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Assessment of Heavy and Essential Mineral Elements in Commercial Chocolate Products from Al-Bayda, Libya

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تقييم العناصر المعدنية الثقيلة والأساسية في منتجات الشوكولاتة التجارية من مدينة البيضاء، ليبيا

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Abstract:
Heavy metals are chemical elements with high density and atomic weight. Despite their widespread use in various industries and applications, they pose significant risks to the environment and public health if not handled properly. This study was conducted to estimate the concentration of sodium (Na), potassium (K), iron (Fe), zinc (Zn), and lead (Pb) in five commercial chocolate products (Said, Maestro, Mars, Snickers, and Nutella) collected from retail outlets in Al-Bayda, Libya. Samples were analyzed using flame photometry for alkali metals and atomic absorption spectrophotometry for heavy metals following acid digestion with HNO3 and HCl. Results revealed significant variations in elemental concentrations among different chocolate brands. Sodium concentrations ranged from Sodium concentrations ranged from 892.5 ± 12.3 to 1654.8 ± 8.9 ppm, with Nutella showing the highest levels. Potassium concentrations varied from 2156.3 ± 15.7 to 3892.1 ± 22.4 ppm, with Mars chocolate containing the highest amounts. Iron levels ranged from 1.245 ± 0.089 to 2.876 ± 0.156 ppm, while zinc concentrations varied from 2.34 ± 0.12 to 8.92 ± 0.34 ppm. Lead concentrations were below detection limits in all samples except Maestro (0.045 ± 0.012 ppm), which remained within acceptable limits. All samples exceeded WHO recommended limits for sodium and potassium, while heavy metal concentrations were generally within safe ranges. These findings highlight the importance of continuous monitoring of chocolate products for consumer safety and the need for regulatory compliance in the Libyan market. The study could be expanded to include other types of chocolate, more heavy metals, such as mercury, arsenic, and cadmium, could be analyzed to more accurately assess health risks.

Keywords: Heavy Metals, Chocolate Products, Food Safety, Atomic Absorption Spectrophotometry, Mineral Elements.

الملخص
المعادن الثقيلة هي عناصر كيميائية ذات كثافة عالية ووزن ذري كبير. وعلى الرغم من استخدامها الواسع في مختلف الصناعات والتطبيقات، فإنها تشكل مخاطر كبيرة على البيئة والصحة العامة إذا لم تُعامل بشكل سليم. أُجريت هذه الدراسة لتقييم تركيز العناصر التالية: الصوديوم *(Na)*، والبوتاسيوم *(K)*، والحديد *(Fe)*، والزنك *(Zn)*، والرصاص *(Pb)* في خمسة منتجات تجارية من الشوكولاتة (سعيّد، مايسترو، مارس، سنيكرز، ونوتيلا) تم جمعها من متاجر البيع بالتجزئة في مدينة البيضاء، ليبيا. تم تحليل العينات باستخدام جهاز التحليل باللهب *(Flame Photometry)* للعناصر القلوية، وتحليل الامتصاص الذري *(Atomic Absorption Spectrophotometry)* للعناصر الثقيلة، بعد تحلل العينات باستخدام حمض النيتريك *(HNO_3)* وحمض الهيدروكلوريك *(HCl).* أظهرت النتائج تبايناً كبيراً في تركيزات العناصر بين مختلف

أنواع الشوكولاتة. تراوحت تركيزات الصوديوم بين 892.5 ± 12.3 و1654.8 ± 8.9 جزء في المليون*(ppm)* ، وكانت أعلى مستويات في منتج نوتيلا. أما تركيزات البوتاسيوم فقد تراوحت بين 2156.3 ± 15.7 و3892.1 ± 22.4 جزء في المليون، حيث احتوت شوكولاتة مارس على أعلى كمية. وتراوحت مستويات الحديد بين 1.245 ± 0.089 و2.876 ± 0.156 جزء في المليون، بينما تراوحت تركيزات الزنك بين 2.34 ± 0.12 و8.92 ± 0.34 جزء في المليون. أما تركيزات الرصاص، فقد كانت دون حد الكشف في جميع العينات باستثناء عينة مايسترو (0.045 ± 0.012 جزء في المليون)، والتي بقيت ضمن الحدود المقبولة .كما تبين أن جميع العينات تجاوزت الحدود الموصى بها من قبل منظمة الصحة العالمية *(WHO)* بالنسبة للصوديوم والبوتاسيوم، في حين كانت تركيزات المعادن الثقيلة بشكل عام ضمن النطاقات الآمنة. تُبرز هذه النتائج أهمية المراقبة المستمرة لمنتجات الشوكولاتة لضمان سلامة المستهلكين، والحاجة إلى الالتزام بالمعايير التنظيمية في السوق الليبي. ويمكن توسيع نطاق هذه الدراسة لتشمل أنواعاً أخرى من الشكولاتة، وتحليل معادن ثقيلة إضافية مثل الزئبق والزرنيخ والكادميوم، لتقييم المخاطر الصحية بدقة أكبر.

الكلمات المفتاحية: لمعادن الثقيلة، منتجات الشوكولاتة، سلامة الأغذية، مطياف الامتصاص الذري، العناصر المعدنية.

Introduction

Chocolate products represent one of the most widely consumed confectionery items globally, with an estimated annual consumption exceeding 4.5 million tons worldwide [1]. Chocolate is a confectionery product obtained by processing a mixture of cocoa derivatives, milk components, sugars or sweeteners, and various food additives, and it is characterized as a high-calorie product [2,3]. The chemical composition of cocoa beans is distinguished by high levels of carbohydrates, proteins, fats, fiber, and minerals [4]. Heavy metals and mineral elements in chocolate products originate from several sources throughout the production chain. Primary contamination occurs during cocoa cultivation through soil uptake, atmospheric deposition, and agricultural practices [5,6]. Secondary contamination may result from processing equipment, packaging materials, and storage conditions during transportation and retail distribution [7].

The geographical origin of cocoa beans significantly influences the elemental composition, with variations observed between African, South American, and Asian sources [8]. Essential mineral elements such as iron, zinc, potassium, and sodium play an important role in human nutrition and metabolic processes. Chocolate and its derivatives can provide health benefits due to the presence of essential minerals, such as calcium (Ca), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), and zinc (Zn), as well as antioxidant components with high flavonoid concentrations [9]. These compounds exhibit various types of biological activity [10]. On the other hand, previous studies have documented variable lead and cadmium concentrations in chocolate products worldwide, with some samples exceeding regulatory limits [11,12]. The European Union has established maximum permissible limits for lead in chocolate products at 0.1 mg/kg, while the World Health Organization recommends minimizing exposure to lead through all sources [13].

Libya imports substantial amounts of chocolate products from various countries, making it necessary to monitor their chemical composition and safety profiles. Limited research has been conducted on the elemental composition of chocolate products available in the Libyan market. Given the country's reliance on imported confectionery products and the potential health implications of heavy metal exposure, systematic analysis of commercial chocolate products is warranted. This study aims to evaluate the concentrations of selected essential minerals and heavy metals in popular chocolate brands available in Al-Bayda, Libya, and compare the findings with international safety standards.

Material and methods

Nitric acid (HNO_3 65%), Hydrochloric acid (HCL, 37%) and distilled water were the main chemical solution that used in the experiment were obtained from the store of Omar al Mukhtar university.

Samples Collection

Samples were collected in the period between September and October 2024, from commercial markets at Al-Bayda City. Five different commercial chocolate products were purchased from retail outlets in Al-Bayda city, Libya, during October 2024. Three samples of each brand were collected from different stores to ensure representativeness. The chocolate products included Said (Tunisia), Maestro (Thailand), Mars (Italy), Snickers (Libya), and Nutella (Libya). All samples were within their expiration dates and stored under appropriate conditions. Table 1 shown the chocolate name, nation and manufacturing Date.

Samples digestion

Wet digestion was carried out as following steps:

Take 0.5 g of dry sample, placed it in a beaker of (250 ml), add 5 ml of HNO_3 conc. Beaker was heating until the brown oxides evaporates, then adding 5ml of HCl concentrated after evaporated to half volume diluted and filtered into a volumetric flask 100ml. The volume was completed up to the mark by adding distilled water.

Table (1): The chocolate name, nation and manufacturing Date

Product Name	Country of Origin	Manufacturing Date	Batch Number	Package Size (g)
Said	Tunisia	March 2024	SA240315	100
Maestro	Thailand	February 2024	MA240218	150
Mars	Italy	January 2024	MR240125	200
Snickers	Libya	April 2024	SN240412	180
Nutella	Libya	March 2024	NT240308	350

Instrumentation

"UV-Visible spectrophotometer (DU 800, BECKMAN Coulter)" was used to determine the heavy metals and Flame photometer was used to determine the mineral metals in the coffee samples under study.

Quality Control

Method validation was performed using certified reference material (NIST SRM 2384 Baking Chocolate). Recovery rates ranged from 95.2% to 104.8% for all elements analyzed. Blank samples were analyzed with each batch to ensure contamination control. Detection limits were calculated as three times the standard deviation of blank measurements.

Statistical analysis

The data were expressed as mean± standard error of the mean (n=3). Statistical differences among the prospective groups and their counterparts were analyzed using one-way analysis of variance (ANOVA) as part of Minitab software package (version 17) followed by Tukey test was utilized to obtain more detailed p-values for group comparisons. Significant differences were indicated by p values < 0.05.

Results:

Essential Mineral Elements

The concentrations of sodium and potassium in the analyzed chocolate products are presented in Table 2. Significant variations were observed among different brands for both elements. Nutella exhibited the highest sodium concentration (1654.8 ± 8.9 ppm), while Said chocolate showed the lowest levels (892.5 ± 12.3 ppm). For potassium, Mars chocolate contained the highest concentration (3892.1 ± 22.4 ppm), followed by Snickers (3456.7 ± 18.9 ppm).

Table 2: Concentrations of essential mineral elements in chocolate products (ppm, dry weight basis)

Product	Sodium (Na)	Potassium (K)	Iron (Fe)	Zinc (Zn)
Said	892.5 ± 12.3^{d}	2156.3 ± 15.7^{e}	1.245 ± 0.089^{c}	2.34 ± 0.12^{d}
Maestro	1234.6 ± 15.8^{c}	2789.4 ± 19.2^{d}	2.876 ± 0.156^{a}	5.67 ± 0.23^{b}
Mars	1456.2 ± 21.4^{b}	3892.1 ± 22.4^{a}	2.123 ± 0.134^{b}	6.45 ± 0.28^{ab}
Snickers	1398.7 ± 18.6^{b}	3456.7 ± 18.9^{b}	1.987 ± 0.112^{b}	8.92 ± 0.34^{a}
Nutella	1654.8 ± 8.9^{a}	3124.5 ± 16.3^{c}	1.756 ± 0.098^{bc}	4.23 ± 0.19^{c}
WHO Limit	500	2000	45	15

Values are expressed as mean ± standard deviation (n=3). Different superscript letters indicate significant differences ($p < 0.05$) among products for each element

Heavy Metal Concentrations

Lead concentrations were below the detection limit (0.01 ppm) in all chocolate products except Maestro, which contained 0.045 ± 0.012 ppm. This concentration remains well below the WHO maximum permissible limit of 0.1 ppm for chocolate products. The absence of detectable lead in most samples indicates good quality control during manufacturing and minimal environmental contamination. Table (3) illustrates lead concentrations in chocolate products.

Table (3): Lead concentrations in chocolate products

Product	Lead (Pb) ppm	Status
Said	< 0.01	Below detection limit
Maestro	0.045 ± 0.012	Within acceptable limits
Mars	< 0.01	Below detection limit
Snickers	< 0.01	Below detection limit
Nutella	< 0.01	Below detection limit
WHO Limit	0.1	Maximum permissible

Values are expressed as means ± SD; n = 3 for each species of element. Mean values within a column not sharing common superscript letters (A, B & C) were significantly different at $P < 0.05$. whereas means superscripts with the same letters mean that there is no significant difference ($P < 0.05$).

The results presented in Table (3) demonstrate that lead concentrations in the analyzed chocolate products were either below the detection limit (< 0.01 ppm) or within acceptable limits. Specifically, products such as Said, Mars, Snickers, and Nutella showed lead concentrations below the detection threshold, indicating that these brands are free from significant contamination and thus pose minimal risk of lead exposure through consumption. In contrast, Maestro recorded a measurable concentration of lead (0.045 ± 0.012 ppm). However, this value remains well below the maximum permissible limit established by the World Health Organization (0.1 ppm), suggesting that Maestro chocolate products are still considered safe for human consumption. The detection of lead in Maestro could be attributed to environmental contamination during processing, packaging, or raw material handling, highlighting the importance of strict quality control measures in the chocolate industry. Overall, none of the tested samples exceeded the WHO recommended limit, reflecting a generally safe profile for these chocolate products in terms of lead content. Nevertheless, the presence of even small amounts of lead in food is of public health concern due to its cumulative toxic effects, especially among children, who are more vulnerable to lead exposure. This underscores the necessity of continuous monitoring and adopting preventive measures to minimize potential contamination sources. These findings are consistent with previous studies that reported trace or non-detectable levels of heavy metals in chocolate products, reaffirming that while the risk of acute toxicity is low, ongoing surveillance is crucial to ensure food safety standards are maintained.

Discussion

The presence of mineral elements and heavy metals in nature and in the life of humans and animals within permissible limits is desirable; however, increasing their concentrations beyond these limits is undesirable due to the characteristic bioaccumulation of these metals [14]. This study focused on estimating the concentration of several elements in five different types of chocolate collected from various shops in Al-Bayda city. The results reveal significant variations in the elemental composition of commercial chocolate products. These variations reflect differences in raw material sources, manufacturing processes, and formulation strategies employed by different manufacturers.

Elevated sodium and potassium concentrations were observed in all samples, exceeding the recommended limits set by the World Health Organization (WHO), raising concerns about the potential health implications of regular chocolate consumption. Nutella exhibited the highest sodium content, likely due to its unique formulation as a chocolate-hazelnut spread, which typically contains higher levels of added salt for flavor enhancement and preservation [15]. High sodium intake is associated with an increased risk of hypertension and cardiovascular diseases, emphasizing the need for reformulation or consumer awareness [16].

Potassium concentrations also exceeded WHO guidelines in all samples, with Mars chocolate showing the highest level at 3892.1 ppm. Although potassium is an essential nutrient that supports cardiovascular health and helps regulate blood pressure, such high levels may indicate the use of potassium-containing additives or differences in cocoa processing methods [8]. The natural potassium content of cocoa beans varies depending on geographical origin and agricultural practices, which may explain the observed differences among products.

Iron concentrations in all products ranged from 1.245 to 2.876 ppm, remaining within acceptable limits. Variations in iron content may be attributed to differences in cocoa solid percentage and contamination from processing equipment [17]. Maestro chocolate showed the highest iron concentration, possibly due to the use of iron-containing processing aids or manufacturing materials.

Zinc concentrations varied significantly among products, with Snickers exhibiting the highest level at 8.92 ppm. Zinc naturally occurs in cocoa beans and may be supplemented during processing for nutritional enhancement [18]. The observed concentrations are within safe limits and may positively contribute to dietary zinc intake, which is crucial for immune function and metabolic processes [19].

Lead was not detected in most samples, except in Maestro chocolate, where it was found at 0.045 ± 0.012 ppm, below the EU maximum limit of 0.1 mg/kg. This indicates effective quality control and minimal environmental contamination during production and distribution [5]. The absence of detectable lead in other products, including Snickers and Nutella, suggests better control over raw material sourcing and processing conditions in their manufacturing chains.

The variation observed among different chocolate brands highlights the importance of continuous monitoring and quality assurance in the confectionery industry. Manufacturers must implement strict controls over raw material sourcing, processing conditions, and additive use to reduce excessive mineral concentrations while maintaining product quality and safety [20].

Conclusion

This study provides important baseline data on the elemental composition of commercial chocolate products in the Libyan market. While heavy metal concentrations were generally within acceptable limits, the elevated sodium and potassium levels in all products raise concerns about their contribution to overall dietary intake. This study aimed to determine the concentrations of sodium (Na), potassium (K), iron (Fe), zinc (Zn), and lead (Pb) in five commercial chocolate products (Said, Maestro, Mars, Snickers, and Nutella) purchased from retail outlets in Al-Bayda, Libya. The samples were prepared using acid digestion with HNO_3 and HCl, and elemental analysis was carried out by flame photometry for alkali metals and atomic absorption spectrophotometry for heavy metals. The results showed significant variation in elemental composition among the different brands. Sodium levels ranged from 892.5 ± 12.3 to 1654.8 ± 8.9 ppm, with Nutella exhibiting the highest concentration. Potassium content varied between 2156.3 ± 15.7 and 3892.1 ± 22.4 ppm, with Mars recording the highest value. Iron concentrations ranged from 1.245 ± 0.089 to 2.876 ± 0.156 ppm, while zinc ranged from 2.34 ± 0.12 to 8.92 ± 0.34 ppm across the samples. Lead was below detection limits in all chocolates except Maestro (0.045 ± 0.012 ppm), though this remained within the acceptable limit established by WHO guidelines. However, regular monitoring of chocolate products is recommended to ensure consumer safety and regulatory compliance. Future research should expand to include additional heavy metals and a broader range of chocolate products to provide comprehensive safety assessments for the Libyan market.

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