EVALUATION OF VARIOUS QuEChERS BASED METHODS FOR THE ANALYSIS OF PESTICIDE RESIDUES IN POLISHED RICE.

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Abstract

There is no universal method for pesticide residue analysis in cereal grains. In spite of their similarities in the bulk chemical composition (starch, proteins and lipids), miniaturized multiresidue methods for pesticide analysis in cereal grains are still under development. Different production technologies and the chemical nature and purpose of the agrochemicals employed to increase crop productivity are the main reason for this situation. The technological package of pesticides, fungicides and herbicides currently employed in cereal cropping can be a serious risk to consumer's health if they are present in the final product, and they must be strictly controlled, but there is no official method available nowadays. In this communication we report our attempts to develop a multiresidue for the analysis of more than 50 pesticides (pre and post emergence herbicides, fungicides and insecticides) in polished rice currently employed in rice production, using a QuEChERS dispersive approach for the extraction and clean-up. Four different sample preparation methodologies based on MeCN extraction for two different sample amounts (5 and 7.5 g) are compared. On one hand, original QuEChERS and Citrate buffered QuEChERS and on the other, acetate buffered QuEChERS without PSA clean-up and Citrate buffered QuEChERS without PSA or C-18 clean-up [1,2]. The performance of the tested methodologies was compared at two fortification levels: 10 and 300 µg Kg^{-1} by LC-QqQ/MS. Matrix effect was evaluated for the four methods using LC-TOF/MS. Linearity, limits of detection (LODs), accuracy and precision were assessed for the two different amounts of sample. For 7.5 grams at 10 µg Kg^{-1} between 67-75 % of the selected pesticides presented recoveries higher than 60 % in the four methods tested with a relative standard deviation below 20%, at 300 µg Kg^{-1} around 62-96 % of the pesticides under study showed good recoveries. When testing 5 g of sample the recoveries were in general lower than the ones obtained with 7.5 g while the limits of detection for both amounts of sample were not significantly different, being below 10 µg Kg^{-1} in general. Eighteen real samples were analyzed by LC-TOF/MS and LC-QqQ/MS using acetate buffered QuEChERS without PSA clean-up.

References

[1] U. K., K. S. and N. L. (2008) Rapid determination of phenoxy acid residues in rice by modified QuEChERS extraction and liquid chromatography–tandem mass spectrometry, Anal. Chim. Acta 626:10–20.

[2] L. L., Y. H.,*, Q. Y., Z. H. and L. J. (2006), Rapid analysis of multiresidual pesticides in agricultural products by gas chromatography-mass spectrometry. Chin J Anal Chem, 34: 783−786.