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EVALUATION OF SOLUBLE OXALATES CONTENT IN INFUSIONS OF DIFFERENT KINDS OF TEA AND COFFEE AVAILABLE ON THE POLISH MARKET

OCENA ZAWARTOŚCI ROZPUSZCZALNYCH SZCZAWIANÓW W NAPARACH RÓŻNYCH RODZAJÓW HERBATY I KAWY DOSTĘPNYCH NA POLSKIM RYNKU

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Key words: *oxalate acid, oxalates, tea, coffee, infusions*
Słowa kluczowe: *kwas szczawiowy, szczawiany, herbata, kawa, napary*

ABSTRACT

Background. Tea and coffee are the potentially rich source of oxalic acid, which can act as a antinutrient.
Objective. The aim of this study was to determine and evaluate the content of soluble oxalates in teas and coffees available on the Polish market.
Material and method. The green, red and black teas, and black natural ground and instant coffees were used for preparing the infusions. The manganometric method was used for the determination of the oxalates in the infusions.
Results. The mean oxalates content in the infusions from 3 g of black teas was 115.68 mg/100cm^3 and was higher as compared to red teas (101.91 mg/100cm^3) and green teas (87.64 mg/100cm^3). Disregarding the variety of analyzed teas, the largest oxalates content was in infusions of pure one-component tea - "Sir Roger" (164.82-174.22 mg/100cm^3), while the lowest oxalates content was noted in the tea containing the components from other plants ("Bio-Active" with grapefruit juice – reaching as low level as 39.00 mg/100cm^3). Instant coffees contained larger amount of oxalates than natural ground coffees. Irrespective of the kind of the tested coffees, the lowest oxalates content was found in the infusions from the following coffees: Tchibo Exclusive - 19.62 mg/100cm^3, Gala *ulubiona* - 37.32 mg/100cm^3, and Maxwell House - 38.40 mg/100cm^3, while the highest oxalates content in instant coffee - Nescafe Espiro 51.80 mg/100cm^3.
Conclusions. The results revealed a significant relation between phytochemical composition of analyzed teas and coffees and the level of soluble oxalates in infusions prepared from the tested products.

STRESZCZENIE

Wprowadzenie. Herbata i kawa stanowią potencjalnie bogate źródło kwasu szczawiowego, który ma działanie antyodżywcze.
Cel badań. Celem badań było zbadanie i ocena zawartości rozpuszczalnych szczawianów w naparach z różnych gatunków herbat zielonych, czerwonych i czarnych oraz naturalnych kaw mielonych i rozpuszczalnych dostępnych na polskim rynku.
Materiał i metoda. Z herbat zielonej, czerwonej i czarnej oraz kaw naturalnych mielonych i rozpuszczalnych przygotowywano napary. Oznaczenia zawartości rozpuszczalnych szczawianów w naparach wykonywano metodą manganometryczną.
Wyniki. Wykazano, że średnia zawartość szczawianów w naparach uzyskanych z 3,0 gramów herbat czarnych (115,68 mg/100 cm^3) jest wyższa, w porównaniu do herbat czerwonych (101,91 mg/100 cm^3) i herbat zielonych (87,64 mg/100 cm^3). Największą zawartość szczawianów zawierały napary z herbat czystych, jednoskładnikowych - „Sir Roger" (164,82-174,22 mg/100 cm^3), natomiast najmniejszą odnotowano w herbacie z komponentem roślinnym („Bio-Active" z sokiem grejpfrutowym - 39,00 mg/100 cm^3). Kawy rozpuszczalne zawierały istotnie większą zawartość szczawianów niż naturalne kawy mielone. Najniższą zawartość szczawianów odnotowano w naparach z kaw: Tchibo Exclusive - 19,62 mg/100 cm^3, Gala ulubiona - 37,32 mg/100 cm^3 i Maxwell House - 38,40 mg/100 cm^3, natomiast najwyższą zawartość w kawie rozpuszczalnej Nescafe Espiro - 51,80 mg/100 cm^3.
Wnioski. Wykazano istotny wpływ składu fitochemicznego badanych próbek herbaty i kawy na zawartość rozpuszczalnych szczawianów w naparach przygotowanych z badanych produktów.

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INTRODUCTION

Tea, due its sensory value is one of the most popular commercial beverages all over the world. In Poland tea constitutes about 40% of all beverages drunk in winter and 26% in summer season. Tea is drunk by approximately 75% of consumers more frequently than once a day, however the average Pole drinks less than 50 cups of tea per month [4, 5].

Teas are available on the market in many forms: as friable products for brewing, granulated products, packed in bags, and as a soluble instant powder [1, 15, 23].

In terms of chemical properties, tea contains alkaloids (caffeine, theobromine, theophylline), flavonoids (polyphenolic compounds, catechins, tannins), minerals and compounds which appear in leaves of other plants that is saccharides, proteins, lipids or chlorophyll [6, 19, 21, 22]. Apart from these components, tea also contains organic acids: oxalic, citric, malic, succinic, pyruvic, fumaric, which significantly increase nutritive and dietetic value of tea [4].

The content of soluble oxalates in teas, reported in the literature, differ significantly due to country of origin, weather conditions during the growth of tea plants, way of leaves processing or time and way of brewing and extraction conditions [9, 10, 13, 24].

However, despite of numerous opinions in the literature underlining positive influence of drinking tea on health it contain, apart from valuable components, also substances which consumed in large amounts can act as antinutrients (*i.e.* oxalic acid) reducing utilization of some minerals from the diet, transforming them into insoluble oxalates of very low bioavailability [11, 14, 16, 20].

It seems that coffees contain less lithogenic oxalic acid, but similarly to tea should not be drunk in unlimited amounts [12, 14, 25]. *Gasińska* and *Gajewska* [8] confirmed that more than 80% of tea infusions contribute in creating of kidney stones in adults in Poland. Lowering the amount of oxalates in the diet can be achieved by reducing consumption of black tea brewed for a long time, strong natural coffees, spinach, sorrel, rhubarb, seeds of leguminous plants, tomato concentrate and chocolate.

The aim of this study was to evaluate oxalic acid content in the infusions of varieties of green, red and black teas and natural ground and instant coffees available on the market as a potentially rich source of lithogenic oxalates [8].

MATERIAL AND METHODS

The study material consisted of nine kinds of commercial teas: green (1 tea bag, 3 leafy), red (1 leafy, 1 tea bag) and black (1 leafy, 2 tea bags) and six commercial black coffees (3 instant and 3 ground) (Tab. 1, 2). The products for the study were bought in supermarkets and groceries in Lublin between February and June 2009. Five samples of each kind of tea and coffee were analyzed.

Table 1. Characteristic data of studied types of teas
Charakterystyka badanych rodzajów herbat

Trade name	Type of tea/producer/country of origin/form of tea
Lipton Morocco (mind and spice)	green tea / Unilever / India / bag
Loyd Tea with petals of marigold and pieces of orange opuntia flavoured	green tea / Mocate / China / leafy
Bio-Active with quince fruit	green tea / Bio-Active / China / leafy
Tetley Green Leaf	green tea / Tetley / India / leafy
Sir Roger Pu-Erh	red tea / Roger / Sri Lanka / leafy
Bio-Active with grapefruit juice	red tea / Bio-Active / China / bag
Sir Roger Earl Grey	black tea / Roger / Sri Lanka / leafy
Saga	black tea / Unilever / India / bag
Minutka	black tea / Mocate / India / bag

Table 2. Characteristic data of studied types of coffees
Charakterystyka badanych rodzajów kaw

Trade name	Type of coffee / Producer / Grade of coffee
Jacobs Velvet	instant / Kraft Foods / 100% natural coffee
Nescafe Espiro	instant / Nestle / Robusta, Arabica
MK Cafe Premium	instant / Strauss Cafe /100% natural coffee
Gala (ulubiona)	ground / Tchibo / 100% Robusta
Maxwell House	ground / Kraft Foods / 100% natural coffee
Tchibo Exlusive	ground / Tchibo / 100% Arabica

For the determination of the soluble oxalates in the products the manganometric method described by *Brzozowska* et al. [2] was used. 3 g of tea and coffee were weighted on laboratory balance and inundated with 100 cm^3 of distilled water (Millipore, France) at 100°C. After 5 minutes of extraction the infusion was passed through qualitative filters - 9 cm diameter made from blotting paper with an average filtration speed 65 g/m^2 (POCH, Poland). 10.0 cm^3 of the infusions were taken for analysis and transferred into the test-tubes of 20 cm^3 volume. 5.0 cm^3 of 5% calcium chloride and 5.0 cm^3 of acetone were added and mixed. Samples were cooled for 30 minutes in the freezer in temperature -6°C. Then, each infusion was transferred from the test tubes into two centrifuge tubes of 12 cm^3 volume, maintaining the same volume 10 cm^3 of the solution in the each centrifuge tube and centrifuged for 10 minutes with 3000 rotations per minute. The supernatant was decanted, and 5.0 cm^3 of 10% sulfuric acid was added to the remaining sediment, then the resulting solution was transferred quantitatively from both centrifuge tubes to

Table 3. Content of soluble oxalates in green (1-4), red (5-6) and black (7-9) teas
Zawartość rozpuszczalnych szczawianów w zielonych (1-4), czerwonych (5-6) i czarnych (7-9) herbatach

No	Trade name	Number of samples	Statistical	mg oxalates/100cm^3 infusion	mg oxalates/100g dry matter
1.	„Lipton" Morocco (mind and spice)"	5	Mean ($\bar{x}$)	65.73^{BC}	1095.3^{BC}
			Range	(59.41-73.81)	(990.0-1230.0)
			Median	63.97	1066.0
2.	„Loyd Tea" with petals of marigold and pieces of orange, opuntia flavoured	5	Mean ($\bar{x}$)	97.01^{B}	1616.7^{B}
			Range	(91.81-106.21)	(1530.0-1770.0)
			Median	93.01	1550.0
3.	„Bio-Active" with quince fruit	5	Mean ($\bar{x}$)	91.21^{BC}	1520.0^{BC}
			Range	(61.21-113.41)	(1020.0-1890.0)
			Median	99.01	1650.0
4.	„Tetley" Green Leaf	5	Mean ($\bar{x}$)	96.61^{B}	1610.0^{B}
			Range	(81.01-124.21)	(1350.0-2070.0)
			Median	84.61	1410.0
5.	„Sir Roger" Pu-Erh	5	Mean ($\bar{x}$)	164.82^{A}	2746.7^{A}
			Range	(152.42-181.82)	(2540.0-3030.0)
			Median	160.22	2670.0
6.	„Bio-Active" with grapefruit juice	5	Mean ($\bar{x}$)	39.00^{C}	650.00^{C}
			Range	(35.10-44.10)	(585.00-735.00)
			Median	37.80	630.00
7.	„Sir Roger" Earl Grey	5	Mean ($\bar{x}$)	174.22^{A}	2903.3^{A}
			Range	(160.22-202.22)	(2670.0-3370.0)
			Median	160.22	2670.0
8.	„Saga"	5	Mean ($\bar{x}$)	89.41^{BC}	1490.0^{BC}
			Range	(88.21-91.81)	(1470.0-1530.0)
			Median	88.21	1470.0
9.	„Minutka"	5	Mean ($\bar{x}$)	83.41^{BC}	1390.0^{BC}
			Range	(79.21-86.41)	(1320.0-1440.0)
			Median	84.61	1410.0

$^{A, B, C}$ - values in the same column marked with different letters differ significantly at $p \leq 0.01$

the 100 cm^3 *Erlenmeyer's* flask, and heated in the water bath in 90°C. Titration in hot temperature was conducted with 0.02 N solution of potassium permanganate until pink color appeared and remained for about 1 minute.

Determinations were made in three repetitions for each infusion. The content of soluble oxalates in the tested teas and coffees was expressed per 100 cm^3 of infusion and 100 g of dry matter of the tested product. The data were analysed statistically by the program STATISTICA, version 6.0 PL, and the results were expressed as mean ($\bar{x}$), range and median. The statistical significance of differences between the mean values was estimated by means of a single-factorial variance analysis ANOVA assuming significance level at $p \leq 0.01$.

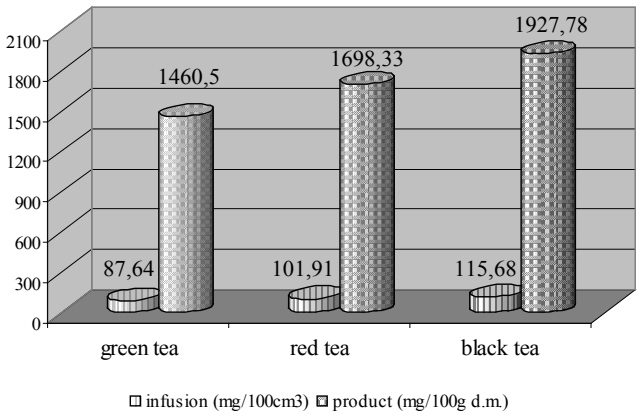

Fig. 1. Content of soluble oxalates depending on the kind of obtained teas
Zawartość rozpuszczalnych szczawianów w zależności od rodzaju pozyskanych herbat

RESULTS AND DISCUSSION

Results of the determinations of oxalates in 45 tested samples of infusions from green, red and black teas are presented in Table 3 and Figures 1 and 2.

The highest oxalates content were found in black teas - 115.68 mg/100 cm^3 of infusion which is equivalent to 1927.78 mg/100 g of dry matter, whereas the lowest content of oxalates was in red and green teas (Fig. 1).

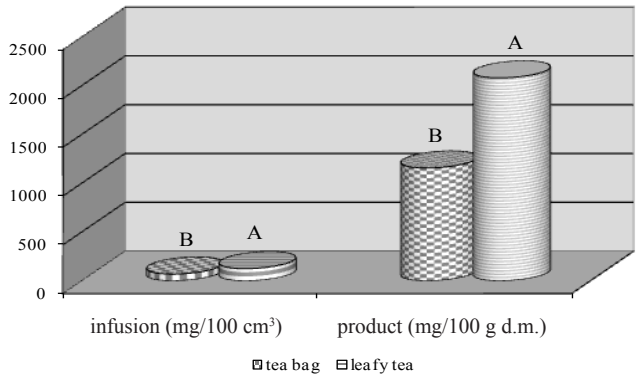

Fig. 2. Content of soluble oxalates depending on the form of obtained teas
Zawartość rozpuszczalnych szczawianów w zależności od formy pozyskanych herbat

The differences presented above appeared to be statistically insignificant. Results of analysis concerning content of these compounds in different kinds of teas available on the Polish market are consistent with the results by *Charrier's* et al. [3] who reported oxalates content in black tea from tea bags on the average level 4.68 mg/g of dry matter, in black leafy teas on the average level 5.11 mg/g of dry matter, and in green teas on the average level 0.68 mg/g of dry matter.

The results of this study showed that the content of oxalates in green teas are comparable or slightly higher than those recently published by *Sperkowska* and *Bazylak* [17], who reported the oxalates content from 38.19 to 78.41 mg/100 cm^3 of infusion which correspond to 636.43 and 1306.61 mg/100g of dry matter.

Divergences of oxalates levels in teas are could result from the use of different analytical techniques (method manganometric, enzymatic, high performance liquid chromatography, capillary electrophoresis), agrotechnical conditions, period of harvest and tea leaves processing technology, as well as different extraction temperatures, different sample mass or different ways of preparing infusions. This fact was confirmed in the study by *Sperkowska* and *Bazylak* [18] where the content of oxalates in black tea in the bags "Saga" was reported at the level - 33.52 mg/100 cm^3 of the infusion, which corresponds to 11.97 mg/g of dry matter. About 2.5-fold higher oxalates content in the analyzed infusions from the tea bags might be due to the fact that the tested samples were heavier than 3 g, as compared to the samples weighted 1.4 g which were used by the cited authors.

The results obtained in this study showed that regardless the type of analyzed products the significantly ($p \leq 0.01$) larger content of oxalates were in the infusions from the one-component teas ("Sir Roger"), as compared to teas containing components from other plants (Bio-Active" with grapefruit juice) (Tab.3). Similar observations concerning content of oxalates in green teas were made by *Charrier* et al. [3] and *Sperkowska* and *Bazylak* [17].

Among all analysed teas the leafy teas: red and black "Sir Roger" contained the largest amounts of oxalates

Table 4. Content of soluble oxalate in instant (1-3) and ground (4-5) coffees
Zawartość rozpuszczalnych szczawianów w rozpuszczalnych (1-3) i mielonych (4-5) kawach

No	Trade name	Number of samples	Statistical	mg oxalates /100cm^3 infusion	mg oxalate/100g dry matter
1.	Jacobs Velvet	5	mean ($\bar{x}$)	*45.60^{AB}*	*760.00^{AB}*
			range	(43.20-48.60)	(720.00-810.00)
			median	45.00	750.00
2.	Nescafe Espiro	5	mean ($\bar{x}$)	*51.80^{A}*	*863.33^{A}*
			range	(47.40-55.81)	(790.00-930.00)
			median	52.20	870.00
3.	MK Cafe Premium	5	mean ($\bar{x}$)	*41.92^{AB}*	*698.67^{AB}*
			range	(36.90-50.41)	(615.00-840.00)
			median	38.46	641.00
4.	Gala (ulubiona)	5	mean ($\bar{x}$)	*37.32^{B}*	*622.00^{B}*
			range	(34.56-39.60)	(576.00-660.00)
			Median	37.80	630.00
5.	Maxwell House	5	mean ($\bar{x}$)	*38.40^{B}*	*640.00^{B}*
			range	(34.20-41.40)	(570.00-690.00)
			median	39.60	660.00
6.	Tchibo Exlusive	5	mean ($\bar{x}$)	*19.62^{C}*	*327.00^{C}*
			range	(17.64-21.96)	(294.00-366.00)
			median	19.26	321.00

$^{A, B, C}$ - values in the same column marked with different letters differ significantly at $p \leq 0.01$

approximately (164.82 mg/100cm^3 which corresponds to 2746.7 mg/100 g of dry matter and 174.22 mg/100cm^3 which corresponds to 2903.3 mg/100 g of dry matter. In the infusions made from the tea bags "Saga" and "Minutka" the oxalates content reached similar level (89.41-83.41 mg/100 cm^3 of infusion corresponding to 1490.00-1390.00 mg/100g of dry matter), while in the infusions prepared from the remaining varieties of teas in bags contained lower concentrations of oxalates.

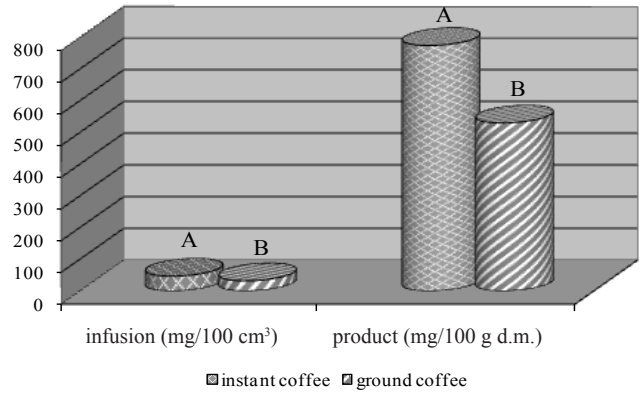

Fig. 3. Content of soluble oxalate depending on the kind of obtained coffees
Zawartość rozpuszczalnych szczawianów w zależności od rodzaju pozyskanych kaw

In the infusions from black tea in bags the significantly ($p \leq 0.01$) lower (ca. 80%) oxalates content as compared to the infusions from leafy teas containing 124.77 mg/100 cm^3 of oxalates corresponding to 2079.33 mg/100 g of dry matter was found (Fig. 2).

The results of the determinations of oxalates in 30 infusions from instant and ground coffees are presented in Table 4 and Figure 3.

Among the analyzed products of significantly ($p \leq 0.01$) the highest average level of oxalates was found in instant coffees from the species Arabica and Robusta beans - Nescafe Espiro (Tab. 4).

In the infusions from 3 different instant coffees the largest concentration of oxalates was found in Nescafe Espiro - 51.80 mg/100 cm^3 corresponding to 863.33 mg/100g dry matter, while in the remaining instant coffees the concentrations were about 18,58±7,05% lower. In *Sperkowska* and *Bazylak* studies [18] the concentration of oxalates in different instant coffees reached average levels from 90.90 mg/100 cm^3 (15.15 mg/g dry matter) to 165.60 mg/100cm^3 (27.60 mg/g dry matter). which does not correspond to the results of our studies showing much lower levels of oxalates in the instant coffees. This discrepancy could be explained by the use of modified analytical method (5-fold higher concentration of calcium chloride, 10-fold lower concentration of potassium permanganate and smaller sample).

In the infusions prepared from ground coffees the lowest oxalates concentrations were in Tchibo Exclusive, as compared to Maxwell House and Gala (ulubiona) coffees. The oxalates content in ground coffees is comparable in Gala and Maxwell House and lower in Tchibo Exclusive than reported by *Sperkowska* and *Bazylak* [18].

It is difficult to compare the studies presenting the results of the soluble oxalates analyses in the instant and ground coffees with our results because of their apparent divergence. The result of determinations of oxalates content in teas and coffees is influenced by the various analytical methods of different sensitivity used, as well as processes related to acquisition followed by burning, grinding and mixing procedures, extraction conditions and the initial mass of the sample.

In conducted in the Sixties *Zarembski* and *Hodkinson's* [25] studies on oxalic acid in the English diets the oxalates were at the level of 57.00 mg in the infusion of instant coffee "Nescafe" from the Arabica beans which was prepared from 2 g sample soaked for 5 minutes with 100 cm^3 water at 40°C. Capillary electrophoresis method was used by *Galli* and *Barbas* [7] for analysis of short-chain organic acids, *inter alia* oxalic acid. The analytical samples were prepared from 1 g sample of ground coffee and extracted for 10 minutes with 10 cm^3 of water at temperature 20°C. These authors found 0.256 mg of oxalates per 1 g of dry matter. *Honow* and *Hesse* [12] compared methods of extraction for the determination of soluble oxalates content in food, using high performance liquid chromatography. They proved that in the infusion prepared from 30 g of roughly grounded beans brewed in 1 liter of water at temperature 70°C the concentration of the analyzed compounds was l0.6 mg/100 cm^3 of infusion.

In this study the instant coffees had significantly ($p \leq 0.01$) higher oxalates content equal to 46.45 mg/100 cm^3 (774.00 mg/100 g dry matter) as compared to the infusions made from ground coffees - 31.78 mg/100 cm^3 (529.67 mg/100 g dry matter) (Fig. 3). It should be emphasized that in *Sperkowska* and *Bazylak* study [18] similar relations were noted. Ground coffees were characterized by lower oxalates concentrations within limits 42.30-56.35 mg/100 cm^3 (7.05-9.39 mg/100 g dry matter), as compared to the infusions from instant coffees 90.90-165.60 mg/100 cm^3 (15.15-27.60 mg/g dry matter of the product). The results of this study may support conscious choice of teas and coffees with lower oxalates content.

CONCLUSIONS

1. Results of this study revealed significant differences concerning the relation between phytochemical com-

position of teas and coffees and the levels of soluble oxalates in infusions prepared from these products.
2. The higher oxalates contents were found in black teas, while lower contents were noticed in red and green teas. In the infusions from tea bags a significantly ($p \leq 0.01$) lower oxalates content was found, as compared to the infusions obtained from leafy teas.
3. Regardless the kind of analyzed teas the significantly ($p \leq 0.01$) higher oxalates contents were in the infusions from pure one-compound teas ("Sir Roger"), while the lowest concentrations were found in the tea containing components from other plants ("Bio--Active" with grapefruit juice).
4. Instant coffees contained significantly ($p \leq 0.01$) higher oxalates content than natural ground coffees. The significantly ($p \leq 0.01$) higher concentrations of oxalates were in the Nescafe Espiro coffee, while the lowest in Tchibo Exclusive, Gala (*ulubiona*) and Maxwell House.

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