Rapid determination of phenol content in extra virgin olive oil

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SUMMARY

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A quick extraction methodology was developed to reduce the time usually required to determine the phenol content in olive oil. The validity of this method, based on SPE technique, was tested against two other phenol extraction techniques.

The statistical analysis of the analytical data showed that over a phenol content range of 110-550 µg/g oil, the proposed method can be a reliable alternative for a rapid extraction of the phenols from olive oil.

KEY-WORDS: Phenol content - Solid phase extraction - Virgin olive oil.

1. INTRODUCTION

Among the various component of the insaponificable fraction of the olive oil, phenol compounds are of major importance for their contribution to flavour, stability and nutritional value of the oil (Cortesi and Fedeli, 1983; Perrin, 1992). The determination of the total phenol content in olive oils is actually carried out using long procedures, mainly based upon liquid-liquid partitioning.

Nowadays, solid phase extraction (SPE) methodology is more and more applied for the isolation or concentration of analytes from a wide variety of substrates. SPE has already been utilized in olive oil analysis for the extraction of pigments (Minguez-Mosquera et al., 1992) and for the extraction of nonvolatile bitter constituents (Gutiérrez et al. 1989; GutiérrezRosales et al. 1992).

The aim of this work was to test the validity of a quick phenol extraction method based on SPE, in comparison with two other currently utilized methods based on liquid-liquid partitioning.

2. EXPERIMENTAL

The proposed method of phenol extraction is reported in Figure 1 (Method 1). The SPE columns contained about 1 g of octadecyl (C_{18}) material and were purchased from J.T. Baker (Phillisburg, NJ, USA). In Figure 2 and Figure 3 are reported the extraction methodologies utilized to compare the extraction efficiency of the proposed method. Method 2 was utilised by Tsimidou et al. (1992) and Method 3 by Montedoro et al. (1992).

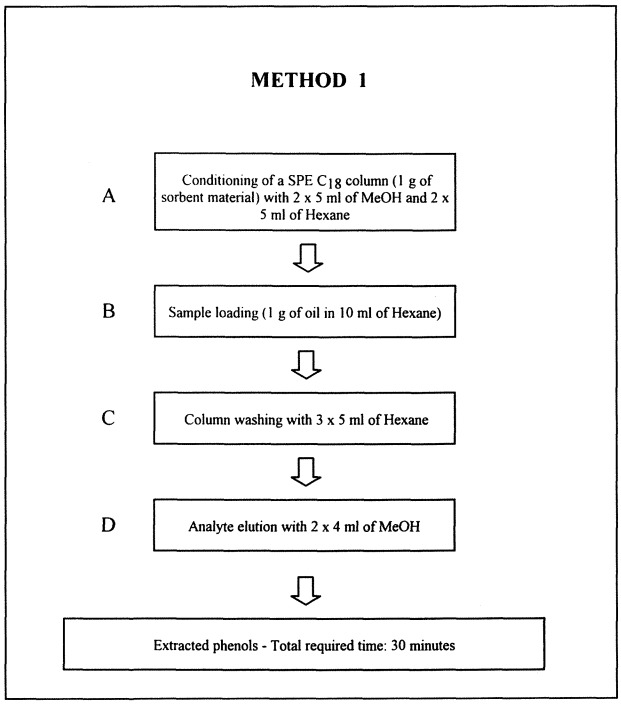

Figure 1
Description of the main steps of the proposed phenol extraction procedure.

A commercial mixture of refined seed oils (Friol-OIO, Unil. It. S.p.A., Milano, ITALY) not containing any detectable amount of phenols, was utilised to test Method 1 *vs.* Methods 2 and 3. The oil was spiked with 3 known amount of tyrosol [2-(p-Hydroxy phenil)-3 ethanol] (Aldrich Chem. Co., Milwaukee, WI, USA), namely 110, 330 and 550 µg tyrosol/g oil. After extraction, the phenol content was determined colorimetrically as follows: 2 ml of the phenol containg solution were transferred into a 20 ml volumetric flask and 5 ml of distilled water were added, followed by 0.5 ml of Folin-Ciocolteau reagent (Carlo Erba Reagents, Milano, ITALY). After 3 min, 4 ml of a 10% solution of sodium carbonate were added, the volume was brought up to 20 ml with distilled water and the flask was stored in the dark. After 90 min the solution was filtered through a 0.2 µm filter (Gelman Sc., Ann Arbor, MI,

USA) and the absorbance read at 765 nm. All reagents were analytical grade and were obtained from Carlo Erba Reagenti (Milano ITALY).

The extraction efficiencies of Method 1, 2 and 3 were assessed and compared applying the following procedure: each method was utilised to extract the phenols from the oil samples spiked with tyrosol at the set concentration; for each sample, five analysis were performed independently by two operators, for a total of ten replicates for each concentration and for each extraction methodology. The acquired data were statistically analysed by ANOVA as reported in Table I.

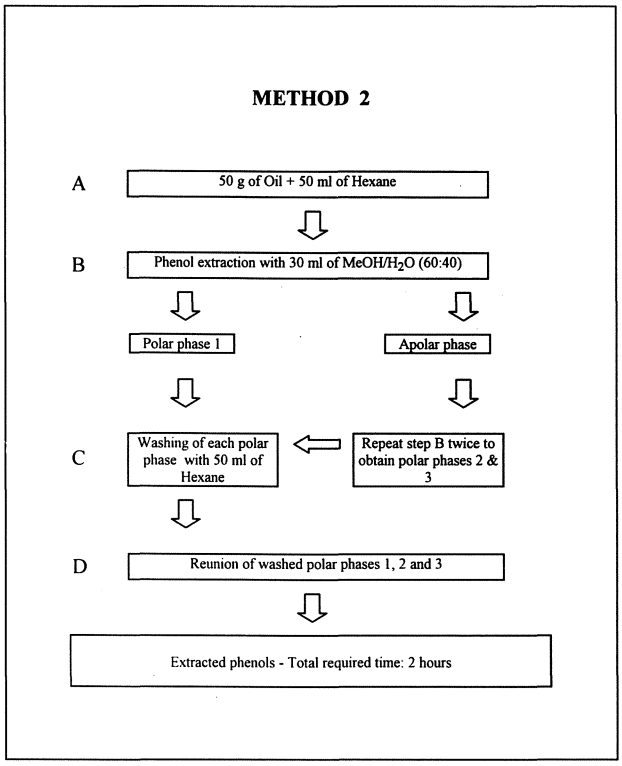

Figure 2
Description of the main steps of the phenol extraction procedure adopted by Tsimidou et al. (1992)

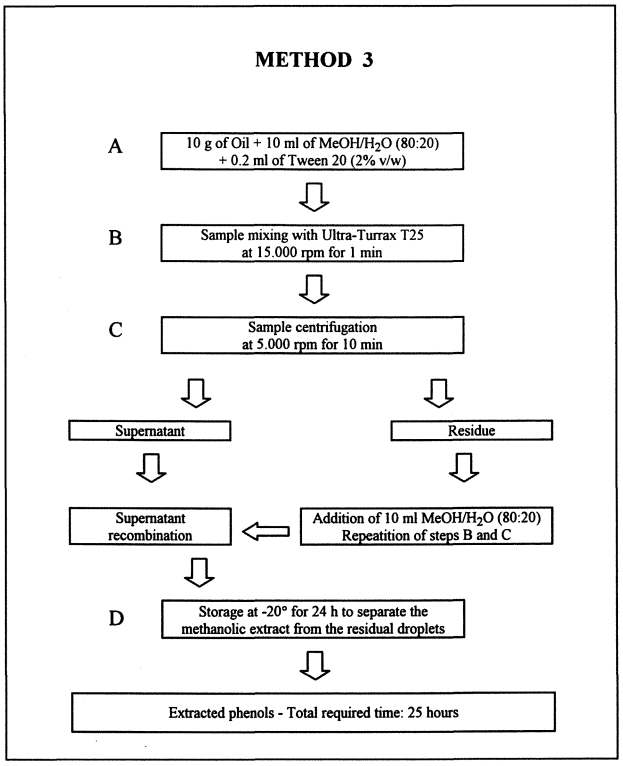

Figure 3
Description of the main steps of the phenol extraction procedure adopted by Montedoro et al. (1992)

Table I
Comparison of three extraction methods: statistical evaluation of the analytical results.

SAMPLES	METHOD 1	METHOD 2	METHOD 3	df	F samples	F replicates	LSD
Spiked oil (110 g tyrosol /g oil)	114	108	109	18, 2	2.89 NS	1.09 NS	-
Spiked oil (330 g tyrosol /g oil)	332^a	310^b	300^b	18, 2	23.75***	1.01 NS	18.7
Spiked oil (550 g tyrosol /g oil)	530^a	501^b	496^b	18, 2	13.42**	2.62 NS	20.7
Oil sample 1	98^a	67^b	81^c	6, 2	14.66**	0.17 NS	14.1
Oil sample 2	323^a	289^b	322^c	6, 2	8.51*	0.33 NS	34.2
Oil sample 3	458^a	528^b	467^a	6, 2	15.31**	3.85 NS	50.5

Method 1 = proposed method, Method 2 = Tsimidou et al., 1992., Method 3 = Montedoro et al., 1992.
a, b, c = values with different superscript are significantly different
NS = not significative
LSD = Least Significant Difference between mean values at the level indicated.
*, **, *** = significant at 5, 1 and 0.1 % level.

The results showed that for a tyrosol content ranging from 110 to 550 µg /g oil, the three methods gave comparable results. Therefore the SPE method was tested also on samples of extra virgin olive oil coming from different countries. Also in this case the SPE method was evaluated in comparison with those utilised by Montedoro et al. (1992) and Tsimidou et al. (1992) (Table I). The analytical results indicate that utilising the proposed methodology, the total phenol content in olive oil can be reliably assessed with a significant reduction of the time required for the extraction of the phenols from the lipid matrix.

REFERENCES

Cortesi, N. and Fedeli, E. (1983). –"I composti polari di oli di oliva vergini. Nota 1".– Riv. Ital. Sost. Grasse **60,** 341-351.

Gutiérrez, F., Albi, M.A., Palma, R., Rios, J.J. and Olías, J.M. (1989). –"Bitter Taste of Virgin Olive Oil: Correlation of Sensory Evaluation and Instrumental HLPC Analysis".– J. Food Sci. **54,** 68-70.

Gutiérrez, F.R., Perdiguero, S., Gutiérrez, R. and Olías, J.M. (1992). –"Evaluation of the Bitter Taste in Virgin Olive Oil".– J. Am. Oil. Chem. Soc. **69,** 394-395.

Minguez-Mosquera, M.I., Gandul-Rojas, B. and Gallardo-Guerrero, M.L. (1992). –"Rapid Method of Quantification of Chlorophylls and Carotenoids in Virgin Olive Oil by HighPerformance Liquid Chromatography".– J. Agric. Food Chem. **40,** 60-63.

Montedoro, G., Servili, M., Baldioli, M. and Miniati, E. (1992). –"Simple and Hydrolizable Phenolic Compounds in Virgin Olive Oil. 1. Their Extraction, Separation and Quantitative and Semiquantitative Evaluation by HPLC".– J. Agric. Food Chem. **40,** 1571-1576.

Perrin, J.L. (1992) –"Les composés mineurs et les antioxigènes naturel de l'olive et de son huile".– Rev. Franç . Corps Gras **39,** 25-32.

Tsimidou, M., Papadopoulos, G. and Boskou, D. (1992) –"Phenolic compounds and stability of virgin olive oil Part I".– Food Chem. **45,** 141-144.