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A GREEN MICROEXTRACTION TECHNIQUE USING NANOMATERIALS ADSORBENT AS VALUABLE TOOLS FOR ELEMENTAL SPECIATION AND PHARMACEUTICAL CONTAMINANTS ANALYSIS

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ABSTRACT

Recently, substantial great innovations in sample preparation techniques have been focused by many researchers to miniaturize analytical laboratories procedure toward green microextraction techniques. Due to the polluting nature of the conventional sample preparation techniques, micro solid-phase extraction (μ-SPE) was developed as a greener alternative technique. This technique eliminates the lengthy workflow such as precipitation, centrifugation, and filtration which increase the sample preparation time and possible error prior to the instrumental analysis. Various nanomaterial adsorbents such as graphene, carbon nanotubes (CNTs), graphene oxide (GO), alginate graphene oxide (Alg/GO), alginate/CNTs, C_{18} have been developed and utilized as adsorbent in micro solid phase extraction for enhancing the extraction efficiency of elemental speciation and pharmaceutical contaminants in various matrices. Performance microextraction was evaluated by selected parameters; sample pH, ionic strength, amount of adsorbent, extraction time and desorption conditions. As a result, this technique offers simple approach, rapid analysis and economical value with superior extraction performance efficiencies with regard to sensitivity, selectivity and recovery. It could also be potentially useful for the analysis of elemental speciation and pharmaceutical contaminants analyses.

Keywords: microextraction technique, adsorbent, nanomaterials, pharmaceuticals

1. INTRODUCTION

In recent years, analytical chemists have been concerned about the future of the environment and the harmful ecological adverse effects of continuing to drown the earth with dangerous materials in the chemical process [1]. It is therefore critical to design green analytical processes with sustainable development of applications to remove environmental degradation and pollution. Among the various techniques of sample preparation, micro solid phase extraction (μ-SPE) was the most recent green analysis to replace SPE and LLE [2]. The good choice of a sorbent is the main role on the efficiency of μ-SPE method. Nanomaterial adsorbents such as carbon nanotubes (CNTs) [3] and graphene oxide (GO) [4] have been received great attention on enhancing the extraction efficiency of pharmaceuticals contaminants.

2. MATERIAL AND METHOD

Carboxylated multi-walled carbon nanotubes (MWCNTs) with specific surface area > 233 m2g1, purity >95 %, 8-15 nm outer diameter × 50 μm in length were purchased from Sun Nanotech (Jiangxi, China). Graphene oxide and C18 powder were purchase from Sigma-Aldrich (USA). Sodium alginate and calcium chloride were obtained from Qrec (New Zealand) and HmbG Chemicals (Germany). Reagent grade methanol, ethanol, 2-propanol, acetone and acetonitrile were obtained from Sigma-Aldrich (USA). HPLC grade acetonitrile (ACN) was obtained from Merck (Darmstadt, Germany). All standard of non-steroidal anti-inflammatory drugs (NSAIDs) and tetracycline antibiotics (TCAs) were purchase from Sigma-Aldrich (USA). For the μ-SPE method, approximately 0.3 g of nanomaterial adsorbents (beads/powder) were dispersed into 10 mL aqueous sample (at pH 3) in 50 mL centrifuge tube. The mixture was vigorously stirred with magnetic stirrer for minutes, to trap the analytes. Subsequently, the adsorbent was isolated from the solution by centrifugation at 4000 r min^{-1} speed for 5 min and the supernatant was discarded. About 2 mL of desorption solvent which is liquid chromatography grade acetonitrile was added to the centrifuge tube and sonicated for another 15 min. The mixture was then centrifuged at 4000 r min^{-1} speed, for another 5 min. The solvent was collected and evaporated to 1 mL under a gentle stream of nitrogen gas. About 1 mL of the extracted analyte was transferred into 1 mL amber glass vial. Finally, 10 μL of the extract was injected into the HPLC system.

3. RESULTS AND DISCUSSION

In this study, the performance of the prepared sorbent has been successfully applied for the extraction of pharmaceutical drugs using μ-SPE method combined with HPLC-UV for chromatographic analysis. Several parameters affecting this method were optimized. The optimum conditions were as follows: pH of the solution at pH 3, 30 min for the extraction time, 15 min for desorption time and 0.3 g for the mass of sorbent. Good linearities were achieved for the analytes with R^2, in the range of 0.9959 - 0.9996. The method was successfully applied for the analysis of river and tap water samples, with good relative recoveries in the range of 75 - 105 %.

Table 1. Tabulated data for method validation and real sample analysis for the nanomaterial adsorbents

Adsorbent	Analyte/ Element	Validation parameters				Real sample analysis		Ref
		Linear Range (μg L^{-1})	Coefficient of determination, R^2	LOD (μg L^{-1})	RSD (%) (*n* = 3)	Recoveries (%)	RSD (%) (n = 3)	
Alginate-Carbon Nanotubes (Alg-CNTs)	NSAIDs	1-500	< 0.9996	0.03 - 0.08	< 7.5	75 -105	< 7.3	[2]
Magnetic Graphene Oxide (GO/Fe_3O_4)	TCAs	50-1000	< 0.9992	0.006-0.011	< 5.6	89-103	< 6.6	[3]
Magnetic-GO (GO/Fe_3O_4)	REEs	10-1000	< 0.9992	0.04 - 0.12	< 4.21	90 -102	< 5.4	-

4. CONCLUSION

As a conclusion, these nanomaterial adsorbents were proven to be suitable alternative trapping media in solid phase extraction for pharmaceutical contaminants analysis and the proposed microextraction technique provide a more selective, cheaper, easy, quick and environmentally friendly compared to the classical SPE.

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