Cumhuriyet Sci. J., 44(3) (2023) 516-521
DOI: https://doi.org/10.17776/csj.1263223

Cumhuriyet Science Journal

| csj.cumhuriyet.edu.tr | Founded: 2002 ISSN: 2587-2680 e-ISSN: 2587-246X Publisher: Sivas Cumhuriyet University

Determination Of Some Trace Elements In Some Lipstick Products By Inductive Coupled Plasma-Mass Spectrometry

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Research Article

History
Received: 10/03/2022
Accepted: 15/08/2023

ABSTRACT

Cosmetics have been attracting the attention of humanity since it existed. Over the years, the reach of the cosmetics industry has increased exponentially. In recent years, cosmetic manufacturers have developed many formulations on cosmetic products that are brighter in color, more permanent and not easily affected by external factors. However, heavy metal powders are used while producing these formulations especially inside in dyestuffs and stabilizers. Cosmetic products can be absorbed through the skin and cause systemic toxicity. Hundreds of chemicals are used in many cosmetic products such as make-up products, shampoos, creams, aftershaves, and these chemicals may cause significant health problems. Thus, this study is designed to investigate the heavy metal content of lipstick samples which are used extensively by women. Determination of aluminum (Al), chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn), elements in 35 different lipstick samples (including 15 brands) were performed by Inductively Coupled Plasma-Mass Spectrophotometer (ICP-MS) device. Before the analysis, two different solubilization methods, wet solubilization and dry ashing, were applied to the samples in order to take the metals in the lipstick samples into inorganic environment.

Keywords: Heavy metals, ICP-MS, Lipstick products, Dry ashing, Wet solubilization.

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Introduction

In the Dictionary of the Turkish Language Association, cosmetics are defined as any substance that helps to beautify the skin and hair and keep them alive, and by the Cosmetic Council of the European Union Cosmetics Legislation 1223/2009, Article 2.1.a, "Cosmetic product; It is prepared to be applied to different external parts of the human body such as the epidermis, nails, hairs, hair, lips and external genitalia, teeth and oral mucosa, with the sole or main purpose of cleaning these parts, giving scent, changing their appearance and/or correcting body odors and/or defined as "all preparations or substances intended to preserve or keep in good condition" [1].

All cosmetic products authorized for use must be completely safe for users and the responsibility for ensuring the safety of these products is the manufacturer, distributor, and importer. In cosmetic products, it is estimated that approximately 10,000 chemicals are present, including parabens, phthalates, p-phenylenediamine, formaldehyde, dioxane, triclosan and numerous metals [2]. Moreover, preservatives, softeners, surfactants, or UV (ultra-violette) protectors may be added to cosmetic products depending on the ingredients in the composition and the purpose of use [3].

Lip products constitute a large proportion of all cosmetic products. The desire to color the lips has attracted the attention of humanity in the historical process. The first records of people using any product to beautify their lips appear in the Sumerians around 7000 BC. While the use of the first lip products started with mixing wax, oil and pigments and applying them to the lips, it took the form of sticks after the 1920s. When looking at the periods of Ancient Egypt, Greek and Roman Empire, it was seen that women applied some red minerals and herbs to their cheeks and lips in order to beautify. Therefore, lip cosmetics can be traced back thousands of years. Lip cosmetics, which moisturize the lips and increase their attractiveness, are one of the most used cosmetics by modern women [4,5]. Lip cosmetics usually consist of three main ingredients which are wax, oil and coloring agents. In addition to these three main ingredients, lip products contain some excipient substances such as antioxidants, preservatives and perfumes. On the other hand, studies are continuing on whether heavy metals are used as in these products and on safe usage intervals. Lip products can be classified as lip balm, lipstick, lip brilliant and lip gloss [6].

Lipsticks basically contain various oils, beeswax, pigments, antioxidants and preservatives. Compounds of some basic elements can also be added to the formulation to improve the quality of cosmetic products. The presence and amounts of metal components in cosmetic products require processes that must be followed continuously since there is direct contact with the skin or mucous membranes. Despite exposure to very low amounts of heavy metals because of the use of lip products, significant chronic health risks may occur due to accumulation in long-term use. Lipsticks may contain heavy metals such as iron (Fe), cadmium (Cd), and copper (Cu) [7]. Serious problems due to excess heavy metals in cosmetics have been reported by the United States Food and Drug Administration (FDA) and the China National Drug Administration [8]. Considering the usage area, it is estimated that the products that cause the most toxic metal intake are lipsticks and lip glosses. There is risk that cosmetic products applied to the lips may be taken directly orally. When lipstick and other lip products are applied to the lips, it is easy to dissolve in the saliva and enter the bloodstream [9].

Microelements are defined as elements required in amounts less than 100 mg per day and present in tissues at mg/kg tissue levels. Iron, copper, zinc, cobalt, manganese, chromium, molybdenum, selenium, fluorine, and iodine are examples of microelements. Since the concentrations of microelements in the body are very low, they are also called trace elements [10]. Trace elements are found in the structure of the organism and play a role in many important functions. These vital elements must be taken in sufficient quantities to maintain a healthy life. Regulation of body functions, oxygen transport, and elimination of free radicals are the most important functions of trace elements. However, their excess amounts have a toxic effect on the body. Moreover, elements that have not been shown to be essential for the body's structure and function, are known as non-essential or toxic elements. All the trace elements, which are necessary for metabolism and listed above, also create a toxic effect if they are taken into the body more than a certain amount. [11].

In recent years, studies on the determination of trace elements in lip products have been increasing. Al-Saleh et al. investigated the levels of lead, cadmium, nickel, chromium, mercury, antimony, and arsenic in 14 brands of lipstick (28 samples) that are widely used in local Saudi markets. According to their results, the levels of all metals were generally lower than the rules of the US Food and Drug Administration for metallic impurities in color additives used in cosmetics. Only one brand of lipstick exceeded the specifications, containing levels of arsenic higher than 3 mg/kg [12]. In another study, lead values of 12 lipsticks, 13 lip glosses and 9 lip balms and the averages were determined by ICP-MS (Inductively Coupled Plasma-Mass Spectrophotometer) device which found as 0.05482 mg/kg, 0.04976 mg/kg and 0.07380 µg/g, respectively [8]. In another study, Cr, Mn, Co, Ni, Cu, Cd, Sb, Pb measurements were made in 32 lip sticks and their values were found as 6.72 mg/kg, 12.27 mg/kg, 0.48 mg/kg, 4.10 mg/kg, 93.93 mg/kg, 0.10 mg/kg, 1.13 mg/kg, 7.42 mg/kg respectively. The study also contained bioaccessibility which was the first study in this area. According to their results, only bioaccessible Pb in all samples significantly exceeded the FDA limit 0.1 mg/kg in candy [6].

In this context, the main goal of this study is to investigate the concentration of aluminum (Al), chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn) in 35 different lipstick samples (including 15 brands) in Turkey market by inductive coupled plasma-mass spectrometry.

Materials and Methods

Chemicals and Devices

All chemicals, used in the analysis were purchased from Sigma Chemical Co. (St. Louis, MO, USA). All chemicals were of analytical grade. HNO_3 %65, HF %40, $HClO_3$ %35 were used for solubilization methods. Quantitative filter paper, (FILTER-LAB® ref. 1238 125 mm diameter) was used for filtration process. Agilent 7700 Series ICP-MS was employed for analysis.

Sample Preparation

Before ICP-MS analysis, two different solubilization techniques were employed to prepare the samples for analysis. The first of these is the wet solubilization method with the acid mixture and the second one is the dry ashing method in the muffle furnace. At first, the microwave solubilization method was tried in order to solubilize the lipstick samples. However, as result of trials with many acid mixtures, explosions, burns, or melted lid boils were experienced in the vials due to high temperature and acid interaction. For this reason, it was decided to solubilize the lipstick samples with acid mixed wet solubilization and dry ashing method.

Wet Solubilization Method

The lipstick samples were numbered after they were categorized. Precise weights of 0.500 grams were taken from the numbered samples with using analytical balance. The weighed samples were placed in equally numbered vials. The elements were added to the inorganic medium by 5.00 mL nitric acid (HNO_3) and 1.00 mL hydrofluoric acid (HF) onto the weighed samples. After the solubilization process of the lipstick samples was carried out, they were diluted to 50.00 mL and stored in HDPE (High Density Polyethylene) storage containers until analysis. Trace element contents in the extracts were determined by ICP-MS device [13].

Dry Ashing Method

The lipstick samples were numbered after they were categorized. Precise weights of 0.500 grams were taken from the numbered samples with using analytical balance. The weighed samples were placed in equally numbered porcelain crucibles. The lids of the crucibles were closed and placed vertically in the muffle furnace. Lipstick samples were allowed to become ashes at 900 ^oC for 4 hours in the muffle furnace. By adding 5.00 mL of HNO_3 to 1.00 mL of chloric acid ($HClO_3$) to the crucibles, the ashes were taken to the liquid medium and filtered with the help of filter paper. After dissolving the lipstick samples, they were diluted to 50.00 mL and stored in HDPE storage containers until analysis. Trace element contents in the extracts were determined by ICP-MS device [13].

ICP-MS Analysis

An Agilent 7700 Series (Agilent Technologies, Tokyo, Japan) was used for all measurements. All measurements were performed in triplicates from each vial. The instrument parameters are described in Table 1.

Table 1. Setting parameters for the ICP-MS method.

Plasma Parameters	*Lens Parameters*	*Cell Parameters*
Power: 1550 W	Omega Lens: 10V	OctP Slope: -8V
Matcher voltage: 1.80V	Cell Input: -30V	OctP RF: 180V
Sample depth: 8mm	Cell Output: -50V	Energy separator: 5V
Carrier gas velocity: 1.05 l/min	Deviation: 13V	Stabilization time: 50 seconds
Nebulizer pump: 0.1 rps	Layer training: -40V	Sampling time: 50 seconds
S/C temperature: 2°C		Calibration curve confidence interval : 0.95

Results and Discussion

In this study, the concentrations of the selected heavy metals; aluminum (Al), chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn) in 35 different lipstick samples were determined by using ICP-MS device. The values detected in lipsticks solubilized by the wet solubilization method (Table 2) and dry ashing method (Table 3). In this study, the standard addition method was used while working with the ICP-MS device to minimize the matrix effects and prevent interference. Although there is widespread suspicion about the toxicity of cosmetics, there have not been enough controlled studies and the reported studies are generally as uncontrolled and case reports. In our literature research, it was realized that there is a great deficiency in this area. Trace elements, which are of great importance especially for human health; aluminum (Al), chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn) concentrations in different lipstick samples was determined by ICP-MS instrument by using 2 different solubilization techniques.

According to the results of the analyze using the wet solubilization method; the values were found for the element aluminum vary between 0.23 mg/kg and 87.93 mg/kg. The results were determined by using the dry ashing method ranged from 0.25 mg/kg to 89.10 mg/kg. The values determined in the wet solubilization method for chromium vary between 0.02 mg/kg and 0.41 mg/kg. The values determined in the dry ashing method for chromium vary between 0.03 mg/kg and 0.14 mg/kg. The values determined in the wet solubilization method for iron vary in a wide range between 3.19 mg/kg and 605.00 mg/kg. The values determined in the iron element dry ashing method vary between 3.71 mg/kg and 54.16 mg/kg. In nickel element, the values determined in the wet solubilization method was very close to each other her but vary between 0.01 mg/kg and 0.25 mg/kg. The amount of nickel determined by the dry ashing method was found between 0.03 mg/kg and 0.13 mg/kg. In the wet solubilization method for copper element, it was ranged between 0.01 mg/kg and 0.22 mg/kg. The results of the dry ashing method of the same element were detected between 0.02 mg/kg and 0.10 mg/kg. The values found for the results obtained in the study are compatible with the studies in the literature in which determinations were made with different or the same device. As a result, the difference in the solubilization method caused the elements to be detected in different amounts. The acids used in the 2 methods are different and therefore the contribution of acidity to ionization in the mass detector is different. Therefore, different results were obtained in wet solubilization and dry ashing methods. According to the Medical Drug Device Agency, the maximum values that should be found in cosmetics are 20 mg/kg for lead, 5 mg/kg for arsenic, 5 mg/kg for cadmium, 1 mg/kg for mercury, and 10 mg/kg for antimony. However, there is no specified limit value for aluminum, chromium, copper, iron, nickel, and zinc determined by Medical Drug Device Agency. Moreover, FDA (US Food & Drug Administration) addresses the maximum values of some trace elements such as 3 mg/kg for arsenic, 20 mg/kg for lead, 1 mg/kg for mercury. However, there is no specified limit value for aluminum, chromium, copper, iron, nickel, and zinc determined by FDA. According to the various studies, chronic and long-term exposure to trace elements can lead to serious health problems [10].

While chromium, when consumed in small quantities, serves as a vital element that aids the body in utilizing sugar, protein, and fat, excessive inhalation of chromium can result in nasal irritation, leading to symptoms such as a runny nose, nosebleeds, and the formation of sores or perforations in the nasal septum. Ingesting large amounts of chromium can lead to gastrointestinal disturbances, ulcers, seizures, as well as damage to the kidneys and liver, potentially resulting in fatality. Contact with specific chromium compounds on the skin can give rise to the development of skin ulcers. Individuals highly sensitive to chromium may experience severe allergic reactions characterized by intense skin inflammation and swelling [14].

Prolonged contact with nickel can lead to allergic reactions of the skin, such as skin rashes and other related symptoms. Moreover, the International Agency for Research on Cancer (IARC) has categorized nickel compounds as belonging to Group 1, indicating they are known to cause cancer in humans, while metallic nickel is classified as Group 2B, suggesting it is possibly carcinogenic to humans. Nickel is a significant contributor to allergic contact dermatitis in people worldwide, affecting both children and adults, and it has a global occurrence rate of approximately 8.6% [15].

Copper is present in plants and animals as a natural component. It is a vital element required by all living organisms, including humans and other animals, in small quantities. However, when found in significantly higher concentrations, copper can have harmful effects that are considered toxic. Prolonged copper exposure may cause cancer, infertility, skin allergy and greenish discoloration of the skin, hair, and teeth. [16].

According to the literature, there are no known risks for healthy people from normal dietary intakes of aluminum. The risks are only harmful longer than the usual consumption of grams of aluminum antacids which is significantly increased in people with impaired renal function. Moreover, long-term intravenous administration always causes serious toxicity. Aluminium may cause gene instability, alter gene expression or enhance oxidative stress in the body [17].

While iron is an essential mineral necessary for various physiological processes, such as oxygen transport and energy production, an overload of iron can lead to toxicity. This can happen in individuals with hereditary disorders like hemochromatosis or through repeated excessive iron supplementation. Prolonged iron toxicity can cause damage to organs such as the liver, heart, and pancreas, leading to conditions like cirrhosis, cardiomyopathy, and diabetes. Symptoms may include fatigue, joint pain, abdominal pain, and skin discoloration. [18].

While zinc is an essential trace element necessary for various physiological functions, such as immune system regulation and enzymatic activity, an excessive intake or chronic exposure to high levels of zinc can lead to toxicity. Prolonged zinc toxicity can occur due to factors such as excessive zinc supplementation, occupational exposure, cosmetic produtcs or genetic disorders affecting zinc metabolism. The adverse effects of prolonged zinc toxicity may include gastrointestinal disturbances, such as nausea, vomiting, and abdominal pain. It can also interfere with copper absorption, leading to copper deficiency and associated neurological symptoms. Additionally, long-term zinc toxicity can impact immune function, disrupt hormonal balance, and affect the liver and kidneys. [19].

When the values of all the elements determined in our study are compared with the literature and the value in lipsticks does not pose any risk. However, long-term, and continuous use may cause adverse effects on health. In continuous use, the accumulation effect in the body should be considered. In addition, there is no limit value determined for these elements by the Medical Drug Device Agency and US Food and Drug Administration. As a result, the accumulation of heavy metals taken from cosmetic products poses a health risk and needs to be inspected with continuous measurements and controls.

Table 2. Amounts of aluminum, chromium, iron, nickel, copper, and zinc (mg/kg) detected in lipsticks using the wet solubilization method.

Samples	*Al*	*Cr*	*Fe*	*Ni*	*Cu*	*Zn*
1	38.55	0.07	191.05	0.05	0.04	8.01
2	28.52	0.09	338.59	0.04	0.05	8.36
3	74.52	0.08	53.62	0.13	0.16	3.98
4	0.23	0.02	3.19	0.01	0.01	0.00
5	11.49	0.04	64.44	0.04	0.03	6.24
6	77.42	0.06	23.55	0.07	0.11	11.79
7	48.73	0.12	176.75	0.13	0.05	10.07
8	20.17	0.05	338.38	0.17	0.06	10.72
9	40.34	0.04	128.03	0.04	0.03	8.15
10	86.93	0.06	14.80	0.07	0.04	8.06
11	50.86	0.06	4.57	0.25	0.03	6.83
12	6.18	0.05	419.68	0.09	0.22	12.00
13	2.32	0.05	239.10	0.24	0.08	11.20
14	33.28	0.08	247.49	0.06	0.13	10.76
15	32.18	0.11	172.99	0.10	0.03	12.70
16	31.50	0.07	56.09	0.06	0.04	10.54
17	20.71	0.06	171.30	0.17	0.03	15.42
18	72.28	0.17	33.76	0.20	0.09	11.73
19	59.40	0.41	29.99	0.20	0.11	11.47
20	20.87	0.05	312.43	0.08	0.13	10.44
21	60.82	0.05	4.09	0.10	0.04	7.29
22	87.93	0.18	23.28	0.18	0.05	10.18
23	80.55	0.04	146.37	0.08	0.04	5.47
24	50.95	0.12	326.64	0.08	0.04	9.22
25	39.04	0.06	200.96	0.05	0.08	8.28
26	12.24	0.03	5.97	0.18	0.03	3.37
27	8.28	0.06	605.00	0.06	0.03	6.33
28	22.03	0.13	11.23	0.10	0.04	6.43
29	12.23	0.04	211.26	0.05	0.03	5.08
30	27.24	0.03	3.26	0.02	0.03	4.37
31	14.79	0.05	66.12	0.05	0.02	2.61
32	23.59	0.22	469.58	0.14	0.15	8.21
33	4.14	0.04	5.16	0.02	0.02	5.65
34	27.65	0.08	360.68	0.04	0.02	11.73
35	12.33	0.06	117.55	0.06	0.08	4.05

Table 3. Amounts of aluminum, chromium, iron, nickel, copper, and zinc (mg/kg) detected in lipsticks using the dry ashing method.

Samples	Al	Cr	Fe	Ni	Cu	Zn
1	25.91	0.07	13.37	0.06	0.10	0.57
2	3.97	0.05	5.76	0.04	0.05	0.53
3	58.86	0.05	4.38	0.03	0.05	0.54
4	18.64	0.05	7.79	0.04	0.05	0.56
5	1.09	0.04	4.45	0.04	0.04	0.56
6	4.77	0.03	4.19	0.03	0.05	0.57
7	9.45	0.04	4.55	0.04	0.09	0.55
8	0.85	0.14	9.90	0.13	0.06	0.82
9	2.82	0.06	4.51	0.05	0.04	0.26
10	4.92	0.05	3.75	0.04	0.04	0.19
11	3.40	0.06	3.71	0.04	0.03	0.19
12	1.76	0.04	4.90	0.06	0.03	0.52
13	1.87	0.03	4.46	0.03	0.03	0.49
14	0.28	0.04	4.43	0.04	0.04	0.53
15	1.36	0.03	4.17	0.04	0.04	0.52
16	3.98	0.05	4.47	0.03	0.04	0.52
17	0.42	0.03	4.05	0.04	0.03	0.52
18	32.77	0.05	4.67	0.04	0.05	0.54
19	41.00	0.07	4.71	0.05	0.06	0.54
20	11.57	0.03	6.82	0.04	0.04	0.54
21	24.73	0.04	3.70	0.04	0.04	0.17
22	3.33	0.03	4.19	0.03	0.02	0.17
23	2.90	0.03	5.10	0.03	0.02	0.17
24	57.42	0.05	54.16	0.04	0.04	0.18
25	1.70	0.03	4.17	0.03	0.03	0.48
26	89.07	0.05	3.97	0.04	0.06	0.54
27	4.14	0.04	5.10	0.04	0.04	0.52
28	7.70	0.05	4.23	0.05	0.03	0.53
29	0.97	0.03	4.03	0.04	0.04	0.52
30	2.52	0.05	3.82	0.04	0.04	0.54
31	0.25	0.03	4.06	0.03	0.03	0.52
32	15.92	0.03	4.98	0.04	0.08	0.26
33	5.81	0.04	4.01	0.04	0.06	0.19
34	2.41	0.03	4.16	0.04	0.03	0.25
35	1.95	0.03	7.83	0.04	0.04	0.49

Conclusion

The results acquired from the study are essential to understanding the risks of trace element exposure and their occurrence in lipsticks. The contents of the trace elements were ordered in the following decreasing order according to the maximum concentrations Al > Fe > Zn > Cr, Ni, Cu and the levels of all investigated metals were generally lower than the limit values determined by literature . Turkey Drug and Medical Device Agency and FDA has set limit values only for lead, arsenic, cadmium, mercury, and antimony. Therefore, risk analysis regarding the concentrations of other trace elements cannot be performed. As a conclude, continuous analysis is essential for all cosmetic products since prolonged exposure of trace elements have significant negative effects on human health.

Acknowledgment

This study was funded by Trakya University Scientific Research Foundation (Project No: TUBAP-2017/136).

Conflicts of interest

There are no conflicts of interest in this work.

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