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Analysis of lipsticks using Raman spectroscopy

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ABSTRACT

In this study, 80 lipsticks were obtained and evaluated using Raman spectroscopy at excitation wavelengths of 532 and 780 nm. Fluorescence severely limited analysis with the 532 nm line while the 780 nm line proved useful for all samples analyzed. It was possible to differentiate 95% of the lipsticks evaluated based on one or more Raman peaks. However, there were no peak trends observed that could be used to identify a manufacturer or categorize a sample. *In situ* analysis of lipstick smears was found to be possible even from several Raman active substrates, but was occasionally limited by background fluorescence and in extreme cases, photodegradation.

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1. Introduction

Cosmetic smears are a type of transfer evidence that can be commonly found at a crime scene. Due to their ease of transfer and prevalence of use, deposits of cosmetics can be encountered on clothing or bedding in rape cases [1], as smears on glasses, cups or cigarettes [2], or even as a medium for writing threatening messages [3]. Identification of cosmetics has been part of forensic investigations since 1912 when Edmund Locard identified pink dust under a homicide suspect's fingernail as being chemically consistent with a face powder found in the victim's room [4]. Though conclusive identification based on a transferred cosmetic smear is impossible due to mass production, cosmetics may be used to form a link between a suspect or victim and a crime scene, corroborate statements or assist in crime scene reconstruction. Additional information obtained from the cosmetic smear, such as brand or type of product, could strengthen the evidential value of cosmetic comparison, especially considering the vast diversity of cosmetic products.

Regulation of cosmetics falls under the jurisdiction of the United States Food and Drug Administration (FDA), but is significantly less stringent than regulation of other products. The FDA does not require a cosmetic product be pre-approved for sale, leaving the burden of testing product safety to the cosmetic companies. The FDA has reported more than 5000 ingredients used in the manufacturing of cosmetics [5]. An analytical scheme for comparably colored cosmetic samples may include high-performance liquid chromatography (HPLC) [1] or thin-layer chromatography (TLC) [2] for indication of dye components, scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDX) [6] for elemental analysis, gas chromatography/mass spectrometry (GC/MS) for examination of oils and waxes, and fluorescence microscopy [2] or infrared (IR) spectroscopy for analysis or comparison of chemical composition [6]. Because of the extreme variation in ingredients, a combination of instrumental techniques is typically needed to reach a high discriminatory power for forensic comparison and identification. One possible complementary instrumental technique for cosmetic analysis is Raman. Lipsticks were selected as the focus of this study.

In recent years, technological advances to Raman spectroscopy have made it increasingly advantageous for forensic science. These include the development of portable Raman spectrometers which could allow for presumptive identification of compounds in the field [7], advancements in optics and optical alignment which help improve signal quality [8], and the coupling of the Raman to a microscope, which made analysis of trace samples possible. Raman spectroscopy also has the advantage of being non-destructive. Raman spectrometry has been used successfully in examination of materials such as fibers [9,10], paints [11–13], drugs [14], and body fluids such as blood [15] and semen [16]. While Raman databases for many compounds are available online [17–19], the variety of ingredients in cosmetics limit the use of these databases for forensic identification.

To our knowledge, only one other report on lipstick identification using dispersive Raman spectrometry exists [20]. Salahioglu and Went showed Raman spectra of lipstick to be stable and

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reproducible over periods of at least one year. They used the 632.8 nm laser source for analysis that was sometimes hindered by fluorescence effects. Nonetheless, it was possible to differentiate between many samples. Their study was also somewhat limited in that it did not account for samples found on substrates or compare wavelengths used for analysis. In a separate 1998 study, the possibility for using surface enhanced Raman spectrometry (SERS) was evaluated for use in examining five red lipsticks on a cotton substrate [21]. While the authors were successful in identifying lipsticks, the added expense and sample preparation makes this technique less desirable than conventional Raman spectroscopy. In this study the lipsticks were analyzed, excitation wavelengths were compared, and the possibility for *in situ* analysis on Raman active substrates was evaluated.

2. Materials and methods

Samples of lip cosmetics were obtained *via* donations. The 80 samples were from 23 different manufacturers and 40 different product lines. Samples were analyzed using a Horiba Labram HR800 confocal Raman microscope with a 532 nm laser and a Thermo Scientific DXR Smart Raman equipped with a 780 nm laser. The Horiba instrument used a 10× objective with a fixed laser output of 60 mW. The Thermo Scientific instrument used a laser set to 50 mW and the 25 μm slit. Pure samples were analyzed as smears on aluminum foil, which had no Raman signal. A typical spectrum was taken as 10 two-second scans for a total exposure time of 20 s. To analyze the influence of substrates cosmetic smears were applied to fabric or other substrates and analyzed *in situ*. Samples were scanned from 50 to 3400 cm^{-1} and compared *via* spectral overlay for chemical differences. Band presence, absence, location and shape were considered during spectral comparison. Excluding subtraction of a constant baseline and multiplication by a constant, no smoothing or corrections for background fluorescence were used on the spectra.

Table 1
Samples for intra-product comparison studies where samples from the same manufacturer and product line in different colors were compared.

Manufacturer	Product line	Number of samples
Mary Kay	Signature	10
Avon	ANEW	5
Avon	Beyond Color	5
Avon	Ultra Color Rich	5
Lancome	Rouge Sensations	5
Clarins	Rouge Prodige	4
Lancome	Rouge Absolu	3
Nars	Lipstick	3
NYC	Ultra Last Lipwear	3
Elizabeth Arden	Exceptional	2
Maybelline	Moisture Extreme	2
NYC	Ultra Moist	2
Revlon	Superlustrous	2

2.1. Evaluation of source laser

A total of 14 lipstick samples were analyzed as smears on aluminum foil using both the 532 and 780 nm lines. The spectra were compared *via* spectra overlay to determine preferred wavelength for analysis.

Table 2
Sample information for the color study samples where similarly colored samples from different product lines and manufacturers were compared.

Color group	Manufacturer	Product line	Color
Pink	Avon	Beyond Color	Electric Pink
	Maybelline	Wet Shine	Pink Splash
	Loreal	Endless	Pink Passion
	Revlon	Superlustrous	Cherry Blossom
	Avon Canada	Beyond Color	Petal
	Avon	Ultra Color Rich	Carnation
	Mary Kay	Signature	Sweet Nectar
	NYC	Ultra Last	Smooch
	NYC	Ultra Moist	Petal
	Maybelline	Moisture Whip	Misty Lilac
Brown	Clinique	Different	Spiced Apple
	Clarins	Rouge Prodige	Fig
	Lancome	Rouge Absolu	Coquette
	Lancome	Rouge Sensations	Coquette
	Avon	Ultra Color Rich	Canela Nacar
	Mary Kay	Signature	Copper Star
	Avon	Beyond Color	Beige Shimmer
	Revlon	Superlustrous	Caramel Glace
	Avon	ANEW	Sandy Beige
	Lancome	Rouge Sensations	Henne
Purple-brown	Lancome	Rouge Sensations	Risque
	Avon	Glazewear	Raspberry Glaze
	NYC	Ultra Last	Rose Petal
	Avon	ANEW	Bordeaux
	Mary Kay	Signature	Whipped Berries
	Rimmel	Lasting Finish	Metallic Shimmer
	Ulta	Silky Wear	Crystal Berries
	Covergirl	Outlast All Day	Blackberry Blush
	Avon	Ultra Color Rich	Copper Plum
	Clinique	Different	A Different Grape
Red	Aziza II	(Information not available)	(Information not available)
	Mary Kay	Signature	Red
	Nars	Lipstick	Fire Down Below
	Clarins	Rouge Prodige	Crimson
	Avon	Beyond Color	Chili
	Maybelline	Revitalizing Matte	Matte Raisin
	Nars	Lipstick	Jungle Red
	Avon	Ultra Color Rich	Sheer Red
	Sally Hansen	Color Fast	Drenched Brick Creme
	Maybelline	Moisture Extreme	Royal Red

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