



Non-targeted chemometric approaches for food and feed quality and safety

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Quality and Authentication of Products Unit
Knowledge and valorization of agricultural products

Department

Walloon Agricultural Research Centre – CRA-W

Gembloux, Belgium



Data sciences **R**aman **I**maging **M**id-IR
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Quentin Arnould / Delphine Delhotte / Antoine Deryck /
Juan Antonio Fernández
Maxime Joissains / Nicaise Kayoka / François Stevens

Quality and authentication of agricultural products Unit

Implementing and validating various operations with a view to being a dynamic, innovative and sustainable research player committed to the quality and authentication of agricultural and agri-food products. The unit also seeks to boost the Walloon agri-food sectors by improving the quality, traceability and typicality of their products, focusing in particular on products from the short distribution and organic sectors.



Objectives

Developing and applying knowledge, analytical methods and innovative tools to ensure product quality and authentication.



Developing **knowledge** in the field of agri-food product quality and authentication



Participating in the **European Research Area (ERA)**.



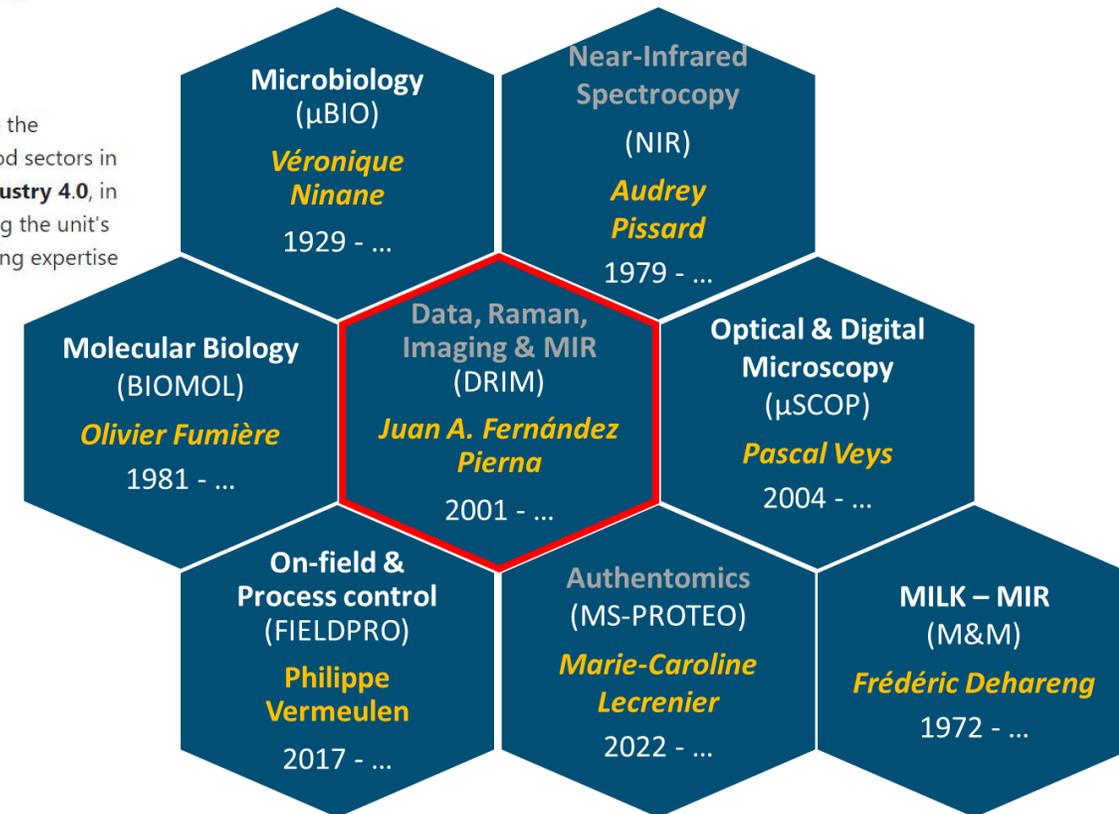
Ensuring a **technological and analytical watch** in the field of molecular biology, chemometrics, bioinformatics, imaging, database management, microbiology, microscopy and vibrational spectroscopy.



Lending support to the agricultural and food sectors in the **roll-out of Industry 4.0**, in particular by sharing the unit's sensor and modelling expertise



Developing **innovative, rapid and sustainable analytical methods**.



Head of Unit: Dr Vincent Baeten

Scientific Director Unit QAP

v.baeten@cra.wallonie.be

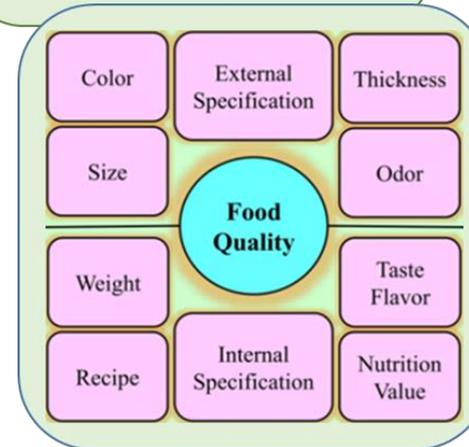
Importance of Ensuring Food/Feed Quality and Safety

- Ensuring food/feed quality and safety is essential to safeguard public health and promote overall welfare.
- Globalization and international trade have increased safety concerns as food/feed products cross geographical borders, increasing the risk of contamination and exposure to unknown hazards.



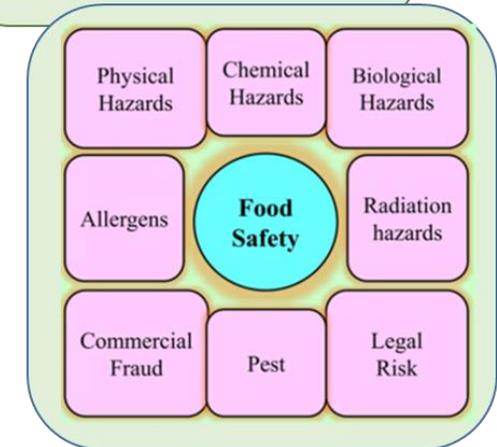
Food Quality

The process of achieving all the required characteristics that are acceptable to the consumer



Food Safety

The process of preventing risks that may affect foodstuffs. And, directly or indirectly affect consumers



Importance of Ensuring Food/Feed Quality and Safety

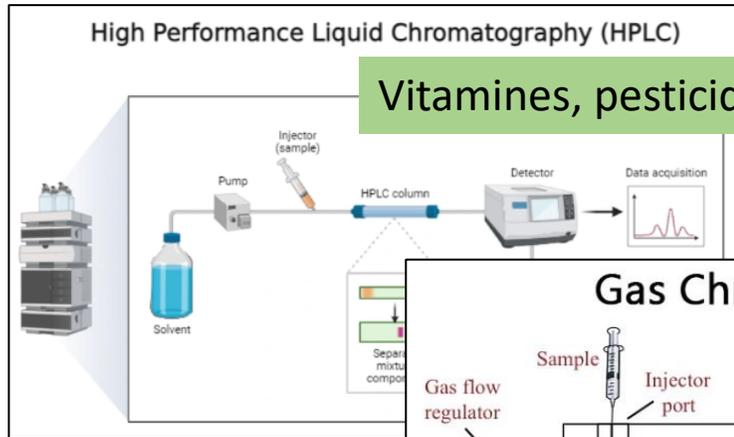
- ❖ Regulatory agencies, food manufacturers, and consumers alike place great importance on ensuring that food/feed products meet **high quality and safety standards**.
- ❖ Robust **food/feed safety management systems, strict regulations and rigorous inspection protocols** are essential to prevent, detect and mitigate risks throughout the food/feed supply chain.
- ❖ **Advances in analytical techniques and scientific methodologies** play a key role in improving our ability to monitor and assess food/feed quality and safety, enabling stakeholders to make informed decisions and take proactive measures to protect public health.

Traditional Targeted approaches for food/feed analysis

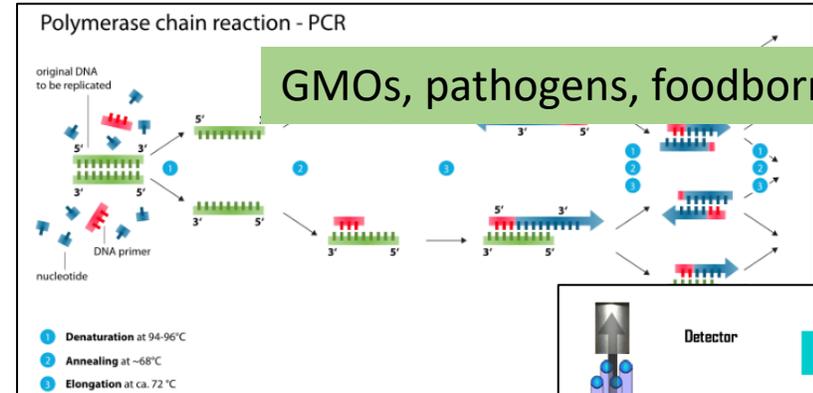


- ❖ Targeted approaches for food/feed analysis are designed to **detect and quantify specific compounds** or classes of compounds within food/feed samples.
- ❖ These methods are characterized by their **specificity and sensitivity to predetermined analytes**.
- ❖ These targeted approaches are essential tools for routine analysis, regulatory compliance, and quality control in the food/feed industry.

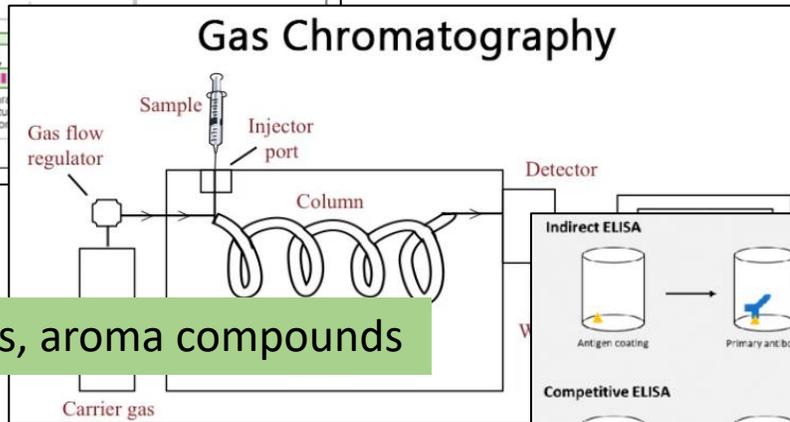
Traditional Targeted approaches for food/feed analysis



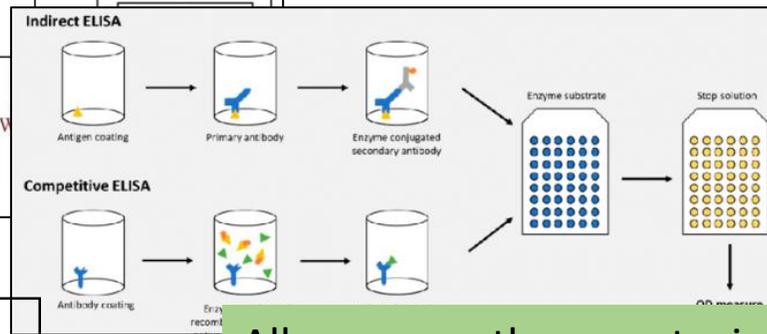
Vitamines, pesticides, mycotoxins



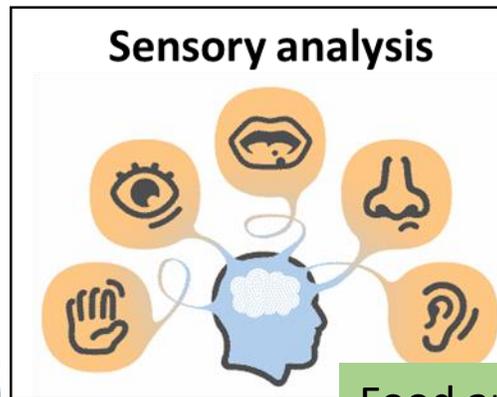
GMOs, pathogens, foodborne bacteria



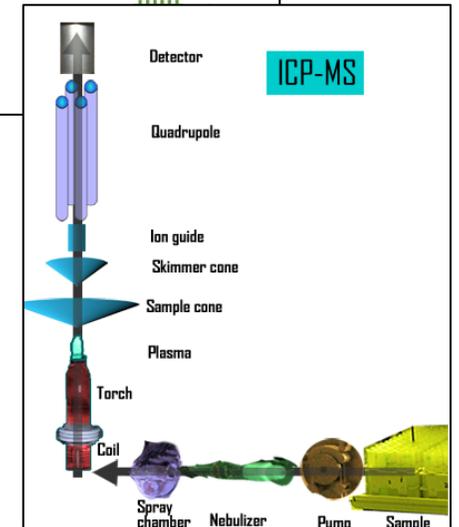
Fatty acids, pesticides, aroma compounds



Allergens, pathogens, toxins



Food appearance, flavor, texture, aroma



Trace elements, heavy metals

From traditional Targeted approaches to Untargeted analysis



- ❖ However, targeted approaches **may lack the ability to detect unknown or unexpected compounds**, highlighting the need for complementary non-targeted approaches.



Vincent Baeten, Philippe Vermeulen, Juan Antonio Fernández Pierna and Pierre Dardenne
Walloon Agricultural Research Centre (CRA-W), Belgium

From targeted to untargeted detection of contaminants and foreign bodies in food and feed using NIR spectroscopy

Untargeted approaches for food/feed analysis

- ❖ Unlike targeted methods, which focus on detecting specific compounds or classes of compounds, non-targeted approaches aim to **characterize the entire chemical profile of a food matrix without prior knowledge of the analytes present.**
- ❖ Non-targeted approaches can contribute significantly to ensuring food safety. They enable the **detection of emerging contaminants, identification of potential hazards, and assessment of overall food quality.**

Untargeted approaches for food/feed analysis

- ❖ These approaches can use a variety of **analytical techniques, such as vibrational spectroscopy (NIR, MIR, Raman...)**, to generate complex datasets containing information about the diverse array of molecules present in a food sample.

Vibrational Spectroscopy

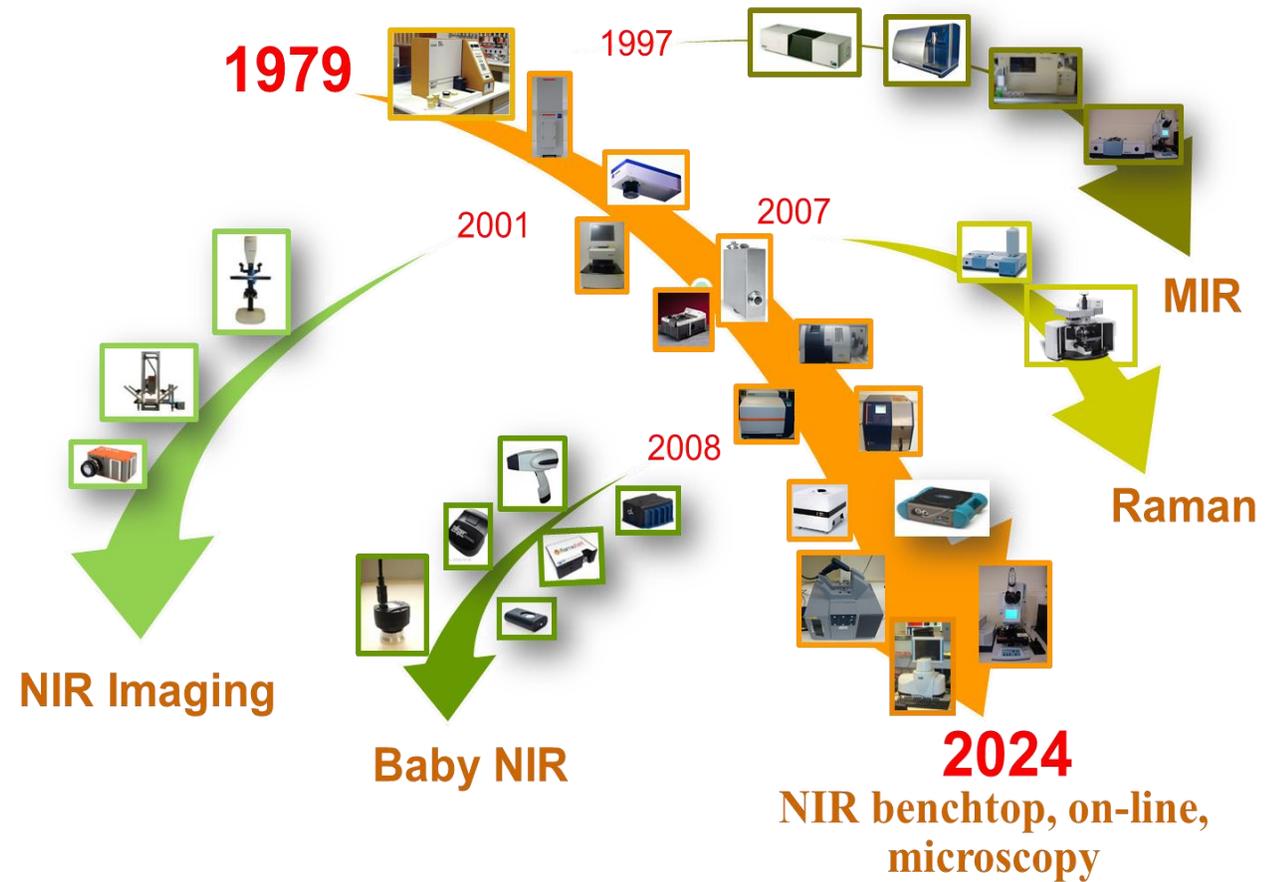
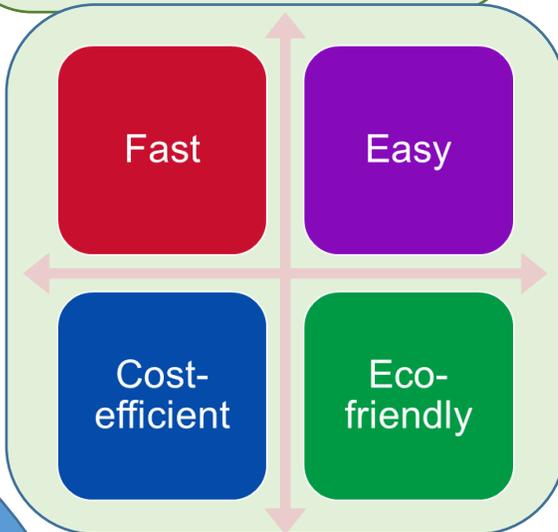
- ❖ Chemometric methods, including multivariate statistical analysis, machine learning algorithms, and pattern recognition techniques, are then applied to **interpret these datasets, uncover meaningful patterns, and identify potential contaminants, adulterants, or quality markers.**

Chemometrics

Spectroscopic approaches for food/feed analysis

Vibrational Spectroscopy

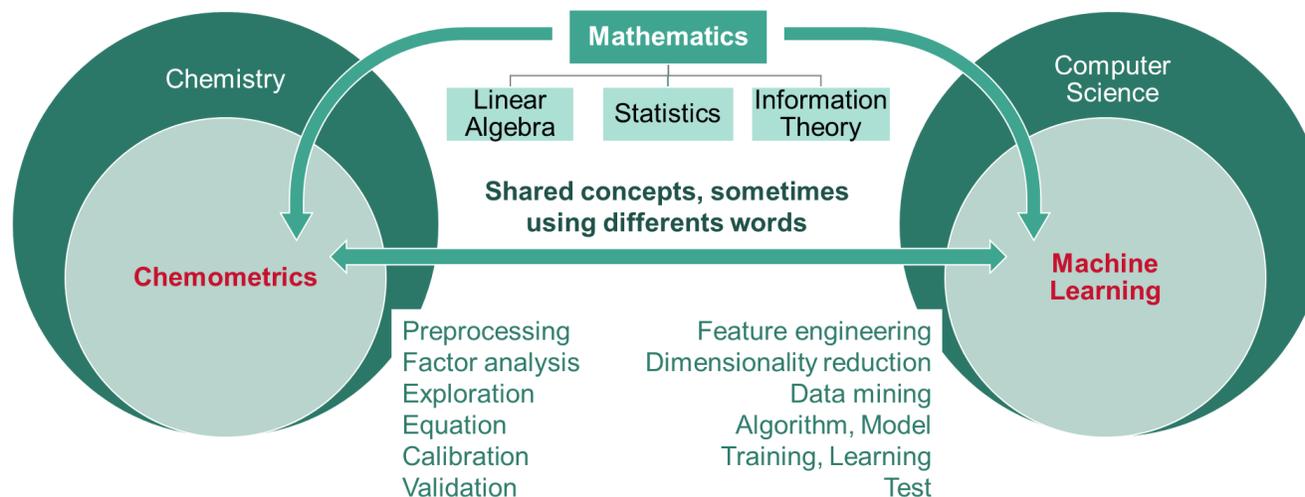
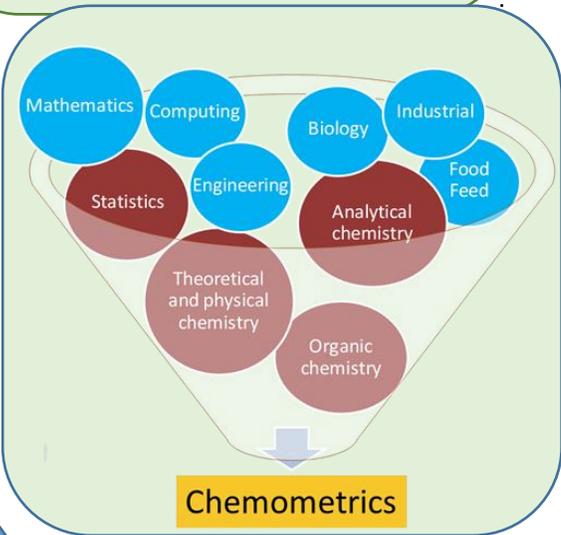
- No or limited sample prep (drying, grinding, ...)
- No additional reagents necessary
- Fast measurement even on moving sample (conveyor belt, ..)
- Non-invasive (do not alter the sample)
- Simultaneous determination of different constituents



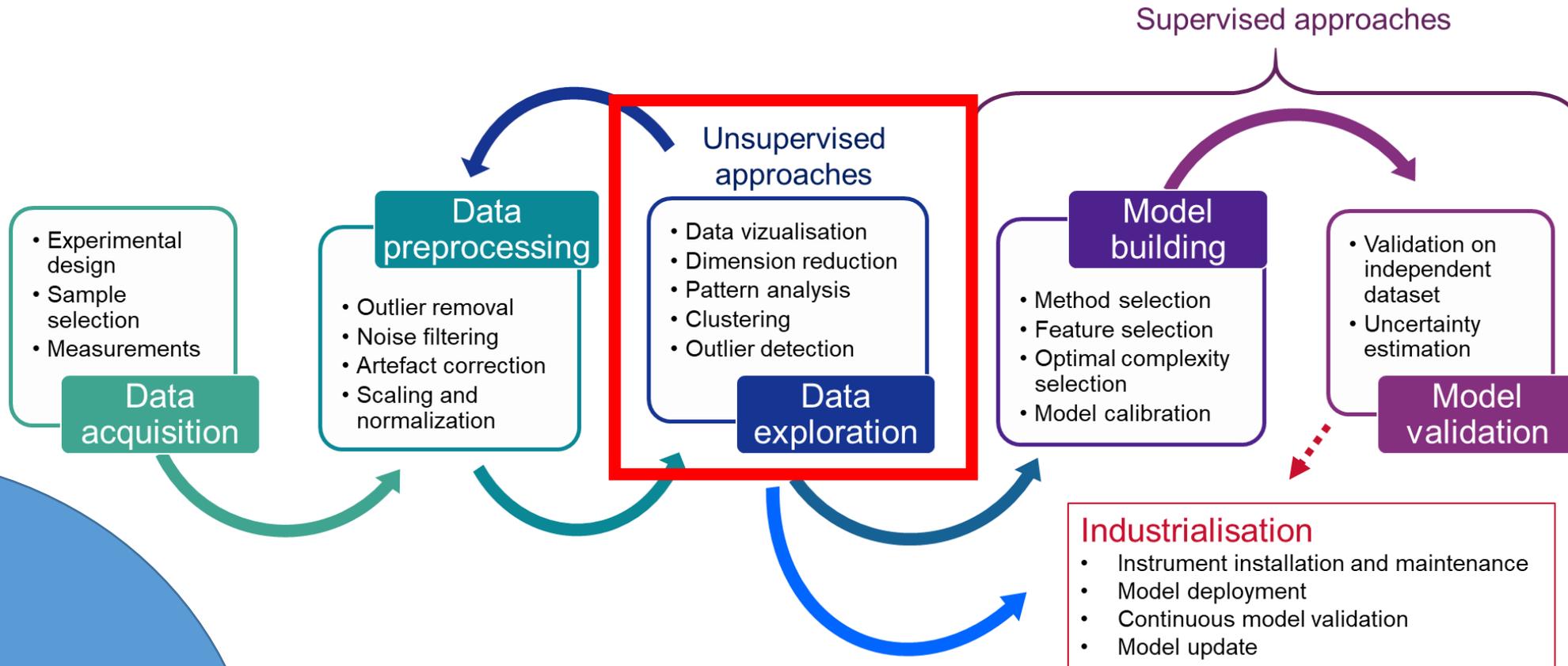
Chemometric approaches for food/feed analysis

Chemometrics

“Chemometrics is a chemical discipline that uses mathematics, statistics and formal logic to design or select optimal experimental procedures; to provide maximum relevant chemical information by analyzing chemical data; and to obtain knowledge about chemical systems.”



Chemometric approaches for food/feed analysis - pipeline



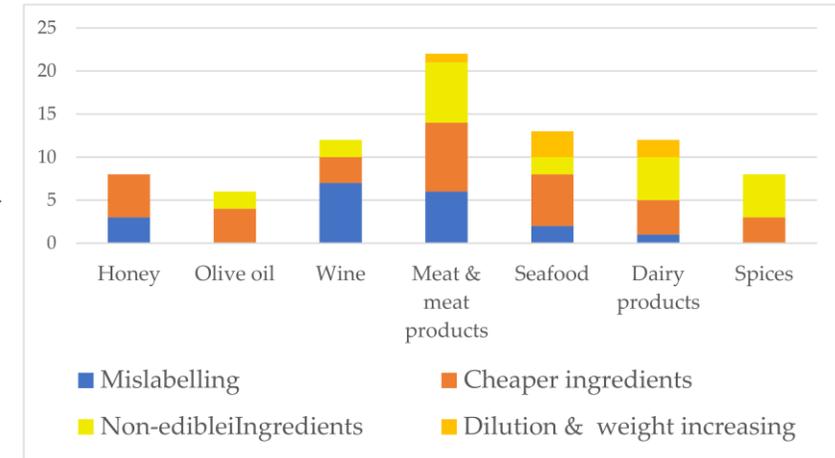
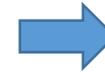
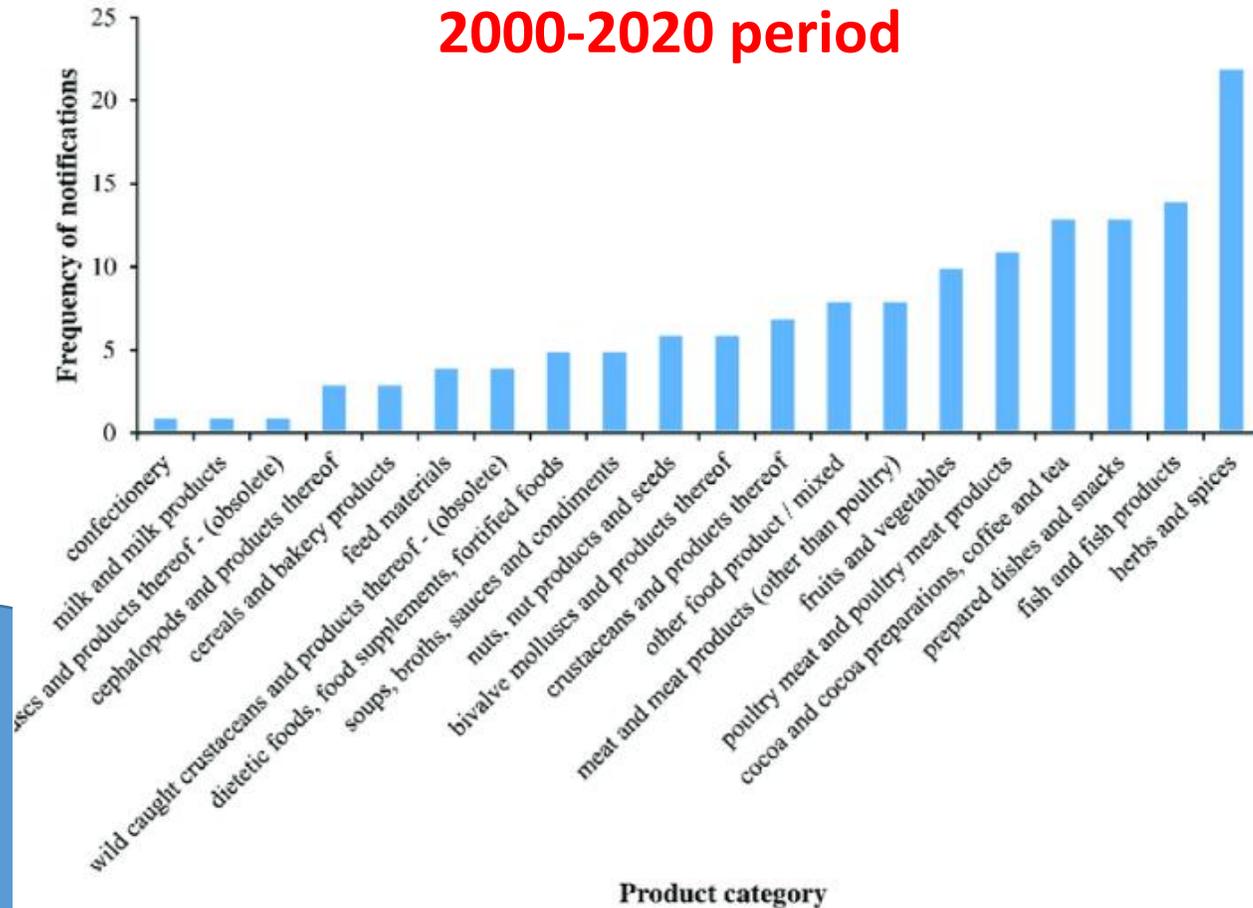
Why we need Untargeted approaches for food/feed analysis ?

In recent years, food and feed safety has become an increased concern for consumers due to several important crises related directly or indirectly to human health.



Why we need Untargeted approaches for food/feed analysis ?

Rapid Alert System for Food and Feed (RASFF)



2023

- Using official reports only, 'Fruit, vegetables & legumes', 'Milk & dairy products' and 'Beverages' are the top three.
- Using official, media & peer reviewed publication reports, 'Honey', 'Herbs & Spices' and 'Meat & Poultry' are the top three.

Frequency of food fraud notifications in RASFF by food/feed product category the year (2000-2020).

Why we need Untargeted approaches for food/feed analysis ?

The authorities closed a spices grinding unit **adulterating the spices** with wood powder, seizing as well 600 kg of adulterated turmeric powder and 300 of red chili powder. The spices were ready to be labelled as branded spices as well.
[The Nation](#)

The authorities seized 230 tons of **fruits and vegetables mislabelled** as "organic."
[Abc](#)

The authorities seized 6 670 litres of **milk adulterated with water and/or chemicals**
[Urdu Point](#) [App](#) [Urdu Point](#) [The Nation](#) [App](#) [Urdu Point](#)

The authorities seized 2 510 litres of **cooking oil sold as olive oil** (total value of 29 000 Euros).
[Cnn Portugal](#) [Tvi](#) [Tvi](#) [Cnn Portugal](#)

The authorities seized 3 tons of **seafood without traceability documentation** or not suitable for human consumption.
[Ansa](#) [Latina Today](#)

The authorities seized 2.7 tons of mussels illegally caught.
[Foggia Today](#) [Oltre free press](#)

Spain
 Fruits and vegetables
 Counterfeit

Pakistan
 Milk and milk products
 Adulteration/ Product tampering

Portugal
 Fats and oils
 Adulteration/ Product tampering

Italy
 Bivalve molluscs and products thereof
 Cephalopods and products thereof
 Crustaceans and products thereof
 Fish and fish products
 Gastropods
 Grey market

JRC Food Fraud

Monthly Report

April 2024



Untargeted approaches for food/feed analysis - applications



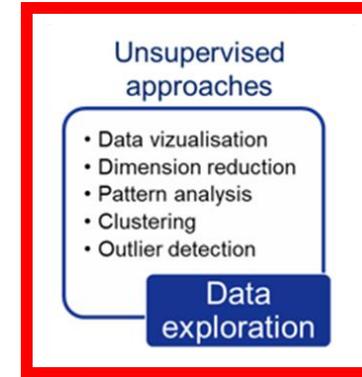
Untargeted

chemometric approaches

Unsupervised methods

Exploratory analysis

Pattern analysis



A classical Chemometric tool: Principal Component Analysis (PCA)

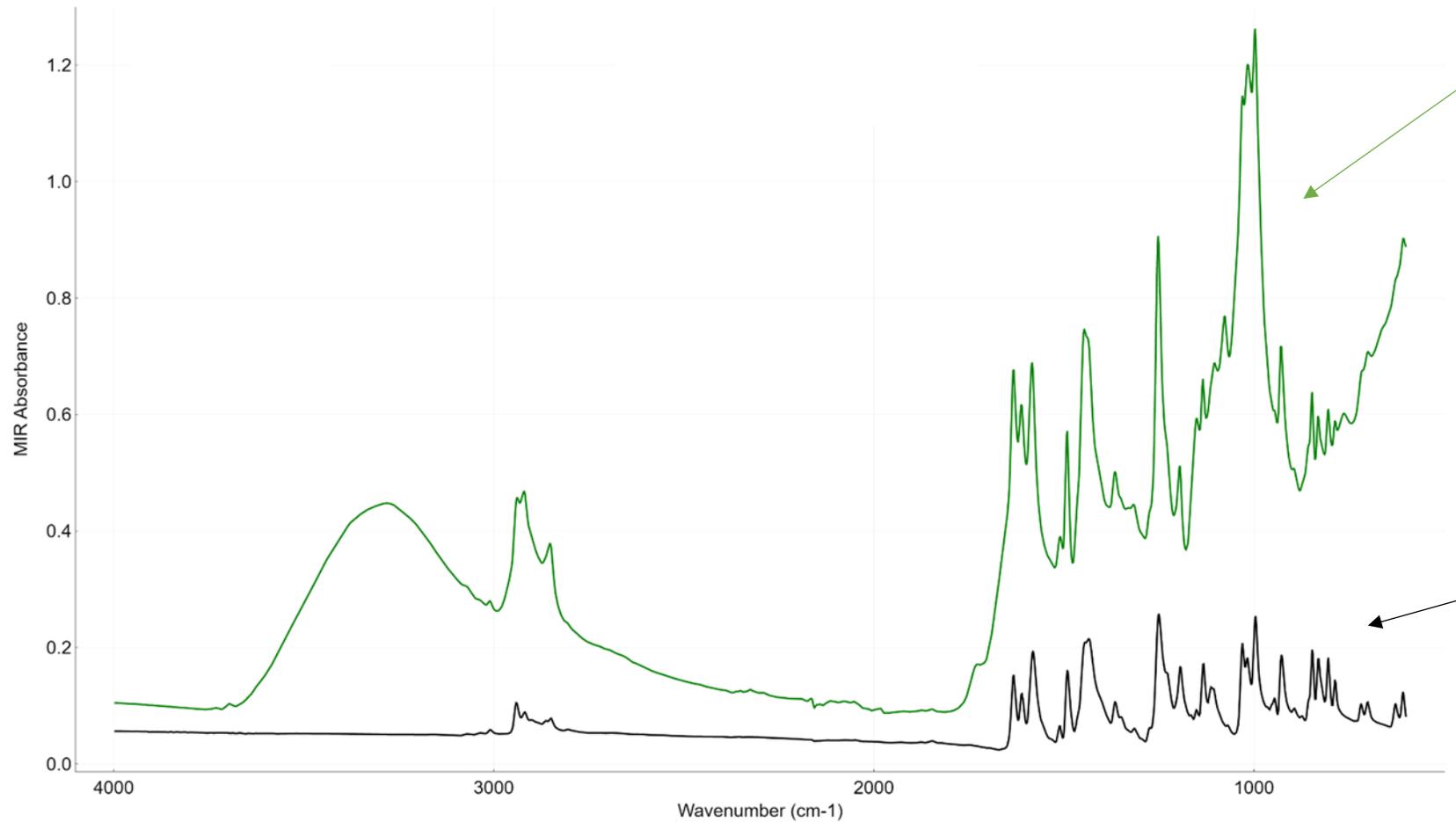
- Moving window PCA, Local PCA
- Determination of different distances : Mahalanobis, GH ...
- t-SNE

are usually used to assess food/feed quality and safety :

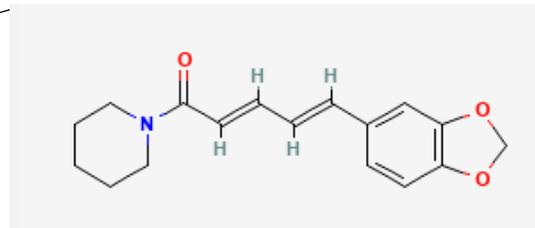
- ✓ to **authenticate** a product,
- ✓ to detect **adulteration** with a harmful or non edible ingredient,
- ✓ To detect **fraud** (mislabelling, deceiving practices, and ingredients replacement),
- ✓ To assess **freshness**,
- ✓ To determine **geographical origin, origin masking** (food authenticity changed – counterfeiting).

Pepper powder fraud / adulteration

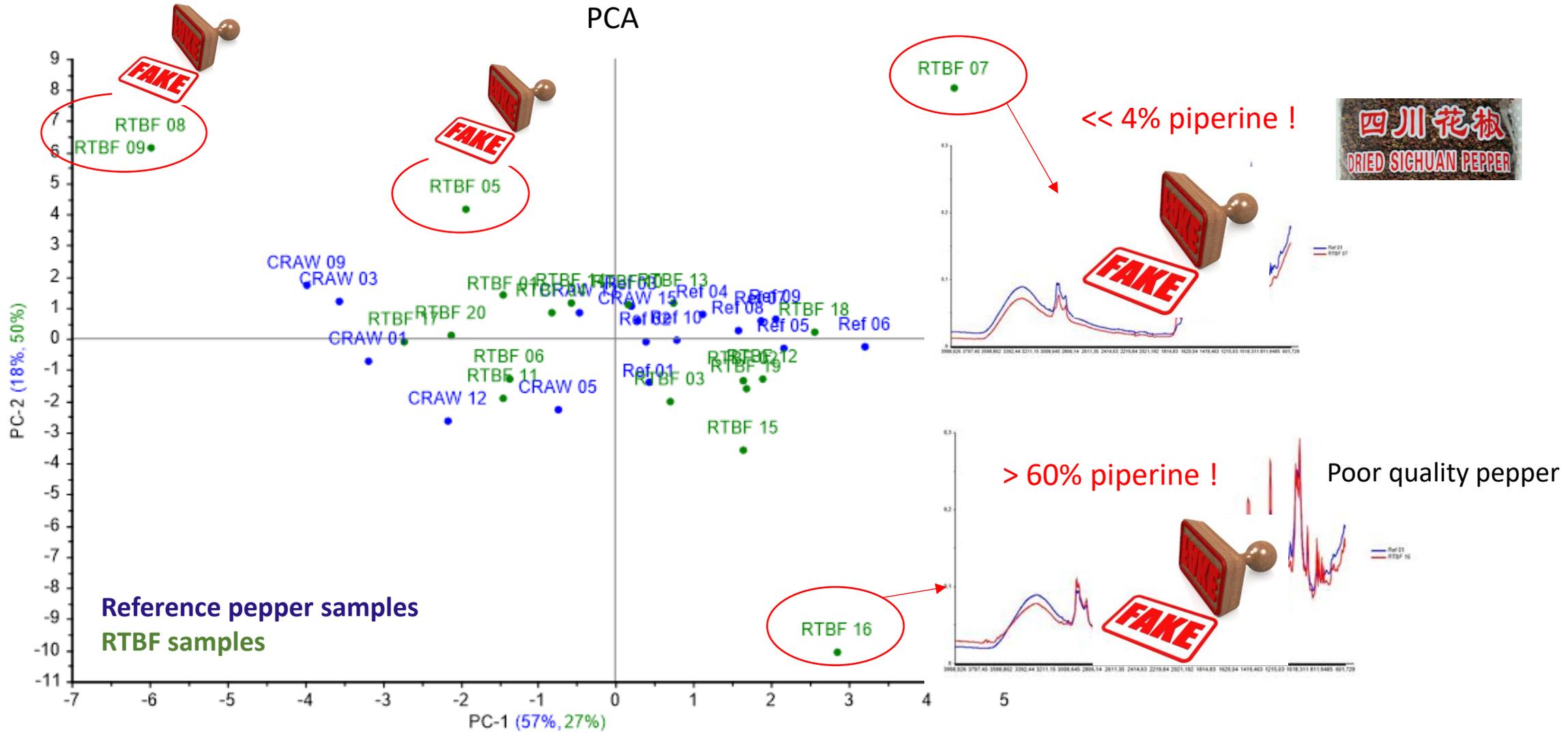
Standard (reference) pepper



Piperine $C_{17}H_{19}NO_3$



Pepper powder fraud / adulteration

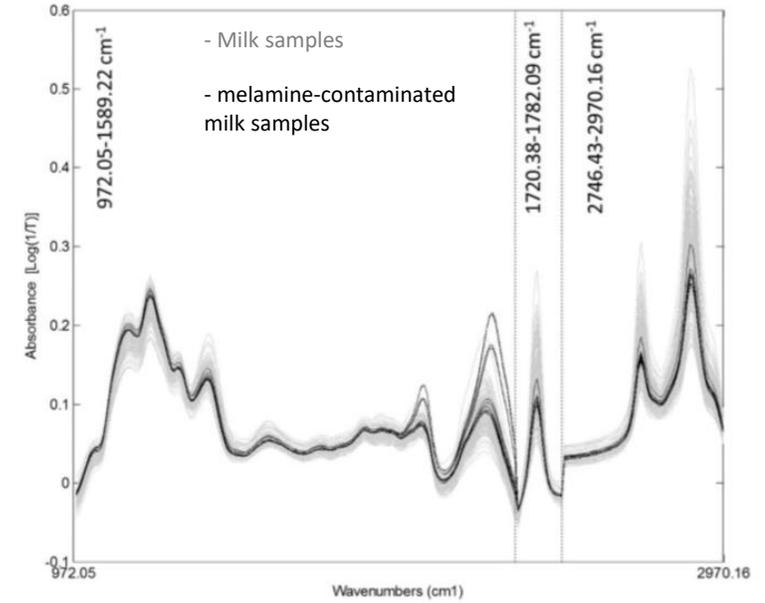
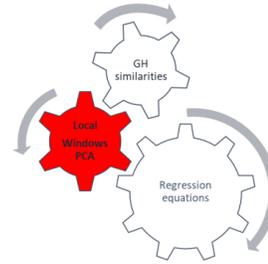


Untargeted approaches for food/feed analysis – Melamine cases

 **Chemometrics and Intelligent Laboratory Systems** 
Volume 152, 15 March 2016, Pages 157–162

Use of a multivariate moving window PCA for the untargeted detection of contaminants in agro-food products, as exemplified by the detection of melamine levels in milk using vibrational spectroscopy ☆

J.A. Fernández Pierna, D. Vincke, V. Baeten, C. Grelet, F. Dehareng, P. Dardenne  



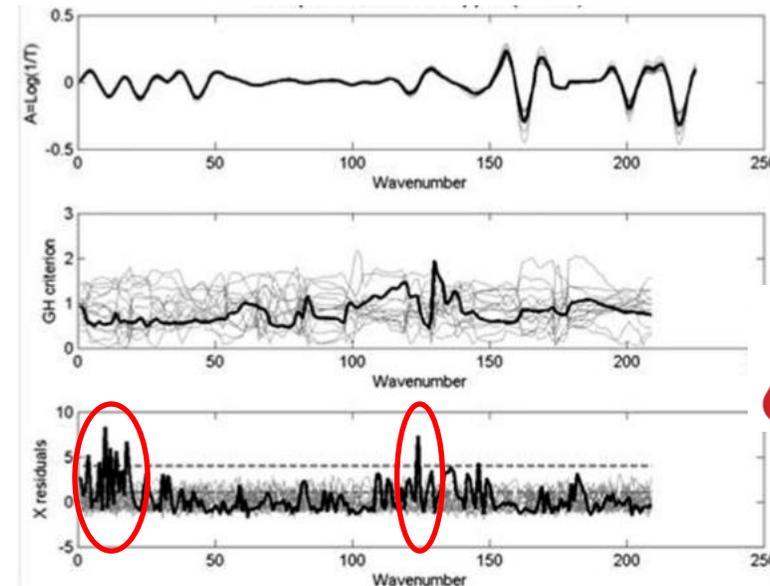
Local Window Principal Component Analysis (LWPCA)

 **Food Control** 
Volume 119, January 2021, 107459

Chemometric non-targeted analysis for detection of soybean meal adulteration by near infrared spectroscopy

O.Ye Rodionova ^a , J.A. Fernández Pierna ^b , V. Baeten ^b , A.L. Pomerantsev ^a 

One Class modelling



Melamine!

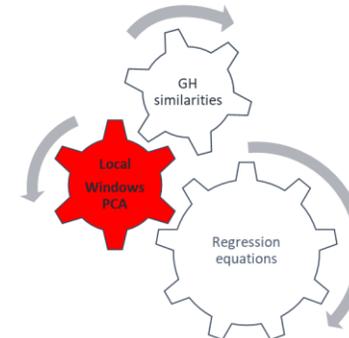
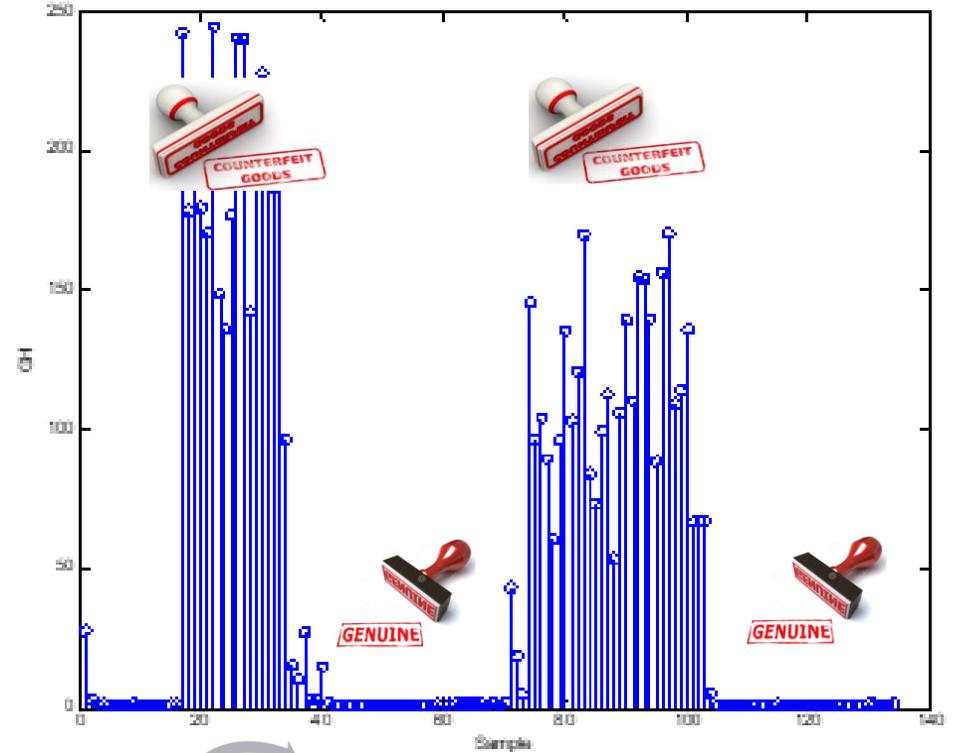
Untargeted approaches for food/feed analysis – Melamine cases



NIR fingerprint screening for early control of non-conformity at feed mills



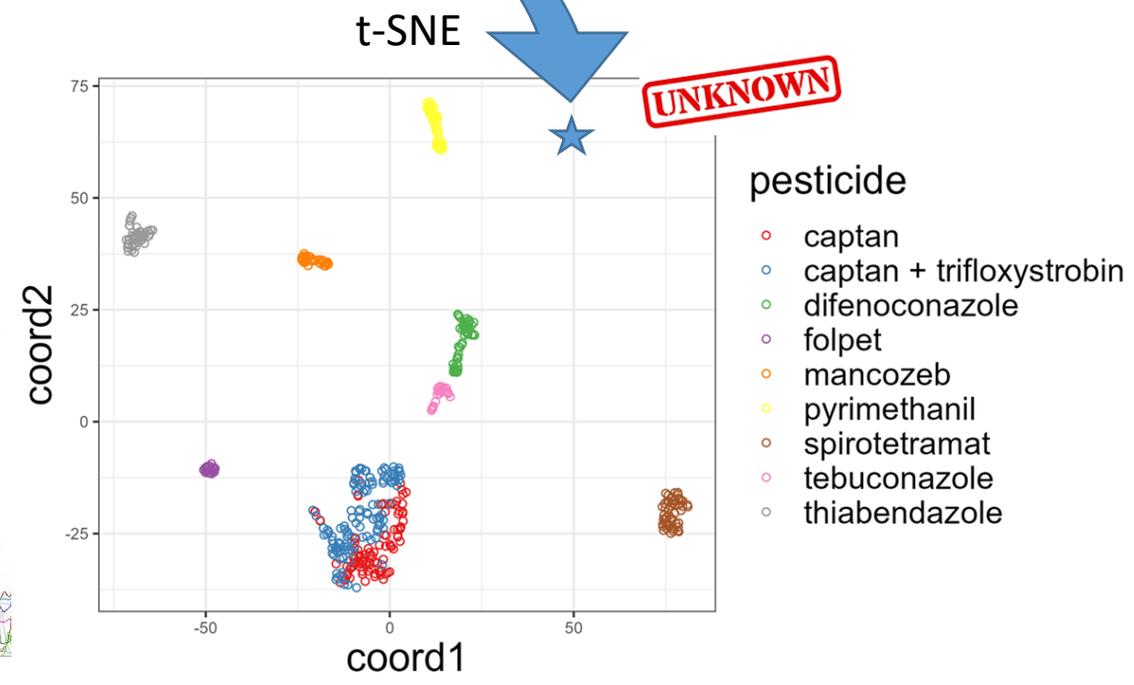
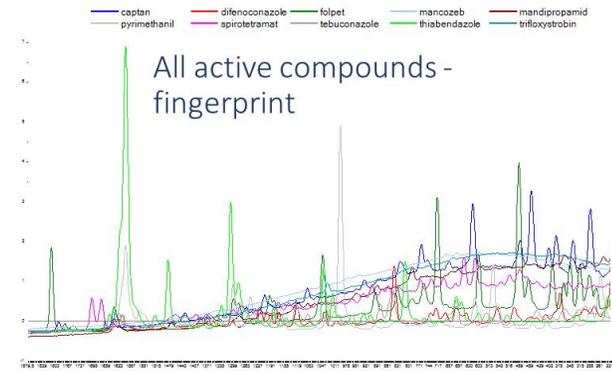
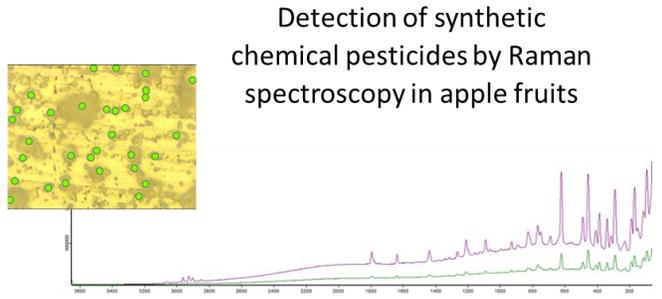
Juan Antonio Fernández Pierna^{a,*}, Ouissam Abbas^a, Bernard Lecler^a, Patrick Hogrel^b, Pierre Dardenne^a, Vincent Baeten^a



provimi
shaping tomorrow's nutrition



Untargeted approaches – pesticides in fruits



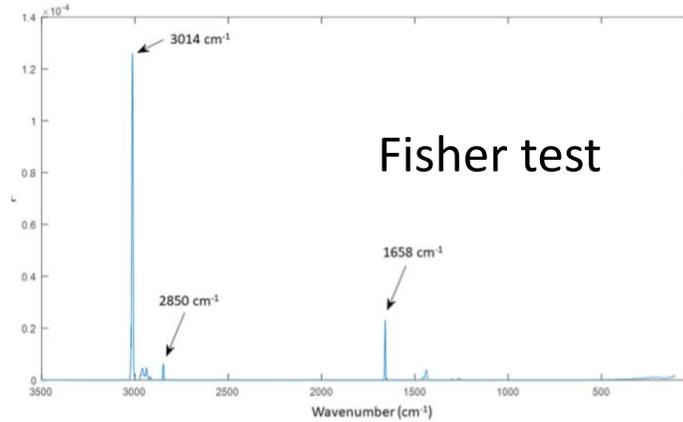
Untargeted approaches – characterization of oils

RESEARCH ARTICLE

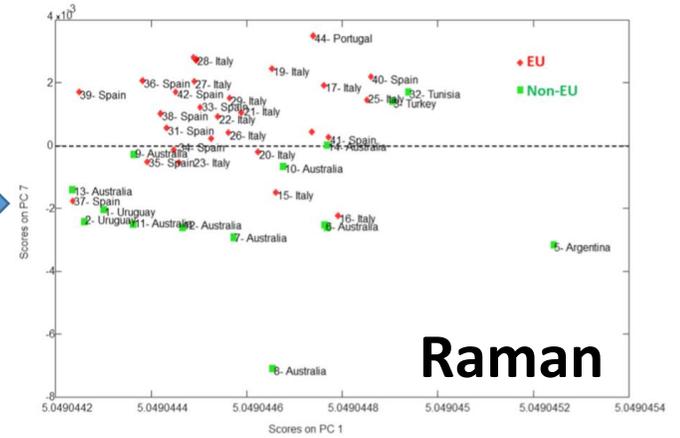
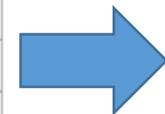
European Journal of
Lipid Science and Technology
www.ejlst.com

Assessment of Vibrational Spectroscopy Performance in Geographical Identification of Virgin Olive Oils: A World Level Study

Noelia Tena, Ramón Aparicio, Vincent Baeten, Diego Luis García-González,* and Juan Antonio Fernández-Pierna



Fisher test



Raman

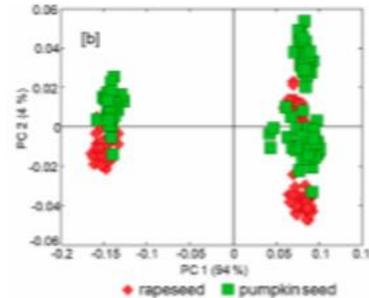
Food Analytical Methods
https://doi.org/10.1007/s12161-023-02568-4

RESEARCH

Comparison of Spectroscopic Techniques Using the Adulteration of Pumpkin Seed Oil as Example

Carolin Lörchner^{1,2}, Carsten Fauhl-Hassek¹, Marcus A. Glomb², Vincent Baeten³, Juan A. Fernández Pierna³, Susanne Esslinger¹

Received: 10 May 2023 / Accepted: 18 December 2023
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Chemometrics and Intelligent Laboratory Systems 240 (2023) 104904

Contents lists available at ScienceDirect

Chemometrics and Intelligent Laboratory Systems

journal homepage: www.elsevier.com/locate/chemometrics



Towards common useable spectra in non-targeted analysis - A feasibility study by mid-infrared spectroscopy, transfer and correction approaches

Carolin Lörchner^{a,b}, Carsten Fauhl-Hassek^a, Marcus A. Glomb^b, Vincent Baeten^c, Juan A. Fernández Pierna^c, Susanne Esslinger^{a,*}

Untargeted approaches for food/feed analysis - Conclusions

Non-targeted chemometric approaches offer valuable capabilities for **comprehensive analysis and characterization of food samples**. They have a large number of advantages. However, they also pose challenges and limitations.



Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:

Comprehensive Analysis

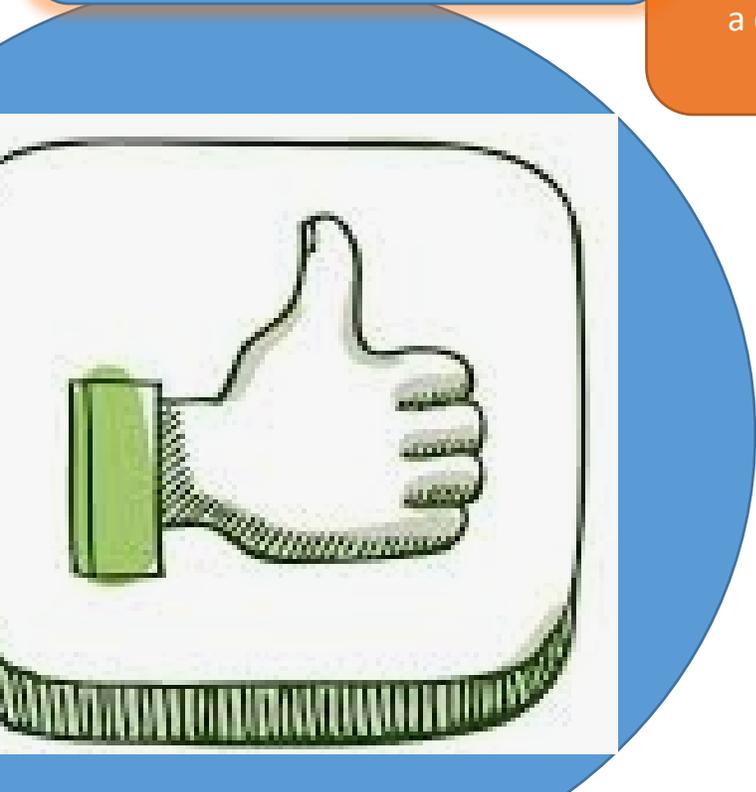


Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:

Comprehensive Analysis

Non-targeted chemometric approaches provide a **more complete picture of the chemical composition** of the food, allowing for a deeper understanding of its quality and safety attributes.

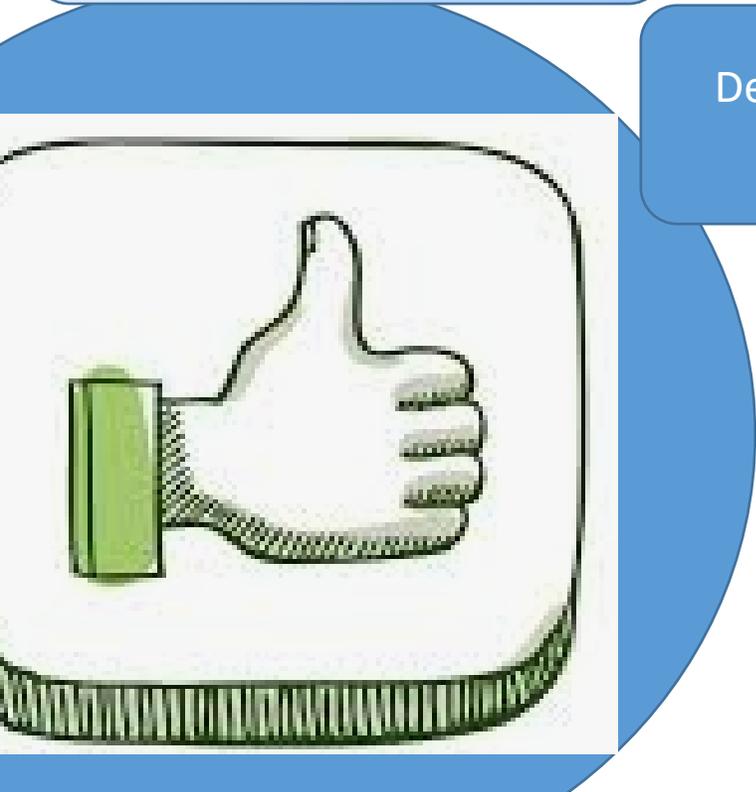


Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:

Comprehensive Analysis

Detection of Unexpected Compounds



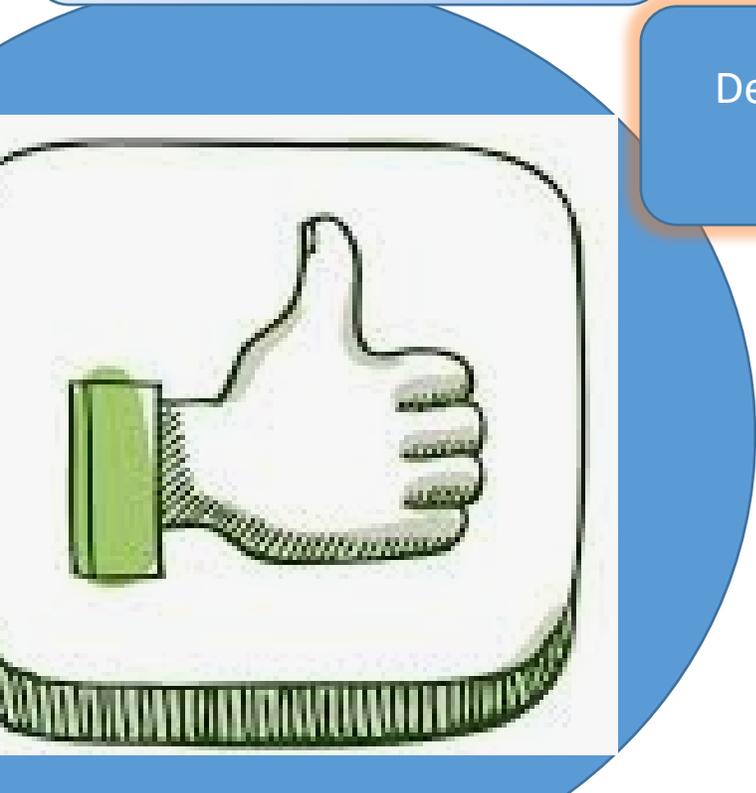
Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:

Comprehensive Analysis

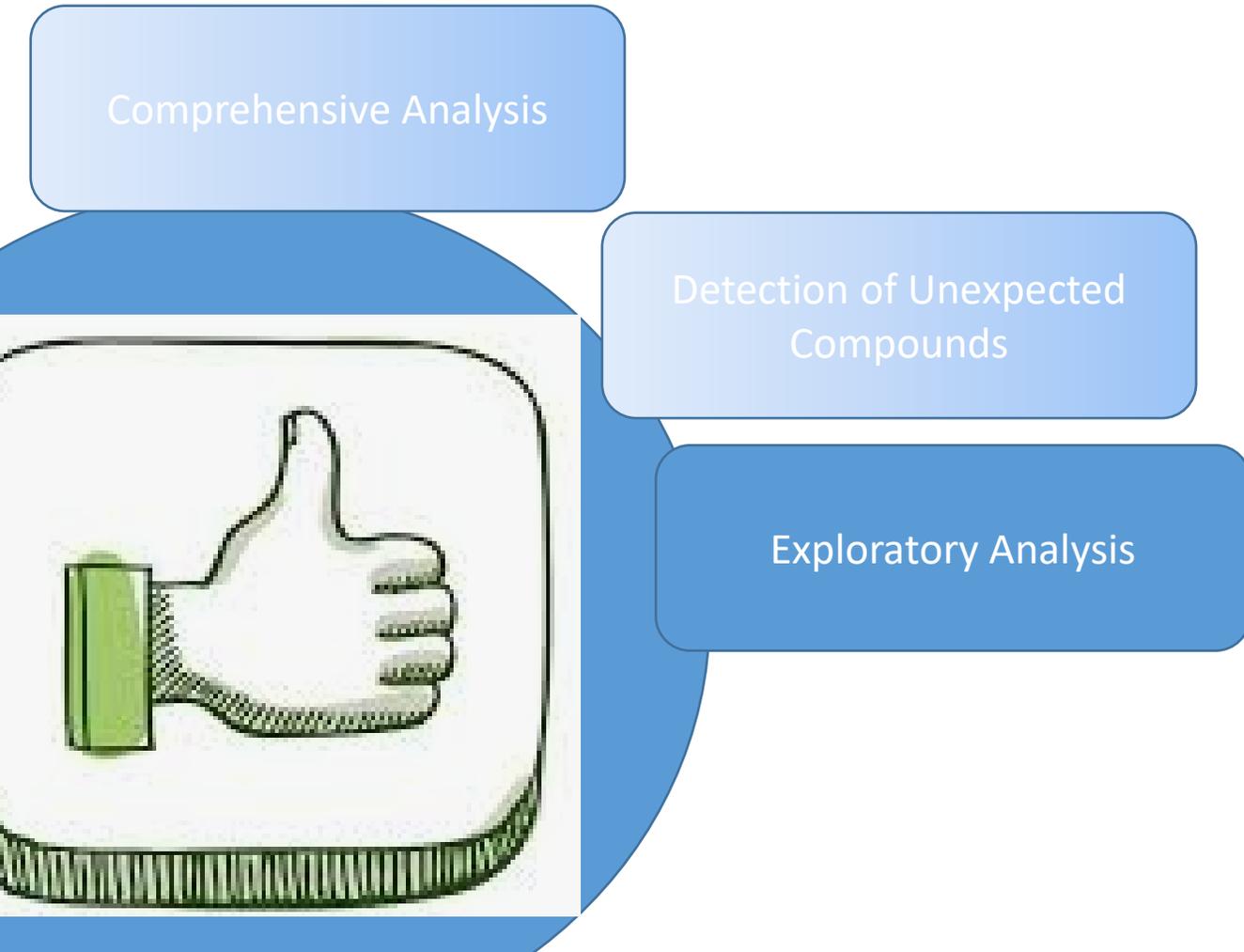
Detection of Unexpected Compounds

These methods do not rely on predefined target analytes, making them well-suited for the discovery of emerging contaminants, adulterants, or unexpected chemical variations that may pose risks to food safety



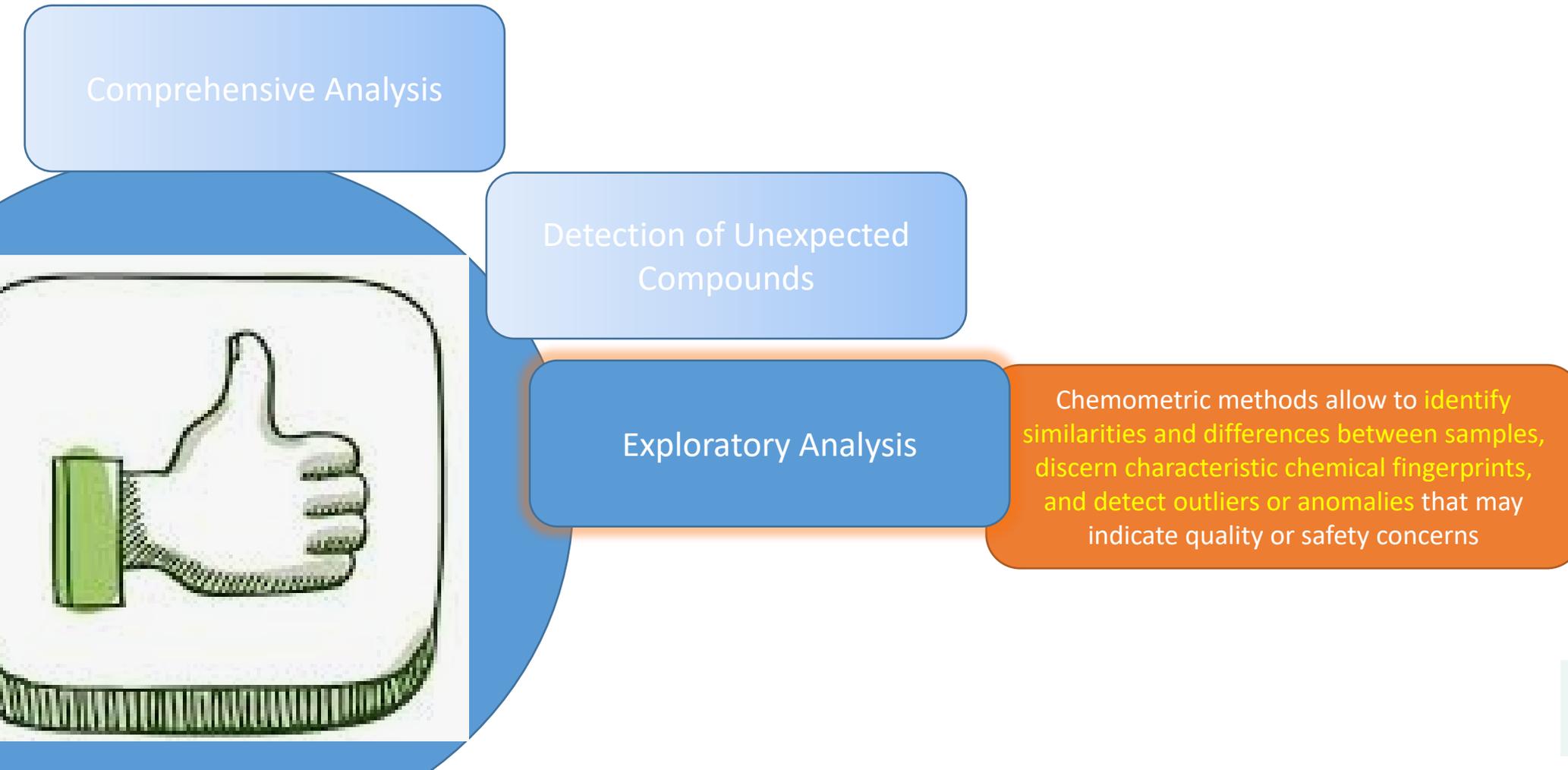
Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:



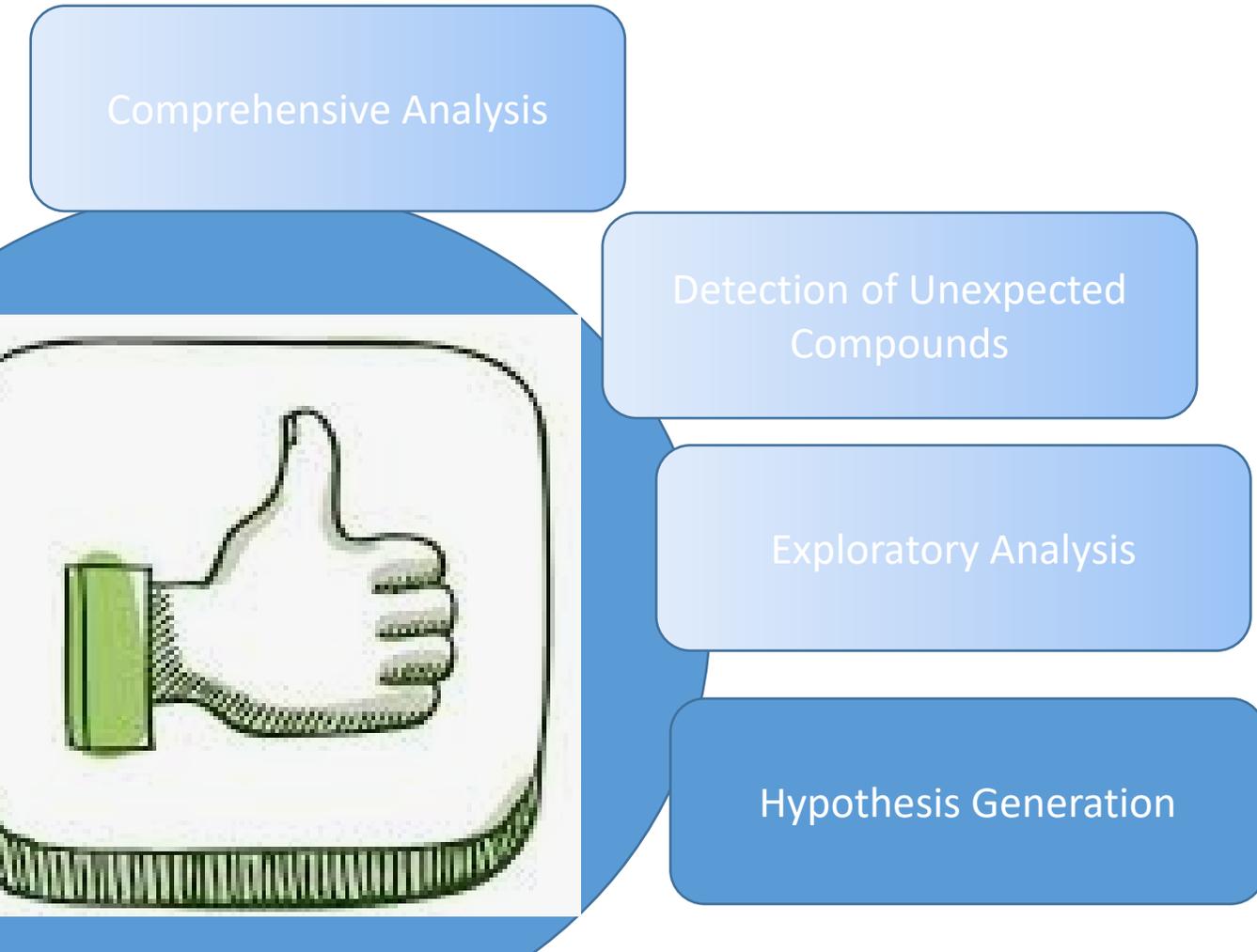
Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:



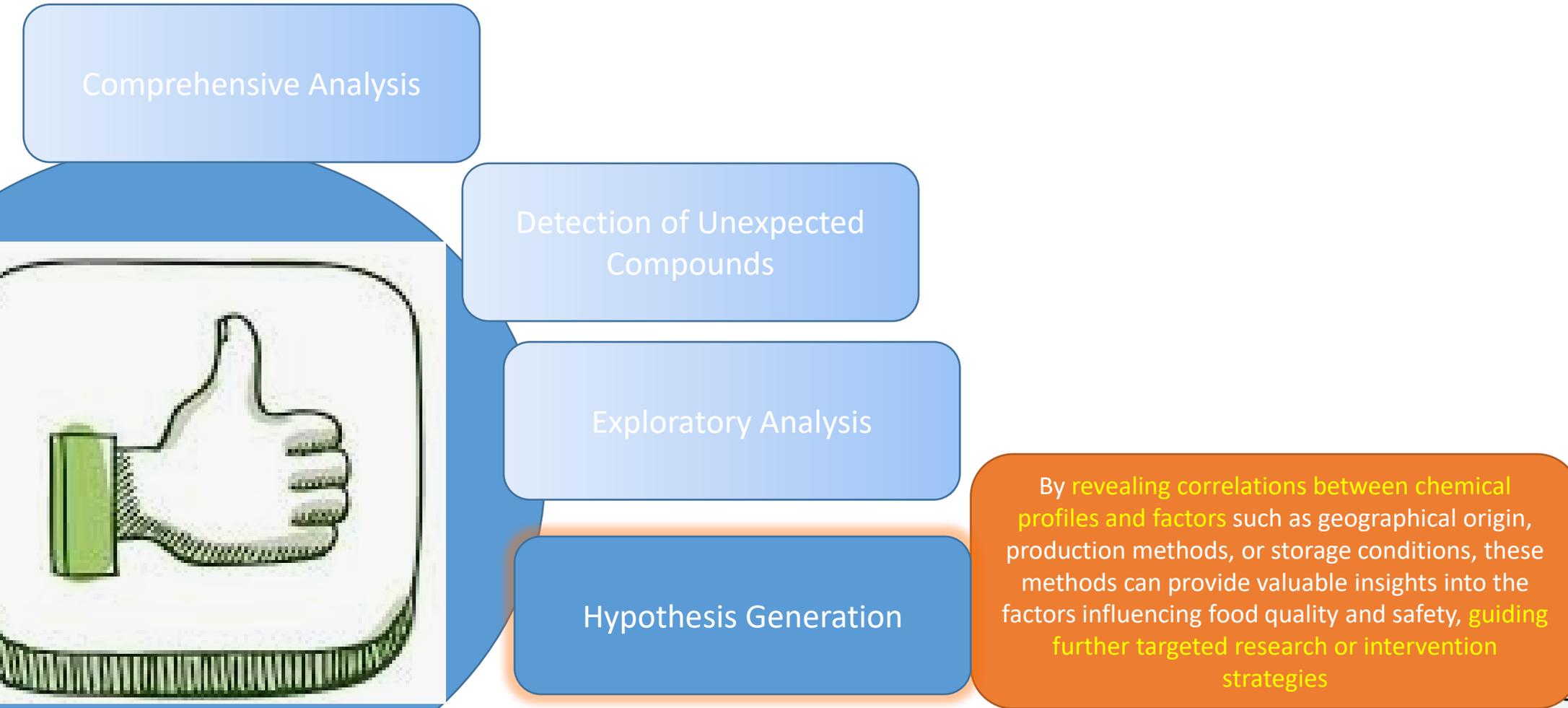
Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:



Untargeted approaches for food/feed analysis - advantages

Some advantages of non-targeted chemometric approaches:



Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:

Complex Data
Interpretation



Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:

Complex Data Interpretation

They require advanced statistical and computational expertise



Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:

Complex Data
Interpretation

Unknown Compounds
and Matrix effects



Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:

Complex Data Interpretation

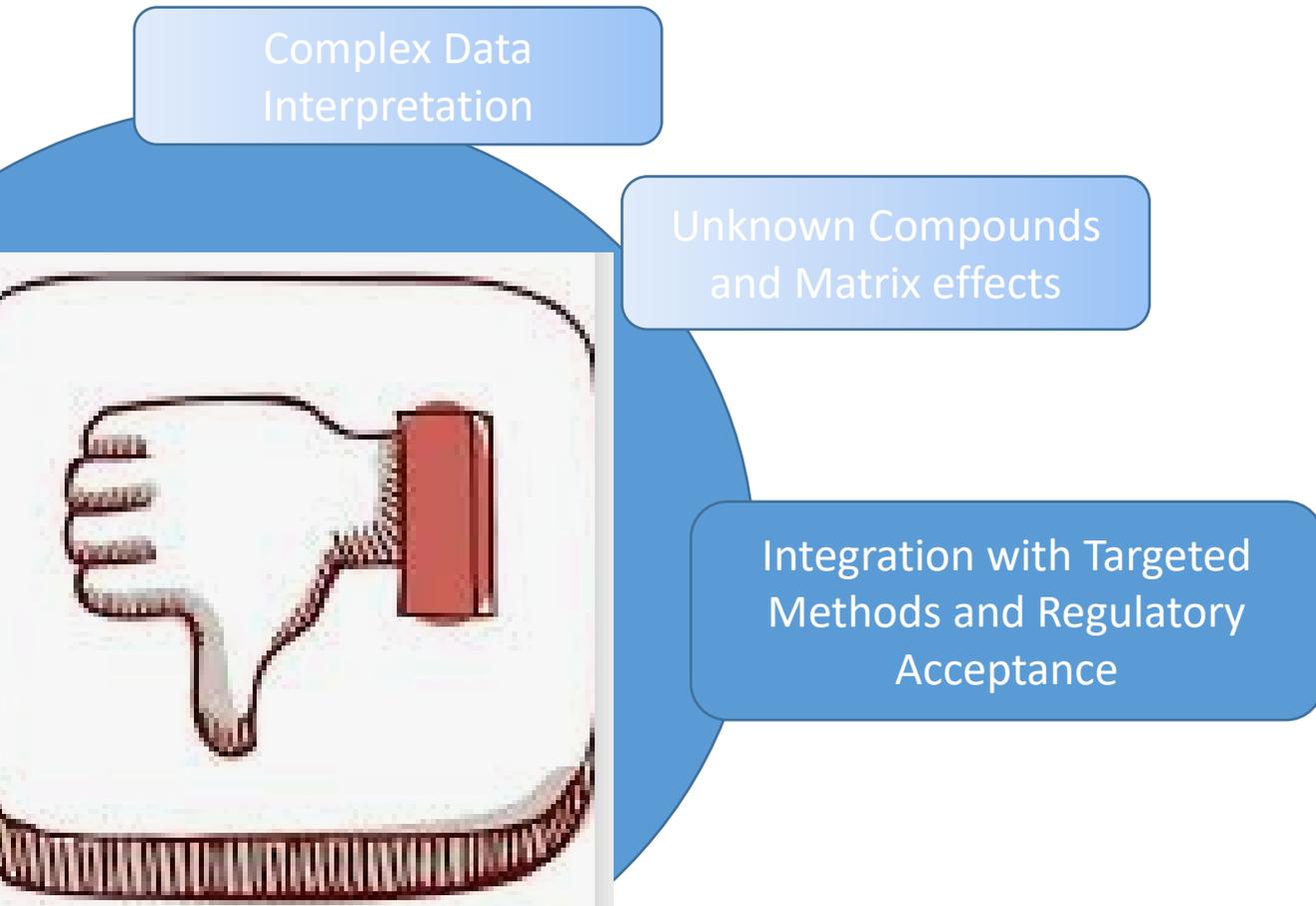
Unknown Compounds and Matrix effects

Without reference standards or databases for comparison, **elucidating the identity and potential hazards** of compounds can be difficult



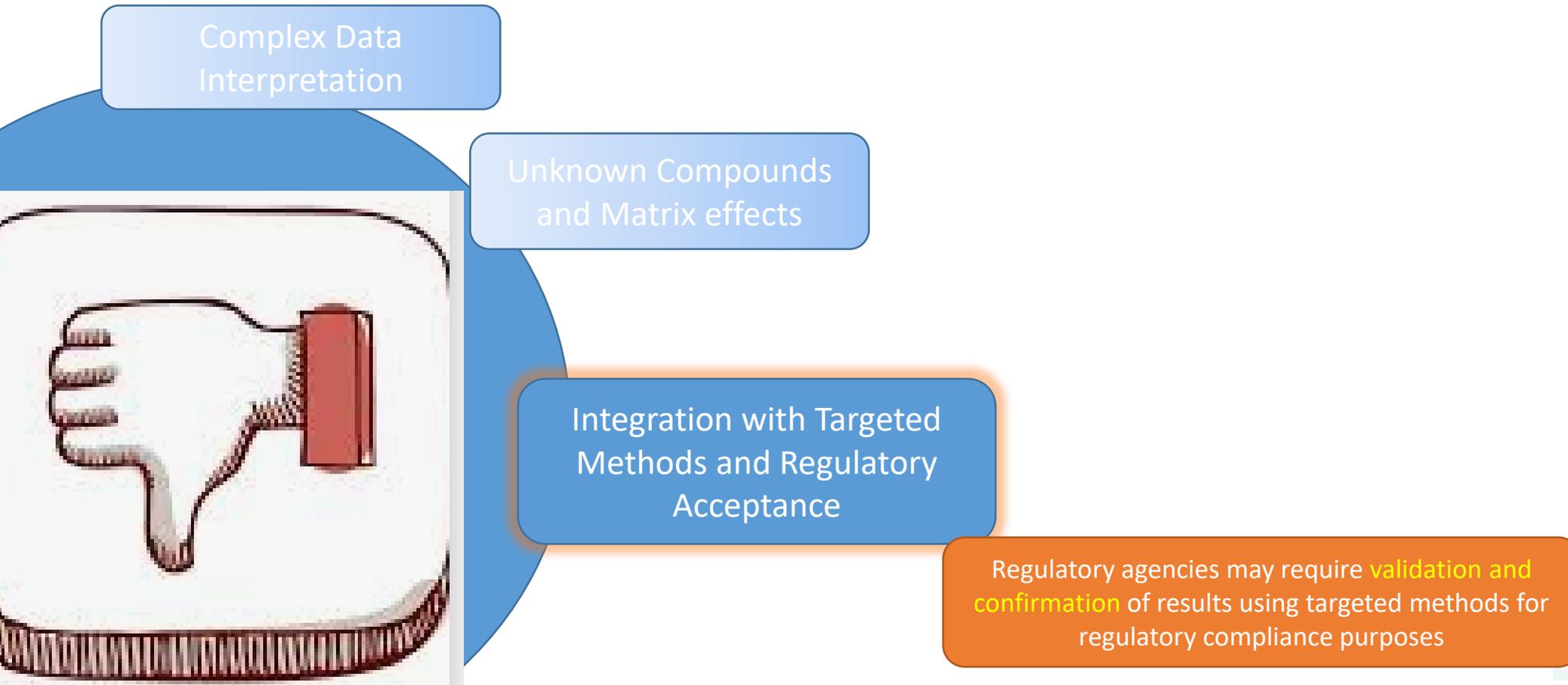
Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:

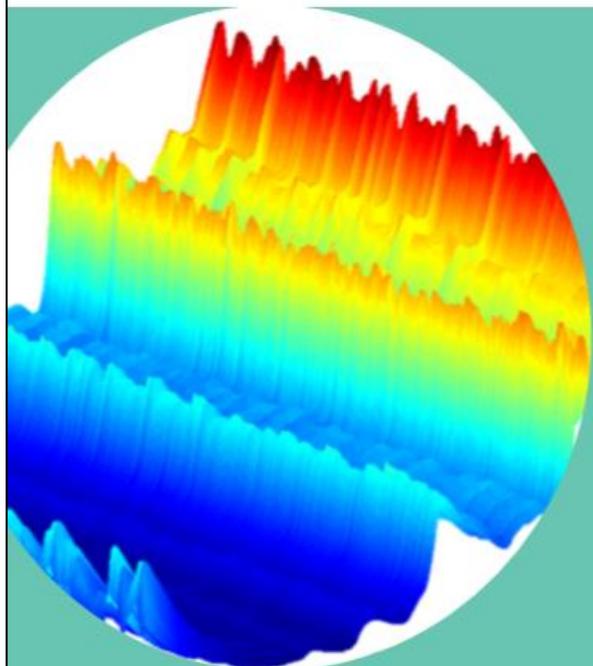


Untargeted approaches for food/feed analysis - limitations

Non-targeted chemometric approaches pose challenges and limitations related to:



Vibrational Spectroscopy and Chemometrics course



14-18 OCTOBER 2024
GEMBLoux BELGIUM



Invited Speakers



Beatriz CARRASCO
(Mining4Quality)



Tom FEARN
(University College London)



Wouter SAEYS
(KU Leuven)



Pierre DARDENNE
(Consultant)

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<http://www.cra.wallonie.be>



Data sciences **R**aman **I**maging **M**id-IR
laboratory



Quentin Arnould / Delphine Delhotte / Antoine Deryck / Juan Antonio Fernández
Maxime Joissains / Nicaise Kayoka / François Stevens

