Saffron valorization by NIR spectroscopy

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INTRODUCTION

- Saffron commercial quality is defined by the ISO 3632 [1] establishing the specifications for dried saffron obtained from the pistils of *Crocus sativus* L. flowers. The ISO testing methods [2] are not particularly complex but require the use of precious grams of products.
- Beyond trading standards, professionals in saffron value chain, especially top quality and PDO producers, are looking for a non-destructive method to assess the quality characteristics saving the precious product, together with the definition of stricter and rewarding quality assignment.

AIM

The main objective of the project is to identify a nondestructive method for saffron analysis and to develop a model to predict sample quality.

MATERIALS & METHODS

Saffron collection:

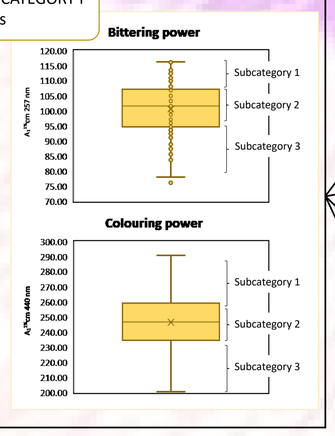
118 Italilan samples (1.5 - 2 g of dry stigmas);

from two harvesting years (2021 and 2022).

Analyses for content of water and the three main metabolites responsible for saffron colour, flavour, and aroma according to ISO 3632 [1,2].

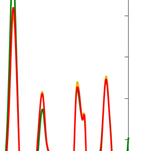
CATEGORY	COLOURING POWER Min value ($A_1^{1\%}$ cm 440nm)	BITTERING POWER Minimum value	AROMATIC POWER Acceptability range	HUMIDITY Max value (%)
I	200	70	20 – 50	Filaments: 12 Powder: 10
II	170	55	20 – 50	Filaments: 12 Powder: 10
III	120	40	20 – 50	Filaments: 12 Powder: 10

Definition of CATEGORY I subcategories



Analyses by FT-NIR spectrometer by mean of an integration sphere

Spectra (9000 - 3800 cm^{-1}) acquired in duplicate by an integration sphere connected to a FT-NIR spectrometer (MPA, Bruker Optics, Milan, Italy).



Spectra pre-treatment
- Smoothing
- Smoothing + SNV
- Smoothing + first derivative
- Smoothing + SNV + first derivate

Exploration of data by PCA
- Geographical area
- Altitude
- Year of harvest distribution
- Quality/subquality categories distribution

Development of regression models by PLS
- Regression for colouring and bittering strength prediction:
  - Calibration
  - Cross-Validation (Venetian blinds, 10 cancellation group)

Development of classification models using PLS-DA
- Classification according to 3 quality subcategories:
  - Calibration
  - Cross-Validation (Venetian blinds, 10 cancellation group)

RESULTS & DISCUSSION

SPECTRA

Figure 1. *NIR spectra of three saffron samples, representative of the three defined subcategories: raw (A) and pre-treated with SNV (B), d1 (C) and SNV + d1 (D)*

REGRESSION & CLASSIFICATION

Table 1. *PLS – Regression statistics*

Property	N_{tot}	Range	Treatment	LVs	R^2_{cal}	RMSEC	R^2_{CV}	RMSECV
Colouring strength ($A_1^{1\%}$ cm 440 nm)	118	187.29-290.97	SNV - 1der	5	0.80	10.0	0.75	11.1
Bittering strength ($A_1^{1\%}$ cm 257 nm)	118	76.26-116.39	SNV - 1der	5	0.80	3.67	0.76	4.06

The most promising PLS models for colouring and bittering strength were obtained with spectra pretreated with SNV and 1st derivative.
The RMSECV were of about 10% of the variation range of each considered variables.

Table 2. *PLS-DA – Classification statistics*

			Calibration			Cross-validation		
Treatment	**LVs**	**Subcategory**	**1**	**2**	**3**	**1**	**2**	**3**
SNV + d1	5	**N**	21	69	28	21	69	28
		Sensitivity	0.857	0.710	0.821	0.810	0.623	0.750
		Specificity	0.907	0.837	0.878	0.845	0.776	0.878

The PLS-DA model well discriminated class one and three, whereas class two was often confused.
Sensitivity and Specificity (weighted average 0.686 and 0.812, respectively) in cross-validation resulted poor, mainly because the misclassification of samples belonging to the second subcategory. However, on the positive side, samples of excellence (first subcategory) are never confused with the third subcategory and vice versa.

CONCLUSIONS

FT-NIR spectroscopy seems promising in predicting both **specific qualities**, such as colouring power and bittering power, and **quality** subcategories.
However, the robustness of the prediction capability must be verified. An independent validation phase is programmed with samples of 2023 harvesting season.

BIBLIOGRAPHY

[1] ISO 3632-1:2011. *Spices – Saffron (Crocus sativus L.) – Part 1: Specification*
[2] ISO 3632-2:2010. *Spices – Saffron (Crocus sativus L.) – Part 2: Test methods*

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