



Determination of Fenvalerate residue in raisin via vortex-assisted surfactant-enhanced emulsification liquid–liquid microextraction (VSLLME) method by using HPLC system

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ABSTRACT

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In this project, ultra-trace amounts of *Fenvalerate* residue in raisin, were determined *via* vortex-assisted surfactant-enhanced emulsification liquid–liquid micro extraction (VSLLME) method and by using high performance liquid chromatography-photo diode array (HPLC-PDA) detector at 225nm. The main parameters relevant to this method were investigated and the optimum condition was established: 20 μL chlorobenzene was used as extraction solvent, 0.9 mmol.L^{-1} CTAB was selected as the surfactant, the extraction time was fixed at 60s, 2% sodium chloride was added and the extraction process was performed under the room temperature. Under the optimum condition, limit of detection (LOD) was 0.3 ng mL^{-1} . The relative standard deviation (RSD, $n=6$) was 2.87%. The linearity was obtained by five points in the concentration range of 0.3 to 100.0 ng mL^{-1} . Correlation coefficients (R^2) was 0.9997, and the enrichment factor (EF) was 114. Finally, the proposed method has been successfully applied for determination of Fenvalerate in real samples. The recoveries of the target analyte in raisins samples were between 84.13% and 92.12%. It seems that the addition of a surfactant, which was used as an emulsifier, could enhance the rate of the mass-transfer from aqueous samples to the extraction solvent.

1. Introduction

Grapes are consumed both in fresh and in processed products such as wine, juice, jelly, seed extract, raisin, vinegar and seed oil. In 2016, the international organization of vine and wine (OIV) had reported that the grape production was estimated at 7.8 million tons (US) of which 39% was produced in Europe, 34% in Asia and 18% in the United States [1]. After the green revolution, the use of pesticides was increased for all products such as grain food, vegetables, fruits, cotton and tobacco [2]. Fenvalerate is an insecticide which is a mixture of four different isomers with various activities. For example, 2- α configuration, which often includes 23%, shows some especial insecticide activity. Fenvalerate has a

modest toxicity for the mammals that can be harmful for the central nervous system through prolonged exposure [3]. The residue levels of this insecticide are directly related to its application. Also, it is more toxic to bees and fishes, and does not significant effect on plants, while, it can stay active for a long time [4]. Since the presence of this pesticide could be harmful to human and the environment, its residues in food must be closely monitored [5].

In the recent years, chromatography methods have been used to measure pesticides (including Fenvalerate) in foods and beverages [6]. One of the most important tasks in determining the amounts of pesticide is the preparation of the sample. In this regard, various methods are used for preconcentration and preparation of those. As an

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