Facile Verification of Edible Oils with Raman Spectroscopy

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Edible oils comprise a significant portion of any diet, and they also have important roles in the production of foods, cosmetics, and skincare products. For these reasons, a convenient and accurate method for materials identification of a variety of fats and oils is highly desirable.

Historically, authentication of fats and oils was performed through intensive laboratory techniques involving chromatographic methods. Here, Raman spectroscopy combined with Principle Component Analysis (PCA) has been used for materials identification with 16 different edible oils, with excellent results. Raman is an ideal technique for evaluation of fats, as carbon-carbon double- and single-bonds give strong Raman signals. PCA analysis in combination with Raman spectroscopy is a powerful tool for qualification and verification of different fats and oils, as there are few visual differences between spectra of edible oils.

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Methods Description

Introduction

The handheld Metrohm Instant Raman Analyzer (Mira) equipped with ORS™ technology is demonstrated to accurately verify the identities of 16 different edible oils. Raman has been used to study saturation, fatty acid composition, decomposition and adulteration of edible oils, and has an important place in industrial process control. The Mira P is uniquely capable of materials identification AND verification within the Metrohm Mira product line. For verification of a known substance, the Mira P is equipped with PCA analysis, which provides a PASS/FAIL result with the associated p-value. This is demonstrated to be an excellent method for discrimination in a study of different fats and oils with very small spectral differences. Analysis of samples with the Mira P is fast and easy, nondestructive, and requires no sample pretreatment. Here, the Mira P is shown to accurately verify the identities of 16 fats and oils 100% of the time using a confidence level of 95%. Included in this report are suggestions for creating and editing the training sets used in the verification method.

Samples

16 edible oils were purchased from local grocery markets. A variety of oils was tested, including 4 distinct types of olive oil, to determine whether the Mira P could discriminate between them.

Sample preparation

The oils were transferred directly from the sales packaging into small glass vials for analysis.

Instrument

The Mira P handheld Raman spectrometer equipped with ORS™ was used to establish training sets and to validate the identity of 16 oils and fats.

Training Sets

The training set for each oil was built upon 60 samples and intentionally included all reasonable variation in testing conditions: laser power, integration times, sample attachment, lighting conditions, and temperature. For 10 of the oils in this study, the training sets were used without editing. For 6 of the oils – African Palm, Avocado, Coconut, Extra Virgin Olive Oil, Pure Olive Oil – visual examination of the training set resulted in the elimination of spectra with unacceptable levels of variation.

Method

After a training set was established for each of the 16 oils, each oil was tested within each training set. For example, Pure Extra Virgin Olive Oil was submitted for verification within the training sets for Corn Oil, Canola Oil, Grapeseed Oil, Pure Olive Oil, etc.

Analysis Parameters	
Mode	Verification
Confidence Level	0.95
Laser Power	3
Integration Time	Autointegration
Average	5
Attachment	Vial Holder

Results and Discussion

Each oil was verified accurately within its training set, with a p-value of 0.145 or greater. A quick look through available literature on analysis of Edible Oils with Raman analysis indicates that comparative studies have found 85.6–93.1% accurate verification of oils with Raman. [1,2,3,] Mira instruments are capable of *100% accurate identification* with smartly-designed training sets and PCA analysis. The data on the following page illustrates the power of this analysis: the complex, fluorescent, and very similar spectra of edible oils do lead to some agreement between samples, resulting in very low p-values (<0.100, indicated with **#**) but stopping short of false positives.

With Mira solutions from Metrohm Raman, real-time qualitative analysis of edible oils can be carried out accurately and efficiently, giving customers and producers confidence in product quality.

References

1) Korifi (2011) J. Raman Spec. 42: 1540
2) Yang et al. (2001) J. of the American Oil Chem. Soc. 78: 889
3) Yang (2005) Food Chem. 93: 25

Table of Results for Verification of Edible Oils

↓Sample / Trainning Set → (# Samples)	African Palm (43)	Avocado (50)	Canola (60)	Coconut (42)	Corn (60)	EL Olive (60)	EV Olive (52)	Grapeseed (60)	OV Coconut (31)	Peanut (60)	Prem. EV Olive (60)	Pure Olive (52)	Pure Palm (30)	Refined Sesame (60)	Sunflower (60)	Walnut (60)
African Palm	P	#		#								#	#			
Avocado		P						#			#	#				
Canola			P												#	
Coconut				P					#							
Corn				#	P	#								#		#
Extra Light Olive				#	#	P				#				#		
Extra Virgin Olive		#					P	#			#					
Grapeseed		#						P				#				
Org Virgin Coconut				#					P							
Peanut				#	#	#				P				#		
P. Extra Virgin Olive		#						#			P					
Pure Olive		#		#				#				P				
Pure Palm	#			#								#	P			
Refined Sesame				#	#	#				#				P		
Sunflower			#	#		#									P	
Walnut			#	#	#											P

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