The Accurrence of Pesticides Residues after the Wheat Expanding Process

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Abstract. Analysis was taken in a lot of pearl barley wheat suspected presence of substances that when expanding cause skin irritation to workers who executed the operation. The objective of this work consisted in the analysis of gas chromatography coupled with mass spectrometry (GS-MS) of wheat samples used in obtaining expanded products, compared with samples of wheat resulting foam, in order, highlighting the residue of chemical contaminants. Since no information about a possible presence of contaminants, was identified from the two drop chromatograms using mass spectra and their comparison with mass spectra library of mass spectra of NIST existence. Further analysis was done on a comparative trickle chromatograms through duplication occurring at the same time retention highlighting to any concentration or dilution of the compounds identified in the two matrices respectively: wheat and expanded wheat.

Keywords: pesticide, expanded wheat, GS-MS.

INTRODUCTION

This paper started from the fact that the department expand a factory in sugar confectionery expanding a lot of pearl barley wheat led to skin irritation on the hands and body workers who carried out this operation. The batch was immediately withdrawn by the manufacturer and have evidence for laboratory analysis to LICSA section of the TPPA USAMV Cluj-Napoca. On this occasion there were made chromatographic analysis both before and after expand lot.

MATERIALS AND METHODS

The material taken in the analysis was a sample from a lot of pearl barley wheat suspected to cause irritation workers performing the expand operation, pearl barley and wheat resulting expanding.

As a method was used chromatography analysis. Preparation of samples was performed according to SR EN ISO 12393-2003. The pesticide residues were extracted from the sample with acetonitrile and petroleum ether, purified by passage on florisil column and eluate with a mixture of ethyl ether and petroleum ether. The elute was concentrated and quantitatively determine the pesticide residues by gas phase chromatography coupled with mass spectrometry.

Equipment: gas chromatograph coupled with mass spectrometer Schimadzu, model QP-2010 equipped with an auto sampler AOC-5000 (CombiPAL). The column used to separate components: AT-5, 30m x 0.25mm x 0.25µm (Alltech, USA). The method of analysis are: 250°C injector temperature, pressure: 37.1 kPa, linear velocity: 32.4 cm/ s; splitare report 1:200. Carrier gas: helium, detector: MS, ion source temperature: 250°C; interface temperature: 250°C MS mode: EI. Mass range: 40-400u. Scan speed: 769u/s. The
temperature for column chromatography: 60°C (5 min) at 160°C to 240°C with 4°C/min (1min) with 15°C/min. Identification of separated compounds was done using the spectra library NIST 127 AND NIST 147.

RESULTS AND DISCUSSIONS

In Fig. 1A and Fig. 1B, are presented the chromatograms obtained for the two food matrices: wheat respectively expanded wheat. As apparent from comparing the two chromatograms common compounds are represented by three major drop in retention time TR: 7.273, 32.322 and 34.320.

![Fig. 1A Chromatogram wheat](image)

![Fig. 1B Chromatogram expanded wheat](image)

In Tab. 1 are presented highlight common compounds, major mass ion (m/z), retention time and their concentration in wheat respectively expanded wheat, expressed in percentage of the area. Analyzed comparative data from Table 1 can find, on this their wheat and wheat expanded, as are the percentages of area for the majority for Compound 1 in expanded wheat, 74.18% to 71.59%, while for Compound 2 and Compound 3 are majority in wheat, the differences for both compounds were approximately around 3%. From the presented facts results that in the expanding process have not produced major changes in the concentration of any of the three compounds in terms of their concentration in wheat expanded, also not in meaning of their dilution. This raises a very delicate issue, on the impossibility decrease concentration of these compounds, in case they would be contaminated class of chemical substances used in the treatment of the wheat plant.
Highlight common compounds and their concentrations in wheat and wheat expanded

<table>
<thead>
<tr>
<th>No.</th>
<th>Compound no.</th>
<th>m/z</th>
<th>Retention time</th>
<th>Concentration in wheat %</th>
<th>Concentration in expanded wheat %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Compound no.1</td>
<td>205</td>
<td>7.273</td>
<td>71.59</td>
<td>74.18</td>
</tr>
<tr>
<td>2.</td>
<td>Compound no.2</td>
<td>57</td>
<td>32.322</td>
<td>8.85</td>
<td>6.11</td>
</tr>
<tr>
<td>3.</td>
<td>Compound no.3</td>
<td>57</td>
<td>34.320</td>
<td>12.73</td>
<td>9.51</td>
</tr>
</tbody>
</table>

In order to identify separately compounds (Tab.1) by chromatography gas, were used in the mass spectra, which were compared with those existing in the libraries of spectra NIST 127 and NIST 147.

In Fig. 2 are presented for the mass spectra of the compound with retention time of 7.273 wheat respectively expended wheat compared with library spectra.

![Mass spectra](image1)

(a) sample of wheat, (b) expanded wheat sample (c) spectrum of the mass spectra library

The pick represented in Fig. 3 is the Compound no.1 (see tab.1) at retention time 7.273 and it can be assigned to him under the bank of spectra, the compound di-tertbutilhidroxitoluen (butylated hydroxytoluene), which is known as Advastab 401. This compound is concentrated around the larger. 3% of the sample area in expanded wheat (74.18%) compared with the sample of wheat (71.59%), which could indicate, in a first approximately,
in the absence of more rigorous quantitative determinations, a slight concentration of this compound during the process of expand, is not just desirable. To clarify this issue, very important in a technological process, further research is necessary to study the influence expand the content in di-tertbutil-hidroxitoluen.

![Fig. 3 Compound no.1 (TR: 7,273) 1- in wheat, 2 - wheat in expanded](image)

The Pick represented in Fig.4 is the Compound no. 2 (see Tab. 1) that appears at the retention time of 32.322 and which can be attributed to a carbohydrate composed of 12 carbon atoms. It couldn’t be determinate exactly with the mass spectrum, what compound it is, that’s why needs further investigation. This compound is in greater concentration in the sample of wheat (8.85% area) compared with the expanded sample of wheat (6.11% area).

![Fig. 4 Compound no. 2 (TR: 32,322) 1 - in wheat, 2 - wheat in expanded](image)

The Pick in Fig. 5 represents Compound no. 3 (see Tab. 1) from retention time of 34.32 and could be assigned to a hexadodecan but with a lower probability, needs further investigation. What can be noticed is that molecular ion has the same m/z as that of compound
2. Compound No.3 in greater concentration in the sample of wheat (8.85% area) compared with the expanded sample of wheat (6.11% area).

![Fig. 5 Compound No. 3 (TR: 34.320) 1 - in wheat, 2 - wheat in expanded](image)

**CONCLUSIONS**

- It has been realized GS-MS analysis of samples of wheat and expanded wheat - derived from wheat by expand - to found a class of contaminants in pesticides using banks spectra of NIST 127 and NIST 147;
- There were compared common compounds identified by overlapping dribble for highlighting changes to the product area picului expanded;
  - The pick at retention time of 7.273 m/z 205 can be attributed di-tertbutil-hidroxitoluenului (butylated hydroxytoluene) with trade name Advastab 401;
  - The pick at retention time is 7.273 in greater concentration in the sample expanded wheat (74.18% area) compared with the sample of wheat (71.59% area), which indicates a concentration of this compound, during expanding process;
- The pick at retention time 32.322 with m/z 57, is in greater concentration in the sample of wheat (8.85% area) compared with the expanded sample of wheat (6.11% area), which indicates reduction during expanding process;
- The pick at retention time 34.320, with m/z 57, is in greater concentration in the sample of wheat (8.85% area) compared with the expanded sample of wheat (6.11% area), which indicates reduction during expanding process.

**REFERENCES**

1. SR EN 12393 (2003) Metode multireziduu pentru determinarea gaz cromatografica a reziduurilor de pesticide