

MIGRATION STUDIES OF NICKEL AND CHROMIUM FROM CERAMIC AND GLASS TABLEWARE INTO FOOD SIMULANTS

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ABSTRACT

Background. In addition to the release of lead and cadmium from ceramic and glass vessels, (acceptable limits being set by the EU 84/500/EC Directive), other harmful metals can migrate, such as nickel and chromium. Permissible migration limits for these latter metals however have not yet been set in the EU legislation. Both the toxic properties of nickel and chromium and the measures taken by the European Commission Working Group on Food Contact Materials for verifying permissible migration limits for lead, cadmium and other metals from ceramics have acted as drivers for studies on nickel and chromium release from ceramic and glass tableware.

Objective. To investigate the migration of nickel and chromium into food simulants from ceramic and glassware, available on the Polish market, which are intended for coming into contact with food. Potential consumer exposure can thereby be estimated from the release of these elements into food.

Materials and Methods. Tableware consisted of ceramics and glass vessels generally available on the domestic market, with inner surfaces being mainly coloured and with rim decorations. Migration of nickel and chromium studied from the ceramics was carried out in 4% acetic acid (24 ± 0.5 hrs at $22 \pm 2^\circ\text{C}$), whilst that from glassware in 4% acetic acid (24 ± 0.5 hrs at $22 \pm 2^\circ\text{C}$) and 0.5% citric acid (2 ± 0.1 hrs at $70 \pm 2^\circ\text{C}$). The concentrations of metals which had migrated into the test solutions were measured by using flame atomic absorption spectrometry (FAAS). This analytical procedure had been previously validated by measuring nickel and chromium released into food simulants from ceramic and glass tableware where working ranges, detection limits, quantification limits, repeatability, accuracy, mean recovery and uncertainty were established.

Results. Migration of nickel and chromium was measured from 172 ceramic and 52 and glass vessels samples, with all results being below the limits of quantification (LOQ = 0.02 mg/L), excepting one instance where a 0.04 mg/L concentration of nickel was found. The validated methods for measuring chromium achieved the following parameters; 0.02 to 0.80 mg/L operating range, 0.01 mg/L detection limit, 0.02 mg/L limit of quantification, 6% repeatability, 2.8% accuracy, 102% average recovery and 11% uncertainty. For the nickel method the corresponding parameters were 0.02 to 0.80 mg/L working range, 0.02 mg/L limit of quantification, 0.01 mg/L detection limit, 5% repeatability, 6.5% accuracy, 101% average recovery and 12% uncertainty.

Conclusions. The tested ceramics and glassware did not pose a threat to human health regarding migration of nickel and chromium, and thus any potential exposure to these metals released from these products into food will be small. However, due to the toxicity of these metals, the migration of nickel and chromium is still required for articles coming into contact with food, which includes metalware.

Key words: *ceramic tableware, ceramics, glassware, food contact articles, nickel, chromium leaching, migration*

STRESZCZENIE

Wprowadzenie. Z wyrobów ceramicznych i szklanych, obok migracji ołowiu i kadmu, dla których w UE określono dopuszczalne limity (Dyrektywa 84/500/EC) może zachodzić migracja także innych szkodliwych dla zdrowia metali, w tym niklu i chromu. Dla chromu i niklu nie ustanowiono jeszcze w UE dopuszczalnych limitów migracji. Właściwości toksyczne niklu i chromu oraz podjęte przez European Commission Working Group on Food Contact Materials prace mające na celu weryfikację dopuszczalnych limitów migracji ołowiu i kadmu z wyrobów ceramicznych oraz wprowadzenie limitowania dla innych pierwiastków, skłoniły do podjęcia badań uwalniania niklu i chromu z ceramicznych i szklanych naczyń stołowych.

Cel. Zbadanie migracji do płynów modelowych niklu i chromu z naczyń ceramicznych i szklanych przeznaczonych do kontaktu z żywnością, dostępnych na rynku w Polsce, w celu oszacowania wielkości potencjalnego narażenia konsumentów na te metale uwalniane z takich wyrobów do żywności.

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Materiał i metody. Materiał do badań stanowiły stołowe naczynia ceramiczne i naczynia szklane, głównie z barwioną powierzchnią wewnętrzną i zdobionym obrzeżem, pochodzące z obrotu handlowego. Migrację niklu i chromu z badanych wyrobów ceramicznych przeprowadzono do 4% kwasu octowego ($24 \pm 0,5$ h w temperaturze 22 ± 2 °C), natomiast migrację z naczyń szklanych do 4% kwasu octowego ($24 \pm 0,5$ h w temperaturze 22 ± 2 °C) i 0,5% kwasu cytrynowego ($2 \pm 0,1$ h w temperaturze 70 ± 2 °C). Zawartość badanych metali w płynach modelowych uzyskanych po migracji, oznaczano metodą płomieniowej atomowej spektrometrii absorpcyjnej (FAAS). Badania poprzedzono walidacją metody oznaczania niklu i chromu uwalnianych do płynów modelowych z ceramicznych i szklanych naczyń stołowych. Wyznaczono następujące parametry metody oznaczania chromu i niklu: zakres roboczy, granicę wykrywalności, granicę oznaczalności, powtarzalność, poprawność, średni odzysk oraz niepewność.

Wyniki. Migracja niklu i chromu z 172 badanych naczyń ceramicznych i 52 naczyń szklanych, w zastosowanych warunkach badania migracji, była poniżej granicy oznaczalności metody analitycznej (LOQ) wynoszącej 0,02 mg/L, z wyjątkiem jednego naczynia, z którego migracja niklu była wyższa i wynosiła 0,04 mg/L. Zwalidowaną metodę oznaczania chromu charakteryzowały następujące parametry: zakres roboczy - od 0,02 do 0,80 mg/L, granica wykrywalności - 0,01 mg/L, granica oznaczalności - 0,02 mg/L, powtarzalność - 6%, poprawność - 2,8%, średni odzysk - 102% i niepewność - 11%. Parametry metody oznaczania niklu: zakres roboczy - od 0,02 do 0,80 mg/L, granica oznaczalności - 0,02 mg/L, granica wykrywalności - 0,01 mg/L, powtarzalność - 5%, poprawność - 6,5%, średni odzysk - 101% i niepewność - 12%.

Wnioski. Zbadane wyroby ceramiczne i szklane nie stanowią zagrożenia dla zdrowia człowieka w zakresie migracji niklu i chromu, a potencjalne narażenie na te metale uwalniane z tych wyrobów do żywności będzie niewielkie. Jednak, ze względu na toksyczność tych metali wskazane jest kontynuowanie badań uwalniania niklu i chromu z wyrobów do kontaktu z żywnością, z uwzględnieniem naczyń metalowych.

Słowa kluczowe: naczynia ceramiczne, naczynia szklane, wyroby do kontaktu z żywnością, migracja niklu, migracja chromu

INTRODUCTION

Nickel and chromium compounds have long been used by manufacturers as pigments for visually enhancing the aesthetic appeal of ceramic and glassware products intended to make food contact. Release of such elements is determined on how the products were manufactured, the temperature and duration that contact is made with the food and what type the foodstuff actually is. By adding nickel compounds to these products, the colour and shade of pigments are deepened which is superior to that achieved by other pigments. This is important in manufacture [6]. For many years, nickel oxide (NiO) has been commonly used in traditional ceramic pigments, despite concerns about its potentially carcinogenicity [15, 32].

The possible leaching of metals from the surface of ceramic and glassware is affected by many factors; improperly formulated composition of glaze, inappropriate production techniques, defective decoration processes and pigmentation and, in particular, insufficiently high firing temperatures [1]. In order to ensure the right quality of products intended to come into contact with food, the principles of good manufacturing practice (GMP) should be adopted [5].

Literature reports suggest there is a greater likelihood migration of elements from ceramics or glassware manufactured by using traditional techniques by individual craftsmen than those coming from industrial manufacture. This is dependent on the sources and how the raw materials were obtained, as well as inadequate temperature control during firing. Because of high migration levels of metals, such products can pose a serious threat to the health of consumers, especially when adequate controls in the pre-market trading phase are absent [3, 19, 21, 22, 32].

The diet constitutes the primary source of exposure to nickel and chromium, excluding those occupationally exposed persons. Concentrations of nickel and chromium compounds are present at low levels in foodstuffs, however the release of these elements from kitchen and table utensils can significantly contribute to increasing dietary intakes of these metals with food [17, 18].

According to the International Agency for Research on Cancer (IARC), nickel compounds are human carcinogens (Group 1), whilst metallic nickel is a possible human carcinogen (Group 2B) [12, 32]. The estimated chronic average exposure to nickel for the general population ranges 2 µg/kg bw/day (for the elderly) to 13 µg/kg bw/day (for small children), which is close to the tolerable daily intake (TDI) of 2.8 µg/kg bw/day and it far exceeds that for the very youngest groups of the population [12].

Despite the IARC classification, the Panel on Contaminants in the Food Chain (CONTAM Panel) of the European Food Safety Authority (EFSA) concluded that nickel inducing cancer through the diet is unlikely in humans [12]. Other health effects of dietary nickel intake concern influence on the gastrointestinal tract, haematological, neurological and immune system. Nickel intake through inhalation or dermal contact also has an allergenic effect [31].

The Maximum Permissible Level of nickel in water intended for human consumption is 20 µg/L, as defined in the national Regulations of the Minister of Health [26, 27] implementing Directive 98/83/EC [8] and Directive 2003/40/EC [4]; this being lower than the level (70 µg/L) established in 1997 by the WHO [12].

Besides nickel, chromium compounds are also used in manufacturing ceramic and glass products. Metallic chromium and chromium compounds (III) are less

harmful than chromium (VI). Chromium (VI) compounds are mutagenic, teratogenic and carcinogenic increases the risk of lung cancer as well as nose and paranasal sinus cancer (Group I according to IARC). Chromium (III) compounds naturally occurs in food, whilst in drinking water mainly components of chromium (VI) are found arising from human activities [11].

The maximum concentration of total chromium in water intended for human consumption is 50 µg/L as defined in the national Regulation of the Ministry of Health [27]. Next to the migration of lead and cadmium from ceramics and glassware, for which the EU has set acceptable limits (Directive 84/500/EC), migration of other harmful metals, including nickel and chromium, can occur.

EU legislation has not yet set the limits of nickel and chromium in food nor given permissible migration limits of these metals from kitchen utensils that make contact with food. The latest European Commission Working Group on Food Contact Materials has begun work on verifying the permissible migration limits of lead and cadmium from ceramic products, that also includes introducing limits on other metals such as nickel and chromium. The Committee of Experts on Packaging Materials for Food and Pharmaceutical Products (P-SC-EMB) recommended a permissible migration limit of 0.14 and 0.250 mg/kg respectively for nickel and chromium released from metal and metal alloys used in the manufacture of articles coming into contact with food [13].

In the literature there a limited number of publications on nickel and chromium migration from ceramics into food simulants. In Poland, only studies on lead and cadmium migration from ceramic and glass have been done [25], but not on the release of nickel and chromium from such articles. However, given the toxic properties of nickel and chromium and the actions undertaken by the European Commission Working Group on Food Contact Materials, it was considered expedient to study nickel and chromium migration from ceramics and glassware into food simulants in order to estimate the potential exposure of consumers to these metals released from these products.

MATERIALS AND METHODS

Test samples

These consisted of ceramic tableware and glassware available on the retail market and manufactured on an industrial scale in China, with the inside surfaces being coloured and a decorated rim. There were 172 ceramic vessels (84 dishes deep and 88 dishes flat) and 52 glassware with decorated rims.

Method validation

Prior to the actual migration study, the analytical method for measuring nickel and chromium in food

simulants was first validated. For method validation white undecorated test samples of ceramic dishes and glassware were used. After migration food simulants were spiked with nickel and chromium at concentrations of 0.02 mg/L and 0.80 mg/L. There were 2 stages in the validation; the release of metals from products into test solutions determination of the nickel and chromium which had migrated into the model solutions by flame atomic absorption spectrometry (FAAS).

Reagents

Consisted of acetic acid (99.5% - 99.9%, Avantor), citric acid (Merck), a nickel standard solution 1g/L and a chromium standard solution 1g/L (GUM, Poland) and deionised water. All were of the required high purity.

Apparatus and instrumentation

These were an ASA Spectr AA 220 spectrometer, Varian, hotplate (Ceran 500, Harry Geestigkeit GmbH, Germany) and class A laboratory glassware.

Migration test conditions

Migration of nickel and chromium from ceramic vessels were into a food simulants comprising 4% acetic acid for 24 ± 0.5 hrs at 22 ± 2 °C, according to the test procedure specified in European Standards EN 1388-1 [23] and EN 1388-2 [24]. Vessels were filled with solvent up to 1 mm below the overflow edge and placed in a dark room at a temperature of 22 ± 2 °C and left for 24 hrs. In the case of the decorated glassware at the rim, then nickel and chromium migration occurred into 4% acetic acid for 24 ± 0.5 hours at 22 ± 2 °C [24] or 0.5% citric acid for 2 ± 0.1 hours at 70 ± 2 °C.

Determination of nickel and chromium

Migrated nickel and chromium was measured by FAAS, under the following conditions: for nickel, an acetylene-air flame; wavelength $\lambda = 232.0$ nm, a slit width: 0.2 nm and for chromium wavelength of $\lambda = 357.9$ nm, a slit width: 0.2 nm. Atomization was via the air-acetylene flame.

RESULTS

As aforementioned, the validation was in 2 stages ie. release of metals into the food simulants from ceramics and glassware followed by measurement of nickel and chromium by FAAS. The assessed analytical parameters were working range, limit of quantification (LOQ), the limit of detection (LOD), repeatability, accuracy, mean recovery and uncertainty; as shown in Table 1.

Table 1. Summarised validation parameters for the analytical method

Validation parameters	Nickel	Chromium
Working range [mg/L]	0.02 – 0.80	0.02 – 0.80
Limit of quantification (LOQ) [mg/L]	0.02	0.02
Limit of detection (LOD) [mg/L]	0.01	0.01
Repeatability [%]	5	6
Accuracy [%]	6.5	2.8
Mean recovery [%]	101	102
Uncertainty [%]	12	11

The method was thus proved to be analytically acceptable for the routine determination of nickel and chromium migrating from ceramic pots and glassware intended to come into contact with food; it was thereby adopted for this study.

Migration of nickel and chromium from the 171 test ceramic vessels into 4% acetic acid over 24 hrs contact was below the limit of quantification of the methods used analytical (LOQ = 0.02 mg/L). On only one occasion, for a decorated ceramic bowl's inner surface, the nickel migration was sufficiently high for measurement; at 0.04 mg/L.

For the test glassware with decorated rims, the migration of nickel and chromium into the 4% acetic acid (for 24 hrs of contact at 22 °C) or into 0.5% citric acid (for 2 hrs of contact at 70 °C) were below the limit of quantification of the analytical method used (LOQ=0.02 mg/L).

DISCUSSION

The migrations of metals from ceramic were depends on several factors, that include their material composition, techniques for manufacture and decoration, as well as the temperature and duration of contact made with the model solution or food.

Comparing the results of our study with others is difficult, as different food simulants and extraction conditions (eg. temperatures and migration times) had been used as well as differing LOQs in the various analytical methods used [1, 2, 16, 28, 30]. The most common model solutions used for studying metal migration were acetic, citric and malic acids [3, 9, 10]; these being widely present in food. It should also be noted that choosing a suitable acid determines not only the pH conditions but also its chelating ability. Now frequently used for measuring metal migration are food-stuffs such as vinegar, orange juice [1], tomato juice [3, 10], sauerkraut juice, pickles, low lactose content milk [28], colas, soft drinks and guarana [14].

A key feature in studying metal release from the

surface of ceramic and glassware is selecting the appropriate conditions for migration (type of model solution, temperature and contact time) so as to reflect actual conditions of culinary usage. This is justified by the diverse use of these products. Ceramic and glass vessels may be used in preparing and cooking food along with food storage. In order to measure the actual migration, this requires choosing appropriate conditions for migration reflecting actual culinary usage of ceramics and glassware. These conditions are a variable temperature and duration of contact and, above all, using a suitable model fluids that can simulate contact with a given foodstuff. Citric and malic acids more aggressively affect enamel and have a greater ability to leach metals, with the exception of aluminium, barium, chromium, iron and magnesium for which 4% acetic acid has proven to be the most effective [9].

Mazinianian et al. [20] studied the effect of citric acid and acetic acid in the leaching of metals from stainless steel cookware, where it was found that 0.3% citric acid was more effective for this purpose than 3% acetic acid, when testing acidic food.

However, it should also be appreciated that the properties of citric acid and acetic acid solutions when used as food simulants will not always reflect the real impact that food makes when coming into contact with the product, due to the latter's very complex composition. It therefore seems reasonable to study migration into food under real conditions so as to simulate the procedures of cooking or storage, which will optimize this metal leaching-process from surfaces [18]. As well as the chemical reactivities of the food dishes, the test method should ideally reflect their preparation process and also how the cooking articles make contact with food. It has been shown that the leaching of lead from the surface of ceramics subjected to microwave energy is similar to the metal's elution after 24 hrs from the product, when 0.5% citric acid is used as the model fluid [27]. It can therefore be expected that in microwaving food in earthenware vessels, it increase the migration of nickel and chromium from their surfaces.

Sheets [30] investigated the release of various metals from glazed porcelain decorated with overglazing coming from Europe and Asia into 4% acetic acid for 24 hrs. Nickel leaching measured from 46 vessels was below 0.05 µg/mL, whilst chromium migration from 44 vessels was below 0.10 µg/mL, however 2 vessels from Japan were found to be at the LOQ of 0.1 µg/mL. *Mohamed* et al. [21] studied nickel migration from glazed earthenware into 4% acetic acid at 30-32 °C and found this to be as low as 0.2 mg/L.

In other studies, nickel and chromium release from ceramic drinking vessels manufactured in India was measured into brewed tea (80 °C for 24 hrs), orange juice (23 °C, 24 hrs) and 4% acetic acid (24 hrs at rt, then at 40 °C and 60 °C). For the tea, the concentra-

tions of nickel and chromium after the migration were respectively 70-80 µg/L and 62-119 µg/L, whilst for orange juice they were respectively 70-134 µg/L and 66-945 µg/L and for 4% acetic acid nickel and chromium was undetectable; with the method's LOQ being 15.0 µg/L (for nickel), and 7.0 µg/L (for chromium) [1].

Findings of the present study demonstrated low migration of nickel and chromium (below the LOQ of 0.02 mg/L) from ceramics and glassware into the tested model fluids, thus indicating that they pose no threat to the health of the consumer. Any potential exposure to these metals released from such products food will thereby be small.

CONCLUSIONS

1. Due to the low levels of nickel and chromium migration, the tested ceramics with the inside coloured surfaces and glassware with decorated rims, available on the Polish market, are not in this respect a health risk to consumers.
2. The validated parameters of the analytical method indicate that it can be used for the routine measurement of nickel and chromium migrating into food simulants.
3. Because nickel compounds are allergenic and carcinogenic and that chromium ones are mutagenic, embryotoxic and teratogenic it is advisable to further investigate migration of these elements from articles intended to come into contact with food, including those metallic items.

Conflict of interest

The authors declare that there are no conflicts of interest.

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