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Determination of lead, cadmium and nickel in hennas and other hair dyes sold in Turkey

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ABSTRACT

The concentrations of lead, nickel and cadmium in various hennas and synthetic hair dyes were determined by high-resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS GFAAS). For this purpose, 1 g of sample was digested using 4 mL of hydrogen peroxide (30%) and 8 mL of nitric acid (65%). The digests were diluted to 15 mL and the analytes were determined by HR-CS GFAAS. All determinations of Pb and Cd were performed using $NH_4H_2PO_4$ as a modifier. The analytes in hair certified reference materials (CRMs) were found within the uncertainty limits of the certified values. In addition, the analyte concentrations added to hair dye were recovered between 95 and 110%. The limits of detection of the method were 48.90, 3.90 and 12.15 ng g^{-1} for Pb, Cd and Ni, respectively and the characteristic concentrations were 8.70, 1.42 and 6.30 ng g^{-1}, respectively. Finally, the concentrations of the three analytes in various synthetic hair dyes with different brands, shades and formulae as well as in two henna varieties were determined using aqueous standards for calibration. The concentrations of Pb, Cd and Ni in hair dyes were in the ranges of LOD-0.56 μg g^{-1}, LOD-0.011 ng g^{-1} and 0.030–0.37 μg g^{-1}, respectively, whereas those in the two hennas were 0.60–0.93 μg g^{-1}, 0.033–0.065 ng g^{-1} and 0.49–1.06 μg g^{-1}, respectively.

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1. Introduction

Cosmetics contain numerous hazardous heavy metal impurities which may cause many health effects such as cancer, birth defects, allergic reactions, mental, respiratory and reproductive problems (Department of Health and Human Services Centers for Disease Control and Prevention, 2009). Hair dyes are one of the most important cosmetics all over the world, and are widely used to cover up grey hair or just to change the shade. All kind of dyes (synthetic and natural) are extensively used, especially by women. Since hair dyes are such commonly used cosmetic products, hair dye ingredients should not be harmful to human health under normal or foreseeable conditions of use (Nohynek et al., 2004). Mainly there are five different kinds of hair dyes: (i) metallic dyes (salts of lead, bismuth, silver), (ii) natural dyes (i.e. henna), (iii) temporary dyes (water soluble), (iv) semi-permanent dyes, and (v) permanent dyes. Permanent dyes are also divided into oxidative hair dyes and progressive hair dyes (Bolduc and Shapiro, 2001; Sampathkumar and Yesudas, 2009). Different kinds of hair dyes with different types of formulations may contain potentially toxic elements. The primary example is lead (as acetate), which is used as a progressive hair dye, which means that the product applied over a period of time, with each application, darkens the hair while combining with the protein in the hair (Cosmetics.info, 2016). Henna is a commonly used herb for cosmetic and medical purposes in Turkey, India, North Africa, the Middle-east countries and other parts of the world. It is either applied to hair, hands, fingernails and body skin (temporary tattoo) or added to shampoos and other hair products. Various herbs or other substances are added to the henna in order to make it more permanent or to give it a stronger or different shade. Lekouch et al. (2001) investigated the lead concentrations in henna and kohl used in Morocco. They found that when henna was mixed with other products (elaborate henna), the lead concentrations significantly increased compared to those in non-elaborate (pure henna) samples. Nevertheless, the lead concentrations in all henna samples, i.e. mixed or pure, were relatively high compared to other hair dyes.

Heavy metal poisoning, especially lead, cadmium and nickel poisoning, is a serious condition, when the metals build up in the body. Lead is a highly poisonous metal which can cause headache,

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abdominal pain, memory loss, kidney failure, male reproductive problems, and weakness, pain, or tingling in the extremities (Pearce, 2007). One study provided evidence of human skin absorption of soluble Pb (acetate and nitrate) in volunteers, with increased levels in sweat, blood and urine within 6 h of skin application (Stauber et al., 1994). Cadmium is an extremely toxic element which is classified as a human carcinogen according to WHO (Friberg and Elinder, 1992). However, incidental ingestion and inhalation are of much greater concern then dermal exposure (Department of Health and Human Services Public Health Service Agency for Toxic Substances and Disease Registry, 2012). Nickel is a carcinogenic metal for which chronic exposure has been connected with increased risk of lung cancer, cardiovascular disease, neurological deficits, developmental deficits in childhood, and high blood pressure (Chervona et al., 2012). Also nickel is known to be an allergic substance which sensitizes the skin (Contado and Pagnoni, 2012). Nielsen et al. showed that repeated skin exposure to low nickel concentration has an effect on vesicle formation and blood flow (Nielsen et al., 1999).

Hair dyes are considered cosmetic products, thus their ingredients are subjected to some regulations. In the European Union, according to Regulation (EC) No. 1223/2009 on cosmetic products, lead and its compounds, cadmium and its compounds and nickel and some of its compounds (i.e. nickel monoxide, dinickel trioxide, nickel dioxide, trinickel disulphide, tetracarbonylnickel, nickel sulphide, nickel dihydroxide, nickel carbonate, nickel sulphate) are listed as prohibited ingredients in cosmetics at ANNEX II (Council of the European Union, 2009). Recently, according to a 'Cosmetic Ingredients Hot List' published in 2015 in Canada, lead and its compounds as well as cadmium and its compounds were prohibited for use in cosmetics (Health Canada, 2015). The U.S. Food and Drug Administration limits the concentration of lead acetate in hair dyes to not higher than 0.6% (weight to volume) (CFR - Code of Federal Regulations Title 21, 2015). A limited number of studies have reported elemental concentrations of henna and other hair dye samples, which are given in Table 1.

The aim of this study was to describe a practical method for the determination of lead, cadmium and nickel in various synthetic hair dyes and hennas by high-resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS GFAAS). The experimental parameters were optimized and the effects of sample preparation and interferences on the accuracy were researched. The results are discussed with respect to shades and brands. The concentrations of analytes in the henna samples were much higher than those in the synthetic hair dye samples and need the user's attention.

2. Experimental

2.1. Instrumentation

All experiments were carried out using an Analytik Jena ContrAA 700 high-resolution continuum-source atomic absorption spectrometer equipped with a transversely heated graphite furnace, MPE 60 autosampler (Analytik Jena, Jena, Germany) and a 300 W xenon short-arc lamp (XBO 301, GLE, Berlin, Germany) operating in hot-spot mode as a continuum radiation source. The equipment includes a compact high-resolution double Echelle monochromator and a CCD array detector with a resolution of approximately 1–5 p.m. per pixel between 200 and 800 nm. All measurements were performed using pyrolytically coated graphite tubes with an integrated PIN platform (Analytik Jena Part No. 407-A81.025). Measurements were performed at wavelengths of 217.005 nm for Pb, 228.801 nm for Cd and 232.003 nm for Ni. All solutions were pipetted as 10 μL. Optimized graphite furnace program conditions for each element are given in Table 2.

2.2. Reagents and solutions

All glassware and polyethylene flasks used for the preparation of solutions were previously immersed in a 10% (v/v) HNO_3 bath overnight, then rinsed with ultra pure water with 18.2 MΩ cm resistivity obtained from a TKA reverse osmosis apparatus connected with a deionizer (TKA Wasseraufbereitungsysteme GmbH, Niederelbert Germany). All reagents were of analytical reagent grade (Merck, Darmstad, Germany). Calibration standards were prepared daily from a mixed stock standard which included 1000 mg L^{-1} of Pb, Cd and Ni (Carlo Erba, Cornaredo, Italy). For Pb and Cd determinations, a 1% solution of $NH_4H_2PO_4$ (Merck, Darmstad, Germany) was used as a modifier.

In order to test the accuracy of the method with respect to some systematic errors due to preparation of inaccurate solutions and imperfections in the weighing and pipetting, etc., two standard human hair reference materials obtained from the China National Analysis Center for Iron and Steel, NCS DC 73347 and NCS ZC 81002b, were used.

2.3. Sample preparation and analysis

Synthetic hair dye and henna samples were purchased from markets in Istanbul, Turkey. A total of 20 synthetic hair dye (five brands with 4 different shades, namely black, yellow, brown and red and 2 henna (black and green)) samples were purchased. All synthetic hair dye samples were labeled as permanent color cream

Table 1
Literature survey for Pb and Cd concentration ranges in hennas and other hair dyes.

	Sample type	Pb	Cd	Ref
Henna	(5 samples)	0.12–1.528 μg g^{-1}	0.017–0.019 μg g^{-1}	(Alwakeel, 2008)
Henna	Red Henna (6 Samples)	2.2–6.2 μg g^{-1}		(Lekouch et al., 2001)
	Black Henna (2 Samples)	15.5–19.9 μg g^{-1}		
Henna	Red Henna (2 Samples)	8.00–9.94 μg g^{-1}[a]		(Jallad and Espada-Jallad, 2008)
	Black Henna (6 Samples)	3.20–65.98 μg g^{-1}[a]		
	Green Henna (4 Samples)	2.29–4.90 μg g^{-1}[a]		
Hair dye		55.3–72.2 μg kg^{-1}		(Sharafi et al., 2015)
Hair dye	Cream Dye (4 Samples)	19.9–187 μg kg^{-1}		(Soares and Nascentes, 2013)
	Dust Dye (4 Samples)	14.0–100 μg kg^{-1}		
	Liquid Dye (4 Samples)	1.00–11.3 μg L^{-1}		
Hair dye	Blonde (5 Samples)	0.41–0.91 μg g^{-1}	0.011–0.017 μg g^{-1}	(Hussein, 2015)
	Brown (3 Samples)	0.61–0.92 μg g^{-1}	0.012–0.016 μg g^{-1}	
	Black (2 Samples)	0.56–0.81 μg g^{-1}	0.013–0.016 μg g^{-1}	
	Red	0.75 μg g^{-1}	0.015 μg g^{-1}	

[a] Units of ppm changed to μg g^{-1} for comparison.

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