



Microwave assisted solid phase extraction for separation preconcentration sulfamethoxazole in wastewater using tyre based activated carbon as solid phase material prior to spectrophotometric determination



K. Mogolodi Dimpe, Anele Mpupa, Philiswa N. Nomngongo *

Department of Applied Chemistry, University of Johannesburg, Doornfontein Campus, P.O. Box 17011, Johannesburg 2028, South Africa

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ABSTRACT

This work was chiefly encouraged by the continuous consumption of antibiotics which eventually pose harmful effects on animals and human beings when present in water systems. In this study, the activated carbon (AC) was used as a solid phase material for the removal of sulfamethoxazole (SMX) in wastewater samples. The microwave assisted solid phase extraction (MASPE) as a sample extraction method was employed to better extract SMX in water samples and finally the analysis of SMX was done by the UV–Vis spectrophotometer. The microwave assisted solid phase extraction method was optimized using a two-level fractional factorial design by evaluating parameters such as pH, mass of adsorbent (MA), extraction time (ET), eluent ratio (ER) and microwave power (MP). Under optimized conditions, the limit of detection (LOD) and limit of quantification (LOQ) were $0.5 \mu\text{g L}^{-1}$ and $1.7 \mu\text{g L}^{-1}$, respectively, and intraday and interday precision expressed in terms of relative standard deviation were $>6\%$. The maximum adsorption capacity was 138 mg g^{-1} for SMX and the adsorbent could be reused eight times. Lastly, the MASPE method was applied for the removal of SMX in wastewater samples collected from a domestic wastewater treatment plant (WWTP) and river water.

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1. Introduction

Antibiotic residues in the environmental matrices have recently gained extensive attention due to their large-scale consumption, wide occurrence, and potential risks for wildlife and human [1,2]. In addition, it has been reported that antibiotics possess high mobility depending on their physicochemical properties such as water solubility and acid dissociation constant [1]. Therefore, high concentrations of antibiotics have been detected in river water [3]. Among others, sulfonamides are a group of antibiotics that are largely used to treat bacterial infections, respiratory infections, urinary and gastrointestinal infection, and as growth promoters in mammalian and avian species [4–6]. These antibiotics are known to be highly stable in the aquatic environment [2]. Among the sulfonamides, the most commonly used antibiotics, is sulfamethoxazole (SMX) and is known to be difficult to be hydrolyzed and biodegraded. The persistence nature of SMX in the environment has been identified to exist in the wastewater coming from the wastewater treatment plant (WWTP). This is because about 85% of ingested SMX molecules are metabolized in the human body and thus in urine and

faeces only 15% of the parent compound remains unaltered [6]. Studies have been conducted to prove that WWTPs cannot completely remove antibiotics such as SMX, hence such pollutants have been discovered to infiltrate water systems [2,7,8]. Therefore, exposure to such a pollutant may lead to adverse effects to humans and animals such as liver cancer, hemopoiesis turbulence, bacterial phytoplankton and zooplankton [4, 6].

Due to the potential risks associated with the presence of trace levels of SMX and their complexity of the matrices in water bodies, it is critical to establish analytical techniques that are capable of determining SMX in wastewater. Techniques such as high performance liquid chromatography (HPLC) [3], UV–Vis spectrophotometry [9–11], and electrochemical methods [1] have been widely used for determination of pollutants in environmental matrices. Some of these methods are not easy to work with because of the complexity and the expensiveness of the techniques. On the contrary, spectrophotometric technique such as UV–Vis spectrophotometry is regarded as a very simple and cost-effective technique for the analysis of pharmaceutical drugs [12]. Over the years, UV–Vis has gained tremendous attention due to its appealing approach for the qualitative and quantitative analysis of sulfamethoxazole in liquid samples [13]. However, UV–Vis spectrophotometer presents some disadvantages such as its low selectivity and sensitivity. However, with

* Corresponding author.

E-mail address: pnnomngongo@uj.ac.za (P.N. Nomngongo).

the employment of relevant sample extraction methods; this technique can overcome the abovementioned drawbacks [10–12]. Thus, the use of extraction methods will assist in resolving the problem of complexed matrices and very low levels of the analyte of interest, which usually results in the limitation of the use of UV–Vis spectroscopy [9].

Sample pretreatment methods such as pressurized liquid extraction (PLE), solid phase extraction (SPE), solid phase microextraction (SPME), microwave assisted extraction (MAE), liquid-liquid extraction (LLE), among others, have been explored for the extraction of emerging pollutants in solid and liquid samples [8,14–18]. Some of these extraction methods have displayed disadvantages such as the usage of large volumes of solvents, time consumption and the utilization of expensive equipment [17]. However, among the above mentioned extraction methods, the MAE has advantages such as saving of time and the usage of smaller volumes of solvents [2]. MAE resembles green chemistry in many ways including reduced waste generation, reduced sample amount required and simultaneous extraction of many samples at a time. The attractiveness of MAE method is its unique abilities of heating the sample directly with a desired speed as compared to the conventional way of heating a sample [18]. The extraction of organic pollutants by adsorbents such as activated carbon using the microwave system, provides minimal parameters to optimize that are influential in assessing the effect of the adsorption process [19]. Furthermore, the use of microwave involves the use of electromagnetic energy for the supply of an adequate amount of energy to transfer the adsorbate from the liquid phase to an adsorbent [20]. The implementation of MAE is easy, rapid and provides good extraction efficiencies when compared to classical techniques that are carried at ambient temperature [19].

Usually the extraction of organic pollutants is complimented by the employment of an adsorbent as a solid phase to remove them from environmental samples. Therefore, the adoption of well-established and efficient adsorbent such as activated carbon sourced from various precursors including coconut shells, wood chips, sawdust, corn cobs, coffee husks, and waste tyres as one of waste material; among others [4] is a growing need for the removal of pollutants in the environment. Activated carbon obtained from a waste tyre has an adsorption capacity that is attractive and is greatly influenced by pore volume, surface area and pore size distribution [21,22].

The objectives of this study was to developed rapid SPE using activated carbon as an adsorbent coupled with MAE (MASPE) for extraction and preconcentration of SMX in wastewater (influent, secondary and effluent) and river samples prior to the UV–Vis spectrophotometric detection. The MAE was chosen to facilitate the transfer of the analyte from the sample solution to the extracting solid phase material. This was achieved by heating the sample under microwave energy for a short period of time. In addition, the MAE was used to extracts the SMX that is associated with small particulate that form part of the wastewater matrix. Furthermore, the AC produced from the waste tyre as a precursor was chosen as a solid phase material due to its high adsorption capacity and affinity for organic pollutants. In addition, AC was chosen because it can be produced from low cost or waste material. Therefore, in this study, the advantages of SPE and MAE were combined to get better extraction and preconcentration efficiency at short analysis time. The optimization of MASPE procedure was carried out using the two-level fractional factorial design. Furthermore, the accuracy of the method was validated by using the spike recovery tests.

2. Experimental

2.1. Instrumentation

The quantification of SMX was performed using the Shimadzu (UV-2450) high performance single monochromator UV–Vis spectrophotometer. The Branson 5800 ultrasonic Cleaner (Danbury, CT, USA) was used for adsorption studies. The pH of wastewater sample and model

solution was measured using the pH meter with electronic glass electrode (Mettler-Toledo FE20, Switzerland). Eppendorf 5702 Centrifuge (Eppendorf, Hamburg Germany), was used to separate the adsorbent from the sample solution.

The tubular electric furnace was used to perform the pyrolytic process under inert conditions using nitrogen. The Microwave Reaction SystemSOLV (Multiwave PRO, Anton Paar), was used as a heating source for the activation of the carbon powder in this study. A drying oven purchased from Xi'an Unique Electronics, UQ 9053A was used to dry all wet glassware adsorbent. The morphology of the adsorbent was observed using scanning electron microscope/energy dispersive x-ray spectroscopy (SEM/EDS) (VEGAS-TESCAN, USA). The specific surface area value was determined from adsorption isotherms by the Brunauer-Emmett-Teller (BET) multipoint method using Surface Area and Porosity Analyzer (ASAP2020 V3. 00H, Micromeritics Instrument Corporation, Norcross, USA). All the gases used for analysis were of instrument grade. X-ray powder diffraction (XRD) measurements were carried out with a Philips X-ray generator model PW 3710/31 a diffractometer with automatic sample changer model PW 1775 (scintillation counter, Cu-target tube, and Ni-filter at 40 kV and 40 mA). The infrared spectrum was recorded using Spectrum 100 FT-IR (PerkinElmer, USA) spectrometer equipped with Universal Attenuated Total Reflectance (ATR).

2.2. Chemicals and Reagents

The chemicals that were used in this study were of analytical reagent grade. Double distilled water was used in all experiments. Hydrogen peroxide (H₂O₂ (30%)) was purchased, absolute methanol (99.9%), acetonitrile, ethanol and nitric acid (69%) were purchased from Sigma-Aldrich (Fluka, St. Louis, MO, USA). The pH values of the model solutions were adjusted with 1.0 mol L⁻¹ acetic acid and 1.0 mol L⁻¹ ammonium hydroxide. Validation of MASPE was performed by spiking sample solution with a SMX standard (Fig. 1, Sigma-Aldrich, South Africa), due to lack of an appropriate standard reference material. The glassware was washed, rinsed and immersed in 10% HNO₃ solution overnight and finally rinsed with deionised water throughout this experiment. Cellulose acetate membrane (Separations Scientific SA (Pty) Ltd) filters (0.45 μm) were used to separate the adsorbent material from model solutions and wastewater samples. The SMX standard solution of 10 mg L⁻¹ was used to obtain the calibration standard solutions for the instruments.

2.3. Sampling of Wastewater

Wastewater samples were collected from the Daspoort wastewater treatment plant (WWTP). This plant largely receives wastewater coming from households (domestic) and a 5% of wastewater was coming from various industries and storm wastewater. The plant is situated in the capital city of South Africa (Pretoria) and the Apies river receives the discharged wastewater.

Sampling points such as the primary stage, secondary treatment stage, tertiary stage and river water were chosen as samples of interest to assess the efficiency of the WWTP. Samples were collected and stored in the refrigerator (at 4 °C) up until they were used.

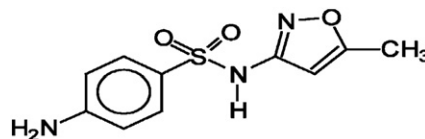


Fig. 1. The structure of SMX (molecular formula: C₁₀H₁₁N₃O₃S molar mass (g mol⁻¹): 253.3 pKa: 1.6/6.4 water solubility (mg L⁻¹): 610 K_{ow}: 7.8).

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