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Efficiency of QuEChERS approach for determining 52 pesticide residues in honey and honey bees

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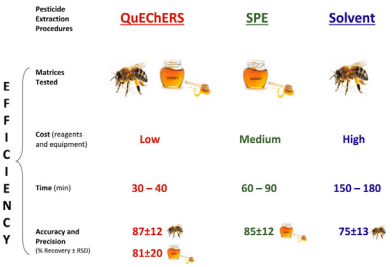
GRAPHICAL ABSTRACT



ABSTRACT

A comparison between QuEChERS and other pesticide extraction procedures for honey and honey bee matrices is discussed. Honey bee matrix was extracted by solvent based procedure whereas solid phase extraction was the protocol for the honey matrix. The citrate buffered QuEChERS method was used for both matrices. The methods were evaluated regarding cost (equipment and reagents), time, accuracy, precision, sensitivity and versatility. The results proved that the QuEChERS protocol was the most efficient method for the extraction of the selected pesticides in both matrices.

- QuEChERS is the most economical and less time-consuming procedure.
- SPE and solvent-based extraction procedures show equivalent recoveries to QuEChERS.
- QuEChERS can be used to extract pesticide residues from both matrices.

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Method details

QuEChERS approach for the extraction of pesticide residues in honey and honey bee matrices [1–3].

1) Weigh 5 g of honey or honey bees into 50 mL centrifuge tubes and add 7.5 mL of water, 10 mL of acetonitrile, 6 g of MgSO4 and 1 g of NaCl. Homogenize the mixture immediately and then, centrifuge for 5 min at 300 rpm.
2) Put 2 mL of the supernatant into another 15 mL centrifuge tube containing 50 mg C18, 50 mg PSA, and 150 mg MgSO4. Vortex the mix and centrifuge it for 5 min at 3000 rpm.
3) Finally, filter the supernatant using a PTFE 13 mm × 0.22 μm into the autosampler vials for LC–MS analysis.

Solvent approach for the extraction of pesticide residues in honey bee matrix [4].

1) Weigh 5 g of honey bees and pound thoroughly in a glass mortar. When homogenized place in a 250 mL flask and mix it vigorously for 10 min with 20 mL of acetone.
2) Filter the mixture in a Kitassato flask through a Buchner funnel of 13 cm with a paper filter packed with a layer of Celite 545 (5–10 mm) and wash the filter cake with 20 mL of acetone.
3) Prepare 100 mL, with 1% weight/volume (w/v) ammonium chloride and 2% volume/volume (v/v) ortophosphoric acid (85%) and add it to the filtrate. Allow it to stand for 30 min with occasional stirring and then filter with Celite 545.
4) After filtration, dilute the sample with 200 mL of 2% aqueous sodium chloride (w/v) and extract twice with 100 mL of dichloromethane.
5) Pass the resultant organic phase through a filter containing anhydrous sodium sulfate and evaporate it to dryness in a rotary evaporator at 35 °C.
6) Dissolve the extract obtained from the honey bee samples in acetone, up to 2 mL, for GC analysis. For LC–MS determination, evaporate to dryness a 1-mL aliquot of the previous extract using a gentle stream of nitrogen and then dissolve it in the same volume of methanol.

Solid phase extraction (SPE) approach for the extraction of pesticide residues in honey matrix [5].

1) Weigh honey (1.5 g) and mix it with 30 mL of hot water (<80 °C). Agitate by a stir bar for 10 min.
2) Pre-condition an Oasis HLB cartridge [poly (divinylbenzene-co-*N*-pyrrolidone)] with 5 mL of methanol and 5 mL of Milli-Q water.
3) Pass the mix through the cartridge at a flow rate of 10 mL min^{-1}.
4) Rinse the cartridge with 5 mL of Milli-Q water.
5) Dry the cartridge under vacuum for 15 min.
6) Elute the retained pesticides by passing 10 mL of methanol–dichloromethane (3:7).
7) Evaporate the eluate to 0.5 mL using a gentle steam of nitrogen.
8) Then, transfer it into 1-mL volumetric flask with methanol, obtaining a final extract in 100% methanol.