SACI in the analysis of complex pesticide mixture

Keywords: Pesticides, vegetables, high sensitivity

Pesticides have been analyzed, in the recent years, by various groups by means of mass spectrometry. In particular, liquid chromatography-mass spectrometry (LC-MS) is an invaluable technique for the control of pesticide residues to ensure food safety . Different mass analyzer (Ion Trap, Time of Flight etc) were coupled with the wide employed Electrospray (ESI) and Atmospheric Pressure Chemical Ionization (APCI) sources to analyze pesticides.

Figure 1 :SACI source on HCT ultra.

Surface Activated Chemical Ionization:

Surface-activated chemical ionization (SACI) is a new ionization technique operating with analyte solutions.

Insertion of a metallic surface in the ionization chamber [Fig.1], allowing for a better ion focalization and hence for an increase in ionization efficiency In particular, while in the case of ESI and APCI, high electrostatic potential (3–6 kV) are employed to lead to the sample ionization, in the case of SACI, the vaporized or sprayed analyte solution experiments employ a low voltage charged surface inserted (50 V) in an APCI ionization source..

In this case, the corona discharge needle apparatus is not used but the ionization effect is activated only by the surface.

Experimental conditions and settings:

Analyte: Mixture af 50 different pesticides

Sample: Spinaches

Sample Preparation and Analytical Conditions:

10g of spinaches were homogenized in a blender containing 100 ml acetone, 75 ml dichloromethane and 15 g sodium chloride for three minutes. The homogenized mixture was allowed to separate into its organic and aqueous layers. The organic phase was transferred to a beaker and 3g of sodium sulphate was added to remove the remaining water. This extract was passed thought a SPE C18 (Supelco, USA).

LC-MS/MS conditions: C_{18} 50 × 2.1 mm, 3µm column was used. The chromatographic analysis was performed under gradient conditions.

A linear gradient of MeOH (0,1% Formic Acid) - H_2O (0,1% Formic Acid); flow 0.5ml/min; injection volume 20 µL per sample.

Instruments Employed:

- HCT ultra, Bruker Daltonics
- HPLC Dionex Ultimate 3000

Experimental Results:

The results achieved in the LC-SACI analysis of 50 pesticides (Fig 2), in terms of limit of detection (LOD), limit of quantitation (LOQ), linearity ranges, quantitation precision and accuracy (Table 1) are shown.

Moreover, the data obtained were compared with these achieved using ESI and APCI, showing that strong benefits in terms of sensitivity, quantitation precision and accuracy are obtained using the SACI source.

Finally, this approach has been used in the analysis of pesticides present in spinaches extract (Fig 3).

Source	Average Lod (pg/injected)	Average Lod (pg/injected)	Precision Error	Accuracy Error	Linearity Range (pg/injected)
ESI	40	100	4%	8%	100 - 100000
APCI	20	50	6%	9%	50 - 100000
SACI	5	10	4%	7%	10- 100000

Table 1: APCI, ESI and SACI linearity ranges, Limit of Detection (LOD), Limit of Quantitation (LOQ), % precision error and % accuracy error. Data are reported as average of all pesticides.

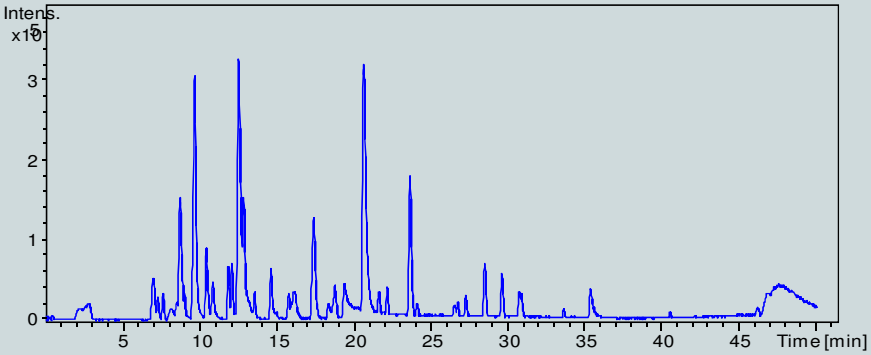

Figure 2: Base peak of 1ng of standard pesticide mixture obtained by SACI

Conclusions:

SACI has shown to increase the instrumental performance with respect with the usually employed ESI and APCI ionization sources. In the analysis of a complex mixture of 50 different pesticides, strong benefits have been obtained main terms of Limit of Detection (LOD), Limit of Quantitation (LOQ) and Linearity range.

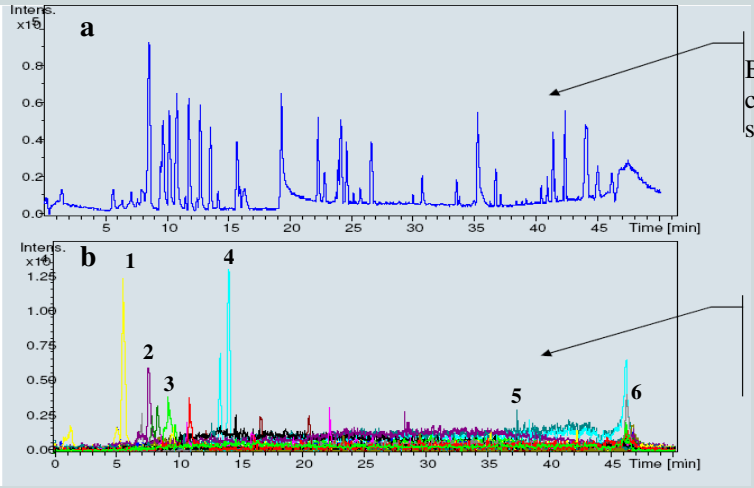

Figure 2: a) Base peak and b) Extracted Ion Current (EIC) of spinaches extract obtained by SACI source; pesticides detectable in the biological sample are reported.

Acknowledgements

1) ISB srl, via Fantoli 16/15, 20138 Milano.
2) Bruker Daltonics S.r.l., Macerata, Italy.

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