Analysis of multiple pesticide residues in olives and olive oil using QuEChERS and LC-MS/MS

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Olive Oil

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Olive oil is a vegetable oil obtained from the fruit of the olive tree (*Olea europaea* L.)

IMPORTANCE
- **Economic**
- **Cultural**
- **Nutritional**

Aim of the Work

- **Apply the QuEChERS method to olives and olive oil for multiple pesticide residues analysis**
- **Optimize and evaluate this method to achieve high pesticide recoveries and adequate detection limits**

Pesticides Used in Olives

Pesticide	MRL (µg/g)						1x Spk to LC-MS/MS (µg/g)
	USA	Codex	EU	Italy	Portugal	Spain	
Omethoate	---	---	---	---	---	---	0.05
Dimethoate	---	0.5	2	---	2	---	0.05
Simazine	0.25	---	---	0.1	0.1	0.1	0.10
Diuron	1	---	---	0.05	0.05	0.2	0.05
Carbaryl	10	30	5	---	1	1	0.20
Malathion	---	---	0.5	---	0.5	---	0.10
Methidathion	0.05	1	1	1	1	1	0.25
Phosmet	---	---	---	---	2	---	0.10

Analysis of Pesticides in Olives

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Sample preparation

QuEChERS method

(10 g sample + 10 ml MeCN + 4 g $MgSO_4$ + 1g NaCl)

Effect of cleanup

Effect of water

Effect of fat

→ Recoveries

Effect of Cleanup

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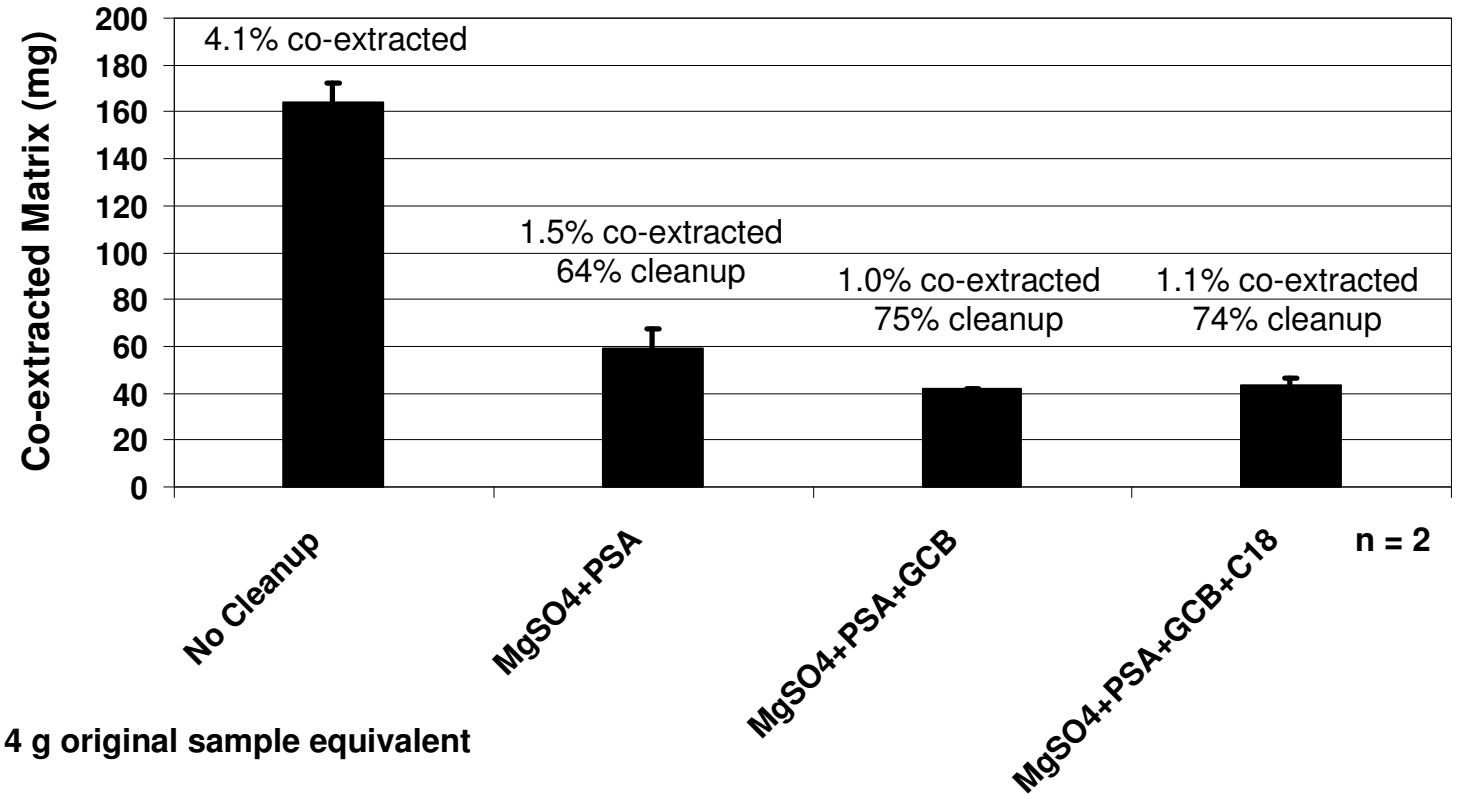

Comparison of different cleanup sorbents for olives extracted with the QuEChERS method

Analysis by LC-MS/MS

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Instrument: Applied Biosystems API 3000 + Agilent 1100 HPLC

Columns:

Analytical - Phenomenex Prodigy, ODS-3 (150 x 3 mm)

Guard - Phenomenex Security guard, ODS–C_{18} (4 x 2 mm)

Mobile phase:

Channel A - water with 0.1% formic acid

Channel B - acetonitrile with 0.1% formic acid

Flow rate: 300 µl/min

Injection volume: 5 µl (MeCN extract)

Detection: Multiple Reaction Monitoring (ESI+)

Effect of Water/Oil

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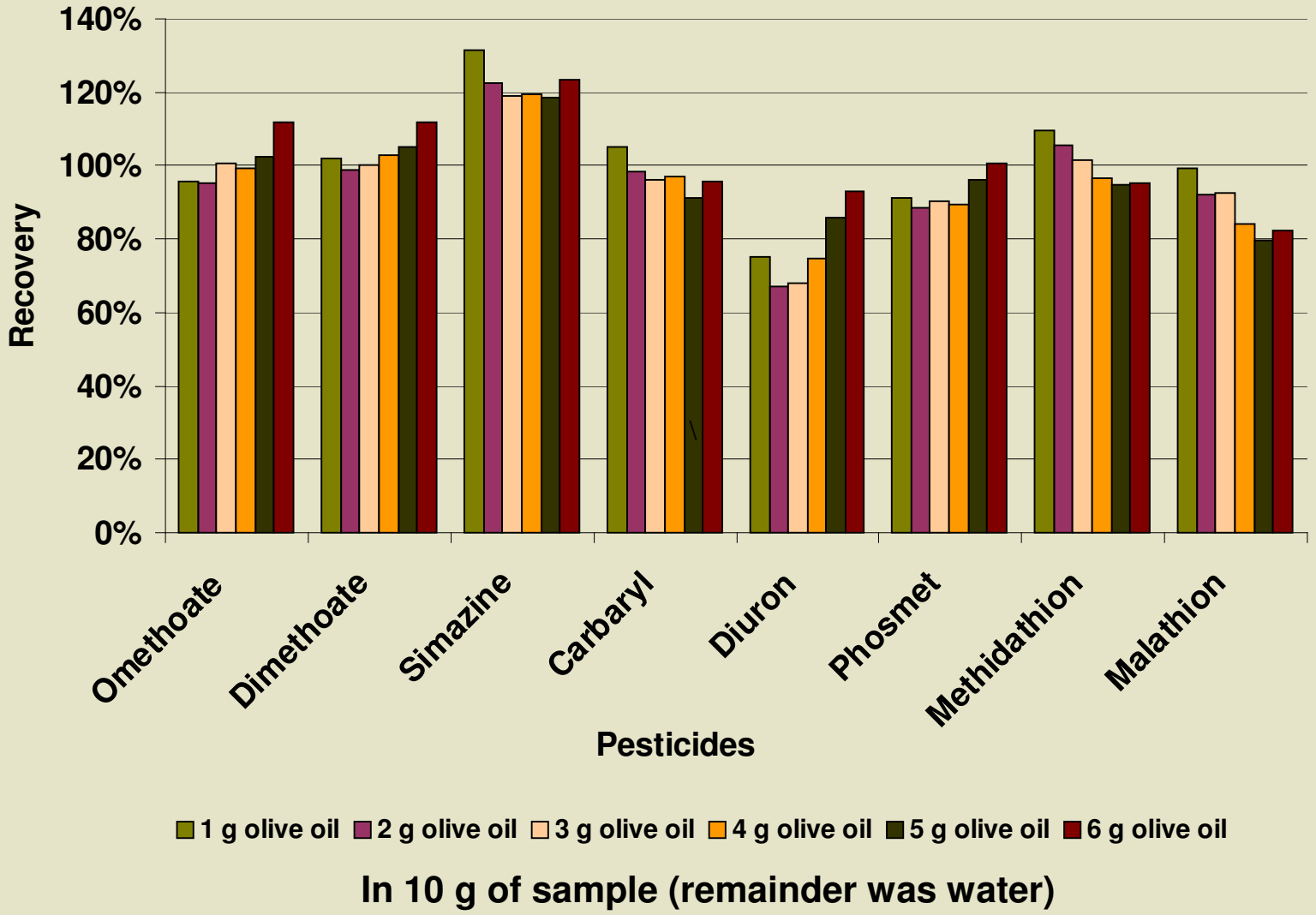

Results of the validation study (mean recoveries and RSDs) obtained with the QueChERS method of olives (*n* = 6)

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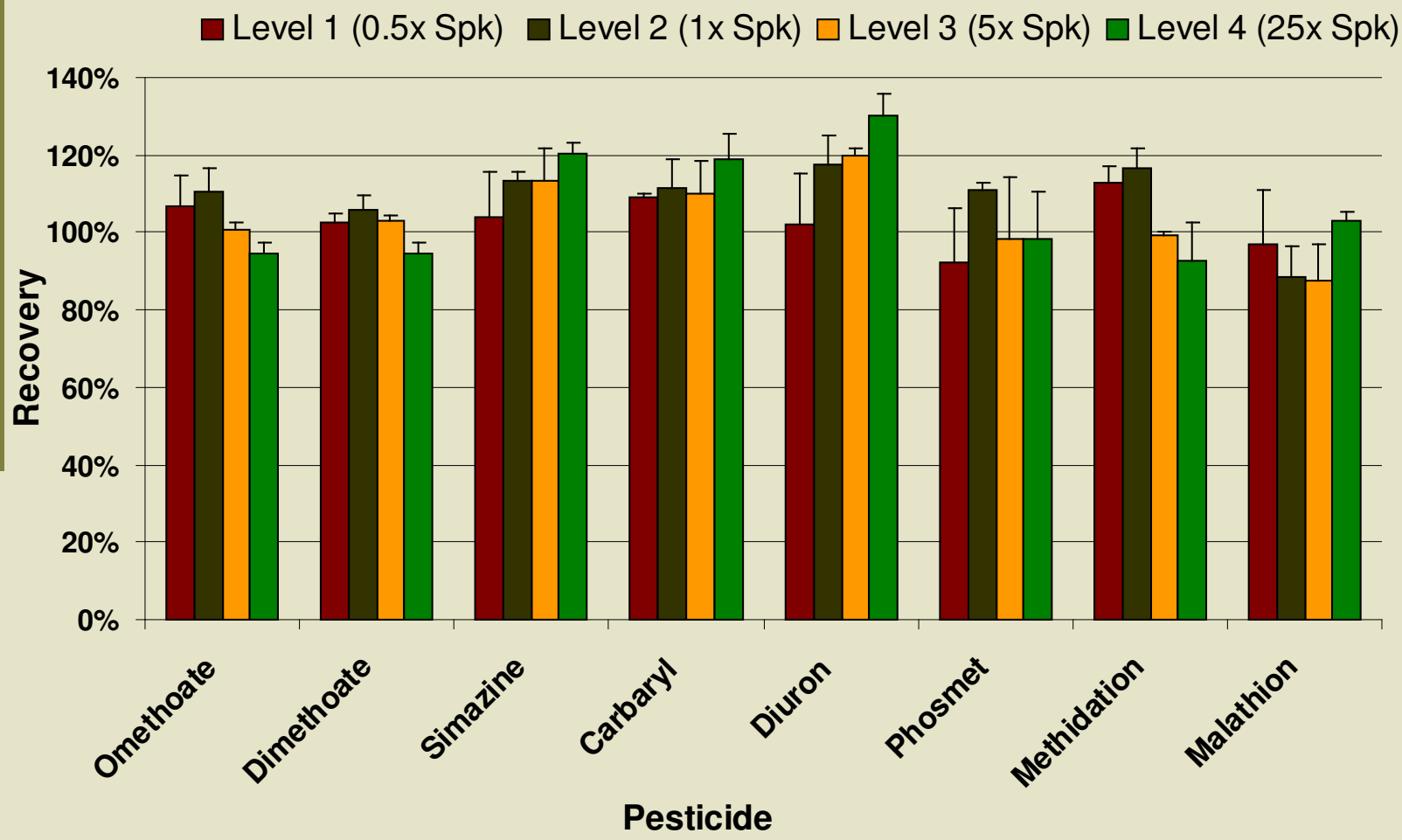

Results of the validation study (mean recoveries and RSDs) obtained with the QueChERS method of olive oil (*n* = 6)

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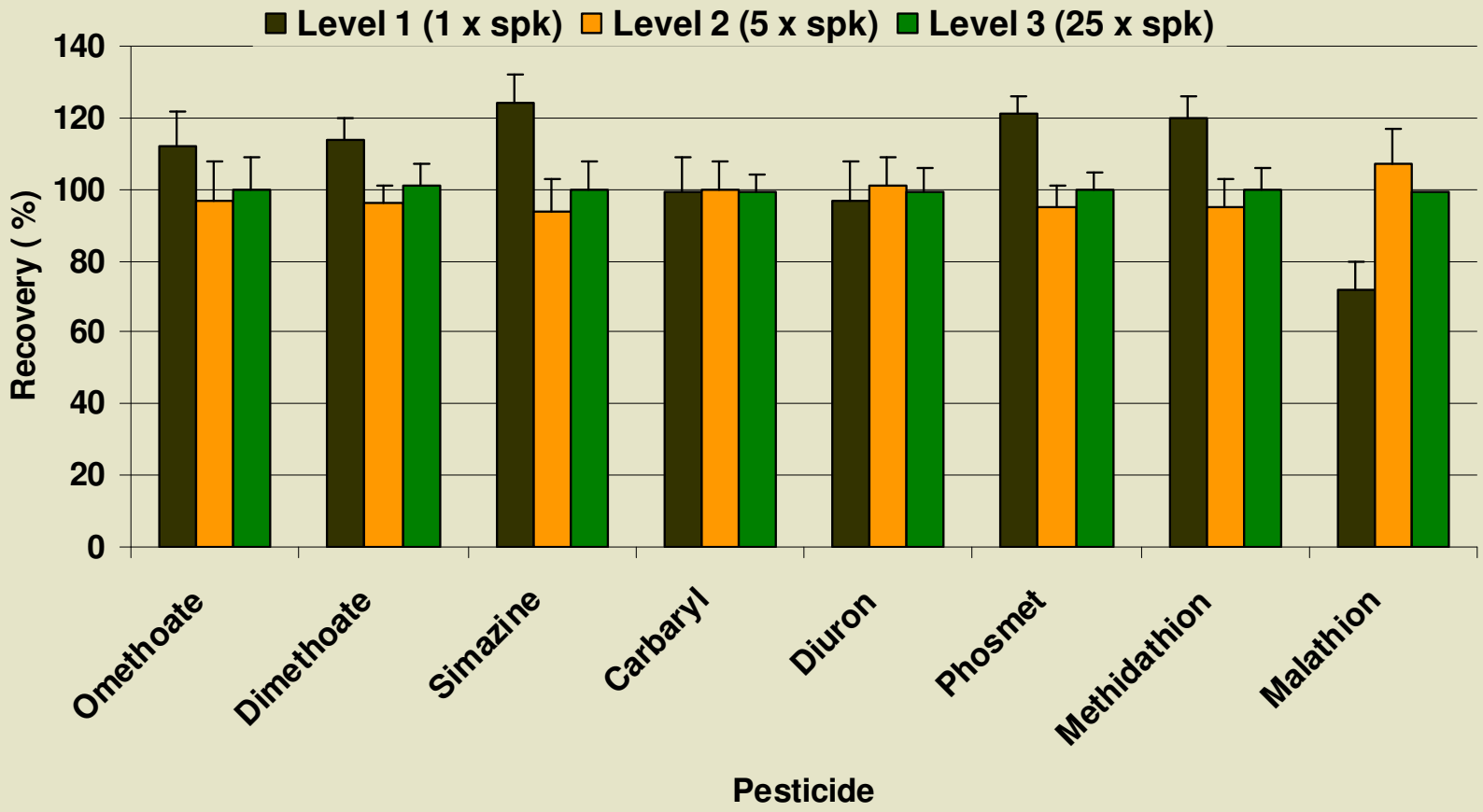

Conclusions

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- ✓ **The best cleanup in dispersive-SPE of olives and its oil used $MgSO_4$+PSA+C_{18}+GCB**
- ✓ **The amount of lipids in the sample did not affect the recoveries of the LC-MS/MS analytes**
- ✓ **The validation obtained in LC-MS/MS for most compounds studied was acceptable with recoveries of 88-130% and RSDs <10%.**

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