Development of an analytical strategy for the field dissolution of targeted species in country foods

Audrey Laberge-Carignan[1], Dominic Larivière[1], Pierre Ayotte[2]

1. Radioecology laboratory, Chemistry department, Laval University; 2. Department of Social and Preventive Medicine, Laval University

Introduction

Country foods play an important role in northern communities diet. These foods include caribou, beluga, birds and fish such as Arctic char. They are rich of essential nutrients, but may also contain levels of contaminants that can pose a health risk to northern communities[1].

Lead (Pb), mercury (Hg) and methylmercury (MeHg) can be found in measurable concentrations in animal flesh. Hg is a toxic heavy metal, that bioaccumulated and biomagnified at the top of the food chain. This implies that its concentration increases for each ascending link in the food chain[1]. Hg has an atmospheric residence time of 0.5 to 1 years greatly facilitate its dispersion. The greater presence of atmospheric oxidants such as halogen radicals (Br •) in northern regions causes the oxidation of Hg (0) to Hg (II) which become deposited[2]. Once deposited, the Hg can infiltrate into the soil and the water where it enters the food chain and can be transformed into MeHg. Thus, the possibility to measure on site the presence or absence contaminants in food could be attractive for population consuming country food. This project will address some scientific aspects associated with the on site monitoring of targeted species.

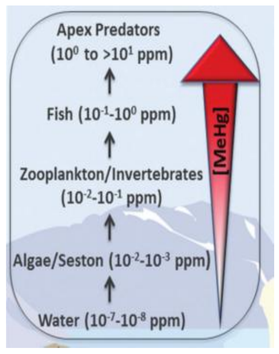

Figure 1: Accumulation of methylmercury (MeHg) in the northern environment[2]

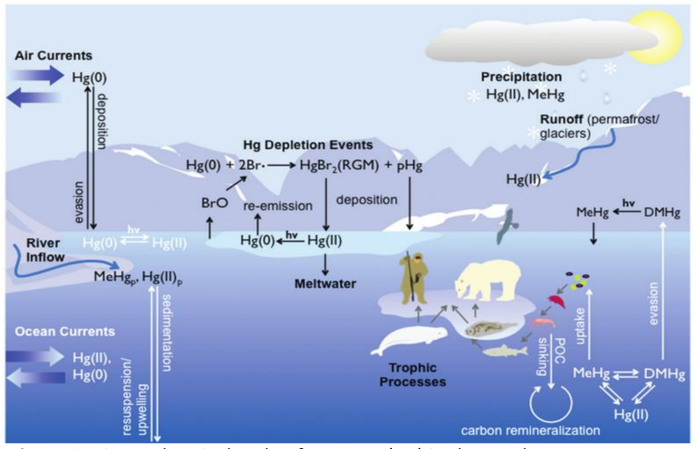

Figure 2: Biogeochemical cycle of mercury (Hg) in the northern environment [2]

Goals

To ensure the foods qualities, the goal of the project is to enabling tools for the monitoring of food quality in the Northern environment

- Development and validation of the dissolution, separation and preconcentration schemes of targeted species
- Designing the preliminary field-deployable instrument
- Development of robust optical detection techniques in microfluidic device
- Parallelization sample preparation and integration of analytical approahces in microfluidic platforms
- Designing the interface, the data mnaging systems and the knowldge user tools

Sample collection

Figure 3: Electron Microscopy Sciences (EMS) biopsy plunger[3]

Since the goal this project is to conduct field analysis, a simple and robust sampling method is required. An analytical scale works efficiently in the laboratory, but is ill-suited for field analysis. Biopsy punches allow the removal of a constant volume.

Table 1:
Sampling test of Arctic char with a biopsy plunger EMS 7mm

Parameters	mass (mg)	Absolute errors (%)
Fresh flesh of the same fish by a sampler (n = 20)	103	6
Fresh flesh of the same fish by different samplers (n = 40)	102	7
Fresh flesh of different fish of the same species (n = 30)	101	8
Refrigerated flesh of the same fish (n = 10)	99	11
Refrigerated flesh of the same fish by different samplers (n = 20)	138	15
Flesh of the same fish by different samplers the same day (n = 30)	107	19
Fresh flesh of different fish of different species(n=20)	106	19
Flesh of different fish of different species by different samplers at different days (n = 150)	118	26
Flesh of the same fish by different samplers at different days (n = 40)	120	37

The absolute errors obtained, although variable, remain acceptable within the framework of our project.

Dissolution

- Take fish tissues
- Add TMAH
- Heat
- Ultrasound
- Filter

Figure 4: Dissolution protocol

Acid or microwave dissolution are the most widely used methods in the laboratory, but they require large amounts of acid, high pressures and a significant amount of heat. These methods therefore hardly apply to field analysis. Alkaline dissolution using tetramethylammonium hydroxide (TMAH) and ultrasonic frequencies is a fast, simple technique that requires little energy and consumable, characteristics sought for field studies[4]. In order to achieve an effective dissolution in the shortest time possible, to optimize the dissolution process was performed.

Fish tissues dissolution optimisation

In order to be able to have an effective dissolution in a short time, it is necessary to optimize the dissolution process which depends on 5 parameters (Figure 5). The optimization of one parameter at a time would be very tedious and would neglect interactions between the different parameters. Experimental designs allow vary several parameters at the same time and to observe their influences using a statistical tool. The first experimental design performed showed that the mass-to-volume ratio and the heating are two parameters that have little impact on the recovery. The mass-to-volume ratio is set at 0.02 which represents 100 mg of flesh per 5 mL of solution. The heating is performed during the ultrasound treatment.

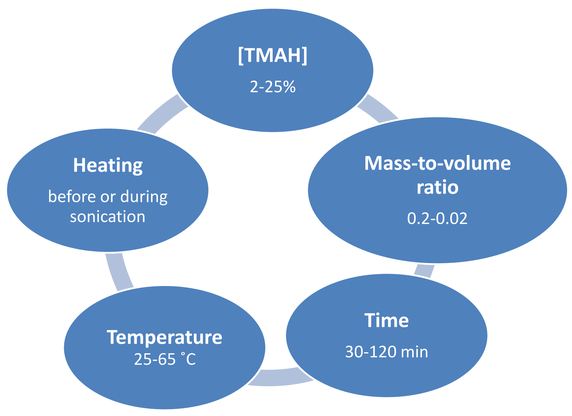

Figure 5: Parameters to optimize the flesh dissolution

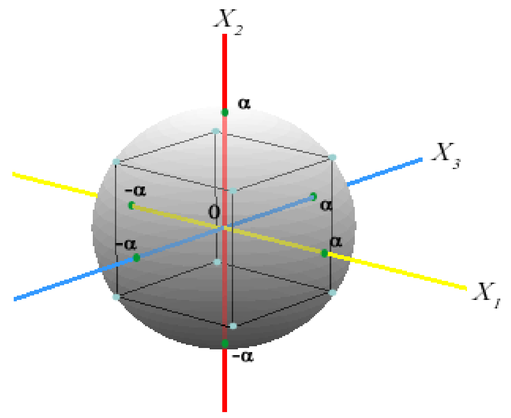

Figure 6: Central composite design[5]

Subsequent plans allowed optimization of time, concentration and temperature. Central composite design (Figure 6) were used to evaluate the system response near the optimum. The tables 2 and 3 show the influence of the various factors on the recovery of Pb and Hg during dissolution.

Table 2:
Response of the optimization of the dissolution parameters for Pb

Term	Prob>|t|
Time(min)*time (min)	0,0294
Temperature (°C)*time (min)	0,1012
Temperature (°C)(50,70)	0,1058
[TMAH] (%)*[TMAH] (%)	0,2075
Temperature (°C)*Temperature (°C)	0,2401
Temperature (°C)*[TMAH] (%)	0,5188
Time (min)*[TMAH] (%)	0,8723
[TMAH] (%)(9,19)	0,9211
Time (min) (5,15)	0,9560

Table 3:
Response of the optimization of the dissolution parameters for Hg

Term	Prob>|t|
Temperature (°C)(50,70)	0,0004
time (min)*time (min)	0,0196
[TMAH] (%)*[TMAH] (%)	0,0757
[TMAH] (%)(9,19)	0,1033
Temperature (°C)*[TMAH] (%)	0,1733
Temperature (°C)*Temperature (°C)	0,2966
Time(min)(5,15)	0,6244
Temperature (°C)*time (min)	0,6676
Time (min)*[TMAH] (%)	0,7946

Results: Recovery of Pb and Hg

Different experimental designs were made to obtain the preliminary results for recovery of Pb and Hg during the fish tissues dissolution.

[TMAH] : 10 %

time : 15 min

Temperature: 65 °C

Hg = 100 ± 6 %
Pb = 96 ± 11%

100 mg of tissues for 5 mL TMAH

Future works

- Evaluate the recovery of MeHg
- Validate dissolution method with certified fish tissues reference material
- Perform separation and preconcentration of Pb and Hg

Thanks

Sentinelle Nord Sentinel North

Project 3.4

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APOGÉE CANADA FONDS D'EXCELLENCE EN RECHERCHE

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