

Non-destructive analytical techniques for food quality determination

Zoltan Kovacs, Flóra Vitális, Mátyás Lukács and the
Department of Food Measurements and Process Control
Institute of Food Science and Technology

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FOOD Quality in Digital Age
(Grant no. 22230075)



● Mendel
● University
● in Brno
●



- **Founded on 1 February 2021** (successor to former Szent Istvan University, Kaposvar University and National Agricultural Research and Innovation Centre)
- **One of the largest agricultural-focused, multi-disciplinary higher education institutions in CEE**
- **provides world-class education in agriculture-related fields**
- **No. of institutes: 21**
- **No. of students: 13 621**
- **No. of international students: 2076** (from 102 countries!) **15.24%**
- **Number of PhD schools: 12**
- **Number of PhD students: 864** (international PhD students 382) **44.2%**
- **Number of academic staff: 1060**
- **Number of staff: 1412**
- **Languages of instruction: Hungarian, English**



— Szent Istvan Campus, Gödöllő – Head Office

— Buda Campus

- **Institute of Food Science and Technology**
- Institute of Horticultural Sciences
- Institute of Landscape Architecture, Urban Planning and Garden Art
- Institute of Plant Protection
- Institute for Viticulture and Oenology

— Kaposvár Campus

— Gyöngyös Campus

— Keszthely Campus



Research Centers and Departments of the Institute of Food Science and Technology

Research Centers	Departments
Center of Food Technology	Department of Postharvest Science, Trade, Supply Chain and Sensory Evaluation
	Department of Grain and Industrial Plant Processing
	Department of Livestock Product and Food Preservation Technology
	Department of Fruit and Vegetable Processing Technology
Center of Food Quality, Safety and Nutrition	Department of Food Chemistry and Analysis
	Department of Nutrition Science
	Department of Food Microbiology, Hygiene and Safety
	National Collection of Agricultural and Industrial Microorganisms
Center of Bioengineering and Process Control	Department of Food Process Engineering
	Department of Food Measurements and Process Control
	Bioengineering and Alcoholic Drink Technology

Institute of Food Science and Technology

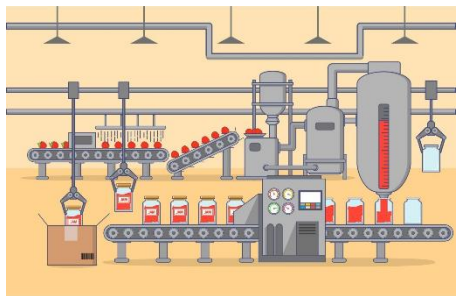
Research topics in focus III.

Across the entire Food Chain:

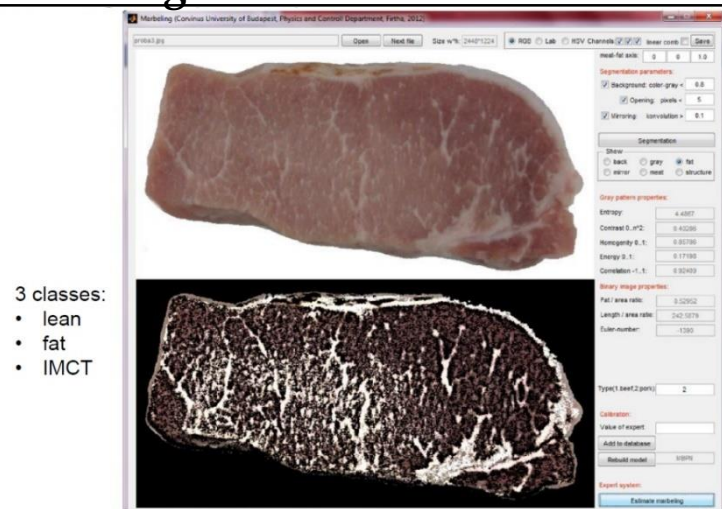
production of food industry raw materials – food processing – food trading

Measurement techniques, automatization, digitalization

- Robotization
- Data processing and evaluation, bigdata, chemometrics
- Digitalization, industry 4.0, IoT, M2M
- Modern measurement methods:
 - Non-destructive methods, contactless measurement technologies
 - Instrumental taste and odor sensing, NIR, hyperspectral, machine vision, etc.



Meat marbling measurement- machine vision



Quality assessment of food - NIRS

<p>Honey Adulterated with rice syrup, self-produced sugar syrup and high fructose content sugar syrup</p>	<p>Coffee drinks Classification and quantification of different origin Arabica and Robusta coffee</p>
<p>Meat mixture extracts Detection and quantification of pork in beef</p>	<p>Yogurt quality With different lactobacillus strains grown with different water</p>
<p>Mung bean juice Monitoring of germination and prediction of ascorbic acid</p>	<p>Tokaj wine Adulterated with grape must concentrate</p>
<p>Monilia (brown rot) on plums Early phase detection of <i>Monilia fructigena</i> infection on plums</p>	<p>Tomato concentrate Adulterated with starch, paprika seed powder, NaCl and sucrose</p>
<p>Paprika powder dilution Adulterated with corn flour at different concentration levels</p>	<p>Mineral water With different mineral content mixed and analyzed</p>



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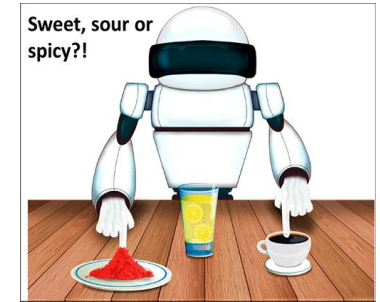


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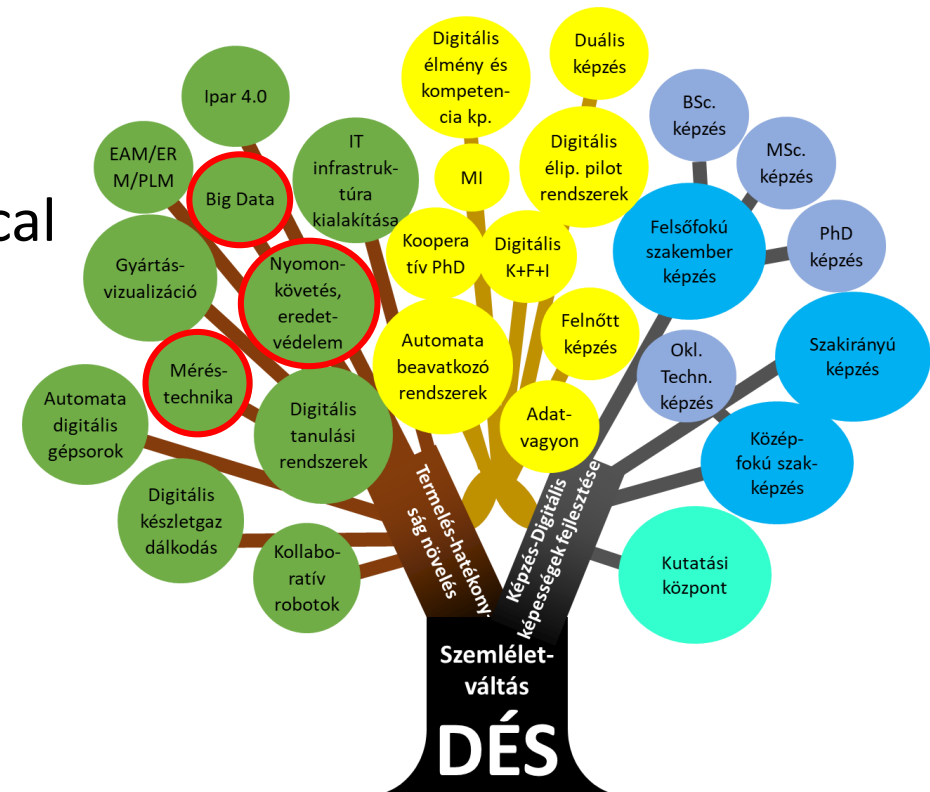


Importance of the non-destructive analytical techniques

- Precision farming and food production
- Increasing quantitative needs \leftrightarrow the standard quality food \leftrightarrow more economically
- Data driven decision making
- Rapid and precise quality determination of raw materials and food products
- Immense need for advanced and non-destructive analytical techniques in the agriculture and food industry:
 - ultrasonic measurement,
 - bioelectric properties, impedance spectroscopy,
 - acoustic measurement,
 - electronic tongue and electronic nose
 - machine vision,
 - **near-infrared spectroscopy** etc.



ACS Sens. 2018, 3, 11, 2375–2384



Hungary's digital food industry strategy (2022)⁷

Objectives of our research

- to develop and adapt non-destructive analytical measurement techniques
 - to explore and expand their application potentials
- for the objective characterization of food industry raw materials, intermediate and final products



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Near-infrared spectroscopy (NIR)

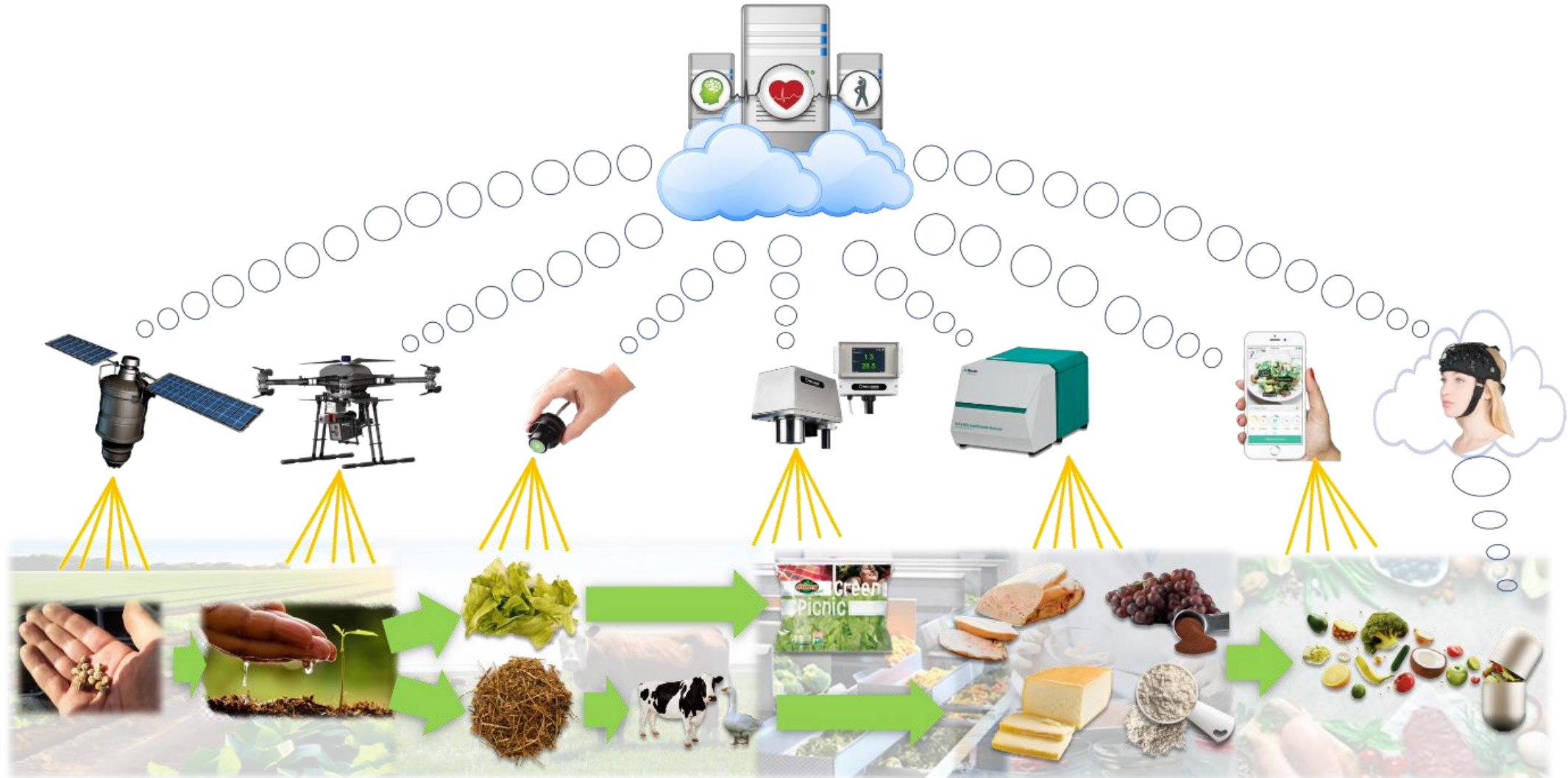


Food Science and Technology 31. 48-50.



Objective of this presentation

to provide insights about recent results of NIRS supporting the monitoring of food supply chain “from farm to fork” – and beyond.



Food quality assessment with NIR



Feed - TMR

Monitoring feed supplements with handheld near-infrared spectrometer



Cheese quality

Monitoring the ripening of cheese at various temperatures



Goose Liver

Determination of the Blood Content in Fattened Goose Liver



Honey

Detection of Heat Treatment on Unifloral Honeys



Whey Protein Powder

Detecting Adulterants in Whey Protein Powder



Chocolate

Authentication of chocolate based on geographical origin



Nutraceuticals

Quantitative evaluation of fruit extracts and fortified fruit juice



Meat mixture extracts

Detection and quantification of pork in beef



Mung bean juice

Monitoring of germination and prediction of ascorbic acid



Monilia (brown rot) on plums

Early phase detection of *Monilia fructigena* infection on plums



Coffee drinks

Classification and quantification of different origin Arabica and Robusta coffee



Yogurt quality

With different lactobacillus strains grown with different water



Tokaj wine

Adulterated with grape must concentrate



Tomato concentrate

Adulterated with starch, paprika seed powder, NaCl and sucrose



Mineral water

With different mineral content mixed and analyzed



Which research would you like to hear about?



Feed - TMR



Cheese quality



Goose Liver



Honey



Whey Protein Powder



Chocolate



Nutraceuticals



Meat mixture extracts



Mung bean juice



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Monitoring feed supplements with handheld near-infrared spectrometer – Objectives

In this study the goals were

- to determine how well various feed supplements can be identified in the feed and
- to monitor the impact of control (rich in saturated fatty acids) and experimental feed supplementations (rich in polyunsaturated fatty acids) on cheese quality



Supported by the project
2020-1.1.2-PIACI-KFI-2021-00245

Monitoring feed supplements with handheld near-infrared spectrometer – M&M

Dairy cows (n = 70 per groups) were fed in 2 trials (6-week of each)

Feed was total mixed rations (TMRs) containing

Trial 1: CTR1 – hydrogenated palm oil (rich in **saturated** fatty acids)

EXP1 – mixture of linseed and linseed oil
(rich in **polyunsaturated** fatty acids)

Trial 2: CTR2 – hydrogenated palm oil (rich in **saturated** fatty acids)

EXP2 – mixture of linseed and algae extract
(rich in **polyunsaturated** fatty acids)

Milk collected in last 2 weeks for cheese production
Fatty acid composition of TMR and cheese samples were measured.

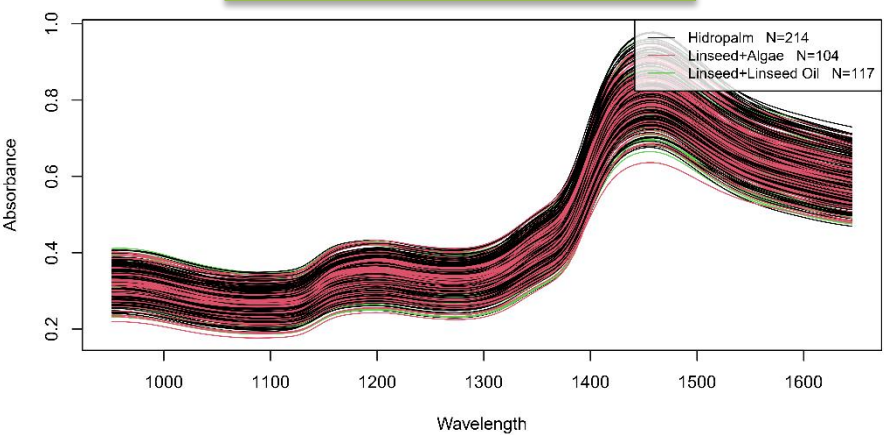
handheld VIAVI MicroNIR spectrometer to measure

- moist TMR samples and
- freshly cut surface of the cheese samples

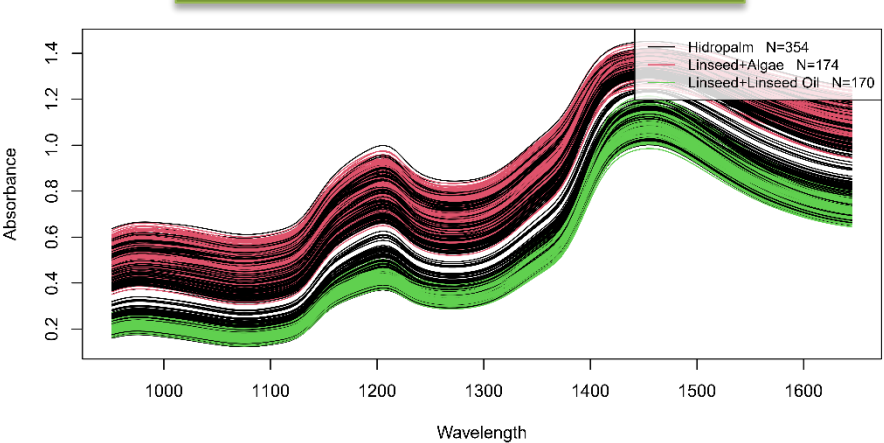


Monitoring feed supplements with handheld near-infrared spectrometer – Results

TMR raw spectra



Cheese raw spectra



The handheld MicroNIR provided good quality NIR spectra of TMR and cheese samples.

TMR SVM Discrimint Analysis

True Class	Predicted Class			
	Hidropalm	Linseed+Algae	Linseed+Linseed Oil	
	Hidropalm	84.6%	5.8%	9.6%
	Linseed+Algae	62.5%	34.2%	3.3%
Linseed+Linseed Oil	52.5%	3.3%	44.2%	

SVM exhibited good discrimination for the cheese samples of three groups: control and two experimental groups

SVM resulted weak discrimination of the two experimental TMR. Which can be due to the notable experimental variations in the feed and the pronounced inhomogeneity of the TMR mixture.

Cheese SVM Discrimint Analysis

True Class	Predicted Class			
	Hidropalm	Linseed+Algae	Linseed+Linseed Oil	
	Hidropalm	84.2%	7.1%	8.8%
	Linseed+Algae	12.6%	87.4%	
	Linseed+Linseed Oil	15.3%		84.7%



Dr. Zoltan Gillay

NIR2023 – P01.21

‘Farm to fork’ quality control during the production of a value-added dairy product



'Farm to fork' quality control during the production of a value-added dairy product



Zoltán Gillay^{1,2}, Tamás Tóth^{1,3}, Viktória Éles¹, Róbert Roszkos¹, Hajnalka Hingyi¹, Éva Csavajda¹, Zsolt Svarka⁴, Norbert Zelei⁴, Miklós Tóth⁵, George Bazar¹

¹ADEXGO Kft., Balatonfüred, Hungary

²Hungarian University of Agriculture and Life Sciences, Institute of Food Science and Technology, Budapest, Hungary

³Széchenyi István University, Agricultural and Food Research Center, Győr, Hungary

⁴Pálhalmi Agrospecial Kft., Dunajváros, Hungary

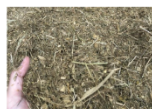
⁵University of Pécs, Faculty of Health Science, Pécs, Hungary



INTRODUCTION

With the growing awareness of consumers, the demand for health-promoting foods rich in bioactive ingredients is increasing. It has long been reported that the nutritional quality of dairy products can be improved through the feeding of cows.

TMR feed refers to "Total Mixed Ration feed". TMR feed is to feeding livestock, especially dairy and beef cattle, where various feed ingredients such as grains, forages, protein and fat supplements, minerals, and vitamins are thoroughly mixed to create a balanced and nutritionally complete diet for the animals.

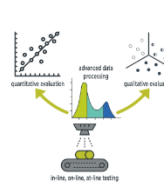


AIM OF THE STUDY

Our vision is to overcome the drawbacks of traditional methods used to analyze the nutritional content of feed and food. These drawbacks mainly include the high costs and time-consuming nature of the analysis.

To tackle this issue, we have tested an affordable, and quick NIR method that can work alongside or replace the traditional methods.

Our scientific goal was to determine how well various feed supplements can be identified in feed and to investigate their impact on cheese production.



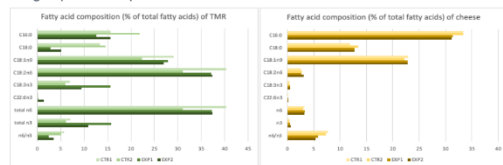
MATERIALS AND METHODS

TMRs containing control feed supplementations (rich in saturated fatty acids) and experimental feed supplementations (rich in polyunsaturated fatty acids) were fed to dairy cows (n = 70 per groups) in 2 trials (6-week of each):

- Trial 1: CTR1 – hydrogenated palm oil
- EXP1 – mixture of linseed and linseed oil
- Trial 2: CTR2 – hydrogenated palm oil
- EXP2 – mixture of linseed and algae extract



On the last 2 weeks of the trials, the collected milk of the groups was used for cheese production. The fatty acid composition of the TMR and cheese samples of the groups showed pronounced differences.



The moist TMR samples and the freshly cut surface of the cheese samples were scanned with a handheld VIAVI MicroNIR spectrometer.

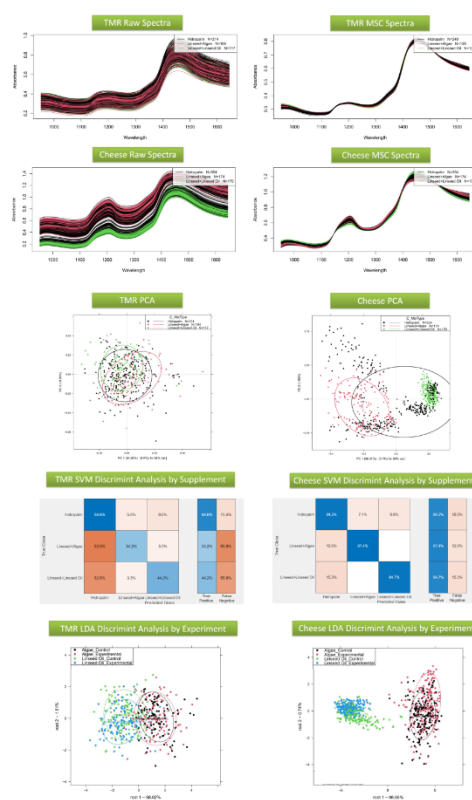


Data analysis was performed in R Project: <https://github.com/bpollner/aquap2> by Bernhard Pollner

ACKNOWLEDGEMENTS

The contribution of our colleagues, Prof. Hedvig Fébel, Prof. András Szabó, and Ali Omeralfaroug in fatty acid analysis (at MATE), Dr. Erika Hanczné Lakatos and Dr. Viktória Kapcsándi in cheese production (at Széchenyi University) is highly acknowledged. The project (2020-1.1.2-PIACI-KFI-2021-00245) has been implemented with the support provided by the Ministry of Innovation and Technology of Hungary from the National Research, Development and Innovation Fund.

RESULTS



DISCUSSION AND CONCLUSIONS

The handheld MicroNIR provided good quality NIR spectra of TMR and cheese samples. We utilized PCA to assess natural separation and the outliers. While PCA indicated clear separation of the cheese samples by experiments, and showed clustering of samples by the fed diets, it failed to show differences of the TMR samples.

The employed Support Vector Machine (SVM) discriminant analysis exhibited reasonable discrimination capabilities for the cheese. The control cheese of the two trials formed one group, while the two experimental groups of the two trials were found to be identifiably different according to the applied diet.

The absence of accurate discrimination of the TMR samples likely originates from notable experimental variations in the supplement-free feed and the pronounced inhomogeneity of the TMR mixture. Ultimately, the supplements' addition did not impact NIR spectra significantly.

Supported by the project
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With different mineral content mixed and analyzed



Monitoring the ripening of different cheese at various temperatures – Objectives

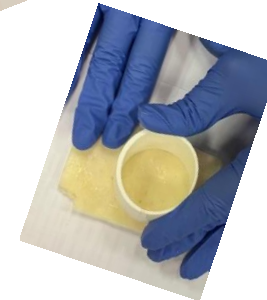
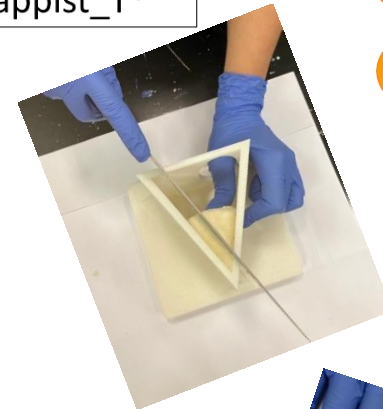
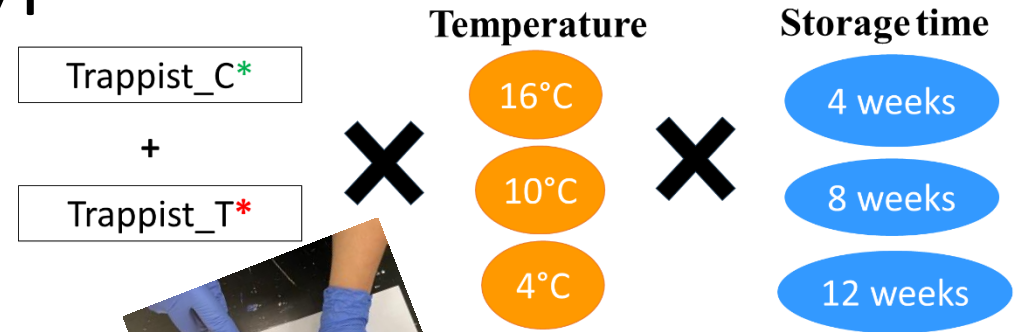
The objectives of this study were

- to determine the potential of NIRS in monitoring the ripening of Trappist style cheese at different temperatures and
- to detect differences in the ripening process caused by the type of diet fed to the cows

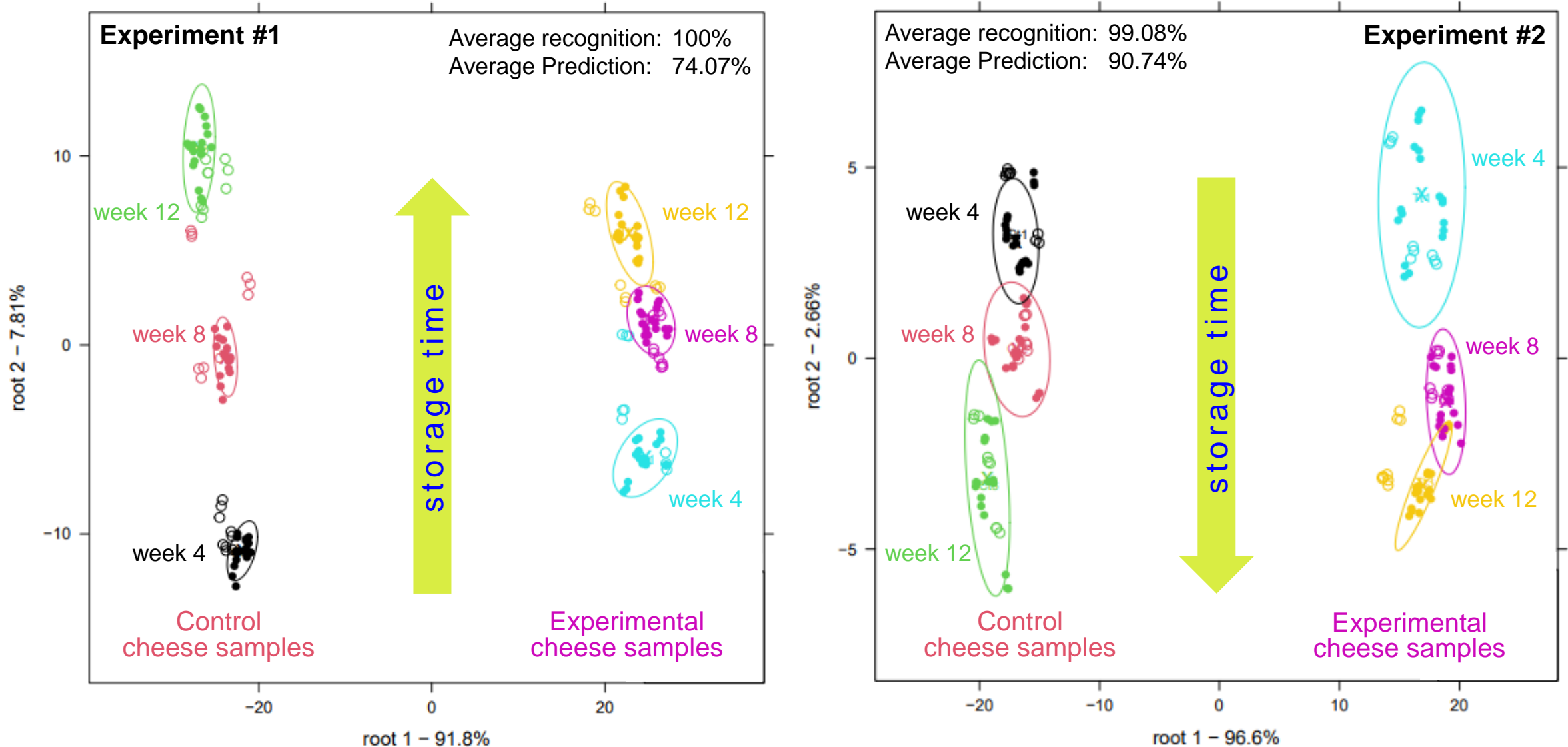


Monitoring the ripening of different cheese at various temperatures – M&M

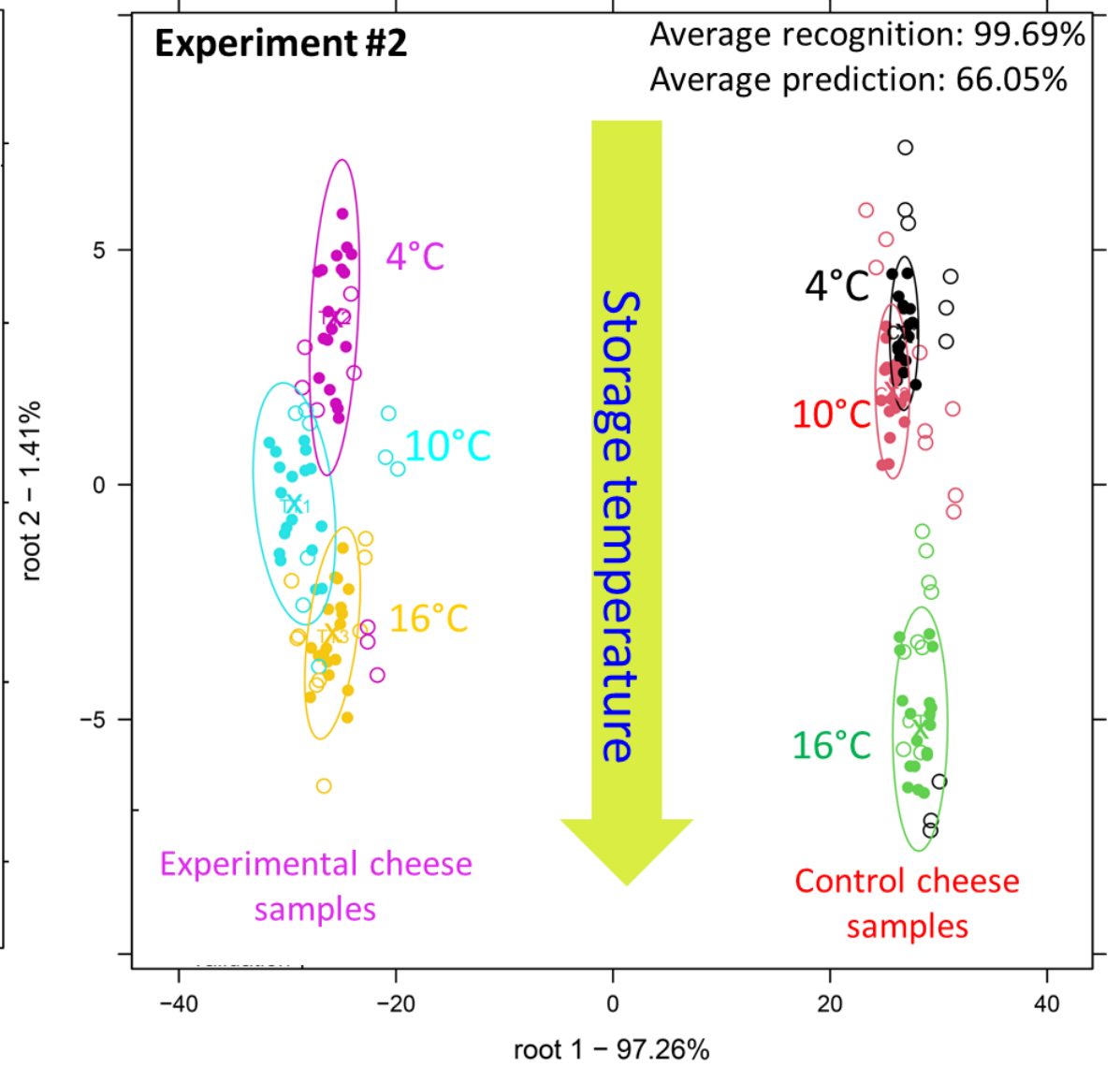
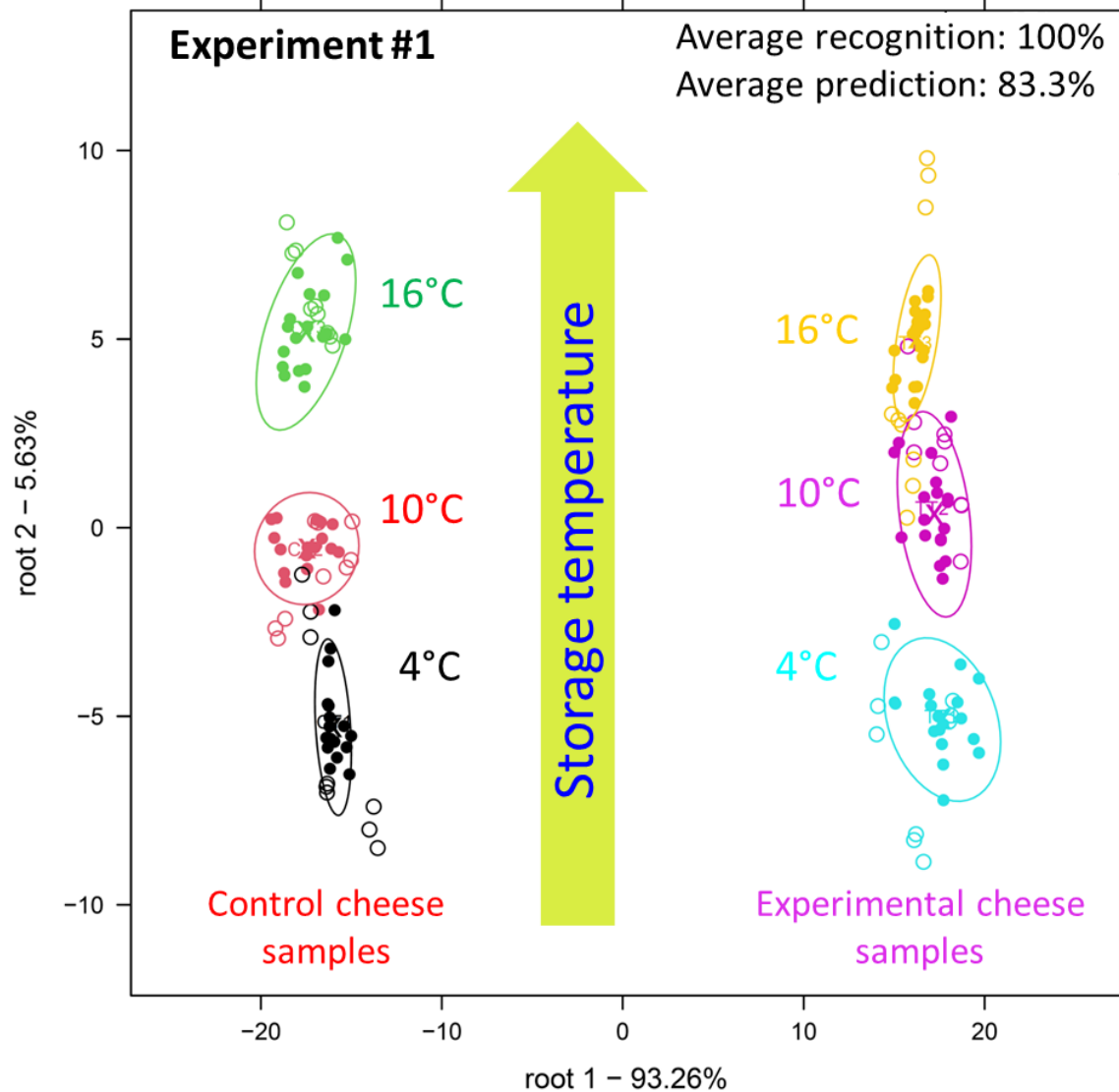
- Trappist style cheese made of cow milk from two different diet (in two repeated experiments):
 - **control**: hydrogenated palm oil (rich in **saturated** fatty acids)
 - **experiment**: mixture of linseed and algae extract (rich in **polyunsaturated** fatty acids)
- Samples stored in triplicates
 - at different temperatures: 16°C, 10°C, 4°C and
 - for 0, 4, 8, and 12 weeks
- Diffuse reflectance NIR spectra acquired with the benchtop XDS rapid content analyzer using a circular glass cuvette
- Freshly cut surface of a sample thickness of 1 cm was scanned
- NIRS data was evaluated with principal component analysis (PCA) and PCA-based linear discriminant analysis (PCA-LDA)



Monitoring the ripening of different cheese at various temperatures – Results – LDA time



Monitoring the ripening of different cheese at various temperatures – Results – LDA temperature





Mariem Majadi

NIR2023 – P01.36

Monitoring the ripening of cheese at various temperatures by means of near-infrared spectroscopy

Monitoring the ripening of cheese at various temperatures by means of Near Infrared Spectroscopy

Mariem Majadi¹ ; Zoltan Kovacs¹ ; Zoltan Gillay^{1,2} ; Tamas Toth^{2,3} ; George Bazar²

¹ Department of Measurements and Process Control, Hungarian University of Agriculture and Life Sciences, Budapest, Hungary

² ADEXGO Kft, Balatonfured, Hungary

³ Szechenyi Istvan University, Agricultural and Food Research Center, Gyor, Hungary

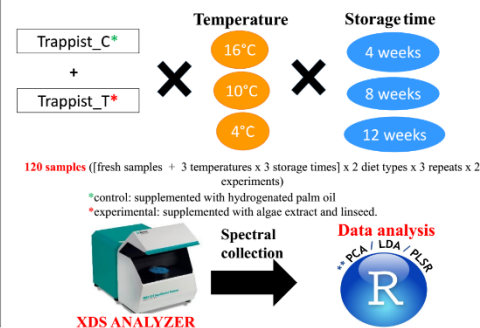
Introduction

- The fatty acid composition depends on many different factors such as breed, season, lactation stage, lactation number, age of dairy cows, geographical location. Most important factor **is the diet**, which is highly responsible for the variance in cow milk fat.
- Lowering intake of saturated fatty acids (FA) and making alterations in the FA profile of the fat consumed is receiving current attention to increase the nutritional value.
- **A need** to evaluate these new products by not only its physicochemical aspects but also by its sensory and general composition.
- Cheese is an important source of a wide variety of biologically active substances among which specific FAs are of utmost importance.
- Lipolysis is one of important biochemical event occurring during cheese ripening which results directly in the formation of flavor compounds by liberating free fatty acids (FFA), which may directly contribute to cheese flavor and also serve as substrates for further reactions producing highly flavored catabolic end products.

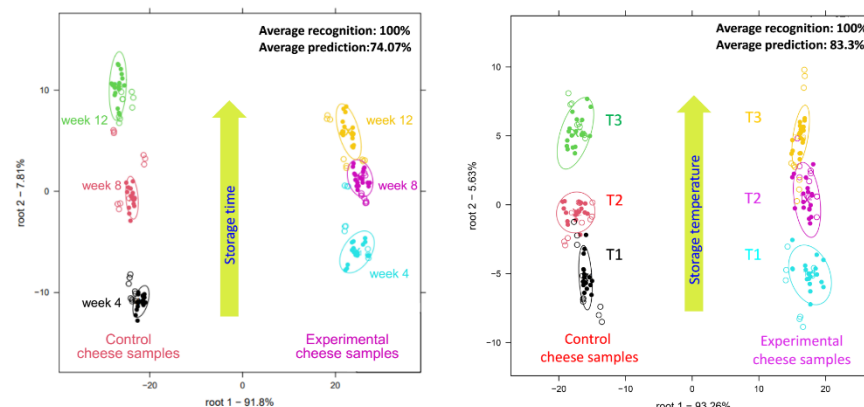
AIM

To determine the potential of NIRS in monitoring the ripening of Trappist style cheese at different temperatures, and to detect differences in the ripening process caused by the type of diet fed to the cows

Materials and Methods



Results & conclusions



NIRS discrimination of control and experimental Trappist cheese samples stored at different temperature for 4, 8 and 12 weeks in two independent experiments

➤PCA-LDA revealed a good discrimination between samples at different storage time and temperature.

➤NIRS coupled with chemometrics is an efficient technique for monitoring cheese quality variations during ripening and to detect alterations related to the cows' diet.

The technology may be applied:

(1) in quality control to confirm the declared storage temperature and ripening duration.

(2) in product development to monitor the differences in the ripening process caused by the altered composition of milk.

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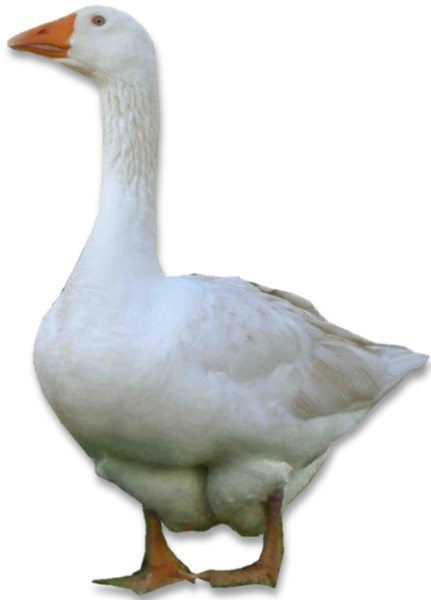
Mineral water

With different mineral content mixed and analyzed



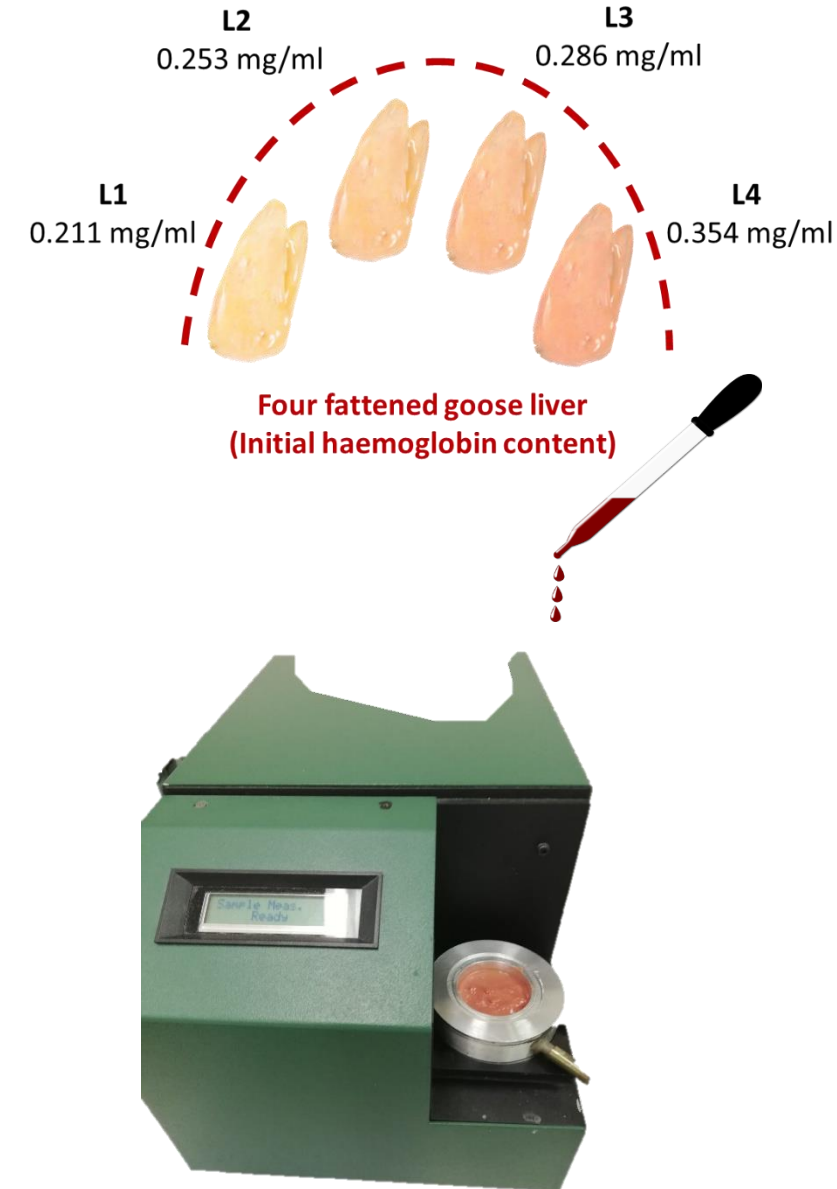
Determination of the Blood Content in Fattened Goose Liver – Objectives

In this study the objective was to determine the applicability of near-infrared spectroscopy to evaluate the blood content and hemoglobin concentration in goose liver

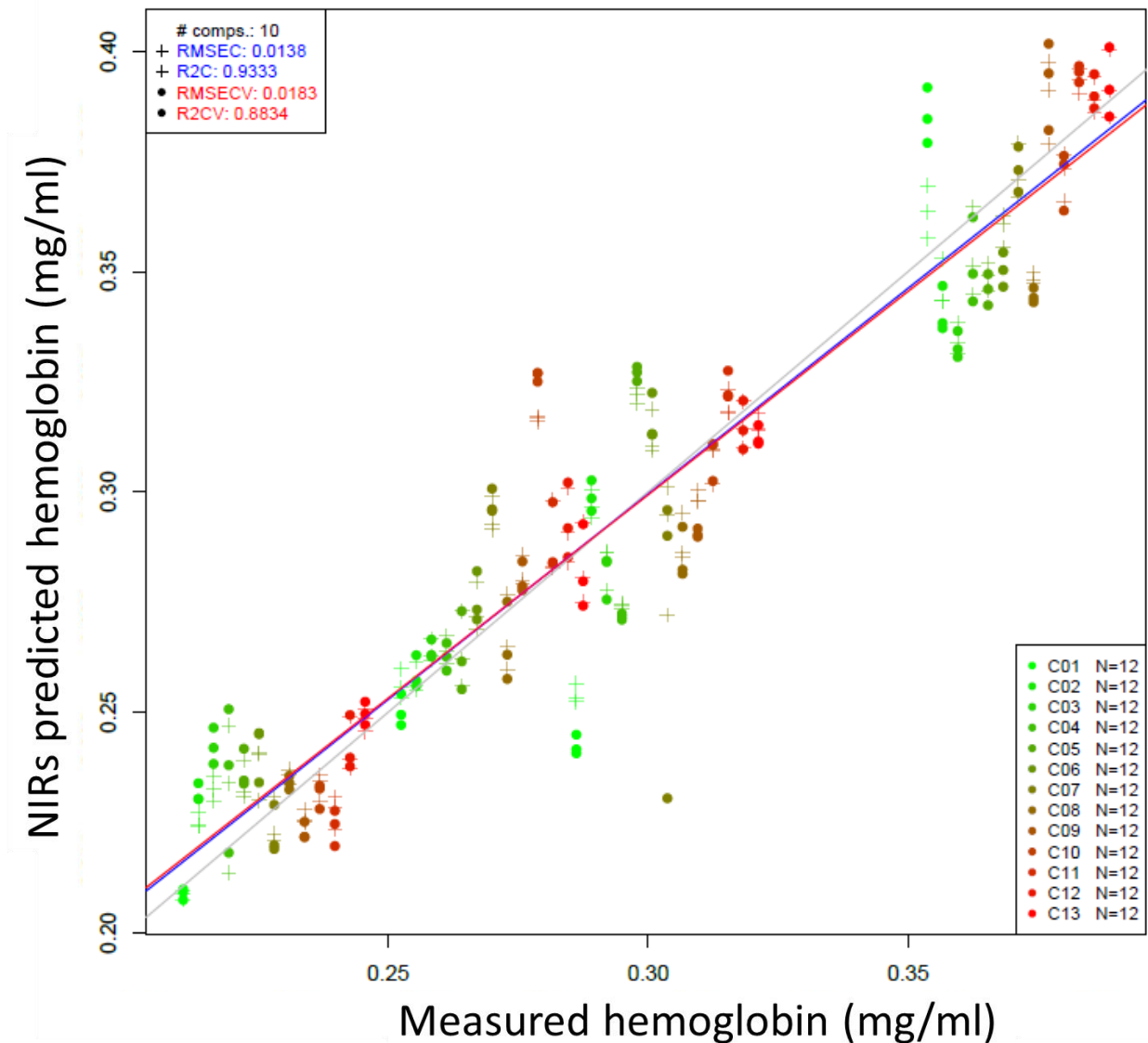


Determination of the Blood Content in Fattened Goose Liver – M&M

- Fattened goose liver from four animals
- Goose blood addition at 13 different concentrations between 0 and 5.5% by 0.5% steps
- Determination of the hemoglobin concentration by Drabkin method
- Measurement of the color of the samples with the Vitalis Colorimeter
- Spectral acquisition of the samples with the MetriNIR benchtop spectrometer in diffuse reflectance mode
- Principal Component Analysis and Partial Least Square Regression to develop models for the four livers together and individually to visualize multidimensional patterns and build prediction models for the blood content determination, respectively



Determination of the Blood Content in Fattened Goose Liver – Results



Prediction of hemoglobin concentration in liver samples

Goose Liver	N	LV	R _c ²	RMSEC (mg/ml)	R _{cv} ²	RMSECV (mg/ml)
L1	39	3	0.984	0.001	0.942	0.003
L2	39	3	0.955	0.002	0.865	0.004
L3	39	3	0.980	0.002	0.929	0.003
L4	39	3	0.972	0.002	0.937	0.003
General model	153	10	0.933	0.014	0.883	0.018

Prediction of color coordinate components (L, a, b, E) of liver samples

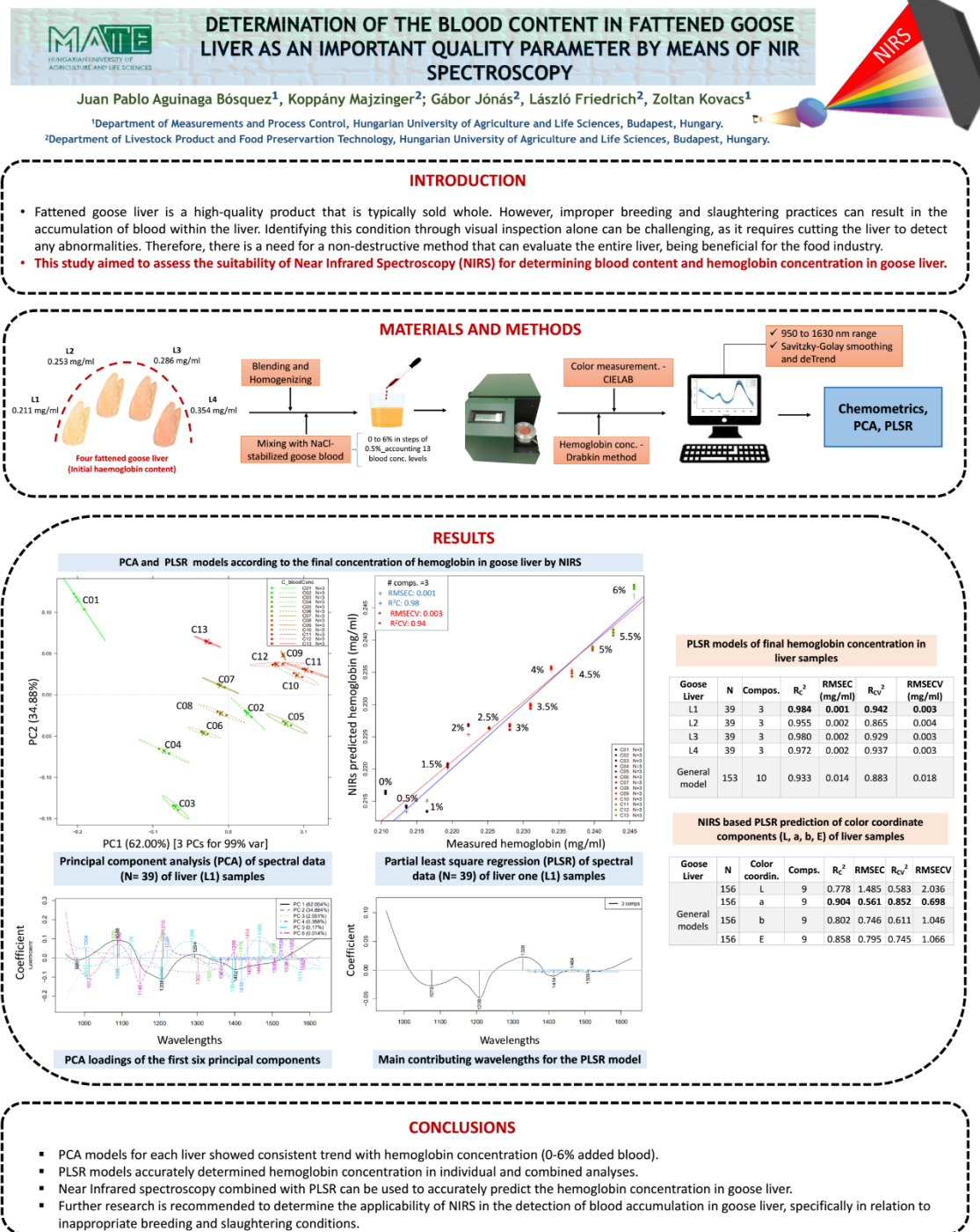
Goose Liver	N	Color coord.	LV	R _c ²	RMSEC	R _{cv} ²	RMSECV
General models	156	L	9	0.778	1.485	0.583	2.036
	156	a	9	0.904	0.561	0.852	0.698
	156	b	9	0.802	0.746	0.611	1.046
	156	E	9	0.858	0.795	0.745	1.066 ²⁶



Juan Pablo Aguinaga
Bósquez

NIR2023 – P01.01

Determination of the Blood Content in Fattened Goose Liver as an Important Quality Parameter by means of NIR Spectroscopy



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Effect of Heat Treatment on the Spectral Pattern of Unifloral Honeys – Objectives

The objectives of this study were to investigate the effect of heat treatment on

- HMF (5-hydroxymethylfurfural) content and
- the spectral pattern of unifloral honeys



Effect of Heat Treatment on the Spectral Pattern of Unifloral Honey – M&M

- Honey samples:
 - acacia (RP-Robinia pseudoacacia),
 - bastard indigo (AF-Amorpha fruticosa) and
 - sunflower (HA-Helianthus annuus) honey
- Samples were heated:
 - at temperatures: 40°C, 60°C, 80°C and 100°C
 - for 60, 120, 180 and 240 minutes
- Determination of the HMF concentration
- Handheld spectrometer in transfectance (0.4 mm layer thickness)
- NIRS data was evaluated with principal component analysis (PCA) and PCA-based linear discriminant analysis (PCA-LDA)



Effect of Heat Treatment on the Spectral Pattern of Unifloral Honeys – results - HMF

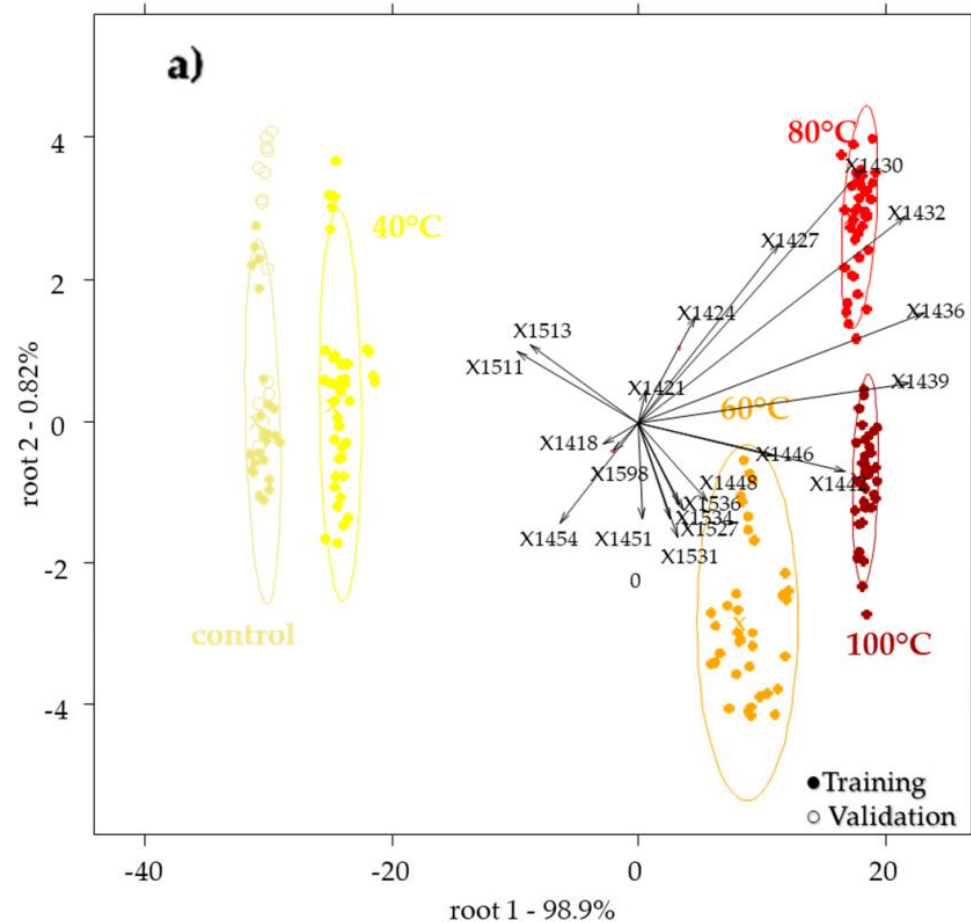
		Hydroxymethylfurfural Content, mg/kg				
		Control	40 °C	60 °C	80 °C	100 °C
Sunflower	Control	18.5 ± 0.3				
	60 min		20.2 ± 1.5 ^{aA}	16.2 ± 1 ^{aA}	17.6 ± 0.2 ^{aA}	40.3 ± 0.8 ^{aB*}
	120 min		17.3 ± 1.3 ^{aA}	20.5 ± 0.7 ^{bB}	31.8 ± 1.3 ^{bC*}	155.1 ± 2.7 ^{bD*}
	180 min		18.4 ± 1.6 ^{aA}	19.9 ± 1.8 ^{bA}	37.2 ± 0.6 ^{cB*}	241.5 ± 7.4 ^{cC*}
	240 min		17.5 ± 1.4 ^{aA}	19.5 ± 2 ^{abA}	52 ± 2.7 ^{dB*}	463.6 ± 28.3 ^{dC*}
Bastard indigo **	Control	14.7 ± 1.6				
	60 min		14.1 ± 2.8 ^{aAB}	18 ± 2.3 ^{abB}	11.9 ± 1.1 ^{aA}	16.7 ± 0.9 ^{aAB}
	120 min		15.1 ± 3.5 ^{aA}	15.8 ± 0.6 ^{abA}	14.3 ± 1 ^{bA}	81.4 ± 4 ^{bB*}
	180 min		15.7 ± 1.1 ^{aA}	21.1 ± 3.5 ^{bA}	19.8 ± 0.6 ^{cA}	146.4 ± 2.3 ^{cB*}
	240 min		12.9 ± 1.4 ^{aA}	13.7 ± 1.3 ^{aA}	28.2 ± 1.1 ^{dB}	306 ± 17.8 ^{dC*}
Acacia	Control	7.0 ± 0.4				
	60 min		9.1 ± 1.3 ^{aA}	7.7 ± 0.3 ^{aA}	8 ± 0.4 ^{aA}	16.1 ± 1.7 ^{aB*}
	120 min		8 ± 0.6 ^{aA}	8.8 ± 1.4 ^{aA}	13.3 ± 0.9 ^{bB*}	44.7 ± 4.3 ^{bC*}
	180 min		8.6 ± 1 ^{aA}	9.6 ± 0.3 ^{aA}	12.2 ± 0.8 ^{bB*}	89.1 ± 2.8 ^{cC*}
	240 min		10 ± 1.1 ^{aA}	9.6 ± 0.9 ^{aA}	18.8 ± 2.4 ^{cB*}	211.6 ± 5 ^{dC*}

Letters represent the significant differences between the samples based on the results of an ANOVA test and pairwise comparisons at $p < 0.05$: lowercase letters (a,b,c,d) stand for the differences between time intervals (columns) within a temperature level; capital letters (A,B,C,D) are for the differences between temperature levels within time intervals (rows); * are for a significantly different level compared to the control sample. ** Results of bastard indigo were previously presented at a conference [29].

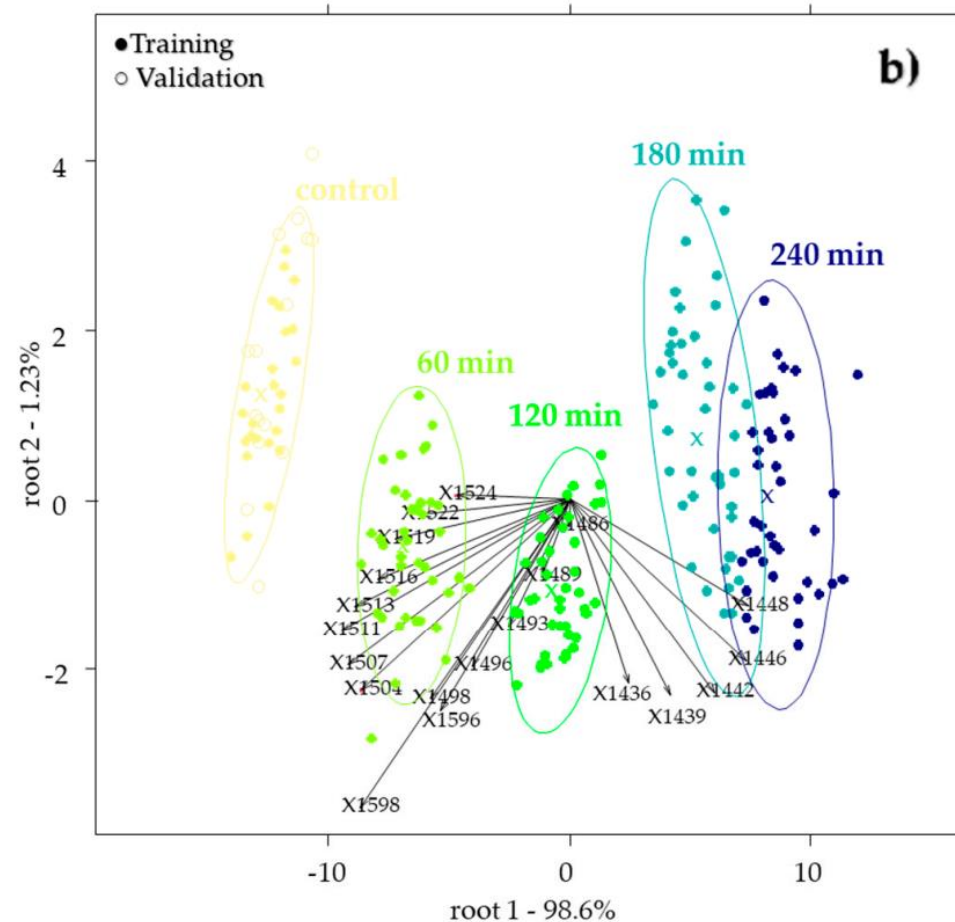
Effect of Heat Treatment on the Spectral Pattern of Unifloral Honeys – results

PCA-LDA score plot of the NIR data of
sunflower honey

temperature level



time interval



Food quality assessment with NIR



Feed - TMR

Monitoring feed supplements with handheld near-infrared spectrometer



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Monitoring the ripening of cheese at various temperatures



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Determination of the Blood Content in Fattened Goose Liver



Honey

Detection of Heat Treatment on Unifloral Honeys



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Detecting Adulterants in Whey Protein Powder



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Authentication of chocolate based on geographical origin



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Adulterated with grape must concentrate



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Adulterated with starch, paprika seed powder, NaCl and sucrose



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With different mineral content mixed and analyzed



Detecting Adulterants in Whey Protein Powder

– Objectives

The objectives of this study were

- to develop models to classify and predict low concentrations of **urea, glycine, taurine, and melamine** in **whey protein powder** using a **benchtop and handheld** spectrometer
- to compare the accuracy of the models achieved by scanning the samples through optical **glass** and commercial **low-density polyethylene (LDPE) plastic bag** using handheld spectrometer



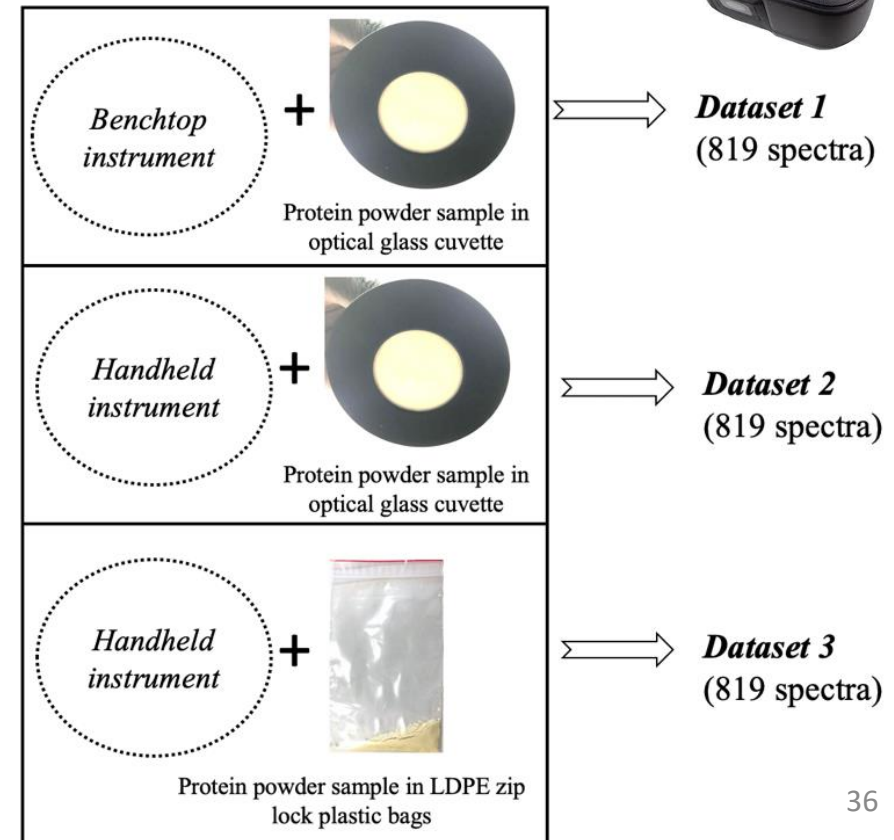
Detecting Adulterants in Whey Protein Powder

– M&M

- Whey protein powder
- Adulterants
 - taurine, glycine, melamine
 - single adulterant mixtures (U, G, T, M),
 - dual mixtures (GT, GU, GM, TU, TM, UM) and
 - multiple mixtures (GTU, GTM, GUM, TUM, GTUM)
 - total 15 different mixture combinations
 - total adulteration level 0.5, 1.0, 1.5, 2.0%, 2.5%, 3% w/w
 - triplicates of each mixture, resulting in 273 samples
- Diffuse reflectance spectra of powders collected by **benchtop and handheld NIR devices**
- Principal component analysis based linear discriminant analysis (PCA-LDA) for classification of type and level of adulteration
- Partial least squares regression (PLSR) for prediction of the level of adulteration



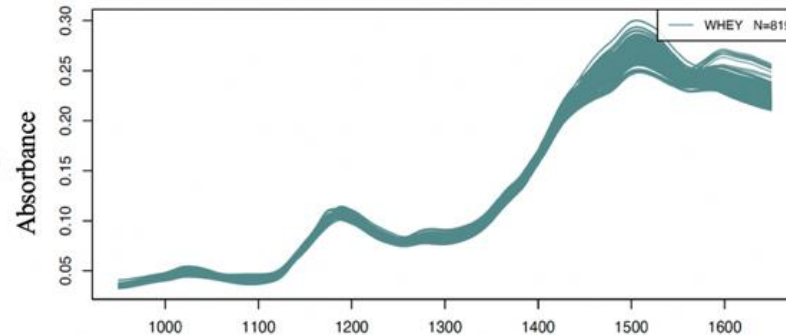
MetriNIR



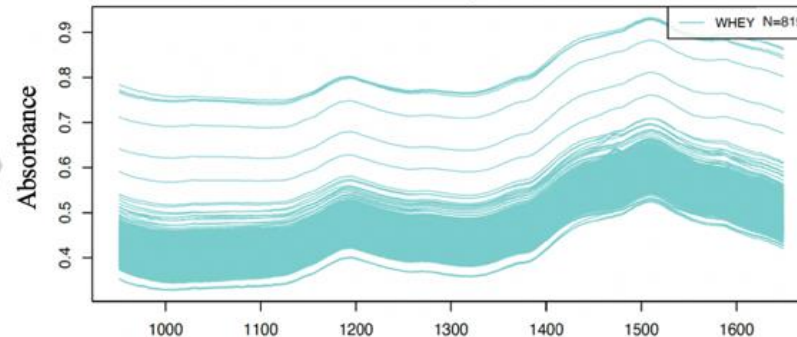
Detecting Adulterants in Whey Protein Powder – Results – PCA-LDA



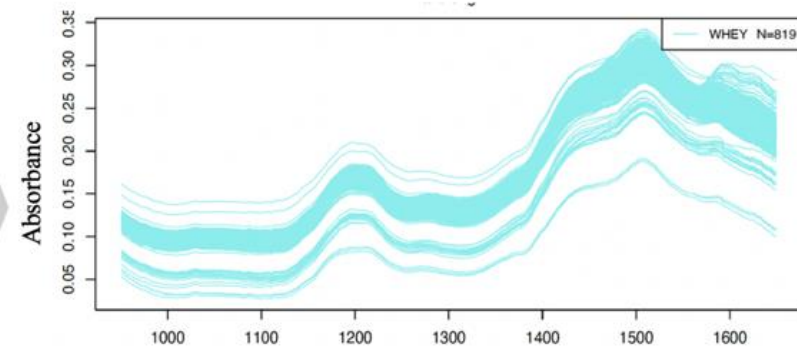
A) Benchtop spectrometer and optical glass



B) Handheld spectrometer and optical glass

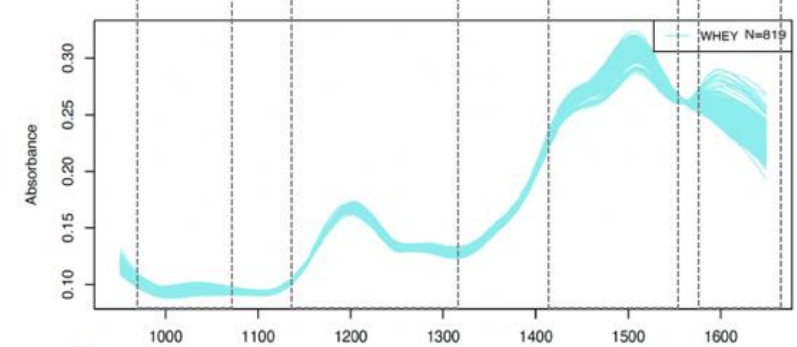
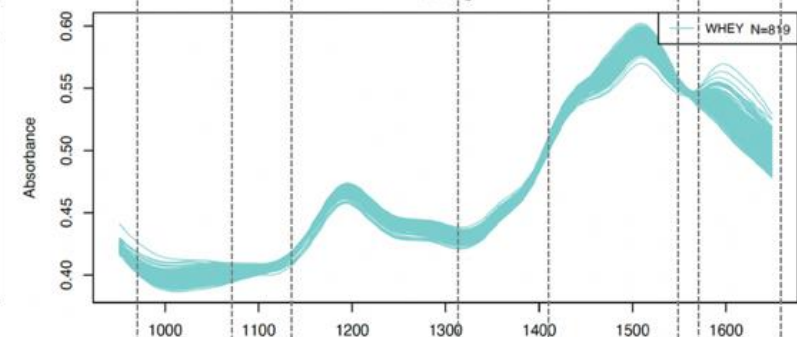
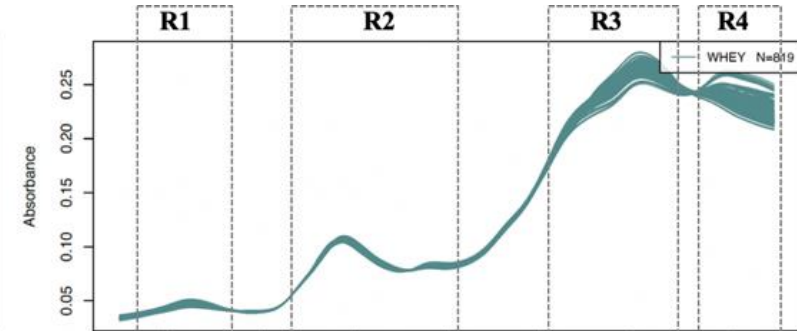


C) Handheld spectrometer and LDPE plastic bag



Wavelength (nm)

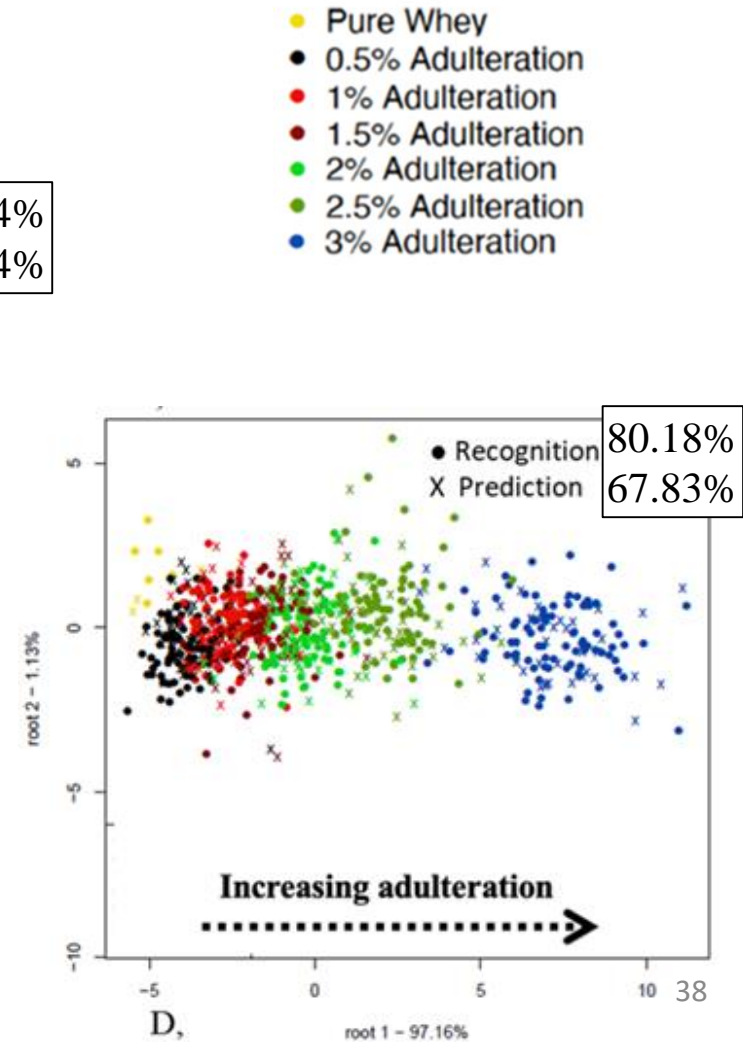
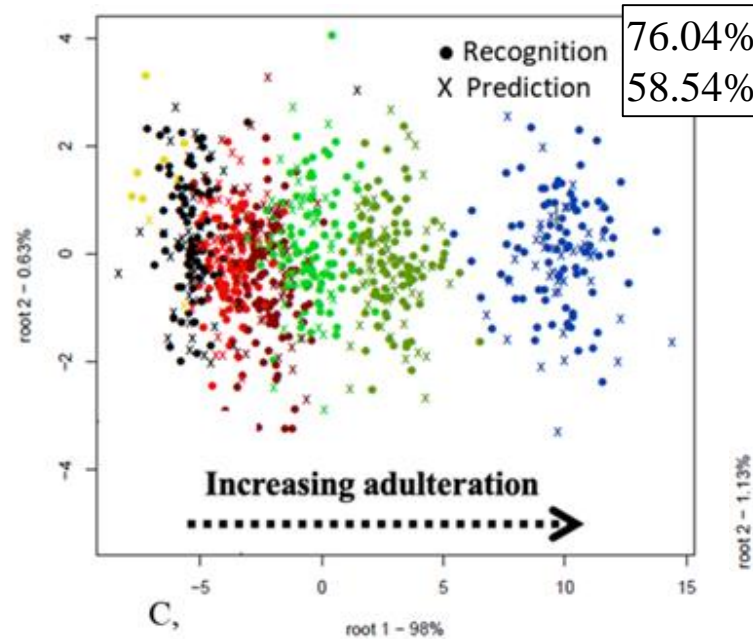
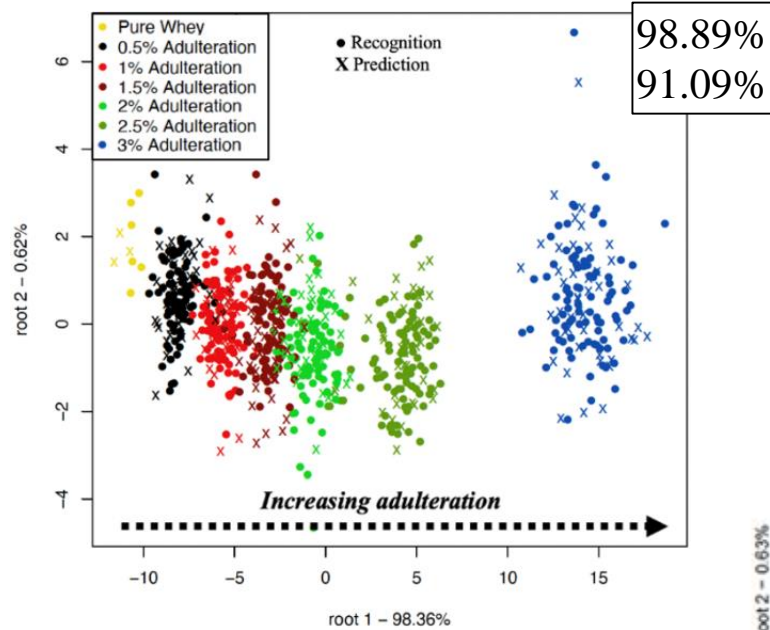
Raw spectra



Wavelength (nm)

Sgolay+MSC

Detecting Adulterants in Whey Protein Powder – Results – PCA-LDA – adulteration levels



Detecting Adulterants in Whey Protein Powder – Results – PLSR – adulteration content



		R^2	RMSEC (g/100g)	R^2_{CV}	RMSECV (g/100g)	R^2_{Pred}	RMSEP (g/100g)
benchtop	Urea	0.94	0.21	0.94	0.21	0.92	0.23
	Glycine	0.91	0.64	0.90	0.65	0.85	0.82
	Taurine	0.92	0.97	0.92	0.99	0.90	1.14
	Melamine	0.90	0.18	0.90	0.19	0.86	0.21
handheld	Urea	0.89	0.26	0.89	0.27	0.91	0.25
	Glycine	0.77	0.98	0.75	1.01	0.75	1.03
	Taurine	0.84	1.39	0.82	1.47	0.85	1.37
	Melamine	0.85	0.23	0.83	0.24	0.87	0.21
Handheld and LDPE plastic bag	Urea	0.93	0.22	0.92	0.23	0.91	0.25
	Glycine	0.79	0.96	0.77	1.03	0.73	1.11
	Taurine	0.78	1.65	0.75	1.77	0.72	1.84
	Melamine	0.79	0.27	0.78	0.28	0.75	0.29

Detecting Low Concentrations of Nitrogen-Based Adulterants in Whey Protein Powder Using Benchtop and Handheld NIR Spectrometers and the Feasibility of Scanning through Plastic Bag

John-Lewis Zinias ¹, Balkis Aouadi ¹, Mátyás Lukács ², Zsannett Bodor ¹, Flóra Vítális ¹, Biborka Gillay ¹, Zoltan Gillay ¹, László Friedrich ³ and Zoltan Kovacs ^{1,*}

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² Department of Quality Management, BioTech USA Ltd, 1033 Budapest, Hungary; lukacs.matyas@biotechusa.com

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Academic Editors: Emanuela Zanardi and Lenka Husáková

Received: 30 April 2020; Accepted: 25 May 2020; Published: 28 May 2020

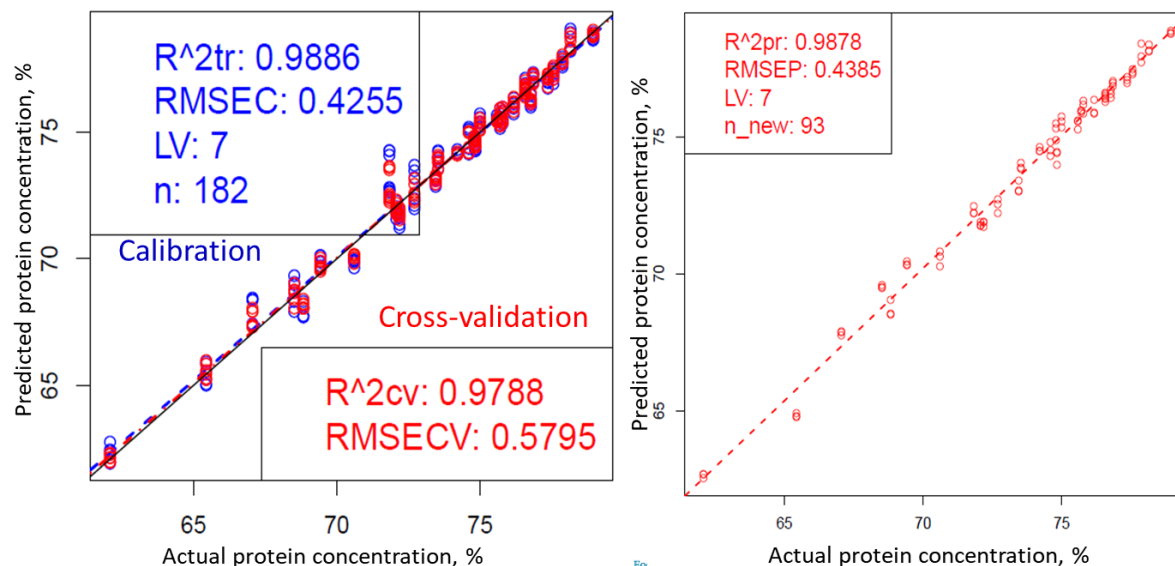
Abstract: Nitrogen-rich adulterants in protein powders present sensitivity challenges to conventional combustion methods of protein determination which can be overcome by near infrared spectroscopy (NIRS). NIRS is a rapid analytical method with high sensitivity and non-invasive advantages. This study developed robust models using benchtop and handheld spectrometers to predict low concentrations of urea, glycine, taurine, and melamine in whey protein powder (WPP). Effectiveness of scanning samples through optical glass and polyethylene bags was also tested for the handheld NIRS. WPP was adulterated up to six concentration levels from 0.5% to 3% w/w. The two spectrometers were used to obtain three datasets of 819 diffuse reflectance spectra each that were pretreated before linear discriminant analysis (LDA) and regression (PLSR). Pretreatment was effective and revealed important absorption bands that could be correlated with the chemical properties of the mixtures. Benchtop NIR spectrometer showed the best results in LDA and PLSR but handheld NIR spectrometers showed comparatively good results. There were high prediction accuracies and low errors attesting to the robustness of the developed PLSR models using independent test set validation. Both the plastic bag and optical glass gave good results with accuracies depending on the adulterant of interest and can be used for field applications.

Keywords: protein-supplements; chemometrics; fingerprinting; near-infrared; spectroscopy optical-glass; commercial LDPE plastic bag; benchtop; handheld

1. Introduction

Proteins are important nutritional requirements with a recommended dietary reference intake (DRI) of 0.8 g of protein per kilogram of body weight [1]. This amounts to 56 g per day for adults with no rigorous daily activities. People engaged in extensive exercises, however, consume more protein due to their intense energy requirements as a result of physical activity. The fast pace style of

Quantification of multiple adulteration of whey protein concentrate by NIRS



Near infrared spectroscopy as an alternative quick method for simultaneous detection of multiple adulterants in whey protein-based sports supplement

Matyas Lukacs^a, George Bazar^b, Bernhard Pollner^c, Raphael Henn^d, Christian G. Kirchler^d, Christian W. Huck^d, Zoltan Kovacs^{a,*}

^a Department of Physics and Control, Faculty of Food Science, Szent Istvan University, 14-16 Somlói Str., 1118 Budapest, Hungary

^b Department of Nutritional Science and Production Technology, Faculty of Agricultural and Environmental Sciences, Kaposvár University, 40 Gábor Sándor Str., 7400 Kaposvár, Hungary

^c Department for Hygiene and Medical Microbiology, Medical University of Innsbruck, Christoph-Probst-Platz 1, Innrain 52, 6020 Innsbruck, Austria

^d Institute of Analytical Chemistry and Radiochemistry, CCB-Center for Chemistry and Biomedicine, Leopold Franzens University, Innrain 80/82, 6020 Innsbruck, Austria

ARTICLE INFO

Keywords:
Sports supplement
Chemometrics
Rapid protein evaluation
Amino acid-spiking
Limit of detection
Chemical compounds studied in this article:
L-tyrosine (PubChem CID 1123)
L-histidine (PubChem CID 6274)
Urea (PubChem CID 1176)

ABSTRACT

Results of the Dumas method and near infrared (NIR) spectroscopy were compared to detect and quantify adulterants (urea, L-tyrosine, L-histidine) and to evaluate protein content in whey protein powder samples. Three different NIR regions were compared using principal component analysis and partial least squares regression models determining the quantity of each component. Calibration models were cross-validated with leave-one-sample-out method and prediction models were tested using data of independent days. Protein values by the Dumas method showed significant differences to the actual protein content in most cases. Predictive models had a minimum R^2 value of 0.97 in all cases and the lowest average prediction error was 0.16%, 0.41% and 0.23% with the respective limit of detection minimum values of 0.09%, 0.22% and 0.15% for urea, tyrosine and histidine. Results show, that NIR spectroscopy coupled with chemometrics can be a useful tool to detect multiple nitrogen-based adulteration simultaneously in whey protein powders.



Food quality assessment with NIR



Feed - TMR

Monitoring feed supplements with handheld near-infrared spectrometer



Cheese quality

Monitoring the ripening of cheese at various temperatures



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Co-funded by the
Erasmus+ Programme
of the European Union

-
- Visegrad Fund
-
-

E³UDRES²

Engaged and Entrepreneurial European University as
Driver for European Smart and Sustainable Regions

FOOD QUALITY IN DIGITAL AGE

Authentication of chocolate based on geographical
origin



Problem



Adulteration

Different contents of cocoa butter

**Substitution of an expensive
raw material for a cheaper
one**

Mixed



**Geographical
origin**

**Increase in
amount and
weight**

Improve sensory properties

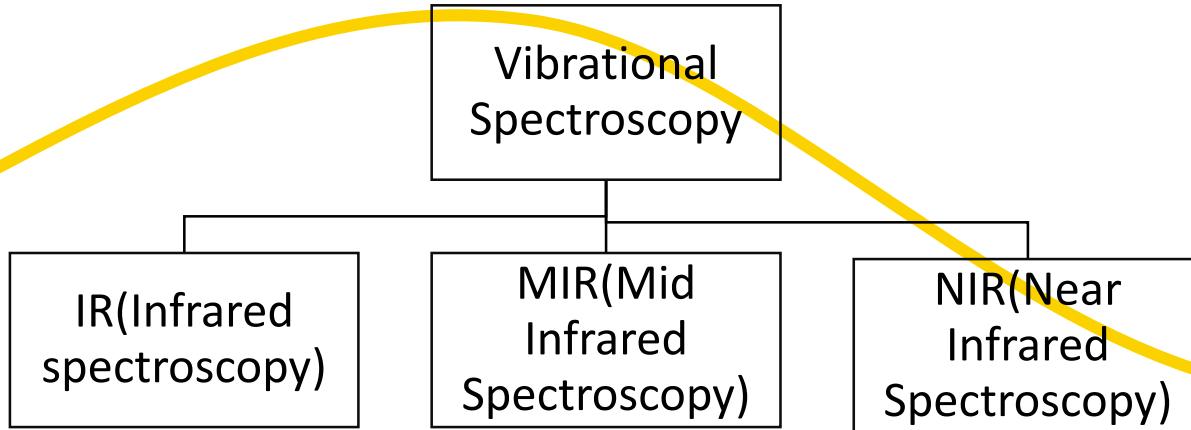
MATERIALS

Chocolate samples:

- Belcolade (100%) – 55% cocoa dry matter
 - Callebaut (100%) – 55% cocoa dry matter
 - Mixtures: Belcolade + Callebaut (10 – 90%)
- 3 replicates each sample group



NIR (Near Infrared Spectroscopy)



- IR Spectroscopy is based on an interaction between the radiation with the bonds in a chemical structure.
- NIR spectroscopy measures chemical bonds on the basis of overtones and combination bands of specific groups.

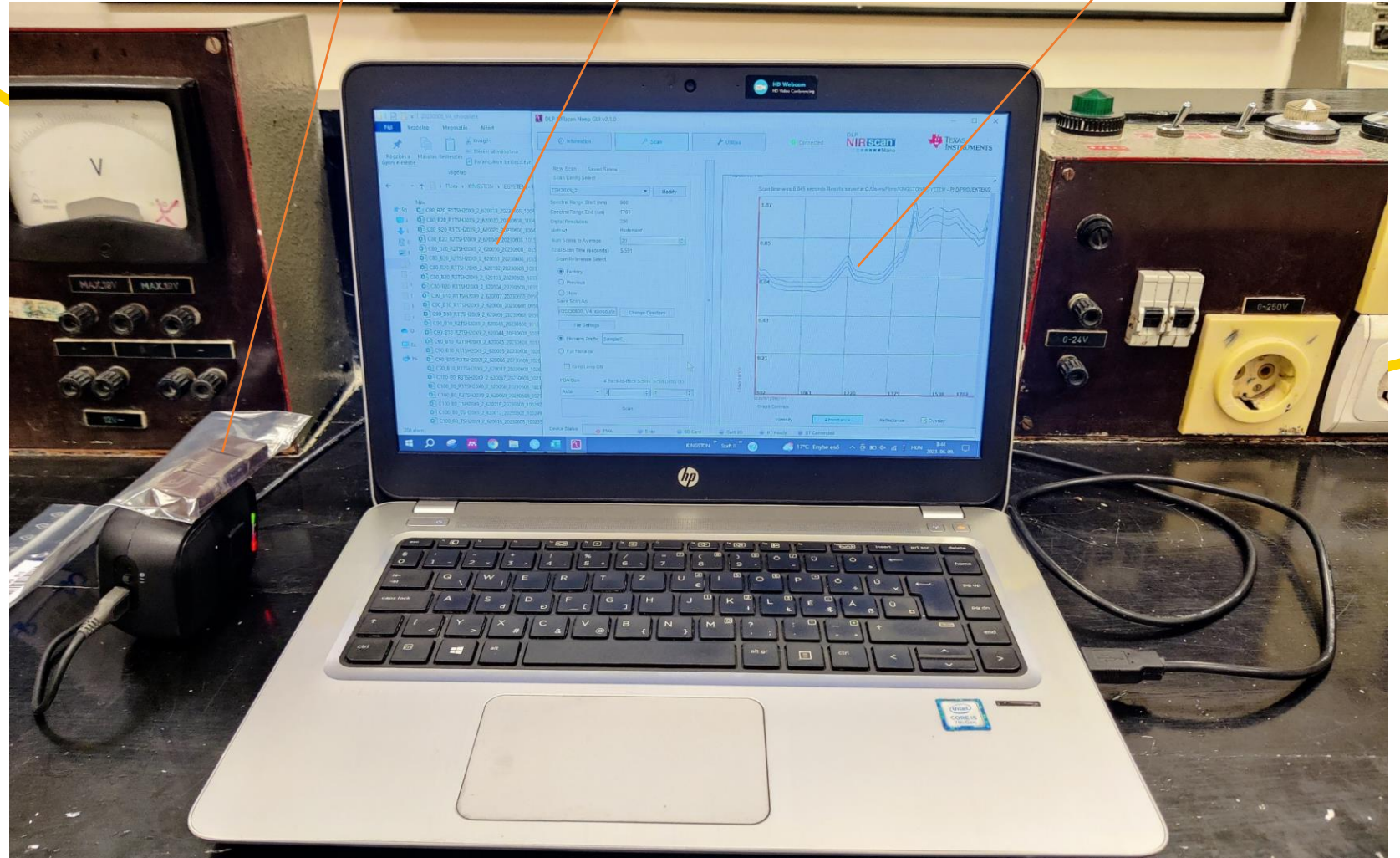


NIR-S-G1 (InnoSpectra Co., Hsinchu, Taiwan)

900-1700 nm

Methods

- NIR spectral region enables indirect analysis of samples contained in glass or plastic containers.
- NIRS is a high energy radiation in vibration spectroscopy that can be used to directly analyse solids with diffuse reflectance

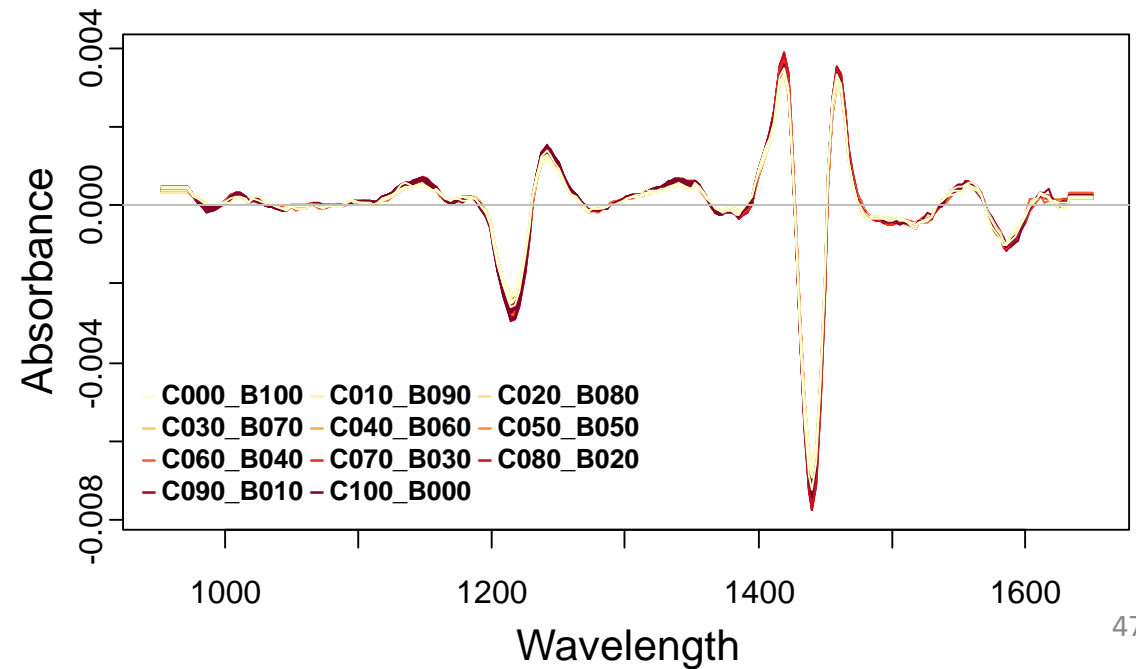
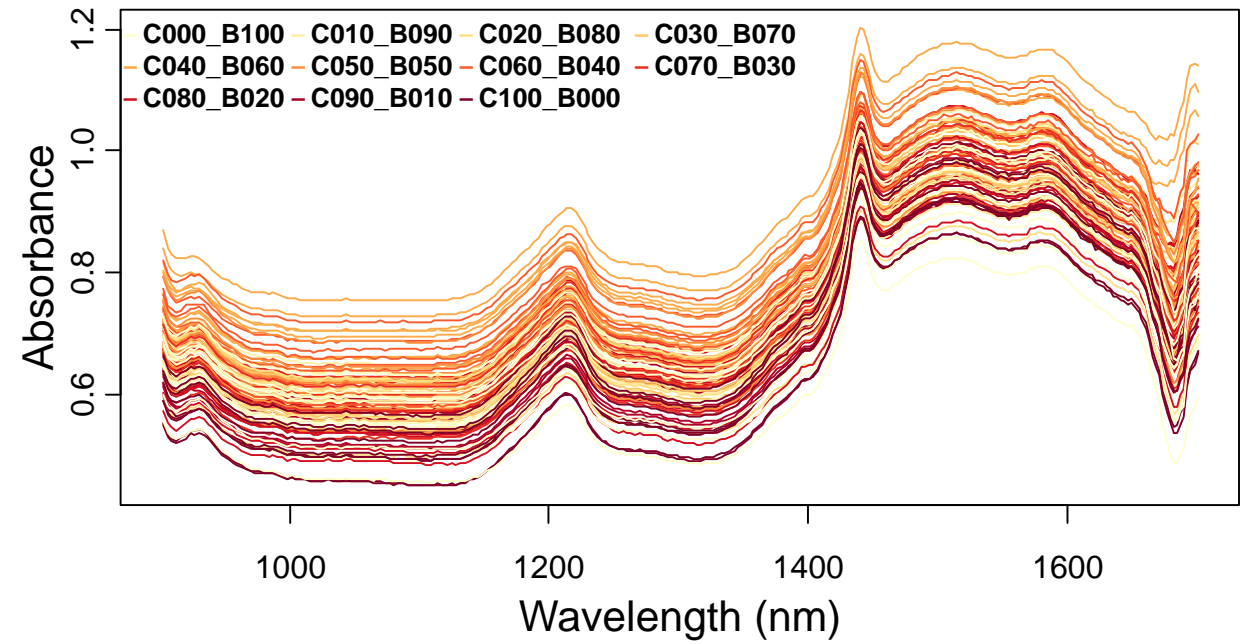


DEVELOPMENTAL RESULTS – Spectra

Raw spectra

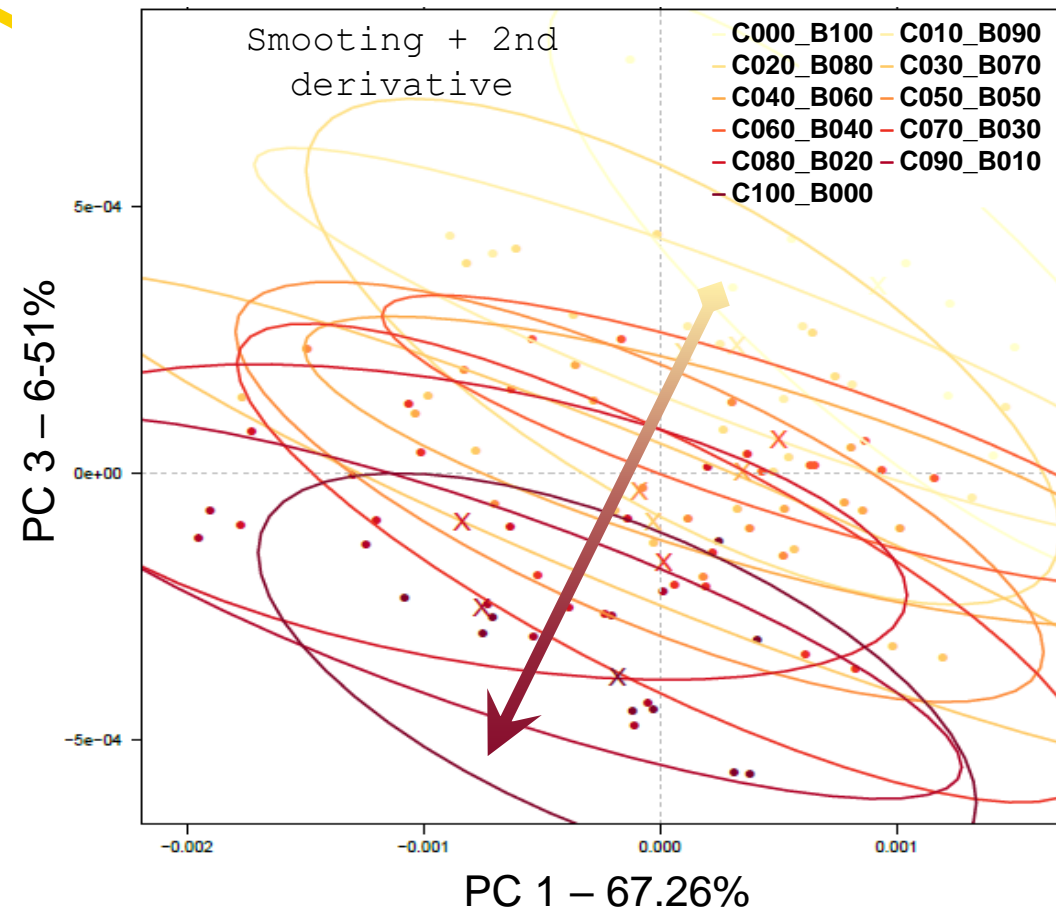
Smoothing + 2nd
derivative

**Pretreated
spectra**



DEVELOPMENTAL RESULTS – Unsupervised analysis

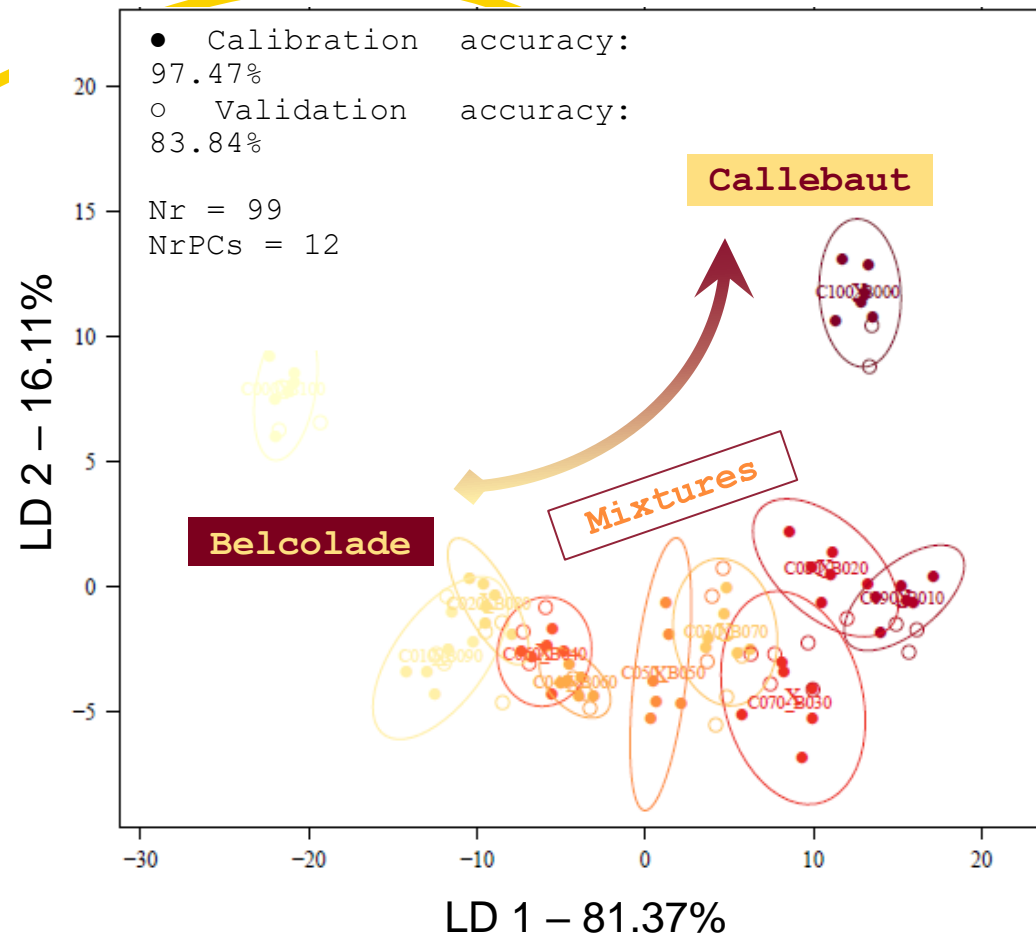
Principal component analysis



DEVELOPMENTAL RESULTS – Supervised data

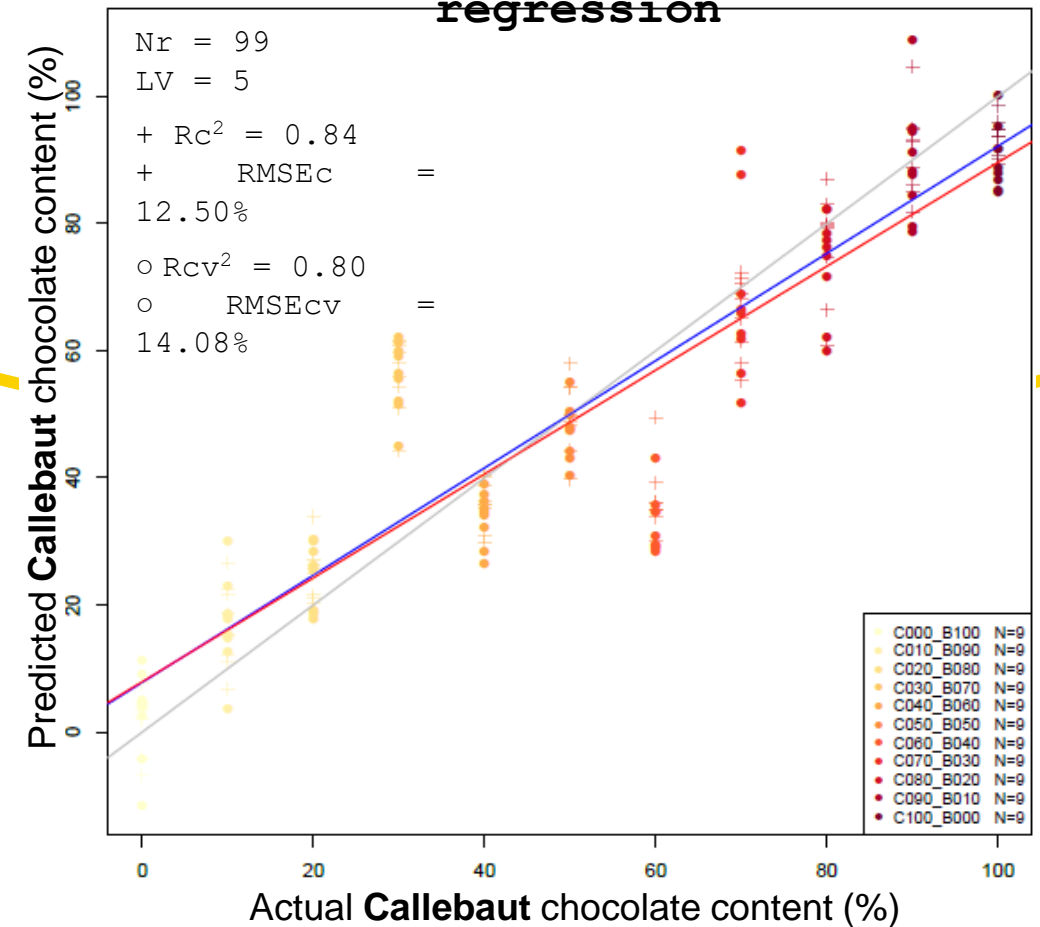
analysis

Linear discriminant analysis



Partial least squares

regression





Adam Kovol (CHE)

Andrea Mesarosova (SLO)

Pavan Kumar Pinnamaraju (IND)

Katarina Polakova (SLO)

Flora Vitalis (HUN)

Robert Waraczewski (POL)

-
- Visegrad Fund
-
-

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Quantitative evaluation of fruit extracts and fortified fruit juice – Objectives

The objectives of this study were

- to develop NIRS methods for the quantitative evaluation of Grape seed extract (GSE) powder when adulterated with chemically similar compounds and
- when used as an additive for fruit juice fortification



NIRS IN THE FIELD OF
NUTRITION

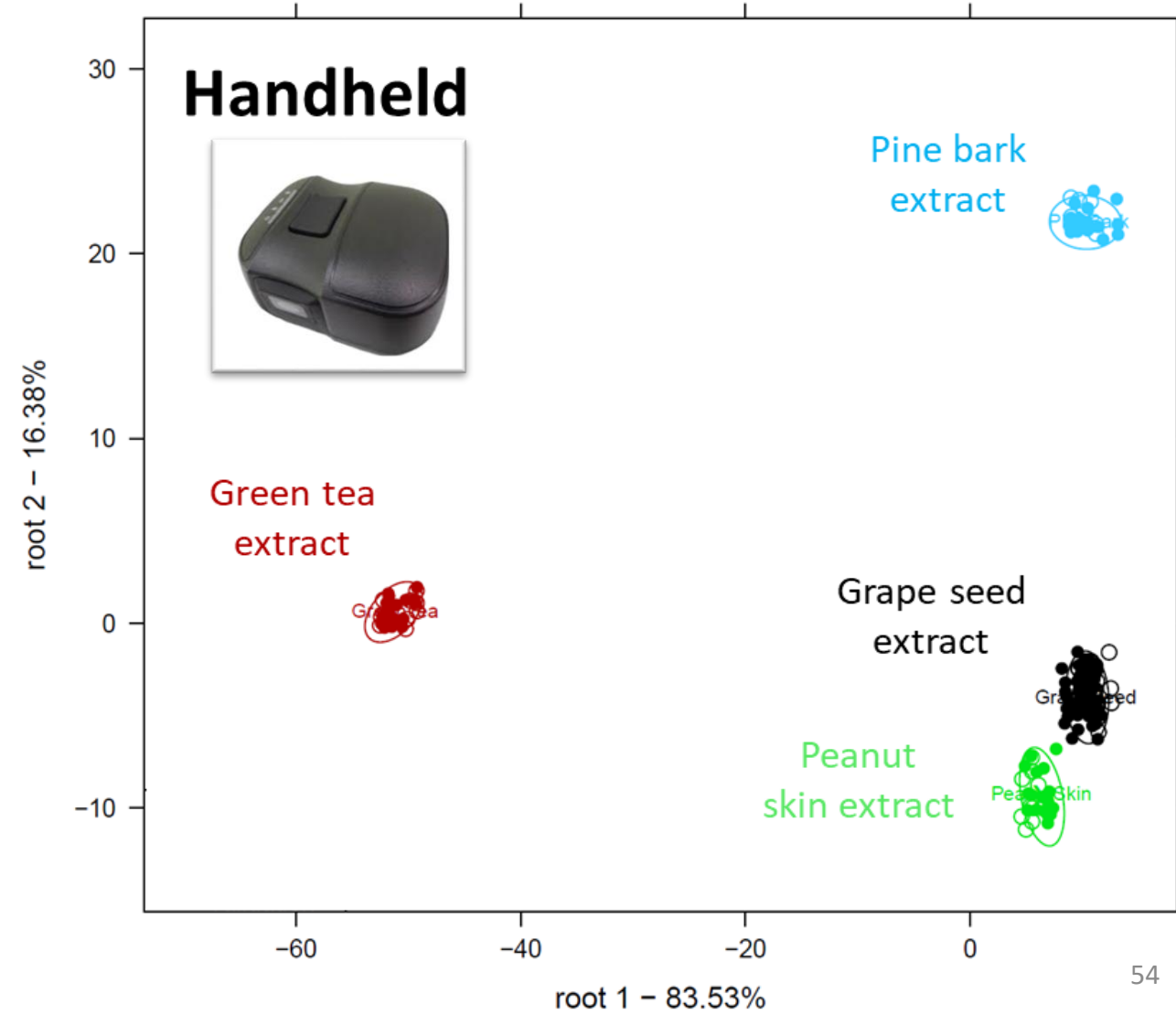
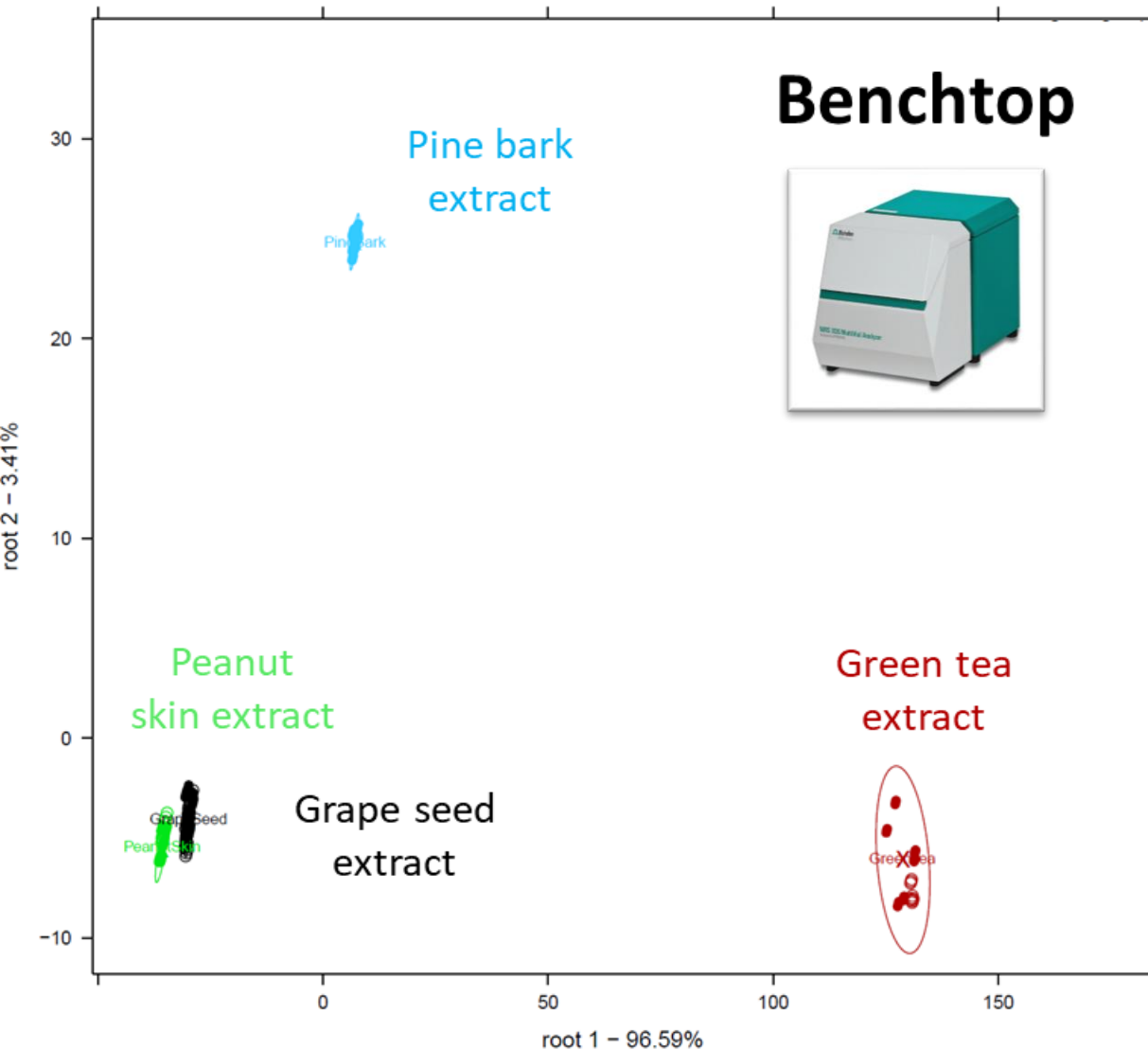


Quantitative evaluation of fruit extracts and fortified fruit juice – M&M

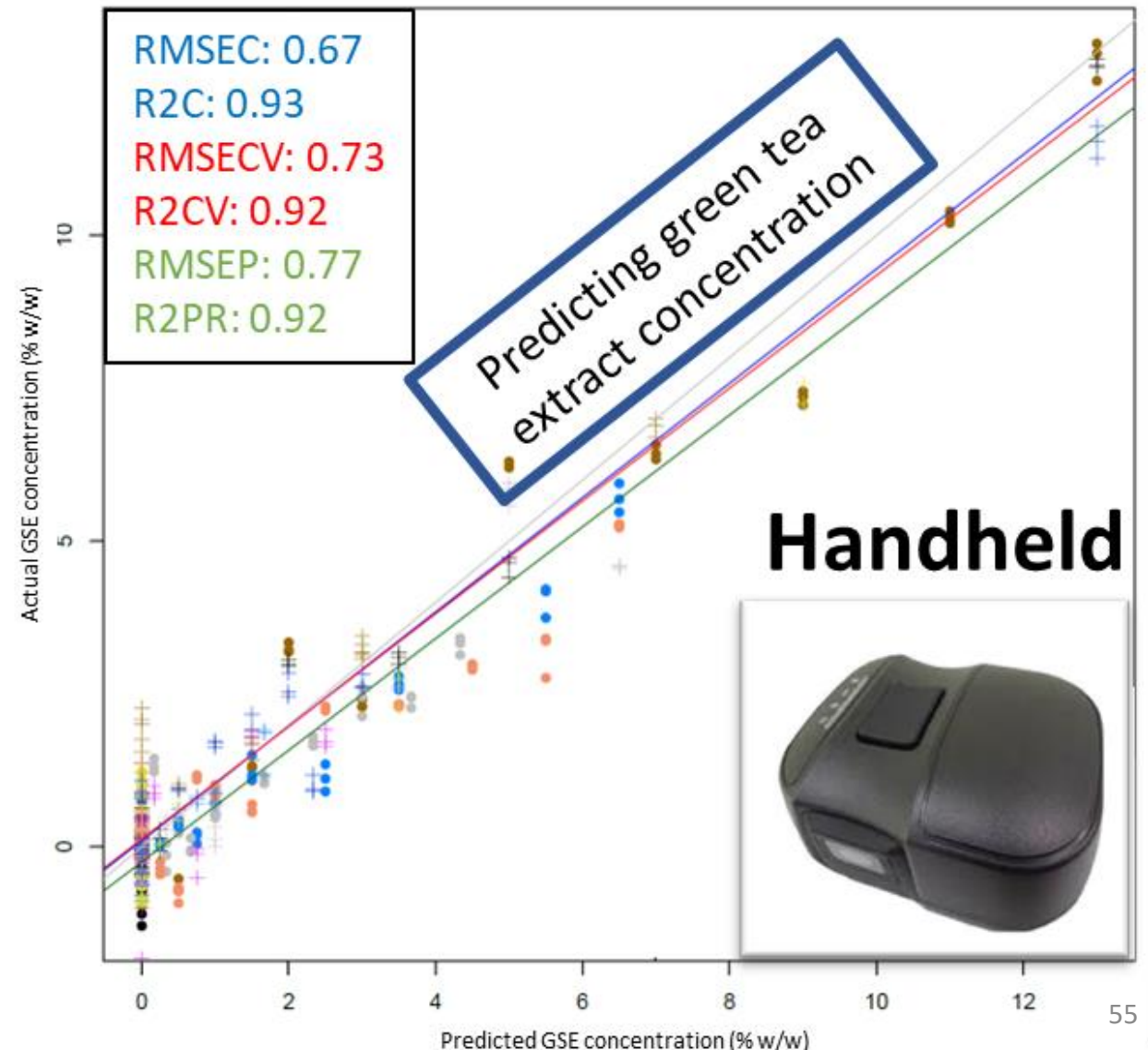
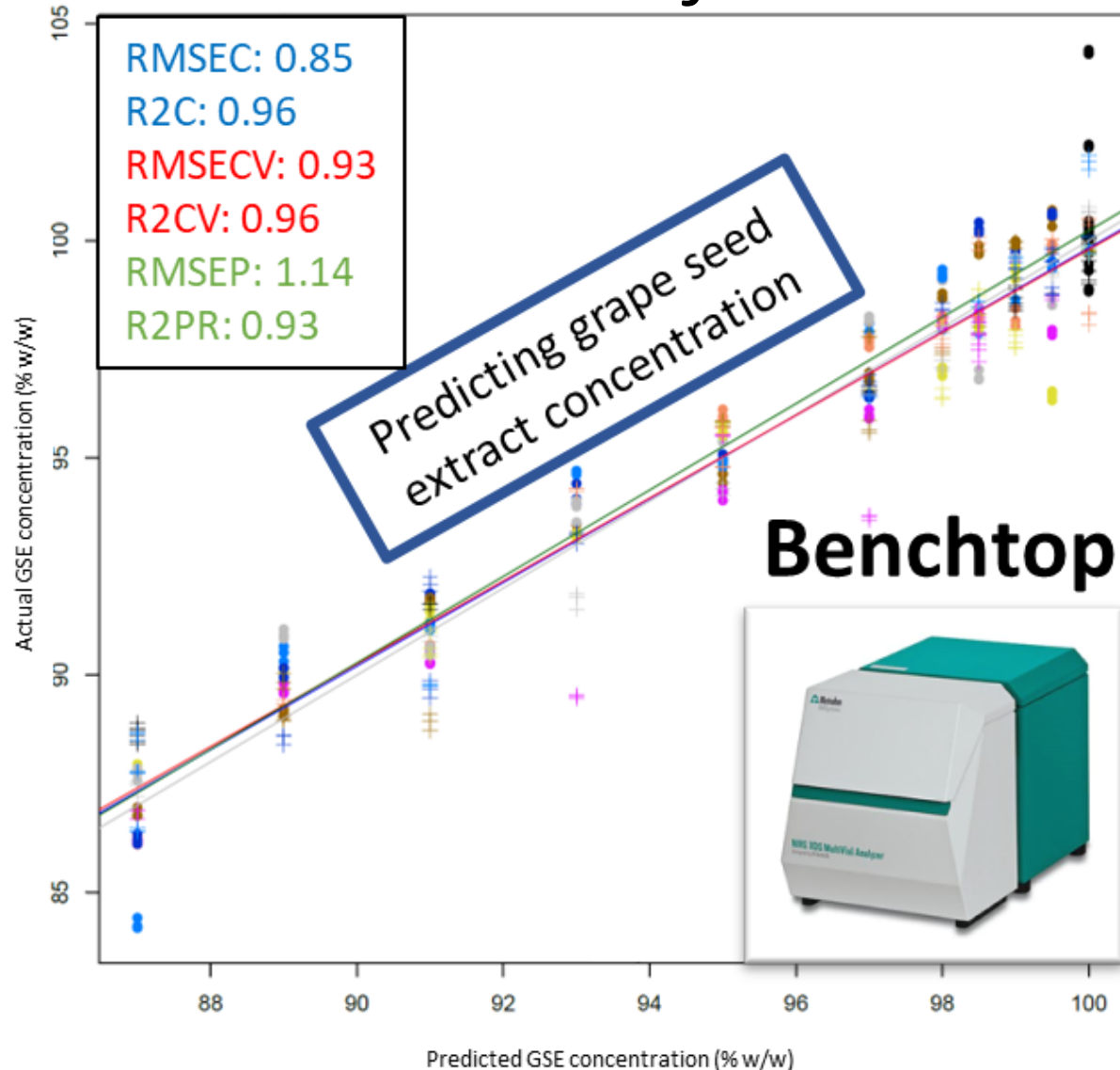
- **Grape seed extract (GSE)** powder mixed with
 - **peanut skin (PSE)**, **pine bark (PBE)** and **green tea** extracts (GTE)
 - Single and multiple mixtures: 11 mixture groups
 - 10 concentration levels/group
- **Juice samples** are fortified with GSE
 - **Plum, sour cherry** and **blue grape** juice
 - 17 concentration levels/juice
- Diffuse reflectance and transmittance spectra of powders and liquids, respectively were collected by different **benchtop and handheld NIR devices**
- Principal component analysis based linear discriminant analysis (PCA-LDA) for classification of type and level of adulteration/fortification
- Partial least squares regression (PLSR) and support vector regression (SVR) for prediction of the level of adulteration/fortification



Quantitative evaluation of fruit extracts and fortified fruit juice – Results – extracts – PCA-LDA



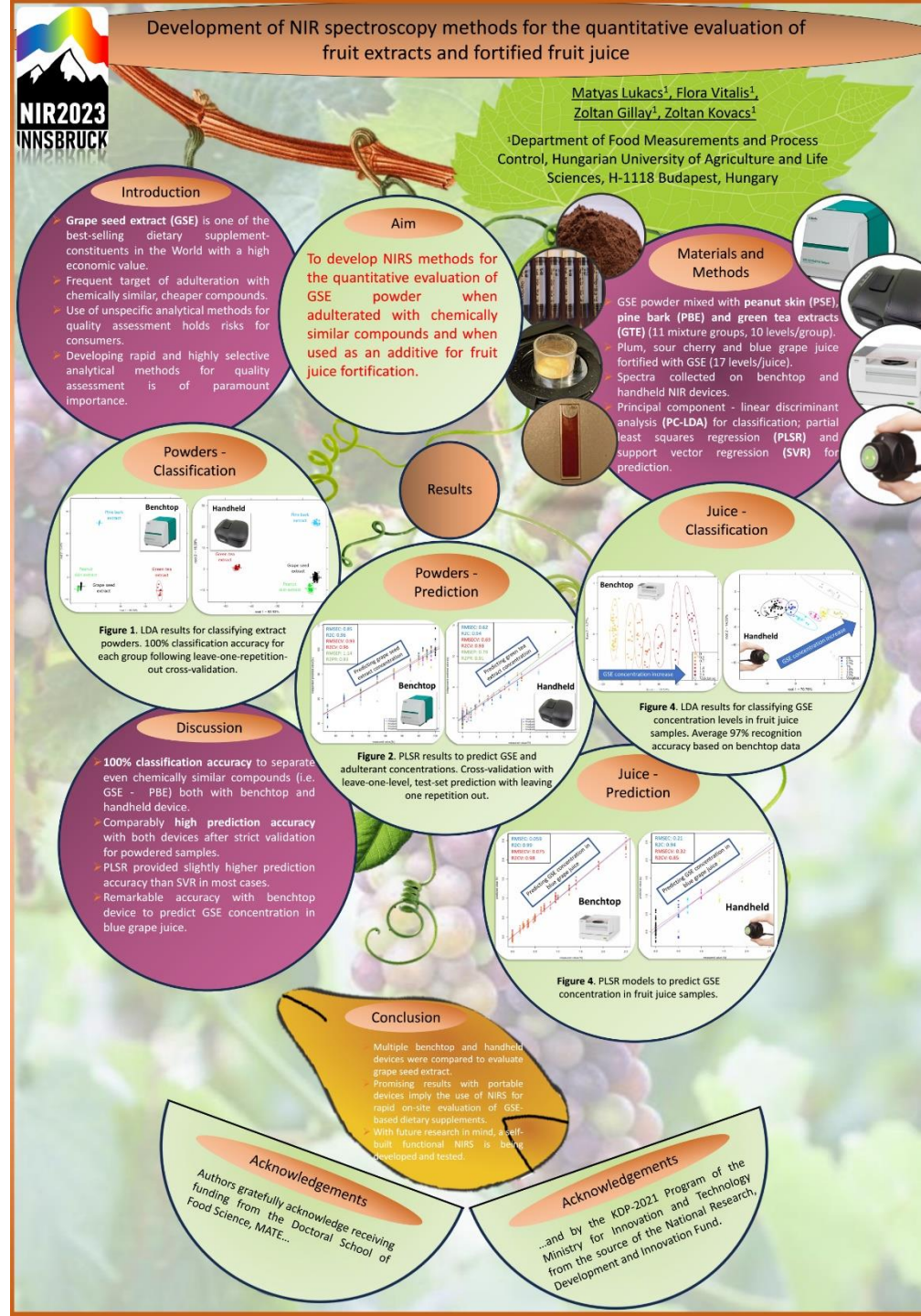
Quantitative evaluation of fruit extracts and fortified fruit juice – Results – extracts – PLSR





Matyas Lukacs NIR2023 – P01.35

Development of NIR
spectroscopy methods for the
quantitative evaluation of
fruit extracts and fortified
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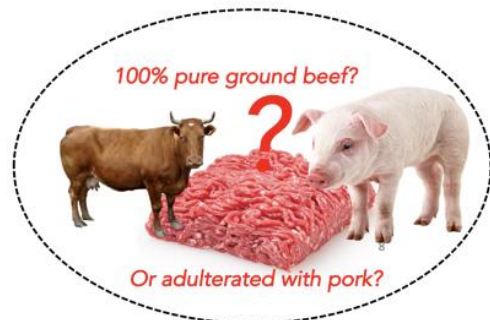
Meat extracts

J.L. Zinia Zaukuu, Z. Gillay, Z. Kovacs

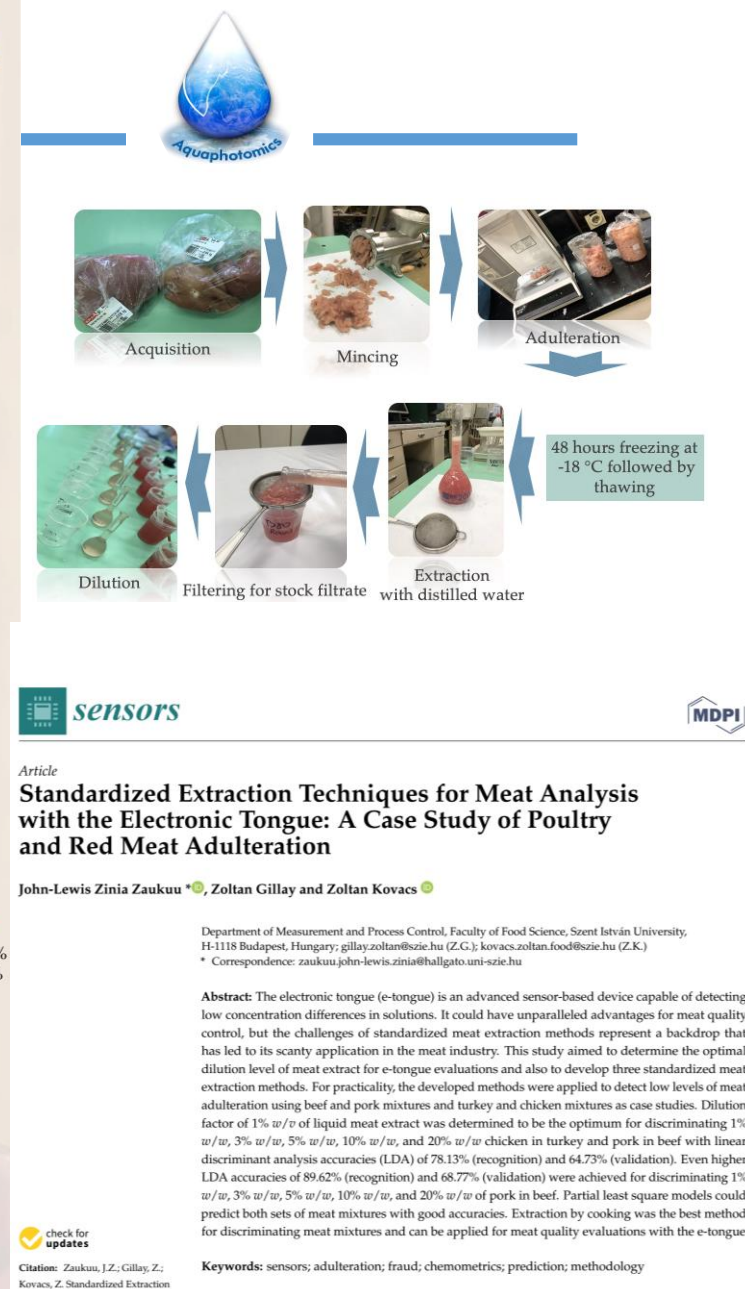
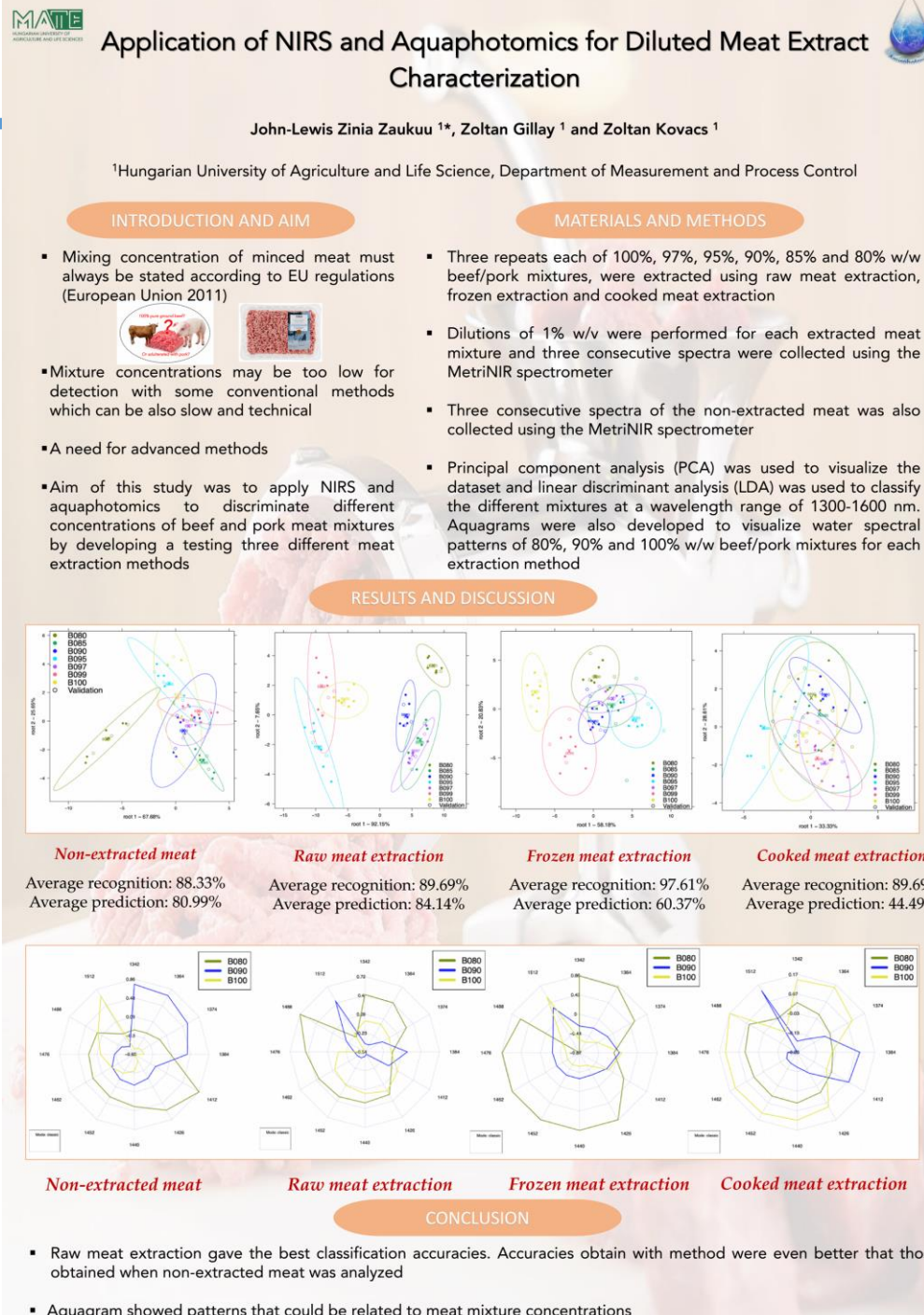
Application of NIRS and Aquaphotomics for Diluted Meat Extract Characterization

The aim of this study was

- to apply NIRS and aquaphotomics to discriminate different concentrations of meat mixtures



The 4th Aquaphotomics International Conference, 2021



Food quality assessment with NIR



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Mung bean juice

D. Tjandra Nugraha, J.L. Zinia

Zaukuu, J.P. Aguinaga Bósquez, Zs.

Bodor, F. Vitalis, Z. Kovacs

Evaluation of Mung Bean (*Vigna radiata*) Sprout and Quantification of Ascorbic Acid Content Using Near-Infrared Spectroscopy and Aquaphotomics

The aim of this study were

- to understand the possibility of rapid quality check and observation of mung bean sprouting process through NIRS and aquaphotomics
- to test the potential of NIR and aquaphotomics to make a correlative model for quality parameters evaluation and ascorbic acid content



The 4th Aquaphotomics
International Conference, 2021



Evaluation of Mung Bean (*Vigna radiata*) Sprout and Quantification of Ascorbic Acid Content Using Near-Infrared Spectroscopy and Aquaphotomics

David Tjandra Nugraha*, John-Lewis Zinia Zaukuu, Juan Pablo Aguinaga Bósquez, Zsannett Bodor, Flora Vitalis, and Zoltan Kovacs

Institute of Bioengineering and Process Control, Department of Measurements and Process Control, Faculty of Food Science, Hungarian University of Agricultural and Life Sciences, 1118 Budapest, Hungary



INTRODUCTION

Mung bean (*Vigna radiata*) is one of the most significant crop in Asia, but it contains anti-nutrient components in the form of phytate, which reduce the bioavailability of mineral [1]. Some processing techniques, especially sprouting have been proven to reduce anti-nutrient components, and increase the quality of mung bean. Sprouting process also produce phenolic components and vitamins; especially ascorbic acid [2]. The use of NIRS within 700-2500 nm interval has been reported to be able to classify mung bean sprout based on sprouting time for quality purpose [3]. However, complex aqueous system of mung bean sprout suggests that water matrix coordinates plays a key role at molecular level [4]. The usage of aquaphotomics is suitable due to the sample preparation from the conventional techniques. The objective of this study was to understand the possibility of rapid quality check and observation of sprouting process through NIRS and aquaphotomics, and test the potential of NIRS and aquaphotomics to make a correlative model for quality parameters evaluation and ascorbic acid determination in low concentration.

MATERIALS & METHODS

The bean was sprouted for 120 h in 6 h interval. The sprouting was done on cellulose paper in an incubator of constant temperature. Grown sprout was crushed, mixed with water (1:2 ratio), and filtered, creating the bean sprout extract used for NIRS scanning. Water content was determined by AOAC method, pH and conductivity based on benchtop instrument, and ascorbic acid content based on direct redox titration with iodine solution. NIRS measurement was done using DLP NIRScanNano instrument (Texas Instrument, Dallas, Texas, United States) in transmission mode using quartz cuvette of 1 mm path length. NIRS scanning was done in triplicate with three consecutive scans. Result from conventional analytical techniques were analyzed using descriptive statistics and one-way ANOVA followed by differentiation test. The spectral data was analyzed using chemometrics methods (PCA and LDA), and correlative model was made using PLSR technique and n-fold cross validation.

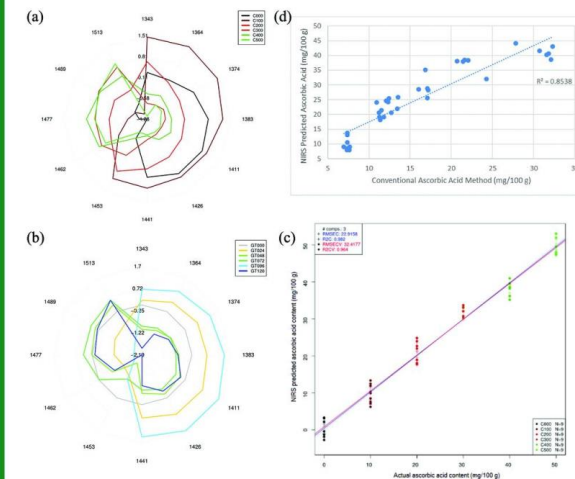


Figure 2. Aquagram (1300-1600 nm) and PLSR plot, after Savitzky-Golay smoothing (SG) and standard normal variate (SNV) pretreatment of the spectra: (a) Aquagram plot of sprouted mung bean sprout with 24-hour interval; (b) Aquagram plot of ascorbic acid (0-500 mg/100 g); (c) Regression of ascorbic acid prediction; (d) Ascorbic acid content plot of NIR predicted values against conventional technique result.



Figure 1. Germinated Mung Bean Sprout from 0-120 h

RESULTS

The aquagram showed a trend of strong bound water formation during germination (Figure 2a.), and its comparison with the ascorbic acid standard (Figure 2b.). PLSR model was made from the spectral data of bean sprout extract, and predicted quality parameters (water content, germination time, and ascorbic acid content). The ascorbic acid content was analyzed for independent prediction of sample with unknown concentration of ascorbic acid. The prediction result was then compared with conventional technique (Figure 2d.)

CONCLUSION

The results showed reliability for mung bean sprout quality evaluation, where it prominently predicted quality parameters. PLSR regression showed high accuracies and low error. Aquagrams showed similar trend of initial weaker bonded water molecules, then forming of stronger bound water (1513 nm) was observed after prolonged sprouting and high ascorbic acid content. This indicated potential of ascorbic acid detection in complex food matrixes with low concentration.

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Article

Near-Infrared Spectroscopy and Aquaphotomics for Monitoring Mung Bean (*Vigna radiata*) Sprout Growth and Validation of Ascorbic Acid Content

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Abstract: Mung bean is a leguminous crop with specific trait in its diet, namely in the form of anti-nutrient components. The sprouting process is commonly done for better nutritional acceptance of mung bean as it presents better nutritional benefits. Sprouted mung bean serves as a cheap source of protein and ascorbic acid, which are dependent on the sprouting process, hence the importance of following the biological process. In larger production scale, there has not been a definite standard for mung bean sprouting, raising the need for quick and effective mung bean sprout quality checks. In this regard, near-infrared spectroscopy (NIRS) has been recognized as a highly sensitive technique for quality control that seems suitable for this study. The aim of this paper was to describe quality parameters (water content, pH, conductivity, and ascorbic acid by titration) during sprouting using conventional analytical methods and advanced NIRS techniques as correlative methods for modelling sprouted mung beans' quality and ascorbic acid content. Mung beans were sprouted in 6 h intervals up to 120 h and analyzed using conventional methods and a NIR instrument. The results of the standard analytical methods were analyzed with univariate statistics (analysis of variance (ANOVA)), and the NIRS spectral data was assessed with the chemometrics approach (principal component



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Food quality assessment with NIR



Feed - TMR

Monitoring feed supplements with handheld near-infrared spectrometer



Cheese quality

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Monilia (brown rot) on plums

Early phase detection of *Monilia fructigena* infection on plums



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Classification and quantification of different origin Arabica and Robusta coffee



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With different lactobacillus strains grown with different water



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Tomato concentrate

Adulterated with starch, paprika seed powder, NaCl and sucrose



Mineral water

With different mineral content mixed and analyzed



Monilia on plums



Monitoring of brown rot caused by *Monilia fructigena* on plums with the aquaphotomics approach

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Monitoring of brown rot caused by *Monilia fructigena* on plums with the aquaphotomics approach

The aim of this study was to apply NIRS and aquaphotomics:

- for monitoring quality change of fruits stored under different conditions,
- early detection of *Monilia fructigena* infection



INTRODUCTION

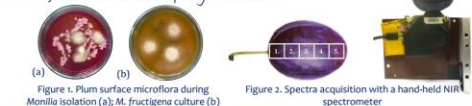
- Plums are particularly sensitive and perishable fruits that ripen quickly after harvest, resulting in a short postharvest lifespan.
- Monilia* species causing brown rot during fruit cultivation, harvest, or storage can result in losses of up to 80% under favorable conditions for the fungus.
- Due to change in the ambient conditions or infection on the fruit result in invisible alterations at first but can be discovered by examining the patterns given by non-invasive near-infrared spectroscopy.
- The "water mirror" approach of aquaphotomics has proven to be suitable to map and determine the physiological state of horticultural products with a high water content.

AIMS

The research focused on tracking changes in plums during storage.

The study aimed to apply NIRS and aquaphotomics:

- for monitoring quality change of fruits stored under different conditions,
- early detection of *Monilia fructigena* infection.



RESULTS AND DISCUSSION

Visible signs of *Monilia* infection were observed after two days only on plums with injured surfaces and stored at room temperature.

The results showed high variability, therefore the constructed models had to be optimized in each case using different spectrum pretreatment methods.

PCA confirmed that the results were considerably influenced by the location of the spectrum acquisition on the fruit surface.

Soft independent modeling of class analogies

Interclass distances indicated demonstrably large differences between infected plums stored under different conditions.

As storage time increased, fruits stored at room temperature showed a crescent interclass distance compared to refrigerated samples.

Plums infected via injury had a larger interclass distance compared to control and intact samples, although there was no incontrovertible distinguishability.

The following wavelengths were relevant for discrimination by storage duration or mode of infection: 1393, 1403, 1415, 1446, 1484, 1539, 1571 nm.

Aquagrams

The spectral patterns of plums showed a dynamic change over the storage time (Figure 3).

The absorbance evolution of injured plums stored in the refrigerator and at room temperature showed the opposite trend in the 1365-1384 nm wavelength range.

The absorption gradually decreased in the range of 1411-1489 nm during storage.

The plums initially showed markedly low absorbance around 1512 nm.

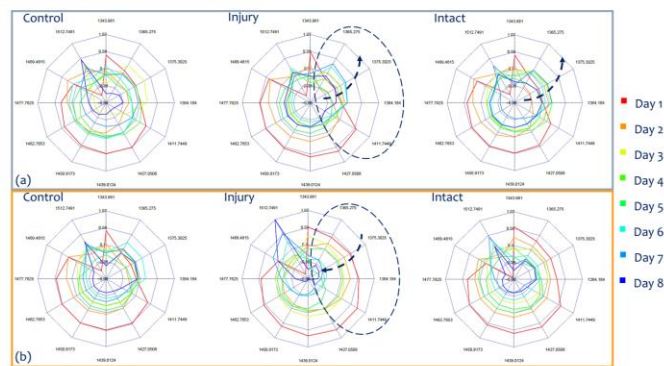


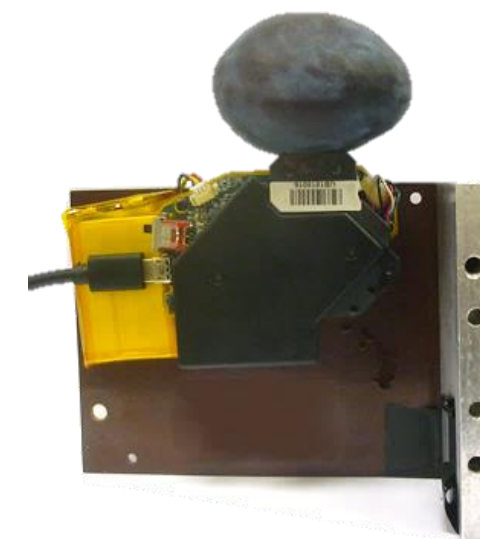
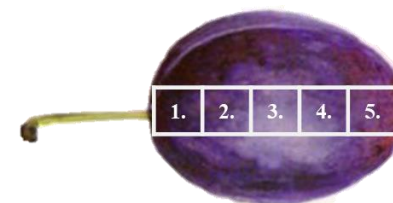
Figure 3. Aquagrams of plums infected with *Monilia fructigena* in different ways and stored refrigerated (a) or room temperature (b)

CONCLUSIONS

There were detectable differences in plums infected with *Monilia fructigena* in different ways during controlled storage. The most striking differences were for fruit stored at room temperature and infected through injury. The approach used proved to be fruitful for predicting the progression of the infection, in any way it would be worthwhile to conduct further experiments with larger sample size by reason of the natural variability of the fruits.

ACKNOWLEDGEMENT

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Conclusions

Near Infrared Spectroscopy

- is well suited for the rapid and objective evaluation of crops and foods
- provides information on non-specific properties of the samples, therefore useful for detecting general quality changes
- has a wide range of applications also in industrial environments and/or field applications
- has been proven a very useful tool in the digitization of agriculture and food production (i.e. in FOOD Quality in Digital Age) all the way “From Farm to Fork” and beyond



We would like to
thank our
collaborators.

I would like to thank
my Team's hard
work.

THANK YOU FOR YOUR KIND ATTENTION!



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