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Determination of some heavy metals in different wheat flour brands in Sulaimani, Kurdistan Region – Iraq

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Abstract: Wheat flour is one of the most important sources of nutrients, and it is widely consumed worldwide as a raw ingredient in bread and other pastries. High heavy metal concentrations in the consumed wheat products could induce higher health risks. This study evaluated the heavy metal concentrations in the most available foreign and domestic wheat flour in Sulaimani, Kurdistan Iraq. The wheat flour samples were collected from different locations, and the heavy metal concentration was measured by inductively coupled plasma optical emission spectrometry (ICP-OES). The mean values in wheat flour samples were roughly within the permissible limit set by Iraqi standard (IQS), GCC Standardization Organization (GSO) (GCC – Gulf Cooperation Council), Codex, and FAO/WHO. Heavy metals were detected in the descending order copper (Cu) > lead (Pb) > arsenic (As) > chromium (Cr) > nickel (Ni) > cadmium (Cd) > cobalt (Co). The results showed that wheat flour samples from the Sulaimani markets were slightly contaminated with Ni, and one domestic wheat flour sample with Pb was probably linked to fertiliser and soil contamination. It is concluded that wheat flour could be a source of chronic exposure to toxic heavy metals such as nickel and lead, resulting in adverse health issues later. Consequently, regular monitoring of soil contamination, water quality, and use of recommended levels of fertilisers and pesticides in the agricultural areas of Sulaimani are recommended.

Keywords: Iraqi standard; inductively coupled plasma optical emission spectrometry (ICP-OES); contamination; domestic; foreign

Wheat (*Triticum aestivum* L.) is an essential global foodstuff among the world's oldest and most widely used crops (Yaradua et al. 2019). It is recognised and consumed as a crucial agricultural source of high levels of carbohydrates and protein (Ran et al. 2016; USDA 2020). Wheat alone plays a crucial role in ensuring the food and nutrition security of the world (Erenstein et al. 2022). Wheat flour is the primary raw material for many confectionery and bakery products, such as bread and biscuits. Agricultural products absorb toxic metals from different soil, water, and air sources. These materials are deposited in other parts of roots, leaves, and grains. Pollution sourced from the air would contaminate water, soil, and plants with heavy metals (Qu et al. 2012; Li et al. 2021). Contamination occurs not only during plant growth and harvesting but also during food processing (Rai et al. 2019). Passing grains through milling rollers can significantly increase the levels of metallic trace elements as the grains degrade (Pirhadi et al. 2022).

In recent years, metallic and potentially toxic elements have become a global concern (Khaneghah et al. 2020; Terzano et al. 2021). More than 90% of exposure to potentially toxic elements (PETs) is estimated

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to result from receiving these compounds in water and food (Khaneghah et al. 2020). Crops contaminated with excessive amounts of PTEs like nickel (Ni), arsenic (As), chromium (Cr), cadmium (Cd), lead (Pb), and copper (Cu) usually come from either anthropogenic sources or human activities like mining, irrigation with sewage sludge, fertilisers, and pesticides (Yaradua et al. 2019). Industrialisation also raised the concentration of toxic metals in soil, water, and air, increasing the concentration of these elements in agricultural products (Karimi et al. 2021). Under the Joint FAO/WHO food standards programme, the permissible limits of lead and cadmium in cereal grain are 0.2 and 0.1 $mg \cdot kg^{-1}$, respectively (Codex 2021). In addition, under GSO/FDS 194/2014 and IQS 37/2010 standards, the permissible limits for lead, cadmium, and arsenic in wheat flour are 0.20, 0.10, and 1.00 $mg \cdot kg^{-1}$, respectively (COSQC 2000; GSO 2014). According to FAO/WHO, the permissible limits of whole grain cobalt, chromium and nickel are 0.03, 1.0, and 0.1 $mg \cdot kg^{-1}$, respectively (Codex 2016).

Heavy metals naturally exist in the Earth's crust at low concentrations. They can enter human, animal, and plant tissues. They bind to vital cellular components, including structural proteins, enzymes, and nucleic acids, interfering with their function (Kosek-Hoehne et al. 2017). Metallic elements may accumulate in the bone, lungs, liver, and kidneys due to their stability, non-biodegradability, and bioaccumulation potential, causing severe organ damage (Dallatu et al. 2016). Many studies have examined the levels of heavy metals in wheat flour from different countries, particularly As, Cd, Pb, Cu, Co, Cr, and Ni. These heavy metals can potentially harm human health due to their cumulative nature and toxicity, even though they are typically found in agricultural soils at low concentrations (Olu et al. 2013). Chronic exposure to lead can adversely affect children, in the form of decreased IQ or growth disorders, while adults may experience high blood pressure (Shokri et al. 2022). Cadmium exposure has been linked to an increased risk of lung, bladder, and prostate cancers. Excessive cadmium levels have also been associated with the painful bone disease known as Itai-itai (Khaneghah et al. 2020). Copper is another heavy metal commonly found in various grains (Demirözü 2002). Excessive copper consumption can harm the liver and kidneys (Barber et al. 2021). Generally, the accumulation of heavy metals in the human body due to chronic exposure can result in various health problems (Tchounwou et al. 2012). The study objective was to determine heavy metal concentrations in the most available wheat flour samples in Sulaimani City to monitor their safety according to national and international standards.

MATERIAL AND METHODS

Flour sampling. In Sulaimani City, two main types of wheat flour, whole and white flour, are readily available and widely used for producing bread and confectionery products. Twenty-seven wheat flour samples, native (domestic) and imported (foreign) ones, were randomly collected from different market locations in Sulaimani. The samples were separately stored in clean bags (polythene), labelled and kept at 5–10 °C until analysis.

Digestion and analysing. The wheat flour sample (1.0 g) was weighed and placed into dry clean polytetrafluorethylene (PTFE) vessels for digestion. Nitric acid (HNO_3; 9.0 mL) and hydrogen peroxide (H_2O_2; 1.0 mL) were added, and the vessels were placed into a microwave digestion system (ETHOS UP; Milestone, Italy) and the recommended digestion method for wheat flour followed as provided by the manufacturer. The method included heating and cooling phases. Throughout the heating stage, the samples were warmed to 210 °C for 15 min and then kept at this temperature for 15 min. Afterwards, during the cooling phase, the samples underwent active cooling to reach a temperature of 50 °C in 15 min. After microwave digestion, the solution was filtered and transferred into a 100 mL flask supplemented with distilled water up to the mark. The metal content was determined in the clear solution by inductively coupled plasma optical emission spectrometry (ICP-OES) (ICP-OES-9820 Plasma Atomic Emission Spectrometer; Shimadzu, Japan) with a mini torch, part number (P/N) S211-81448. The optimum functioning parameters produced a maximum output of 1.6 kW at a frequency of 27 MHz; argon gas grade 5 was used for plasma, nebuliser, and auxiliary gas. The gas pressure was 450 ± 10 kPa, based on the gas purity. The flow values for plasma (coolant) gas, auxiliary gas and carrier gas were set to 20, 1.5, and 1.5 $L \cdot min^{-1}$, respectively. Also, the time consumed for stabilisation, rinsing sample measurement, and rinsing were 20−30, 1.5, and 4.5 min, respectively (3 repetitions). All the analyses were performed in three technical replicates.

Statistical analysis. Statistical data analysis was performed using SPSS software (version 22.0). One-way analysis of variance (ANOVA) and post hoc test (Tukey) were used to determine significant differences. The values were expressed as mean ± stand-

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ard deviation (mean ± SD), and the probability value of $P < 0.05$ was followed.

RESULTS AND DISCUSSION

The contents (mean ± SD) of heavy metals in 27 wheat flour samples of various brands and the maximum permissible levels of the elements in wheat flour according to Iraqi standard (IQS), GCC Standardization Organization (GSO) (GCC – Gulf Cooperation Council), Codex, and FAO/WHO standards are summarised in Table 1. Generally, concentrations of As, Cd, Co, Cr, and Cu all samples were lower than the acceptable limits of 1.0, 0.1, 0.03, 1.0, and 10.0 mg·kg^{-1}, respectively, set by IQS, FAO/WHO, and Codex standards (COSQC 2000; Codex 2007; Codex 2016). In contrast, the concentration of Pb was also lower than the limit of detection in all the samples except F0, with a concentration of 0.64 mg·kg^{-1}, and referred to domestic white flour, which roughly exceeded the permissible level of 0.2 mg·kg^{-1} (COSQC 2000; Codex 2001; GSO 2014). In addition, nickel concentrations in some domestic and foreign samples, including F0, F7, F9, F16, F18, and F24 exceeded the acceptable limit of 0.1 mg·kg^{-1} set by Codex (2016). However, the study results were compared with those from the specialised literature in Table 2.

Arsenic (As). Significant variations in arsenic concentrations were observed among the samples. The lowest values were below the limit of detection (LOD) of 0.002 mg·L^{-1} for both domestic and foreign wheat flour samples, and the highest value was 0.277 mg·kg^{-1} in domestic white flour. Generally, the As concentration was lower than the permissible level set by IQS and GSO 1.0 mg·kg^{-1} (COSQC 2000; GSO 2014). This finding is within the range reported by Mahdi and Omran (2021), which was roughly 0.256 mg·kg^{-1} for wheat flour in Iraq. A study conducted to determine As in wheat plants showed that the minimum and maximum levels were recorded between 0.049 and 0.363 mg·kg^{-1} (Al-Othman et al. 2016), which is relatively similar to the results of the present study. Moreover, the obtained results showed higher As levels than in a previous study on foreign white flour samples in Sulaimani City, where no arsenic was detected (Abdul et al. 2023). However, these results were lower than those reported by Rasheed (2021), where concentrations of As in bread ranged from 0.009 to 0.019 mg·kg^{-1} (Rasheed 2021).

Cadmium (Cd). The cadmium levels in all analysed samples were below the IQS and GSO permissible limit of 0.1 mg·kg^{-1} (COSQC 2000; GSO 2014). The amount observed in this study was within the range Nejabat et al. (2017) reported in Iran, which was 0.008 to 0.031 mg·kg^{-1} in wheat flour. In contrast, this range is notably lower than that reported by Mahdi and Omran (2021), where cadmium levels in wheat flour ranged from 0.4 to 12.6 mg·kg^{-1} in Iraq. The results of this study were similar to those obtained by Abdul (2023) for foreign white flour and higher than those reported by Rasheed (2021) for bread, with concentrations of 0.034 mg·kg^{-1} and 0.011 mg·kg^{-1}, respectively (Rasheed 2021; Abdul et al. 2023). Predicting the metallic content of the crops is complicated as it depends on many factors, including variety, land of cultivation, fertilisation rate and weather conditions (Ekholm et al. 2007). The statistical analysis revealed a significant difference in cadmium content between samples F4, F10, F11, F18, F22, F23, and F26 compared to the other samples ($P < 0.05$).

Lead (Pb). The statistical analysis indicated a significant difference in lead content, with sample F0 from the others ($P < 0.05$). The highest recorded Pb concentration was 0.64 mg·kg^{-1} in F0. Conversely, all other samples exhibited Pb levels lower than the detection limit (LOD) of 0.003 mg·L^{-1} and an acceptable limit of 0.2 mg·kg^{-1} set by IQS and GSO (COSQC 2000; GSO 2014). These results are consistent with a previous study conducted in Iraq, when the levels of Pb in wheat flour samples ranged from 0.092 to 1.021 mg·kg^{-1} (Mahdi and Omran 2021). Nevertheless, this finding is roughly higher than that reported by Alhendi and Al (2018) in a study of wheat in Iraq, where the Pb concentration ranged from 0.023 to 0.192 mg·kg^{-1}.

In contrast, the findings of this study are not consistent with a study conducted by Nejabat et al. (2017) and Ghanati et al. (2019) in Iran, when they reported that Pb content ranged from 0.013 to 0.140 mg·kg^{-1} and 0.049 to 0.083 mg·kg^{-1}, respectively (Nejabat et al. 2017; Ghanati et al. 2019). Since lead is a constituent of the Earth's crust and is naturally present in soil and subsoil, industrial activity could also increase the Pb content during the process. Air pollution significantly influences lead content in grains, resulting from automobile traffic that could affect irrigation water and farm soils (Hammed and Koki 2016; Bossou et al. 2021). In a study by Wild et al. (2005), it was found that contaminated soils are the main pathway by which heavy metals, especially lead, enter the food chain, particularly bread.

Cobalt (Co). The concentration of cobalt was found to be 0.00 mg·kg^{-1}. The cobalt content in all analysed samples was below the permissible limit of 0.03 mg·kg^{-1} in foods as recommended by Codex (2016). These results are lower than those reported

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Table 1. Heavy metal concentrations in 27 wheat flour samples (mean ± SD)

Code	Origin	Type of flours	As	Cd	Pb	Co	Cr	Cu	Ni
						(mg·kg^{-1})			
F0	Iraq		0.243 ± 0.05	0.05 ± 0.043	0.64 ± 0.07	ND	0.24 ± 0.05	5.55 ± 0.08	0.21 ± 0.05
F1	Türkiye		< LOQ	0.05 ± 0.031	< LOQ	ND	ND	2.45 ± 0.05	ND
F2	Iraq		< LOQ	0.04 ± 0.030	< LOQ	ND	0.017 ± 0.00	1.87 ± 0.05	0.05 ± 0.00
F3	Iraq		0.253 ± 0.05	0.05 ± 0.041	< LOQ	ND	0.010 ± 0.00	2.13 ± 0.05	0.03 ± 0.00
F4	Germany		< LOQ	ND	< LOQ	ND	< LOQ	2.63 ± 0.05	ND
F5	Türkiye		0.234 ± 0.05	0.00 ± 0.021	< LOQ	ND	0.090 ± 0.00	1.65 ± 0.04	0.08 ± 0.01
F6	Türkiye		0.223 ± 0.05	0.04 ± 0.024	< LOQ	ND	0.021 ± 0.01	1.33 ± 0.04	0.07 ± 0.00
F7	Türkiye		0.245 ± 0.05	0.05 ± 0.031	< LOQ	ND	ND	1.93 ± 0.05	0.10 ± 0.05
F8	Türkiye	white flour	0.221 ± 0.05	0.05 ± 0.024	< LOQ	ND	0.120 ± 0.03	1.41 ± 0.05	0.00 ± 0.04
F9	Türkiye		0.227 ± 0.05	0.00 ± 0.030	< LOQ	ND	0.100 ± 0.03	1.79 ± 0.05	0.10 ± 0.05
F10	UAE		< LOQ	ND	< LOQ	ND	ND	2.10 ± 0.05	ND
F11	Kuwait		< LOQ	ND	< LOQ	ND	ND	1.90 ± 0.05	ND
F12	Russia		< LOQ	0.03 ± 0.043	< LOQ	ND	ND	1.21 ± 0.02	ND
F13	Russia		< LOQ	0.00 ± 0.025	< LOQ	ND	0.027 ± 0.00	1.42 ± 0.03	0.04 ± 0.01
F14	Russia		0.231 ± 0.05	0.00 ± 0.027	< LOQ	ND	0.021 ± 0.01	1.30 ± 0.02	0.08 ± 0.02
F15	Russia		< LOQ	0.04 ± 0.036	< LOQ	ND	0.019 ± 0.01	1.70 ± 0.05	0.03 ± 0.00
F16	Iraq		0.229 ± 0.05	0.05 ± 0.051	< LOQ	ND	0.140 ± 0.05	2.00 ± 0.05	0.14 ± 0.05
F17	Iraq	whole wheat flour	0.241 ± 0.05	0.00 ± 0.048	< LOQ	ND	0.013 ± 0.02	2.65 ± 0.05	0.04 ± 0.02
F18	Russia		0.207 ± 0.05	ND	< LOQ	ND	0.018 ± 0.01	1.33 ± 0.04	0.11 ± 0.05
F19	Türkiye		0.232 ± 0.05	0.03 ± 0.029	< LOQ	ND	0.090 ± 0.01	1.90 ± 0.05	0.06 ± 0.00
F20	India		< LOQ	0.04 ± 0.031	< LOQ	ND	ND	1.66 ± 0.05	0.05 ± 0.00
F21	Iraq	white flour	0.277 ± 0.05	0.05 ± 0.039	< LOQ	ND	0.050 ± 0.01	1.32 ± 0.05	ND
F22	France		< LOQ	ND	< LOQ	ND	ND	1.02 ± 0.00	ND
F23	Italy		< LOQ	ND	< LOQ	ND	ND	1.00 ± 0.00	0.07 ± 0.00
F24	Türkiye		< LOQ	0.05 ± 0.039	< LOQ	ND	0.013 ± 0.01	2.10 ± 0.05	0.10 ± 0.05
F25	Iran	whole wheat flour	< LOQ	0.00 ± 0.035	< LOQ	ND	ND	2.73 ± 0.05	ND
F26	Türkiye	white flour	0.221 ± 0.05	ND	< LOQ	ND	0.023 ± 0.02	1.64 ± 0.05	0.04 ± 0.00
Permissible limits (mg·kg^{-1})			1.0[a]	0.1[a]	0.2[a]	0.03[b]	1.0[b]	10.0[c]	0.1[b]

[a] Permissible limits (mg·kg^{-1}) according to (GSO 2014 and IQS 37/2010 standards; [b] permissible limits (mg·kg^{-1}) according to (FAO/WHO); [c] permissible limits (mg·kg^{-1}) according to Codex 1993; LOQ – limit of quantification; ND – not detected; As – arsenic; Cd – cadmium; Pb – lead; Co – Cobalt; Cr – chromium; Cu – copper; Ni – nickel

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Table 2. Summary of heavy metal contents found in this study and a comparison with other studies focused on wheat flour or bread

No.	References	As	Cd	Pb	Co	Cr	Cu	Ni	Country
		(mg·kg^{-1})							
1	in this study	< LOQ – 0.253	ND–0.043	< LOQ – 0.640	ND	< LOQ – 0.240	1.0–5.550	ND–0.210	Iraq
2	Abdul et al. 2023	ND	0.034	0.405	ND	1.157	1.908	0.208	Iraq
3	Rasheed 2021, in bread	0.009–0.019	0.003–0.011	0.008–0.110	0.008–0.012	ND	ND	0.160–0.243	Iraq
4	Ölmez et al. 2023	ND	ND	1.574–2.201	ND	ND	2.150–3.180	ND	Türkiye
5	Ghanati et al. 2019	7.200–12.280	0.477–0.821	0.049–0.083	0.060–0.063	0.152–0.270	0.026–0.036	> 0.010	Iran
6	Nejabat et al. 2017	ND	0.008–0.031	0.013–0.140	ND	ND	0.480–6.200	ND	Iran
7	Mustatea et al. 2020	ND	0.008–0.024	0.002–0.137	ND	ND	ND	ND	Romania
8	Hammed and Koki 2016	ND	< 0.005	0.100–0.250	ND	ND	ND	ND	Malaysia
9	Doe et al. 2013	ND	0.250–0.600	0.220–0.340	ND	ND	ND	ND	Ghana
10	Tejera et al. 2013	ND	0.023–0.027	0.037–0.056	ND	ND	2.800	ND	Spain

LOQ – limit of quantification; ND – not detected; As – arsenic; Cd – cadmium; Pb – lead; Co – Cobalt; Cr – chromium; Cu copper; Ni – nickel

by Rasheed (2021) in bread, 0.008 to 0.012 mg·kg^{-1} in Sulaimani City. In addition, these results are also lower than the results from the finding of a study from Iran, which obtained Co concentration between 0.015 to 0.1 mg·kg^{-1} for bread. In addition, results were lower than Co concentrations in Iranian wheat flour, 0.06 to 0.079 mg·kg^{-1} reported by Ghanati et al. (2019).

Chromium (Cr). This study revealed significant variations in chromium concentrations among the samples. The lowest values were observed in imported white flour samples, with a concentration of 0.00 mg·kg^{-1}, while the highest concentration was found in domestic samples at 0.24 mg·kg^{-1}. Notably, all analysed samples exhibited chromium concentrations below the permissible limit of 1.0 mg·kg^{-1} set by Codex (2016). These results contrast with a study conducted in Sulaimani City, which reported Cr concentrations of 1.15 mg·kg^{-1} in Turkish white flour samples (Abdul et al. 2023). However, these results are lower than the Cr concentration of 0.152 to 0.27 mg·kg^{-1} for wheat flour reported by Ghanati et al. (Ghanati et al. 2019).

Copper (Cu). In this study the copper concentration in white flour was found to range from 1.0 to 5.55 mg·kg^{-1}, which is consistent with a study conducted in Sulaimani City that reported a copper concentration of 1.90 mg·kg^{-1} in a Turkish white flour sample (Abdul et al. 2023). In contrast, a study conducted in Baghdad, Iraq, found that Cu concentrations ranged from 0.22 to 0.63 mg·kg^{-1} (Jawad and Allafaji 2012). The result indicates that the Cu concentration in all studied samples was lower than the permissible level of 10 mg·kg^{-1} set by Codex (2007).

Nickel (Ni). The nickel concentration ranged from the lowest value of 0.00 mg·kg^{-1} in imported white flour to the highest value of 0.21 mg·kg^{-1} in domestic white flour, which was more than the permissible level of 0.1 mg·kg^{-1} set by Codex (2016). Previous studies on cereal crops found that nickel tends to accumulate in soils, possibly due to external sources such as fertilisers and pesticides (Martínez-Cortijo and Ruiz-Canales 2018). Compared to the results of this study, Abdul et al. reported that the concentration of Ni was 0.208 mg·kg^{-1} in white flour samples collected from one sampling site in Sulaimani City (Abdul et al. 2023). Although nickel can be flushed from the human system after consumption, food contamination with nickel should still be considered. Mixing Ni with other harmful active compounds in the human system can cause severe toxicity (Abd Rashid et al. 2018).

Table 1 shows the opposite results regarding the Pb content for flour samples imported from Türkiye,

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which is less than the permissible level of 0.2 mg·kg^{-1}. In contrast, Olmez's study shows that the lead level in wheat flour samples in Türkiye was higher than the permissible level, as shown in Table 2. Ölmez et al. (2023) indicated that air pollution is a source of Pb contamination due to rapid industrialisation in the study area Çorum. The metallic content is significantly different and will rely on the type of land where the crop has been cultivated, fertilisation used, and the weather (Ekholm et al. 2007).

CONCLUSION

The findings of this study reveal the presence of As, Cd, Pb, Co, Cr, Cu, and Ni in the most available wheat flour samples in Sulaimani City. Notably, heavy metal concentrations like Ni and Pb exceeded the permissible levels established by IQS, GSO, Codex, and FAO/WHO. It is a significant achievement since wheat is an essential cereal and staple food in Kurdistan, Iraq. This study revealed that domestic wheat flour was more contaminated than imported flour. While it is impossible to address all contributing factors, the study results align with elevated air pollution parameters and rapid industrialisation observed in Sulaimani City. These findings suggest a critical monitoring system, particularly in addressing potential sources of heavy metals, such as air pollution, fertilisers, and pesticides. Overall, based on the results of this study, wheat flour can be considered safe for local consumption in terms of heavy metal content.

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