ORGANIC-INORGANIC SOL-GEL HYBRID AND MAGNETIC SPOROPOLLENIN-BASED COMPOSITES AS SOLID PHASE-BASED EXTRACTION OF SELECTED ALDEHYDES AND DRUGS ANALYSIS

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"……Act! Allah will behold your actions, and (so will) His messenger and the believers, and ye will be brought back to the Knower of the Invisible and the Visible, and He will tell you what ye used to do" (A Taubah: verse 105)

This thesis is dedicated to my beloved family.

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ABSTRACT

The effectiveness of microextraction method is strongly dependent on the selection of suitable sorbent material for the target analyte/s. Production of an efficient and easily handled sorbent material is challenging. The use of sorbent based on a single material often exhibits limited physicochemical properties. Thus, combining the advantages of different materials especially from renewable or less toxic sources is preferred to obtain an eco-friendly composite with enhanced performance. In this study, three organic-inorganic composites were successfully synthesized as new extraction materials for selected organic compounds from different aqueous samples. The first sorbent, methyltrimethoxysilane-(3-mercaptopropyl)trimethoxysilane (MTMOS-MPTMOS) was successfully used as coating material in hollow fiber-solid phase microextraction (HF-SPME) and applied to the extraction of hexanal and heptanal in urine samples. MTMOS-MPTMOS HF-SPME method showed good limits of detection (LOD) (3 S/N) (0.010 µg mL^{-1} for hexanal and 0.015 µg mL^{-1} for heptanal), excellent accuracies (recovery = 91.21 - 97.51%) and good precisions (RSD < 5.6%). The ease of sorbent preparation and simplicity in use made the MTMOS-MPTMOS HF-SPME as a potential tool for detecting volatile organic compounds in real samples. The second sorbent, magnetic sporopollenin-3-cyanopropyltriethoxysilane (MS-CNPrTEOS) was synthesized and successfully evaluated as a new sorbent candidate for use in dispersive micro-magnetic solid phase extraction (D-µ-MSPE) for the determination of selected non-steroidal anti-inflammatory drugs (NSAIDs) and tricyclic antidepressants (TCAs) in water samples. Magnetic sporopollenin composite functionalized calix[4]arene derivative (Calix-MS) was prepared as the third new sorbent and successfully employed for D-µ-MSPE of selected NSAIDs in water samples. The developed methods using both MS-CNPrTEOS and Calix-MS provided low LODs (3*s*) at ppb levels (< 0.50 µg L^{-1}), good linearities ($R^2 \geq 0.9951$) and accuracies (> 86%). Findings revealed that the bio-based sporopollenin composites exhibited good extraction capability, good stability (reusable) and sufficient magnetic property to greatly simplify the extraction process. Based on the analytical eco-scale, the greenness of MTMOS-MPTMOS HF-SPME method is excellent while D-µ-MSPE using MS-CNPrTEOS and Calix-MS as sorbents are classified as acceptable green analytical methods. The three developed methods showed high potential as green alternative methods for determination of organic compounds in aqueous samples.

ABSTRAK

Keberkesanan kaedah pengekstrakan mikro amat bergantung kepada pemilihan bahan pengerap untuk analit sasaran. Penghasilan bahan pengerap yang cekap dan mudah dikendalikan adalah agak mencabar. Penggunaan bahan pengerap berasaskan bahan tunggal kebiasaannya mempamerkan sifat fizikokimia yang terhad. Oleh itu, kelebihan penggabungan bahan berbeza terutamanya daripada sumber yang boleh diperbaharui atau kurang toksik lebih digemari untuk menghasilkan komposit mesra alam dengan peningkatan prestasi. Dalam kajian ini, tiga komposit organik-tak organik telah berjaya disintesis sebagai bahan pengekstrakan baharu untuk sebatian organik terpilih daripada sampel akueus berbeza. Bahan pengerap pertama, metiltrimetoksisilana-(3-merkaptopropil)trimetoksisilana (MTMOS-MPTMOS) telah berjaya digunakan sebagai bahan salut dalam pengekstrakan fasa pepejal-gentian berongga (HF-SPME) dan diaplikasikan untuk pengekstrakan heksanal dan heptanal di dalam sampel air kencing. Kaedah MTMOS-MPTMOS HF-SPME telah menunjukkan had pengesanan yang baik (LOD) (3 S/N) (0.010 μg mL^{-1} untuk heksanal dan 0.015 μg mL^{-1} untuk heptanal), ketepatan cemerlang (pengembalian semula = 91.21 - 97.51%) dan kepersisan yang baik (RSD < 5.6%). Penyediaan bahan pengerap yang mudah dan senang digunakan menjadikan MTMOS-MPTMOS HF-SPME suatu peranti yang berpotensi untuk mengesan sebatian organik mudah meruap di dalam sampel sebenar. Bahan pengerap kedua, sporopollenin-sianopropiltrietoksisilana bermagnet (MS-CNPrTEOS) telah disintesis dan diuji dengan jayanya sebagai bahan pengerap baharu dalam pengekstrakan mikro fasa pepejal bermagnet terserak (D-μ-MSPE) untuk penentuan dadah anti-keradangan bukan steroid (NSAIDs) dan dadah anti-kemurungan trisiklik (TCAs) terpilih di dalam sampel akueus. Komposit sporopollenin bermagnet terfungsi terbitan kaliks[4]arena (Calix-MS) telah disediakan sebagai bahan pengerap baharu ketiga dan telah berjaya digunakan untuk D-μ-MSPE bagi NSAIDs terpilih di dalam sampel air. Kaedah yang dibangunkan menggunakan kedua-dua bahan pengerap MS-CNPrTEOS dan Calix-MS telah menghasilkan LOD (3*s*) yang rendah pada tahap ppb (< 0.50 μg L^{-1}) dan kelinearan baik ($R^2 \geq 0.9951$) serta kejituan (> 86%) yang baik. Penemuan menunjukkan bahawa komposit sporopollenin berasaskan-bio mempamerkan keupayaan pengekstrakan yang baik, kestabilan yang baik (kebolehgunaan semula) dan sifat bermagnet yang mencukupi untuk mempermudahkan proses pengekstrakan. Berdasarkan analisis skala-eko, tahap kehijauan kaedah MTMOS-MPTMOS HF-SPME adalah cemerlang manakala kaedah D-μ-MSPE menggunakan MS-CNPrTEOS dan Calix-MS sebagai bahan pengerap dikategorikan sebagai kaedah analisis hijau yang memuaskan. Ketiga-tiga kaedah dibangunkan menunjukkan potensi tinggi sebagai kaedah alternatif hijau untuk penentuan sebatian organik di dalam sampel akueus.

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LIST OF ABBREVIATIONS

AES	-	Analytical eco-scale
ANOVA	-	Analysis of variance
ATR	-	Attenuated total reflectance
BAµE	-	Bar adsorptive microextraction
BBD	-	Box Behnken design
BSE	-	Back-scattered Electron
Calix-MS	-	Alkylbenzonitrile substituted *p-tert*-butylcalix[4]arene-immobilized magnetic sporopollenin
CAR/PDMS	-	Carboxen/polydimethylsiloxane
CCD	-	Central composite design
CME	-	Capillary microextraction
CNPrTEOS	-	Cyanopropyltriethoxysilane
DSPE	-	Dispersive solid-phase extraction
D-µ-SPE	-	Dispersive-micro-solid phase extraction
D-µ-MSPE	-	Dispersive-micro-magnetic solid phase extraction
EDX	-	Energy-dispersive X-ray
EPPTMS	-	3-(2,3-epoxypropoxy)propyltrimethoxysilane
FPSE	-	Fabric phase sorptive extraction
FTIR	-	Fourier transform infrared
GAC	-	Green analytical chemistry
GC	-	Gas chromatography
GC-FID	-	Gas chromatography with flame ionization detection
HF-SPME	-	Hollow fiber-solid phase microextraction
HPLC-UV	-	High performance liquid chromatography with ultraviolet detection
HS-SPME	-	Headspace-solid phase microextraction
LLE	-	Liquid-liquid extraction
LOD	-	Limit of detection
LOQ	-	Limit of quantification
MEPS	-	Microextraction by packed sorbent

MNPs	-	Magnetic nanoparticles
MPTMOS	-	Mercaptopropyltrimethoxysilane
MS	-	Magnetic sporopollenin
MSPE	-	Magnetic solid-phase extraction
MTMOS	-	Methyltrimethoxysilane
MWCNT	-	Multi-walled carbon nanotubes
NSAIDs	-	Non-steroidal anti-inflammatory drugs
NTD	-	Needle trap device
PDMS	-	Polydimethylsiloxane
PDMS/DVB	-	Polydimethylsiloxane/divinylbenzene
p*K*a	-	Acid dissociation constant
PMHS	-	Polymethylhydroxysiloxane
PMME	-	Polymer monolith microextraction
PSA	-	Primary and secondary amines
RDSE	-	Rotating disk sorptive extraction
RSD	-	Relative standard deviation
RSM	-	Response surface methodology
SBSE	-	Stir bar sorptive extraction
SDGs	-	Sustainable Development Goals
SEM	-	Scanning electron microscopy
SPE	-	Solid-phase extraction
SPME	-	Solid phase microextraction
TCAs	-	Tricyclic antidepressants
TEM	-	Transmission electron microscope
TEOS	-	Tetraethoxysilane
TFA	-	Trifluoroacetic acid
TGA	-	Thermogravimetric analysis
UPLC-PDA	-	Ultra-performance liquid chromatography with photodiode array detection
VSM	-	Vibrating sample magnetometer
VOCs	-	Volatile organic compounds

LIST OF SYMBOLS

AU	-	Absorbance unit
D, d	-	Diameter
emu	-	Electromagnetic unit
G	-	Gravity
L	-	Litre
mg	-	Milligram
mL	-	Millilitre
ng	-	Nanogram
ppb	-	Part per billion
R^2	-	Coefficient of determination
µ	-	Micro
µg	-	Microgram
µL	-	Microlitre

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Chromatographic analysis of organic compounds including endogenous volatiles and environmental persistence pharmaceutical pollutants are inevitably challenging due to the overwhelming matrix effects and the trace levels of the analytes in sample matrices. Direct chromatographic sample introduction is often avoided. Even though modern analytical instrumentation is utilized, sample preparation is still mandatory to remove the interferences as well as impurities to obtain clean extracts before instrumental separation and detection for analysis of the intended analyte/s. In laboratory practice, coupling liquid chromatography and solid phase extraction (SPE) is a typical technique to determine non-volatile organic compounds. For volatile organic compounds (VOCs) determination, gas chromatography (GC) is a convenient option and combination with a solid phase microextraction (SPME) method has been proven a perfect match. With time, a variety of sample preparation and preconcentration techniques have been developed and adopted for different classes of organic compounds prior to chromatographic analysis. Undeniably, the choice of sample preparation determines the reagents and solvent used, cost and time required as well as influences the quality of the analysis. Besides, appropriate selection of preconcentration approach is exceptionally an imperative way to improve method reliability and sensitivity for intended organic compound/s analysis.

Among broad groups of organic compounds, aldehydes are important volatile fingerprint that have been quantified in a variety of sample matrices such as biological samples (Wei *et al.*, 2019; Monedeiro *et al.*, 2020), foodstuffs (Liu *et al.*, 2020) as well as beverages (Bueno *et al.*, 2014; Moreira *et al.*, 2019). Aldehydes play a role in oxidative stress and several aliphatic aldehydes particularly hexanal and heptanal have received a spotlight in research studies as potential markers of a few diseases including

lung cancer and bladder cancer (Liu *et al.*, 2014; Yu *et al.*, 2018; Wei *et al.*, 2019). In addition, hexanal and heptanal are among volatile aldehydes that are commonly found as food processing by-products and their presence in foodstuffs could be a marker of food quality (Aisala *et al.*, 2019; Ghiasvand *et al*., 2019). Since they are useful volatile markers, the two aldehydes have been selected as model compounds in the current study.

Prior to analysis, SPME is a promising technique to determine volatile aldehydes in multiple samples (Buszewski *et al.*, 2012; Ghiasvand *et al*., 2018a; Filipowska *et al.*, 2020). SPME offers the advantage of one step extraction and preconcentration which is rapid for sampling. Commercial SPME using polydimethylsiloxane/divinylbenzene (PDMS/DVB) (Guadagni *et al.*, 2011; Monteiro *et al.*, 2014) and carboxen/polydimethylsiloxane (CAR/PDMS) fibers (Manousi and Zachariadis, 2019) are superior for aldehydes analysis. However, commercial SPME fiber suffers from low recommended operating temperature, limited lifetime and is relatively expensive. The commercially available fibers are not chemically stable due to the mere physical deposition or partial cross linking of the polymer coating. These limitations have been the driving force to emergence of diverse fiber coatings for SPME (Wan Ibrahim *et al.*, 2010; Aziz-Zanjani and Mehdinia, 2014; Ghiasvand *et al*., 2018b; Zhang *et al.*, 2018; Xiang *et al.*, 2020). Perpetual effort has been devoted to creating materials for effective SPME and sol-gel approach offers a convenient and simple pathway to produce sorbent materials with good properties and stability (Wan Ibrahim *et al.*, 2010; Sarafraz-Yazdi *et al.*, 2011; Pena-Pereira *et al.*, 2014; Ghader *et al.*, 2017; Kong *et al.*, 2019; Dowlatshah and Saraji, 2020; Xiang *et al.*, 2020).

Sol-gel technology represents a simple chemical route to synthesize multicomponent composites and different kinds of sol-gel derived materials are proven to be very useful for ultra-trace analysis (Wan Ibrahim *et al*., 2010; 2011a; 2013; Kabir *et al*., 2013; Abd Rahim *et al*., 2016; Kong *et al*., 2019; Fan *et al*., 2020). Typical sol-gel process involves hydrolysis and polycondensation of precursors in the presence of other polymer or metallic derivatives. Some inherent advantages of sol-gel process include the possibility of creating hybrid organic-inorganic materials at lower processing temperature, homogeneous system and good properties of the final

materials including the ability to enhance stationary phase stability (Kumar *et al.*, 2008; Singh, 2008; Li *et al.*, 2013; Moein *et al.*, 2019; Amiri *et al.*, 2020). Sol-gel method is an established approach to prepare various form of materials, therefore, designing new materials via this approach is expected to overcome the drawbacks of commercial sorbents and is used in the current work.

Pharmaceutical drugs are among organic clusters that have received much attention as intended analytes in scientific journals, including in the current study. Pharmaceuticals occurrence in environmental waters has been considered as an emerging issue pertaining to the tendency risk posed by their presence. Concern has been raised for non-steroidal anti-inflammatory drugs (NSAIDs) and antidepressant drugs as emerging contaminants since the drugs are among a wide range of pharmaceuticals that have been detected in many environment compartments (Afonso-Olivares *et al*., 2012; Nannou *et al*., 2015; Mezzelani *et al*., 2018; 2020). Most of the pharmaceutical drugs are not extensively degraded; consequently, the drugs are highly potential to be detected in environment waters. Pharmaceuticals occurrence can create possible threats to the aquatic life and water ecosystem. Worst, the micropollutants occasionally enter the groundwater and drinking source in which at certain level it can be dangerous to embryos, infants, children, and person with weak constitutions or allergic to pharmaceuticals (Lee *et al.*, 2014; Mezzelani *et al.*, 2016). Concerning the unforeseen threat exposed to aquatic life as well as indirectly endanger to humans, monitoring and measuring the concentration levels of the pharmaceutical drugs in environment are of central importance.

In analytical practice, solid phase extraction (SPE) is an established technique used for a wide spectrum of water contaminants including acidic and basic drugs (Wen *et al*., 2014; Paíga *et al*., 2017; Azzouz *et al*., 2018; Montes *et al.*, 2019; Alygizakis *et al.*, 2020). Even though SPE is a reliable sample pretreatment for decades, many attempts have made to shift the matured technology to meet the green analytical chemistry (GAC) requirement. GAC becomes an intense focus and safeguard for environmental-friendly laboratory practice wherein a few GAC components such as minimal use of sorbent or reagents, reducing sample number and size, minimal use of energy, less generation of waste have been imparted as attractive perspectives and has

been adhered as much as possible in analytical methods (Mohamed, 2015; Saroj *et al.*, 2018). As an effort to meet the requirement of GAC, research has geared toward micro-scale and miniaturized sample preparation which termed as microextraction.

Microextraction techniques offer the greatest benefits over conventional method to congruent with GAC. SPME represents the first generation of microextraction and evolution in existing analytical methods brings the second generation of microextraction where different configurations of solid phase-based extractions have been developed and improved from time-to-time (Kabir *et al*., 2017; Reyes-Garcés *et al.*, 2018; Soares da Silva Burato *et al.*, 2020). Amongst the microextraction options, dispersive-micro-solid phase extraction offers ease-of-use procedure and close contact with analytes in sample solution without utilizing the particles-filled cartridge or any auxiliary tools is the foremost method advantage (Du *et al*., 2018a; Ji *et al*., 2018; Chisvert *et al*., 2019; Nascimento *et al*., 2019). The term of dispersive-micro-solid phase extraction (D-µ-SPE) was coined in 2009 and the D-µ-SPE based on silica/primary and secondary amines (PSA) was first applied to isolation and preconcentration of veterinary drugs from swine muscle samples (Tsai *et al.*, 2009).

The evolution in solid phase-based extraction comes together with the emergence of new sorbent materials. Sorbent materials play a vital role in extraction methods to practically upgrade extraction efficiency and sensitivity. The advanced materials technology in analytical methods has been widespread and dominated research interests. Sorbents such as carbon nanotubes (Jiménez-Soto *et al*., 2012), mesoporous carbon (Yahaya *et al.*, 2013; 2019; Omidi *et al.*, 2020), graphene/graphene oxide (Mahpishanian and Sereshti, 2014; Amiri *et al*., 2019; Duval *et al.*, 2020), silica materials (Khezeli and Daneshfar, 2015), metal-organic frameworks (Lu *et al.*, 2016; Gutiérrez-Serpa *et al.*, 2020), carbon graphite nitride (Karbalaie *et al*., 2020), etc. have been applied in D-µ-SPE. Several constraints involved in conventional sorbents including specific-task, lack of selectivity, poor capacity as well as inefficient towards analytes with different polarities have opened the door to exploitation of various classes of materials. The deployment of neat or single materials has also many drawbacks. Thus, much effort has been put to position

composites or hybrid materials as attractive alternatives to conventional sorbents. The integration of magnetic nanoparticles (MNPs) into D-µ-SPE makes the method more feasible since the magnetized sorbents could be easily retrieved from sample solution by use of an external magnet.

Typical magnetic sorbents constitute simple magnetic composites and complex magnetic composites. Combination of magnetic nanoparticles with a non-magnetic material is classified as simple magnetic composite. For instance, simple magnetic composite based on solely magnetite and carbon nanotubes was employed to determine selected NSAIDs in environmental water samples (Abujaber *et al*., 2018; El-Sheikh *et al*., 2019). Complex magnetic composites consist of more than two non-magnetic components and the multifunctional composites possess additional desired properties that can elevate the sorbent efficiency in extracting the target compounds. For example, a new D-µ-SPE sorbent based on combination of metal-organic framework, multiwalled carbon nanotubes and magnetite was synthesized and applied in toxic environmental chemicals extraction (Jalilian *et al*., 2019). The resulting magnetic composite exhibited high surface area and porosity, good adsorption capacity and superior magnetic property and the enhanced merits of the sorbent provided significant advantages of excellent extraction performance and fast extraction kinetic.

The use of composites based on advanced materials such as graphene/graphene oxide nanocomposites (Rashidi Nodeh *et al.*, 2017; Ferrone *et al.*, 2018; Li *et al*., 2018a; Sarp and Yilmaz, 2019; Medina *et al.*, 2020), metal organic frameworks-based composites (Wang *et al.*, 2017; Boontongto and Burakham, 2019; Jalilian *et al*., 2019), conducting polymers (Marsin *et al.*, 2018; 2020) is top-notch and expanding over the years. Due to the extraordinary properties and excellent extraction capability, such extraction sorbents have been widely used for drugs determination in water samples. Jalilian and co-workers synthesized a new nanocomposite made of magnetite, carbon nanotubes and poly(2-aminopyrimidine) for determination of acidic and basic drugs in wastewater (Jalilian *et al*., 2018). Determination of polar NSAIDs was accomplished using a new magnetic sorbent based on magnetite, polyethyleneimine and reduce graphene oxide (Li *et al*., 2019). Despite their fascinating extraction performance, producing sorbent materials made of two or three advanced materials might incur high

cost and lengthy synthesis routes. Seeking the alternatives or complementary materials from renewable and natural sources is advantageous from economic and environmental perspective. In addition, employing substance from renewable resource is among the listed philosophy of "12 Principles of Green Chemistry" (Anastas and Kirchhoff, 2002). One of the attractive natural source materials is sporopollenin.

Sporopollenin is an abundant and low-cost natural organic polymer derived from exoskeletal or exine of plant spores and pollen (Binks *et al.*, 2011). Among the pollen grains species, sporopollenin from *Lycopodium Clavatum* is more accessible and readily marketed. The unique morphology of the exines and the polymeric nature comprises hydroxyl, carboxyl, carbonyl is thought to be a useful green extraction sorbent. Several literatures described the potential of sporopollenin as an inexpensive sorbent for removal of toxic pollutants (Şener *et al*., 2014; Sargin and Arslan, 2015; Ahmad *et al.*, 2017; Hassan *et al.*, 2020). Sporopollenin is relatively unexplored as extractant usage and the fact that such less processing and abundant natural polymer is highly desirable as it is cheaper, safer and is a sustainable alternative offer a good option. The unique properties such as chemical and physical robustness and incredibly consistent in size, morphology and composition as well as possess various group functionality make the biopolymer attractive over the available synthetic polymers (Erzengin *et al*., 2011; Şener *et al*., 2014; Kamboh *et al*., 2016; Kaya *et al*., 2017; Pomelli *et al*., 2020).

To date, exploitation of sporopollenin and its composites in sample preparation and as enrichment sorbent media is still limited. Pollen grains from Pinus Massoniana Lamb were demonstrated as a hydrophilic SPE sorbent for analysis of selected pesticides in fruits and vegetables (Lu *et al*., 2014). However, classical SPE involves multi-step procedure that is time-consuming and clogging issue often associated with the packed-bed column. Alternatively, D-µ-SPE technique is more convenient and time-saving approach that could circumvent the weakness of conventional SPE. In this context, the merits of naturally occurring as microparticles with great uniformity which is difficult to obtain by man-made and it is well-dispersed in water due to the abundant oxygen-containing functional groups in its structure grants the biopolymer to fulfill the criteria of dispersive-based extraction sorbent. Additionally, sporopollenin is

amenable to surface modification which enables the natural biopolymer to combine with different kinds of compounds and materials to serve as efficient composites (Syed Yaacob *et al*., 2018a, b). Since development of safer, benign, and sustainable materials shows great promise for environmental-friendly sample preparation method, such sustainable biopolymer is explored as material of choice in this study.

The GAC has stimulated the green wave in research field where research interests are not only specific to microextraction methods development but also designing material with less impact on the ecosystem. Thus, in this study, special attention has been devoted to producing three new sorbents entailing the use of less toxic or/and natural substance with the attempt to develop green analytical solid phase-based extraction for determination of selected aldehydes, NSAIDs and tricyclic antidepressants (TCAs). Interestingly, the green criterion of a method could be evaluated using an ecological scale. This scale is evaluated based on deduction of 100 points with penalty points derived from the volume/toxicity of reagents used, consumption of energy, emission, operator hazard as well as generation of waste. An excellent green analysis is given to a method with > 75 points whereas acceptable green analytical method is > 50 points (Armenta *et al*., 2015). This analytical eco-scale is employed in the current work to assess the green character of the developed methods using the sorbents produced.

1.2 Problem Statement

SPME is an outstanding green analytical method and the technique has been widely used to analyse volatiles including aldehydes (Poli *et al.*, 2010; Ma *et al.*, 2014; da Silva *et al.*, 2015; Aisala *et al.*, 2019; Filipowska *et al*., 2020). Interestingly, many in-house SPME fibers adopted sol-gel method to address limitations related to thermal stability and mechanical strength (Ghader *et al*., 2017; Yarazavi *et al*., 2018; Shnayder *et al*., 2019). The use of sol-gel precursors to produce hybrid organic-inorganic material as SPME extraction phase is relatively cost-effective. Great performance of hybrid materials derived from two alkoxysilane precursors has been clarified in

previous works (Wan Ibrahim *et al*., 2012; 2013; Muhamad *et al*., 2014). Inspired by the success of these works, herein, sol-gel hybrid based on methyltrimethoxysilane-(3-mercaptopropyl)-trimethoxysilane (MTMOS-MPTMOS) was proposed for aldehydes analysis. However, there are several constraints associated with the development of in-house SPME fiber including cumbersome pre- and post-gelation treatment, long ageing time and conditioning is needed before first use. Fiber breakage as well as sample carryover during analysis are other limitations that have outweighed the advantage of SPME. These shortcomings have been highlighted in this work by proposing hollow fiber-solid phase microextraction (HF-SPME) as an alternative and simpler approach to extract volatiles such as aldehydes from aqueous samples. Rather than using a fiber as support, HF-SPME makes use of a small segment of polypropylene hollow fiber to hold the extracting phase. In this regard, sol-gel MTMOS-MPTMOS could be coated in a simple manner without heat treatment via in-situ polymerization in the lumen of the HF and thus, could serve as a convenient extraction media for the determination of hexanal and heptanal.

Designing sorbent materials derived from biopolymer and natural sources is an effective strategy to give low environmental impact. Sporopollenin is nontoxic, biodegradable, cheap and from renewable sources that fit the green sorbent criteria. The property of high dispersibility in water allows the biopolymer to be used in D-µ-SPE format. Separation of sporopollenin sorbent from sample solution is quite challenging. In addition, pollen grains suffer from low surface area (Thio *et al*., 2011) which may cause lack of selectivity to reach satisfactory fast kinetic extraction efficiency. Functionalizing the sporopollenin surface serves as an appropriate solution to improve and enhance its extraction capability and incorporation of magnetic property allows for easy separation of the sorbent, avoiding filtration step. As concern has surfaced over the effect of sorbent materials toxicity, choosing low toxicity and biodegradable substances as functional materials is much pronounced to attend to the demand for eco-friendly extraction strategy. Herein, two attractive substances that are nontoxic, inert and biocompatible namely cyanopropyltriethoxysilane and alkylbenzonitrile substituted *p-tert*-butylcalix[4]arene were selected as viable functional groups in magnetic sporopollenin composites. They are easy to be functionalized and possesses hydrophobic, hydrophilic as well as polarizable

characteristic that is expected to conceivably enhance the selectivity and versatility of the sporopollenin composites in extracting NSAIDs and TCAs. To the best of our knowledge, this is the first attempt on the use of magnetic sporopollenin-cyanopropyltriethoxysilane (MS-CNPrTEOS) and alkylbenzonitrile substituted *p-tert*-butylcalix[4]arene immobilized-magnetic sporopollenin (Calix-MS) in D-µ-SPE for analysis of selected NSAIDs and TCAs in water samples.

1.3 Aims and Objectives of Study

The study aims to produce three new efficient sorbents namely MTMOS-MPTMOS, MS-CNPrTEOS and Calix-MS and to use these materials to develop green analytical chemistry methods for selected aldehydes and two classes of drugs analysis in aqueous samples. The objectives of the study are to;

[1] develop a convenient and simple HF-SPME based on sol-gel hybrid MTMOS-MPTMOS for analysis of hexanal and heptanal in urine samples.

[2] prepare MS-CNPrTEOS as a new D-µ-MSPE biosorbent for extraction of selected NSAIDS and TCAs in water samples.

[3] demonstrate an efficient method for determination of selected NSAIDs by Calix-MS as a new D-µ-MSPE biosorbent in water samples.

1.4 Scope of Study

This work focuses on the development of solid phase-based microextraction with a special emphasis on the use of MTMOS-MPTMOS, MS-CNPrTEOS and Calix-MS as material of choice for analysis of selected aldehydes, NSAIDs and TCAs in aqueous samples. This study includes preparation and characterization of the three new sorbents and method development which covers optimization, validation, and

application to real samples. In this context, sol-gel hybrid MTMOS-MPTMOS was synthesized as HF-SPME coating material and the MTMOS-MPTMOS HF-SPME was demonstrated for hexanal and heptanal in urine samples. Gas chromatography-flame ionization detector (GC-FID) was used for detection and quantification. Both MS-CNPrTEOS and Calix-MS were synthesized as D-µ-MSPE sorbents and used for the determination of ketoprofen, ibuprofen, diclofenac and mefenamic acid in tap water, lake water, river water and wastewater. The D-µ-MSPE based on MS-CNPrTEOS was also evaluated and applied for the determination of three TCAs (amitriptyline, imipramine and chlorpromazine) in water samples. High performance liquid chromatography-ultraviolet (HPLC-UV) was used for detection and quantification of the selected NSAIDs and TCAs. The last part of study involves the green assessment of the developed methods using analytical eco-scale (penalty points approach).

1.5 Limitation of Study

This study has some limitations. In this work, environment water samples were collected at nearby source (viz., Skudai, Johor Bahru) and used for validation purpose in method development. The collection of water samples involved only a snapshot of the situation at the sampling area and this small sample size might fail to provide more realistic information. In addition, the possibility of non-presence of the target analytes might be associated to the inavailability of sensitive detection for trace amount (low ng/L) since laboratory-based instrumental analysis was performed using UV detector. In order to acquire more intensive monitoring of environmental levels, multi-channel sampling stations and liquid chromatography-tandem mass spectrometry can be used in the future work.

1.6 Significance of Study

MTMOS-MPTMOS HF-SPME and D-µ-MSPE based on bio-based sporopollenin composites are new extraction media that offer fast, easy handling and efficient miniaturized approach for determination of selected target organic compounds. The developed methods are expected to satisfy the GAC aspect and have great potential to serve as promising cost-effective alternatives to established methods. This work emphasizes on the minimal use of hazardous solvents, user-friendly procedure, and the employment of eco-compatible materials and from renewable source which bring the eco-friendly advantage to mitigating environmental impact. Since environmental component is included in Sustainable Development Goals (SDGs), this work represents an academic effort to support SDGs and the 2030 Agenda to be pursued.

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LIST OF PUBLICATIONS

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Abd Wahib, S. M., Wan Ibrahim, W. A., Sanagi, M. M., Kamboh, M. A. and Abdul Keyon, A. S. (2018), 'Magnetic Sporopollenin-Cyanopropyltriethoxysilane-Dispersive-Micro-Solid Phase Extraction Coupled with High Performance Liquid Chromatography for the Determination of Selected Non-Steroidal Anti-Inflammatory Drugs in Water Samples', *Journal of Chromatography A*, 1532, 50-57. **(Q1; IF: 4.049).**

Indexed Journal

Abd Wahib, S. M., Wan Ibrahim, W. A., and Sanagi, M. M. (2016), 'New Methyltrimethoxysilane-(3-Mercaptopropyl)trimethoxysilane Coated Hollow Fiber-Solid Phase Microextraction for Hexanal and Heptanal Analysis', *Malaysian Journal of Analytical Sciences*, 20 (1), 51-63 **(Indexed by Scopus).**

Non-Indexed Conference Proceeding

Abd Wahib, **S. M**., Wan Ibrahim, W. A., and Sanagi, M. M. (2014). Optimization of Sol-Gel Hybrid-Mediated Hollow Fiber-Solid Phase Microextraction for Analysis of Selected Aldehydes. *Proceeding of 2nd International Science Postgraduate Conference 2014*. ISBN: 978-967-0194-39-4.

LIST OF PRESENTATIONS

Siti Munirah Abd Wahib, Wan Aini Wan Ibrahim, Mohd Marsin Sanagi. Sol-Gel Hybrid Coating Hollow Fiber-Solid Phase Microextraction Coupled with Gas Chromatography-Flame Ionization Detection for Analysis of Hexanal and Heptanal. Paper presented at the 27th Regional Malaysian Symposium of Analytical Sciences (SKAM-27), Johor Bahru, 9-10 December 2014.

Siti Munirah Abd Wahib, Wan Aini Wan Ibrahim, Mohd Marsin Sanagi. Magnetic Sporopollenin-Grafted Cyanopropyltriethoxysilane for Analysis of Non-Steroidal Anti-Inflammatory Drugs. Paper presented at International Science Postgraduate Conference 2016, Johor Bahru, 22-24 February 2016.

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