UNIVERSITI TEKNOLOGI MARA

DISPERSIVE MICRO-SOLID PHASE EXTRACTION USING ALGINATE GRAPHENE OXIDE FOR THE DETERMINATION OF SELECTED NON-STEROIDAL ANTI-INFLAMMATORY DRUGS FROM WATER SAMPLES

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ABSTRACT

The aim of this study was to synthesize alginate incorporated graphene oxide (Alg/GO) for the extraction of selected non-steroidal anti-inflammatory drugs (NSAIDs) using dispersive micro-solid phase extraction (D-μ-SPE) technique. The effects of GO and Alg amounts on the performance of the composite beads were optimized. The sorbent Alg/GO (1.0:0.3 w/v %) was characterized using scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and Brunauer- Emmett-Teller (BET). FTIR results of Alg/GO beads showed intermolecular hydrogen bonds, π-π stacking and electrostatic interactions within the Alg/GO matrix. BET linear plot of adsorption/desorption isotherms showed that Alg/GO relates to the type 1V mesoporous materials with a hysteresis loop of type H_3. SEM analysis of Alg/GO beads revealed homogenous pores on the relatively rough surface of the sorbent. The synthesized material was utilized as a sorbent in D-μ-SPE for extraction of three targeted analytes (ibuprofen, naproxen, and diclofenac) from water samples prior to analysis by high-performance liquid chromatography-ultraviolet detector (HPLC-UV). Furthermore, eight most effective parameters of D-μ-SPE technique were optimized using the prepared sorbent beads. The obtained optimum parameters are 0.15 g of mass of sorbent, sample pH 3,1000 rpm stirring speed, 30 min extraction time, 0 % (w/v) salt addition, isopropyl alcohol (IPA) as desorption solvent, 300 μL IPA as desorption solvent volume and 10 min desorption time. Under the optimized conditions, good linearity ($r^2 \geq 0.9993$) over a concentration range 10-1000 μg L^{-1} and detection limits between 3.5 - 4.8 μg L^{-1} with a relative standard deviation of ≤ 6 % were obtained. The proposed method also provided acceptable relative recoveries in the range of 95.6 -102.2 %. Alg/GO is found to be a potentially good alternative sorbent for the monitoring of pharmaceuticals in water samples using D-μ-SPE technique.

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TABLE OF CONTENTS

CHAPTER ONE
INTRODUCTION

1.1 Background of Research

Pharmaceuticals refer to any kind of products which can be used for medical treatment of humans and animals [1]. Pharmaceuticals are becoming omnipresent in the environment due to their wide-ranging usage in various areas such as medicine, industry, aquaculture, livestock farming and domestic needs. Nevertheless, pharmaceutical wastes have been identified as one of the sources of emerging contaminants and has become a serious scientific and public concern in aquatic environments [2].

Pharmaceutical remains are constantly discharged into shallow waters, adversely affecting and endangering the lives of aquatic organisms. [2]. Sources of water pollution include direct streaming or discharge of agricultural, poultry, fishery, wastewaters and sewage from houses, dormitories or any other public places into rivers, lakes and coastal waters [3]. The presence of pharmaceutical waste in environmental waters, especially those that contaminate drinking water, should be considered as an important issue in terms of human safety and health. It is known that pharmaceutical waste can create health problems [3]. Thus, an efficient, rapid and reliable analysis is needed for the determination of pharmaceutical waste at low concentration levels in different environmental matrices in order to ensure public safety and health.

Sample preparation is a crucial step in analytical chemistry, especially in pharmaceutical, wastewater and biological analysis [4]. Various sample preparation techniques have been used in the analysis of drugs and wastewaters. Conventional extraction techniques such as liquid-liquid extraction (LLE) [5] and solid phase extraction (SPE) [6] have been used in the analysis of pharmaceutical compounds [7]. However, these extraction techniques are time consuming and require large volumes of organic solvents [4]. Micro-solid phase extraction (MSPE) techniques are currently the most promising sample preparation techniques. These techniques offer several advantages such as the minimum use of solvents, low cost, simple and excellent