

EVALUATING THE EFFECTIVENESS OF INCORPORATING CERAMIC HOMOGENIZERS IN THE QUECHERS EXTRACTION METHOD

BRIAN KINSELLA, XIAOYAN WANG AND THOMAS AUGUST
UCT, INC. 2731 BARTRAM ROAD, BRISTOL PA 19007 • 800.385.3153 • WWW.UNITEDCHEM.COM



INTRODUCTION

The QuEChERS sample preparation method was first introduced in 2003 by Anastassiades et al for the analysis of pesticide residues in food [1]. QuEChERS involves the extraction and partitioning of residues from an aqueous sample (water is added to dry samples) into acetonitrile (MeCN) with the addition of salts to induce phase separation. This is followed by dispersive solid phase extraction (dSPE) to remove matrix co-extractives. Since its inception, there has been a large increase in the use of QuEChERS in chemical residue analysis, including in veterinary drug residue analysis and environmental analysis. Several modifications have been made to the method to improve sample clean-up and recovery of particular analytes, such as the use of buffered extraction and different dSPE sorbents. Recently, ceramic homogenizers have been introduced to the environmental and food safety area to enhance the recoveries and decrease the variability of the method. The aim of this study was to compare the use of ceramic homogenizers in the extraction step versus no homogenizers. Three different matrices (fish, strawberries and multigrain cereal) and two different classes of residues (polycyclic aromatic hydrocarbons and organophosphate/organochlorine pesticides) were evaluated.

EXPERIMENT 1

16 PAHs in fish QuEChERS extraction

- Weigh 5 g homogenized sample into a 50 mL centrifuge tube and spike with standard/internal standard
- Vortex and equilibrate for 15 min
- Add 2 ceramic homogenizers to each tube to be evaluated with homogenizers
- Add 5 mL DI H₂O and 10 mL MeCN
- Add contents of Mylar pouch containing 4 g MgSO₄ and 2 g NaCl extraction salts (UCT# ECQUUS2-MP) to each tube, shake for 1 min, and centrifuge at 4000 rpm for 5 min

Dual layer SPE cleanup

- Condition dual layer (PSA/C18) SPE cartridges (UCT# ECPSAC1856) topped with ~0.5 cm of muffled Na₂SO₄ with 2-3 mL MeCN and turn on full vacuum for 1 min
- Add 5 mL sample extract to the cartridge and collect by applying 3" Hg vacuum, (up to 60% of PAHs remain in the cartridge) add an additional 4 mL MeCN, and collect
- Add 0.5 mL toluene and concentrate to 0.5 mL with a gentle stream of nitrogen at 35 °C
- Transfer the concentrated extract into a GC vial

RESULT

16 PAHs IN FISH

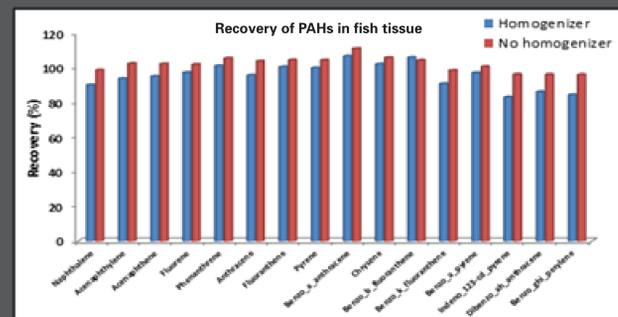


Figure 1. Recovery data of 16 PAHs in fish tissue fortified at 10 ng/g (n = 3)

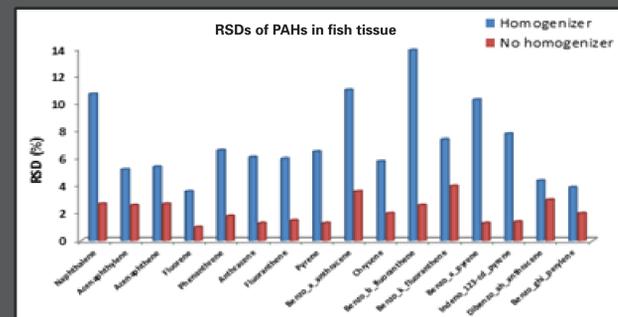


Figure 2. Reproducibility data of 16 PAHs in fish tissue fortified at 10 ng/g (n = 3)

EXPERIMENT 2

25 organophosphate and organochlorine pesticides in strawberries QuEChERS extraction

- Weigh 10g homogenized sample into a 50 mL centrifuge tube and spike with standard/internal standard
- Vortex and equilibrate for 15 min
- Add 2 ceramic homogenizers to each tube for extractions to be evaluated with homogenizers
- Add 10 mL MeCN
- Add contents of Mylar pouch containing 4000 mg magnesium sulfate, 1000 mg sodium chloride, 500 mg sodium citrate dibasic sesquihydrate and 1000 mg sodium citrate tribasic dihydrate extraction salts (UCT# ECQUEU750CT-MP) to each tube, shake for 1 min, and centrifuge at 4000 rpm for 10 min

SPE cleanup with Quick QuEChERS cartridge

- Transfer 1 mL supernatant to a 10 mL syringe attached to the Quick QuEChERS Push Thru cartridge containing 110 mg MgSO₄ and 180 mg PSA (UCT# ECPURMP5MC)
- Samples were filtered through the Push Thru directly into GC vials

RESULT

25 PESTICIDES IN STRAWBERRIES

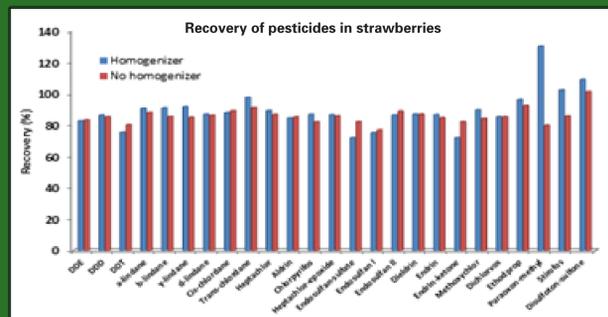


Figure 3. Recovery data of 25 organophosphate and organochlorine pesticides in strawberries fortified at 250 ng/g (n = 3)

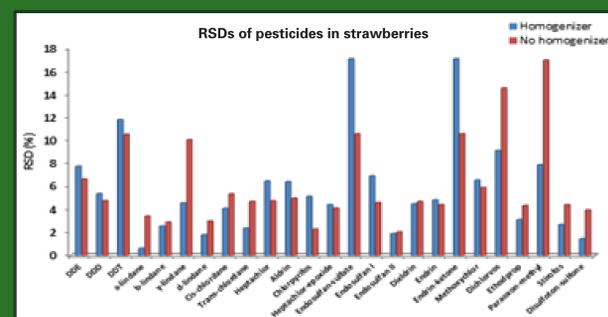


Figure 4. Reproducibility data of 25 organophosphate and organochlorine pesticides in strawberries

EXPERIMENT 3

16 organochlorine pesticides in multigrain cereal QuEChERS extraction

- Weigh 5 g homogenized sample into a 50 mL centrifuge tube and spike with standard/internal standard
- Vortex and equilibrate for 15 min
- Add 10 mL H₂O and allow sample to hydrate for 15-30 min, then add 10 mL MeCN
- Add 2 ceramic homogenizers to each tube to be evaluated with homogenizers
- Add contents of Mylar pouch containing 4000 mg magnesium sulfate, 1000 mg sodium chloride, 500 mg sodium citrate dibasic sesquihydrate and 1000 mg sodium citrate tribasic dihydrate extraction salts (UCT# ECQUEU750CT-MP) to each tube, shake for 1 min, and centrifuge at 4000 rpm for 10 min

dSPE cleanup

- Transfer 1 mL of supernatant to a 2 mL dSPE centrifuge tube containing 150 mg MgSO₄ and 50 mg PSA (UCT# CUMPS2CT)
- Vortex samples for 30 sec, centrifuge at 5000 rpm for 5 min, and transfer 500 µL of the extract into GC vials

RESULT

16 PESTICIDES IN MULTIGRAIN CEREAL

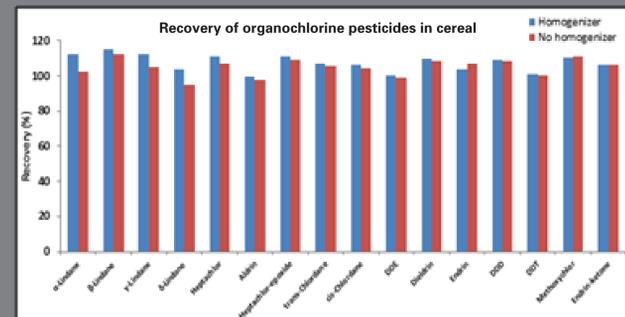


Figure 5. Recovery data of 16 organochlorine pesticides in strawberries fortified at 250 ng/g (n = 3)

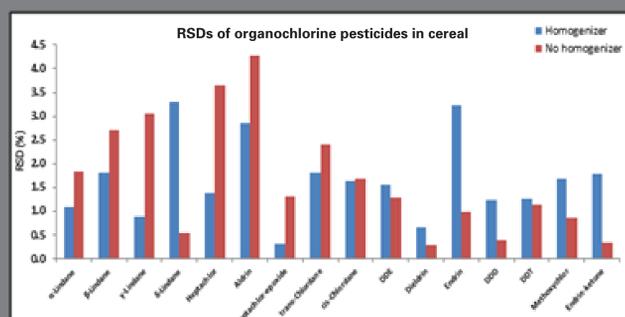


Figure 6 - Reproducibility data of 16 organochlorine pesticides in strawberries fortified oat 250 ng/g (n=3)

INSTRUMENTATION

GC-MS CONDITIONS

Two instruments were used during this study:

1. Thermo Scientific Trace GC Ultra coupled with ISO single quadrupole MSD with TriPlus Autosampler and Xcalibur (V 2.1) software. Column: Restek Rtx-5MS (30m x 0.25 mm x 0.25 µm) with 10 m guard column.
2. Agilent 6890N GC coupled with 5975C MSD, equipped with 7683 auto sampler and controlled by Chemstation software. Column: Restek Rxi-5sil MS 30m*0.25mm*0.25um integrated with 10 m guard column.

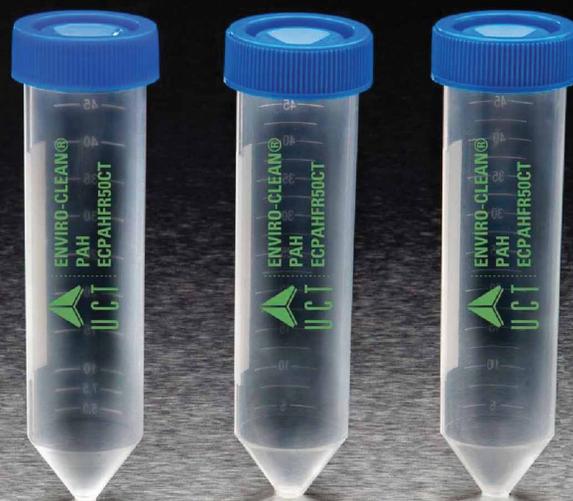
Additional GC-MS parameters are available by contacting the corresponding author (bkinsella@unitedchem.com).

CONCLUSIONS

- The results of this study illustrate that ceramic homogenizers do not have a significant effect on the recovery or variability of residue concentration when used in QuEChERS extraction. While in some cases (e.g. difficult matrices) the use of ceramic homogenizers may provide a better degree of homogenization, for the matrices and analytes tested in this study no significant differences in the recovery or variability was found.
- For PAHs in fish tissue, higher recoveries and less variability was obtained when the homogenizers were not used. The results for the pesticides in strawberries were almost identical. For pesticides in cereal the homogenizers also gave similar result as the regular QuEChERS method.

REFERENCES

- [1] M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenk, J. AOAC Int. 86 (2003) 412.



WWW.UNITEDCHEM.COM