

UNIVERSITI TEKNOLOGI MARA

**ALGINATE INCORPORATED
MULTI-WALLED CARBON
NANOTUBE AS NEW SORBENT FOR
SELECTIVE EXTRACTION OF NON-
STEROIDAL
ANTI-INFLAMMATORY DRUGS**

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ABSTRACT

Alginate incorporated multi-walled carbon nanotubes (Alg-MWCNT) was utilized as sorbent in dispersive micro solid phase extraction (D- μ -SPE) and online solid phase extraction (online-SPE) for the extraction of non-steroidal anti-inflammatory drugs (NSAIDs) in water samples prior to high performance liquid chromatography (HPLC) analysis. Selected commonly used NSAIDs namely salicylic acid (SAL), naproxen (NAP), diclofenac (DIC), ibuprofen (IBU) and mefenamic acid (MEF) were used as target analytes. Alg-MWCNT sorbent was prepared and characterized by Fourier Transform Infrared (FTIR) Spectroscopy, Field Emission Electron Microscope (FESEM) and Brunauer-Emmet-Teller (BET) analyzer. Several parameters were optimized in D- μ -SPE method. The optimum conditions were as follows: pH of the solution at pH 3, 30 min for the extraction time, 15 min for the desorption time and 0.3g for the mass of sorbent. All the five NSAIDs were successfully extracted using the same conditions in D- μ -SPE. Good linearities were achieved for the analytes with coefficients of determination, R^2 in the range of 0.9959 - 0.9996. The method was successfully applied for the analysis of river water and tap water samples, with good relative recoveries in the range of 75 - 105 %. The prepared sorbent was applied for the extraction of NSAIDs by online solid phase extraction liquid chromatography (online SPE-LC). Several important online SPE parameters such as valve switching time, solvent composition and buffer pH were optimized using Box-Behnken Design of Response Surface Methodology (RSM). Analysis of variance (ANOVA), revealed that the regression was statistically significant with second order equation. Under the optimized conditions (valve switching time: 1.5 min, composition of acetonitrile:MSA, 60:40 and buffer pH: pH 2), Alg-MWCNT-online-SPE-LC provided good linearity in the concentration range of 1 to 500 $\mu\text{g L}^{-1}$ with R^2 of 0.9971 - 0.9996 and low limits of detection $\leq 0.018 \mu\text{g L}^{-1}$. The method showed high relative recoveries in the range of 75 – 110 % for river water and tap water samples, respectively with RSDs of ≤ 7.8 (n = 3). The developed method showed a good tolerance to aqueous sample matrices. Based on the results, online SPE-LC method by using Alg-MWCNT as sorbent proved to be a rapid, selective and efficient technique for the extraction and separation of acidic drugs in aqueous matrices.

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