

A Dispersive Micro-Solid Phase Extraction Using Ionic Liquid – Modified Graphene Oxide Sorbent for Pre –Concentration and Extraction of Acid Drug From Water Sample Using Liquid Chromatography

Siti Nor Ziana Mat Zain^a, Nor Suhaila Mohamad Hanapi^{a,b*}

Structured Abstract

Background: Pharmaceutical waste has recently been identified as a significant cause of novel toxins and a grave ecological health concern. Dispersive micro-solid phase extraction (D- μ -SPE) is a miniaturized method that adheres to the same principle as the solid phase extraction (SPE) approach, based on the dispersion of miniature or milligram level of sorbent within a solution. The advancement of dispersive micro-solid phase extraction (D- μ -SPE) has offered a novel method that is straightforward, quick, and effective. Nowadays, graphene oxide (GO), combined with ionic liquid, has found applications as sorbents for diverse purposes.

Methods: Approximately 0.01g of sorbent was dispersed into a 10 mL aqueous sample in a 50 mL centrifuged tube. The mixture was vortex for 2 minutes to trap the analytes. Afterwards, the adsorbent was isolated from the solution by centrifugation at 9000rmin⁻¹ speed for 6 min, and the supernatant was discarded. About 1 mL of a desorption solvent, which is liquid chromatography grade isopropyl alcohol, was added to the centrifuge tube and vortex for another 2 min. The mixture was then centrifuged at 9000rmin⁻¹ speed for another 6 min. The solvent was collected and evaporated into 0.5 mL under a gentle stream of nitrogen gas. About 0.5 mL of the extracted analyte was transferred into a 1 mL amber glass vial. Lastly, 5 μ L of the extract was injected into the HPLC system.

Results: This study shows that the performance of the prepared sorbent has been successfully applied for the extraction of acidic drugs using the D- μ -SPE method combined with HPLC-DAD for chromatographic analysis. Several parameters affecting this method were optimized. The optimum conditions were as follows: 6 min for desorption time, IPA for the desorption solvent and 0.01g for the mass of the sorbent. Good linearities were achieved for the analytes with r² of 0.9971. The relative recoveries demonstrate an 82% yield with acceptable reproducibility, expressed as a percentage relative standard deviation (RSD) of 3.20%.

Conclusion: In conclusion, this sorbent has been demonstrated as a viable alternative for trapping pharmaceutical contaminants in solid-phase extraction. The suggested dispersive micro-extraction technique offers a more cost-effective, convenient, and environmentally friendly option compared to traditional SPE methods.

Keywords: Microextraction technique, pharmaceuticals, graphene oxide, ionic liquids.

*Correspondence: norsuhaila979@uitm.edu.my

^aSchool of Chemistry & Environment, Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, Malaysia