

Survey on pesticide residues in virgin olive oils consumed in France

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Introduction

As virgin olive oil (VOO) is not submitted to refining process, there is more risk that traces of contaminants still remain in the oil compared to refined oils. At the present time, the most critical area is the possible presence of residues of insecticides used against "olive fly" especially organophosphorus (OP) and pyrethroids (PYR) compounds. Due to their toxicity and the increase of olive oil consumption (2,7 millions tons), special attention has to be paid to the monitoring of pesticide residues in VOO. The method developed by ITERG allows us to measure 15 OP and 5 PYR at the µg/kg level. 142 samples were analyzed in a two year survey (2001 and 2002) in order to assess the average contamination of VOO produced, imported and sold in France.

Compound	LOQ	MRL Codex in OO	Compound	LOQ	MRL Codex in OO
Azinphos-ethyl	0,060	-	Cyfluthrin	0,055	-
Azinphos methyl	0,040	-	Cypermethrin	0,060	-
Carbophenothion	0,020	-	Deltamethrin	0,025	-
Chlorpyrifos	0,015	-	Lambda-cyhalothrin	0,015	-
Diazinon	0,010	-	Permethrin	0,220	-
Dichlorvos	0,025	-			
Dimethoate	0,010	0,050			
Etrimfos	0,010	-			
Fenitrothion	0,010	-			
Fenthion	0,010	1			
Heptenophos	0,025	-			
Malathion	0,010	-			
Methidathion	0,010	2			
Parathion (ethyl)	0,015	2			
Pirimiphos-methyl	0,010	-			

LOQ : Limit of quantification (mg/kg) MRL : Maximum Residue Limit (mg/kg)
OO : Olive Oil

Materials and Methods

Samples

VOO samples came from several geographic origins : Spain (61), France (44), Italy (10), Greece (6), Portugal (1) and Crete (1). Samples were collected from different suppliers: 3 major French companies provided samples directly from the containers imported before mixing, French mills gave samples from two harvest seasons 2000/2001 and 2001/2002, some samples were bought in French supermarkets.

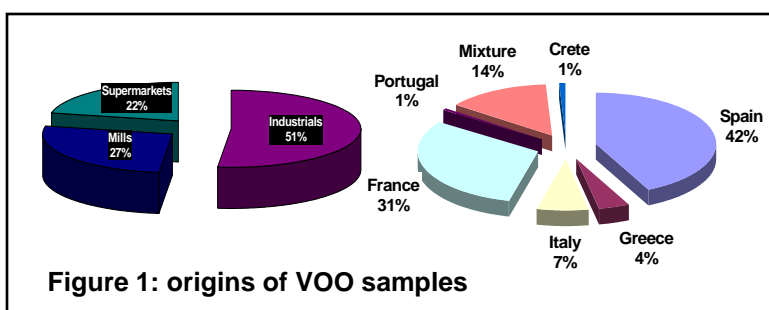


Figure 1: origins of VOO samples

Extraction and clean-up procedure

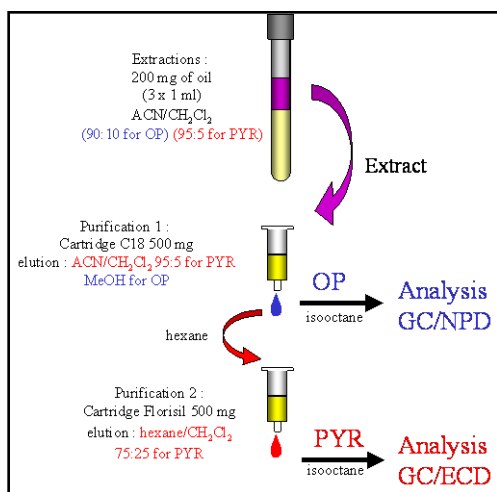


Figure 2: analytical diagram

Apparatus

GC/NPD : Varian Star 3400 CX.

Column : DB 1701, 14% cyanopropyl-phenyl, 86% méthylpolysiloxane, 30 m, Ø 0,32 mm, 0,25 µm. Progr. T° : 80°C (5 min.), 6°C/min. to 270°C, 270°C (20 min). Inj. : 80°C (0,5 min.), 250°C/min. to 250°C, 250°C (55 min.) T° detector : NPD, 300°C

GC/ECD : Varian Star 3400

Column : DB 1, 100% diméthylpolysiloxane, 15 m, Ø 0,25 mm, 0,1 µm. Progr. T° : 90°C (0 min.), 10°C/min. to 280°C, 280°C (11 min.). Inj. : 90°C (0,3 min.), 180°C/min. to 300°C, 300°C (30 min.). T° detector : ECD, 300°C.

Table 1: List of pesticides determined, Limits of quantification and Codex MRLs

Results and discussion

This survey shows that *fenthion* is the major pesticide residue present in virgin olive oil (Figure 3), 31 % of the samples analysed contain traces of this insecticide (Limit of quantification = 0,010 mg/kg) with a maximal value of 0,839 mg/kg (fenthion + fenthion sulfoxide) and an average value of 0,087 mg/kg. However, this is very far from the maximum residue limit (MRL) 1 mg/kg. *Chlorpyrifos*, *methidathion*, *dimethoate*, *fenitrothion* and *diazinon* are sometimes found in virgin olive oil (5 to 15 % of the samples depending on the molecule) with a maximum value of 0,065 mg/kg for *methidathion*. Traces of *pirimiphos-methyl*, *parathion-ethyl*, *malathion* and *etrimfos* were found in less than 5% of samples.

Overall, 95% of the analysed samples contain less than 0,050 mg/kg of total organophosphorous pesticides (Figure 4). In all cases, no excess of residue was found.

Concerning pyrethroids, 34 samples were analysed : no contamination was observed.

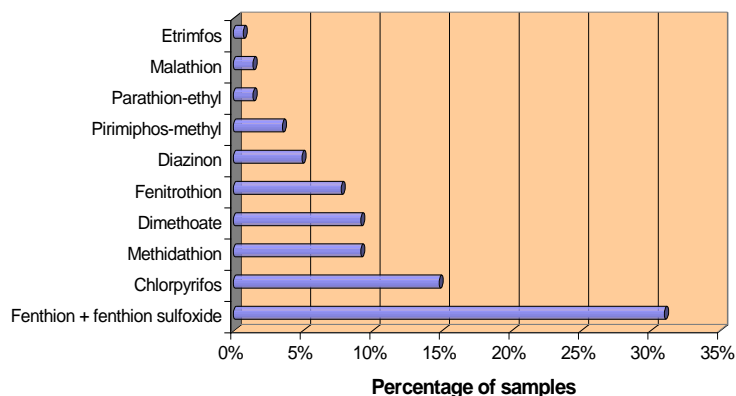


Figure 3: Major pesticides found in VOO

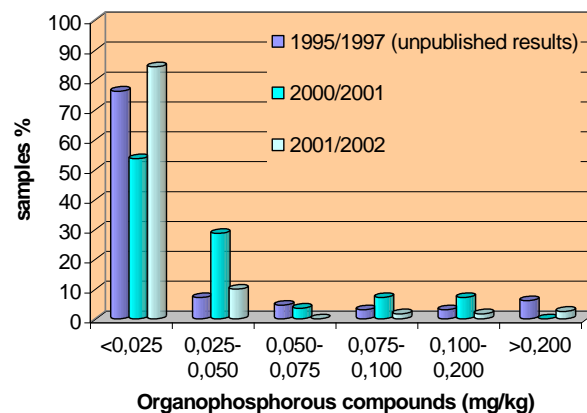


Figure 4: Total concentration of OP pesticides found in VOO

Conclusion

This survey shows that virgin olive oils consumed in France are weakly contaminated by organophosphorous and pyrethroids pesticides and thus present no risk for human health. All the concentrations determined are under the few existing MRLs of the Codex Alimentarius. However, special attention has to be paid to *fenthion* and its metabolite *fenthion sulfoxide* which are the major compounds found in samples.