shaken at room temperature for 24 h. After, this suspension was filtered and washed until the filtrate gave a negative reaction for Cl^-. The product was dried at 110 °C for 24 h and then sized with a 50 μm sieve. The product, acid modified baby powder based talc, was named modified-BPT.

Preparation of thin layer plates from modified-BPT

Slurries of modified-BPT in ethanol (1:1.5, w/v) were spreaded onto clean glass plates (7.5x15 cm) with a thickness of 250 μm using a spreader kit. Non activated plates were obtained by keeping the plates at room temperature for 2 h. They were activated by heating in an oven at 110°C for 1 h. For TLC applications, activated plates were used.

TLC applications

The red and blue ink samples and a mixture of both (1:1, v/v) were spotted with micropipettes on the starting line which was 2 cm from the bottom of the activated modified- BPT plates. The original spots on the layers were dried at room temperature for 5 min. A pencil line was marked 8 cm above the starting line of each plate. Two developing chambers measuring 10x50x20 cm were used. Sixty milliliters of butanol-ethanol-2M ammonia (3:1:1, v/v) was poured into one chamber and 60 mL of butanol-acetic acid-water (12:3:5, v/v) into the other. The lids of the chamber were closed and the chambers allowed standing for 25 min to ensure saturation of the air in each chamber with solvent vapors. Then, activated modified BPT plates with their ink samples were carefully immersed in the chambers. The plates, which were taken out of the chambers when the solvent fronts reached 8.0 cm above the starting line of each plate, were dried. The migration distances of the solvent (Z_f) and of each spot (Z_x) were then measured.

The Measurements of pH

10 % süspensions of BP, modified BPT and Si-60GF_{254} were individually prepared. The pH values of these süspansions, butanol-ethanol-2M ammonia (3:1:1, v/v) and butanol-acetic acid-water (12:3:5, v/v) mixtures were individually measured using pH meter. From the measurations, when the pH values of acidic and basic mobile phases were respectively 3.00 and 11.00, pH values of the slurries of Si-60GF_{254}, BP and modified-BP as stationary phases were respectively 7.00, 9.00 and 7.50.

Result

Successful TLC separation depends on the properties of the sample and also those of the mobile and stationary phases. Finding suitable resolution for a TLC application usually involves changing the properties of the mobile phase only. It does not involve changing the properties of the stationary phase.

Table 1. The R_f values for red and blue ink components obtained by using a acidic and basic mobile phases on activated Si-60GF_{254} (Ergül & Savaşcı, 2008; Ergül, 2009), BP (Ergül, 2009) and modified-BP layers

Component Couple	Si-60GF254		Activated BP		Modified-BPT	
	R_F^a	R_F^b	R_F^a	R_F^b	R_F^a	R_F^b
Pink	0.91±0.05	0.71±0.04	0.91±0.05	0.86±0.04	0.94±0.05	0.84±0.04
Yellow	0.38±0.02	0.40±0.02	0.67±0.03	0.72±0.04	0.34±0.02	0.73±0.04
Green	0.56±0.03	0.56±0.03	0.67±0.03	0.54±0.03	0.59±0.03	0.60±0.03
Dark Blue	0.49±0.03	0.56±0.03	0.08±0.01	0.05±0.01	0.09±0.01	0.11±0.01

a: Butanol:acetic acid:water mixture (12:3:5, v:v:v), **b:** Butanol-ethanol-(2M) ammonia mixture (3:1:1, v/v) Number of repeated runs: 3

In this study, to prepare a new stationary phase, the baby powder based talc was modified by refluxing with 3M HCl and modified BPT was used as stationary phase for normal-phase TLC applications of commercial red and blue ink samples. Because of the ink components being acidic, basic or neutral, and also soluble in water, basic butanol-ethanol-2M ammonia (3:1:1, v/v) and acidic butanol-acetic acid-water (12:3:5, v/v) mixtures were used as mobile phases. The chromatograms were given in Figs 1- 2 for the activated modified BPT plates. The developed spots of ink compenents could be determined easily with the naked eye. The retention factors (R_F) of the ink components were calculated from $R_F=Z_x/Z_f$. The same procedure was also applied to the activated BP and the modified-BPT layers. From the chromatograms, the R_F values of ink components were given in Table I.

Separability on TLC and expected resolution on CC

On TLC application, the resolution is relation to separability parameter (*r*) in context of different between R_F values of component couple. In addition, the separations by TLC are precursor for the applications on column chromatography. The retention factors and separability of components in the sample using TLC is related to the elution time and separability on column chroma-

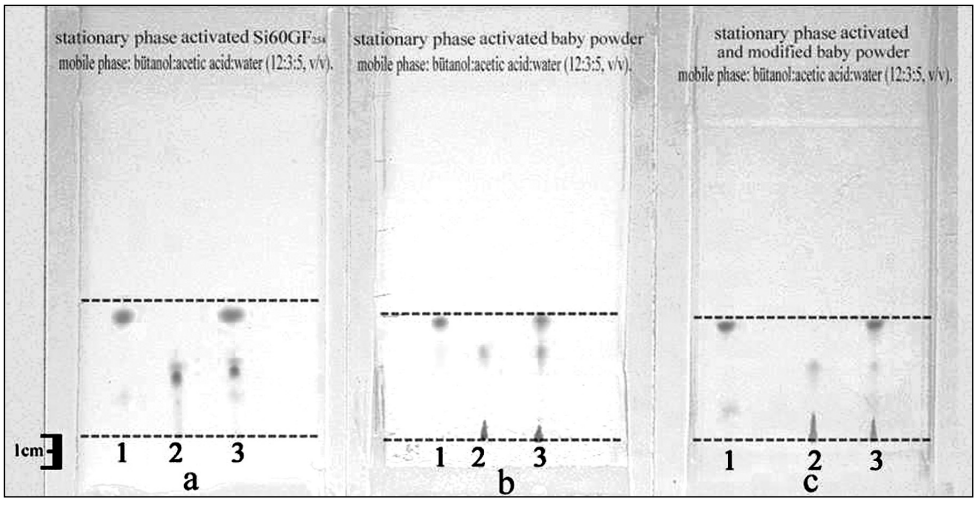

Figure 1. Chromatograms of ink samples with butanol-acetic acid-water mixture (12:3:5, v/v). **(a)** on activated Si-60GF$_{254}$ plate **(b)** on activated BP plate **(c)** on activated modified-BPT plate [1: red ink, 2: blue ink, 3: mixture of 1 and 2]

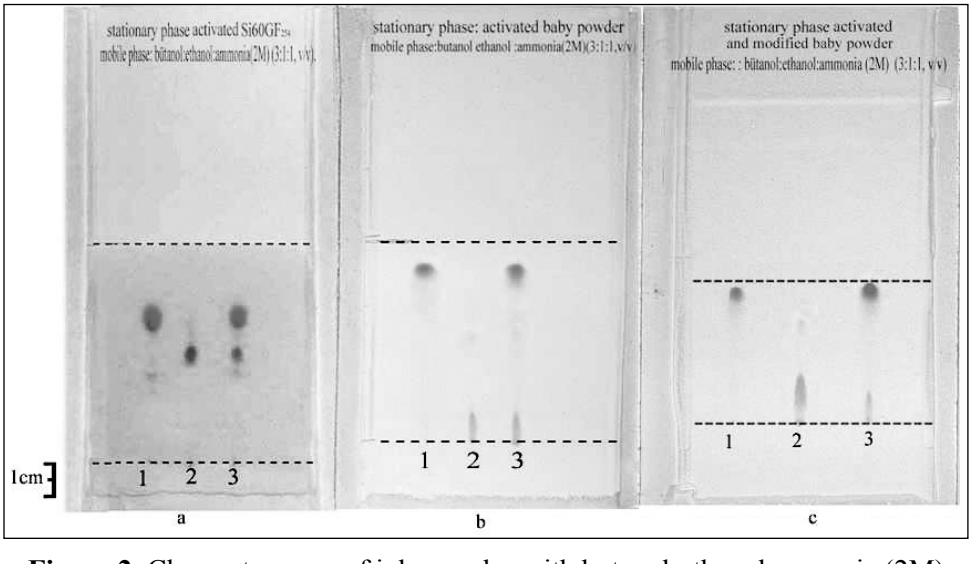

Figure 2. Chromatograms of ink samples with butanol-ethanol-amonnia (2M) mixture (3:1:1, v/v). **(a)** on activated Si-60GF$_{254}$ plate **(b)** on activated BP plate **(c)** on activated modified-BPT plate [1: red ink, 2: blue ink, 3: mixture of 1 and 2]

tography. In this context, the *r* parameter is quantitatively the measurement of expected resolution on column chromatography in this context of the separability of components on thin layer chromatography. It is calculated using Eq. (1) (Duncan, 1962).

$$r = \frac{a}{b + 0.1a} \quad (1)$$

In Eq. (1), *a* is R_F value of the fast moving substance A and *b* is R_F value of the slow moving substance B on the chromatograms respectively. The separability parameter is dimensionless number, which indicates whether separation on column chromatography is carried out or not. When mobile and stationary phases are same for column and thin layer chromatographic systems, it is said that the separation of two components may be possible when *r* is >1 (Duncan, 1962). In this study, *r* values of red and blue ink component couples obtained on thin layer chromatographic applications were given in Table 2.

Table 2. Expected resolution data on CC of component couples in ink samples

Component couple	Si-60GF254		Activated BP		Modified-BPT	
	ra	rb	ra	rb	ra	rb
Pink-Yellow	1.932	1.507	1.196	1.067	2.166	1.032
Pink-Green	1.398	1.125	1.196	1.374	1.374	1.228
Pink-Dark Blue	1.566	1.125	5.322	6.324	5.109	4.330
Green- Dark Blue	1.026	0.909	4.558	5.192	3.960	3.529
Green-Yellow	1.284	1.228	0.909	1.177	1.479	1.085
Yellow-Dark Blue	1.142	0.909	4.558	5.902	2.742	3.989

a: Butanol:acetic acid:water mixture (12:3:5, v:v:v), **b:** Butanol-ethanol-(2M) ammonia mixture (3:1:1, v/v) Number of repeated runs: 3.

Discussion

Evalation of Modification

To prevent interference and the reacting of activated BP layer with acidic mobile phase was modified by refluxing with 3M HCl solution. The reacting and interfering inorganic compounds (such as ZnO, magnesium stearate) were converted to soluble salts and removed from baby powder based talc by refluxing with acid processes. When pH value of baby powder based talc was 9.0, pH of modified BPT was 7.5. Through the modification process, the pH of the baby powder based talc changed from 9.0 to 7.5. Although the layers of baby powder based talc react with acidic mobile phase, modifie BPT layers do not

react with acidic mobile phase. In addition, **c**hromatographic parametres (such as R_F and *r*) of both layers were differend from each other. These qualitative and quantitative indicators suggested that some interfering organic and inorganic compounds had been successfully removed and mofidifaciton was succcesfuly carried out.

Evalation of Chromatograms

As shown in Figs 1 – 2, on the activated BP and modified BPT layers, the red ink was separated into yellow and pink spots when the solvent front values were ≥2.0 cm for both acidic and basic mobile phases. The blue ink was separated into two main spots (dark blue and green) on running with the acidic and basic mobile phases when the solvent front was ≥8.0 cm. However, the blue ink components were not separated even when the solvent front was ≥8.0 cm on running with basic mobile phase on Si-60GF_{254} layer. In addition, dark blue spot has undergone to tailing, the other spots have not. The reason for tailing lies in the fact that it is related with both load capacity of the stationary phase and the amount of the components in the spots. Although the same amount of sample has been spotted to all of three kind layers, the dark blue spot on activated BP and activated modified BPT layers has been developed with tailing on running with acidic mobile phase, but the one on Si-60GF_{254} has been developed without tailing. This situation indicates that the load capacity of Si-60GF_{254} layer for dark blue is higher than those of activated BP and activated modified BPT layers. On the other hand, the area of dark blue spot is larger than those of green for three kinds of layers. This observation reveals that the amount of dark blue component on the sample is higher than those of green.

As shown in Figs 1 – 2, the chromaogram in Fig. 2b obtained with basic mobile phase on the activated BP layer is beige although the bottom section chromatogram in Fig. 1b obtained with acidic mobile phase on the activated BP layer is tight and turns into dirty beige. Those spoilt structures and color changing are qualitative indicators of chemical interaction of the stationary phase with mobile phase. The spoilt structure and color change are results of chemical interaction between the talc, $Mg_3[Si_4O_{10}](OH)_2$, on activated BP layer and the CH_3COOH in acidic mobile phase. On the other hand, no physical deformation and color change have been observed on the activated Si-60GF_{254} and modified BPT layers qualitatively. Consequently, it can be said that the modified BPT and Si-60GF_{254} layers do not react with acidic and basic mobile phases although the activated BP layer undergoes to chemical decomposition on the application with the acidic mobile phase. However, it is possible to conclude that layers of activated BP layer have been successfully applied to the separation of components of ink samples also.

Effect of mobile phase changing to R_F

In Table I, for modified BPT layers, when the acidic mobile phase was replaced by the basic one, R_F value changed from 0.34 ± 0.02 to 0.73 ± 0.04 for the yellow component. R_F values for the pink, dark blue and green components did not change significantly although acidic mobile phase was replaced by the basic one. For Si-60-GF_{254} layers, when the acidic mobile phase was replaced by the basic one, R_F values changed significantly from 0.91 ± 0.05 to 0.71 ± 0.04 for the pink component. R_F values for the yellow, dark blue and green components did not change although mobile phase changed by basic one. For the activated BP layers, when acidic mobile phase was replaced by basic one, R_F values of components of ink samples change. On the basis of the experimental data and their changing patterns, the polarity, acidic and basic properties of the mobile phase was effective in contributing to the chromatographic behavior of all the dye components because of water soluble property.

Effect of Stationary Phase Changing to R_F

In Table I, when the modified BPT layer was replaced with the activated BP one, when using acidic mobile phase, the R_F value changed significantly from 0.34 ± 0.02 to 0.67 ± 0.03 for the yellow component only. R_F values for the pink, dark blue and green components did not change significantly by both acidic and basic mobile phases although the modified BPT layer was replaced by the activated BP one. On the other hand, when the modified BPT layer was replaced with the activated Si-$60GF_{254}$ one, when using acidic mobile phase, the R_F value changed significantly from 0.09 ± 0.01 to 0.49 ± 0.03 for the dark blue component. In addition, when the modified BPT layer was replaced with the activated Si-$60GF_{254}$ one, when using basic mobile phase, the R_F values changed significantly the dark blue, pink and yellow components. R_F value for the green component did not change significantly by basic mobile phase although the modified BPT layer was replaced by the activated Si-$60GF_{254}$ one. The chemical composition, pH and physical properties of the modified BPT, activated BP and Si-$60GF_{254}$ are significantly different from each others. Thus, these data and chromatographic behavior patterns could not be explained easily. As a result, the retention of various molecules on stationary phase is generally explained by considering the interactions between polar Si-OH groups on surface of layer and molecules in sample, e.g., hydrogen bridging, dipole-dipole and Van der Waals. Thus, the change of R_F values of ink components on TLC applications on both adsorbents is related to surface activity of the layers.

Separability on TLC and expected resolution on CC

The data for the red and blue ink components obtained by using the acidic and basic mobile phases on the activated Si-$60GF_{254}$, the activated BP and the modified BPT layers are given Tables II. In Table 2, the r values for pink-yellow couple which belong to red ink on the activated Si-$60GF_{254}$, the activated BP and the modified

BPT layers, when using acidic mobile phase are 1.932, 1.196 and 2.166, respectively. In Table 2, the r values for pink-yellow couple on the activated Si-60GF_{254}, the activated BP and the activated BPT layers, when using basic mobile phase are 1.507, 1.67 and 1.032, respectively. Because the r values are >1.0 for pink-yellow couple, these chromatographic systems are effective to separate this couple. As a result, pink and yellow spots belong to red ink are successfully separated on all of chromatographic systems.

In Table 2, the r values for green-dark blue couple which belongs to on the activated Si-60GF_{254}, the activated BP and the modified BPT layers, when using acidic mobile phase are 1.026, 4.558 and 3.960, respectively. *r* values for green-dark blue couple which belongs to on the activated Si-60GF_{254}, the activated BP and the modified BPT layers, when using basic mobile phase is 0.909, 5.192 and 3.529, respectively. Yet these components of blue ink have developed only one spot without separation on activated Si-60GF_{254} layer. Because r value is >1.0 for green-dark blue-green couple, the activated BP and the activated BPT layers are successful to separate this couple on TLC applications using both kinds of mobile phases. However, Si-60GF_{254} layer is not successful to separate this couple because of r <1.0 (r=0.909) and spot overlapping. Consequently, to separate the components of blue ink, the activated BP and activate BPT layers are more successful than Si-60GF_{254} layers on TLC applications using both kinds of mobile phases.

As seen in Table 2, *r* parameters of pink-yellow, pink-green, pink-dark blue, green-dark blue, green-yellow and yellow-dark blue ink component couples were bigger than 1.000 on all of chromatographic systems using the activated modified-BPT layers. Therefore, it can be said that all of ink component couples were successfully separated on all of thin layer chromatographic systems using the modified BPT layers because of $r > 1.000$. In addition, the best analytical separations of pink-yellow and green-yellow ink component couples on all of the chromatographic applications were obtained when using acidic mobile phase butanol:acetic acid:water mixture (12:3:5, v:v:v) as mobile phase on the activated modified BPT layer.

As seen in Table 2, *r* parameters of pink-yellow, pink-green, pink-dark blue ink compenent couples were bigger than 1.000 on all chromatographic systems. Therefore, it can be said that these complex couples were successfully separated on all TLC systems because of $r > 1.000$. In addition, the best analytical separation of pink-green ink compenent couple was obtained when using butanol:acetic acid:water mixture (12:3:5, v:v:v) as mobile phase on the activated Si-60GF_{254} layer. The best analytical separations of pink-dark blue, green-dark blue and yellow-dark blue ink compenent couples were obtained when using butanol-ethanol-(2M) ammonia mixture (3:1:1, v/v) as mobile phase on the activated BP layer.

As seen in Table 2, *r* parameters of green-dark blue, green-yellow and yellow-dark blue were smaller than 1.00 on some chromatographic systems. On these

chromatographic systems, the mobile phase was butanol-ethanol-(2M) ammonia mixture (3:1:1, v/v) for green- dark blue and yellow-dark blue ink component couples when the stationary phase was activated Si-60GF_{254} phase. In addition, *r* value for green-yellow ink component couple was smaller than 1.0 when using butanol:acetic acid:water mixture (12:3:5, v:v:v) as mobile phase on activated BP layer. As a result, although these chromatographic systems were not successfully separated ink component couples cited, other chromatographic systems were successfully separated ink component couples. The mutual separation for all of ink components was obtained when using butanol:acetic acid:water mixture (12:3:5, v:v:v) as mobile phase on activated Si-60GF_{254} and modified BPT layers. In addition, the mutual separation for all of ink components was obtained when using butanol-ethanol-(2M) ammonia mixture (3:1:1, v/v) as mobile phase on activated BP and modified BPT layers. As a result, the best mutual separations are carried out on these chromatographic systems cited. It can be said that this result is also valid for column chromatography in context of *r* parameters of ink component couples.

Conclusions

This work was carried out to modifie baby powder based talc and to investigate the useful for TLC and the adaptability or validity on column chromatography of modified BPT. In light of these studies, conclusions are as follows: (a) the baby powder based talc was succesfully modified to the modified BPT by refluxing with 3 M HCl. Being different of the pH values and chromatographic properties of BP and modified BPT are quantitative indicators of modification; (b) the modified BPT and Si-60GF_{254} layers do not react with acidic and basic mobile phases although the activated BP layer undergoes to chemical decomposition on the application with the acidic mobile phase; (c)pink and yellow spots belong to red ink are successfully separated on all of chromatographic systems; (d) to separate the components of blue ink, the activated BP and activate BPT layers are more successful than Si-60GF_{254} layers on TLC applications using both kinds of mobile phases; (e) the best analytical separations of pink-yellow and green-yellow ink component couples on all of the chromatographic applications were obtained when using acidic mobile phase butanol:acetic acid:water mixture (12:3:5, v:v:v) as mobile phase on the activated modified BPT layer.

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✉ **Dr. Soner Ergül**
Department of Science Education
Ondokuz Mayıs University
55200, Atakum Yerleşkesi-Samsun, Turkey
E-mail: sergul@omu.edu.tr