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Development and validation of analytical method for chlorpyrifos determination in biobeds

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Improper management of pesticides can cause serious environmental contamination. The key strokes are resulting from the filling operation, the spraying equipment leaks, equipment remains in the tank, internal cleaning wash water tank, among others. These spills can contaminate seriously the environment in case of contact with surface water and / or groundwater. As an alternative to minimize these potential problems, the use of biological treatments in biobeds systems can be efficient in the adsorption and degradation of pesticides in addition to being an inexpensive alternative and easy to operate. The aim of this study was to develop and validate a method for determination of chlorpyrifos pesticide residues in biobeds based on the method proposed by Racke, K. D. et al [1] and Coppola et al [2]. The extraction procedure used 5 g of sample, to which was added 30 mL of extraction solution containing acetone acidified with phosphoric acid and water in a ratio of 98:1:1 (v/v/v) and quinalphos at 500 ng/mL as procedure internal standard. This mixture was homogenized by ultraturax for one minute. After homogenization and centrifugation, an aliquot of 2 mL of supernatant was filtered. An aliquot of 125 μ L of organic extract was evaporated in a sample concentrator and reconstituted in 5 mL of methanol containing 0.1% acetic acid and internal standard (propoxur at 40 ng/mL). The extract was homogenized and analysed by UPLC-MS/MS. The method was linear in the concentrations studied (0.1; 0.5; 1; 5; 20; 100 and 250 ng/mL) with values of determination coefficient (r^2) of 0.9955 and 0.9909 for analytical curves prepared in matrix blank extract and the solutions prepared in organic solvent, respectively. The RSD% values obtained ranged between 9-24% for analytical solutions prepared in organic solvent and 6-21% for analytical solutions prepared in matrix blank extract. Estimated LOD were 0.1 ng/mL for the instrument and 23.5 mg/kg for the method and estimated LOQ were 0.3 ng/mL for the instrument and 78.4 mg/kg for the method. Accuracy (% recovery) and precision (as RSD%) were evaluated for 2, 10 and 50 mg/kg levels that showed recoveries of 115, 100 and 96%, and RSD values of 8.5; 3.2 and 5.1%, respectively. The intermediate precision was assessed by performing the fortification testing and recovery by two different analysts in different days. The %RSD obtained were 1.8; 4.1 and 3.6 for levels 2; 10 and 50 mg/kg, respectively. The real LOQ determined was the lowest concentration which showed recovery between 70% and 120% and RSD less than 20%, in this case 2 mg/kg. There was no significant matrix effect when comparing the results for the solutions of the calibration curves prepared in organic solvent and matrix blank extract.

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MAPA, MCTI, FINEP, CAPES, EMBRAPA, NWWA

[1] Racke, K.D.; Steele, K.P.; Yoder, R.N.; Dick, W.A.; Avidov, E.; J. Agric. Food Chem., 1996, 44, 1582-1592.

[2] Coppola, L.; Castillo, M.D.P.; Monaci, E.; Vischetti, C.; Journal of Agricultural and Food Chemistry, 2007, 55, 396-401.