CHARACTERIZATION OF SOME OLIVE OIL QUALITY ASPECTS BY NIRS ANALYSIS OF ITS FATTY ACIDS AND TRIGLYCERIDES

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INTRODUCTION

The development of new rapid techniques to characterize aspects of the quality of olive oils is of great interest, specially when they do not depend on the use of solvents and reagents. Two main areas of application of these techniques are 1) determining the varietal origin and 2) olive oil authentication against fraudulent mixtures of plant oils. In this work we have developed predictive models based on spectroscopy Vis/NIR that allow analysis of the composition of fatty acids ($FAME_S$) in olive oil and accurately estimating their triglyceride composition. Strategies for developing fraud detection techniques on olive oils, based on the Vis/NIR analysis of their triglycerides composition and 'Equivalent Number of Carbon' (ECN), are being studied.

MATERIAL AND METHODS

OLIVE OILS

$FAME_S$ N = 233

Triglycerides N = 166

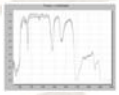
INSTRUMENTATION

Bath – 33 ºC

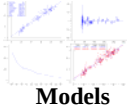
Labspec (ASD, USA)
(Vis/NIR 350-2500 nm)

Transmittance
(Ocean Optics, USA)

SOFTWARE AND MODELING



Acquisition
IndicoPro (ASD, USA)



Models
The Unscrumbler 9.7
(CAMO, Norway)

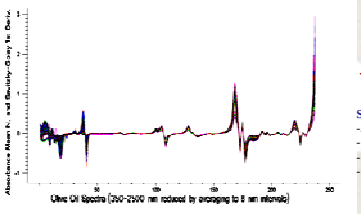
Reference Analysis

$FAME_S$ → 50 mg oil, 2 mL heptane, 300 µL 2N KOH in methanol, supernatant collection, GC, 180 to 220ºC at 3ºC/min, FID detector.

Triglyceride composition→ Purification: silica column, 50 mg oil, Hexane: dietyl-eter (87:13), 10 mL 1 mL propionitrile, RP-18 HPLC, 20ºC, RI detector

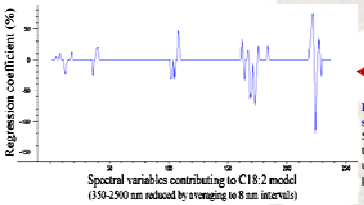
ECN_s → Aritmeticallty calculated by integration

RESULTS



Spectral data treatment
-Averaging to 8 nm intervals
- Transmittance to Absorbance
- Mean Normalization
- First Savitzsky-Golay Derivative

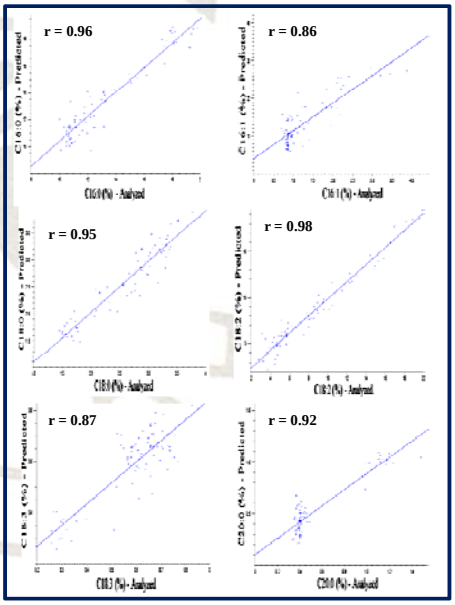
Spectra from the olive oil samples
Absorbance, treated by mean normalization and Savitzsky-Golay 1st Derivative



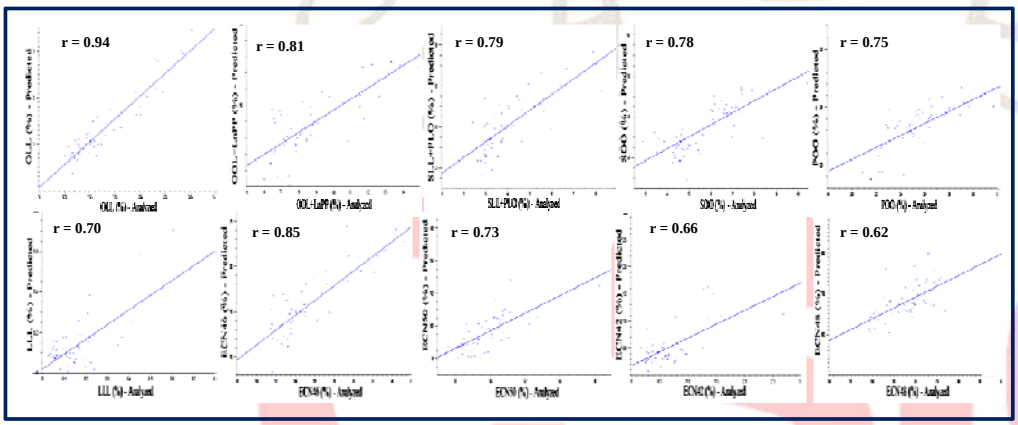
Example of spectral variable selection.
Sucesive cicles were carried out to eliminate wavelengths uncorrelated with the analytical parameter.

	Calibration				Validation			
	N	Range	$\bar{x}$	σ	N	Range	$\bar{x}$	σ
C16:0	147	9.40-19.42	13.14	2.94	76	10.05-19.72	13.27	3.02
C16:1	142	0.51-2.95	1.26	0.59	77	0.7-3.89	1.3	0.72
C17:0	100	0.04-0.17	0.07	0.02	60	0.03-0.15	0.08	0.03
C17:1	104	0.05-0.26	0.12	0.04	56	0.07-0.29	0.14	0.06
C18:0	135	1.49-3.86	2.64	0.66	72	1.44-3.76	2.59	0.67
C18:1	146	53.86-81.25	73.05	7.54	74	42.84-80.16	71.52	8.63
C18:2	144	2.26-19.82	8.29	4.53	75	2.84-19.87	8.75	4.72
C18:3	146	0.15-0.82	0.57	0.15	75	0.19-1.02	0.58	0.16
C20:0	149	0.14-1.16	0.53	0.27	73	0.22-1.48	0.55	0.3
C20:1	141	0.11-0.92	0.38	0.23	75	0.21-1.19	0.38	0.25
C22:0	132	0.08-0.19	0.11	0.02	76	0.06-0.19	0.11	0.02
C24:0	107	0.03-0.08	0.05	0.01	58	0.03-0.08	0.05	0.01

Table 1. Statistical data from the calibration and validation sets



External validations using the predictive models for olive oil fatty acids
C16:0, Palmitic acid; C16:1 Palmitoleic acid; C18:0 Estearic acid; C18:2 Linoleic acid; C18:3 Linolenic acid; C20:0 Araquidic acid. r, correlation coefficient between the predicted and analyzed values



External validations using the predictive models for olive oil triglycerides and ECN
OOL, Dioleolinolein; OOL+L_2PP, Dioleolinolein + Dipalmitolinolenin; SLL+PLO, Stearodilinolein+ Palmitooleolinolein; SOO, Stearodiolein; POO, Palmitodiolein; LLL, Trilinolein; ECN46, Equivalent Carbon Number 46; ECN50, Equivalent Carbon Number 50; ECN42, Equivalent Carbon Number 42; ECN48, Equivalent Carbon Number 48. r, correlation coefficient between the predicted and analyzed values.

CONCLUSIONS

This work demonstrates the feasibility of determining $FAME_S$, and estimating the olive oil triglyceride composition by Vis/NIRS, using multivariate models. The predictive exercises for estimating dioleolinolein and dioleolinolein+dipalmitolinolenin provided r 0.94 and 0.81, and for ECN46 and ECN50 provided r 0.85 and 0.73. The proposed techniques are fast, non-destructive and potentially multi-parametric. The goodness of statistical models and the evaluation tests shows that these techniques can be useful together other methods for analyzing these quality parameters of olive oil.

7th International Symposium on Recent Advances in Food Analysis