ASSESSMENT OF EMERGING POLLUTANTS IN SKUDAI RIVER AND ITS TREATABILITY AT DOWNSTREAM WATER TREATMENT PLANT

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Civil Engineering)

School of Civil Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

AUGUST 2021

DEDICATION

This thesis is dedicated to:

Special dedication to my beloved father Talib @ Harun Lebai Hashim and my beloved mother Siti Aishah Saidin, for their love and prays, without them none of my success would be possible.

To my beloved husband Mohamad Husaini Paboh, he has been a great source of motivation and strength during the moments of discouragement and despair.

To my lovely son and daughter, Mohamad Hakimi and Arissa Maisarah,

mama loves you all so much.

And to my beloved sisters and brothers for their support.

ACKNOWLEDGEMENT

Praise to Allah SWT, The Almighty for giving me strength, passion, and blessed to complete my PhD study. The experience gained during the process of completing this study has been deeply enriching and meaningful. My deepest gratitude goes to my supervisor Professor Dr. Azmi Aris for his dedication and commitment in guiding me throughout the process of completing my PhD. Your time and energy, your knowledge and your invaluable experience have enriched my interest in academic and scientific research. Not forgetting to my co-supervisor Associate Professor Dr. Jafariah Jaafar and Associate Professor Dr. Zaiton Abdul Majid for their invaluable guidance and encouragement.

I would like to thank the authority of Universiti Teknologi Malaysia (UTM) for providing me with a good environment and facilities to complete my PhD. I also place on record, my sense of gratitude to one and all, who directly or indirectly, have lent their hand to this venture.

Last but not the least, I would like to thank; my family, parents, brother and sisters for supporting me spiritually throughout the process of writing this PhD thesis and my life in general.

Above all, I thank the Almighty Allah SWT for His unmerited favour and wonderful grace in guiding every step of my life and giving me the faith to believe that all things are possible to him who believes.

ABSTRACT

Emerging Pollutants (EPs) are synthetic or naturally occurring compounds currently detected in water environment. These chemicals such as surfactants, pharmaceuticals, personal care products (PCPs) and pesticides could cause adverse ecological and human health effects which include alteration of the normal function of endocrine systems of human and animals. With variation of potential sources, determination of their presence is also a difficult and costly. Different treatment technologies to remove the EPs for drinking water have been studied, which include adsorption, chemical oxidation and membrane filtration. Nevertheless, these technologies are relatively costly in terms of capital, operation and maintenance. This study was carried out to identify the best technique in extracting the EPs from water for screening purposes, to assess their presence in River Skudai and to determine the ability of downstream water treatment plant (WTP) to remove the EPs. Identification approach and solvent was carried out through extensive literature review and trial tests. Samples were taken from eight sampling points in Skudai River and five points in the WTP. Samples were pretreated using solid phase extraction (SPE) method and were analysed using Liquid Chromatography-Mass Spectrometry Detection (LCMS-QTOF) for the river water sample and using Gas Chromatography-Mass Spectrometry (GCMS) for the treatment plant water sample. It was surmised that the extraction of EPs is largely based on polarity. The acetonitrile and methanol are highly polar solvents that can achieve high yields of EPs. EPs detected in Skudai River can be categorized into three groups, namely pharmaceutical (decylamine, hexadecyl isocyanate, methotrexate, butirosin A, tridodecylamine and 4-vinylcyclohexene), PCPs (tetradecylamine, limonene, oleylamine, and diethanolamine) and EDCs (styrene, ethylbenzene, phthalic and alfa-methyl styrene). The concentration of styrene ranged from 45 μg/L to 203 μg/L with an increasing trend towards downstream of the river. All the EPs detected are classified as toxic and carcinogenic compounds. As for the WTP, the coagulation process successfully removed endosulfan, chlorothalonil, and ethylbenzene while sedimentation removed 50% of benzene, 50% of triazine, along with 100% of ibuprofen and bisphenol A (BPA). Filtration and chlorination process did not remove styrene or triazine. Trihalomethanes (THMs) which are classified as EDCs were formed after chlorination process. Using polynomial multivariate, the removal rate of triazine in the water treatment plant was modelled. A nonlinear regression design was successfully applied to model the response as a polynomial function based on selected independent. Polynomial multivariate was further used to conduct and evaluate the effectiveness of coagulation and sedimentation process. The findings of this study indicate that different types of EPs can be found in Skudai River. While many can be successfully removed in the conventional water treatment plant, more efforts are needed to ensure that the environment and human health are protected from the hazardous EPs.

ABSTRAK

Bahan cemar baharu (EP) adalah sebatian sintetik atau semula jadi yang kini dikesan di dalam persekitaran air. Bahan kimia seperti surfaktan, farmaseutikal, produk penjagaan diri (PCP) dan racun perosak boleh menyebabkan kesan buruk kepada ekologi dan kesihatan manusia yang merangkumi pemindaan fungsi normal sistem endokrin manusia dan haiwan. Dengan kepelbagaian sumber berpotensi, penentuan kehadiran bahan cemar ini adalah satu tugasan yang susah dan juga mahal. Pelbagai teknologi rawatan bagi menyingkirkan EP untuk air minuman telah dikaji, meliputi penjerapan, pengoksidaan kimia dan penapisan membran. Walaupun begitu, teknologi-teknologi ini agak mahal dari segi modal, operasi dan penyelenggaraan. Kajian ini dilakukan untuk mengenal pasti teknik terbaik untuk mengekstrak EP dari air bagi tujuan saringan, untuk menilai kehadiran EP di Sungai Skudai dan untuk menentukan kemampuan loji rawatan air (WTP) di hilir sungai dalam menyingkirkan EP. Pengenalpastian pendekatan dan pelarut dilakukan melalui tinjauan literatur yang luas dan melalui ujian percubaan. Sampel diambil dari lapan tempat persampelan di Sungai Skudai dan lima tempat di loji rawatan air. Sampel dipra-rawat dengan menggunakan kaedah pengekstrakan fasa pepejal (SPE) dan dianalisis menggunakan kromatografi cecair spektroskopi jisim masa penerbangan (LCMS-QTOF) untuk air sampel sungai dan menggunakan kromatografi gas spektroskopi jisim (GCMS) untuk air sampel WTP. Adalah disimpulkan bahawa pengekstrakan EP sebahagian besarnya berdasarkan kepada polariti. Asetonitril dan metanol adalah pelarut berpolariti tinggi yang dapat mencapai hasil EP yang tinggi. EP yang dikesan di Sungai Skudai dapat dikategorikan kepada tiga kumpulan iaitu farmaseutikal (desilamina, heksadesil isosianat, metoreksat, butirosin A, tridodesilamina dan 4 vinilsikloheksena), PCP (tetradesilamina, limonena, olelamina, dan dietanolamina) dan EDC (stirena, etilbenzena, ftalat, metil stirena). Kepekatan stirena adalah di antara 45 µg/L hingga 203 µg/L dengan trend peningkatan ke hilir sungai. Semua EP yang dikesan dikelaskan sebagai sebatian toksik dan karsinogenik. Bagi WTP, proses pengentalan berjaya menyingkirkan endosulfan, klorotalonil, dan etilbenzena sementara pengenapan dapat menyingkirkan 50% benzena, 50% triazina, bersama dengan 100% ibuprofen dan bisfenol A (BPA). Proses penapisan dan pengklorinan tidak dapat menyingkirkan stirena atau triazina. Trihalometana (THM) yang diklasifikasikan sebagai EDC terbentuk selepas proses pengklorinan. Dengan menggunakan polinomial pelbagai, kadar penyingkiran triazina di loji rawatan air telah dimodelkan. Reka bentuk regresi bukan linear berjaya dilaksanakan untuk model tindak balas sebagai fungsi polinomial berdasarkan pilihan bebas. Polinomial multivariat selanjutnya digunakan untuk menjalankan dan menilai keberkesanan proses pengentalan dan pengenapan. Hasil kajian ini menunjukkan bahawa pelbagai jenis EP boleh didapati di Sungai Skudai. Walaupun banyak yang berjaya dikeluarkan di loji rawatan air konvensional, lebih banyak usaha diperlukan untuk memastikan bahawa persekitaran dan kesihatan manusia dilindungi dari EP berbahaya.

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LIST OF ABBREVIATIONS

APCI	-	Atmospheric pressure chemical ionisation
APPI	-	Atmospheric pressure photoionisation
AOP	-	Advance Oxidation Processes
BOD	-	Biological Oxygen Demand
BPA	-	Bisphenol A
COD	-	Chemical Oxygen Demand
CWs	-	Constructed wetlands
DDT	-	Dichloro-diphenyl-trichloroethane
DEET	-	N, N-Diethyl-meta-toluamide
ED	-	Electrodialysis
EDC	-	Endocrine Disrupting Chemical
ELISA	-	Enzyme-linked immunosorbent assays
EP	-	Emerging Pollutants
GAC	-	Granular Activated Carbon
GC	-	Gas chromatography
GCMS	-	Gas Chromatography Mass Spectrometry
HAAs	-	Halo Acetic Acids
HNO_3	-	Nitric Acid
HPLC	-	High-Performance Liquid Chromatography
HFLPME	-	Hollow fibre liquid-phase microextraction
IT	-	Ion Trap
LCMS	-	Liquid Chromatography and Mass Spectrometry
LLE	-	Liquid-liquid extraction
LLME	-	Liquid-liquid microextraction
LSM	-	Least Square Method
MEKC	-	Micellar electrokinetic chromatography
MF	-	Microfiltration
MLR	-	Multiple Linear Regression
MPR	-	Multivariate Polynomial Regression
MATLAB	-	Multi-paradigm Numerical Computing Environment

MPR	-	Multivariate Polynomial Regression
NaOH	-	Natrium Hydroxide
NF	-	Nano-Filtration
NOM	-	Natural Organic Matter
NSAIDs	-	Non-steroidal Anti-inflammatory Drug
NTU	-	Nephelometric Turbidity *Unit*
OC	-	Organochlorine
PAEs	-	Phthalate esters
PFCs	-	perfluorochemicals compounds
PMRs	-	Photocatalytic Membrane Reactors
RO	-	Reverse Osmosis
RSM	-	Response Surface Methodology
SPE	-	solid-phase extraction
SPME	-	solid-phase microextraction
STP	-	Sewage Treatment Plant
THM	-	Trihalomethanes
TOC	-	Total Organic Compound
TC	-	Total Coliform
UF	-	Ultrafiltration
UV	-	Ultraviolet
VA	-	Veterinary antibiotics
WTP	-	Water Treatment Plant
WWTPs	-	Wastewater Treatment Plants
Zn (II)	-	Zinc ions

LIST OF SYMBOLS

β_1 β_2	-	Linear effect variables
β_{11} β_{22}	-	Quadratic effect parameters
β_{12}	-	Interaction effect parameter
$E(y)$	-	Regression function
M	-	Concentration of the moles of solute equal to molarity
ng/L	-	Nanogram per Litre
µg/L	-	Microgram per Litre
V	-	Volume of Solution

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Water demand in Malaysia was projected to increase by 113% to 20,338 m3/day in 2020 (Fulazzaky *et al.*, 2010). Such an increase in demand indicates the importance of protecting the quality of raw water. This is because slight contamination or deterioration of water quality can lead to the scarcity of water resources and jeopardise the water supplied to the consumers. Besides the conventional organic and inorganic pollutants, emerging pollutants (EPs) can contribute to the contamination or deterioration of water quality. These newly detected contaminants are classified as EPs or also known as contaminants of emerging concern (CEC).

Emerging pollutants are synthetic or naturally occurring compounds that are currently detected in the aquatic environment. These compounds have been detected in their original form or sometimes as metabolites. These include natural organic matter (NOM), veterinary antibiotics and hormones (Ho *et al.*, 2014) and various classes of micro-pollutants such as pharmaceuticals and personal care products (PPCPs), pesticides, estrogenic hormones, polyaromatic hydrocarbon, dioxins, bioactive chemicals, polymers and polychlorinated compounds (Omar *et al.*, 2019; Ismail *et al.*, 2020). Due to their ability to alter the normal function of the endocrine systems in human and animals, some of them are also known as endocrine-disrupting chemicals (EDC).

Typically, some of the outlined chemical compounds and their metabolites are naturally present in water bodies. Numerous synthetic forms or derivatives are produced from daily human activity. These essential compounds include biocides, medications, and pesticides. Other notable household or general use chemicals include flame retardants, plastics and cleaning products for furniture.

Depending on the type and characteristics of the EPs, detection of the compounds in a water sample is normally carried out using high end spectrophotometers such as Gas Chromatography-Mass Spectrometry (GCMS) and Liquid Chromatography-Mass Spectrometry (LCMS). The detection depends on the sensitivity of the equipment, which is improving with time.

The presence of EPs has been reported in several studies in Malaysia. Omar *et al.*, (2019) evaluated the occurrence and level of selected emerging organic contaminants (EOCs) in fish and mollusc samples from the estuary of the Klang River. The findings demonstrated the presence of diclofenac, bisphenol A, progesterone, and amoxicillin with diclofenac and progesterone gave the highest concentrations in fish and mollusc samples ranging from 1.42 ng/g to 10.76 ng/g and from 0.73 ng/g to 9.57 ng/g, respectively. Although their findings will unlikely pose any health risk to consumers, this preliminary result is an important finding for pollution studies in Malaysian tropical coastal ecosystems.

Praveena *et al.*, (2019) studied the presence of nine pharmaceutical residues in drinking water (amoxicillin, caffeine, chloramphenicol, ciprofloxacin, dexamethasone, diclofenac, nitrofurazone, sulfamethoxazole, and triclosan) at the Putrajaya residential area in Malaysia. The authors found the presence of pharmaceutical residue concentrations up to 0.38 ng/L, with the highest concentration of caffeine (0.38 ng/L) and the lowest concentration of diclofenac (0.14 ng/L). Based on the findings, the authors conclude that the nine pharmaceuticals are not only ubiquitous in daily drinking water but can increase the likelihood of potential human health risks.

Over the years, the growing existence of these micropollutants has become a source of concern for governments, academia, and industry around the globe on their potential of causing harmful effects on human health, animals, and the ecosystem. Furthermore, the progressively multifaceted mixture of these chemical compounds is seeping into the drinking water supplies obtained from water bodies. Studies have shown that long-term exposure to these chemical compounds even at low concentrations can potentially result in catastrophic effects on humans, animals, and the environment. It has also become evident that the mixtures of these compounds and chemical substances in natural water bodies can result in complex, additive, and new stressor effects (Jokiel *et al.*, 2004). The presence of such chemicals has been reported to elevate water temperatures or higher levels of ultraviolet (UV) radiation, which is reportedly linked to climate change and other harmful effects on animals in the environment (Kraemer *et al.*, 2005; Blaustein *et al.*, 2015). Additionally, these chemicals can alter the normal function of the endocrine systems in human and animals (Esplugas *et al.*, 2007; Seyhi *et al.*, 2013).

Therefore, various treatment technologies have been explored to remove the EPs for drinking water. These include adsorption, chemical oxidation, and membrane filtration (Azimvand *et. al.*, 2016; Toczyłowska-Mamińska, 2017). Currently, the removal of the micropollutants using state-of-the-art technologies such as ozonation and membranes is widely reported in the literature (Joss *et al.*, 2008; Lee *et al.*, 2012, 2019; Margot *et al.*, 2013; Stylianou *et al.*, 2018). However, these technologies are relatively expensive in terms of capital, operation, and maintenance. This scenario is particularly evident in developed countries where these technologies are widely available. However, the utilisation of such highly advanced water treatment technologies is gravely lacking in the developing world. Therefore, there is a need to identify, examine, and highlight more environmentally sustainable, economically viable, and technologically efficient approaches for the removal of the EPs and other micropollutants in water bodies.

1.2 Problem Statement

The upsurge in human activity and agricultural land use has significantly impacted the occurrence of EPs, particularly in water bodies such as rivers. Most studies have determined targeted compounds. However, with the density of land use and pollution sources within Skudai River catchment including industrial and domestic effluents, and commercial and township activities, there is a high variation of EPs that are presence in the river. So, it is interesting to know the types of EPs in the river and how they vary from upstream to downstream of the river.

The increase in point and non-point water pollution, combined with the high demand for water due to rapid population growth and development has prompted the government, under the Ministry of Natural Resources and Environment to launch the National Water Resource Policy in 2012. Conventionally, only water physico-chemistry metrics are monitored in Malaysian rivers. However, without considering the dynamics of biological communities and the variances in landscape physical that can affect river integrity, this approach alone is not enough. Malaysia does not have a unified river assessment protocol yet. Thus, it would be more efficient to utilize the available accepted systems, metrics, and readapt them to the local environment where appropriate to ensure the widest acceptance and adoption (Ng *et al.*, 2020).

While analytical equipment plays important role in the EPs analysis, sample preparation usually takes 70 to 90 % of the analysis time (Zuloaga *et al.*, 2012). There are many extraction steps depending on the type of water to be analysed. The most classic and common methods are soxhlet and soxtec (use of large volume of solvent), ultrasound-assisted extraction (leaching of organic), and stirring and mechanical shaking. Novel techniques include the use of focused ultrasound-assisted liquid extraction (FULSE), pressurized liquid extraction, pressurized hot water extraction, microwave-assisted extraction, supercritical fluid extraction, matrix solid-phase dispersion, liquid-liquid extraction, and liquid-phase microextraction with porous hollow fibre (HF-LPME) (Yoon *et al.*, 2010; Vulliet and Cren-Olivé, 2011; Wille *et al.*, 2012; Zuloaga *et al.*, 2012; Neale *et al.*, 2017; Rousis *et al.*, 2017; Hebert *et al.*, 2018; Kiefer *et al.*, 2019). Many studies have reported of various extraction techniques and solvent for various targeted EPs; however, these have not been well addressed when dealing with screening various unknown compounds.

As EPs in the environment exist at trace levels, extracting the EPs from a sample is carried out with the use of a solvent (Tang *et al.*, 2019). The extraction method depends on the sample and instrument detection used. The most commons are liquid-liquid extraction (LLE), solid-phase extraction (SPE), solid-phase microextraction (SPME), sulfur elimination, combined extraction, selective pressurized liquid extraction (SPLE), and gel permeation extraction (GPC) (Wille *et al.*, 2012; Zuloaga *et al.*, 2012). Solid-phase extraction (SPE), followed by gas chromatography or liquid chromatography coupled with mass spectrometry, is still a reliable method for analysis of a huge number of EPs because of its speed, accuracy and reliability (Tang *et al.*, 2019). Extraction of the EPs depends on the solubility, selectivity, and cost of the solvents. In theory, polar EPs easily dissolve in a polar solvent, and the less polar EPs solutes in a less polar solvent (Zhang *et al.*, 2018). For targeted compounds, the selection of solvent is much easier as the polarity of the compounds are known. However, this is difficulty in screening of unknown EPs. The solvent need to be able to deal with the presence of polar and non-polar compounds. Unfortunately, work on this aspect is apparently very limited.

Numerous studies have been conducted to detect, monitor or remove the EPs present in water bodies (Yoon *et al.*, 2010; Vulliet *et al.*, 2011; Neale *et al.*, 2017; Rousis *et al.*, 2017; Hebert *et al.*, 2018; Kiefer *et al.*, 2019). However, most of the EPs detected in this study are not stated in the drinking water quality standard of Malaysia as presented in Table 4.4. As observed, Table 4.4 (Chapter 4) accounts for only THM, endosulfan, and benzene, which leaves out various other critical EPs detected in the SIWTP in this study. The nature, composition, occurrence, and treatment processes for the removal of EPs reported for river water sources in Malaysia are limited in the literature. Furthermore, the gap in the knowledge on the toxicity and potential impacts of these EPs on human health, animals and the water environment in Malaysia requires further attention. This is crucial for Skudai River, which is in the densely populated city of Johor Bahru in Malaysia.

While the conventional water treatment plants are designed and operated to eliminate suspended solids and other particulate contaminants, the removal of soluble contaminants particularly EPs is not well documented. With the recent advances in chemical analysis of environmental compounds, EPs and related compounds can be detected in aqueous samples even at low concentrations. This advanced approach will enhance the detection of various levels of EPs detected in surface, ground or tap water, which have increased over the years (Chen *et al.*, 2000; Sui *et al.*, 2011). Furthermore, modelling of the EPs removal in the treatment plant processes is apparently very limited.

1.3 Objectives of the Study

This study was conducted to examine the presence of EPs in Skudai River and its water treatment plant. The work is based on the following objectives:

1. To identify appropriate techniques and solvents suitable for EPs detection.
2. To characterize the presence of EPs in the Skudai River in relation to land use and human activities.
3. To characterize EPs in the water treatment plant and to evaluate the efficiency of conventional water treatment plant processes in removing EPs.
4. To quantify/determine the removal of EPs by simulating the coagulation-flocculation-sedimentation-filtration processes using polynomial multivariate for the removal of triazine in the water treatment plant.

1.4 Scope of the Study

The assessment of EPs in this study has been focused on the Skudai River located in the southern part of Johor, covering from the Sedenak area to the water intake of Sultan Ismail Water Treatment Plant (SIWTP). The water samples were collected five times from eight sampling points distributed along the river from January 2014 to May 2015. The selection of the sampling points accounts for the contribution of pollutants from the residential, industrial, and agricultural activities within the river basin. The river water was screened for various NOMs and EPs using Gas Chromatography-Mass Spectrometry (GCMS) and Liquid Chromatography-Mass Spectrometry (LCMS) method. The study also assessed the presence of the EPs at various stages of the process at the SIWTP. The data were analysed to determine the relationship between the EPs and the land use within the basin. The data were also used to develop a polynomial multivariate model that describes the relationship between the process operating conditions and the EPs removal and finally to optimise and validate the model based on the collected data.

1.5 Limitation of Study

The main limitation of the study is related to the analytical equipment and sample collection at SIWTP. The Gas Chromatography-Mass Spectrometry (GCMS) and Liquid Chromatography-Mass Spectrometry (LCMS-QTOF) encountered problems particularly with the procurement of spare parts, which lasted almost 2 years. This affects the number of sampling activities and determination of styrene concentration in the river and at the SIWTP. Furthermore, the limited number of sampling was affected by the time and budget provided for the study.

The collection of samples at SIWTP was significantly affected by the frequent close-down of the treatment plant due to high ammonia concentration in the Skudai River. This is exacerbated by the closure of the treatment plant from public assess due to security reason. Hence, the data for the third and fourth objectives of the study was limited to the available data gather prior to the closure of the treatment plant.

1.6 Organization of the Thesis

This thesis comprises five chapters. Chapter One presents the introduction and background, statement of the problem, aims, scope and significance of the study. Chapter Two presents the review of the literature, discussions on the WTP process, along with the various treatment processes employed in previous research. This chapter will also highlight the nature, operational issues, and research trends related to the removal of EPs. Chapter Three presents the research methodology, while Chapter Four presents the results, analyses, and discussion based on laboratory experiments and fieldwork. Lastly, Chapter Five presents the conclusions and recommendations for future work in the area.

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