

1st ISO-FOOD International Symposium on Isotopic and Other Techniques in Food Safety and Quality

Portorož, Slovenia

April 1-3, 2019



Organised by

ERA Chair ISO-FOOD in Isotope Techniques in Food Quality,
Safety and Traceability
Department of Environmental Sciences
Jožef Stefan Institute

Programme and Book of Abstracts

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Edited by:

David Heath, Milena Horvat, and Nives Ogrinc

Lunch Break and Poster Session Apr 01, 12:45 - 14:00

PO-01

Geographical origin of plant food: Implementation of robust screening tests

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Recently, increased interest in production with declared and guaranteed geographical origin has resulted in higher market prices for these products, and thus mislabelling and mixing higher class products with cheaper products have become a serious problem in many areas of the food industry. The need to monitor the authenticity and quality of various types of plant samples led to increasing demand for rapid, inexpensive and reliable screening methods in order to check their origin. Therefore, our research work was focused on evaluation of potential traceability parameters for geographical characterization of different types of plant samples through the application of a broad spectrum of analytical techniques in combination with multivariate statistical analysis. Geographical characterization and classification of plant samples into groups was performed through applying different parameters such as stable isotopic compositions of four major bio-elements ($\delta^{13}\text{C}$, $\delta^{15}\text{N}$, $\delta^{34}\text{S}$, $\delta^{18}\text{O}$), elemental content (Na, Mg, P, S, Cl, K, Ca, Mn, Fe, Ni, Cu, Zn, Mo, Br, Rb, Sr) including rare earth elements (Sc, Y, Nb, La, Ce, Pr, Nd, Dy, Er), and physico-chemical parameters (total antioxidant potential, total phenolic compounds, ascorbic acid, lutein, nitrates and nitrites, ammonium), and data treatment using discriminant analysis. Subsequently, efficient preliminary statistical models were created. Classification efficiencies of suggested multivariate statistical models were found out to be sufficient for fast and robust screening purpose, giving the overall success rate of correctly reclassified samples for particular model from 71.1% to 100.0%. Different approaches were tested to assign geographical provenance at different scales- the macro-region origin at national scale (i.e. Slovenian) to organically grown garlic and potato, or country of origin at the larger European and Mediterranean scale to commercially distributed tomato, lettuce and sweet pepper. Present studies were performed to initiate the collection of data for the creation of fundamental and reliable traceability models since an important prerequisite for implementation of any official surveillance of origins is the availability of the relevant databanks, at both national and international levels. These protocols enable traceability and verification of provenance on selected realistic physico-chemical key parameters, and not only traceability through documentation, which is unfortunately the main common practice in our food trade market.

PO-02

On the traces of the *Cannabis sativa*

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Hemp (*Cannabis sativa*) is becoming increasingly important year by year, especially in Italy, mainly in the food sector (oils, flours, seeds).

The aim of this PhD study is to define a method to trace and guarantee the geographic provenience of the Italian *Cannabis sativa* and its derived products. Nowadays, this method does not exist for the “industrial hemp”, except for Marijuana traffics (Elisa K. Shibuya et al., 2007, Janet M. Hurley et al., 2010).

This traceability study will be based on stable isotope ratios and on trace elements fingerprints. Food authentication has successfully been using this method, mainly through C, N, H, S and O stable isotope ratios, for a long time now.

First samples of hemp were collected in 2018 in Veneto, Emilia Romagna and Friuli Venezia Giulia regions. Further samplings are planned in 2019 and 2020, starting from early Summer (South Italy) to Autumn (North Italy). Two specific sites will be considered for the so called “Study of Hemp Factory”: Udine and Catania, that were chosen because of their difference in climatic conditions and geological background, helping us to understand which isotopic ratios will be more effective in the traceability along the whole production chain. Indeed, in the “Study of Hemp Factory”, we will evaluate oil, plant, inflorescences, seeds and fertilizers. 2H/1H and 18O/16O isotopic ratios of local precipitation and irrigation waters will be compared to the ones obtained from the plant tissues to understand if these ratios can be used as tracers. A further sampling campaign will be carried out in other areas of Italy for defining a spatial variability: here, only plants and oils will be analysed.

The isotopic composition of C, N, H, O and S, will be analysed by Isotope Ratio Mass Spectrometry at Fondazione Edmund Mach and the IDPA-CNR laboratories. H and O isotopic analysis on waters will be done at DAIS-Cà Foscari University.

Moreover, isotopic composition of Sr and Pb will be determined in plant, soil and oil samples at the Jožef Stefan Institute laboratories in Ljubljana, by means of Multi Collector Inductively Coupled Plasma – Mass Spectrometry. Simultaneously, to reinforce the method, ICP-MS analysis on trace elements will be performed on selected samples,

at IDPA-CNR Venice.

This multiparametric approach will allow us to define which parameters are more useful for trace back the geographic provenience of *Cannabis sativa* and its derived products.

PO-03

Application of chemometric tools for determination of authenticity of Serbian pear spirits

Biljana Marosanovic, *Maja Lojovic*, Aleksandra Bauer
SP Laboratorija, Serbia

In recent years, many food products have been losing their authenticity, uniqueness and characteristic of the area from which they originate. The authenticity of fruit spirits is one of the important issue regarding the consumer protection and it is based on the identification and elimination of fraud in the market. Pear spirit is a national, Serbian, alcoholic beverage with a great tradition and it becomes subject of falsification. Counterfeiting is related to ethanol content increasing, by adding sugar from sugar beet (C3 plant) or sugar cane and corn (C4 plant).

The determination of $\delta^{13}\text{C}$, $\delta^2\text{H}$ and $\delta^{18}\text{O}$ isotope ratios by Elemental Analyzer - Isotope Ratio Mass Spectrometry (EA-IRMS by Thermo) is a precise analytical method that can be used to check the authenticity, botanical and geographical origin of fruit spirits.

For detection of the adulteration, we have made laboratory control samples of spirits of pear, sugarcane and corn in SP Laboratorija. At the same time, we have prepared samples of pear spirits with different amount of added beet sugar (3%, 5%, 10%, 20% sugar was added on the weight of fruit during fermentation), from different geographical origin (South and North part of Serbia). Also, we collected pear spirits produced in Serbia in the period from 2005 until 2017. Combined results of $\delta^2\text{H}$ and $\delta^{13}\text{C}$ values gives an important information about botanical origin of ethanol and possibility of distinguishing between fruit spirits and spirits with non-fruit origin (made from beet sugar, maize, cane sugar). In the aim of geographical classification for spirit samples, originating from different locations in Serbia, spirits has been determined by isotope ratio $\delta^2\text{H}$ and $\delta^{18}\text{O}$.

Application of Chemometrics has established a linear correlation between the concentration of sugar values (derived from sugar beet) and $\delta^2\text{H}$ values. Applying this equation, it is possible to calculate the amount of added sugar in the unknown sample.

The application of the formed database enabled the classification of commercial pear spirits on the basis of botanical and geographical origin.

PO-04

Characterization of truffles (*Tuber* sp.) in Slovenia using stable isotope approach and elemental composition

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The high cost of several species from the genus *Tuber* attracts more or less exquisite adulteration practices. The most known of the *Tuber* species in Europe are: *Tuber magnatum* Pico 1788 and *Tuber melanosporum* Vittadini 1831, followed by other commercially interesting species such as *Tuber aestivum* Chatin 1887, *Tuber macrosporum* Vittadini 1831, *Tuber brumale* Vittadini 1831, *Tuber borchii* Vittadini 1831, etc. Truffles are used in culinary as fresh fruiting bodies or as ingredients in the processed product. Since 2011 the truffle hunting is again legal in Slovenia and majority of Slovenian truffles comes from hunting wild truffles. While some of the species can be found in different parts of Slovenia, the two most appreciated *T. magnatum* and *T. melanosporum* are found only in Slovenian Istria.

In season 2017-2018, six truffle samples were collected in Slovenian Istria. Two samples of *T. magnatum*, one *T. borchii*, one *T. melanosporum*, and two *T. aestivum* were analyzed for elemental composition by X-ray fluorescence (XRF) and for stable isotope composition (H, O, C, N, and S) by isotope ratio mass spectrometry (IRMS). Stable isotope composition of carbon was also determined for the main volatile organic compounds by the use of headspace solid-phase microextraction (HS-SPME) coupled with IRMS.

There was high variability of $\delta^2\text{H}$, $\delta^{15}\text{N}$, and $\delta^{34}\text{S}$ observed between different truffle species. The statistical evaluation of elemental and stable isotope compositions indicated that samples can be separated according to $\delta^{15}\text{N}$, Pb and P. $\delta^{13}\text{C}$ values in VOC were also significantly different between different truffle species. It was also speculated that different VOC components can be produced not only by the truffle bur also or exclusively by the microbes such as yeasts or bacteria.

These preliminary results of the ongoing study show that elemental composition and stable isotope composition could be used for determination of species variety and trophic status of truffles. It was also indicated that truffles offer a unique opportunity to better understand the ecological function of microbes associated with fungi and their involvement in aroma formation.

Acknowledgement: The research was performed within ARIMnet REALMed project with the aim of "Pursuing authenticity and valorization of Mediterranean traditional products".

PO-05

Establishing reproducibility in compound-specific stable isotope measurements for food authentication and adulteration

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