applied sciences

MDPI

Editorial

Application of Spectroscopy in Food Analysis: Volume II

Federico Marini [1,*] and Alessandra Biancolillo [2,*]

[1] Department of Chemistry, University of Rome "La Sapienza", Piazzale Aldo Moro, 5, 00185 Roma, Italy
[2] Department of Physical and Chemical Sciences, University of L'Aquila, Via Vetoio, Coppito, 67100 L'Aquila, Italy
* Correspondence: federico.marini@uniroma1.it (F.M.); alessandra.biancolillo@univaq.it (A.B.)

1. Introduction

"Man is What He Eats": food represents one of the fundamental needs of human beings, and, therefore, food analysis is a field of utmost importance. At the same time, given its inherent complexity, this subject needs to be investigated by employing advanced analytical methods. In light of this, this Special Issue aimed at collecting studies describing interesting/relevant problems in food analysis and, ideally, suggesting strategies for solving/handling them. In total, 15 manuscripts (13 research works, 1 commentary, and 1 review) have been published. The submitted papers encompass different aspects and scopes: authenticating and/or characterizing agro-foods/products and ensuring law/sanitary compliance and quality.

2. Exploratory Analysis, Class-Modeling, and Food Quality

In three of the published articles, the investigation was directed towards the solution of real practical problems, and/or focused on authenticating/characterizing products that had not been extensively or comprehensively studied yet.

Of these, two have a biological background, and they are the works by Butsenko et al. and Eady and Park. In the first one [1], the random amplification of polymorphic DNA (RAPD)-PCR (polymerase chain reaction) profiles were used to identify the causative agent of the basal glume rot of wheat. Eventually, using cluster analysis, the authors highlighted that the strains isolated from weeds have a high similarity with the causative agent of the basal glume rot of wheat *P. syringae* pv. *Atrofaciens.* Consequently, they concluded that, to avoid basal glume rot, an alternative solution to crop rotation should be inspected.

In the work from Eady and Park [2], the possibility of developing a rapid detection tool for foodborne pathogenic bacteria was investigated. To this purpose, different bacterial cultures were prepared (isolated and purified), stocked, and analyzed using hyperspectral microscope imaging (HMI); then, the soft independent modeling of class analogies (SIMCA) was used to model class Salmonella. A sensitivity of 95.4% with a specificity of 97% was achieved. These results indicate that HMI can be used for the early (<8 h) and rapid (<1 h) detection of salmonella in foodstuffs.

The latter paper in this group is the one by Sciubba and collaborators [3] where a study aimed at monitoring the metabolome of purple carrots using high-resolution ^{1}H NMR spectroscopy for a period of four months is reported. Principal components analysis (PCA) revealed that, during the development of the carrot, it is possible to appreciate an increase in the content of amino acids, NAD, and caffeic acid. Moreover, the analysis pointed out that tardive harvest (December) of the plant provides an increase in the concentration of luteolin-7-O-glucoside, chlorogenic acid, falcarinol, and γ-aminobutyrate together with a decrease in carotenoids and ω-6 fatty acid.

3. Regression Approaches and Quality Assessment

Two articles focus on the assessment of the freshness/maturity of foods. This is the case of the papers by Kasampalis et al. and Legner et al. [4,5]. In the former one, the authors

check for updates

Citation: Marini, F.; Biancolillo, A. Application of Spectroscopy in Food Analysis: Volume II. *Appl. Sci.* **2023**, *13*, 5633. https://doi.org/10.3390/app13095633

Received: 1 March 2023
Revised: 7 March 2023
Accepted: 15 April 2023
Published: 3 May 2023

demonstrated that Vis/NIR spectroscopy coupled with multiple linear regressions (MLR) can be used on potato tubers to assess their postharvest storage life, whereas its combination with partial least squares discriminant analysis (PLS-DA) represents a suitable tool for cultivar discrimination [4].

The work from Legner and collaborators [5] proposes a portable Raman-based tool to estimate the maturity of fruits containing carotenoids. Hot pepper samples were analyzed using a Raman instrument, and then the spectra were processed by means of PCA, showing clear grouping tendencies according to their ripening. From the intensity of the signals at 1156 cm^{-1}, 1515 cm^{-1}, and 1001 cm^{-1}, the authors also derive a maturity index which allows the assessment of the maturities of different carotenoid-containing fruits. One of the main practical outcomes of this tool is that it can be used in greenhouses or in automatic harvesting processes to assist with fruit picking.

A similar purpose has been investigated by Baeva et al. [6] who have developed a FT-MIR, FT-NIR, and FT-Raman approach finalized to the optimization of the picking of *Pleurotus ostreatus* mushrooms. In fact, they have demonstrated that the vibrational spectra of basidiocarp samples handled by PLS can be used to quantify protein and total glucan contents, and, therefore, this approach can be exploited to assess the quality of mushrooms as culinary ingredients.

The synergy between FT-NIR and PLS was also exploited by Aykas et al. [7] in order to develop a method that allows the quantification of total sugars in various breakfast cereals. Briefly, 164 breakfast cereal samples were analyzed using a miniaturized FT-NIR. Reference analysis for sucrose, glucose, fructose, and total sugars was carried out using high-performance liquid chromatography (HPLC). Eventually, PLS was used to assess individual and total sugars based on the NIR spectra. The authors concluded that the proposed approach represents an efficient and rapid alternative to standard methods for the quantification of individual and total sugar content in breakfast cereals.

The coupling of FT-NIR and PLS was also exploited in the work published by Alamu et al. [8] who have relied this efficient combination for the quantification of dry matter, protein, and starch content in fresh yam samples. Eventually, they demonstrated that the coupling of FT-NIR (collected on blended yam) and PLS represents a suitable alternative to the wet-chemistry procedure.

The last paper belonging to this group is the one from Liu and collaborators [9] who have developed a spectroscopy-based procedure for the prediction of the frying time of various edible oils. To achieve this goal, 15 frying cycles were carried out using 3 different oils: soybean, peanut, and rapeseed. FT-NIR spectra were collected on oils after each frying cycle. Eventually, the correlation coefficient between the sequence of frying times and absorbance was used to select characteristic wavelengths which were at the basis of a differential prediction model for the estimation of frying time. The proposed approach led to a prediction accuracy of 100% $\pm$ 1 cycle.

4. Classification Approaches and Spectroscopy for Fraud Detection

In this section, the works where spectroscopic data have been analyzed using different classifiers for the determination of possible frauds will be summarized. Within this Special Issue, three papers belong to this field. One of them is the work published by Socaciu et al. [10], where the botanical origin of edible oils was investigated. Oil samples belonging to eight different classes were analyzed by FT-IR and UV-Vis and then classified (according to the botanical origin) by PCA, PLS-DA, cluster analysis, heatmap, random forest analysis, and ANOVA post hoc analysis. Eventually, the authors concluded that PLS-DA, cluster analysis, and random forest achieved the highest predictive ability.

The other two works, proposed by Biancolillo et al. and Le Nguyen et al., exploit IR and NIR spectroscopy for adulteration detection in agro-foods. In the former paper [11], the investigation is focused on a PGI Italian dry bell pepper sold ground and used as a seasoning. The work aims at developing a tool that determines whether a product consists of pure Senise bell pepper or has been adulterated with similar ground foods (for example, paprika). For this purpose, mixtures of Senise bell pepper and paprika (at different concentrations) were prepared with the intent of mimicking adulteration. Eventually, samples were analyzed using FT-IR and FT-NIR spectroscopies; then, multi-block classification models (sequential and orthogonalized partial least squares linear discriminant analysis and sequential and orthogonalized covariance selection linear discriminant analysis) were used to discriminate blends from the pure Senise bell pepper spice. Both procedures achieved successful results in external validation, correctly classifying all the test samples, and confirming the suitability of the proposed strategies.

Le Nguyen et al. [12] investigated the adulteration of rice and developed a NIR-based tool for its detection. On this product, adulteration can be performed by mixing a high-valued cultivar with rice with a lower market value. In particular, blends of high-commercial-value rice with other cheaper ones were prepared and analyzed using NIR spectroscopy. Subsequently, a discriminant classifier, PLS-DA, and a class-modeling one, SIMCA, were applied for the identification of the adulterated mixtures. The outcome of the analysis revealed that, contrary to SIMCA, PLS-DA was suitable for the purpose, achieving a high accuracy on a validation set of samples.

5. Method Development, Review, and Commentary on This Topic

The article by Sadat and Joye [13] proposed a method finalized to the identification and separation of hidden peaks in the IR and Raman spectral ranges associated with the amide I band. To test this novel approach, the authors prepared solutions of hydrated zein, gluten proteins immunoglobulin G, concanavalin A, lysozyme, and trypsin. The use of the second derivative allowed the identification of different peaks; eventually, the separation of peaks was achieved by curve fitting using the Voigt function.

The commentary from Grootveld et al. [14] presents a case study focalized on the generation of aldehydic lipid oxidation products (LOPs) in culinary frying oil (CFO) products undergoing laboratory-simulated shallow-frying episodes (LSSFEs). The main outcome of this study lies in the result that LOPs reached higher levels in oils rich in monounsaturated and saturated fatty acids (sunflower), and the lowest levels occurred in extra-virgin olive oils. Nevertheless, thanks to its low content in saturated fatty acids, coconut oil also shows low levels of aldehydes. The authors conclude that high-temperature frying in PUFA-rich oils leads to a high concentration of LOPs in food which may contribute to the development/progression of communicable chronic diseases.

The review by Lozada Ramirez and collaborators [15] discusses the identification, quantification, and characterization of bioactive compounds in edible plants. The authors have reported the procedures for the identification and quantification of a large number of families of molecules, by reference methods (mainly chromatographic) and/or spectroscopic strategies.

Author Contributions: Writing—original draft preparation, A.B. and F.M.; writing—review and editing, A.B. and F.M. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Butsenko, L.; Pasichnyk, L.; Kolomiiets, Y.; Kalinichenko, A.; Suszanowicz, D.; Sporek, M.; Patyka, V. Characteristic of Pseudomonas syringae pv. atrofaciens Isolated from Weeds of Wheat Field. *Appl. Sci.* **2021**, *11*, 286. [CrossRef]
2. Eady, M.; Park, B. An Unsupervised Prediction Model for Salmonella Detection with Hyperspectral Microscopy: A Multi-Year Validation. *Appl. Sci.* **2021**, *11*, 895. [CrossRef]
3. Sciubba, F.; Tomassini, A.; Giorgi, G.; Brasili, E.; Pasqua, G.; Capuani, G.; Aureli, W.; Miccheli, A. NMR-Based Metabolomic Study of Purple Carrot Optimal Harvest Time for Utilization as a Source of Bioactive Compounds. *Appl. Sci.* **2020**, *10*, 8493. [CrossRef]
4. Kasampalis, D.S.; Tsouvaltzis, P.; Ntouros, K.; Gertsis, A.; Moshou, D.; Siomos, A.S. Rapid Nondestructive Postharvest Potato Freshness and Cultivar Discrimination Assessment. *Appl. Sci.* **2021**, *11*, 2630. [CrossRef]
5. Legner, R.; Voigt, M.; Servatius, C.; Klein, J.; Hambitzer, A.; Jaeger, M. A Four-Level Maturity Index for Hot Peppers (Capsicum annum) Using Non-Invasive Automated Mobile Raman Spectroscopy for On-Site Testing. *Appl. Sci.* **2021**, *11*, 1614. [CrossRef]

6. Baeva, E.; Bleha, R.; Sedliaková, M.; Sushytskyi, L.; Švec, I.; Čopíková, J.; Jablonsky, I.; Klouček, P.; Synytsya, A. Evaluation of the Cultivated Mushroom Pleurotus ostreatus Basidiocarps Using Vibration Spectroscopy and Chemometrics. *Appl. Sci.* **2020**, *10*, 8156. [CrossRef]
7. Aykas, D.P.; Ball, C.; Menevseoglu, A.; Rodriguez-Saona, L.E. In Situ Monitoring of Sugar Content in Breakfast Cereals Using a Novel FT-NIR Spectrometer. *Appl. Sci.* **2020**, *10*, 8774. [CrossRef]
8. Alamu, E.O.; Adesokan, M.; Asfaw, A.; Maziya-Dixon, B. Effect of Sample Preparation Methods on the Prediction Performances of Near Infrared Reflectance Spectroscopy for Quality Traits of Fresh Yam (*Dioscorea* spp.). *Appl. Sci.* **2020**, *10*, 6035. [CrossRef]
9. Liu, Y.; Sun, L.; Bai, H.; Ran, Z. Detection for Frying Times of Various Edible Oils Based on Near-Infrared Spectroscopy. *Appl. Sci.* **2020**, *10*, 7789. [CrossRef]
10. Socaciu, C.; Fetea, F.; Ranga, F.; Bunea, A.; Dulf, F.; Socaci, S.; Pintea, A. Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) Coupled with Chemometrics, to Control the Botanical Authenticity and Quality of Cold-Pressed Functional Oils Commercialized in Romania. *Appl. Sci.* **2020**, *10*, 8695. [CrossRef]
11. Biancolillo, A.; Di Donato, F.; Merola, F.; Marini, F.; D'Archivio, A.A. Sequential data fusion techniques for the authentication of the P.G.I. senise ("crusco") bell pepper. *Appl. Sci.* **2021**, *11*, 1709. [CrossRef]
12. Le Nguyen Doan, D.; Nguyen, Q.C.; Marini, F.; Biancolillo, A. Authentication of rice (*Oryza sativa* LA.) using near infrared spectroscopy combined with different chemometric classification strategies. *Appl. Sci.* **2021**, *11*, 362. [CrossRef]
13. Sadat, A.; Joye, I.J. Peak Fitting Applied to Fourier Transform Infrared and Raman Spectroscopic Analysis of Proteins. *Appl. Sci.* **2020**, *10*, 5918. [CrossRef]
14. Grootveld, M.; Percival, B.C.; Moumtaz, S.; Gibson, M.; Woodason, K.; Akhtar, A.; Wawire, M.; Edgar, M.; Grootveld, K.L. Commentary: Iconoclastic Reflections on the 'Safety' of Polyunsaturated Fatty Acid-Rich Culinary Frying Oils: Some Cautions regarding the Laboratory Analysis and Dietary Ingestion of Lipid Oxidation Product Toxins. *Appl. Sci.* **2021**, *11*, 2351. [CrossRef]
15. Lozada-Ramírez, J.D.; Ortega-Regules, A.E.; Hernández, L.R.; de Parrodi, C. Spectroscopic and Spectrometric Applications for the Identification of Bioactive Compounds from Vegetal Extracts. *Appl. Sci.* **2021**, *11*, 3039. [CrossRef]