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Development of Method for Detecting Low Concentration of Organophosphate Pesticides in Vegetables Using near Infrared Spectroscopy

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Abstract : Vegetables are frequently contaminated with pesticides residues resulting in the most food safety concern among agricultural products. The objective of this work was to develop a method to detect the organophosphate (OP) pesticides residues in vegetables using Near Infrared (NIR) spectroscopy technique. Low concentration (ppm) of OP pesticides in vegetables were investigated. The experiment was divided into 2 sections. In the first section, Chinese kale spiked with different concentrations of chlorpyrifos pesticide residues (0.5-100 ppm) was chosen as the sample model to demonstrate the appropriate conditions of sample preparation, both for a solution or solid sample. The spiked samples were extracted with acetone. The sample extracts were applied as solution samples, while the solid samples were prepared by the dry-extract system for infrared (DESIR) technique. The DESIR technique was performed by embedding the solution sample on filter paper (GF/A) and then drying. The NIR spectra were measured with the transflectance mode over wavenumber regions of 12,500-4000 cm^{-1}. The QuEChERS method followed by gas chromatography-mass spectrometry (GC-MS) was performed as the standard method. The results from the first section showed that the DESIR technique with NIR spectroscopy demonstrated good accurate calibration result with R^2 of 0.93 and RMSEP of 8.23 ppm. However, in the case of solution samples, the prediction regarding the NIR-PLSR (partial least squares regression) equation showed poor performance (R^2 = 0.16 and RMSEP = 23.70 ppm). In the second section, the DESIR technique coupled with NIR spectroscopy was applied to the detection of OP pesticides in vegetables. Vegetables (Chinese kale, cabbage and hot chili) were spiked with OP pesticides (chlorpyrifos ethion and profenofos) at different concentrations ranging from 0.5 to 100 ppm. Solid samples were prepared (based on the DESIR technique), then samples were scanned by NIR spectrophotometer at ambient temperature (25+2°C). The NIR spectra were measured as in the first section. The NIR- PLSR showed the best calibration equation for detecting low concentrations of chlorpyrifos residues in vegetables (Chinese kale, cabbage and hot chili) according to the prediction set of R2 and RMSEP of 0.85-0.93 and 8.23-11.20 ppm, respectively. For ethion residues, the best calibration equation of NIR-PLSR showed good indexes of R^2 and RMSEP of 0.88-0.94 and 7.68-11.20 ppm, respectively. As well as the results for profenofos pesticide, the NIR-PLSR also showed the best calibration equation for detecting the profenofos residues in vegetables according to the good index of R^2 and RMSEP of 0.88-0.97 and 5.25-11.00 ppm, respectively. Moreover, the calibration equation developed in this work could rapidly predict the concentrations of OP pesticides residues (0.5-100 ppm) in vegetables, and there was no significant difference between NIR-predicted values and actual values (data from GC-MS) at a confidence interval of 95%. In this work, the proposed method using NIR spectroscopy involving the DESIR technique has proved to be an efficient method for the screening detection of OP pesticides residues at low concentrations, and thus increases the food safety potential of vegetables for domestic and export markets.

Keywords : NIR spectroscopy, organophosphate pesticide, vegetable, food safety
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