

Food Authenticity and Food Fraud Research: Achievements and Emerging Issues

HH Grundy^a, SD Kelly^a, AJ Charlton^a, JA Donarski^a, SJ Hird^a, HJ Hird^a and MJ Collins^b

- a) The Food and Environment Research Agency (FERA), Sand Hutton, York, YO41 1LZ UK
b) BioArCh, The University of York, UK

Introduction

In order to protect consumer interests and to combat the continuing problems of food fraud and adulteration scientific expertise and technologies are constantly being developed and advanced to test the authenticity of foods and feeds. Such methods, using the latest developments in DNA fingerprinting techniques and mass spectrometry, have been applied during recent high profile cases of food fraud and adulteration reported in the media such as the inclusion of illegal Sudan dyes in foods and food ingredients, the addition of bovine material to chicken fillets, the counterfeiting of popular wines and species determination of meats.

Adulteration can occur for a variety of reasons, often linked to financial gain. Increases in profitability may be achieved by adulteration to improve the perceived quality of products, mimic an established brand, reduce manufacturing costs or for product extension purposes.

Analytical approaches employed at FERA to screen for food fraud are discussed below, focussing mainly on methods involving mass spectrometry and spectroscopy. These techniques include targeted approaches when the analyte of interest is known and specifically screened for, non-targeted approaches, isotopic measurements and the so-called “*omics*” technologies including metabolomics and proteomics. DNA profiling techniques are also available and are briefly discussed.

Targeted Analysis

Targeted analysis involves screening for pre-determined components in a sample. Foods are analysed using techniques such as liquid chromatography-mass spectrometry and gas chromatography-mass spectrometry (LC-MS and GC-MS) or nuclear magnetic resonance spectroscopy (NMR). The resulting data can then be compared to known masses or information stored in databases in order to identify the contents of a food and screen for a given adulterant. Targeted approaches, for example, have been used to interrogate foods for the presence of illegal Sudan dyes¹, inclusion of meat binding products or “*glues*” of bovine or porcine blood origin^{2,3} and for determining the authenticity of manuka-factor honeys⁴.

Stable Isotope and Elemental Analysis

Analysis of stable isotopes in foods can reveal economically-motivated adulteration such as:

- addition of cheap sugar syrups to extend honey and maple syrup
- watering down of wine

- preparation of fruit juice described as “*freshly squeezed*” from concentrate
- verification that chicken has been “*corn-fed*”
- determination of whether ethanol and vinegar and flavourings are natural or synthetic
- differentiation between organic and conventional farming methods^{5,6}.

In more sophisticated applications of multi-element stable isotope analysis the geographic origin (rearing location) of animals used in meat production can be determined⁷. This approach can be applied to any agricultural product where provenance adds value, such as Saffron spice⁸.

Metabolomics and Proteomics

Meats which are allowed to mature for longer periods of time can carry a premium price at market. Beef, for example, can be labelled as being matured for 21 days as a marketing tool to promote sales. Metabolomic methods have been developed at FERA to verify labelling claims linked to beef ageing and also to the temperature used for meat storage during ageing. Samples of meat stored under ideal (4°C), sub-ideal (-20°C) and above-ideal (20°C) temperatures for wet ageing were prepared and analysed daily over a period of 28 days to cover typical labelling claims. Samples of meat were analysed by ¹H-NMR spectroscopy using a simple extraction strategy to generate a non-targeted profile. The ¹H-NMR spectra were analysed statistically using principle components analysis (PCA) to determine underlying trends. Several analytes that varied with both temperature and age were identified and could be used to verify the age of the sample⁹.

Proteomic methods use high resolution mass spectrometry to identify unique proteins or peptides to determine food components. Further, the technology can be used in a more forensic approach, for example to identify the plant or animal species incorporated within a food.

Protein-based methods are often useful for analysing highly processed samples in which DNA is denatured during the production procedure which hinders using DNA as a marker of authenticity. A proteomics method was developed at FERA in collaboration with the University of York to determine the species origin (pork, beef, fish or poultry) of gelatine and other highly processed hydrolysed proteins made from skin and bone and added to foods. Such proteins are incorporated into many foods and beverages as thickeners, clarifying agents and to enhance “*mouth feel*”. The method employs a proprietary database built up over a number of years and containing scores of species and phylogeny data which is used to match unique peptides which are specific to a species or to a tissue (skin or bone). The method was recently employed to investigate the suspected addition of hydrolysed protein to samples of chicken fillets as a water-binding agent¹⁰. The fillets, labelled as “*chicken only*” or as “*Halal-slaughtered*”, were shown to be adulterated with hydrolysed proteins derived from cow material.

In a variation of the same method, the species contained in animal feed (meat and bone meal) can be determined. This method may be useful in supporting the relaxation of the European Commission Extended Feed Ban. This legislation prohibits the inclusion of mammalian proteins in ruminant feed and was implemented as a result of the BSE outbreak of 1987 in order to eradicate the disease in Europe. Scientific and epidemiologic evidence shows that meat and bone meal is the most probable vector of the disease. The EU has come to the stage where amendments to certain parts of the feed ban could be envisaged, without endangering

health or prejudicing the policy of eradicating BSE, provided that scientific conditions are in place to screen feeds. Our gelatine method could determine the species provenance of skin and bone within a highly processed feed sample to determine whether ruminant material was present.

Fingerprinting Techniques

Finally, DNA fingerprinting techniques can be applied to authenticate foods. DNA techniques are used to screen for adulteration of basmati rice, for identification of meat and fish species^{11,12} and to determine the floral content of honey¹³.

Conclusion

A wide variety of scientific techniques are employed to screen for authenticity and/or adulteration in the food and feed chain. The analytical testing services at the Food and Environment Research Agency (FERA) are underpinned by scientific expertise, detailed knowledge of current and emerging regulations and by internationally-recognised quality standards. A range of sophisticated tools and continuing research effort are applied to address emerging food quality issues and to ensure brand and consumer protection.

Acknowledgements

We would like to thank all of our colleagues at FERA and collaborators who contributed to the various research projects outlined above. We thank the team at BioArCh for their collaboration. We also gratefully acknowledge all of the customers and funding bodies who funded the work including the Food Standards Agency, Defra, EU (FP6) Safeed-PAP project, EU (FP6) Trace project, the English Beef and Lamb Executive and Interact funding.

References

- 1 http://www.scientistlive.com/European-Science-News/Food_Safety/LC-MS-MS%20in_the_hunt_for_%26lsquo%3Billegal%26rsquo%3B_dyes_in_foodstuffs/14486/
- 2 HH Grundy, P Reece, MD Sykes, JA Clough, N Audsley, R Stones (2007), Screening Method for the Addition of Bovine Blood-based Binding Agents to Food using Liquid Chromatography/Triple Quadrupole Mass Spectrometry, *Rapid Commun Mass Spectrom*, **21**(18), 2919-25
- 3 HH Grundy, P Reece, MD Sykes, JA Clough, N Audsley, R Stones (2008), Method to Screen for the Addition of Porcine Blood-based Binding Products to Foods using Liquid Chromatography/Triple Quadrupole Mass Spectrometry, *Rapid Commun Mass Spectrom*, **22**(12), 2006-8
- 4 JA Donarski, DPT Roberts and AJ Charlton (2010), Quantitative NMR Spectroscopy for the Rapid Measurement of Methylglyoxal in Manuka Honey, *Anal Methods*, **2**, 1479-1483

- 5 SD Kelly (2003), Using Stable Isotope Ratio Mass Spectrometry in Food Authentication and Traceability, Food Authenticity and Traceability, Michele Lees(Ed), Woodhead Publishing, Cambridge, UK, ISBN 0-8493-1763-0, 156-183
- 6 SD Kelly, AS Bateman (2009), Comparison of Mineral Concentrations in Commercially Grown Organic and Conventional Crops - Tomatoes (*Lycopersicon esculentum*) and Lettuces (*Lactuca sativa*), Food Chemistry, 119, 738–745
- 7 K Heaton, SD Kelly, J Hoogewerff and M Woolfe (2007), Verifying the Geographical Origin of Beef: The Application of Multi-element Isotope and Trace Element Analysis, Food Chemistry, 107, 506–515
- 8 L Maggi, M Carmona, SD Kelly, N Marigheto and GL Alonso (2011), Geographical Origin Differentiation of Saffron Spice (*Crocus sativus* L. stigmas) – Preliminary Investigation using Chemical and Multi-element (H, C, N) Stable Isotope Analysis, Food Chemistry, 128, 543–548
- 9 JS McKenzie, JA Donarski, JC Wilson and AJ Charlton (2011), Analysis of Complex Mixtures using High-resolution Nuclear Magnetic Resonance Spectroscopy and Chemometrics, Progress in Nuclear Magnetic Resonance Spectroscopy, 59(4) 336-359
- 10 www.food.gov.uk/news/newsarchive/2009/jun/chicken
- 11 J Chisholm, C Conyers, C Booth, W Lawley and H Hird (2005), The Detection of Horse and Donkey using Real-time PCR, Meat Science, 70 (4), 727-732
- 12 H Hird, J Chisholm, J Kaye, A Colyer, G Hold, C Conyers, J Irazu Nunez and R Macarthur (2012), Development of Real-Time PCR Assays for the Detection of Atlantic Cod (*Gadus morhua*), Atlantic Salmon (*Salmo salar*) and European Plaice (*Pleuronectes platessa*) in Complex Food Samples, European Food Research and Technology, 234 127-136
- 13 I Laube, H Hird, P Brodmann, S Ullmann, M Schöne-Michling, J Chisholm and H Broll (2010), Development of Primer and Probe Sets for the Detection of Plant Species in Honey, Food Chemistry, **118 (4)**, 979-986