

Multiresidue determination of 75 pesticides in asparagus and sweet pepper by QuEChERS method and LC-MS/MS detection

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Amount exported of agrarian commodities from Peru in 2008 reached to US\$ 2,597 millions which meant an increase of 31.7% compared with 2007, the main commodities exported were asparagus and sweet pepper and the most important destiny countries were United States (31%), Spain (12%) and Netherlands (10%) [1]. However; it has been attenuated by rejections mainly due to presence of pesticide residues at levels greater than MRL. During 2008, 13% of rejection was due to pesticide residues [2]. For this reason, the Control Center of Toxic Residues and Agricultural Products (official Peruvian laboratory for analysis of pesticide residues in food) has been implementing multiresidue pesticide analytical methods. The aim of this study was to verify the performance of a multi-residue method for 75 pesticides including mainly conazole, carbamate, benzimidazoles, pyrimidine fungicides and insecticides in asparagus and sweet pepper using QuEChERS method [3] and detection by LC-MS/MS in positive and negative ESI mode. A representative portion of the sample (15 g) was subjected to extraction with acetonitrile containing acetic acid, followed by the addition of salts (MgSO₄ and NaOAc) and then perform dispersive solid-phase extraction with a mixture of magnesium sulfate, primary secondary amine (PSA) and graphitized carbon black (GCB). The polarity switching capability enabled the determination of 68 of the compounds in the positive ionization and 7 of the compounds in the negative ionization mode in a single run. The recovery and precision were assessed at levels of 10, 20 and 50 µg/kg, and linearity with matrix-matched calibration in 5 levels (5 to 250 µg/kg). Results for the 75 pesticides assessed in both matrix of this study ranging from 50 - 120% for recovery, 3.9 to 26% for precision (%RSD) and 2 to 24 µg/kg for LOD. The recoveries were found satisfactory for most pesticides in according with the criteria of Codex with exception for seven pesticides (thiabendazole, tiadicarb, triflumizol, carboxin, tiophanate metil, etofenprox, and bensulfuron metil) in the asparagus and fenazaquin in the sweet pepper. Precision (%RSD) values fulfilled the AOAC criteria (<15%). The range of the analytical curve was linear and the correlation coefficients were mostly >0.99. The MRM chromatograms also showed good selectivity. The results show that the method QuEChERS is adequate for official control and could be used for the national program of monitoring pesticides residues.

[1] Evolución de las exportaciones peruanas Enero – Diciembre 2008.

http://www.siicex.gob.pe/siicex/portal5ES.asp?_page_=458.42200

[2] Import Refusal Reports for OASIS.

http://www.accessdata.fda.gov/scripts/ImportRefusals/ir_index.cfm

[3] Lehotay, S., 2007. Determination of Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate: Collaborative Study. *Journal of AOAC International*. 90:485-520.