Partial least squares- residual bilinearization for simultaneous determination of ten pesticides in milk using QuEChERS-dispersive liquid-liquid microextraction followed by gas chromatography

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ABSTRACT

Milk is one of the best sources of proteins, lactose, and minerals which virtually all ages among people consume it at least one time in a week. Unfortunately, sometimes due to an inferior feeding of cows, it could be possible to detect some kinds of pesticides in their product. As a result, the quality control of milk to achieve the healthiest production is incredibly considerable for users. Plenty of efforts have been developed to determinate the amount of pesticides in complex matrices such as water, fruits, vegetables and milk [1]. In the present study, ten pesticides including dichlorvos, carbofuran, carbaryl, atrazine, ametryne, diazinon, pirimiphos methyl, chlorpyrifos, prothioconazole, and tebuconazole were extracted from milk by utilizing quick, easy, cheap, effective, rugged, and safe (QuEChERS) combined with dispersive liquid-liquid microextraction (DLLME) [1]. It should be noted that central composite design (CCD), multiple linear regression (MLR), and Nelder-Mead simplex optimization method were used in order to design, model, and optimize all of the effective factors of QuEChERS-DLLME for simultaneous determination of these mentioned pesticides with gas chromatography-flame ionization detector (GC-FID). Accordingly, global optimum conditions were gained which were 0.25 mL of acetonitrile, 42.1 µL of chloroform, 25 min of sonication time and 3.8 % (w/v) NaCl in pH 6.1. In the next section, partial least squares-residual bilinearization (PLS-RBL) [2] was utilized to build a multivariate calibration model in concentration range of 0.5-100 $ngmL^{-1}$. R^2 for calibration and cross validation were 0.997 and 0.973 respectively. Moreover, RMSEC and RMSECV were 1.758 and 7.332, and analytical figures of merit (AFOM) including sensitivity (the first and most important analytical parameter), analytical sensitivity, selectivity, limit of detection (LOD), and limit of quantitation (LOQ) were evaluated [3]. The validity of this proposed method was confirmed based on the comparison between calculated LOD and MRL guideline of European Union (EU). the value of recovery and RSD were 77.69-147.69% and 1.57-9.67%. This validated method was successfully applied for quantitative determination of 10 pesticides in milk samples and would be powerful to exploit in quality control laboratories as a reliable method.

Keywords: **"**Pesticides", "QuEChERS", "Dispersive Liquid-Liquid micro extraction", "Gas chromatography", "Multi-response optimization", "partial least squares- residual bilinearization"

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