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To Identify the Concentration Level of Various Pigments & to Determine Suitable Solvent System for Different Lipstick Samples by Using TLC

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Abstract

30 lipstick samples of different brands of similar color were selected for this study coloring agent was analyzed by thin layer chromatography (TLC) and UV. Using four different solvent systems [Toluene/Benzene (12:8), Toluene/Acetone (16:4), Toluene/Benzene/Cyclohexane (4:12:4), Toluene/Benzene/Diethyl ether (12:6:2)]. Lipstick samples of colors indistinguishable on visual observation could be grouped into eight subgroups. The work that follows goes on this line of investigation. The study compares the usefulness of two kinds of fluorescent reagents -Yellowescent Fluorescent Latent Prints Powder and Nile Red- for developing latent lip prints, older than one and a half year, on multi colored surfaces. The reagents were used in powder form and luminescence was observed by an alternate light source and ultraviolet light. In principle Nile Red, like lysochromes, have advantage over other Fluorescent powders because it react with fats and physical agents.

The main purpose of this study is to identify concentration level of various pigments & to determine which solvent system is best for the particular brand of the lipstick using Thin Layer Chromatography by checking the differences in the respective chromatogram & UV.

Keywords: Lipstick; Lip-print; Crime scene; Forensic analysis; Solvent system; UV; TLC; Forensic chemistry

Introduction

Cosmetic evidence such as lipstick recovered from a crime scene can prove useful to link a suspect with the victim or crime scene and therefore need to be carefully analyzed during crime investigation. At present with an aesthetic purpose or also for protection of the lips, the use of protecting lipsticks and permanent lipsticks is more and more widespread. These lipsticks do not leave any visible print but can make a latent one. This characteristic has opened a field of investigation about the possibility of developing latent lip prints, and some results have been already obtained [1-5].

Forensic scientists use cosmetics lipstick prints and smears as evidence for solving crimes. Traces of lipstick, lipstick smears could be found left on drinking cups, glasses, cigarette butts, tissue papers or handkerchief. In certain cases, trace amount of lipstick was transferred to the clothing of perpetrator who attacked a female. By comparing the composition of a lipstick smear with that of a victim, forensic scientists can demonstrate indirect proof of contact or a relationship between victim and suspect. Also, it is sometimes possible to extract saliva DNA from the print. Thus, forensic analysis of lipsticks was often found to be crucial in the investigation of criminal cases. The identification and determination of components in a lipstick sample have to be conducted with rapid methods.

Lipsticks contain wax, oil and coloring agents as three main ingredients. Wax enables the adjustment of the staying power properties to heat and hardened texture on application. Meanwhile, oil provides shiny and glide quality. During manufacturing process, the number of dyes or coloring agents used is limited and combination of dyes give rise to variation in shade. Coloring agents can be either synthetic or natural dyes which can further be categorized into oil-soluble or water soluble dyes. Therefore, lipstick of same color may contain varied coloring agents [6-14].

Various methods of forensic lipstick analysis were reported. Small amount of lipstick (approximately 10 μg) could lead to good comparisons in TLC. Oil soluble and water soluble dyes were separated in different solvent systems respectively. Several authors have described various TLC solvent systems for separation in respective studies. In addition, TLC gives rise to different retardation factor (Rf) value for different components present in a lipstick sample.

Materials and Methods

Jar with lid, Lipstick samples (Branded & Local), TLC Plates, Mobile Phase/Solvent Phase, Clean Cotton Piece, Sterilized Scissor, Fine capillaries, Ruler, Pencil, Iodine pellets / U.V lamp, Titer plates, Graduated cylinder, Gloves, Protective Mask and Thirty samples of lip impressions bearing lipstick were obtained on cloth using various brands and types of lipsticks, which were available from volunteers [15-23].

Thin-Layer chromatography

Thin-Layer Chromatography (TLC) is a method use to separate components from each other in a mixture. It is normally experimented on glass, aluminum foil or plastic which is coated by some kind of absorbent material (ex: silica gel, aluminum oxide). It takes the RF

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values of each color component and compares them; RF values are experiments that depend on the polarity of the substance on the paper chromatography [23,24].

1. A piece of cotton cloth was washed in a detergent solution, immersed in a hot water, and dried.
2. This cloth was cut into small pieces (2 cm×2 cm) and lipstick smears were rubbed onto these pieces.
3. The stained areas from the cloth pieces were cut and transferred into serially marked small bowls.
4. These samples were mixed with an extracting solution and each bowl was then shaken for about 5-10 minutes to remove the stain from the cloth piece.
5. Cloth piece is taken out, and the extract was used for further examination with TLC.
6. Obtain jar with lid, piece of filter paper and a TLC plate (20 cm×20 cm). Handle TLC plate by the edges only, avoid touching the white silica layer.
7. With a pencil and ruler, GENTLY draw a line across the short side of the TLC plate about 1.5 cm from the bottom of the plate. At even intervals label the top of the plate with the letters: 4×1B, 2B, 3B, 4B, 5B, 6B for six different well known brands of different shades of Revlon lipsticks on a single TLC plate. Similarly 1L, 2L, 3L, 4L, 5L, 6L for six different local brands of lipsticks.
8. Using a capillary, place a dot of each lipstick sample along the bottom pencil line directly under the corresponding label on the top of the plate, the dots should be about 0.2 cm in diameter and dark enough to be clearly visible.
9. Using measuring cylinder, dispense approximately mentioned ratio of mobile phase/solvent into the jar. The mobile phase/solvent should be about 0.5 cm deep. Place a small piece of filter paper in the jar, secure the lid and tilt the jar to saturate the filter paper with mobile phase/solvent.
10. Carefully insert the TLC plate into the jar, sample end down. The lipstick dots must be above the mobile phase/solvent. Secure the lid.
11. Allow the mobile phase/solvent to rise to within one cm of the top of the plate (5-10 minutes). Watch – do not allow the mobile phase to rise to the very top of the plate. Remove the TLC plate and mark the solvent front with a pencil.
12. Measure the distance the mobile phase/solvent moved in cm (the distance from the spotted pencil line to the solvent front end). Also measure the distance in cm each component of the lipsticks moved from the spotted pencil line. Some lipsticks have only two or three components, and some have more. Enter these measurements on the Data Table.
13. Determine the Fro for each lipstick component of all lipstick samples. Enter those values on the Data Table. To calculate the Fro value, divide the distance traveled by each lipstick component by the distance traveled by the solvent.

$$\text{Fro} = \frac{\text{Distance traveled by one lipstick component from the spotted pencil line}}{\text{Distance the solvent moved from the spotted pencil}}$$

Mobile or solvent phase

Samples are run for both the samples of branded and local lipstick. Use hood if possible. Self life is about one month (Table 1).

Lipstick examined

(Table 2).

For local brand of lipstick

(Table 3).

Steps followed

(Figures 1,2).

Solvent system

Toluene/Benzene (Figures 3-5).

Solvent system

Toluene / Acetone (Figures 6,7).

Solvent system

Toluene/Benzene/Diethyl ether (Figures 8,9).

Solvent system

Toluene/Benzene/Cyclohexane (Figures 10,11).

Sample No.	Solvent System
1	Toluene/Benzene (12:8)
2	Toluene/Acetone (16:4)
3	Toluene/Benzene/Cyclohexane (4:12:4)
4	Toluene/Benzene/Diethyl ether (12:6:2)

Table 1: Solvents used.

Sample No.	Name & Number	Brand	Color
1B	Frost Rose Pearls 334	Revlon	Pink
2B	Frost Coffee Bean 66	Revlon	Brown
3B	Flirtatious Violet 111	Revlon	Violet
4B	Crème Vive Violet 379	Revlon	Purple
5B	Cherry Sparkles 57	Revlon	Bright Red
6B	Bridal Dream 104	Revlon	Maroon

Table 2: Branded Revlon Lipstick Shades.

Sample No.	Name & Number	Brand	Color
1L	Biros 14	Local	Pink
2L	Biros 8	Local	Light Brown
3L	Matte Lip Color 293	Local	Violet
4L	Aver Matte 147	Local	Purple
5L	Iffy Super Soft 15	Local	Red
6L	Iffy Matte Soft 17	Local	Maroon

Table 3: For Local Brand of Lipstick.

Results

The data collected from the chromatograms were recorded, and the Fro values were calculated using distance travelled by the solute (Table 4-11).

Figure 1: Samples rubbed on a clean cotton piece.

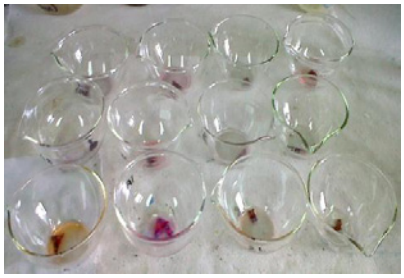

Figure 2: Bowls contains the extracted sample.

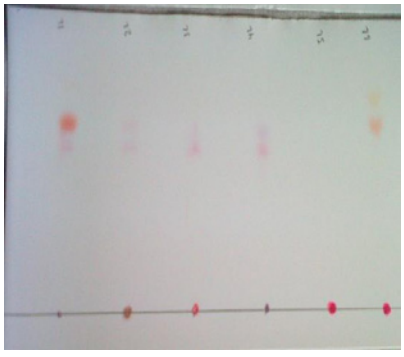

Figure 3: Spots seen when the sample in run in a solvent system (Toluene/Benzene).

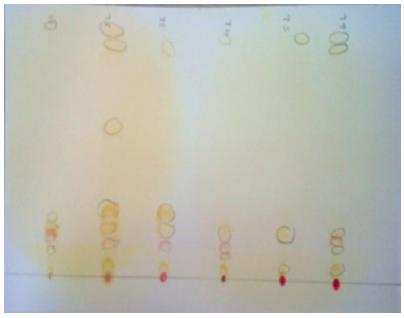

Figure 4: Spots seen for local brand of Lipstick when viewed under Iodine Fumes.

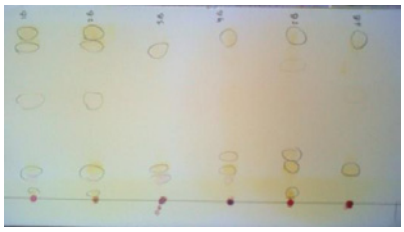

Figure 5: Spots seen for the Revlon lipstick when viewed under Iodine fumes.

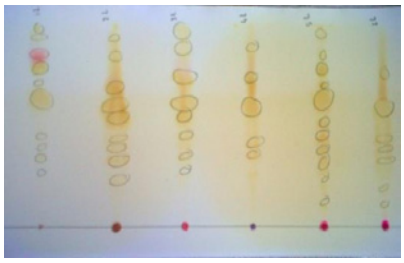

Figure 6: Spots seen for local brand of Lipstick when viewed under Iodine Fumes.

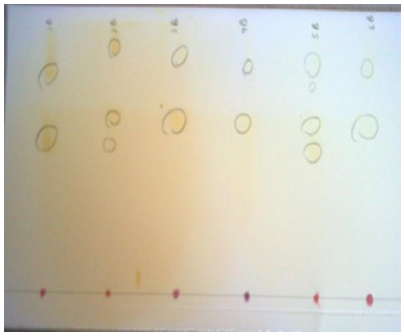

Figure 7: Spots seen for the Revlon lipstick when viewed under Iodine fumes.

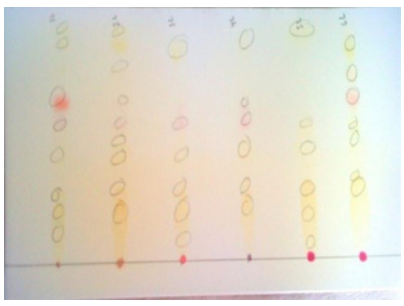

Figure 8: Spots seen for local brand of Lipstick when viewed under Iodine Fumes.

Discussion and Conclusion

The conclusion of the above work done is that with the help of Thin Layer Chromatography, the branded Revlon lipstick separates best in a solvent system (Toluene/Benzene) and local brand of lipstick separates best in a solvent system (Toluene/Acetone).

When viewed under UV light the more number of spots are observed in the local brand of different color of lipsticks, where as for

the branded Revlon lipstick less number of spots are seen which are comparatively less soluble in the solvent system used. It is possible that when working on very dark or multicoloured surfaces problems of contrast may make it difficult to visualize a lip print. In these cases it could be advantageous to use a fluorescent reagent to avoid those problems. Previous studies indicate that Nile Red is useful for developing old prints on different dark colour surfaces, specifically blue, red and black.

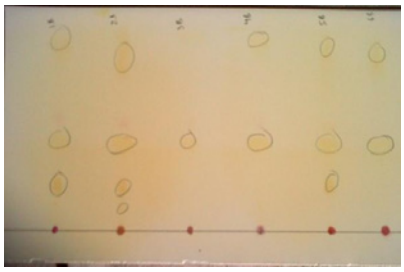

Figure 9: Spots seen for the Revlon lipstick when viewed under Iodine fumes.

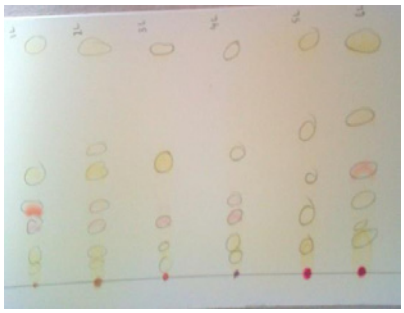

Figure 10: Spots seen for local brand of Lipstick when viewed under Iodine Fumes.

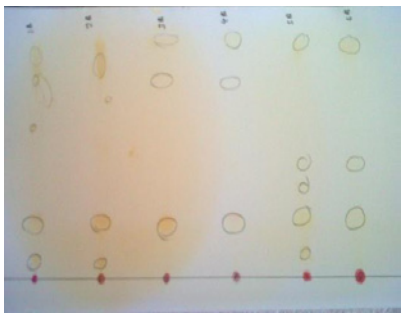

Figure 11: Spots seen for the Revlon lipstick when viewed under Iodine fumes.

Sample No.	Number Of Spots	her
1B	2	47 68
2B	3	47 54 77
3B	2	54 75
4B	2	53 72
5B	4	44 53 65 73
6B	2	53 73

Table 4: For Branded Revlon Lipstick in solvent system Toluene/ Acetone (16/4).

Sample No.	Number of Spots	her
1L	10	21 26 32 35 50 56 63 69 74 78
2L	9	18 26 31 35 43 48 55 67 75
3L	8	23 28 36 44 48 60 72 78
4L	5	28 33 47 60 73
5L	11	10 18 25 30 36 42 50 56 63 68 78
6L	7	9 15 26 31 35 48 62

Table 5: For Local Brand of Lipstick in solvent system Toluene/ Acetone (16/4).

Sample No.	Number Of Spots	her
1B	5	5 17 49 65 73
2B	4	5 17 59 70
3B	3	15 65 78
4B	3	17 64 77
5B	5	7 20 28 37 77
6B	3	20 37 77

Table 6: For Branded Revlon Lipstick in the solvent system Toluene/ Benzene/ Cyclohexane (4/12/4).

Sample No.	Number Of Spots	her
1L	6	6 10 19 25 35 78
2L	7	6 10 18 25 36 44 76
3L	5	5 12 19 39 75
4L	6	6 13 20 25 42 74
5L	5	10 20 31 46 75
6L	6	13 16 25 34 52 75

Table 7: For Local Brand of Lipstick in the solvent system Toluene/Benzene/ Cyclohexane (4/12/4).

Sample No.	Number Of Spots	her
1B	5	3 13 45 67 73
2B	5	3 13 45 67 75
3B	3	10 15 67
4B	4	10 13 20 73
5B	5	5 16 23 63 75
6B	3	15 50 74

Table 8: For Branded Revlon lipstick in the solvent system Toluene/Benzene (12/8).

The work that follows goes on this line of investigation. The study compares the usefulness of two kinds of fluorescent reagents -Yellowescent Fluorescent Latent Prints Powder and Nile Red- for developing latent lip prints, older than one and a half year, on multicoloured surfaces. The reagents were used in powder form and luminescence was observed by an alternate light source and ultraviolet light. In principle Nile Red, like lysochromes, have advantage over other Fluorescent powders because react with fats and physical agents.

Lip-print comparison need be addressed in the forensic science community before it can be regarded by an accepted technique. Our method of development can be useful to help establish the validity of the technique. Also the possibility to obtain DNA from a latent lip print could be other useful application for crime investigation.

Sample No.	Number Of Lipstick	her
1L	5	9 13 16 19 82
2L	6	12 16 23 48 76 81
3L	4	10 15 21 75t
4L	4	8 12 15 79
5L	3	4 15 79
6L	7	4 10 13 16 38 75 79

Table 9: For Local Brand of Lipstick in solvent System Toluene/Benzene (12/8).

Sample No.	Number Of Lipstick	her
1B	3	17 35 75
2B	4	9 17 35 69
3B	1	35
4B	2	35 75
5B	3	17 34 73
6B	2	33 70

Table 10: For Branded Revlon Lipstick in the solvent system Toluene/Benzene/ Diethyl ether (12/6/2).

Sample No.	Number Of Spots	her
1L	8	10 19 27 38 47 57 76 82
2L	8	17 26 36 43 47 56 68 81
3L	6	8 17 26 37 48 75
4L	6	17 26 38 48 55 78
5L	6	6 16 25 38 47 82
6L	7	25 28 42 46 56 65 76

Table 11: For Local Brand of Lipstick in the solvent system Toluene/ Benzene/ Diethyl ether (12/6/2).

It is hypothesized that through Thin Layer Chromatography analysis & UV light analysis of the various pigments of Lipstick in different solvent system will provide characteristic data to determine the best solvent system for a particular lipstick.

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