DISPERSIVE MICRO-SOLID PHASE EXTRACTION USING SILICA BASED SOL-GEL HYBRID ORGANIC-INORGANIC MATERIAL FOR ANALYSIS OF ORGANOPHOSPHORUS PESTICIDES IN WATER SAMPLES

Wan Aini Wan Ibrahim*[1,2] and Norfazilah Muhamad[1]

[1] Separation Science and Technology Group (SepSTec), Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[2]Nanotechnology Research Alliance, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
(E-mail: wanaini@kimia.fs.utm.my, norfazilah2412@gmail.com)

INTRODUCTION

In 1930s, the insecticidal properties of organophosphorus pesticides (OPPs) and carbamate compounds were found and the compounds were developed for pesticides use in 1940s. They have been extensively used since 1970s when the environmentally persistent organochlorine pesticides were banned for use in the United States [1].

Although OPPs compounds are considered less dangerous than organochlorine, they are still highly neurotoxic to human and in some cases their degradation products have the potential to be more toxic with chronic exposure. The common method used for the analysis of pesticides is solid phase extraction (SPE) since it is more rapid, simple and economical than traditional liquid-liquid extraction. However, this method still has tendency to produce large secondary wastes, solvent loss, a long procedure and need for complex equipment [2,3]. Therefore to overcome this limitation, a quick, easy, cheap, rapid and simple (QuECheRS) microextraction method named dispersive micro-solid phase extraction (D-µ-SPE) has been developed. The developed method used synthesized silica based sol-gel hybrid cyanopropyltriethoxysilane-methyltrimethoxysilane (CNPrTEOS-MTMOS) as sorbent for the analysis of selected pesticides namely methamidophos, monocrotophos and chlorpyrifos in water sample.

The parameters affecting the extraction efficiency of the selected analytes were studied and extracted analyte were analyzed using gas chromatography-mass spectrometry (GC-MS). The optimum extraction conditions involved the use of 100 mg CNPrTEOS-MTMOS as adsorbent, 10 mL of water samples, 7 min of extraction time, 300 µL of methanol as desorption solvent and 3 min of desorption time. The adsorbent was collected on a filter paper and transferred to a micro-vial before been dried under a gentle stream of nitrogen. The extract was desorbed and the resulting aliquot was filtered through 0.45 µm membrane filter before subjected to GC-MS.

MAIN RESULTS

Under the optimum conditions, the method showed excellent detection limits and linear ranges were achieved which are 0.03, 0.05-10 µg L^{-1} for methamidophos, 0.02, 0.05-10 µg L^{-1} for monocrotophos and 0.004, 0.01-10 µg L^{-1} for chlorpyrifos, respectively. The sol-gel hybrid CNPrTEOS-MTMOS sorbent showed superior extraction performance (based on peak area obtained) compared to commercial C18 sorbent as shown in Figure 1. The CNPrTEOS-MTMOS sorbent offers an alternative sorbent material for the extraction of Opps of wide polarity. The developed method was applied for the analysis of OPPs in water samples and showed potential as an alternative microextraction technique.

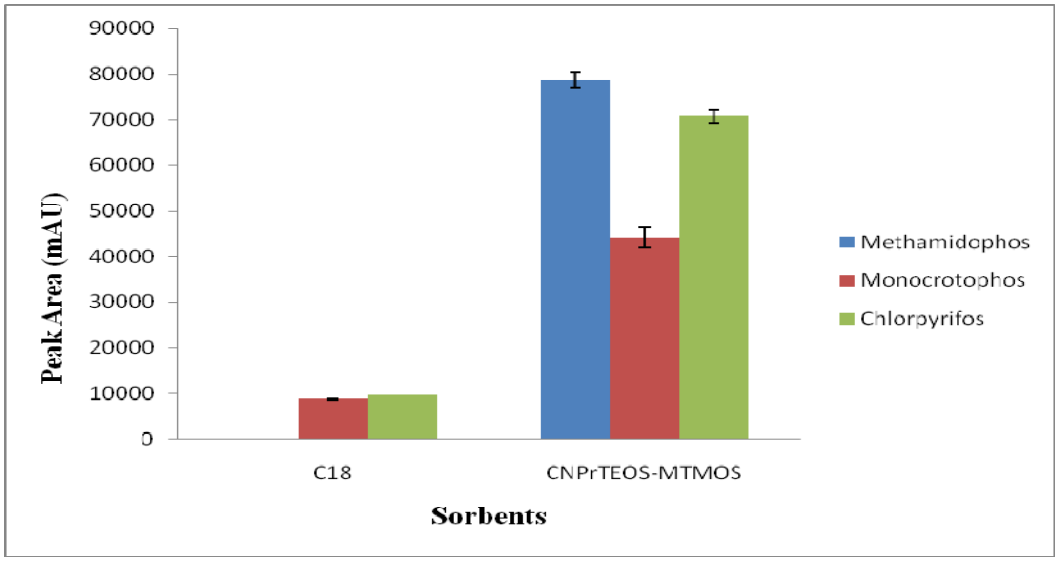

Figure 1: Comparison of extraction efficiency of the in-house silica based sol-gel hybrid CNPrTEOS-MTMOS as D-µ-SPE adsorbent material and commercial C18 sorbents towards three selected OPPs.

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