Chemical Analysis of Seagreens® Arctic Wild Wrack Seaweed

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FOOD INNOVATION

Executive Summary

- No organotin, PCBs or aromatic hydrocarbons present in certified reference standards could be detected in the three samples analysed.
- Iodine was detected at levels of 375 in pure arctic wild rack and 25 in chipolata plus arctic wild wrack
- No heavy metal contaminants were detected in the samples tested.

From all the tests carried out, we see no reason that the product[1] would not be safe to consume within the regulatory definition of food safety for human consumption. The product has satisfied all the requirements as regards determination of regularly examined toxins and shown that they are absent. Based on these analyses, there is no evidence to suggest that the consumption in the specified amounts would cause any detrimental health effects.

[1] **Certified analysis for contaminants, toxic metals and microbial pathogens in Seagreens® Ascophyllum nodosum (knottedwrack), Fucus spiralis (spiral wrack), and Pelvetia canaliculata (channel wrack).**

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Analysis

Analysis was carried out on arctic wild wrack, pork chipolata and pork chipolata plus wild wrack. All certified reference standards were obtained from LGC Promochem (UK) and all other chemicals were obtained from Sigma-Aldrich (UK). All analysis was carried out on triplicate samples, with duplicate GC-MS or GC analysis of each replicate.

Element Analysis

The concentration of the following elements was determined using inductively coupled plasma - optical emission spectrometry (ICP-OES):

Arsenic
Cadmium
Calcium
Copper
Iron
Lead
Magnesium
Mercury
Potassium
Sodium

Samples were acid digested by adding 1g of sample to 9ml of 2% HNO_3 plus 1 ml of H_2O_2. Samples were microwaved at 360 W and 20 psi for 6 minutes and then cooled for 5 minutes. This process was then repeated.

Samples were then analysed using ICP-OES and values determined based on calibration curves created using certified reference standards. The limits of detection for all elements were less than 1 µg/ml (~1ppm). ICP analysis was carried out in duplicate, from triplicate samples.

	Ca	Cd	Cu	Fe	Hg	K	Mg	Na	Pb	As
Wild Wrack	17841	1.8	BLD	345.66	BLD	20100.6	9636	35100	BLD	BLD
Chipolata plus Wild Wrack	372.885	0.6	BLD	8.1	BLD	2265.6	285.15	2835	BLD	BLD
Chipolata	165.3	0.9	BLD	3.15	BLD	2057.25	202.185	2606.25	BLD	BLD

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All results are in µg/g of sample. BLD = below limits of detection

The results show that Cadmium, Copper, Mercury and Lead are negligible in both the wild wrack and Chipolata products. Calcium, Potassium and Magnesium are present in high quantities in the Seaweed and the so the Chipolata products with the wild wrack in have higher levels compared to the Chipolatas without wild wrack. The wild wrack contains iron but this seems to have only carried over into Chipolata samples 1 and 2. Also the sodium levels in the wild wrack are high but the Sodium levels in all the chipolatas are similar.

Iodine Analysis

Iodine is difficult to analyse using ICP-OES and so a spectrophotomic absorbance analysis was carried out. Briefly 2 g of sample was freeze dried and then ground to a fine powder under liquid nitrogen. Ground samples were then dissolved in 50 ml sterile distilled water and the solid matter allowed to settle. 4 ml of supernatant was removed and added to 1 ml of KSCN, 1 ml of water and 1 ml of $NH_4Fe(SO_4)_2$

After 90 s 1 ml of sodium nitrite solution was added and absorbance read at 450 nm.

Valued were read against a calibration curve to determine iodine concentration.

	Iodine concentration (µg/g)
Arctic Wrack	375
Chipolata plus Arctic Wrack	25
Chipolata	Below limits of detection

Organotins

10 g of sample was freeze dried then ground to a fine powder under liquid nitrogen. 25 ml of sterile distilled water was added followed by 0.25 ml of acetic acid/acetate buffer solution (5 M, pH 5) and 0.9 g of sodium chloride. The samples were shaken briefly and divided into two. One half was spiked with 100 µl of standard solution mixture. If necessary, the pH was adjusted to 5.0 with acetic acid or 0.1 M NaOH. The samples were again shaken prior to addition of 150 µl of a freshly prepared 1.5% (w/v) $NaBEt_4$ aqueous solution. Samples were then incubated at room temperature for 2 hours.

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2 ml of hexane was then added, samples tubes were sealed and shaken overnight at 25 ºC. Samples were allowed to settle and the hexane layer removed. Samples were then analysed using GC-MS.

A calibration curve was created using certified standards diluted in methanol. Limits of detection were determined to be between 4 and 8 ng/g for the following organotin molecules.

Monobutyltin trichloride

Di-n-butyltin dichloride

Tributyltin chloride

Tetrabutyltin

Monooctyltin trichloride

Dioctyltin dichloride

Triphenyltin chloride

Tricyclohexyltin chloride

All organotins could be detected using GC-MS from spiked samples. No organotins were detected in arctic wrack, chipolata, or chipolata plus arctic wrack samples.

Polychlorinated biphenyls

10 g of freeze dried and ground sample were extracted in dichloromethane, the extract was then treated with sulphuric acid. Samples were incubated at room temperature for 2 hours after which the supernatant was removed and allowed to evaporate under nitrogen. Residues were re-suspended in 1 ml hexane and analysed using GC-MS

This process was then repeated with samples spiked with 10 µl of certified standard solution containing the following PCBs

2-chlorobiphenyl
3,3-dichlorobibiphenyl
2,4,5-trichlorobiphenyl
2,2,4,4-tetrachlorobiphenyl
2,3,4,5,6-pentachlorobiphenyl
2,2,3,3,6,6-hexachlorobiphenyl

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2,2,3,4,5,5,6-heptachlorobiphenyl
2,2,3,3,4,4,5,5-octachlorobiphenyl
2,2,3,3,4,4,5,5,6-nonachlorobiphenyl
decachlorobiphenyl

All ten PCBs were recovered from spiked samples. No PCBs were detected in arctic wrack, chipolata, or chipolata plus arctic wrack samples.

Aromatic Hydrocarbons

1 g of freeze dried and ground sample was added to 20 ml of dichloromethane and sonicated on ice for 30 min. The extracts were then filtered and concentrated under nitrogen to 200 µl. This process was repeated with samples spiked with certified standards containing the following aromatic hydrocarbons.

Benzene
Toluene
o-Xylene
m-Xylene
p-Xylene
Ethylbenzene
Propylbenzene
Cumene
Butylbenzene
Isobutylbenzene
sec-Butylbenzene
tert-Butylbenzene
p-Cymene
1,2,4-Trimethylbenzene
Mesitylene
p-Diisopropylbenzene
Styrene
alpha-Methylstyrene
beta-Methylstyrene
1,2,4,5-Tetramethylbenzene (2% w/v in p-Xylene)

Concentrated samples were analysed using gas chromatography, with reference peaks created using the certified reference samples. All aromatics were successfully analysed with the exception of butylbenzene and isobutylbenzene. Again none of the molecules present in the reference standards were detected in arctic wrack, chipolata, or chipolata plus arctic wrack samples.

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