UNIVERSITI TEKNOLOGI MARA

FACILE SYNTHESIS OF REDUCED GRAPHENE OXIDE/Fe_3O_4 NANOCOMPOSITE FOR PRECONCENTRATION AND TRACE DETERMINATION OF TETRACYCLINES AND RARE EARTH ELEMENTS IN WATER

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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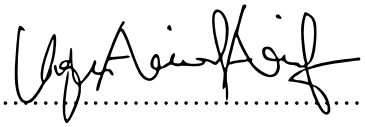
Signature of Student	:	
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ABSTRACT

A green magnetic adsorbent, reduced graphene oxide/Fe_3O_4 nanocomposite, was synthesized *via* one-pot reaction method and characterized *via* X-Ray Diffraction, Field Emission Scanning Electron Microscope, Fourier-transform infrared spectroscopy, Energy Dispersive X-ray spectroscopy, Point of Zero Charge and Vibrating-sample magnetometer. The adsorbent was used to develop magnetic solid-phase extraction (MSPE) technique for tetracycline antibiotics (TCAs) and rare earth elements (REEs) from water samples. The MSPE procedure for TCAs was optimised with respect to the necessary parameters; pH 4 for sample solution, 10 mg of adsorbent, 8 mL of sample, 5 min of extraction time, ethanol as the eluent and 2 min of desorption time. Under the optimised MSPE conditions, good linearity was achieved for the selected TCAs at concentrations ranging 0.05–1.0 mg L^{-1}. The limit of detection and limit of quantification were 0.006–0.011 mg L^{-1} and 0.019–0.036 mg L^{-1}, respectively. The intra-day and inter-day precisions were less than 3.38% and 5.59%, respectively. Satisfactory recoveries were obtained from real sample analysis ranging from 89.77–106.33%. Meanwhile, the MSPE procedure for REEs was optimised with respect to the necessary parameters; sample solution at pH 4, 20 mg of adsorbent, 100 mL of sample, 5 min of extraction time, 0.5 M of HNO_3 as the eluent and 3 min of desorption time. The method also revealed good linearity for the selected REEs, ranging from 10–1000 µg L^{-1}. The limit of detection and limit of quantification were obtained in the range of 0.044–0.115 µg L^{-1} and 0.147–0.389 µg L^{-1}, respectively. The intra-day and inter-day precisions were less than 3.87% and 4.21%, respectively. Satisfactory recoveries were obtained from real sample analysis ranging from 90.19–102.43%. Both analytes obeyed Langmuir adsorption isotherm model with adsorption capacity up to 52.64 and 53.56 mg g^{-1}, respectively. The adsorbent can be reused up to 10 cycles without a significant loss of performance.

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"Without your past, you could never have arrived—so wondrously and brutally, by design or some violent, exquisite happenstance…here."

—Why She Disappeared, T.S.

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