



Contents lists available at ScienceDirect

Trends in Food Science & Technology

journal homepage: <http://www.journals.elsevier.com/trends-in-food-science-and-technology>

Review

Stable isotope techniques for verifying the declared geographical origin of food in legal cases

Federica Camin^{a,*}, Markus Boner^b, Luana Bontempo^a, Carsten Fauhl-Hassek^c, Simon D. Kelly^d, Janet Riedl^c, Andreas Rossmann^e^a Department of Food Quality and Nutrition, Research and Innovation Centre, Fondazione Edmund Mach (FEM), via E. Mach 1, 38010, San Michele all'Adige, Italy^b Agroislab GmbH, Prof. Rehm Str. 6, D-52428, Jülich, Germany^c Department Safety in the Food Chain, BfR – Federal Institute for Risk Assessment, Max-Dohrn-Straße 8-10, D-10589, Berlin, Germany^d Food and Environmental Protection Laboratory, Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, Department of Nuclear Sciences and Applications, International Atomic Energy Agency, Vienna International Centre, PO Box 100, 1400, Vienna, Austria^e Isolab GmbH, Laboratorium für Stabile Isotope, Woelkestrasse 9/I, 85301, Schweitenkirchen, Germany

ARTICLE INFO

Article history:

Received 30 June 2016

Received in revised form

5 December 2016

Accepted 21 December 2016

Available online 5 January 2017

Keywords:

H, C, N, O, S and Sr

Food authenticity

PDO

PGI

Legal cases

Fraud

Mislabeling

ABSTRACT

Background: Consumers are increasingly interested in the provenance of the foods and European laws require protection against the mislabelling of premium foods. Methods for testing authenticity require robust analytical techniques that can be utilised by the various regulatory authorities. Of the many techniques, the most widely-used method is stable isotope ratio analysis.

Scope and approach: Focus is on the use of stable isotope ratios of H, C, N, O, S and Sr for verifying the geographical origin of food, cross-referencing it with examples of legal cases. State of the art including rules for building an authentic sample reference database (commonly called databank) and for interpreting the results obtained in actual cases is described. The overall objective is to provide stakeholders and competent authorities dealing with fraud, with a best-practice guide for its use.

Key findings and conclusions: Stable isotope ratios can differentiate foods on the basis of their geographical origin and, especially for light elements, can be measured reliably in routine work in different matrices and compared successfully between different laboratories. Examples of legal applications are grape products, orange juices, olive oil, cheese, butter, caviar. Sometimes, the cases are not brought directly to the court, but before further verifications (e.g. paper traceability, forensic accounting) are conducted. The system can satisfy the court when a robust databank of authentic samples exists, the methods used are officially recognized, validated and accredited, and the expert demonstrates that the conclusions are sufficiently robust and reliable to stand up to the required level of proof.

© 2017 Elsevier Ltd. All rights reserved.

1. Introduction

Consumers are increasingly interested in the provenance of the foods they consume. The reasons include patriotism; specific culinary or organoleptic qualities associated with regional products; decreased confidence in the quality and safety of products produced outside their local region, country, the EU, or those of unknown provenance. Consequently, products from highly esteemed geographical origins can be sold at markedly higher prices than similar products of other or unknown provenance. For certain

products, such as wines, indicating geographical origin has been an established practice for many years, but for other foods and drinks this kind of marketing is more recent. In some European countries, marking certain products with geographical indication or brand has a long tradition (e.g. French and Italian cheese). Today in Europe a number of products with a specific geographical origin and production method are officially protected (Protected Designation of Origin = PDO; Protected Geographical Indications = PGI) and this number is increasing annually, reaching a total number of 1328 registered products at the beginning of March 2016. As these products may obtain higher prices on the market, it is possible that unscrupulous producers might buy cheaper raw materials from other regions and illegally sell the product with the PDO or PGI

* Corresponding author.

E-mail address: federica.camin@fmach.it (F. Camin).

label. This is increasingly likely if the demand exceeds domestic supply from a PDO defined geographical region. European laws EC N. 510/2006 and 1151/2012 require protection against the mislabelling of PDO and PGI foods. In this respect, among others, appropriate analytical strategies are required to avoid consumers being cheated, and to protect honest producers.

Methods for testing authenticity and providing analytical data on traceability require robust analytical techniques that can be utilised by the various regulatory authorities. Of the many techniques available to aid in authenticity and traceability testing, the most widely-used method is isotope ratio mass spectrometry (IRMS). The reasons for this are the high accuracy of the method, the small amount of sample required for analysis and the fact that the same technique can be used for almost any type of food or beverage and the results are generally not altered by the production technique. Stable isotope analysis has been used in food control, since around 1975, to detect adulteration of products like wine, honey, fruit juice, or maple syrup with cheaper extenders, such as sugar syrup made from maize or cane carbohydrates, industrially produced acids, or simply water (Rossmann, 2001).

These “traditional” applications of stable isotope analysis in food control rely on the measurement of the isotopic ratio of only one or two elements ($^{13}\text{C}/^{12}\text{C}$ and $^{18}\text{O}/^{16}\text{O}$, $^{13}\text{C}/^{12}\text{C}$ and D/H). This can confirm if a certain component of a food material was a non-natural (exogenous) additive from other plants or industrial production. Several of these methods have been officially validated and acknowledged as AOAC, CEN, or EU methods (Table 1). More recently other applications based on multi-isotope ratio analysis ($^2\text{H}/^1\text{H}$ or D/H, $^{13}\text{C}/^{12}\text{C}$, $^{18}\text{O}/^{16}\text{O}$, $^{15}\text{N}/^{14}\text{N}$, $^{34}\text{S}/^{32}\text{S}$, $^{87}\text{Sr}/^{86}\text{Sr}$) have included geographical origin verification studies of olive oil (Camin et al., 2010), orange fruit (Rummel, Hoelzl, Horn, Rossmann, & Schlicht, 2010), honey (Schellenberg et al., 2010), cereals (Goitom Asfaha et al., 2011; Luo et al., 2015), tomato (Bontempo et al., 2011; Trincherini, Baffi, Barbero, Pizzoglio, & Spalla, 2014), Chinese cabbages (Bong et al., 2012), animal products (Rees et al., 2016; Vinci, Preti, Tieri, & Vieri, 2013) and feeds (Nietner, Haughey, Ogle, Fauhi-Hassek, & Elliot, 2014), seafood (Kim, Suresh Kumar, & Shin, 2015), and coffee (Santato, Bertoldi, Perini, Camin, & Larcher, 2012).

To address the question of geographical origin, the methods developed and used for testing food authenticity are useful as a starting point because they already confirm that stable isotope data can be measured reliably in routine work and compared successfully between different laboratories. Nevertheless, the methods, instrumentation, reference materials, and data evaluation have to be adapted to enable the verification of geographical provenance. Adulterant identification usually works successfully with one or two stable isotope parameters (see Table 1), while geographical origin determination often requires multiple stable isotope parameters. This should include stable isotope data for H, C, N, O and S (the light elements, which form biological materials, so called “bio elements”), and sometimes heavier elements, such as Sr, and their isotopic ratios (Hölzl, Horn, Rossmann, & Rummel, 2004; Kelly, Heaton, & Hoogewerff, 2005). The heavier elements (or “geo elements”), because of their importance in geochemical research) are non-essential or at least not quantitatively important components of living organisms, nor are they actively integrated into biological molecules, and consequently are unaffected by measurable isotopic fractionation in biological systems. However, they can be very important indicators of the local geology (rocks, soils), where living matter originates from, and may indicate anthropogenic contamination as well.

The applications of stable isotope ratio analysis are based on the fact that stable isotope ratios of these elements change with the geographical origin, the climatic conditions, and the soil pedology and geology of the location from where the products originate. In

broadly generalised terms, the H and O stable isotopic composition of organic matter in food is linked to the H and O isotope composition of water from the source region that exhibits geographical variability. N and C isotopes are related to the climate and the agricultural practices, and S isotopes are affected by geology, volcanic activity, distance from the sea, and by certain anthropogenic effects. Sr isotopes depend on bedrock geology, especially on their chemical composition (content of alkaline and earth alkaline metals, that is the ratio of K and Ca, and from that of Rb and Sr), their age (as with time ^{87}Rb decays into ^{87}Sr), and geological history (crystalline rocks, metamorphic rocks, or sediments) (Drivelos & Georgiou, 2012; Kelly et al., 2005; Rossmann et al., 2000).

In this paper we focus on the use of stable isotope ratio analysis to independently verify the geographical origin declared on the food label and cross-reference it with examples of legal cases where the method was used in enforcement exercises. We aim to describe the state of the art including limits of reliable interpretation, rules for building an authentic sample reference database (commonly called databank) and for interpreting the results obtained in actual cases. The overall objective is to propose a reliable implementation strategy for stable isotope ratio analysis in geographical origin determination of food and provide stakeholders and competent authorities, dealing with food fraud, with a best-practice guide for its use.

2. Methodological background

2.1. Commodities

Currently standard methods are available for wine, fruit juice, cheese and olive oil (Table 1), which are commodities susceptible to adulteration or extension of their major nutrient components, e.g. water in wine, sugar in fruit juice; requiring specific sample preparations for each. These methods can be used for other similar commodities (e.g. the methods for cheese can be used for meat or butter, and those for fruit juice for any food containing sugar).

2.2. Sample preparation

With a few exceptions (e.g. $\delta^{18}\text{O}$ of food water and of $\delta^{13}\text{C}$, $\delta^{18}\text{O}$ and $\delta^2\text{H}$ of olive oil) treatment of the sample is needed because it permits the isolation of nutrient components that record the geographical fingerprint better than the bulk sample and with less interferences (e.g. fat has different isotopic values than proteins (DeNiro & Epstein, 1978)). Note that it is essential that any sample preparation avoids isotopic fractionation i.e. variation of the original isotopic ratio due to concentration or reduction of heavy versus light elements.

According to the official methods (Table 1) vegetable products containing sugar (e.g. grape must and fruit juices) are often fermented and subjected to distillation to recover ethanol which is subsequently analysed. This is because ethanol reflects the isotopic composition of the parent sugar and is much more amenable to isotopic analysis by both isotope ratio mass spectrometry and quantitative deuterium Nuclear Magnetic Resonance. Beverages that are already fermented (e.g. wine) only need to be distilled before isotopic analysis.

From fruit juice, the pulp is separated through juice centrifugation and washing with water and then with acetone, as described in the ENV 13070 method. The sugars are extracted from the remaining supernatant, through the addition of calcium hydroxide and then sulphuric acid to eliminate acids and amino acids according to the ENV 12140 method.

Samples containing lipids (e.g. meat, milk, and cheese) are usually defatted because fat has a different C and H composition

Download English Version:

<https://daneshyari.com/en/article/5523735>

Download Persian Version:

<https://daneshyari.com/article/5523735>

[Daneshyari.com](https://daneshyari.com)