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Rocz Panstw Zakl Hig 2020;71(4):371-376

https://doi.org/10.32394/rpzh.2020.0142

ORIGINAL ARTICLE

ASSESSMENT OF EXPOSURE TO NICKEL INTAKE WITH SELECTED CEREAL GRAINS AND CEREAL-BASED PRODUCTS

Monika Mania¹, Małgorzata Rebeniak¹, Oksana Orshulyak¹, Jacek Postupolski¹

¹National Institute of Public Health - National Institute of Hygiene, Department of Food Safety, 24 Chocimska str., 00-791 Warsaw, Poland

ABSTRACT

Background. Cereal grains and their products are among the basic components of the diet all over the world. Their consumption varies depending on the dietary habits in each country. Apart from ingredients valuable from a health point of view, these products can also be a source of contaminants. The European Food Safety Authority (EFSA) included these foodstuffs in the group of main contributors to the dietary exposure to nickel by different groups of the population.

Objective. The aim of the studies was to determine the nickel content in cereal grains and selected cereal products commercially available in Poland and to assess the exposure of consumers to nickel intake with these foodstuffs.

Material and methods. A total of 56 samples of cereal grain and cereal products from trade were tested. Nickel content was determined after microwave mineralization of the samples by atomic absorption spectrometry with a graphite furnace atomization (GFAAS). The estimated exposure for the different groups of the population was assessed and compared with the Tolerable Daily Intake (TDI) value established by the European Food Safety Authority based on an updated risk assessment at 13 μ g/kg body weight (b.w.) per day.

Results. The mean (MB) and 95th percentile (MB) nickel content in investigated samples of cereal grains and their products were 0.66 mg/kg and 1.93 mg/kg. The mean content of Ni in the analyzed samples of grains was 1.16 mg/kg. Obtained results in this group of products ranged from 0.10 mg/kg for rye to 4.80 mg/kg for millet. In the group of grain products, the mean (MB) concentration of nickel was 0.61 mg/kg (95th percentile (MB) 1.84 mg/kg). The highest nickel level was determined in the samples of bran, groats, and flakes compared to other grain-based products. The highest content of Ni in the group of cereal products was found in the samples of roasted buckwheat 1.81 mg/kg and oat flakes 2.53 mg/kg. Significantly lower nickel content was observed in barley groats as well as for pasta and flour.

The estimated average exposure of adults and children to nickel intake with grains and grain-based products ranged from 1.1% to 13.4% of TDI and it does not pose a health risk for consumers.

Conclusions. Based on the obtained results, it was found that the nickel content in the tested samples of cereals and their products does not pose a health risk, even though the contamination of individual samples was significant.

Key words: nickel, cereals grains, cereal-based products, intake, exposure assessment, Tolerable Daily Intake, TDI

STRESZCZENIE

Wprowadzenie. Ziarna zbóż i produkty na ich bazie są zaliczane do podstawowych składników diety na całym świecie. Ich spożycie jest zróżnicowane w zależności od nawyków żywieniowych panujących w poszczególnych krajach. Poza cennymi ze zdrowotnego punktu widzenia składnikami produkty te mogą również stanowić źródło pobrania zanieczyszczeń. Europejski Urząd ds. Bezpieczeństwa Żywności zaliczył te środki spożywcze do grupy głównych składników mogących wnosić istotny udział do pobrania niklu z dietą przez różne grupy populacji.

Cel badań. Celem badań było oznaczenie zawartości niklu w wybranych ziarnach zbóż i produktów zbożowych dostępnych w obrocie handlowym w Polsce oraz ocena narażenia konsumentów na pobranie niklu z tymi grupami środków spożywczych.

Material i metody. Zbadano łącznie 56 próbek zbóż oraz produktów zbożowych pochodzących z obrotu handlowego. Zawartość niklu oznaczono po mineralizacji mikrofalowej próbek metodą absorpcyjnej spektrometrii atomowej z wykorzystaniem kuwety grafitowej (GFAAS). Oszacowane narażenie w odniesieniu do różnych grup populacji porównano z wartością tolerowanego dziennego pobrania – TDI (Tolerable Daily Intake) ustaloną przez Europejski Urząd ds. Bezpieczeństwa Żywności na podstawie uaktualnionej oceny ryzyka na poziomie 13 μg/kg m.c./dzień.

Wyniki. Średnia zawartość niklu (MB) w badanych próbkach ziaren zbóż i ich produktów wyniosła: 0,66 mg/ kg (95-percentyl MB 1,93 mg/kg). Średnia zawartość Ni w analizowanych próbkach ziaren zbóż wyniosła 1,16 mg/ kg. Uzyskane wyniki w tej grupie produktów wahały się od 0,10 mg/kg dla żyta do 4,80 mg/kg dla prosa. W grupie produktów zbożowych średnie (MB) stężenie niklu wyniosło 0,61 mg/kg (95 percentyl (MB), 1,84 mg/ kg). Najwyższą

Corresponding author: Monika Mania, National Institute of Public Health-National Institute of Hygiene, Department of Food Safety, 24 Chocimska str., 00-791 Warsaw, Poland, phone +48 22 54 21 369, fax +48 22 54 21 392, e-mail: mmania@pzh.gov.pl

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zawartość Ni stwierdzono w próbkach: otrąb, kasz i płatków w porównaniu z innymi produktami zbożowymi. W grupie przetworów zbożowych najwyższą zawartość Ni stwierdzono w próbce gryki prażonej 1,81 mg/kg i płatków owsianych 2,53 mg/kg. Znacząco niższą zawartość Ni otrzymano w kaszach jęczmiennych oraz w makaronie i mące.

Oszacowane średnie narażenie dorosłych i dzieci na pobranie Ni z ziarnami zbóż i produktami zbożowymi wyniosło od 1.1% do 13,4% TDI i nie stanowi ono zagrożenia dla zdrowia konsumentów.

Wnioski. Na podstawie uzyskanych wyników stwierdzono, że zawartość niklu w zbadanych próbkach zbóż i produktów zbożowych nie stanowi zagrożenia dla zdrowia, pomimo iż zanieczyszczenie pojedynczych próbek było istotne.

Słowa kluczowe: nikiel, zboża, produkty zbożowe, pobranie, ocena narażenia, TDI, Tolerowane Dzienne Pobranie

INTRODUCTION

Cereals and cereal products are important components of the daily diet [5]. These products are the source of valuable minerals, vitamins mainly from group B, fiber, protein, and carbohydrates [4, 16, 34].

The average monthly consumption of cereal products and bread in Poland is high and amounts to 5.36 kg per person. We eat bread the most, on average 2.89 kg per person per month, compared to other cereal products such as flour 0.59 kg or pasta 0.40 kg [15]. These foodstuffs can be also a source of contaminants such as heavy metals (lead, cadmium, and nickel), pesticides, and herbicides [1, 3, 5, 31]. The presence of nickel is connected mainly with environmental contamination or can be connected with the migration of this metal during the production process from equipment used in the food industry or during food preparation [22, 23, 24, 29, 30, 33]. This element is also a natural component of the Earth's crust easily bioaccumulated [29].

The toxicity of nickel and its compounds has been confirmed in numerous scientific studies, including the International Agency for Research on Cancer (IARC), which has classified nickel compounds in Group I as carcinogenic to humans, and metallic nickel and nickel alloys in Group 2B as potentially carcinogenic to humans. Intake of Ni with a diet is associated rather with non-carcinogenic effects [22, 23, 24]. However, the mechanism of the toxic effect of nickel on the human body still requires understanding.

In 2015, European Food Safety Authority (EFSA) based on risk assessment and available data published scientific opinion on the risk to public health related to the presence of nickel in food and drinking water. EFSA indicated that grain and grain-based products are the main contributors to the dietary exposure to nickel for the general population, same as non-alcoholic beverages (except milk-based beverages), sugar and confectionery, legumes, nuts and oilseeds, and vegetables and vegetable products (including fungi), milk and dairy products [28, 29]. Based on the risk assessment a tolerable daily intake (TDI) of 2.8 μ g/kg b.w. was established.

In its recent opinion from 2020 CONTAM Panel of EFSA established a tolerable daily intake (TDI) at 13

 μ g Ni/kg body weight (b.w.) per day which is 4.6 times higher than in the previous risk assessment [30].

The newly established TDI value was influenced, inter alia, by an updated version of the guidance on the use of the reference dose method (BMD) in the risk assessment [10]. According to newly collected occurrence data, EFSA confirmed that grains and grain-based products mainly contributing to the mean LB (lower bound) chronic dietary exposure to nickel across all age classes with contributions reaching up to 49% in infants and toddlers Ni/kg body weight (b.w.) per day [30].

Currently, studies on the determination of nickel contents in food are conducted in the Member States to a slightly greater extent than a few years ago. This is due to the recommendation developed in 2016 by the European Commission on monitoring the level of this contamination in various groups of foodstuffs to collect more representative data covering the entire territory of the European Union, which would allow taking appropriate measures in the area of risk management in the future [6].

Research on the absorption of nickel in the human body are still scarce, which makes it impossible to reliably assess the exposure to this element in the diet. The available information in this regard indicates a large spread in absorption (1%-30%), depending on many factors such as chemical form and thus, the solubility of the nickel compound, which additionally increases the uncertainty of such assessments [13, 22, 23, 24, 29, 30].

As a critical effect for the risk characterization of acute oral exposure a systematic contact dermatitis was identified by EFSA and the lowest observed adverse effect level of 4.3 μ g Ni/kg bw was selected as the reference point. The margin of exposure (MOE) approach was also applied. MOE of 30 or higher was considered as being indicative of a low health concern.

Estimated mean chronic dietary exposure was below or at the level of the TDI. Whilst the 95th percentile chronic dietary exposure was below the TDI in adolescents and in all adult age groups, but generally exceeded the TDI in toddlers and in other children, as well as in infants in some surveys and raise a health concern in these young age groups. Calculated MOE values for mean acute dietary exposure to 95th percentile exposures raise health concerns in nickel allergic individuals [30].

Currently applicable legislation of the European Union does not take into account the requirements for nickel contamination of foodstuffs, apart from the requirements for food additives [7]. For the time being, such requirements apply at national level to drinking water, natural mineral, spring and table waters [26, 27].

Accordingly, there is a need to protect the most vulnerable groups of the population by implementing appropriate risk management measures.

MATERIALS AND METHODS

Samples

The samples were taken from the Polish market in the years 2019-2020. They consisted of 56 samples of grain and grain-based products. The profile of the grain products under analysis was: 11 samples of pasta; 13 samples of flours; 12 samples of groats; 10 samples of flakes and 5 samples of bran. In on the group of grains were: wheat, rye, barley, and millet.

Concentration of nickel was measured by using in-house fully validated and accredited method according to standard PN-EN ISO/IEC 17025:2018. The validation was performed based on the analysis of the certified reference material - SRM 1515 (Apple leaves NIST) with certified mass fraction for nickel $0.936 \text{ mg/kg} \pm 0.094 \text{ mg/kg}.$

The obtained validation paramteres of the method were as follows: LOD: 0.95 μ g/L; LOQ: 1.25 μ g/L; repeatability - RSDr 7%; correctness (relative error): 3.4%, recovery: 97%; range: from LOQ to 25.0 μ g/l, uncertainty 24%.

Equipment

VARIAN SpectrAA 880Z atomic absorption spectrometer equipped with graphite furnace and Zeeman background correction was used. For graphite furnace measurements, argon was used as an inert gas. Pyrolytic-coated graphite tubes (Agilent) without platform were used. The spectrometer settings were as follows: wavelength 232.0 nm, slit width 0.2 nm, lamp current 4.0 mA, argon (flow) 3.0 l/min

The atomic absorption signal was measured as a peak surface mode against an analytical curve. Milestone MLS 1200 (Italy) microwave closed system at the stage of sample preparation was used.

Reagents

Nitric acid (HNO3)65% (Merck), hydrogen peroxide 30% (Merck), palladium matrix modifier for graphite furnace (Pd (NO3)2/HNO3 (Merck) hydrochloric acid 35%-38% (Chempur), L (+) - ascorbic acid (Poch), deionized water, argon. The element standard solution used for calibration was prepared by diluting a stock solution of 1g/L (GUM). All reagents used were on an appropriately high degree of purity.

Elements of control and verification of the method

The accuracy of the method was verified by the analysis of certified reference material SRM-1515 (Apple leaves NIST). As part of the external validation of results, the laboratory regularly participates in proficiency tests organized by reputable suppliers such as the UK Food Science Laboratory (FAPAS) and the EU reference laboratory for metals and nitrogenous compounds (EURL-MN), Denamrk.

Samples digestion

Approximately 0.5 g of sample was and transferred into clean teflon vessel then 5 mL of concentrated HNO_3 and 1 mL of hydrogen peroxide were added. The digestion was lead by using a microwave in the closed mineralization system. To prevent clumping the contents of the reaction vessel were thoroughly mixed. After finished of the spontaneous reaction, the reaction vessels were quickly closed. The mineralization was carried out following the guidelines of the mineralizer producer.

Calibration curve

Scope of calibration curve: from 0 μ g/L to 25 μ g/L. The standard curve was prepared from one calibration solution thanks to the automatic dilution of the reagents.

Statistical evaluation of data

Statistical assessment of results was performed according to the substitution method used by EFSA for the treatment of left-censored data – LC (below limits of quantification (LOQ was 0.06 mg/kg). For results reported to be below the LOQ, the value equal to the LOQ (upper bound – UB), zero (lower bound – LB), or half the LOQ (medium or middle bound – MB) were used [31].

RESULTS AND DISCUSSION

Left-censored data (below limit of quantification), represented 27% of the analytical results for all investigated samples of grain and their products. The results are presented in Table 1. The mean (MB) and 95th percentile (MB) nickel concentration in investigated samples of grain and their products were 0.66 mg/kg and 1.93 mg/kg. The average concentration of the nickel in the analyzed samples of grains was 1.16 mg/kg (MB). Nickel contents for this group of products ranged from 0.10 mg/kg for rye to 4.80 mg/kg for millet. Similar results were obtained in the surveys conducted by other authors [16, 21].

Cereal grains and grain-based products	Number of samples	Nickel content		
		Mean (MB)	P95 (MB)	SD (MB)
Cereal grains (millet, rye, wheat, barley)	5	1.16	3.91	2.04
Pasta	11	0.26	1.04	0.52
Flour	13	0.35	1.35	0.58
Groats	12	0.63	1.72	0.67
Flakes	10	0.93	1.97	0.66
Bran	5	1.34	1.76	0.36
All grain-based products	51	0.61	1.84	0.66
Cereal grains and grain-based products	56	0.66	1.93	0.85

Table 1. Nickel content in tested samples of cereal grains and grain-based products, mg/kg

MB – middle bound, P95 – 95th percentile, SD – standard deviation

In the group of grain products, the mean (MB) concentration of nickel was 0.61 mg/kg (95th percentile (MB) 1.84 mg/kg). The highest nickel level was determined in the samples of bran, groats, and flakes compared to other grain-based products. In the case of bran mean, MB was 1.34 mg/kg, for flakes was 0.93 mg/kg whilst for groats 0.63 mg/kg. Another survey indicates a comparable concentration of Ni, for groats and flakes, mean was from 0.07 mg/kg to 2.68 mg/kg, and from 0.25 mg/kg to 1.16 mg/kg respectively.

The highest content of Ni in the group of cereal products was found in the samples of roasted buck wheat 1.81 mg/kg and oat flakes 2.53 mg/kg. The obtained results are comparable with the results from other EU countries, indicating higher nickel contamination of buck wheat and oat and products from these grains as compared to other products, mean were 2.0 mg/kg and 1.8 mg/kg [29]. A study conducted in the United States and Australia also confirmed higher nickel contamination of oats and its products, obtained results were in the range: 0.36 mg/kg - 2.10 mg/kg and 0.35 mg/kg - 0.41 mg/kg, respectively [11, 36].

The survey conducted in Sweden indicates lower contamination of cereal-based products than in our studies, the mean was 0.14 mg/kg [20].

Previous studies by other authors concerning cereal products indicate even higher nickel contamination of buckwheat groats, reaching up to 2.68 mg/kg and significantly lower values in the case of oatmeal – mean 1.16 mg/kg [18]. According to new EFSA occurrence data, the mean (LB-UB) concentration of nickel in grains and grain-based products was is in the range: 0.31 mg/kg - 0.33 mg/kg (95th percentile 1.25 mg/kg - 1.25 mg/kg) [30]. Average contamination of investigated samples of pasta and flour with nickel was lower than other investigated samples of grain-based products, mean MB was 0.26 mg/kg (95th percentile 1.04 mg/kg), and 0.35 mg/kg (95th percentile 1.35 mg/kg) respectively. In the case of wholemeal pasta, higher levels of nickel were observed than in the case

of regular pasta made from wheat flour. The obtained results for both groups were in the range: from 0.20 mg/kg to 1.79 mg/kg and 0.03 mg/kg to 0.08 mg/kg for pasta made from wheat flour. The survey conducted in the UK indicates comparable contamination of pasta and wheat flour, mean was about 0.03 mg/kg and 0.04 mg/kg, accordingly [12]. Similar lower contents of Ni in pasta were observed in France, reported mean values were as follows: 0.02 mg/kg and 0.05 mg/kg [13, 19]. Other authors reported results for pasta in a wide range, from 0.05 mg/kg to 0.12 mg/kg [2].

A similar connection as for pasta was observed in the case of whole grain flour and wheat flour. For the first group concentration of Ni was much higher and ranged between 0.23 mg/kg and 2.12 mg/kg. In the case of wheat flour obtained results were below LOQ value whilst contamination of spelt flour was significantly higher than wheat flour and ranged from 0.14 mg/kg to 0.18 mg/kg.

The survey conducted in Spain concerning contamination of wheat flour indicates slightly higher values form 0.08 mg/kg to 0.09 mg/kg [34].

To assess the dietary exposure to nickel from commercially available grain and grain products mean and high contamination levels (P95), middle bound (MB), were taken into account. The data of the Central Statistical Office [15], and data of WHO, GEMS/ Food Consumption Cluster Diets [35] on consumption of cereals were used to assess consumption. The intake of nickel from these foodstuffs was assessed and compared to the Tolerable Daily Intake (TDI) at 13 μ g/kg body weight per day, recently established by EFSA.

Taking into account the data of WHO, GEMS/ Food Consumption Cluster Diets on the consumption of cereal grains only (excluding rice) the assessed mean (MB) intake of nickel by adults from cereal grains would be 13.4% TDI; and do not raise a health concern. Intake of nickel by adults and children based on average consumption of cereal products [14] (excluding bread, rice and bakery products) and taking into account the maximum absorption in the gastrointestinal tract would be 1.1 % of the TDI recently determined by EFSA for adults and 3.9% TDI for children. At the 95-percentile level of contamination, it would be 3.4% and 11.8% of this value. Studies conducted in Sweden indicated slightly higher exposure associated with the intake of Ni in cereal products by adults in comparison to our results. Mean MB intake of nickel with pasta by adults and children would be low, below 0.5% TDI and 1.3% TDI, respectively which also does not pose a health threat.

Assessed exposure was much lower than exposure to Ni intake with pasta calculated by Italian and French authors for both groups of the population [8, 19].

CONCLUSIONS

The content of nickel in the tested samples of selected grain and grain products does not pose a health concern

The estimated exposure do not indicate excessive nickel intake with cereal grains and their products and is unlikely to be of toxicological concern.

The obtained results may contribute to legislative work allowing for proper risk management in this area.

Acknowledgements

The study was performed under the scientific projects financed by the National Institute of Public Health National Institute of Hygiene, Warsaw, Poland (BŻ-1/2020(FB-1/2020)).

Conflict of interest

The authors declare no conflict of interest.

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Received: 17.11.2020 Accepted: 04.12.2020

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