A NOVEL THIOPHENE FIBER MODIFIED WITH CLAY FOR THE GAS CHROMATOGRAPHIC DETERMINATION OF CHLORPYRIFOS IN JUICE SAMPLES

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Contamination of water resources by pesticide residues is one of the major challenges for the preservation and sustainability of the environment. Their extensive use in world-wide agricultural practice in addition to industrial emission during their production has led to substantial occurrence of pesticide residues in food commodities. Chlorpyrifos (CP) is an organophosphorus pesticide defined as an endocrine disruptor [1] that has been used extensively on fruits to control various pests. Chromatographic techniques are widely employed for the determination of CP. In recent years, simplification and increasing automation of sample preparation steps are one of the modern trends in analytical chemistry. Solid-phase micro extraction (SPME) is a good alternative to these trends. In fact, it can be easily automated and allows, in a single step, pre-concentration and a rapid direct extraction, without the use of organic solvents. SPME methods have been developed for the determination of a large number of pesticides in different matrices [2]. In recent years, studies have been focused on fabricating low cost, simple, robust and long-life fiber for the target analyte [3]. A variety of methods for the production of fibers have been developed and among them, electrochemical polymerization is the method of choice as the film thickness can be controlled easily and polymers with different functional groups can be formed under controlled electrochemical conditions.

Present study deals with the modification of polythiophene (PTh) coated SPME fiber with a Montmorillonite (MMt) clay to improve the adsorption characteristics of the fiber to offer the analyst to exploit it as an innovative sorbent in pesticide analysis. These novel materials possess the advantages of both PTh polymer and nanofiller clay supports and represents to be alternative surfaces for separation for improving the adsorption activity of the analyte. For this purpose, PTh composite film was directly electrodeposited on the surface of a stainless-steel wire electrode from a 0.1 M NaClO₄ solution containing 0.3 M thiophene including clay in acetonitrile by using cyclic voltammetry. The surface structure of modified MMt-PTh-SPME fiber was characterized by Scanning Electron Microscopy (SEM). Operational parameters effecting the extraction, mainly, pH, sample volume, adsorption temperature and time, desorption temperature, stirring rate and salt amount were optimized. The regression coefficients of two pesticides relating to linearity were at least 0.99. For the analysis of apple juice samples 6 mL of wine was placed in a headspace vial, SPME fibre suspended in the headspace above the sample and after deposition analyte was desorbed in the inlet at 250°C. Calibration curves were linear in a range of 0.05 – 0.25 ng/mL with the detection limit of 0.009 ng/mL. The recovery value was calculated as 99% for 0.15 ng/mL concentration. The method was validated with spiked matrix samples. The advantages of the method developed are the less solvent consumption, being cost effective and fast.

KEYWORDS: Pesticide, Gas chromatography, Chlorpyrifos, SPME, Thiophene

REFERENCES: