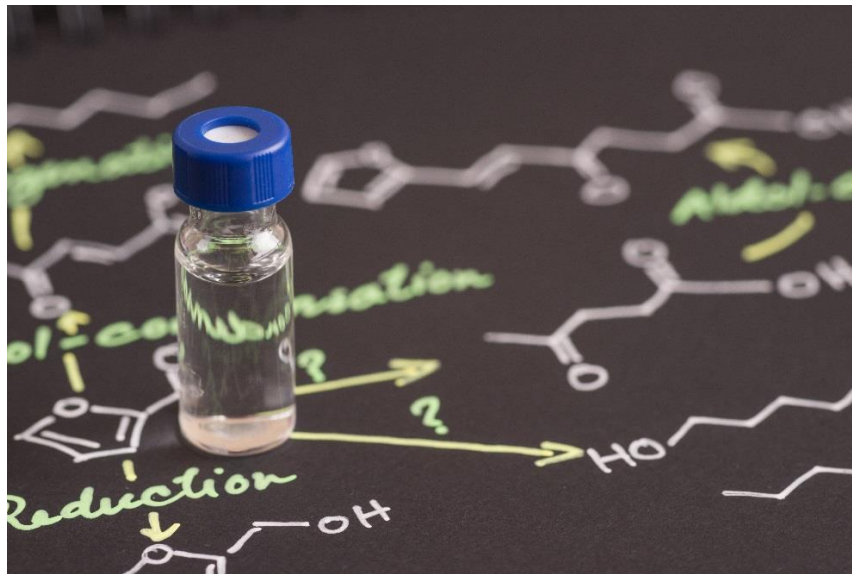


Technical bulletin 2: testing of new technologies - REIMS



Fish Fraud: Speciation, the Lipidomic approach

Globally, food fraud is estimated to cost \$40 billion with the seafood industry implicated. In a report from 2016, Oceana reviewed more than 200 published studies from 55 countries, and found that one in five of more than 25,000 samples of seafood tested worldwide was mislabelled. This included a study from the UK & Ireland which claimed that 44 of 226 samples (19.5%) were mislabelled. Processed products such as smoked and battered fish were found to be mislabelled more often i.e. in Ireland specifically, 28.2% (37 of 131) samples were mislabelled, higher than the UK level of mislabelling at 7.4%. This points to the need for rapid tests to determine speciation of fish samples.

Lipidomics is the study of the lipidome i.e. the lipid profile of found within a cell, tissue, organism, or ecosystem. Rapid evaporative ionisation mass spectrometry (REIMS) is one of the newest forms of AMS and as is the case with many analytical innovations was created for medical research purposes. This lipidomics based technique uses an electrosurgical knife which creates an aerosol (smoke) when cutting into a tissue sample (Figure 1). The aerosol is evacuated from the surgical site through a vent line into a mass spectrometer, where a heated capillary is situated and the ionisation process occurs.

This technical bulletin describes research carried out at Queen's University Belfast on the application of REIMS technology in the speciation of fish samples. The study was based upon five commercially popular white fish species i.e. cod, coley, haddock, pollock and whiting. The spectra produced were then data-mined, pre-processed and used to generate chemometric models such as Linear Discriminate Analysis (LDA) calibration (Figure 2). As part of the method development, internal and external validation sets of samples were used. With internal validation the correct classification rate was measured at 99.4% whilst the external validation gave 99% (Table 1).

Figure 1: REIMS source and QToF-MS coupled to the electrosurgical knife

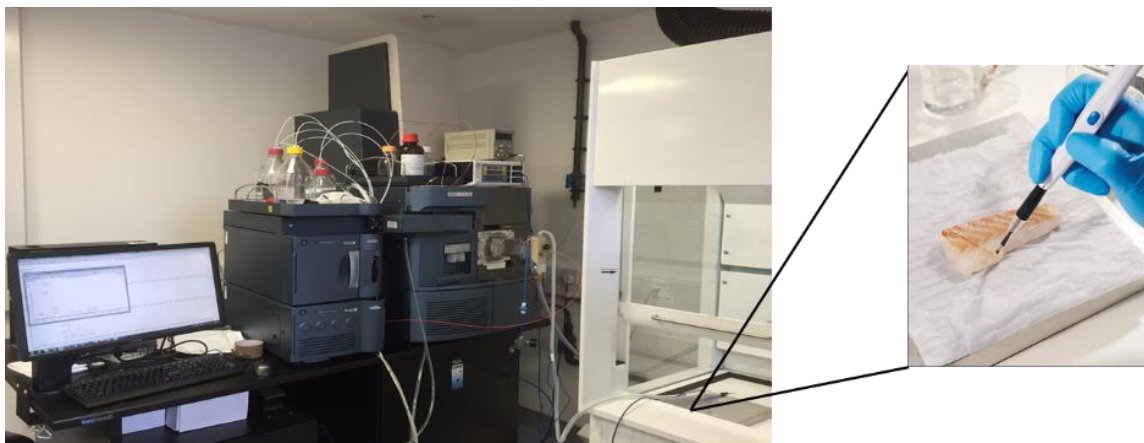


Figure 2: LDA chemometric model based the lipodomic profile from REIMS-QToF-MS data.

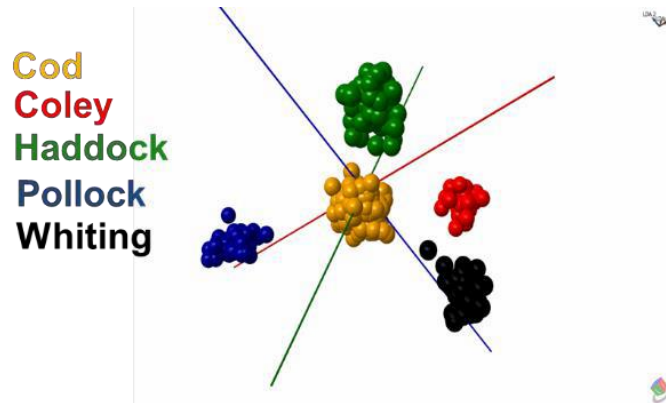


Table 1: Internal and external validation of fish speciation model.

Internal	Cod	Coley	Haddock	Pollock	Whiting	Outlier	Total	Correct Classification Rate
Cod	193	0	0	0	0	1	194	99.48%
Coley	0	51	0	0	0	0	51	100.00%
Haddock	0	0	133	0	0	0	133	100.00%
Pollock	0	0	0	49	0	1	50	98.00%
Whiting	0	1	0	0	49	0	50	98.00%
Total							478	99.37%
External	Cod	Coley	Haddock	Pollock	Whiting	Outlier	Total	Correct Classification Rate
Cod	21	0	0	0	0	1	22	95.45%
Coley	0	20	0	0	0	0	20	100.00%
Haddock	0	0	20	0	0	0	20	100.00%
Pollock	0	0	0	20	0	0	20	100.00%
Whiting	0	0	0	0	17	0	17	100.00%
Total							99	98.99%

This proof of concept lipidomic study using the REIMS technology has indicated that this is a possible approach to identifying fish fraud. It is evident that REIMS has the capability to accurately differentiate between five similar white fish species rapidly using their phospholipid profiles through incorporation into chemometric models.