



Determination of pesticide residues in beans using QuEChERS technique coupled to gas chromatography-mass spectrometry: Multivariate optimization of CEN and AOAC methods

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ABSTRACT

The use of pesticides has led to environmental pollution and posed a global health risk, since they remain as residues on foods. Beans one of the most widely cultivated crop in Africa, and susceptible to attack by insects both on field and during storage, leading to the application of pesticides to control pests' infestation. However, misuse of these chemicals by farmers on beans has resulted in the rejection of beans exported to European countries, due to the presence of pesticide residues at concentrations higher than the maximum residues levels (MRLs). In this study, the effectiveness of the Association Official Analytical Chemists (AOAC) Official Method and the European Committee of Standardization (CEN) Standard Method, were determined using multivariate approach for the analysis of organochlorine pesticide residues in 6 varieties of beans samples. The significance of factors (mass of sample, volume of acetonitrile, mass of magnesium sulphate, sample pH, centrifugation time and speed) affecting the efficiency of extraction was estimated using Plackett-Burman design, while central composite design was used to optimize the significant factors. The following optimum factors were subsequently used for method validation, recovery tests, and real sample analysis: 4 g of sample sludge (1:1 v/v), 10 mL of acetonitrile, 4.45 g of MgSO₄, and 5 min of centrifugation at 5000 rpm. The figure of merit of analytical methodology estimated using matrix-matched internal standard calibration method gave linearity ranging from 0.25 to 500 µg/kg, with correlation coefficient (R²) greater than 0.99, the recovery ranged from 75.55 to 110.41 (RSD = 0.70–16.65), with LOD and LOQ of 0.23–1.77 µg/kg and 0.76–5.88 µg/kg, respectively.

1. Introduction

The capacity of food production, availability and stability is faced with an ever-growing number of challenges (Calicioglu, Flammini, Bracco, Bellu, & Sims, 2019), including a world population growing at geometrical rate to nearly 10 billion by 2050 (UN., 2019) and a falling ratio of arable land to population explosion (Akpan & Ebong, 2021). The imbalance in population growth and arable land utilization have led to high demand for food and increased in the use of pesticides to increase production and meet the ever-increasing demand for food supply

(Abdulra'uf, Hammed, & Tan, 2012). The presence of pesticide residues on foods have become worrisome due to their health risks (including disruption of the endocrine system, alteration of hematological and hepatic systems, allergies and asthma (Pathak et al., 2022)). This has led to the establishment of maximum residue levels through the Codex Alimentarius Commission (Abdulra'uf, Chai, & Tan, 2012). Sample preparation stage is vital since the residues are present at trace level and embedded in the complex food matrices with diverse chemical and physical properties (Costa, Caldas, & Primel, 2014).

QuEChERS method was introduced in 2003 as "Quick, Easy, Cheap,

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