The Quality Control of Eye Shadow, Eyeliner, and Mascara Products that Sold on Saudi Markets

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ABSTRACT

This study aimed to assess the quality control of different types of eye cosmetics products (eye shadow, eyeliner, and mascara), including the detection of their elements' content. Thirty-four samples were selected based on a survey done in Jeddah markets in Saudi Arabia; they had different prices and sources (China, Saudi Arabia, Italy, Canada, and the USA). The Saudi Standards, Metrology and Quality Organization (SASO) specification tests were applied to detect the quality control of the products. Concentrated acids HNO3: HClO3 in ratio 4:1 was used to digesting all samples before being analyzed with Inductivity coupled plasma – optical emission spectroscopy (ICP-OES) to quantification selected elements(toxic and nontoxic). An elemental analyzer determined the percentage of carbon and sulfur. In general, the results of this research revealed that heavy metals current in eye cosmetics products were under the acceptable limits while a part of those lower price products imported from China might be dangerous. Also, eye shadow products contained the largest concentration of heavy metals compared with eyeliner and mascara products. Sadly, Mascara contained high bacteria accounts than the others but under permissible limits. The constant use of these products can be a hidden hazard to human health since heavy metals can concentrate over time and cause emotional problems. It was sincerely suggested to control the quality of these products and to inform consumers to be careful when they use low priced products.

Key Words: *Cosmetics, eye shadow, eyeliner, mascara, ICP-OES, CHNS, heavy metals, toxicity, SASO.*

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INTRODUCTION

Cosmetics have been considered as a part of daily body care since the dawn of civilization. Eye make-up is used to spotlight the beauty of the eyes. It consists of three categories: eyeliner, mascara, and eye shadow [1]. Makeup cosmetic products contain various types of pigments (organic, inorganic, pearly pigments, etc.) in their base formula. Heavy metals exist broadly in cosmetics. As ingredients, some metals are added to cosmetic formula when otherns are contaminants. Even though these metals accumulate in the human body, they may have dangerous effects. Health and environmental concerns have been linked to exposing to metals [2].

There have been several studies conducted in eye makeup products to determine the presence of heavy metals using different techniques [3-10].

Women in Saudi Arabia widely use cosmetic products. According to statistics issued by the General Saudi Customs, the value of imports of cosmetics exceeded SR 3 billion during the past two years; volumes amounted to about 58.7 million kg [11]. These cosmetics contain heavy metals as a part of various chemicals. This study aimed to assess the quality control of 34 eye makeup products available in the Saudi market according to Saudi Standards, Metrology and Quality Organization (SASO) tests, then determined their content of some metals and non-metals using ICP-OES and CHNS (an Elemental analyzer technique).

EXPERIMENTAL

1. Conducting a statistical study on a group of Saudi women, and determining the level of their knowledge on the use of eye makeup products, and the extent of the damage which they might suffer from using them, and choosing some samples under study according to their use.

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2. Considering Saudi Standards, Metrology and Quality Organization and Saudi specialized laboratories company (Motabaqa) for chemical and microbiological analysis standard, the following experiments have been performed: Visual inspection, melting point test, microbiological tests, and determination of some metals.

The results were compared with the standards of the Saudi Standards, Metrology and Quality Organization, which have been shown in Table.1 below.

Table 1: The requirements of eye makeup according to GSO 1846/2008 and GSO 2170/2011[12, 13]

Characteristic	Requirement
Melting point ^{0}C (minimum limit)	50
Micro-organism number per gram (maximum limit)	100
As ppm (maximum limit)	2.5
Pb ppm (maximum limit)	10
Sb ppm (maximum limit)	2
Hg ppm (maximum limit)	0.5

Samples Collection:

According to the questionnaire that was conducted on 500 Saudi women to determine their awareness of eye makeup products, 34 samples with different brands were selected under study and purchased from Jeddah markets in Saudi Arabia (12 samples of eye shadow, 11 samples of eyeliner, and 11 samples of mascara). Different colored eye makeup samples were divided into four groups representing four different brands from different countries of origin, and each group had three colors: black, blue, and brown. Nine of the investigated samples were Saudi manufactured while twenty-five samples were manufactured in China, Italy, Canada, and the USA. Tables .2,3, and 4 below represents all of the details of the samples.

Table 2: Eye shadow samples and their details.

Sample's Type	Sample's Number	Colour	Manufacturing Country	Expiry date	Price
Eye shadow	S1	Black	China	none	2 $
	S2	Blue			
	S3	Brown			
	S4	Black	Saudi Arabia	06/2017	12 $
	S5	Blue		11/2018	17 $
	S6	Brown			
	S7	Black	China	18 months after open	14 $
	S8	Blue			
	S9	Brown			
	S10	Black	Italy	36 months after open	26 $
	S11	Blue	Canada		
	S12	Brown	USA		

Chemicals, equipment, and instruments:

- Chemicals: The materials used in the study included: cyclohexane, distilled de-ionized water (Milli-Q, France), ethanol (EtOH), nitric, acid (65%, Sigma Aldrich), Nutrient Agar (OXOID, England) and perchloric acid (60%, Sigma Aldrich)
- Equipment: filter papers (Whatman no. 42), glass beaker 150 ml, measuring cylinder, Petri dishes, and volumetric flask 100 ml.
- Instruments: The instruments used in the study included: Inductively Coupled Plasma Optical Emission Spectrometry (Perkin Elmer-Optima 7300DV), Elemental C, H, N, S Analyzer (Vario El M Germany), Milli-Q Ultrapure Water System, balance (Adventurer Analytical), hotplate stirrer, melting point apparatus (Stuart SMP10), Autoclave, incubator (Binder), and automatic colony counter (Interscience Scan 500).

Table 3: Eyeliner samples and their details.

Sample's Type	Sample's Number	Colour	Manufacturing Country	Expiry date	Price
Eyeliner	L1	Black	China	18 months after open	0.5 $
	L2	Blue			0.5 $
	L3	Brown			0.5 $
	L4	Black	Saudi Arabia	-	5.60 $
	L5	Blue			4.27 $
	L6	Brown			4.27 $
	L7	Black	Germany	24 months after open	13 $
	L8	Blue			13 $
	L9	Brown			13 $
	L10	Black	Italy	-	25.60 $
	L11	Brown			25.60 $

Table 4: Mascara samples and their details.

Sample's Type	Sample's Number	Colour	Manufacturing Country	Expiry date	Price
Mascara	M1	Black	China	6 months after open	1.33 $
	M2	Blue			1.33 $
	M3	Brown			1.33 $
	M4	Black	Saudi Arabia		12 $
	M5	Blue			12 $
	M6	Brown			12 $
	M7	Black	Italy	6 months after open	21 $
	M8	Blue			24 $
	M9	Brown			24 $
	M10	Black	Canada	-	38.40 $
	M11	Brown			

Visual inspection:

The samples were checked by the naked eyes to look for flaws. This required no equipment to ensure the

compliance of the product, in terms of color, smell, and structure of the samples, and they were visually examined by the naked eyes to ensure the compliance of the products with the Saudi Standards.

Melting point:

Measuring the melting point was essential to check the quality of the product. Eye makeup must have had a melting point more than 50ºC.

Microbiological examination:

Microbiological safety is one of the most dynamic and critical cosmetics' quality parameters. Cosmetic products must be free of pathogenic microorganisms, and the total aerobic microbial count must be lower than 100 CFU/ml (CFU=colony forming units).

Procedure: The media were reconstituted and sterilized according to the direction of the manufacturer(Oxoid). After isolating samples, all the plates were incubated at 37**º**C for 24 hours followed by colony count using an Interscience Scan 500 automatic colony counter. Results were expressed as colony forming unit per milliliter (CFU/ml).

ICP-OES Experimental:

- **Sample Preparation**

The overall sample preparation method followed previously published procedures [14]. Each day, calibration standards for each metal were prepared from the certified standard stock solution (High-Purity Standards ICP-OES-68B Solution A, 100 mg/L in 4% HNO_3) in the range from 0.5 to 10 ppm. In deionized water, all solutions were prepared. Diluted or concentrated sample during analysis were undergone for the dilution correction.

- **Sample Analysis:**

There was a narrow range between safe and toxic levels, so a precise determination of heavy metal content in cosmetic products was essential. This study used ICP-OES for the selected elements. The system was conducted in air mode and collision mode; the latter, using helium, was used for the investigation of elements with atomic weights under 100. The line choice and method were selected to reduce the possible interference. The concomitants for the ICP-OES operation were as follows: plasma gas, 15 L/min; power, 1550 W; nebulizer, 0.8 L/min; aux gas, 0.2 L/min; sampling rate, 0.3 mL/min. The readings were culminated according to the standard deviation from measurements that were achieved in triplicate.

CHNS Elemental Analyzer experimental:

- **Preparation of the sample:**

The sample was weighted in the range of (1-3 mg) then it was put in a tin boat which stood at the high temperature of the furnace (First furnace 850 ºC, Second furnace 1150 ºC). Then, the sample was placed inside the instrument through the sample feeder.

- **The performance of the instrument:**

High purified He gas (the carrier gas), with the flow of 200 ml/min, the pressure of 1.2 bar was used. O_2 gas was used (for the combustion process) from 1-2 min splashing the gas Oxidation-reduction process. The columns were absorbed for trapping the oxidized after they undergo a reduction process through Cu – cc, H, N, S). TCD (Therma conductively detector) was used to detect the gasses, and translate them into signals. (peaks of the samples) N, C, S, H.

RESULTS AND DISCUSSION:

Visual inspection results:

The samples under study were visually examined by naked eyes and sense of smell to ensure the compliance of the product with the Saudi Standards. All the samples were acceptable considering color, smell and texture (pressed powder for eye shadow, paste for eyeliner, and cream for mascara) according to the Saudi standards specification.

Melting point test:

Melting point test was performed on eyeliner and eye shadow samples using melting point apparatus (Stuart SMP10). According to the standards, the melting point should be higher than 50ºC. Eyeliner samples showed melting point range from 55ºC to 79ºC, which was accepted by SASO specification.

For the eye shadow samples, the melting point for all samples exceeded 400ºC, which was the highest degree the instrument can reach. The explanation for this high value was due to the main components of eye shadow which were Talc and Mica; these minerals had a high melting point estimated about 1500°C for talc and 700–1000 °C for mica.

Microbiological testing:

The results of the microbiological examination were suitable with the limits obtained in GSO 1846/2008.

Table 5: microbiological examination results of eye shadow, eyeliner, and mascara samples

Eye shadow sample No.	Mean of bacterial count ±SD (CFU/ml)	Eyeliner sample No.	Mean of bacterial count ±SD (CFU/ml)	Mascara sample No.	Mean of bacterial count ±SD (CFU/ml)
S1	9 ± 5	L1	1 ± 1	M1	67 ± 20
S2	ND	L2	5 ± 3	M2	2 ± 1
S3	1 ± 1	L3	ND	M3	1 ± 1

S4	2 ± 3	L4	ND	M4	20 ± 24
S5	ND	L5	ND	M5	7 ± 5
S6	1± 1	L6	1 ± 1	M6	18 ± 8
S7	ND	L7	ND	M7	20 ± 10
S8	ND	L8	1± 1	M8	10 ± 9
S9	1 ± 2	L9	1 ± 1	M9	34 ± 1
S10	1 ± 1	L10	1 ± 2	M10	8 ± 11
S11	ND	L11	ND	M11	27 ± 9
S12	5 ± 3	-	-	-	-
Diacritics with SASO specification	Approving	-	Approving	-	Approving

*No. of determinations: 3 *ND: Not Detected

The bacterial count results were minimal in the eye shadow and eyeliner samples, as well as in mascara samples, because the tested samples were new and not used. The low bacterial count presence was due to manufacturing or exposure to the air before the analysis. However, mascara samples had more bacteria count than the eye shadow and eyeliner that was due to the brush (inside mascara box), which gave a suitable place for the presence and growth of bacteria.

ICP-OES Experimental results:

- **Validation of Methods and Procedures:**

Method validation is vital in the practice of an analytical process; it's the process of defining an analytical requirement ,and confirming that the current method has performance abilities consistent with what the application requires.

Linearity, LOD, and LOQ for all the elements under study have been shown in table 6. Coefficient of determination $\mathbf{r^2}$ values ranged from 0.999 to 1, which indicated a good correlation of linearity through all the concentrations.

- **Precision:**

Precision is how close the measured values are to each other. The samples were analyzed with two different procedures; in the first one, the samples were analyzed two times in the same day with one hour apart; this procedure is called intra-day analysis. Second: the samples were analyzed in two different days with one week apart, it's called intra-day analysis. The concentration of the elements on different days was close, as shown in Table 7. These results confirmed that the samples were stable, and ICP-OES was an accurate and suitable tool for this analysis.

Table 6: Linearity, LOD and LOQ.

Metal	Range	(r^2)	r	LOD	LOQ
Ag	0.125 - 5	0.9995	0.9997	0.180	0.600
Al	0.25 - 10	1.0000	1.0000	0.058	0.195
As	0.25 - 10	0.9998	0.9999	0.210	0.701
Ba	0.25 - 10	0.9999	0.9999	0.202	0.675
Ca	0.25 - 10	1.0000	1.0000	0.050	0.168
Co	0.25 - 10	0.9993	0.9996	0.439	1.464
Cr	0.25 - 10	0.9999	0.9999	0.175	0.583
Cu	0.25 - 10	1.0000	1.0000	0.058	0.193
Hg	0.25 - 10	0.9885	0.9942	1.821	6.070
Ni	0.25 - 10	0.9992	0.9996	0.472	1.574
Pb	0.25 - 10	1.0000	1.0000	0.093	0.309
Sb	0.25 - 10	0.9999	0.9999	0.164	0.546
Ti	0.25 - 10	0.9998	0.9999	0.248	0.825
Zn	0.25 - 10	0.9990	0.9995	0.530	1.768

*LOD= Limit of detection *LOQ= Limit of quantitation
*r= Coefficient of correlation *r^2= Coefficient of determination

Table 7: Evaluation of intra-day and inter-day precision:

Metal	Content in mascara sample mean±SD (n=3)		Relative standard deviation RSD %	
	Intra-day	Inter-days	Intra-day	Inter-days
Ag	ND	ND	ND	ND
Al	ND	ND	ND	ND
As	ND	ND	ND	ND
Ba	ND	ND	ND	ND
Ca	1071.40 ± 19.40	901.20 ± 150.8	1.81	16.73
Co	281.10 ± 7.30	239.40 ± 34.40	2.60	14.37
Cr	3.70 ± 0.10	3.60 ± 0.00	2.70	0
Cu	ND	ND	ND	ND
Hg	ND	ND	ND	ND
Ni	3.50 ± 0.10	3.20 ± 0.20	2.86	6.25
Pb	3.50 ± 0.30	1.90 ± 1.90	8.57	100.00
Sb	ND	ND	ND	ND
Ti	22.80 ± 0.40	20.00 ± 2.40	1.75	12.00
Zn	ND	ND	ND	ND

*No. of determinations: 3 *ND: Not Detected

- **Toxic Heavy Metals:**

The following table (Table 8) illustrates the organizations and their allowed limits of heavy metals.

Table 8: Heavy metals limits in cosmetic

Metal Organization	Lead	Arsenic	Cadmium	Mercury	Antimony	Ref.
German Federal Government	20 ppm	5 ppm	5 ppm	1 ppm	10 ppm	[8]
Health Canada	10 ppm	3 ppm	3 ppm	1 ppm	5 ppm	[15]
FDA	20 ppm	3 ppm	-	1 ppm	-	[16]
SASO	10 ppm	2.5 ppm	1 ppm	0.5 ppm	2 ppm	[13]

The results of heavy metals As, Hg, Pb, and Sb have been summarized in the following tables (Tables 9, 10, & 11):

Table 9: ICP Results for Heavy Metals Exists in Eye shadow Samples

Sample No.	Elements concentration ppm ±SD				
	As	Hg	Pb*	Sb	Total
S1	122.80 ±0.10	ND	4.00 ±0.01	ND	**126.800**
S2	139.00 ±0.16	ND	10.40 ±0.01	ND	**149.40**
S3	135.00 ±0.12	ND	16.60 ±0.04	ND	**151.60**
S4	22.00 ±0.00	ND	7.60 ±0.11	ND	**29.60**
S5	ND	ND	ND	ND	**0.00**
S6	2.6.00 ±0.01	ND	1.20 ±0.04	ND	**3.80**
S7	ND	ND	9.20 ±0.07	ND	**9.20**
S8	ND	ND	ND	6.40 ±0.06	**6.40**
S9	ND	ND	2.80 ±0.02	ND	**2.80**
S10	ND	ND	3.80 ±0.05	ND	**3.80**
S11	57.00 ±0.21	20.8 ±0.00	7.80 ±0.00	ND	**85.60**
S12	ND	0.2 ±0.00	4.6 ±0.03	ND	**4.8**

*No. of determinations: 3 *ND: Not Detected
* Pb results [17]

Table 10: ICP Results for Heavy Metals Exists in Eyeliner Samples

Sample No.	Elements concentration ppm ±SD				
	As	Hg	Pb	Sb	Total
L1	ND	ND	ND	0.20 ±0.08	**0.20**
L2	ND	ND	ND	17.60 ±0.09	**17.60**
L3	ND	ND	6.60 ±0.02	ND	**6.60**
L4	17.00 ±0.08	0.20 ±0.00	5.40 ±0.05	ND	**22.60**
L5	ND	ND	0.20 ±0.01	ND	**0.20**
L6	6.80 ±0.14	ND	6.40 ±0.07	ND	**13.20**
L7	ND	ND	7.200 ± 0.04	ND	**7.20**
L8	ND	ND	ND	ND	**0.00**
L9	15.80 ± 0.18	ND	1.20 ± 0.02	ND	**170**
L10	ND	ND	7.60 ± 0.02	ND	**7.60**
L11	11.20 ± 0.07	ND	1.00 ± 0.03	ND	**12.20**

*No. of determinations: 3 *ND: Not Detected

Table 11: ICP Results for Heavy Metals Exists in Mascara Samples

Sample No.	Elements concentration ppm ±SD				
	As	Hg	Pb	Sb	Total
M1	ND	ND	ND	4.60 ±0.06	**4.60**
M2	1.40 ±0.06	ND	ND	11.40 ±0.05	**12.80**
M3	7.60 ±0.05	ND	7.00 ±0.11	ND	**14.60**
M4	9.00 ±0.092	ND	ND	ND	**9.00**
M5	40.00 ±0.29	ND	ND	ND	**40.00**
M6	ND	ND	ND	ND	**0.00**
M7	ND	ND	3.20 ±0.04	ND	**3.20**
M8	ND	4.40 ±0.00	ND	ND	**4.40**
M9	0.80 ±0.18	ND	1.80 ±0.02	ND	**2.60**
M10	22.20 ±0.05	ND	3.60 ±0.04	ND	**25.80**
M11	9.60 ±0.12	ND	7.00 ±0.02	ND	**16.60**

*No. of determinations: 3 *ND: Not Detected

Arsenic can cause acute toxicity, genetic toxicity, reproductive and developmental toxicity, biochemical toxicity, and chronic toxicity [18]. Arsenic concentrations existing in eye shadow samples ranged from 2.6 ppm in sample S6 (which was locally made) to 139 ppm in S2 (the Chinese brand). Six samples were free of arsenic. Samples S1, S2, and S3 (which were all manufactured in China and with low price) exceeded the safe limits with high concentrations. The maximum level reported for arsenic in eye shadow was 2.95 ppm [8]. Arsenic concentrations existing in eyeliner samples ranged from 6.8 ppm (L6) to 17 ppm (L4), which were both locally made products. Seven samples were free of arsenic. The four samples containing arsenic exceeded the allowed limits of arsenic in cosmetics. Mascara results ranged from 0.8 ppm (M9) to 40 ppm (M5). Four samples were free of arsenic. Five samples exceeded the allowed limits of arsenic.

Mercury can cause anxiety, autoimmune diseases, difficulty with balance, fatigue, hair loss, irritability, memory loss, restlessness, recurrent infections, tremors, damage to brain, lungs, and kidney [19]. It was detected in only two eye shadow samples S11 and S12 with

concentrations of 20.8 ppm and 0.2 ppm; respectively. Sample S11 exceeded the limits of the four organizations, while sample S12 was safe. For eyeliner samples, Mercury was detected in only one eyeliner sample L4 (the locally made) with a concentration of 0.2 ppm, which is considered to be safe. Likewise, in mascara samples, Mercury was detected in only one sample, which was M8 (which was manufactured in Italy) with a concentration of 4.4 ppm. This sample exceeded the maximum allowed limit for mercury in cosmetics, which was 1 ppm.

Lead toxicity is a general problem; it is a substantial critical disease in children. Exposure to low levels of lead has been dangerous to pregnant women [8]. The results in eye shadow samples ranged from 1.2 ppm (S6 the locally made) to 16.6 ppm (S3 the Chinese made with low price). Samples S5 (which was locally made), and S8 was lead-free. The analyzed samples had a concentration of lead less than 20 ppm [17]. The maximum level in eye shadow reported for Pb was 153.89 ppm [20]. The results in eyeliner ranged from 0.2 ppm (L2) to 7.6 ppm (L10). Three samples were lead-free. The maximum levels of lead reported in eyeliner were 123.2 ppm [21] and 33.8 ppm [21], which were higher than the concentrations in this study. For mascara samples, the results ranged from 1.8 ppm (M9) to 7 ppm (M3 and M11). Six samples were lead-free. All samples that contained lead had concentrations less than 10 ppm, which has been the maximal lead limit allowed in cosmetics. The maximum level in Mascara reported for lead was 18.5 ppm.

Antimony Chronic exposure may intensively cause the irritation of the eyes, skin, and lungs [22]. It was detected in only one sample S8, which was Chinese manufactured with medium priced with a concentration of 6.4 ppm. This sample exceeded the allowed limit according to Health Canada, in which the permitted limit is 5 ppm (Table 8). Based on the German federal government, it is safe since the maximum limit is 10 ppm. For eyeliner samples, Antimony was detected in only two eyeliner samples L1 and L2 (which were both Chinese manufactured) with concentrations of 0.2 ppm and 17.6 ppm; respectively. Sample L2 exceeded the allowed limit according to the German federal government. In mascara samples, Antimony was detected in two samples, M1 and M2, which were also Chinese made with concentrations of 4.6 ppm and 11.4 ppm; respectively. Both exceeded the allowed limits.

Overall, the highest total of heavy metals concentration in the tested sample was found in eye shadow samples of S1, S2, and S3. These samples were made in China, they were cheap, and had low quality. Only two samples were free of heavy metals, mascara sample M6, which was locally manufactured, and the other sample was eyeliner sample L8, which was made in Germany.

Table 12: Comparative Values in PPM for Heavy Metals in Eye Cosmetic Products Reported in The Literature.

Type	Reff.	As	Hg	Pb	Sb
Eye shadow	**This Study**	**139**	**20.8**	**16.6**	**6.4**
	[20]	-	-	1.35 -153.89	-
	[8]	2.95		11.9	2.12
	[5]	2.3	-	16.8	-
	[14]	-	-	11.9	-
	[23]	-	-	0.0815	-
	[24]	0.058	1967.704	45.859	-
	[25]			55	
	[26]	1.85			0.76
Eyeliner	**This study**	**17**	**0.2**	**7.6**	**17.6**
	[10]	2	-	1	-
	[14]	-	-	1071	-
	[27]	-	-	123.2	-
	[21]	-	-	33.8	-
Mascara	**This study**	**40**	**4.4**	**7**	**11.4**
	[8]	-	0.0095	2.18	-
	[21]	-	-	18.5	-

Associated Elements (Ag, Al, Ba, Ca, Co, Cr, Cu, Ni):

Allowed limits in cosmetics were not settled for these elements (Ag, Al, Ba, Ca, Co, Cr, Cu, Ni) due to their small toxicity. Although, long-time exposure to these elements might lead to reaching levels causing some health hazards. Tables below have illustrated ICP results for these elements.

Ag, Al, Ba, Ca

No trace amount of silver was detected in any of the samples. Meanwhile, **Aluminum** represents about 8% of the Earth's crust and has no known biological function. There have been several studies on how aluminum might have a role causing Alzheimer's Disease. These studies supported the view that reducing aluminum exposure might provide significant public health benefits [23]. Eye shadow results of Aluminium varied from 55320 ppm in sample S11 (which was the highest level of Al in this study) to 1806 ppm in sample S3, and three samples were free of Aluminium. Similar high values were reported with 50000 ppm [8] (Table.13). For eyeliner, the results varied from 226.2 ppm (Sample L1) to 10190 ppm (sample L4), and four samples were Al-free. Mascara results varied from 11204 ppm in sample M5 to 5.80 ppm in sample M1.

Barium plays no critical biological roles, as mentioned in the literature. There are signs of Ba poisonousness such as

nausea, headache, agitation, dyspnea, and cardiac arrhythmia [28]. Ba was detected in 16 samples. The highest level in eye shadow was 41.2 ppm in the sample S3, 17.2 ppm in eyeliner sample L4, 258 ppm in mascara sample M3.

Calcium is necessary for many functions in human health. It was detected in all the samples under the study with high levels, likewise in the literature, as shown in Table.16. The highest level detected among all the samples under the investigation was in eye shadow sample S2 with 80000 ppm.

Table 13: ICP results for (Ag, Al, Ba, Ca, Co, Cr, Cu, Ni) in Eye shadow samples

Sample No.	Elements concentration ppm ±SD							
	Ag	Al	Ba	Ca	Co	Cr	Cu*	Ni*
S1	ND	2312 ±0.10	95.4 ±0.01	28540 ±1.60	8.2 ±0.00	9.6 ±0.00	167 ±0.01	2.8 ±0.00
S2	ND	10448 ±0.86	74.6 ±0.00	80000 ±11.70	1.6 ±0.01	11.2 ±0.01	337.4 ±0.02	10.2 ±0.01
S3	ND	1806±0.12	41.2 ±0.00	66580 ±3.60	70 ±0.01	10.2 ±0.00	7.2 ±0.00	10.2 ±0.02
S4	ND	ND	0.6 ±0.00	1387.2 ±0.20	879.2 ±0.05	43 ±0.00	ND	1.4 ±0.00
S5	ND	ND	23.4 ±0.00	3130 ±0.30	ND	42380 ±1.11	0.6 ±0.00	6 ±0.01
S6	ND	3030 ±0.19	7.2 ±0.00	3166 ±0.32	493.2 ±0.02	80 ±0.04	4.4 ±0.00	11.8 ±0.01
S7	ND	ND	ND	3402 ±0.42	515.2 ±0.02	16 ±0.00	ND	0.8 ±0.00
S8	ND	4580 ±0.1	ND	3202 ±0.04	ND	8.4 ±0.00	ND	ND
S9	ND	6320 ±1.03	3.6 ±0.00	5702 ±0.15	654.6 ±0.02	8.4 ±0.00	ND	8 ±0.00
S10	ND	ND	ND	4098 ±0.43	731.6 ±0.01	1.6 ±0.00	ND	ND
S11	ND	55320 ±1.00	81.2 ±0.00	17234 ±1.6	28.6 ±0.012	75.4 ±0.00	8 ±0.00	14.8 ±0.01
S12	ND	4430 ±0.35	3.8 ±0.00	3020 ±0.328	1356.2 ±0.07	8 ±0.00	ND	6.6±0.017

Cr, Cu, Ni) in Eye shadow samples *No. of determinations: 3 *ND: Not Detected * Cu and Ni results [17]

Table 14: ICP results for (Ag, Al, Ba, Ca, Co, Cr, Cu, Ni) in Eyeliner samples

Sample No.	Elements concentration ppm ±SD							
	Ag	Al	Ba	Ca	Co	Cr	Cu	Ni
L1	ND	226.2 ±0.02	ND	45 ± 0.02	ND	10.8 ±0.00	ND	2.8 ±0.01
L2	ND	6026 ± 0.40	8.6 ± 0.00	420 ±0.02	ND	3.4 ±0.00	137.2±0.004	ND
L3	ND	ND	3.8 ±0.00	802.4 ±0.03	331.2 ±0.02	139.8 ±0.01	ND	0.6 ±0.01
L4	ND	10190 ± 0.83	17.2 ±0.00	1361.2 ±0.14	1071.8 ±0.04	ND	ND	ND
L5	ND	823.6 ±0.08	ND	445 ±0.03	18.4 ±0.00	139.4 ±0.00	ND	ND
L6	ND	ND	ND	871± 0.07	1188.2 ±0.06	ND	ND	9.6 ±0.02
L7	ND	ND	ND	932.2 ± 0.09	866.6 ± 0.02	7.6 ± 0.00	ND	ND
L8	ND	2550 ± 0.10	4 ± 0.00	79.4 ± 0.01	45.8 ± 0.01	12.2 ± 0.00	ND	1.6 ± 0.01
L9	ND	287 ± 0.17	ND	666.4± 0.06	262 ± 0.02	12.6 ± 0.00	ND	4.8 ± 0.01
L10	ND	ND	ND	1132.2 ±0.20	921.8 ± 0.02	ND	ND	1 ± 0.01
L11	ND	1058.4 ± 0.06	1.6 ± 0.00	606.6 ± 0.06	576.6 ± 0.01	7.6 ± 0.00	ND	5.8 ± 0.01

*No. of determinations: 3 *ND: Not Detected

Table 15: ICP results for (Ag, Al, Ba, Ca, Co, Cr, Cu, Ni) in Mascara samples

Sample No.	Elements concentration ppm ±SD							
	Ag	Al	Ba	Ca	Co	Cr	Cu	Ni
M1	ND	5.80 ±0.013	ND	80.40 ±0.03	ND	14.40 ±0.00	0.80 ±0.00	ND
M2	ND	6544 ±0.34	258.0 ±0.01	1667.4 ±0.18	1.80 ±0.00	11.20 ±0.00	0.40 ±0.00	2.60 ±0.00
M3	ND	ND	ND	473.0 ±0.04	163.8 ±0.01	48.00 ±0.00	43.40 ±0.00	16.60 ±0.01
M4	ND	1192 ±0.23	ND	2530 ±0.28	89.60 ±0.01	8.80 ±0.00	ND	1.00 ±0.00

M5	ND	11204 ±0.00	10.40 ±0.20	11918 ±0.00	7.400 ±0.76	10.80 ±0.00	0.60 ±0.01	1.80 ±0.00
M6	ND	ND	ND	95.00 ±0.03	263.8 ±0.01	4.20 ±0.00	ND	2.20 ±0.01
M7	ND	ND	ND	1091 ±0.09	288.4 ±0.02	3.80 ±0.01	ND	3.60 ±0.01
M8	ND	821.4 ±0.09	ND	936.4 ±0.05	1.80 ±0.02	5.20 ±0.00	ND	0.40 ±0.01
M9	ND	ND	ND	289.0 ±0.07	303.0 ±0.00	4.00 ±0.00	ND	ND
M10	ND	374.0 ±0.02	0.60 ±0.00	3138 ±0.27	152.8 ±0.01	5.40 ±0.00	ND	2.80 ±0.00
M11	ND	ND	ND	510.4 ±0.14	195.8 ±0.01	8.20 ±0.00	ND	10.00 ±0.01

*No. of determinations: 3 *ND: Not Detected

Co, Cr, Cu, Ni

These metals are fundamental to human beings, although there have been some arguments around Chromium. Cobalt in vitamin B12 has a biological role that is necessary for methylation reactions and various rearrangements [28].

Cobalt was found in 29 samples out of 34. The highest level of Co found in eye shadow samples was in the sample S12 which was 1356 ppm, Eyeliner samples was in the sample L6 which was 1188.2 ppm, and for mascara, it was in the sample M9 which was 303ppm.

Chromium is essential for glucose regulation and insulin function. Only three samples were free of Chromium; it was found in a significant value in eye shadow (sample S5) 24380 ppm, 139.8 ppm in eyeliner (sample L3), and 43 ppm in mascara (sample M3).

Copper is essential for many procedures like biological electron transfer, and oxygen atom transfer [28]. Copper was detected in 11 samples out of 34. The highest value in eye shadow was in sample S1, and it was 167 ppm [17]. All eyeliner samples were free of Cu except sample L2 which had 137.2 ppm of Cu. For mascara, the highest value was 43.4 ppm in M3.

Nickel is fundamental for the action of urease, acetyl coenzyme synthase, and many hydrogenases [28]. Nickel was detected in 26 samples. The highest value detected in eye shadow samples was in S11, and it was 14.8 ppm [17], 5.8 ppm in eyeliner sample L11, and 16.6 ppm in mascara sample M3.

Regardless of their significance to a human, these metals would cause a few health problems; the allergy is the common issue. Cu is known as a weak allergen, but Ni was named allergen of the year in 2008 by the American Contact Dermatitis Society (ACDS). These four metals were also suggested to play a role in breast cancer [28].

Table 16: Comparative Values in PPM for Elements in Eye Cosmetic Products Reported in The Literature

Type	Reff.	Ag	Al	Ba	Ca	Co	Cr	Cu	Ni
Eye shadow	This study	-	55320	95.4	80000	1356.2	42380	337.4	14.8
	[20]	-	-	-	-	-	-	-	31.91
	[8]	-	50000	2000	2000	31.3	7000	37.3	46.8
	[5]	-	-	-	-	41.2	5470	-	49.7
	[23]	-	-	-	-	0.30	0.29	-	4.15
	[24]	-	-	878.23	-	-	-	102.01	13.71
	[26]	-	-	74	4700	2.42	15.3	-	-
	[25]	-	-	-	-	258.33	150	465	359.44
	[21]	-	-	-	-	17.1	146	194	30.8
Eyeliner	This study	-	10190	17.2	1361.2	1188.2	139.8	137.2	5.8
	[10]	-	19321	-	-	-	-	-	120
	[14]	-	-	-	-	0.72	0.078	302.2	1.014
	[27]	-	-	-	-	-	39.9	-	12.1
	[21]	-	-	-	-	43.6	45.1	67.2	55.7
Mascara	This study	-	11204	258	11918	303	48	43.4	16.6
	[8]	3.76	-	-	-	20.4	17.1	1.04	31.4
	[21]	-	-	-	-	16.6	21.3	16.9	589

Titanium Dioxide and Zinc Oxide:

Two metals: titanium and zinc were investigated in the samples under the study. Typically, Ti and Zn were present as titanium dioxide and zinc oxide in cosmetics. Tables 17,18, and 19 show the percentage of the compounds TiO_2 and ZnO in the samples. The rates of titanium dioxide and Zinc oxide in the samples were found by applying the following equations:

$$TiO_2\% = \text{Ti results} \times \left(\frac{\text{Mwt. } TiO_2}{\text{At wt. Ti}}\right) \times 10^{-7}$$

$$ZnO\% = \text{Zn results} \times \left(\frac{\text{Mwt. ZnO}}{\text{At wt. Zn}}\right) \times 10^{-7}$$

Where Mwt.: molecular weight, and At wt.: atomic weight

The rates of titanium dioxide and Zinc oxide in the samples have been shown in the following Tables:

Table 17: Titanium Dioxide and Zinc Oxide Percentages in Eye shadow

Sample	Ti results ppm±SD	TiO_2 %	Zn results ppm±SD	ZnO %
S1	11.8 ±0.01	1.97 ×10 $^{-6}$	15.4 ±0.01	1.91×10 $^{-6}$
S2	121.8 ±0.00	2.03×10 $^{-5}$	1.8 ±0.00	2.23×10 $^{-7}$
S3	28.6 ±0.00	4.77×10 $^{-6}$	110 ±0.01	1.36×10 $^{-5}$
S4	15 ±0.00	2.50×10 $^{-6}$	426.2 ±0.02	5.28×10 $^{-5}$
S5	513.6 ±0.04	8.56×10 $^{-5}$	ND	ND
S6	620.8 ±0.05	1.04×10 $^{-4}$	18.4 ±0.01	2.28×10 $^{-6}$
S7	65.2 ±0.01	1.09×10 $^{-5}$	12.4 ±0.01	1.54×10 $^{-6}$
S8	234 ±0.01	3.90×10 $^{-5}$	ND	ND
S9	136 ±0.01	2.27×10 $^{-5}$	435.8 ±0.03	5.40×10 $^{-5}$
S10	7.2 ±0.01	1.20×10 $^{-6}$	4912 ±0.044	6.09×10 $^{-4}$
S11	78.2 ±0.01	1.30×10 $^{-5}$	13790 ±0.23	1.71×10 $^{-3}$
S12	18.2 ±0.00	3.03×10 $^{-6}$	8898 ±0.49	1.10×10 $^{-3}$

*No. of determinations: 3 *ND: Not Detected

Table 18: Titanium Dioxide and Zinc Oxide Percentages in Eyeliner

Sample	Ti results ppm±SD	TiO_2 %	Zn results ppm±SD	ZnO %
L1	0.6 ± 0.00	1.00×10 $^{-7}$	137.4± 0.01	1.70×10 $^{-5}$
L2	491.4 ±0.02	8.19×10 $^{-5}$	23.8 ±0.01	2.95×10 $^{-6}$
L3	54.6 ±0.02	9.10×10 $^{-6}$	57 ±0.00	7.07×10 $^{-6}$
L4	25.8 ±0.00	4.30×10 $^{-6}$	175.8 ±0.02	2.18×10 $^{-5}$
L5	157 ±0.05	2.62×10 $^{-5}$	ND	ND
L6	2.4 ±0.00	4.00×10 $^{-7}$	ND	ND
L7	7.4 ± 0.00	1.23×10 $^{-6}$	ND	ND
L8	664.6 ± 0.00	1.11×10 $^{-4}$	87.8 ± 0.01	1.09×10 $^{-5}$
L9	566.2 ± 0.04	9.44×10 $^{-5}$	ND	ND
L10	1.2 ± 0.00	2.00×10 $^{-7}$	0.2 ± 0.00	2.48×10 $^{-8}$
L11	759.2 ± 0.07	1.27×10 $^{-4}$	2 ± 0.00	2.48×10 $^{-7}$

*No. of determinations: 3 *ND: Not Detected

Table 19: Titanium Dioxide and Zinc Oxide Percentages in Mascara

Sample	Ti results ppm±SD	TiO_2 %	Zn results ppm±SD	ZnO %
M1	ND	ND	58.8±0.00	7.29×10 $^{-6}$
M2	70.6±0.01	1.18×10 $^{-5}$	257.4±0.02	3.19×10 $^{-5}$
M3	17.8 ±0.01	2.97×10 $^{-6}$	240.6 ±0.02	2.98×10 $^{-5}$
M4	1.2 ±0.002	2.00×10 $^{-7}$	21.4 ±0.01	2.65×10 $^{-6}$
M5	189.4 ±0.01	3.16×10 $^{-5}$	78 ±0.00	9.67×10 $^{-6}$
M6	0.8 ±0.00	1.33×10 $^{-7}$	267 ±0.01	3.31×10 $^{-5}$
M7	23.2 ±0.00	3.87×10 $^{-6}$	ND	ND
M8	0.6 ±0.00	1.00×10 $^{-7}$	42 ±0.00	5.21×10 $^{-6}$
M9	14.6±0.00	2.43×10 $^{-6}$	73±0.01	9.05×10 $^{-6}$
M10	4.8±0.00	8.00×10 $^{-7}$	27.4±0.00	3.4×10 $^{-6}$
M11	0.4±0.00	6.67×10 $^{-8}$	83.8±0.00	1.04×10 $^{-5}$

*No. of determinations: 3 *ND: Not Detected

Titanium (IV) oxide is the naturally occurring compound. It helps to minimize the transparency of product formulas, and increase the opaqueness. TiO_2 as well as scatters, reflects or absorbs light (including UV radiation from the sun), which can damage products [29].

FDA and SASO have approved the use of titanium dioxide for the purpose in cosmetic products at concentrations up to 25% [30, 31]. While zinc oxide has been approved to be used as a white pigment in all cosmetic formulation under the percentage of 25%. As shown in the previous tables, all samples under the investigation showed that titanium dioxide and zinc oxide proportions were at levels below the acceptable limit, so they were considered to be safe.

Titanium was found in all eye shadow samples with concentrations ranging between 7.2 (S10) to 620.8 ppm (S6). The highest concentration of titanium was found in sample S6. For eyeliner samples, titanium was detected in all samples with the level from 0.6 ppm (sample L1) which had a dark colour, to 759.2 ppm (sample L11) which had a bright colour. Meanwhile, Titanium was found in all mascara samples except M1, with concentrations between 0.4 ppm in sample M11, to 189.4 ppm in sample M5. The highest level was found in M5 which had a light blue colour. However, the higher

proportion of titanium dioxide was found in the sample S6 which had a bright colour with 1.04×10^{-4} %.

Zinc was found in all eye shadow samples except S5 and S8. The concentrations ranged from 1.8 ppm in S2 to 13790 ppm in S11. Four eyeliner samples were free of zinc. The concentrations ranged from 0.2 (L10) to 175.8 ppm (L4). The highest concentration of zinc was found in L4. In mascara samples, zinc was found in all samples except M7; the levels ranged from 21.4 ppm (M4) to 267 ppm (M6). Overall, the highest proportion of zinc oxide was found in three high priced samples S10, S11, and S12 with 6.09×10^{-4}, 1.71×10^{-3}, 1.10×10^{-3}% ; respectively.

Elemental analyzer CHNS results:

Elemental analyzer equipment was used to confirm the presence of carbon and sulfur on eye cosmetics samples. The results of CHNS for eye shadow samples have been given in the following table:

Table 20: Carbon and sulfur results for eye shadow, eyeliner and mascara samples using Elemental analyzer CHNS

Eye shadow sample	C %	S%	Eyeliner sample	C %	S%	Mascara sample	C %	S%
S1	20.88	ND	**L1**	77.79	ND	**M1**	37.73	ND
S2	10	ND	**L2**	57.14	ND	**M2**	25.29	ND
S3	15.65	ND	**L3**	63.96	ND	**M3**	54.36	ND
S4	14.11	0.3	**L4**	38.69	0.41	**M4**	66.04	ND
S5	17.16	0.3	**L5**	34.36	0.04	**M5**	29.47	ND
S6	13.63	ND	**L6**	40.47	ND	**M6**	21.25	ND
S7	9.18	0.4	**L7**	53.16	ND	**M7**	32.88	ND
S8	9.48	ND	**L8**	57.12	ND	**M8**	34.66	ND
S9	9.52	0.4	**L9**	57.43	ND	**M9**	33.92	ND
S10	8.42	ND	**L10**	46.77	ND	**M10**	29.43	ND
S11	9.22	3.71	**L11**	59.2	0.12	**M11**	39.05	ND
S12	47.76	0.12	-	-	-	-	-	-

*ND: Not Detected

SASO has allowed to carbon black to be used in cosmetic products as a colorant [32]. Coal was found in all eye shadow samples. The highest level of carbon was 47.76% in sample S12, which was manufactured in the USA. The lowest level was detected in the Italian sample S10 with 9.52%. Meanwhile, sulfur was detected in only five samples. Sample S11 contained the highest level of sulfur with 3.71%, by returning to ICP-OES results for lead, it was found that this sample contained lead with 7.8ppm, also XRF results for the same sample confirmed the presence of lead sulfide PbS (Galina) [17]. For eyeliners, the highest level was 77.97% in the Chinese sample L1, and the lowest level was 34.36% in the Saudi sample L5. Sulfur was detected in 3 samples with low levels (Table 20). Finally, the highest level of carbon discovered in mascara samples was 66.04% in the sample M4 which was manufactured in Saudi, and the lowest level was 21.25% also in the Saudi made sample M6. Furthermore, the mascara samples were free of sulphur, which indicated that all of the mascara samples were free of Galina (Table 20).

Comparing these results, it could be seen that carbon existed in higher levels in eyeliner samples.

Relationship between results of all experiments:

Eye shadow samples S1, S2 and S3 (the cheaper priced samples and made in China) successfully passed the microbiological examination and the melting point test, the total heavy metals' concentration of these samples was high, with values reaching to 151.6 ppm in sample S3 due to the high level of lead and arsenic in these samples. Therefore, these samples were not safe for use. Samples S4, S5, and S6 which were locally manufactured have passed all SASO tests except the limits of arsenic in samples S4 and S6. Samples S7, S8, and S9 were intermediately priced and passed all SASO tests, so these samples were safe. The expensively priced samples S10, S11, and S12 have levels of arsenic and mercury above the permissible limits, but the total of heavy metals concentration was low compared to the cheaper samples.

All of the eyeliner samples have passed all SASO tests. In the same way, mascara samples have passed the microbiological examination, the sample M1 which was cheaply priced and made in China had a higher count among all samples, it reached 67 CFU/ml, and it was slightly below the permissible limit. Overall, all mascara samples passed the SASO tests.

CONCLUSION

ICP-OES determined the levels of heavy metals in different eye cosmetic products after wet digestion for the samples. Precise and accurate results were obtained with this method, which confirmed that it is usable for routine tests. The overall results of this research revealed that heavy metals present in eye cosmetics products were within the acceptable limits, while some of those lower price products imported from China can be harmful, as they failed some tests of SASO. The prolonged use of such products can be a potential threat to human health since heavy metals can accumulate in human tissues over time and induce allergic problems.

There is, therefore, a definite need for minimizing health risks related to cosmetic products use, and it is highly recommended to control the quality of these products, and warn the consumers to be careful when they purchase low price products.

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