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DTU Food
National Food Institute

Levels of perchlorate and chlorate in foods available in Denmark

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Background:

Sources of both perchlorate and chlorate in food may be multiple though chlorinated water used for irrigation/washing/blanching/ disinfection is meant to be an important and primary source of perchlorate and chlorate in foods, respectively. In Denmark 100% of the water supply is covered by groundwater which is only filtered and aired (thus no chlorination). Consequently the risk of water being a source of perchlorate and chlorate is low. This makes Denmark unique among European countries (Fig. 1).

The **aim** of this study was to gain data on the occurrence of perchlorate and chlorate in products of Danish origin.

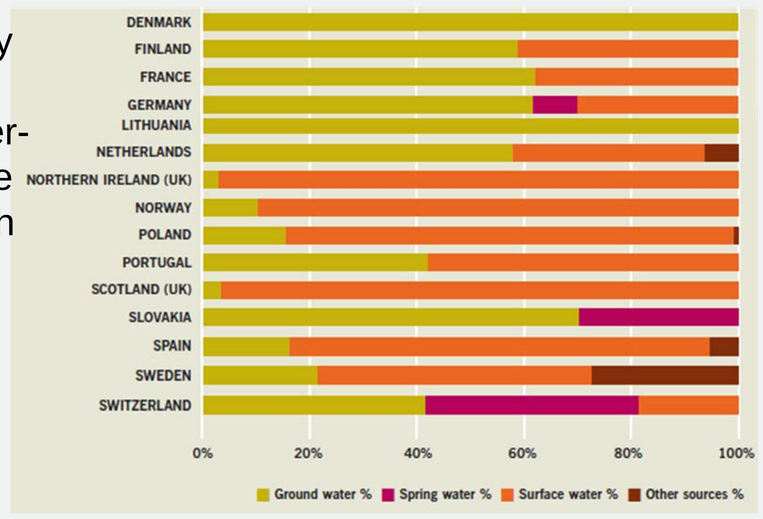

Figure 1: Sources of water covering the supply in varies EU countries (1).

Results:

High risk commodities were collected and analysed in 2014-2017. A total of 119 (89 domestic, 29 foreign) were analysed for perchlorate (sampling period 2014-2017). Additionally, 77 of the samples from 2016-2017 (48 domestic, 29 foreign) were analysed for chlorate (LOQ 0.01 mg/kg).

Figure 2 illustrate that the frequency with which perchlorate and chlorate were found (≥0.01 mg/kg) generally were higher among non-domestic samples than among domestic samples. The number of foreign samples were low but the frequency of positive perchlorate findings are in line with the findings of e.g. Arcella et al. 2017(2) and Vejdovszky et al. 2018(3) (Table 1). Roughly 40% of the foreign samples in the present study were found to contain chlorate (≥0.01 mg/kg) compared to roughly 14% of domestic samples. The high frequency of chlorate in foreign samples are in line with findings presented in EFSA Journal 2015;13(6):4135. No clear difference in the levels perchlorate or chlorate found in domestic vs non-domestic samples are indicated (Fig. 3).

Conclusion:

The presented results strongly indicate that the frequency with which perchlorate and chlorate are found in commodities of Danish origin generally is lower than in commodities of foreign origin. This may be related to the fact that the water supply in Denmark is based 100% on non-chlorinated groundwater.

Extraction by QuPPe method:

- 10 gram homogenous sample
- Add 10 ml methanol w. 1% formic acid
- Add ILIS ($^{18}O_3$ Chlorate and $^{18}O_4$ Perchlorate)
- Add ceramic homogeniser and shake 1 min.
- Centrifuge (5 min at 4500 *g)*
- Dilute the extract w. eluent A (1:1 or more to reduce matrix interferences) and filter
- LC(ESI neg)-MS/MS w. Hypercarb

Instrument setup:

- Analysis on Water/Bruker LC(ESI)-MSMS
- Chromatographic separation on a Hypercarb column (100x2.1, 5 µm)
- Column primed w. multiple injections of spinach extract
- Injection volume 5 µl, flow rate 0.4 ml/min
- To avoid source contamination resulting in decreasing **sensitivity, water** was injected between each analysis (Waters system) or source exhaust was turned on (Bruker)
- LOQ: 0.01 mg/kg for both perchlorate and chlorate

LC gradient programme

Time (min)	Eluent A (%) (water w. 1% acetic acid)	Eluent B (%) (methanol w. 1% acetic acid)
0	95	5
14	95	5
18	100	0
19	50	50
23	50	50

	MRM transistion (quant, qual)	
Compound	**Quantifier**	**Qualifier**
Chlorate	83.00 > 67.00	85.00 > 69.00
Chlorate, $^{18}O_3$	89.00 > 71.00	
Perchlorate	99.00 > 83.00	101.00 > 85.00
Perchlorate, $^{18}O_4$	107.00 > 89.00	

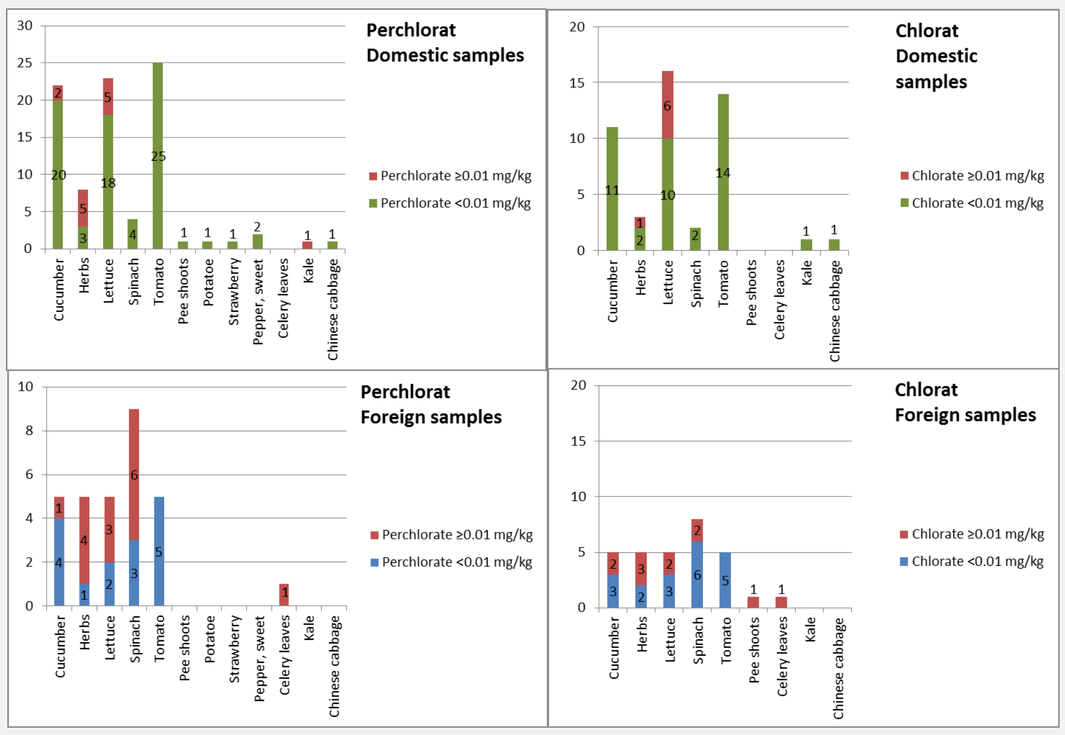

Figure 2: Number of samples with residues of perchlorate and chlorate ≥0.01 mg/kg and <0.01 mg/kg. Sampling were performed in 2014-2017 for perchlorate and in 2016-2017 for chlorate.

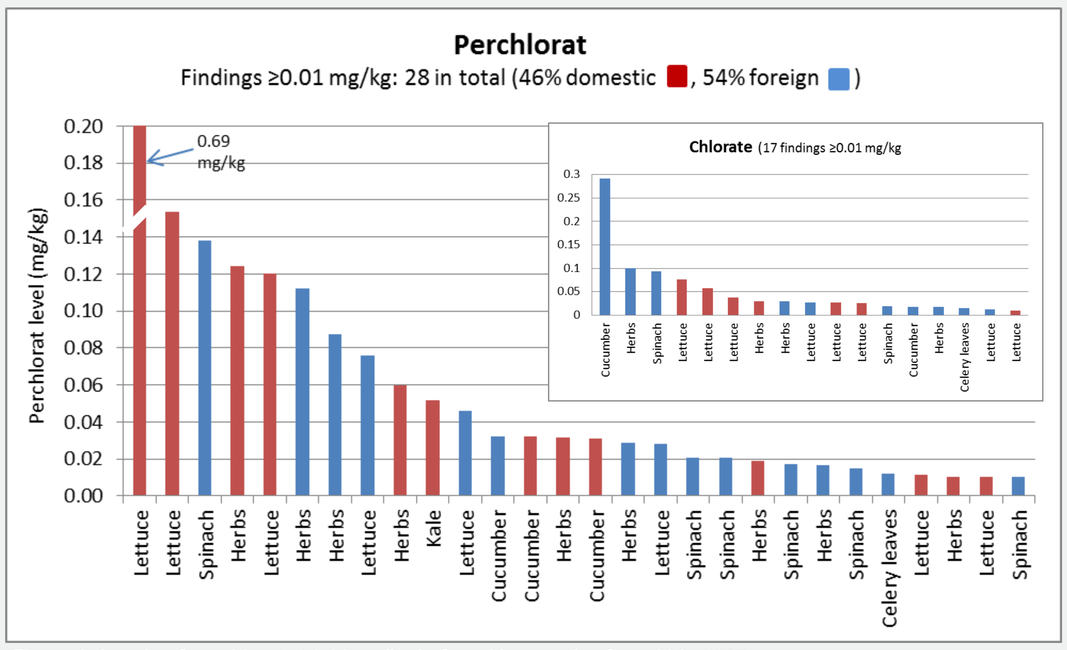

Figure 3: Levels of perchlorate ≥0.01 mg/kg in found in samples from 2014-2017.

Table 1: Findings of perchlorate in commodities of which more than two samples have been analysed.

	Perchlorate in samples of Danish origin			Perchlorate in samples originating from other EU countries			
Commodity	No. samples analysed	Positive findings ≥LOQ (%)	Average of positive findings (mg/kg)	No. samples analysed	Positive findings ≥LOQ (%)	Average of positive findings (mg/kg)	Positive findings reported by others (%)
Cucumber	22	9%	0.03	5	20%	0.03	37% (2)
Herbs	8	63%	0.05	5	80%	0.06	60% (2)
Lettuce	23	22%	0.20 (0.06)*	5	60%	0.05	42%^ (3)
Spinach	4	0%	0.00	9	67%	0.04	51% (2)
Tomato	25	0%	0.00	5	0%	0.00	7% (3)

(1): Iwa, 2010. International Statistics for Water Services. Water Supply, p.20.
(2): Arcella, D., Eskola, M. & Gómez Ruiz, J.A., 2016. EFSA Journal, 14(12). https://www.efsa.europa.eu/en/efsajournal/pub/5043.
(3): Vejdovszky, K. et al., 2018. Food Addit Contam Part A, Chemistry, Analysis, Control, Exposure and Risk Assessment, 35(4), pp.623–631. https://doi.org/10.1080/19440049.2018.1426889.
* Value if the finding of 0.69, which was exceptionally high, is excluded.
^ Leaf vegetables (Lettuce, rocket, field salad, mixed leaf salad)