Supercritical fluid chromatography coupled to tandem mass spectrometry for the analysis of captan and folpet in fruits and vegetables.

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INTRODUCTION

Captan and folpet are pesticides widely used in agriculture practices; however, they present some difficulties to be analyzed. These troublesome compounds are not amenable compounds by liquid chromatography (LC), and, as they are thermally labile pesticides, experience degradation in the source during gas chromatography (GC) injection. Their metabolites phthalimide (PI) and 1,2,3,6-tetrahydropthalimide (THPI) can be analyzed; nevertheless, the origin of these metabolites can be produced from other sources. As opposed to gas chromatography injection, no degradation is observed in the electrospray (ESI) source during supercritical fluid chromatography (SFC) analysis. The analysis of captan and folpet is necessary to avoid quantitation uncertainties.

EXPERIMENTAL

System: Shimadzu Nexera UC coupled to LC-MS 8060

SFC parameters:
- Injection volume: 5µL
- Flow rate: 1.3 mL/min
- Make up flow: 0.08 mL/min
- Oven temperature: 40ºC
- BPR pressure: 150 bar
- BPR Temperature: 50ºC
- Mobile Phases: Modifier: MeOH 1mM HCOONH₄, Make up: MeOH 5mM HCOONH₄ 0.1% HCOOH

MS parameters:
- Ion source: ESI
- Dwell time: 5 ms
- Nebulizing Gas Flow: 3 L/min
- Heating Gas Flow: 10 L/min
- Drying Gas Flow: 10 L/min
- Interface temperature: 125ºC
- DL temperature: 125ºC
- Heat Block Temperature: 150ºC

RESULTS AND DISCUSSION

Ion source optimization

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>Interface (ºC)</th>
<th>DL (ºC)</th>
<th>Heat Block (ºC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>100</td>
<td>100</td>
<td>150</td>
</tr>
<tr>
<td>12</td>
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<td>200</td>
<td>300</td>
</tr>
<tr>
<td>15</td>
<td>300</td>
<td>250</td>
<td>400</td>
</tr>
</tbody>
</table>

SFC vs LC

This same set was injected changing the type of chromatography (ESI) parameters maintained.

Recoveries

Tomato and pepper samples were spiked at 10 and 50 µg/kg. The recoveries were studied by SFC and GC.

Conclusions

- The ion source optimization proves that the optimum temperatures for captan and folpet are 125, 125 and 200ºC for interface, DL and heat block, respectively.
- A direct comparison between SFC and LC demonstrates that higher sensitivity can be obtained for captan and folpet using supercritical fluid chromatography.
- The recoveries of both compounds using SFC were between the 70-120% range for tomato and pepper matrices at 10 and 50 µg/Kg concentration levels.
- The reproducibility results were below 10% in all the studied cases.

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