Clean-up experiments of oat extracts for pesticide residues analysis by PSA, C18, Z-sep or EMR-lipid, individually and combinations

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Clean-up experiments of oat extracts for pesticide residues analysis by PSA, C18, Z-sep or EMR-lipid, individually and combinations

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Introduction

The level of co-extracted matrix in QuEChERS cereal extracts (EN 15662) is generally high but the highest levels are found in oat extracts due to the high fat content. Especially for oat flour stored at room temperature the matrix can result in poor method performance.
With the present study the EURL-CF wanted to elucidate whether an optimised clean-up procedure, employing more than one sorbent, could be suggested for oat flour stored 4 weeks at room temperature. Besides PSA were Z-sep (zirconium based sorbent), EMR-lipid (enhanced matrix removal-lipid) and C18 found relevant to include in the study.

Study design

Extracts, un-cleaned or cleaned by dSPE with PSA, C18, Z-sep, EMR-lipid or ultracentrifugation, were evaporated to dryness and the amount of matrix determined (mg/ml extract)(Figure 1). GCMS chromatograms of these extract were obtained (Figure 2, ultracentrifugation not shown).
A four factor 2-level factorial experiment (full resolution) was setup to study the effect of the four sorbents on the matrix level and analyte recoveries of 101 pesticides (spike level 0.02 mg/kg). Test sample 1-16 (double determination) were extracted with the QuEChERS method and clean-up in accordance with the design presented in the Table 1. Analyses of the final extracts were performed on GC-MS/MS.

Table 1: Design of 2-level factorial experiment (full resolution).

Sample	PSA (extract)	C18 (extract)	Z-sep (extract)	EMR	Analytes w. recov. 60-130%
1	0	0	0	0	41
2	150	0	150	0	68
3	150	150	150	EMR-lipid	42
4	0	150	0	0	39
5	0	150	0	EMR-lipid	53
6	150	0	0	EMR-lipid	65
7	**0**	**0**	**150**	**EMR-lipid**	**78**
8	0	0	150	0	37
9	0	150	150	0	39
10	150	0	0	0	42
11	0	150	150	EMR-lipid	51
12	150	150	150	0	75
13	**150**	**0**	**150**	**EMR-lipid**	**84**
14	150	150	0	0	71
15	0	0	0	EMR-lipid	48
16	150	150	0	EMR-lipid	42

Conclusion

All four sorbents reduced the amount of co-extracted matrix in the oat extracts, demonstrated by reduction of the matrix residue (mg/ml extract)(Figure 1) and the intensity of the major matrix peaks in the GCMS chromatogram (Figure 2). Though, PSA had the most pronounced reducing effect on both the residual matrix as well as the matrix peaks. Ultracentrifugation had virtually no effect and the amount of co-extract.

From the results of the factorial experiment we found that PSA, Z-sep as well as EMR-lipid increased the number of analytes for which acceptable recoveries (60-130%) were obtained (Table 1 and Figure 3.1, 3.3. and 3.4). Though PSA has the most pronounced effect. C18 on the other hand reduced the number of analytes with acceptable recoveries (Figure 3.2) also if used in combination with other sorbents (Figure 3.5, 3.7 and 3.9).

The study showed that a combination of more than one sorbent provide better analytical results when analysing a problematic matrix as oat flour. A combination of PSA, Z-sep and EMR-lipid or Z-sep and EMR-lipid provided the highest number of analytes with recoveries between 60-130%.

Results

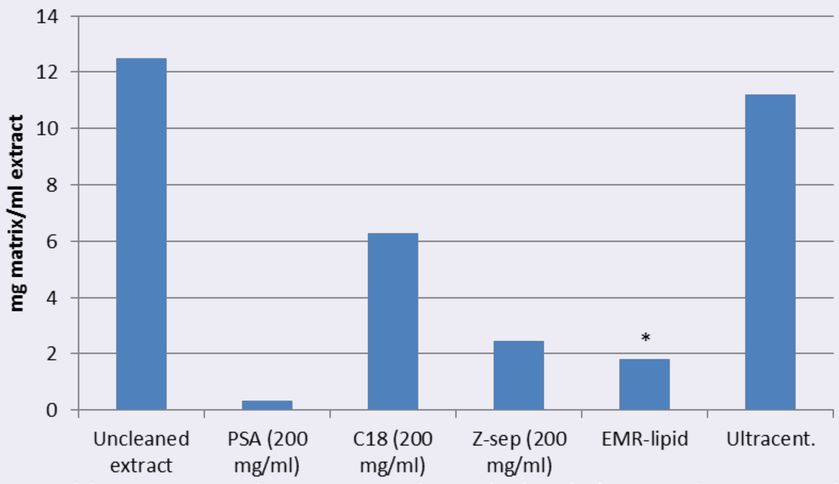

Figure 1: The residue amount of co-extracted matrix (mg/ml extract) measured after evaporation to dryness of oat extracts. Mean of double determinations except for EMR-lipid which was a single determination (market with an asterisk).

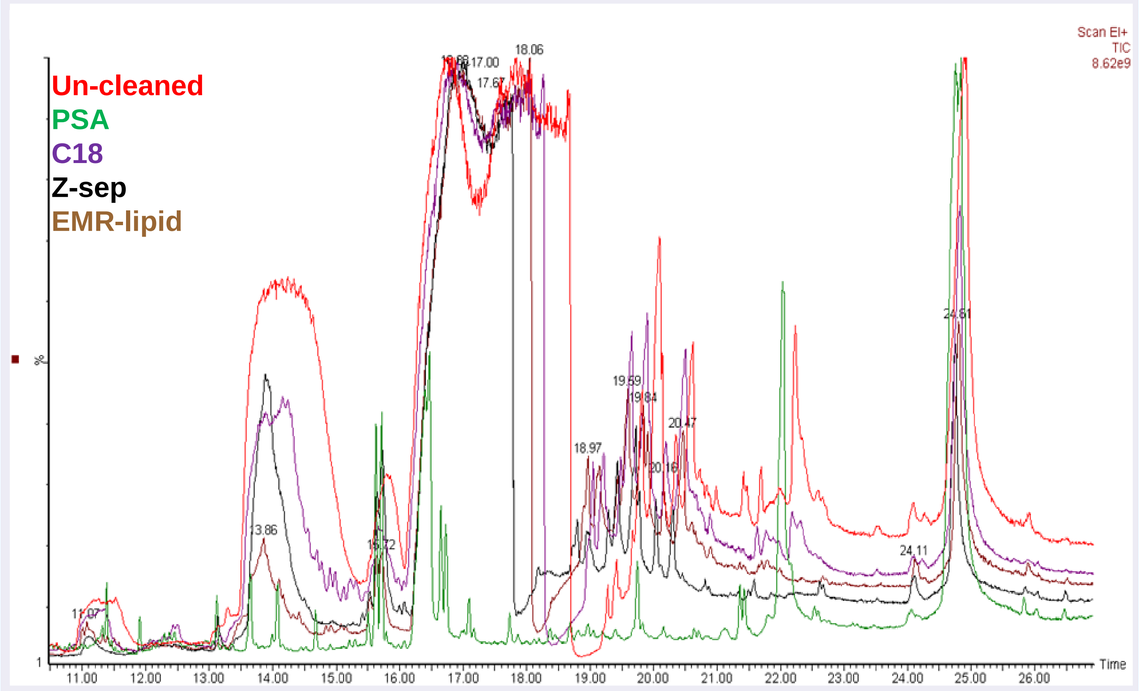

Figure 2: GCMS chromatogram of oat extracts after no cleanup or dSPE cleanup with PSA, C18, Z-sep (200 mg/ml) or EMR-lipid.

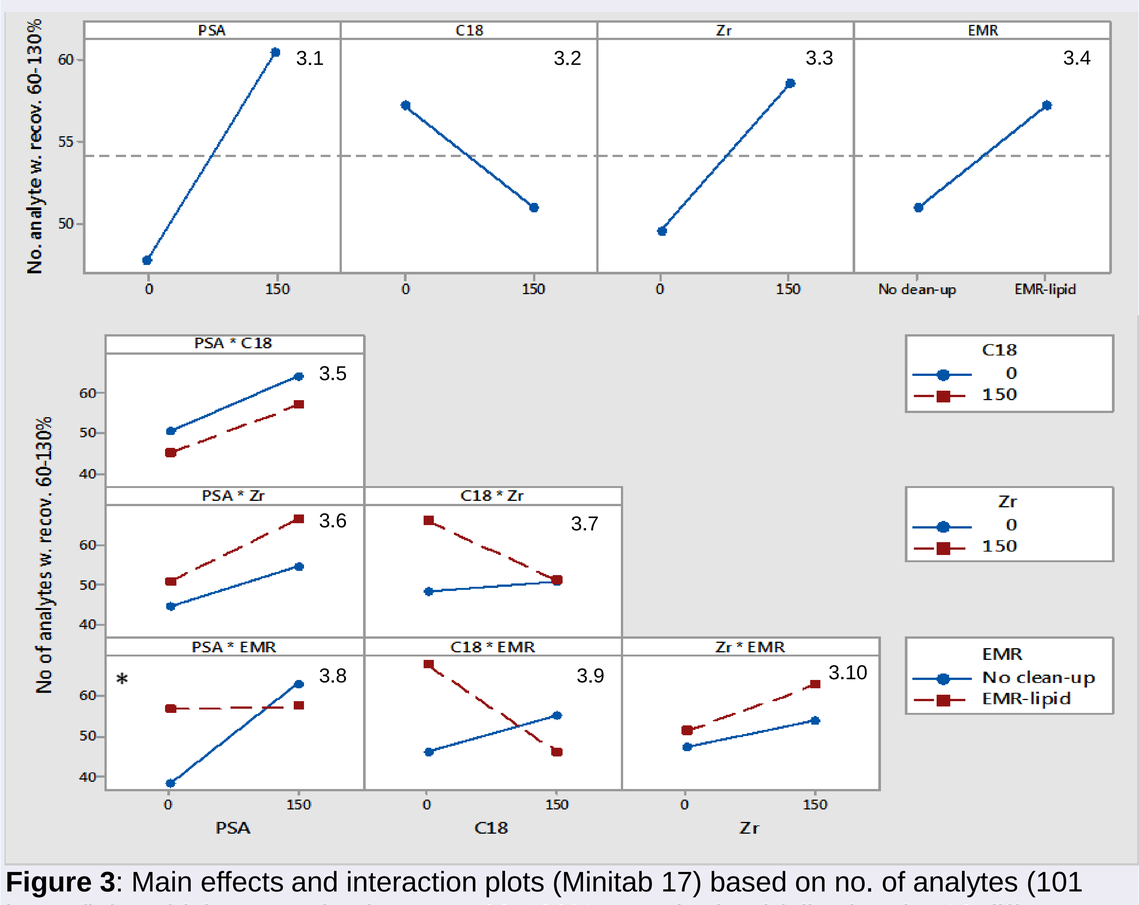

Figure 3: Main effects and interaction plots (Minitab 17) based on no. of analytes (101 in total) for which recoveries between 60-130% was obtained following the 16 different clean-up procedures (an asterisk indicate that the effect is significant P<0.05). Zr: Z-sep.