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Risk assessment of rare earth elements, antimony, barium, boron, lithium, tellurium, thallium and vanadium in teas

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Abstract

In recent years, a great intensification in the use of various elements especially in modern technology can be observed. However, the anthropogenic activities, including industrialisation, urbanisation or intensive agriculture, have led to the release of many of the elements into the environment. The consequence of the accumulation of the elements both in soil and water systems is their presence in the food chain. Inhalation and consumption of the contaminated food and beverages have been indicated as the main pathways of the exposure to many elements. Due to the fact, that tea is considered the second most popular beverage worldwide and its consumption is constantly increasing, it is crucial to evaluate the safety of the product, especially for toxic elements contamination. Thus, the aim of the project was to evaluate the contamination levels of rare earth elements (REEs) including lanthanides, scandium (Sc) and yttrium (Y) and also antimony (Sb), barium (Ba), boron (B), lithium (Li), tellurium (Te), thallium (Tl) and vanadium (V) in teas. Subsequently, the risk assessment was carried out. Additionally, the Fellowship provided hands-on training on the evaluation of applications of new biocides and participation in the science-based advises given to the Danish Food and Veterinary Administration, Danish Environment Protection Agency and Danish Medical Agency.

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1. Introduction

In the last few decades, there has been an intensive increase in technological development, which has involved the use of many chemical elements. Anthropogenic activities, including industrialisation, urbanisation or intensive agriculture, have already altered the natural occurrence state of many of the elements (Fedele et al., 2008). Release to the environment, and subsequent accumulation in soil, water and organisms has consequently led to the presence of many potentially toxic elements in the food chain, which may affect animal and human health.

Until now, some of the elements including lead (Pb), cadmium (Cd), mercury (Hg), arsenic (As) or aluminium (Al) have been widely known and studied, especially as food contaminants (Pearson and Ashmore, 2020). However, it can be expected that humans, through consumption of various foods can be exposed to several other elements, which may reveal toxic potential. Still, there is a wide group of elements that has been poorly studied, both toxicologically and as food contaminants. Especially, little is known about the dietary exposure to a group of elements called rare earth elements (REEs).

REEs include 17 elements out of which 15 are named lanthanides, including lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), promethium (Pm), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb) and lutetium (Lu). Two additional elements have been classified as REEs, namely yttrium (Y) and scandium (Sc) (Gwenzi et al., 2018). REEs are chemically uniform with similar physical and chemical properties. Due to their specific physical properties, they found a broad application in the modern technology e.g. catalysis, electronics, mobile communication, LED light bulbs, wind turbines, electric cars, fuel cells and fuel additives (Gwenzi et al., 2018; Squadrone et al., 2018; Doulgeridou et al., 2020). REEs has also been used in the production of fertilisers, especially in China (Wang et al., 2003; Gwenzi et al., 2018; Squadrone et al., 2018; Doulgeridou et al., 2020), and it was estimated that 5200 tons of REE-enriched fertilisers, used as growth promoters, were released into the cultivated soil in China only in 2002 (Li et al., 2013). Due to the broad use of REEs and a potential release to the environment, they began to be perceived as emerging contaminants (Squadrone et al., 2018).

Similarly to REEs, lithium (Li) has numerous industrial and commercial applications e.g. as catalyst of chemical reactors, component of batteries or as sanitising agent for swimming pools, hot tubs and spas (EPA, 2008). Some additional elements including tellurium (Te) and thallium (Tl), despite their high toxicity, are also widely used. Thallium is used in semiconductor materials, photocells, infrared measuring devices and as a catalyst in various organic synthesis procedures. In some countries, it is also used for the production of pesticides (Willner et al., 2021). Tellurium is mainly applied in the production of cadmium telluride thin-film solar cells, followed by thermo-electrics (Willner et al., 2021). In the case of barium (Ba), it has been already evaluated that the industrial (e.g. petroleum industry, steel industry, production of semiconductors) and medicinal application more than doubled during the last 40 years (Kravchenko et al., 2014).

Industrial intensification and increasing presence of electronic waste worldwide are becoming a paramount problem leading to contamination of soil and water. It can be therefore expected that elevated concentrations of many elements will be present in various plants and consequently contribute to an increased consumer exposure to REEs.

Due to the fact that tea is considered the second most popular beverage worldwide (Wang et al., 2020) and its consumption is constantly increasing (Vieux et al., 2019), it is crucial to evaluate the safety of the product, especially for toxic elements contamination.

Aside of the essential trace elements such as potassium (K), manganese (Mn), selenium (Se), zinc (Zn), strontium (Sr) and copper (Cu), teas can contain various chemical elements that can be harmful and which cannot be eliminated while processing or tea infusions preparation (Zhang et al., 2018). Depending on tea origin, accumulation of various elements can occur naturally or result from manufacturing and agronomic processes (de Oliveira et al., 2018).

As tea has the ability to accumulate REEs in a higher degree than other major food crops (Wang et al., 2020), and fertilisers containing REEs have been also used for tea production (Wang et al., 2003, 2020), it can be expected that tea may contain elevated concentrations of REEs compared to other plants (Wang et al., 2020). As many other elements can also be present in tea plants, besides REEs, a set of elements including antimony (Sb), barium, boron (B), lithium, tellurium, thallium and vanadium (V) was also investigated.

2. Description of work programme

2.1. Aims

The aim of the project was to estimate the exposure of the adult Danish population to REEs and other selected elements, including Sb, B, Ba, Li, Te, Tl, V resulting from tea consumption. Finally, for each element, the risk assessment was performed. In the case of REEs, due to their similar properties and lack of toxicological data on all of the elements, the exposure and risk assessments were carried out for the sum of the analysed elements.

2.2. Activities/Methods

2.2.1. First part included a literature search (PubMed, Scopus, Science Direct) for the elements, besides REEs that could be incorporated into the scope of the project. The next step regarded identification of the most important dietary sources of REEs and other elements included in the study.

2.2.2. Analysis of a selection of dry teas (black, green – *Camellia sinensis*, and rooibos – *Aspalathus linearis*) for their content of REEs, Sb, B, Ba, Li, Ta, Tl and V with inductively coupled plasma triple quad mass spectrometry (TQ ICP-MS, Thermo Scientific). Samples of teas were grinded and homogenised before the analysis, and subsequently, a test portion (0,3 g) was subjected to acidic digestion with the use of microwave oven. Afterwards, samples were analysed by iCAP™ TQ ICP-MS with Standard, Kinetic Energy Discrimination and Oxygen Reaction modes. The quality of the analytical methods was assured by simultaneous analysis of a certified reference material for REEs (BCR-670), and adherence to European standard methods EN 13805:2014 and EN 15763:2009.

Since teas are consumed as an infusion, to evaluate the real ingestion of the investigated elements, the transfer rates of the elements to the infusion were also measured.

2.2.3. Exposure assessment combined data on REEs from the present study with consumption data from a consumer survey among Danish citizens. Only adult (> 18 years old) consumers were included. A consumer was defined as a person consuming min. one cup of tea per week. The estimated average consumption was 350 mL per day, and the high consumption (1,084 mL per day) was represented by 95th percentile (P95). For the purpose of risk assessment, an average body weight of 70 kg was adopted. Exposure was estimated using the average concentrations corrected with the transfer rates and the average consumption (scenario 1). However, as regards tea, one can expect the phenomenon called 'brand loyalty', which in the case of high contamination of the product can lead to long-term exposure to elevated concentrations of the hazards, additional exposure scenarios were taken into consideration. One, representing the case of the average consumption with exposure to high concentration (P95) (scenario 2), and the other, representing high consumption (P95) and high contamination (P95) of the products (scenario 3).

2.2.4. Risk assessment of the dietary exposure to REEs, Sb, B, Ba, Li, Ta, Tl and V resulting from tea consumption by Danish consumers.

3. Conclusions

3.1. Risk assessment

Rare Earth Elements: The group of REEs in the study consisted of 16 elements (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Gd, Tb, Dy, Tm, Yb, Y, Sc). A key toxicological characteristic of the REEs is their common ability to displace calcium from calcium-binding sites in living systems, resulting in enzyme inhibition or other biochemical dysfunctions (Palasz and Czekaj, 2000). Some other potential human health effects include dysfunctional neurological disorders e.g. reduced intelligence quotient (IQ) in children, associated mainly with La (Gwenzi et al., 2018; Wang et al., 2019) and bone alteration (Zaichick et al., 2011). Additionally, genotoxicity and fibrotic tissue injury associated with several REEs were observed (Gwenzi et al., 2018). In the case of La, multiple adverse effects on various organs including liver, kidney and lung as well as the nervous system of animals were reported. The effects were related to the oxidative stress, disturbance of the homeostasis of essential elements and enzymes as well as histopathological changes (Liu et al., 2010).

Since the interest in REEs is only recently increasing, there is no sufficient toxicological data on all individual elements. Consequently, there are no health-based guidance values for REEs derived by any of the authorities. For the purpose of risk assessment, we have adopted the values from toxicity studies on some of the REEs.

Ninety-day studies conducted according to the guidance of the Organization for Economic Co-operation and Development (OECD) with the established no observed adverse effect levels (NOAEL) were available for La, Ce, Y. Based on the NOAELs, tolerable daily intake (TDI) values were derived for La, Ce and Y. For La, 51.3 $\mu\text{g}/\text{kg}$ body weight (bw) per day for a decreased body weight was derived (Fang et al., 2018). In the case of Ce, TDI was set at 161.5 $\mu\text{g}/\text{kg}$ bw per day from NOAEL for induced weight loss, decreased erythrocyte, albumin, total bilirubin and phosphocreatine kinase as well as increased leukocytes in animals (Wu et al., 2019). For yttrium, the TDI of 145.5 $\mu\text{g}/\text{kg}$ bw per day was proposed, based on NOAEL for multiple changes in mortality, clinical signs, daily food consumption and weekly body weights, urinalysis, haematology, blood coagulation, clinical biochemistry and histopathology of all the main organs/tissues except lung (Wang et al., 2017). The lowest TDI, derived for La of 51.3 $\mu\text{g}/\text{kg}$ bw per day, was adopted as the reference value for the sum of all REEs. The decision was based on the fact that La was one of the predominant elements in all analysed teas, as it was determined at one of the highest concentrations together with Ce and Y, thus had a major contribution to the overall contamination.

High uncertainty is associated with the adaptation of TDI derived for La, for the sum of REEs, as there is insufficient toxicological data on other elements. It is possible that other elements could have higher toxicity comparing to La, and adapted TDI could be insufficiently protective, and risk could have been underestimated.

The evaluated exposure to REEs (Table A.1, Appendix A) ranged from 7.57 to 58.06 ng/kg bw, depending on the tea type. However, as the highest exposure would constitute only 0.1% of the TDI, it can be said that the risk of adverse effects caused by REEs is rather not expected from the prolonged ingestion of teas.

Antimony: In the case of Sb, oral exposure predominantly affects the gastrointestinal system resulting with burning stomach pains, colic, nausea and vomiting (Sundar and Chakravarty, 2010). The health effects observed in animals orally exposed to higher doses of Sb included hepatocellular vacuolisation, haematological alterations such as decreases in red blood cell counts and haemoglobin levels and histological alterations in the thyroid (Atsdr, 2019). For Sb, the suggested NOAEL in the subchronic drinking water study in rats was established as 6.0 mg/kg bw per day based on decreased body weight gain and reduced food and water intake. By the application of an uncertainty factor of 1,000 (100 for intra- and interspecies variation and 10 for the use of a subchronic study), a TDI of 6 $\mu\text{g}/\text{kg}$ bw was determined (WHO, 2003). Taking into the consideration the evaluated exposure to Sb, the daily intake from tea consumption would represent only 0.03% of a TDI, confirming the negligible risk to the health of the consumers.

Barium: Human and animal high-dose exposure to soluble Ba compounds results in a number of effects including electrocardiogram abnormalities, ventricular tachycardia, hypertension and/or and hypotension, muscle weakness and paralysis (SCHER, 2012). However, kidney effects are considered the most sensitive health effect associated with long-term ingestion of Ba (Kravchenko et al., 2014). Due to the marked severity of nephropathy, the lower confidence limit of the benchmark dose for a 5% response (BMDL_{05}) was selected over the typically 10% incidence as a point of departure. By applying an assessment factor of 300, a TDI of 0.2 mg/kg bw per day was derived (SCHER, 2012). In the case of the highest exposure resulting from black tea consumption, the TDI would be covered only in 0.3%, meaning that there is a negligible risk of the adverse health effect caused by Ba ingestion with tea.

Boron is not an essential nutrient for humans and any specific biochemical functions have not been identified. There is however, some evidence that, in humans, B may influence the metabolism and utilisation of other nutrients, especially calcium, and may have a beneficial effect on bone calcification and maintenance (EFSA, 2004a; Ziola-Frankowska et al., 2014). Symptoms related to B intoxication includes gastrointestinal disturbances, granular degeneration of tubular cells, exfoliate dermatitis, epilepsy, cardio-circulatory collapse. Congestion of the brain, hair loss, lethargy, anorexia and mental confusion were other identified effects (EFSA, 2006). The most sensitive endpoint of toxicity of B was, however, a developmental toxicity (Murray and Schlekot, 2004). A tolerable upper intake level (UL) was based on the decreased fetal body weight in rats resulting from maternal boron intake during pregnancy. The NOAEL for this effect (9.6 mg/kg bw per day) was extrapolated to humans by application of uncertainty factor of 60 (including intra- and interspecies variability) to give an UL of 0.16 mg/kg bw per day (EFSA, 2004a).

Consumption of rooibos tea would lead to the highest exposure to B compared to consumption of the other analysed teas. However, it would represent only 2.6% of the allowed UL. Based on the outcome, it can be said that the risk for the tea consumers related to the B would be very low.

Lithium is used as a treatment in the bipolar affective disorder; hence, most of the toxicological studies are based on clinical investigations of the patient subjected to Li treatment. The element has been identified as having an adverse renal effect, with the most common being nephrogenic diabetes insipidus. However, some additional adverse effects on thyroid function, primarily asymptomatic hypothyroidism have been observed in patients treated with Li (McKnight et al., 2012). In the case of lithium, the provisional subchronic and chronic reference dose (p-RfD) was derived from the lowest observed adverse effect level (LOAEL) of 2.1 mg/kg per day for adverse effects in several organs and systems. The LOAEL was divided by an assessment factor of 1,000, yielding a subchronic and chronic p-RfD of 2 µg/kg per day (EPA, 2008). The highest exposure from tea consumption was evaluated for rooibos. However, it would constitute only 8.14% of p-RfD, reflecting low risk to the health of the tea consumers.

Tellurium is an element with chemical properties resembling those of non-metals, such as sulfur, however, if the toxicity is concerned, the properties are closer to the effects caused by selenium (Health Council of the Netherlands, 2014). The clinical manifestation of the ingestion of substantial concentrations of Te includes vomiting, nausea, metallic taste, black discoloration of the oral mucosa and skin, corrosive gastrointestinal tract injury and a characteristic garlic-like odour of the breath (Vávrová et al., 2021). In long-term drinking water studies in rats and mice, no evidence of carcinogenic effects were found (Greim, 2005).

Concentrations of Te determined in teas were very low and often below the limit of quantification of the method; in some samples, the element was not determined (data not shown). Due to the absence of the elements or very low determined concentration, tellurium was not included in the exposure assessment and in the general risk assessment.

Thallium: In the case of thallium, it is known that its salts can cause a wide spectrum of adverse effects in humans and animals, and thallium is considered a cumulative poison (EPA, 2009). Acute thallium poisoning is usually accompanied by gastrointestinal symptoms, while neurological findings (sensory and motor changes) predominate in chronic exposure. Other symptoms include polyneuritis, encephalopathy, tachycardia and degenerative changes of the heart, liver and kidneys (Cvjetko et al., 2010). P-RfD values for thallium of 1×10^{-5} mg/kg per day (for hair follicle atrophy) were derived by Environmental Protection Agency (EPA) (EPA, 2009, 2012). Taking into account the highest estimated exposure of 1.48 ng/kg bw, resulting from drinking black tea, it can be said that the risk for the human health is low, as the exposure covers 14.8% of the p-RfD.

Vanadium has not been shown to be essential for humans or possess any nutritional value. The most common non-occupational sources of vanadium exposure are contaminated food and drinking water (Rodríguez-Mercado et al., 2011; Crebelli and Leopardi, 2012). High concentrations of V may cause irreversible damage to the kidneys (EFSA, 2004b). However, vanadium in mammalian species can accumulate in the liver, kidneys, bones, lungs and spleen (Rodríguez-Mercado et al., 2011; Crebelli and Leopardi, 2012). Vanadium compounds may initiate some gastrointestinal problems such as diarrhoea, vomiting, general dehydration with weight reduction, intestinal inflammation and a characteristic green tongue (Wilk et al. 2017). In the case of V, the reference dose (RfD) of 7 µg/kg bw per day was derived by the EPA. The dose was based on gastrointestinal disturbance (intestinal cramping and diarrhoea) observed in human studies (EPA, 2006). The exposure to V through consumption of teas is low, ranging from 0.1 to 2.6 ng/kg bw, contributing only to 0.04% of RfD in the case of the highest exposure. Thus, the risk of adverse effects is not expected from the prolonged ingestion of teas.

Due to the lack of substantial information on the toxicity of most of the elements from the REEs group, the related uncertainty should be evaluated as high. Some of the REEs may reveal higher toxicity than this established for lanthanum. Thus, the applied TDI would not be enough protective. Consequently, the assessment would lead to the underestimation of the risk. However, due to the fact that lanthanum, cerium and yttrium constituted 60% of total REEs contamination, the contribution from other elements is significantly lower, and performed risk assessment should provide a reliable outcome. An additional factor contributing to the uncertainty of the assessment is the fact that some of the consumers can brew tea longer than 3 min that was used in the transfer rate study. Therefore, higher rates of the elements can be leached into infusions and thus contribute to higher exposure. As the exposure to most of the elements was contributing to a small per cent of the tolerable daily intakes, the whole uncertainty of the risk assessment could be evaluated as low, and moderate in the case of REEs.

3.2. Final conclusions

The analysis of tea samples from the Danish market for the determination of REEs, Sb, B, Ba, Li, Te, Tl, V was carried out. The aim of the analysis was to determine the contamination levels and subsequently evaluate the risk related to the exposure of adult consumers of tea to these elements. The risk assessment revealed that exposure to all investigated elements through consumption of tea poses a negligible risk to the consumers and no adverse effects are expected even for high consumers.

Overall, the work programme allowed the fellow to gain knowledge and practical skills on risk assessment. Additionally, the fellow gained a practical knowledge on ICP-MS analysis and sample preparation for elemental analysis. Results of the project were presented as a EUROTOX conference poster and are planned to be published as a scientific paper in a peer-reviewed scientific journal.

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Abbreviations

BMDL ₀₅	Lower confidence limit of the benchmark dose for a 5% response
Bw	Body weight
DTU	Technical University of Denmark
EPA	Environmental Protection Agency
ICP-MS	Inductively coupled plasma mass spectrometry
LOAEL	Lowest observed adverse effect level
NOAEL	No observed adverse effect level
p-RfD	Provisional reference dose
REEs	Rare earth elements
RfD	Reference dose
TDI	Tolerable daily intake
UL	Tolerable upper intake level

Appendix A – Exposure assessment

Table A.1: Estimated exposure to the investigated elements resulting from the consumption of black, green and rooibos tea, expressed in ng/kg bw per day

	REEs	B	Ba	Li	Sb	Tl	V
Black tea							
Scenario 1	9	482	132	2	0.4	0.2	0.1
Scenario 2	18	578	196	5	0.4	0.5	0.3
Scenario 3	56	1,789	606	16	1	1.5	0.8
Green tea							
Scenario 1	8	636	109	2	0.3	0.03	0.5
Scenario 2	9	714	133	5	0.5	0.6	0.8
Scenario 3	27	2,211	412	14	2	0.2	2.5
Rooibos tea							
Scenario 1	15	991	44	43	0.05	0.01	0.6
Scenario 2	19	1,345	64	53	0.07	0.02	0.8
Scenario 3	58	4,167	199	163	0.2	0.04	2.6

Appendix B – Secondary activities

- 1) Webinar on ICP-MS. 'Heavy Metals in Baby Food' (28 April 2021).
- 2) Participation in 9th BfR-Summer Academy 2021: Lecture Series on Risk analysis in Food safety (23–27 August 2021).
- 3) Participation in the postgraduate course 'Risk Analysis in Food Safety' consisting of two main modules: first focusing on microbiological, and a second on chemical risk assessment. Each module consisted of 12 submodules, including case studies intended to the elaboration of a risk assessment on a specific microbiological/chemical hazard, finalised with the preparation of the reports and poster presentations (31 August to 3 December 2021).
- 4) Participation with a poster presentation in the 56th Congress of the European Society of Toxicology – EUROTOX 2021. The poster presentation: 'Rare earth element as the emerging contaminants in black tea – risk assessment resulting from the dietary exposure' was related to the project on risk assessment carried out by the fellow at DTU (27 September to 1 October 2021).
- 5) Webinar on the application procedure for active substances in pesticides and maximum residue levels (28 October 2021).
- 6) Hands-on training on the evaluation of applications and requests related to biocides products, mainly destined to be used as disinfectant/cleaning agents.
- 7) Taking part in advice-giving to the Danish Food and Veterinary Administration, the Danish Environment Protection Agency and the Danish Medical Agency.
- 8) Participation in division meetings.