

EXPOSURE ASSESSMENT TO LEAD, CADMIUM, ZINC AND COPPER RELEASED FROM CERAMIC AND GLASS WARES INTENDED TO COME INTO CONTACT WITH FOOD

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ABSTRACT

Background. One of the important source of food contamination with elements harmful to human health such as lead or cadmium is migration from the articles intended to come into contact with food. Ceramic and glass articles can also be a source of zinc and copper which compounds are used during the production process of vessels.

Objective. Determination of migration of lead, cadmium, zinc and copper from ceramic and glass wares available on the market in Poland and assessment of consumer exposure to these elements.

Material and methods. Ceramic and glass articles which were available on the retail market and mainly produced in China were tested for release of lead, cadmium, zinc and copper. Samples for testing were prepared in accordance with European Standards: EN 1388-1 and EN 1388-2 and the migration of elements into a food simulant (4% acetic acid) was measured using flame atomic absorption spectrometry (FAAS). Validated and accredited methods according to EN ISO/IEC 17025:2005 meeting the criteria set in the legislation were applied. Exposure assessment was performed taking into account actual reference doses introduced by the European Food Safety Authority (EFSA) and the Joint FAO/WHO Expert Committee on Food Additives (JECFA).

Results. 736 samples of ceramic and glass wares were tested. Among investigated samples for migration of lead 82% of results were below limit of quantification (LOQ = 0.1 mg/L) of the analytical method used. For cadmium zinc and copper results below LOQ were found in 94%, 79% and 100% tested samples, respectively (Cd LOQ = 0.01mg/L; Zn LOQ = 0.05 mg/L; Cu LOQ = 0.03 mg/L). Only one sample release lead in amount higher than limit set in Polish Standard PN-B-13210:1997, it was rim of the glass mug and migration value was 4.81 mg/article. In case of cadmium there were two samples which exceeded permissible migration limit, it was flat ceramic dish and the rim of the glass mug, migration values were: 0.14 mg/dm² and 0.42 mg/article respectively. The most measurable results (above LOQ) in case of lead and cadmium were for the samples of ceramic and glass drinking vessels. Quantifiable amounts of zinc were found also in other groups of articles tested: flat and deep ceramic dishes. None of the tested articles released copper in amount higher than LOQ value. The risk assessment showed that lead and cadmium exposure for some of the samples far exceed the reference doses. For children, assuming a single use only of these vessels, these doses may be exceeded: in the case of lead, 69 times and in the case of cadmium, 8 times.

Conclusions. It is the urgent need to introduce requirements into the applicable legislation for the rim area of drinking ceramic and glass vessels to reduce exposure especially by the most vulnerable groups of population such as children.

Key words: lead migration, cadmium migration, copper migration, zinc migration, ceramic wares, glass wares, food contact articles, exposure assessment

STRESZCZENIE

Wprowadzenie. Jednym z istotnych źródeł zanieczyszczenia żywności pierwiastkami szkodliwymi dla zdrowia człowieka, takimi jak ołów lub kadm, jest ich migracja z wyrobów do kontaktu z żywnością. Wyroby ceramiczne i szklane mogą być również źródłem pobrania poza ołowiem i kadmem innych pierwiastków, których związki są wykorzystywane podczas procesu produkcji naczyń, takich jak cynk i miedź.

Cel. Celem badań było zbadanie migracji ołowiu, kadmu, cynku i miedzi z wyrobów ceramicznych oraz wyrobów szklanych dostępnych na rynku w Polsce oraz oszacowanie narażenia konsumentów na te pierwiastki.

Material i metody. Wyroby ceramiczne i szklane dostępne na rynku detalicznym i produkowane głównie w Chinach, zostały zbadane pod kątem uwalniania ołowiu, kadmu, cynku i miedzi. Próbkę do badań przygotowano zgodnie z normami europejskimi: EN 1388-1 i EN 1388-2, a migracja pierwiastków do płynu modelowego (4% kwasu octowego) była mierzona metodą płomieniowej atomowej spektrometrii absorpcji (FAAS). Stosowano zwalidowane i akredytowane metody zgodne z EN ISO/IEC 17025: 2005 spełniające kryteria określone w ustawodawstwie. Ocenę narażenia przeprowadzono

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przy uwzględnieniu aktualnych dawek referencyjnych określonych przez Europejski Urząd ds. Bezpieczeństwa Żywności (EFSA) i Połączony Komitet Ekspertów FAO/WHO ds. Dodatków do Żywności (JECFA).

Wyniki. Zbadano 736 próbek wyrobów ceramicznych i szklanych. Wśród badanych próbek w kierunku migracji ołowiu 82% wyników było poniżej granicy oznaczalności (LOQ = 0.1 mg/L) zastosowanej metody analitycznej. Dla kadmu, cynku i miedzi wyniki poniżej LOQ stwierdzono odpowiednio w: 94% 79% i 100% zbadanych próbek (Cd LOQ = 0.01 mg/L; Zn LOQ = 0.05 mg/L; Cu LOQ = 0.03 mg/L). Tylko dla jednej próbki stwierdzono uwalnianie ołowiu w ilości wyższej niż dopuszczalny poziom określony w polskiej normie PN-B-13210:1997, był to kubek szklany, a wartość migracji z obrzeża naczynia wyniosła 4,81 mg/wyrób. W przypadku kadmu dwie próbki przekroczyły dopuszczalny limit, było to płaskie ceramiczne naczynie i obrzeże szklanego kubka, wartości migracji wynosiły odpowiednio: 0,14 mg/dm² i 0,42 mg/wyrób. Najwięcej mierzalnych wyników (powyżej LOQ) w przypadku ołowiu i kadmu było wśród próbek ceramicznych i szklanych naczyń do picia. Wyniki mierzalne dla cynku znaleziono również w innych grupach badanych produktów tj.: płaskich i głębokich naczyń ceramicznych. Żadna z badanych próbek nie uwalniała miedzi w ilości wyższej niż wartość granicy oznaczalności (LOQ). Ocena ryzyka wykazała, że narażenie na ołów i kadm w przypadku niektórych zbadanych naczyń znacznie przekracza dawki referencyjne. W przypadku dzieci, przyjmując jednorazowe tylko użycie tych naczyń, dawki te mogą zostać przekroczone: dla ołowiu - 69 razy, dla kadmu - 8 razy.

Wnioski. Istnieje pilna potrzeba ustalenia wymagań dla obrzeża naczyń ceramicznych i szklanych w obowiązujących przepisach, w celu zmniejszenia narażenia, zwłaszcza przez najbardziej wrażliwe grupy populacji takie jak dzieci.

Słowa kluczowe: migracja ołowiu, migracja kadmu, migracja miedzi, migracja cynku, naczynia ceramiczne, naczynia szklane, wyroby do kontaktu z żywnością, ocena narażenia

INTRODUCTION

Ceramic and glassware can be an important source of intake of elements harmful to human health such as lead and cadmium [1, 21, 35]. The accessible data from other countries, related to the migration of metals from ceramic articles point to potential risk of adverse health effects also in case of others metals such as zinc and copper [3, 29, 30, 31, 32]. All these metals are used in pigments or glaze. The glaze on properly made and kilned plates and dishes should normally be unaffected by food and should not release metals during cooking or standing [10, 16]. If ceramic articles are baked at the wrong temperature, the glazing will not have the desired sealing properties and harmful to human health elements can migrate into food and drink with a low pH [2, 3, 7, 12, 15, 37].

The amount of migration depends on many factors, namely: the quality of the raw materials used for production, the course of the technological process, the type of food contacting, time and temperature of contact [14, 21]. Particular risk may be created by products produced on a small scale in small craft workshops, where the principles of good manufacturing practice do not apply [31, 32, 35]. Numerous of Rapid Alert System for Food and Feed (RASFF) notifications in the last years in this area indicate potential threat to human health. In 2017 from 43 notifications concerning heavy metals migration from food contact materials 11 concerned ceramic and glass articles [28]. (Figure 1). An example of a country where especially children are exposed to significant amounts of lead intake due to its migration from traditional ceramics is Mexico. The studies shows that children of families who used lead-glazed ceramics had blood lead levels 18.5% higher than children from other families were such articles were not used [25].

The European Food Safety Authority (EFSA) and the Joint FAO/WHO Expert Committee on

Food Additives (JECFA) in the recent years re-evaluated the risk assessment to human health related to these toxic elements [4, 8, 9, 13, 24]. JECFA identified neurodevelopmental effects in children and cardiovascular effects in adults as a critical effects of adverse lead action; in addition to that EFSA CONTAM Panel identified nephrotoxic effects as relevant to the risk assessment in adults. The PTWI at 25 µg/kg b.w. (body weight) was withdraw because did not guarantee health safety but JECFA do not established new reference dose that would be considered to be health protective [8, 9].

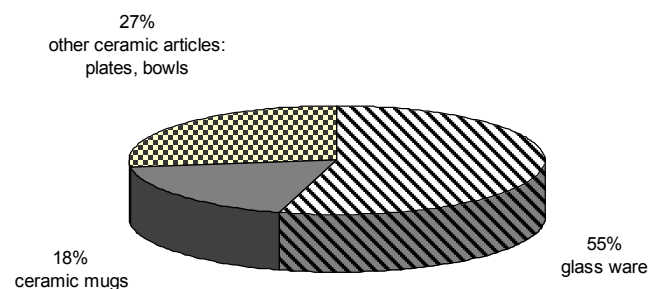


Figure 1. RASFF notifications in 2017 concerning ceramic and glass articles intended into contact with food

EFSA has established the lowest Dose Lower Confidence Limit for lead: BMDL₀₁ for children (neurotoxic effect) 0.50 µg/kg b.w./day; for adults BMDL₁₀ 0.63 µg/kg b.w./day (nephrotoxicity) and BMDL₀₁ 1.50 µg/kg b.w./day (cardiovascular disorders). For cadmium EFSA established a tolerable weekly intake (TWI) of 2.5 µg/kg b.w., factor 2.8 lower than the previous JECFA PTWI of 7 µg/kg b.w. [24, 27]. JECFA, using different statistical approach than EFSA, established for cadmium the Provisional Tolerable Monthly Intake (PTMI) of 25 µg/kg b.w. [9]. Visible, clinical symptoms at low levels of lead and cadmium exposure do not occur immediately, these

elements accumulate in the body. The toxic effects of their activity, even at low levels of exposure, do not occur immediately but after a few months and a few years (carcinogenic) or in a next generations (mutagenic effect); early changes can only be observed at the physiological or biochemical level. Copper and zinc are essential elements in the nutrition of mammals, but their high levels can be toxic for organism. Recommended Dietary Allowance (RDA) of zinc was set at the levels: for children 5 mg/day and for adults 10 mg/day. High levels of zinc are toxic for the hematopoietic system, kidney and pancreas during long-term exposure. JECFA established provisional maximum tolerable daily intake (PMTDI) for zinc at the range: 0.3 mg/kg b.w. - 1 mg/kg b.w. [33]. For copper RDA value was established for children at the level 0,44 mg/day and for adults 1 mg/day. High levels of copper can cause adverse effects in humans, as liver and kidney damage, gastrointestinal effects. Derives PMTDI by JECFA was established at 0.05 mg/kg b.w. and 0.5 mg/kg b.w. for children and adults respectively [34].

The existing evaluations of population dietary exposure levels to metals noxious to health do not take into consideration such important source of oral heavy metals intake as migration from articles intended to come into contact with foodstuffs. Dietary exposure is clearly the dominating source of overall lead and cadmium exposure for non-smokers, with a small potential contribution from dust in particularly contaminated areas [8, 24].

Additional exposure via migration of such and toxic elements, with similar mechanism of activity, from glass and ceramic should be reduced to minimum [3, 25, 26]. Manufacturers of materials and articles intended to come into contact with food should apply the principles of good manufacturing practice (GMP) to ensure safety of their products [5]. According to regulation (WE) No 1935/2004 [22], materials and articles intended to come into contact with foodstuffs must not transfer their constituents to foodstuffs in quantities which could endanger human health. Council Directive No 84/500/EEC on ceramics, implemented in Polish Regulation of the Minister of Health [23], regulates only lead and cadmium. However, other toxic elements such as zinc, copper, cobalt, barium, aluminum, nickel, antimony, vanadium can be leached from ceramics and pose a risk to public health, especially when colored glazes are applied [7]. Limits of migration of lead and cadmium from the rim area are set in Polish Standard PN-B-13210:1997 at the levels 2.0 mg/article and 0.2 mg/article respectively [17].

As part of the working group on materials for contact with food, intensive work is already under way to reduce the current limits on the migration of lead and cadmium from ceramic articles and set requirements in legislation for other elements posing a threat to public

health, as well as set the requirements of enamel and glass products including the drinking rim.

Based on the health data in opinions published by EFSA a significant reduction of the migration limits for a lead and cadmium was proposed, 400 and 60 times lower values were introduced respectively. Currently draft document regarding the revision of Directive 84/500 is at the stage of consultations with industry representatives [36]. The finalization of works in the above mention scope is expected in the near future.

The study aims were to determine lead, cadmium, zinc and copper migration into the food simulant (4% acetic acid) from ceramic and glass articles intended to come into contact with food and estimate consumer exposure to these elements.

MATERIAL AND METHODS

Test Samples

736 articles of ceramic and glassware (with 184 decorations) that were available on the retail market and mainly manufactured in China, with the inside surfaces being coloured and a decorated rim were tested in scope of lead, cadmium, copper and zinc migration. The products under analysis were:

Ceramic articles: 248 flat dishes; 276 deep dishes; 120 drinking vessels with decorated rims;

Glass articles: 36 deep dishes; 56 drinking vessels with decorated rims.

According to the European Standards: EN 1388-1, EN 1388-2 and EU legislation on ceramics the laboratory sample consisted of 4 individual identical articles produced from the same material, identical shape, dimensions, decoration and glaze [6, 18, 19, 23].

Reagents

Acetic acid (99.5% - 99.9%, Avantor), standard solution 1g/L (GUM, Poland): lead, cadmium, copper, zinc and deionized water. All reagents used were on an appropriately high degree of purity.

Apparatus and instrumentation

For determination of migration of lead and cadmium atomic absorption spectrometer ASA Spectr AA 220 Varian was used, while for copper and zinc spectrometer, Philips Pye Unicam, model 9200. At the stage of sample preparation class A laboratory glassware was used.

Migration test conditions

Migration of lead, cadmium, copper and zinc from the surface of ceramic and glass articles into a food simulant 4% acetic acid for 24 ± 0.5 hrs at 22 ± 2 °C was performed, according to the test procedure specified in European Standards EN 1388-1 and EN 1388-2 [18, 19]. Vessels were filled with acid up to 1 mm below the overflow edge and placed in a dark at

a temperature of 22 ± 2 °C and left for 24 hrs. In the case of the decorated glass and ceramic ware at the rim, the same analytical conditions were used.

Determination of lead and cadmium was measured by FAAS, under the following conditions: an acetylene-air flame; lead wavelength $\lambda = 217.0$ nm, a slit width: 0.5 nm; cadmium wavelength of $\lambda = 228.8$ nm, a slit width: 0.5 nm, copper wavelength of $\lambda = 324.8$ nm, a slit width: 0.5 nm and zinc wavelength of $\lambda = 213.9$ nm, a slit width: 0.5 nm.

Methods for determining of lead and cadmium meets the efficiency criteria set forth in the prevailing legislation [6, 23] and have been accredited by the Polish Accreditation Center (PCA) according to EN ISO/IEC 17025:2005 [20]. Methods for determination of migration

of copper and zinc were fully validated in the Laboratory of Department of Food Safety in the National Institute of Public Health – National Institute of Hygiene.

RESULTS

736 articles made from ceramic or glass were tested for migration lead, cadmium, zinc and copper. Among tested samples for lead migration 82% samples were below limit of quantification (LOQ) of the analytical method used. In case of other elements tested results below LOQ value for cadmium, zinc and copper were found in 94% 79% and 100% respectively. The results of lead, cadmium, zinc and copper migration from ceramic and glass articles are presented in the Tables 1-3.

Table 1. Lead migration from ceramic and glass articles

Article	Number of individual articles***	Distribution of samples					Maximum migration	Units
		below permissible levels				above permissible level		
Flat ceramic dishes	248 (62)	< 0.1	0.1-0.2	0.2-0.4	0.4-0.8	> 0.8	0.75	mg/dm ²
		47	7	7	1	-		
Deep ceramic dishes**	276 (69)	< 0.1	0.1-0.5	0.5-2.0	2.0-4.0	> 4.0	0.87	mg/L
		55	12	2	-	-		
Ceramic drinking vessels (rims)	120 (30)	< 0.1	0.1-0.5	0.5-1.0	1.0-2.0	> 2.0	0.29	mg/article
		28	2	-	-	-		
Glass drinking vessels (rims)	56 (14)	< 0.1	0.1-0.5	0.5-1.0	1.0-2.0	> 2.0	4.81	mg/article
		12	-	-	1	1		
Glass deep dishes**	36 (9)	< 0.1	0.1-0.5	0.5-2.0	2.0-4.0	> 4.0	<LOQ*	mg/L
		9	-	-	-	-		

* LOQ for lead 0.1 mg/L

** with vessels for beverages

*** the number of laboratory samples is given in brackets

Table 2. Cadmium migration from ceramic and glass articles

Article	Number of individual articles***	Distribution of samples					Maximum migration	Units
		below permissible levels				above permissible level		
Flat ceramic dishes	248 (62)	< 0.01	0.01-0.02	0.02-0.04	0.04-0.07	> 0.07	0.14	mg/dm ²
		60	-	-	1	1		
Deep ceramic dishes**	276 (69)	< 0.01	0.01-0.05	0.05-0.15	0.15-0.30	> 0.30	0.15	mg/L
		65	1	3	-	-		
Ceramic drinking vessels (rims)	120 (30)	< 0.01	0.01-0.05	0.05-0.10	0.10-0.20	> 0.20	0.02	mg/article
		29	1	-	-	-		
Glass drinking vessels (rims)	56 (14)	< 0.01	0.01-0.05	0.05-0.10	0.10-0.20	> 0.20	0.42	mg/article
		12	-	-	1	1		
Glass deep dishes**	36 (9)	< 0.01	0.01-0.05	0.05-0.15	0.15-0.30	> 0.30	< 0.01*	mg/L
		9	-	-	-	-		

* LOQ for cadmium 0.01 mg/L

** with vessels for beverages

*** the number of laboratory samples is given in brackets

Table 3. Zinc and copper migration from tested ceramic and glass articles

Article	No individual articles (No laboratory samples)	Distribution of samples range of results					Maximum migration		Units
		Zn			Cu		Zn	Cu	
Flat ceramic dishes	248 (62)	<0.05	0.05-0.20	0.20-0.30	<0.03	>0.03	0.26	<0.03*	mg/dm ²
		48	13	1	62	-			
Deep ceramic dishes**	276 (69)	< 0.05	0.05-0.30	0.30-0.90	<0.03	>0.03	0.85	<0.03*	mg/L
		53	12	4	69	-			
Ceramic drinking vessels (rims)	120 (30)	< 0.05	0.05-1.0	1.0-3.0	<0.03	>0.03	2.31	<0.03*	mg/article
		25	4	1	30	-			
Glass drinking vessels (rims)	56 (14)	< 0.05	0.05-3.0	3.0-10.0	<0.03	>0.03	9,90	<0.03*	mg/article
		12	-	2	14	-			
Glass deep dishes**	36 (9)	< 0.05	0.01-0.05	0.05-0.15	<0.03	>0.03	<LOQ*	<0.03*	mg/L
		9	-	-	9	-			

*LOQ: for zinc: 0.05 mg/L; for copper: 0.03 mg/L

**with vessels for beverages

DISCUSSION

Flat ceramic dishes

Out of 248 analysed samples (62 decorations) of flat ceramic dishes 47 analytical results (76%) for lead, 60 (97%) for cadmium, 48 (77%) for zinc and all results for copper were below LOQ i.e. 0.1 mg/L; 0.01 mg/L; 0.05 mg/L and 0.03 mg/L respectively. Measurable concentrations of lead and cadmium were significantly below permissible levels for lead (0.1 mg/dm²) and cadmium (0.01 mg/dm²) except one result for cadmium which exceeded this limit. Concentration of zinc was low and does not pose health risk for public health. The results obtained by other authors regarding the migration of zinc and copper from ceramic products are similar to those obtained in our research [11].

Deep ceramic dishes

In case of 276 samples (69 decorations) of deep ceramic dishes tested for migration of lead and zinc about 80% obtained results were below LOQ of analytical methods for lead and zinc, respectively. Quantifiable amounts were reported for 14 samples of lead and 16 samples of zinc. The highest reported values were: 0.87 mg/L for lead and 0.85 mg/L for zinc, respectively and in case of lead was significantly lower than permissible level set in legislation for this category of dishes – 4.0 mg/L. For cadmium 94% of obtained results were below LOQ value. None of the articles transferred more cadmium than migration limit of 0.3 mg/L. The highest obtained result was 0.15 mg/L. No quantifiable amounts of copper were measured from the surface of deep ceramic dishes.

Ceramic drinking vessels

From among ceramic drinking vessels including vessels for beverages, tested for migration of elements from the rim area they were only a few measurable results. Above 90% results were below LOQ values. Lead migration from the rims of ceramic vessels did not exceed the 2.0 mg/article limit according to Polish Standard PN-B-13210 “Glass and ceramic products intended for contact with food. Permissible amounts of released lead and cadmium“, [17]. Quantifiable amounts of lead but significantly lower from the limit, were measured in 2 samples of ceramic mugs richly decorated on the rim. Trace of cadmium but considerably lower from the limit 0.2 mg/article were measured in one of the 30 tested rims of drinking vessels. 97% of results were below limit of quantification of the analytical method. None of the 30 samples of ceramic drinking vessels tested transferred quantifiable amounts of copper and only in 5 samples from 30 tested migration of zinc was measurable. The maximum result of migration obtained for zinc was 2.31 mg/kg.

Glass drinking vessels

The highest migration of lead was reported for the rim of richly decorated on external surface tumbler it was 4.81 mg/article. This value exceeded, over 2.4 times limit of release of lead set in PN-B-13210: 1997 amounting to the edge for drinking 2.0 mg/article. In case of cadmium they were 2 measurable results, one of them was higher than the limit set in Polish standard for migration of this element from the rim. Only 2 out of 14 samples had quantifies levels of zinc. Maximum value 9.90 mg/article was detected for the sample of glass mug. From this sample also migration

of cadmium was observed. No quantifiable amounts of copper were measured from the rims of the 56 tested glass drinking vessels.

Glass deep dishes

None of the 36 glass deep dishes including drinking vessels tested transferred quantifiable quantities of lead, cadmium, zinc and copper.

Exposure assessment

Health risk assessment was performed taking into account current reference doses for elements introduced by EFSA and JECFA [4, 8, 9, 13, 24] and the highest obtained results for migration of tested elements.

Taking into account the highest value for lead - 4.81 mg/article migrated from the rim of glass cup and assuming that the cup will be used once a week, exceedance of the BMDL₁₀ dose (nephrotoxicity effects - adults) and the dose BMDL₀₁ (cardiovascular disorders - adults) would be more than 15-fold and 6-fold; in the extreme case of daily use of cups with the same decoration, respectively, almost 110-fold and 46-fold. If the above-mentioned cups will be used by children, exceedance the value of the BMDL₀₁ dose – (neurotoxic effect) would be 69-fold; in the extreme case of everyday use of cups with the same decoration 481-fold respectively.

For cadmium the highest observed migration was from the rim of glass cup - 0.42 mg/article, leading to exceedance the TWI value with a single use of the vessel more than 2-fold for an adult, and in the extreme case of daily use of glasses with the same decoration, about 17-fold; for a child with a body weight of 20 kg, the reference dose would be exceeded 8-fold and about 59-fold respectively. Taking into account as a reference point the PTMI value (JECFA) and assuming the use of these cups once a week, reference dose for adults would be exceeded about 1-fold; when using daily -7-fold, for children 3.6-fold and 25-fold respectively. If we take into account the intake of lead and cadmium from other sources: such as contaminated food, air and water the real exposure will be much higher. The migration of lead and cadmium under real conditions of use is usually lower than for the food stimulant (acetic acid) depending on the properties of the foodstuffs contacted with the vessel. The real contact time of the food with the dish is usually shorter than during the tests. Other important aspect of migration is that migration of elements from ceramics or glass surfaces decreases upon repeated use of the vessels, the highest migration is observed from new articles [21].

The exposure to zinc migrated from the rim of glass drinking vessels at the highest level of 9.90 mg/article was low and for adults and children did not exceeded PMTDI value established by JECFA.

However, additional sources of zinc intake, including diet, should also be considered for the complete

exposure assessment and evaluation health hazard. The obtained migration results indicate a necessity to extend the Directive 84/500 of setting limits for drinking rim area of ceramic and glass vessels.

CONCLUSIONS

1. Most of the tested ceramic and glassware samples do not show the release of the tested elements in amounts exceeded limits set in legislation and they do not pose a health concern. In the case of individual samples, the intake of cadmium and lead, especially by children, can be an important from the health point of view.
2. High migration of elements observed in single cases may indicate that the principles of good manufacturing practice during the production of these products were not used.
3. It's advisable to introduce requirements for the rim area of drinking vessels, including glassware, as soon as possible.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgements

This study was undertaken as a scientific project No 4/ZŚ.1 during the years 2014-2017 financed by the National Institute of Public Health-National Institute of Hygiene, Warsaw, Poland.

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Received: 12.09.2018

Accepted: 07.11.2018