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Roya Mirzajani, Fatemeh Kardani, Zahra Ramezani

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## ACCEPTED MANUSCRIPT

Preparation and characterization of magnetic metal–organic framework nanocomposite as solid-phase microextraction fibers coupled with high-performance liquid chromatography for determination of non-steroidal anti-inflammatory drugs in biological fluids and tablet formulation samples

## Roya Mirzajani<sup>a</sup>\*, Fatemeh Kardani<sup>a</sup>, Zahra Ramezani<sup>b</sup>

<sup>a</sup> Chemistry Department, College of Science, Shahid Chamran University of Ahvaz, Ahvaz, Iran

<sup>b</sup> Department of Medicinal Chemistry, School of Pharmacy, Ahvaz Jundishapur University of Medical

Sciences, Ahvaz, Iran

A novel solid-phase microextraction (SPME) fiber based on a capillary glass tube coated with magnetic Fe<sub>3</sub>O<sub>4</sub>/Cu<sub>3</sub>(BTC)<sub>2</sub> metal organic frameworks nanocomposite was prepared by solgel technique. The magnetic Fe<sub>3</sub>O<sub>4</sub>/Cu<sub>3</sub>(BTC)<sub>2</sub> metal organic frameworks nanocomposite were synthesized by a simple hydrothermal reaction and the resultant powder was mixed with sol-gel precursors to prepare sol-gel solution of the magnetic Fe<sub>3</sub>O<sub>4</sub>/Cu<sub>3</sub>(BTC)<sub>2</sub> coating material. In this study, glass tubes with a specific diameter were used as substrates. The magnetic Fe<sub>3</sub>O<sub>4</sub>/Cu<sub>3</sub>(BTC)<sub>2</sub> MOF nanocomposites coating was characterized using Fourier transform infrared (FTIR) spectroscopy, powder x-ray diffraction (XRD) and scanning electron microscopy (SEM). Then, the synthesized fiber as novel solid-phase microextraction (SPME) fiber combined with high-performance liquid chromatography (SPME-HPLC) was applied for the determination and quantification of non-steroidal anti-inflammatory drugs (NSAIDs) (ibuprofen, diclofenac, naproxen and nalidixic acid) in real samples including human urine, serum, plasma, and tablet formulation. To found optimum microextraction conditions, the influences of effective variables were investigated using one-factor-at-a-time experiments and the significant variables were optimized using a Box-Behnken design (BBD) combined with desirability function. Under optimized conditions, calibration graphs of analytes were linear in a concentration range of 0.1-400  $\mu$ g L<sup>-1</sup> with correlation coefficients more than 0.9966. Limits of detection and quantification were in the ranges of 0.03-0.0 5 µg  $L^{-1}$  and 0.12-0.18 µg  $L^{-1}$ , respectively. This procedure was successfully employed in

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