International Conference on
EXTRACTION METHODS
for Valorising Fibers from
FOOD & AGRO-PROCESSING WASTE
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Near-Infrared Spectroscopy applications for fiber quantification and characterization as a strategic tool for waste valorization in food and feed systems

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ENFiber
European Network for Valorising Food Processing Waste into Sustainable Fibers

cost
EUROPEAN COOPERATION IN SCIENCE & TECHNOLOGY

Funded by the European Union

INTRODUCTION

Transition towards a circular economy

Valorisation strategies require...

PRECISE, REAL-TIME CHEMICAL PROFILING

...to drive the sustainable transformation of residues into **valuable resources**.

Do valuable resources meet **standards of the food and non-food industries**?

Without understanding *the exact* ***chemical composition*** *of the initial waste stream, integrated* ***recovery efforts risk inefficiency*** *or* ***failure***.

A comprehensive **determination of fiber fractions** is essential prior to processing

NEAR INFRARED SPECTROSCOPY

Key tool in the valorisation strategy. Rapid, non-destructive determination of fiber fractions.

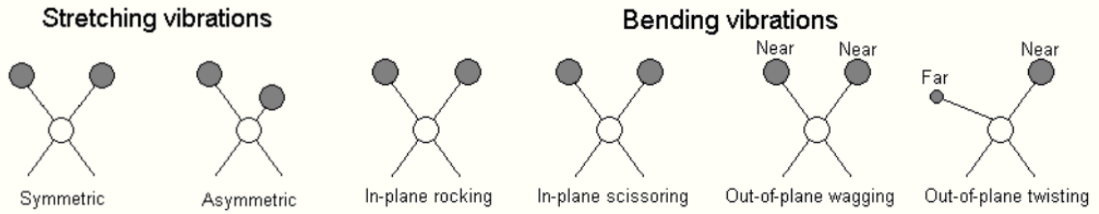
INTRODUCTION

NEAR INFRARED SPECTROSCOPY

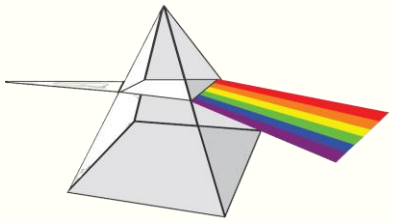
(Shenk et al., 2001)

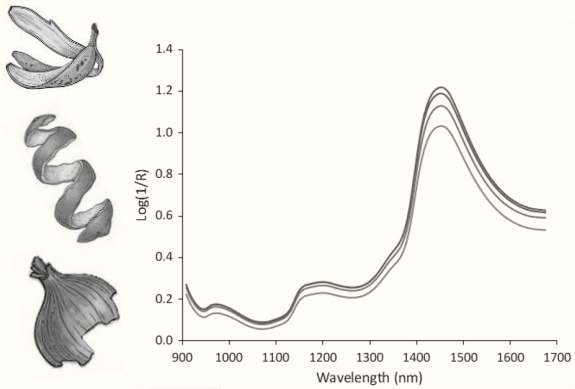
Based on the **absorption** that occurs when **near-infrared radiation** vibrates at the same specific **frequency** as the **molecular bonds** within the analysed sample.

Chemical bonds → **C – H**, **O – H**, **N – H**, **C – O** →



NIR devices





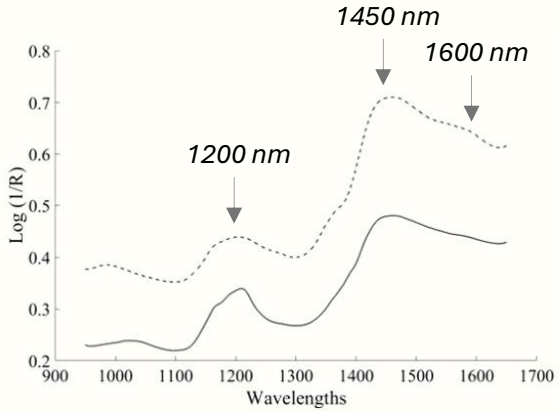
OBJECTIVE

The **objective** of this work is to provide a **comprehensive review** of the research conducted by the Department of Animal Production and the Department of Bromatology and Food Technology of the **University of Cordoba (Spain),** with particular emphasis on the optimisation of **NIR applications for fiber analysis across the food and feed value chains**.

MATERIAL AND METHODS

1. Spectral analysis and spectral repeatability

Spectra visualization Identification of bands and spectral anomalies



- 1200 nm, second overtone absorbance of C-H
- 1450 nm, first overtone absorbance O-H
- 1600 nm, first overtone absorbance of O-H

C-H, O-H, N-H

SUGARS, LIPIDS, WATER, PROTEINS, FIBERS…

RMS Similarity between different spectra of a single sample

Shenk and Westerhaus (1991)

Assurance of spectral data quality

$$RMS_{j,k} = \sqrt{\frac{\sum_{i=1}^{n} D_{ij}^2}{n}} \qquad STD_k = \sqrt{\sum_{j=1}^{N} (RMS_j)^2/(N-1)} \qquad STD_{limit} = 1.036 \times \sqrt{\sum_{k=1}^{k=m} \frac{STD_k^2}{m}}$$

MATERIAL AND METHODS

2. Structure of the population and spectral outliers

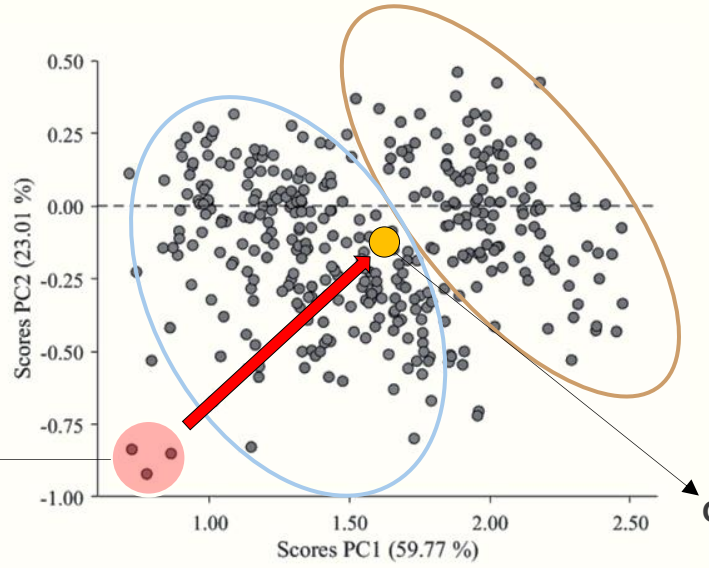
Data dimensionality reduction - Principal components analysis (PCA)

- PCA scores and loadings plot analysis, groups and trends.
- Distance of each sample to the center of the population.

Mahalanobis distance (GH)

$GH_{threshold} = 3.5$

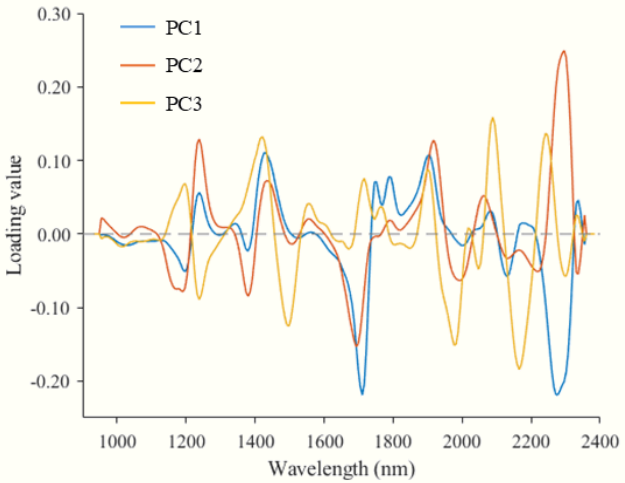
Shenk and Westerhaus (1995)



Potential outlier samples

GH > 3.5

Center of the population



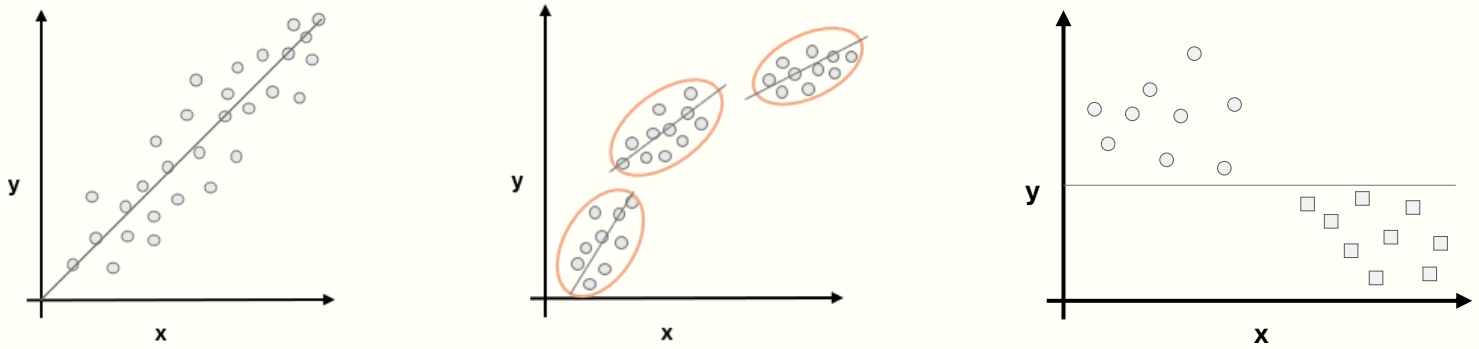
MATERIAL AND METHODS

3. Calibration of quantitative and qualitative models

- Calibration and validation groups.
- Linear and non-linear approaches for quantitative and qualitative models.
  - **Partial Least Squares Regression (PLSR)**
  - **Local Regressions (LR)**
  - **Partial Least Squares Discriminant Analysis (PLSDA)**

Optimization through cross-validation

SECV, R^2_{cv}, RPD_{cv}, NER



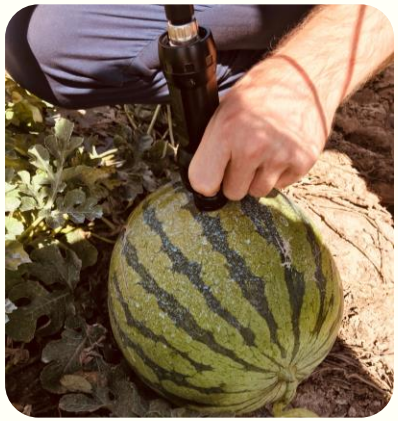
4. External validation

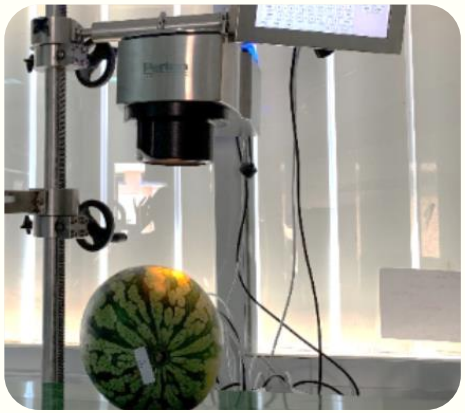
Protocol: **Windham et al. (1989)**

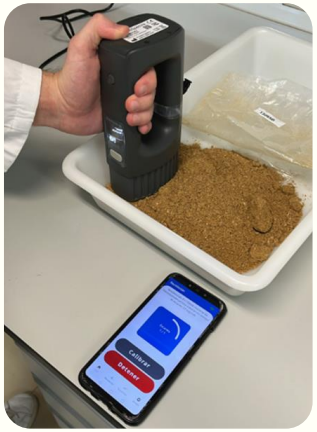
SEP, Bias, SEP(c), R^2_p, RPD_p, Slope

MATERIAL AND METHODS

5. Applications







Bec et al. (2020)
Huang and Yu (2020)

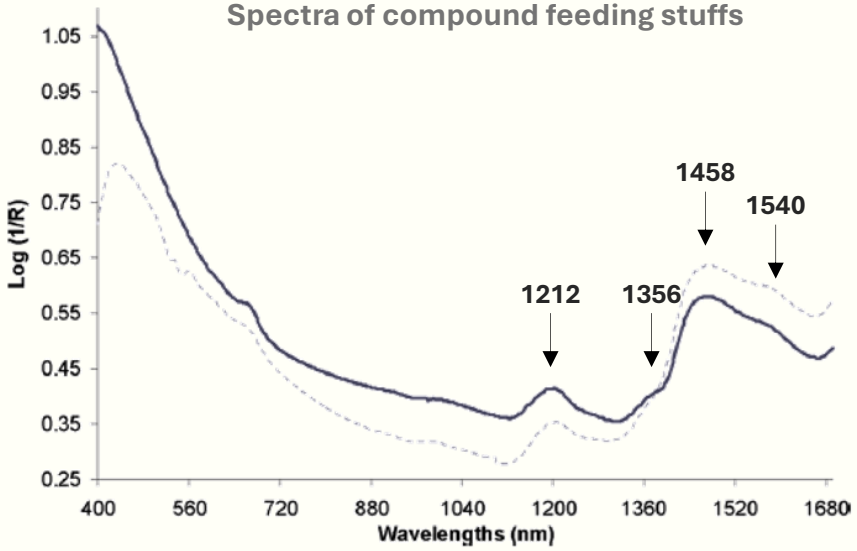
RESULTS AND DISCUSSION

Most important fiber-related absorption bands

Spectral band	Molecular bond vibration
1212 nm	C-H stretching
1356 nm	C-H stretching
1458 nm	C-H and O-H stretching
1540 nm	O-H stretching

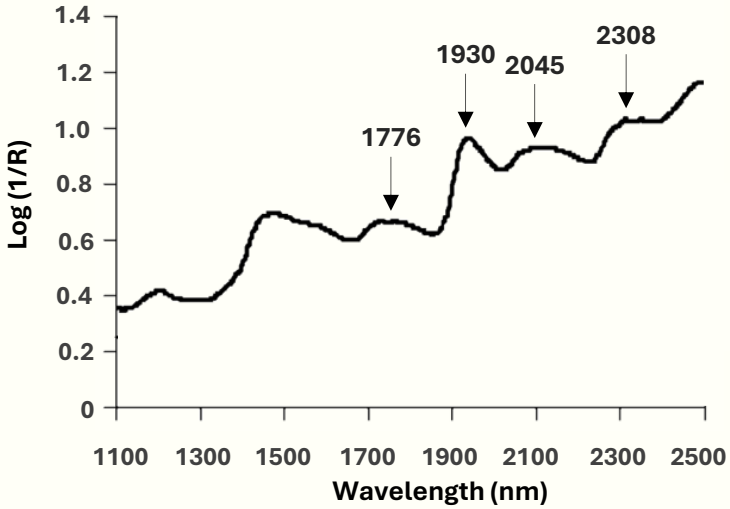
Spectral band	Molecular bond vibration
1776 nm	C-H and C-H_2 stretching
1930 nm	O-H asymmetric stretching
2095 nm	C-H & O-H stretching
2308 nm	C-H_2 bend & O-H stretching

Spectra of compound feeding stuffs



Fernández-Ahumada et al. (2008)

Spectrum of compound feeding stuffs



Pérez-Marín et al. (2004)

RESULTS AND DISCUSSION

WILEY Online Library
Journal of Food Quality

PREDICTION OF NEUTRAL AND ACID DETERGENT FIBER CONTENT OF GREEN ASPARAGUS STORED UNDER REFRIGERATION AND MODIFIED ATMOSPHERE CONDITIONS BY NEAR-INFRARED REFLECTANCE SPECTROSCOPY

A. GARRIDO, M.T. SÁNCHEZ, G. CANO, D. PÉREZ, C. LÓPEZ

Calibration models on asparagus (*Asparagus officinalis*). *Garrido-Varo et al. (2001)*

- Study of changes of the fiber content during harvesting and cold storage.
- Foss-NIRSystems 6500 SY-II monochromator (400 to 2498 nm, every 2 nm)
- Linear approach for quantitative models, Modified PLSR.

Calibration statistics for the equations obtained for the prediction of NDF and ADF for green asparagus stored under refrigeration conditions

Constituent	N	Math treatment	SECV	r^2_{cv}	RPD
NDF	46	0,0,1,1	0.91	0.98	7.19
	48	1,4,4,1	1.07	0.97	5.99
	48	1,5,5,1	1.07	0.97	6.01
	46	2,4,4,1	0.84	0.98	7.81
	46	2,5,5,1	0.84	0.98	7.80
ADF	50	0,0,1,1	0.64	0.97	5.91
	51	1,4,4,1	0.62	0.97	6.07
	51	1,5,5,1	0.62	0.97	6.07
	50	2,4,4,1	0.60	0.97	6.32
	50	2,5,5,1	0.60	0.97	6.38

RESULTS AND DISCUSSION

Asparagus classification by fiber content.

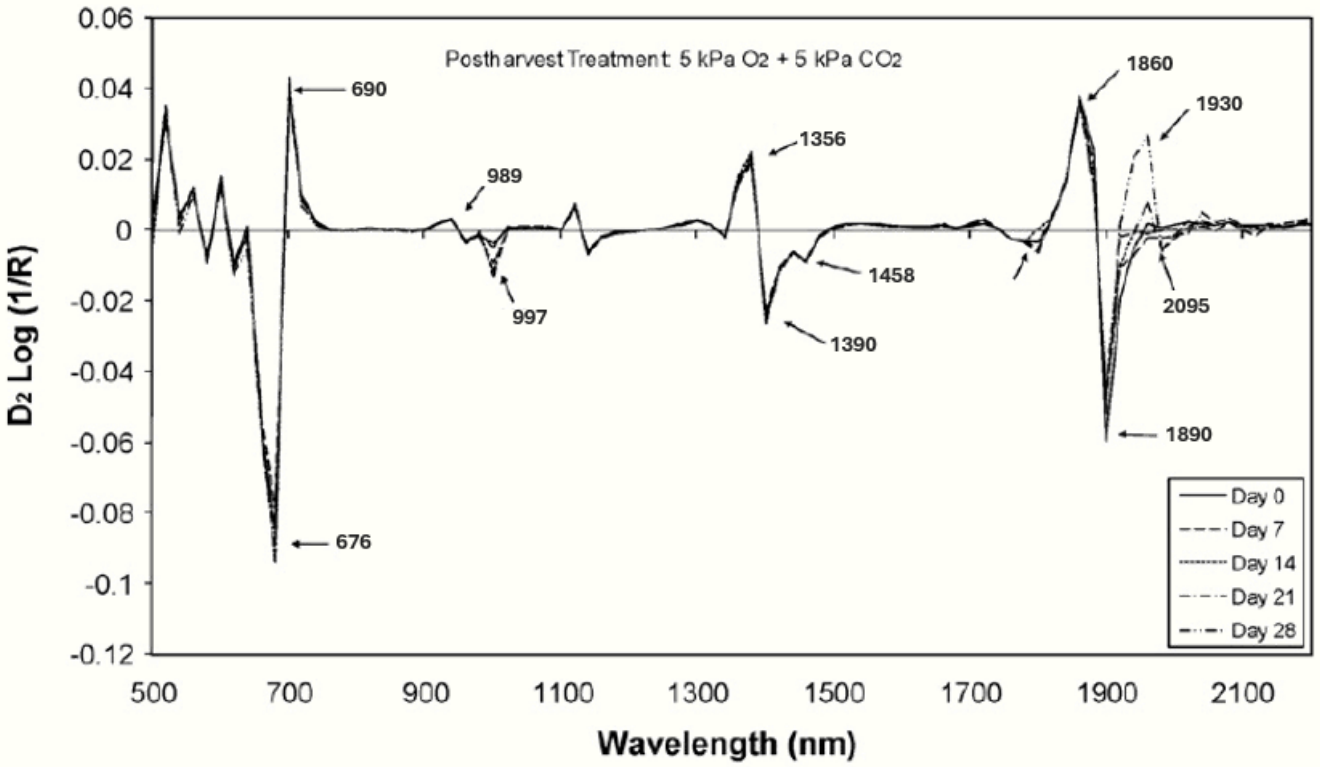
Sánchez et al. (2009)

Talanta
Volume 78, Issue 2, 30 April 2009, Pages 530-536

Use of near-infrared reflectance spectroscopy for shelf-life discrimination of green asparagus stored in a cool room under controlled atmosphere

María-Teresa Sánchez [a], Dolores Pérez-Marín [b], Katherine Flores-Rojas [a], José-Emilio Guerrero [b], Ana Garrido-Varo [b]

- Foss-NIRSystems 6500 SY-II monochromator (400 to 2498 nm, every 2 nm)
- Discrimination of asparagus by shelf-life. Changes in O-H absorptions (fiber)



RESULTS AND DISCUSSION

Calibration models on forage.

Fernández-Cabanás et al. (2023)

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy
Volume 285, 15 January 2023, 121922

Optimisation of the predictive ability of NIR models to estimate nutritional parameters in elephant grass through LOCAL algorithms

Víctor M. Fernández-Cabanás [a], Dolores C. Pérez-Marín [b], Tom Fearn [c], Joadil Gonçalves de Abreu [d]

- Calibration models on neutral (NDF) and acid detergent fiber (ADF).
- Foss-NIRSystems 6500 SY-II monochromator (400 to 2498 nm, every 2 nm)
- Linear (PLSR) and non-linear (LR) approaches for quantitative models.

PLSR, prediction statistics

Constituent	Statistic	Value
NDF	SEP	3.42
	Bias	-0.21
	$SEP_{(c)}$	3.42
	Slope	0.94
	R^2_p	0.45*
ADF	SEP	4.00
	Bias	-0.17
	$SEP_{(c)}$	4.01
	Slope	1.03
	R^2_p	0.64

LR, prediction statistics

Constituent	Statistic	Value
NDF	SEP	3.11
	Bias	-0.19
	$SEP_{(c)}$	3.11
	Slope	0.98
	R^2_p	0.55*
ADF	SEP	3.70
	Bias	-0.06
	$SEP_{(c)}$	3.71
	Slope	1.07
	R^2_p	0.70

**Exceed validation limits. Protocol: Windham et al. (1989)*

RESULTS AND DISCUSSION

Data transfer between NIR devices.

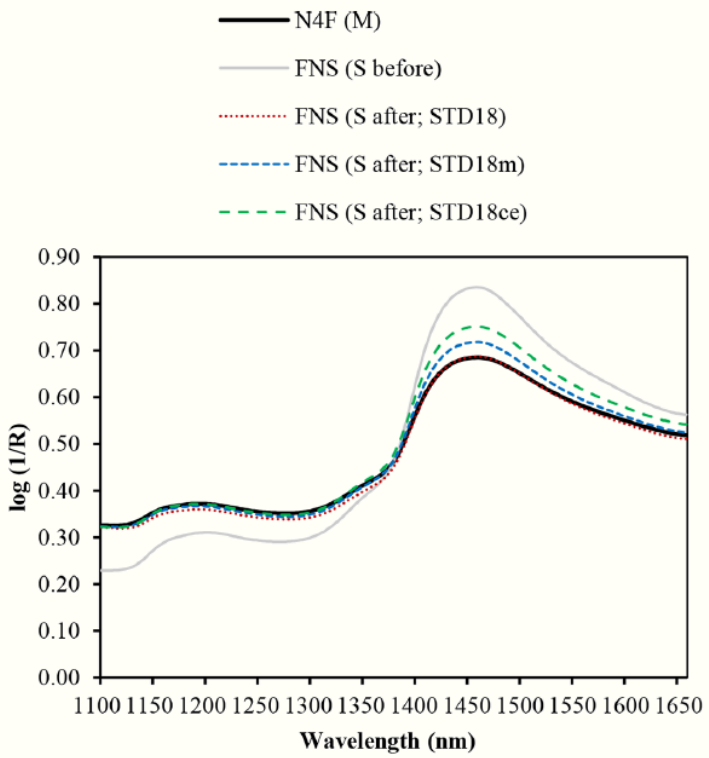
Pérez-Marín et al. (2022)

J. Dairy Sci. 105:2380–2392
https://doi.org/10.3168/jds.2021-21032

Transferring a large data library of fresh total mixed rations from a benchtop to 2 portable near-infrared spectrometers for on-farm real-time decisions

Dolores Pérez-Marín,[1] Begoña de la Roza-Delgado,[2] J. Antonio Entrenas,[1]* Mar Garrido-Cuevas,[1] and Ana Garrido-Varo[1]

- Data library transfer (NIR benchtop to portable devices) for fiber prediction.
- Instrumental standardization Shenk and Westerhaus (1989) algorithm.



CONCLUSIONS

1. The application of **NIR spectroscopy** in plant-based matrices like asparagus demonstrated that this technology enables a**ccurate quantification of fiber fractions (NDF and ADF)** achieving strong predictive performance.

2. **NIR spectroscopy** has also proven effective in **discriminating samples according to fiber content**, due to spectral variations, particularly those associated with O–H functional groups.

3. Within forage matrices, the adoption of **non-linear modelling approaches enhances predictive performance** relative to conventional linear methods for the determination of **NDF** and **ADF**. This finding highlight the importance of selecting appropriate chemometric strategies when analysing complex matrices.

4. The successful **transfer of data libraries** from benchtop instruments to portable devices demonstrates that predictive accuracy for **fiber** can be preserved following instrumental standardisation. This constitutes a significant step towards practical, **real-world implementation, enabling real-time analysis** at the point of waste generation or processing, and thereby facilitating efficient fiber valorisation.

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Vega-Castellote, M. & Pérez-Marín, D.

Thank You
For Your Attention

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