Effect of the homogenization process on the analysis of target pesticide residues in tomato and lemon

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Overview

Pesticide residues are not uniformly present in agricultural raw materials. In order to analyze them correctly, it is necessary to obtain a perfectly homogeneous sample before the extraction process. Therefore, it is very important to carry out a correct and representative processing. During method validation, the most common spiking procedure is performed on the homogenate commodity; consequently, the homogenization process is not considered.

With the aim to understand how the homogenization process affects the analysis of a sample, spiking was carried out by injecting the pesticides directly inside the raw commodities with a micro syringe, before milling. The variables evaluated were the type of blender, the grinding temperature and the degree of uniformity of the residues in the total sample. A total of 18 pesticides were analysed in "injected" tomato and lemon samples.

Compounds analysed

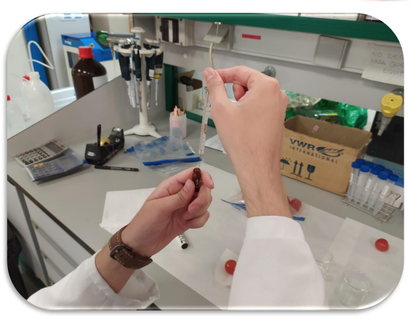
LC-MS/MS: Acephate, acetamiprid, chloridazon, dicrotophos, dimethoate, monocrotophos, propamocarb, terbuthylazine desethyl, thiamethoxam

GC-MS/MS: Bromopropylate, endosulfan-alpha, etrimfos, flamprop-methyl, HCB, lindane, quintozene, sulfotep, tecnazene

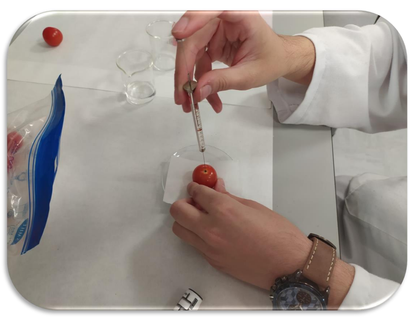
Cherry tomato

A needle was heated with a lighter.

A hole was made in the top of the tomato until it reached approximately to the centre.



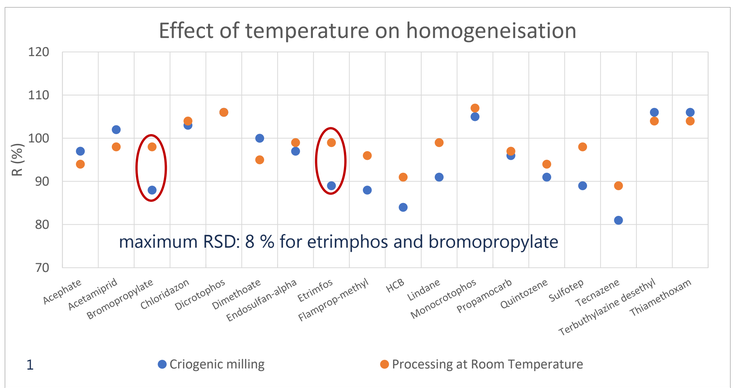
The volume of interest was taken from the prepared pesticide mix with a 10 µL GC micro syringe.

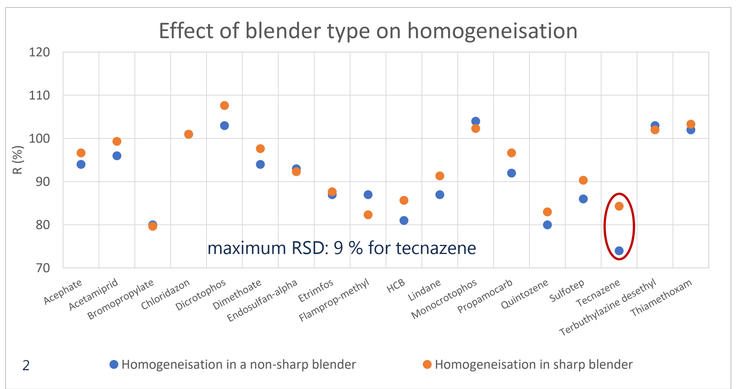


The pesticides were introduced into the tomato through the previously formed hole (5-10 µL in each tomato for a total concentration of 0,02 mg/kg).

All tomatoes were homogeneised (200 g, 15-17 units)

Studied variables





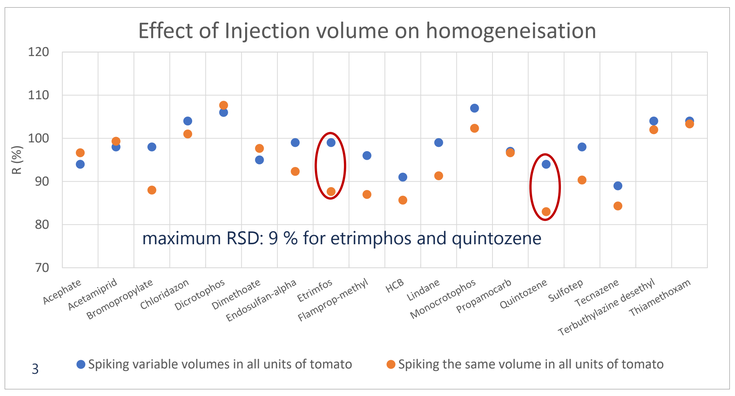
For each experiment, three recovery replicates were extracted

Recoveries studied at 0,02 mg/kg

All recoveries were within the range 70-120 %

Each experiment of spiking by injection was repeated three times, in order to evaluate the repeatability of the procedure. RSD was in all cases below 11 %.

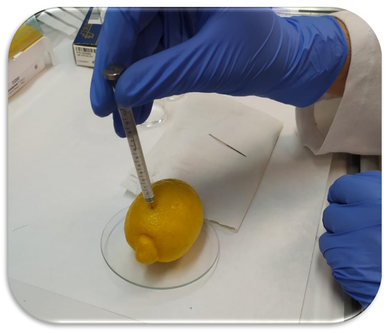
Each point in the graph represents the average of the three replicates



Lemon

A cold needle was inserted in several different places into the lemon.

Each lemon had several holes.

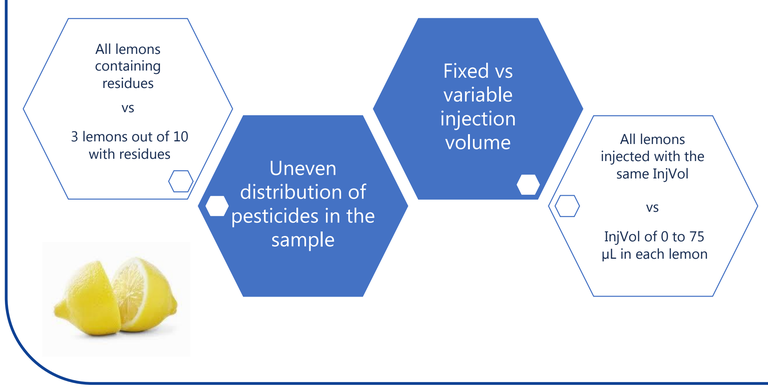


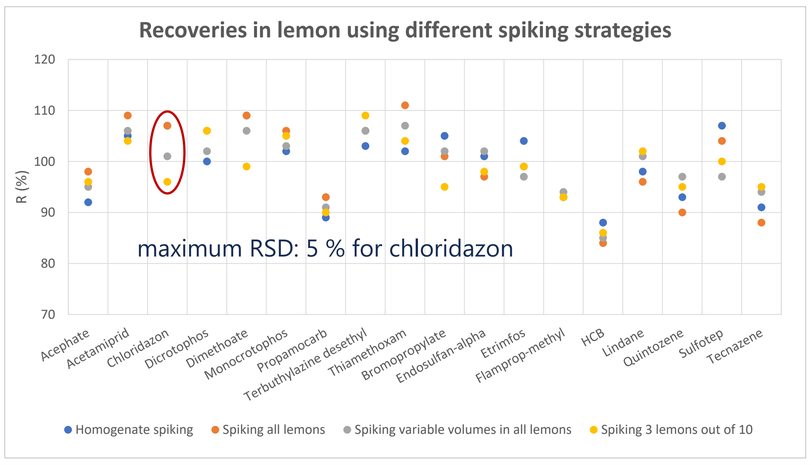
The pesticides were introduced into the lemon through the previously formed holes (5-75 µL in each lemon for a total concentration of 0,02 mg/kg).

Lemons were homogeneised (1000 g, 10 units).

Lemon homogenate after milling.

Studied variables





Conclusions

- The preliminary results show that the homogenisation process does not have a significant effect on the variability of the results of the target residues studied.
- For this study it is not relevant to include a factor related to the uncertainty data derived from the homogenisation process.
- When strict protocols are applied in sample processing, the homogenisation step does not have a major impact on the results.

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