SOLAR PHOTOCATALYTIC DEGRADATION OF BASIC RED 51 IN HAIR DYE BATHROOM GREYWATER USING ZINC OXIDE NANOPARTICLES SYNTHESIZED WITH *Corriandrum Sativum* LEAF EXTRACT

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A thesis submitted in fulfilment of the requirement for the award of the Doctor of Philosophy

Faculty of Civil Engineering and Built Environment
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FEBRUARY 2021

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged.

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ABSTRACT

The high tendency of chemically synthesized ZnO NPs to aggregate causes drop in the solar photocatalytic degradation (SPD). The present study is aimed to formulate ZnO NPs synthesized using *Corriandrum sativum* leaf extract as a capping agent to prevent aggregation of ZnO NPs for hair dye bathroom greywater degradation (HDBGW). The ZnO NPs were prepared at 100°C [[ZnO [A]] and calcined at 550°C [[ZnO [B]]. The produced ZnO NPs were characterized by field emission scanning electron microscopy with energy dispersive X-ray spectroscopy (FESEM-EDX), transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray powder diffraction (XRD), X-ray photoelectron spectroscopy (XPS), fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis/ differential scanning calorimetry (TGA/DSC) and Raman spectroscopy. The SPD of artificial hair dye bathroom greywater (AHDBGW) was optimized based on ZnO loadings, pH and initial Basic Red 51(BR51) concentrations while the kinetic model was investigated using Langmuir-Hinshelwood in an aqueous solution of BR51. The SPD of BR51 degradation pathway was studied using gas chromatography-mass spectrometry (GC-MS). The XRD analysis confirmed the hexagonal wurtzite structure of both types ZnO NPs. The TEM analysis of ZnO NPs [A] and ZnO NPs [B] were between 117 and 149 nm and 71.1 and 102 nm, respectively. FTIR revealed the presence of –C–O, =C-H, C=C, –C–O–C and O–H bonds in both types of ZnO NPs. The best operating parameters for SPD of BR51 in AHDBGW was 0.10 g of ZnO NPs, at pH 5 and with 1 ppm of BR51. It can be observed that SPD of AHDBGW and RHDBGW by ZnO NPs reduces COD and BOD_5 concentrations by 80.92% and 79.14% (COD) and 82.91% and 70.18% (BOD_5) respectively. The SPD BR51 in aqueous solution follows pseudo first-order kinetics with average rate constants of 0.019, 0.014, 0.008 and 0.005 min^{-1} for 5, 10, 15 and 20 ppm of BR51 at optimum condition respectively. The SPD pathway determines hexadecanoic acid, methyl ester and octadecanoic acid and methyl

ester as safer final products. Hence, it is found that the study represents an effective solution for HDBGW treatment to ensure the environmental sanitation for future generations.

ABSTRAK

Kecenderungan tinggi ZnO NP yang dihasilkan menggunakan kaedah kimia untuk bergumpal menyebabkan penurunan dalam penguraian fotokatalitik suria (SPD). Oleh itu, kajian ini bertujuan untuk mencirikan zink oksida (ZnO NPs) yang dihasilkan menggunakan teknologi hijau menggunakan ekstrak daun ketumbar sebagai agen pelindung untuk mengelakkan pergumpalan ZnO NP dalam penguraian air sisa bilik mandi yang mengandungi pewarna rambut (HDBGW). ZnO NP dikeringkan terlebih dahulu pada suhu 100°C [[ZnO [A]] dan dikalsinasikan pada suhu 550°C [[ZnO [B]]. Ciri-ciri ZnO NP dikaji menggunakan mikroskop elektron pengimbasan pelepasan medan dengan spektroskop X-ray tenaga dispersif (FESEM-EDX), mikroskop elektron penghantaran (TEM), mikroskop berkuatsa atom (AFM), difraksi serbuk sinar-X (XRD), spektroskop fotoelektron sinar-X (XPS), Spektroskop inframerah transformasi (FTIR) analisis termogravimetri / kalorimetri imbasan pembezaan (TGA/DSC) dan spektroskop Raman. Proses SPD air sisa bilik mandi yang mengandungi pewarna rambut yang dihasilkan secara sintetik (AHDBGW) dioptimumkan berdasarkan dos ZnO NPs, pH dan kepekatan awal Basic Red 51 (BR51) sementara model kinetik disiasat menggunakan Langmuir-Hinshelwood dalam larutan berair BR51. Hasil penguraian SPD BR51 dikaji menggunakan kromatografi gas-spektrometri massa (GC-MS). Analisis XRD mengesahkan struktur wurtzit heksagon kedua-dua jenis ZnO NP. Analisis TEM mengesahkan ukuran zarah ZnO NP [A] dan ZnO NP [B] masing-masing berada dalam lingkungan 117 hingga 149 nm dan 71.1 hingga 102 nm. FTIR mendedahkan kehadiran ikatan –C–O, =C–H, C = C, –C–O– C, dan O–H dalam kedua-dua jenis ZnO NPs. Parameter operasi terbaik untuk SPD BR51 di AHDBGW direkodkan dengan 0.10 g ZnO NP, pada pH 5 dan dengan 1 ppm BR51. Kajian ini dapat diperhatikan bahawa SPD oleh AHDBGW dan RHDBGW menggunakan ZnO NPs masing-masing mengurangkan kepekatan COD and BOD_5 dengan kadar 80.92% dan 79.14% (COD) dan 82.91% dan 70.18% (BOD_5).

SPD BR51 dalam larutan berair mengikuti kinetik pseudo first-order dengan pemalar kadar purata masing-masing 0.019, 0.014, 0.008 dan 0.005 min^{-1} untuk 5, 10, 15 dan 20 ppm BR51 pada keadaan optimum. Hasil penguraian SPD menentukan bahawa BR51 dapat diuraikan kepada sebatian yang lebih selamat iaitu asid hexadecanoic, metil ester dan asid octadecanoic, metil ester. Oleh itu, sistem kajian didapati berkesan untuk rawatan HDBGW untuk memastikan kebersihan persekitaran untuk generasi akan datang.

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LIST OF SYMBOLS AND ABBREVIATIONS

AFM	-	Atomic Force Microscopy
AGW	-	Artificial Greywater
AHDBGW	-	Artificial Hair Dye Bathroom Greywater
AOP	-	Advanced Oxidation Process
BOD	-	Biological Oxygen Demand
BR51	-	Basic Red 51
CB	-	Conduction Band
COD	-	Chemical Oxygen Demand
DO	-	Dissolved Oxygen
DOC	-	Dissolved Organic Carbon
EDX	-	Energy-Dispersive X-ray Spectroscopy
EQA	-	Environment Quality Act
Eg	-	Band Gap Energy
FESEM	-	Field Emission Scanning Electron Microscopy
FWHM	-	Full Width Half Maximum
FTIR	-	Fourier Transform Infrared Spectroscopy
GC-MS	-	Gas Chromatography Mass Spectrometry
HDBGW	-	Hair dye bathroom greywater
h	-	Planck’s constant
k	-	Scherer’s constant
LLE	-	Liquid-Liquid Extraction
NPs	-	Nanoparticles
NR	-	Not Reported
Ns	-	Nanosecond

O_2	-	Oxygen
O_2•-	-	Superoxide anion radical
OCP	-	Organochlorine Pesticide
OH•	-	Hydroxyl Radical
PCP	-	Personal Care Products
PPCP	-	Pharmaceuticals and Personal Care Products
PZC	-	Point of zero charge
RHDBGW	-	Real Hair Dye Bathroom Greywater
ROS	-	Reactive oxygen radicals
SPD	-	Solar Photocatalytic Degradation
TEM	-	Transmission Electron Microscopy
TGA/DSC	-	Thermogravimetric Analysis / Differential scanning calorimetry
TiO_2	-	Titanium Dioxide
TS	-	Total Solids
TSS	-	Total Suspended Solids
TN	-	Total Nitrogen
TP	-	Total Phosphorus
UV	-	Ultraviolet
UV-DRS	-	UV-Vis Diffuse Reflectance Spectra
UV-Vis	-	Ultraviolet–visible spectroscopy
UV-Vis NIR	-	UV-Visible Near Infrared
VB	-	Valence Band
XOC	-	Xenobiotic Organic Compound
XRD	-	X-ray Diffraction
XPS	-	X-ray Photoelectron Spectroscopy
Zn^{2+}	-	Zinc ion
ZnO	-	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Background of the research

The rapid increase in the global population has associated with high pollution of the water resource and increased the demand for clean water. It has been indicated that 90 % of the wastewater produced globally is discharged without treatment, thereby exacerbating the level of pollutants in the natural water bodies (Karlsson *et al*., 2013). The domestic greywater represents one of the main point sources of water pollutants (Mohamed *et al*., 2018). Greywater has pH (6.4-8.1), chemical oxygen demand (COD) (100-633 mg/L), biological oxygen demand (BOD) (50-300 mg/L), total suspended solids (TSS) (7-505 mg/L), turbidity (44-375 mg/L), total nitrogen (TN) (3.6-19.4 mg/L), total phosphorus (TP) (0.11-48.8 mg/L), and total coliforms ($10\text{-}2.4 \times 10^7$ CFU/100 mL) which is lower than that reported in the blackwater (Abdel-Shafy *et al*., 2019). However, greywater contains a qualitative pollution such as xenobiotic organic compounds (XOCs) resulted from the utilization of personal care products (PCPs) such as hair dyes, soap, shampoo, toothpaste and cleaning products (Patel *et al*., 2020).

XOCs are emerging contaminants containing low level of pollutants in water which can cause harmful effect on the environment and ecosystem. For instance, polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), polycyclic aromatic hydrocarbons (PAHs), polychlorinated dibenzofurans (PCDFs) and polychlorinated dibenzo-p-dioxins (PCDDs) are among XOCs which cause impaired reproduction and sexual anomalies for aquatic and terrestrial ecosystems (Rochman *et al*., 2013; Oteng-Peprah *et al*., 2018). Moreover, XOCs such as colorant compounds in hair dye products have been proved to be mutagenic, carcinogenic, toxic to mammals and can be absorbed percutaneously (Fraga *et al*., 2017). The synthetic dyes with azo-aromatic chromophore group such as Basic Red 51 (BR51) has high stability in the environment (Abe *et al*., 2018). Coagulation and flocculation-sedimentation, adsorption, electrochemical techniques and fungal decolonization are among the methods which have a significant contribution in removing the dyes from the wastewater (Ghaedi *et al*., 2014). Nonetheless, these methods have limitations in their application such as cost-ineffective as well as generation secondary by-products which need further treatment (Hameed *et al*., 2011). Therefore, an appropriate treatment of bathroom greywater technologies should be introduced.

One of the most recent technologies for the degradation of XOCs is the photocatalytic degradation which is one of most environmentally friendly advanced oxidation process (AOP). This technology utilizes the semiconductor/catalyst such as titanium dioxide (TiO_2) and zinc oxide (ZnO) to oxidize harmful organic chemicals. Photocatalytic degradation has gained significant attention due to its ability to degrade a wide range of the recalcitrant compound. Photocatalytic degradation is a term used to define the reactions that occur in a semiconductor and its ability to absorb a photon of light more energetic than its band gap (Ghosh *et al*., 2019). In recent times, ZnO is one of the most extensively studied multifunctional nanocrystalline semiconductors. As a result, it attracts significant attention based on numerous applications in luminescent, solar cells, electrical and chemical sensors due to the wide band gap (3.2 eV) (Mirzaei *et al*., 2016). Moreover, the semiconductor materials from the nano-sized particle have received much more interest in recent years for their advantageous properties such as the large surface area to volume ratio, thermal and electronic properties (Surya *et al*., 2019). ZnO NPs have enormous advantages including high UV absorption, large surface area, catalytic activity and long life span that can be applied to a catalytic reaction process (Fowsiya *et al*., 2016). In addition to that, green synthesised ZnO NPs and their mediated azo dye degradation are the latest and effective methods used for treatment of hazards effluent samples (Sreedharan *et al*., 2019). Green synthesis is the bottom-up approach where the metal precursors is reduced by plant extracts and this method is eco-friendly and nontoxic to the environment (Kumar *et al*., 2019). Besides, green synthesis utilises pollutant-free chemicals and solvents such as water and natural extracts to synthesise NPs (Gnanasangeetha and Saralathambavani, 2013).

Plant-mediated NPs synthesis has gained much attention since the plant itself could act as both reducing and capping agent as it contains phytochemicals such as terpenoids, flavonoids, alkaloids and phenols (Deepak *et al*., 2019). Therefore, in this study, *Coriandrum sativum* was explored as an alternative reducing and capping agent for synthesizing ZnO NPs due to its abundance in phytochemicals such as flavonoids and terpenoids which are responsible for the synthesis of metal NPs (Senthilkumar *et al*., 2018). Hence, the aim of this study is to formulate ZnO NPs by green method to be used in the solar photocatalytic degradation (SPD) of BR51 in artificial hair dye bathroom greywater (AHDBGW).

1.2 Problem statement

Among various PCPs, hair dyes (Basic Red 51, BR51) are widely used to groom ourselves either for beauty or youthful looking. BR51 is utilized in formulations of direct hair dye at concentrations up to 20000 µg/mL. Lately, BR51 has been studied by the European Union (EU) and it tested positive for gene mutation in the prokaryotic cells (Zanoni *et al*., 2014). Even though hair dyeing is very pertinent in this modern era, significant amount of greywater is produced from hair dyeing. This greywater contains huge quantity of dye that can cause effects on the aquatic organisms, water resources, soil fertility and human health through food chains. The existing treatment technologies for dye greywater such as constructed wetland, activated sludge and filtration focus mainly on removing the basic parameters in terms of BOD, COD, TSS, turbidity and pH. In contrast, dyes removal requires an advanced treatment technology such as advanced oxidation process (AOPs). Solar photocatalytic degradation (SPD) using ZnO NPs is one of AOPs with high efficiency due to its high activity and high potential to adsorb UV irradiation.

Nonetheless, the synthesis of ZnO NPs by chemical methods has huge tendency of aggregation due to the high surface area-to-volume ratio which lead to reduce its photocatalytic activity. Furthermore, ZnO has a high recombination rate of the photo-generated electron-hole pairs ($e^- + h^+$) and photo-instability in aqueous solution leading to drastically decrease the photocatalytic activity of pure photocatalyst. The chemicals used for synthesis of ZnO NPs and for their stabilization are toxic and lead to hazardous by products. These gaps offer an opportunity for the researches to find alternative method to synthesize non-toxic and stabilized ZnO NPs.

The green methods are the best alternative for producing more bio-compatible NPs. *C. sativum* contains phytochemicals with antioxidant properties responsible for the synthesis of metal and metal oxide NPs. *C. sativum* contains polyol which act as a both reducing and capping agent for the NPs. Moreover, most of the researchers proved that green leaves are most suitable to synthesize ZnO NPs as they are the site of photosynthesis and availability of more H^+ ions to reduce the zinc acetate dehydrate (precursor) into ZnO NPs. *C. sativum* leaf extract as a both reducing and capping agent in the synthesis ZnO NPs to reduce aggregation is not widely reported, thus this process was conducted in objective one.

Next, since the main factors such as ZnO NPs loadings, pH and initial BR51 concentration were selected to be the manipulated variables as they have dominance effects and work differently on different type of wastewater samples, thus, the optimization and kinetic studies were needed in this study and achieved in objective two and three. The photocatalytic degradation of hair dye bathroom greywater is considered a few, since most of the researchers preferred to bathroom greywater instead of hair dye bathroom greywater. Furthermore, the degradation pathway of BR51 by green ZnO NPs still unclear as different catalyst exhibited a distinctive degradation pathway and therefore the degradation pathway of BR51 was studied in objective four. Thus, synthesis of ZnO NPs using leaf extract of *C. sativum*, as well as the optimization and kinetics model of the photocatalytic degradation of BR51 in artificial hair dye bathroom greywater (AHDBGW) using ZnO NPs with the explanation for the degradation pathway has not reported before and this emphasizes the novelty in the current work.

1.3 Hypothesis of the research

The current research hypothesized that ZnO NPs prepared by green synthesis approach using *C. sativum* leaves have novel characteristics which can contribute effectively in the efficiency of ZnO NPs for SPD. It is hypothesised that ZnO NPs increase the solar absorbance and accelerate the SPD process of BR51. It is hypothesised that the use of SPD by ZnO NPs able to degrade BR51 in hair dye bathroom greywater (HDBGW). Lastly, SPD is expected to reduce the pollution load in HDBGW effluent and meet the discharge limit.

1.4 Objectives of the research

The objectives are the following;

i. To formulate ZnO NPs synthesized by leaf extract of *Corriandrum sativum*
ii. To optimize the best operating parameters for solar photocatalytic degradation of BR51 in artificial hair dye bathroom greywater
iii. To determine the kinetics model of BR51 by solar photocatalytic degradation
iv. To determine the solar photocatalytic degradation pathway of BR51

1.5 Research questions

Central to the issues of solar photocatalytic degradation of BR51 in HDBGW, the main research questions developed for this research are:

i. What are the characteristics of ZnO NPs synthesized by leaf extract of *C. sativum*?
ii. What are the best operating parameters for solar photocatalytic degradation process of BR51?
iii. How BR51 response for solar photocatalytic degradation (kinetics model)?
iv. What are the degradation products of BR51 (Degradation pathway of BR51)?

1.6 Scopes of the research

The study tends to focus on the efficiency of SPD of BR51 using green synthesized ZnO NPs. The HDBGW were obtained from four houses in Taman Universiti which were ascertained using questionnaire while AHDBGW was prepared in the laboratory (Wurochekke, 2017). The extraction of *C. sativum* leaves, UV-Vis absorption analysis of *C. sativum* leaf extract, phytochemical analysis of *C. sativum*, fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis and differential scanning calorimetry (TGA/DSC) of *C. sativum*. The synthesis of ZnO NPs was conducted by green synthesis using precipitation method at two different temperatures which are at 100°C [ZnO [A]] and 550°C [ZnO [B]]. The characteristics of ZnO NPs were determined using Ultraviolet–visible (UV-Vis) absorption spectroscopy, field emission scanning electron microscopy with energy dispersive X-ray spectroscopy (FESEM-EDX), transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray powder diffraction (XRD), FTIR,TGA/DSC, X-ray photoelectron spectroscopy (XPS) and Raman analysis. The point of zero charge (PZC) and phytotoxicity of ZnO NPs were evaluated.

The characteristics of real hair dye bathroom greywater (RHDBGW) collected from sampling area and the prepared AHDBGW are discussed. Identification of XOCs in RHDBGW by gas chromatography-mass spectrometry (GC-MS) was conducted. The factors that affect the SPD such as ZnO NPs loading (0.05, 0.10, 0.15, 0.20 g), pH (3, 6, 9) and initial BR51 concentration (1, 5, 10 ppm) were optimized under direct sunlight irradiation. Moreover, COD, BOD, TSS, turbidity removal efficiency and final pH were determined. Reusability performances of ZnO NPs in degradation of AHDBGW under optimized condition were investigated. Moreover, characterizations of ZnO NPs after SPD of AHDBGW [ZnO [AP]] were explored. Phytotoxicity analysis of AHDBGW, RHDBGW and treated AHDBGW and RHDBGW were analysed.

The SPD kinetic models of BR51 were studied by varying the ZnO NPs loadings (0.05, 0.10, 0.15, 0.20 g), pH (3, 5, 7, 9, 11), and initial BR51 concentrations (5, 10, 15, 20 ppm). This is followed by evaluation of reusability performance of ZnO NPs in SPD of BR51 in aqueous solution under optimized condition. Phytotoxicity analysis of BR51 and treated BR51 were investigated. Lastly, the degradation pathways of BR51 during SPD were investigated by GC-MS to explain the role of ZnO NPs in the degradation process.

1.7 Significance of the research

The present study is in line with the government policy in which 11th Malaysia Plan (11MP) indicated that by 2020, 99% of the population will have clean and treated water. Moreover, it has mentioned that the nanotechnology research will focus on the development of applications in the energy sector and environment, medical and healthcare, electronics and systems and food and agriculture. This research is in consistence with the future directions of the Government of Malaysia for protecting environment and biodiversity of the natural waters. Since the country has adopted a regulation of wastewater, this project will contribute effectively in the improving the quality of natural water in the country, based on the strategy as mentioned by Policy Directions for Core Area 2: Water Resources Sustainability in Target 9 To 11 (Strategy 15-18).

The current work focused on finding a green alternative technique to remove XOCs which represent the main challenges in the greywater. The technique used in this study will be helpful to remove those contaminants from greywater more efficiently than the traditional methods (constructed wetland and sand filtration column) which have many disadvantages and are insufficient to totally remove these pollutants from greywater within a single treatment process. Photocatalytic degradation of organic pollutants is promising technology owing to its benefits of degradation on pollutants instead of their transformation under ambient conditions. Since our country is located near the equator, Malaysia's climate is categorized as equatorial, being hot and humid throughout the year making the SPD possible throughout the year with free access to the presence of sunlight. Moreover, photocatalytic degradation induced by solar light has gained wide recognition due to its concession with the idea of green chemistry in encouraging advanced technologies. This process is able to remove a wide range of micropollutants such as personal care products, hormones, industrial chemicals, cosmetics, pharmaceuticals and storm water runoff from cities and organic pollutants such as pesticides and herbicides. To follow and support Malaysia's national environmental policy, this research will help our government and nation to deal with polluted water which is increasing at an alarming rate. Since Malaysia is one of the most visited tourist countries in the world, the protection of the coastline and natural water bodies is one of the main considerations for the country.

1.8 Thesis structure

There are 5 chapters in this thesis summarized as below:

Chapter 1 presents a brief introduction to greywater, photocatalytic degradation, ZnO NPs as a potential catalyst and *C. sativum* as a capping agent. Additionally, Chapter 1 explains the issues this research attempts to tackle, summarizes the objectives and presents a brief overview of this thesis. It also includes the background, problem statement, objectives, scope, significance of the study and organisation of the thesis.

Chapter 2 presents the literature reviews of previous studies that are useful for this research work, background study of greywater treatment, azo dyes in greywater, ZnO NPs as photocatalyst, green synthesis of ZnO NPs, photocatalytic degradation and its mechanism are covered. From the literature reviews, information is gathered for the use and justifications or the experiment conditions and results.

Chapter 3 explains in detail of the method development used in this study including sampling locations, characteristics of hair dye bathroom greywater samples, identification XOCs present in hair dye bathroom greywater samples using GC-MS, preparation of ZnO NPs and SPD experiments.

Chapter 4 presents the results obtained from the experiments and also the analysis of the results. This chapter covers the chemical properties of the ZnO NPs produced. Other than that, the efficiency and the removal mechanism of BR51 from hair dye bathroom greywater and aqueous solution were discussed.

Chapter 5 concludes the findings from based on the objectives of this study. In order to improve this research work, several recommendations were also suggested.

CHAPTER 2

LITERATURE REVIEW

2.1 Characteristics of greywater

Greywater is defined as the wastewater produced from the household's activities such as laundry, showers, bathing, washing basins and kitchen sinks, except the toilet wastewater (black water) which is input to the sewerage system. Greywater represents 60-75% of the domestic wastewater typically with a relatively low content of nutrients and pathogens (Ding *et al*., 2017; Zhu *et al*., 2018). Some sanitary specialists described the greywater as water that is lower in quality than potable water, but of higher quality than black water (Leong *et al*., 2018). The term greywater is used because the colour of the water turns grey when it is stored without treatment, although laundry greywater has a grey colour without the storage (Chaillou *et al*., 2011). Greywater has a high volume with a lower level of pollution. The physical and chemical characteristics of greywater are similar to dilute sewage. Therefore, it contains the similar contaminants such as organic compounds, nutrients and pathogens. Furthermore, its chemical oxygen demand (COD) and the five-day biological oxygen demand (BOD_5) ratios are generally around 4:1 indicating a high chemical content (Shaikh and Ahammed, 2020). However, pathogens and nutrients such as phosphorus and nitrogen are usually lower than in industrial wastewater (Etchepare and van der Hoek, 2015). In general, greywater is divided into four categories which are laundry, bathroom, washbasin and kitchen as illustrated in Figure 2.1.

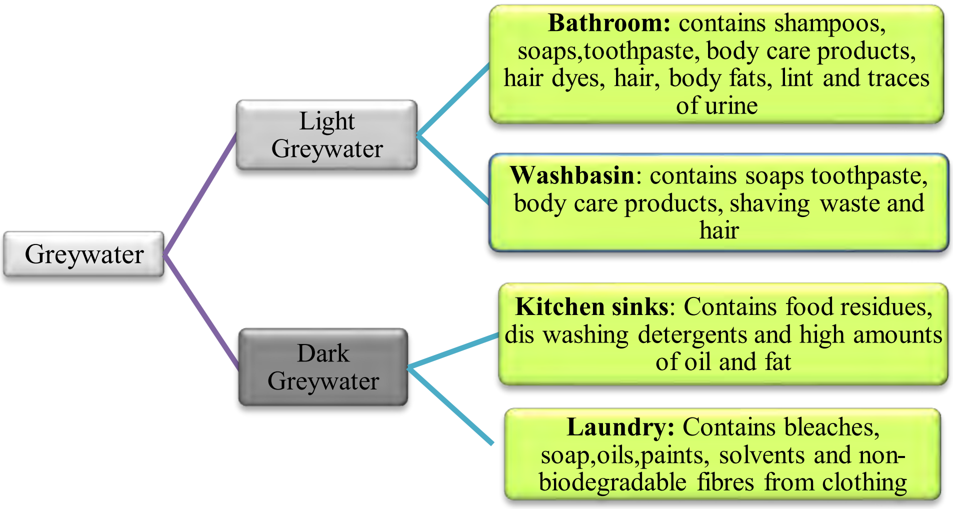

Figure 2.1: Types and sources of greywater (Arden and Ma, 2018)

The quantity of generated greywater is vary depending on the number of residents, age and water usage pattern. Other factors include time of usage, availability of water and lifestyle of households (Barişçi and Turkay, 2016). For instance, per capita production of greywater is 15-55 L per person and varies from 90 to 120 L in a day (Eslami *et al*., 2018). The major chemical contaminants detected in the laundry and bathroom greywater are surfactants (anionic, cationic and amphoteric) coming from shampoos and detergents. Other sources include high levels of chemicals from soaps (such as sodium, phosphorous, surfactants and nitrogen), organic matter, suspended solids and turbidity (Barişçi and Turkay, 2016). The major volume share of greywater is produced in bathrooms (bathtub and hand basin) (Noutsopoulos *et al*., 2017). Wastewater from clothes, washing machines, showers, tubs and bathroom is expressed as light greywater as it has highest volume and the lowest pollutant concentration (Thompson *et al*., 2017). The general characteristics of bathroom greywater such as pH, BOD, COD, total nitrogen (TN), total phosphorus (TP), total solids (TS) and *E. coli* that has been reported by the previous researchers are presented in Table 2.1.

Table 2.1: Physicochemical properties of bathroom greywater from previous researchers

Bathroom Greywater							Researches
Parameter							
pH	BOD (mg/L)	COD (mg/L)	TN (mg/L)	TP (mg/L)	TSS (mg/L)	*E. coli* (CFU/100 mL)	
9.9	NR	848	17	60.6	377.5	NR	Raphael *et al.* (2020)
9.7	120	554	14	8	NR	NR	Patel *et al.* (2020)
7.03±0.14	380±7.6	505±10.1	NR	12±0.24	155±3.1	NR	Abdel-Shafy *et al.* (2019)
6.18-6.35	NR	107.9-112	3.9-5.1	NR	66.67-86.92	NR	Mohamed *et al.* (2018)
7.5 ± 0.1	263±83	390 ± 125	2.7 ± 2.2	0.10 ± 0.14	73.5 ± 38	NR	Noutsopoulos *et al.* (2017)
7.2-7.6	NR	112-346	1.0-7.2	0.19-3.0	38-99	NR	Thompson *et al.* (2017)
NR	123.1	271.8	50.3	5.3	155.8	NR	Chrispim and Nolasco (2016)
6.4-8.1	50-300	100-633	3.6-19.4	0.11- 48.8	7-505	$10\text{-}2.4\times10^{7}$	De Gisi *et al.* (2016)
6.4-10	NR	26-645	3.6-21	0.1-101	7-250	$0\text{-}3.4\text{x}10^{5}$	Fountoulaks *et al.* (2016)
6.30 ± 0.42	NR	15.0 ± 0.8	NR	NR	305.0 ± 6.0	NR	Eze *et al.* (2015)
NR	75.1 ± 4.1	433±4	NR	NR	NR	NR	Grčić *et al.* (2015)

Table 2.1 (continued)

Bathroom Greywater							Researches
Parameter							
pH	BOD (mg/L)	COD (mg/L)	TN (mg/L)	TP (mg/L)	TSS (mg/L)	*E. coli* (CFU/100 mL)	
6.13	349	445	NR	NR	81	NR	Teh *et al*. (2015)
6.1- 6.4 ± 0.21	40 ± 0.25 - 105 ± 0.42	445 ± 2.52 - 621 ± 4.02	10 ± 2.90 - 38 ± 0.56	3 ± 0.87 - 20 ± 1.76	78 ±4.55 - 163 ± 7.12	NR	Mohamed *et al*. (2014)

*NR = Not Reported

*BOD = Biological Oxygen Demand, COD = Chemical Oxygen Demand, TN= Total Nitrogen (TN), TP = Total Phosphorus, TSS = Total Suspended Solids

It can be noted from the data presented in Table 2.1 that the bathroom greywater specifications are different depending on the type of the product used by the residents. For example, Mohamed *et al*. (2018), revealed that the ranges of bathroom greywater parameters were ranged from 6.18 to 6.35 for pH, 107.9-112 mg/L for COD and 66.67- 86.92 mg/L for suspended solids and 3.9-5.1 mg/L for TN. Thompson *et al*. (2017) mentioned that greywater has pH 7.2-7.6, 112-346 mg/L of COD, 1.0-7.2 mg/L of TN, 0.19-3.0 mg/L of TP and 38-99 mg/L of TSS. Besides that, Chrispim and Nolasco (2016) analysed that COD of for bathroom greywater was 271.8 mg/L, 123.1 mg/L for BOD, 50.3 mg/L for TN, 5.3 mg/L for TP and 155.8 mg/L for TSS. These differences in parameters could be related to the geographical location, nature of living, social habits demographics, water usage patterns, types of personal care products used, level of occupancy and time. Bathroom greywater comprises xenobiotic compounds originating from soap, shampoo, hair dyes, toothpaste and other cleaning products (Edwin *et al*., 2014).

2.1.1 Hair dye bathroom greywater (HDBGW)

Bathroom greywater contains hair colorant components from the utilization of hair dye that are categorized as hazardous micro-pollutants which are classified as xenobiotic organic compounds (XOCs) group. Bathroom greywater containing hair dye compounds should gain significant attention as dyes have high tectorial values and even release of less than 1 ppm of dye into the water bodies can cause modifications in the physical characteristics of water (Noman *et al*., 2019). However, very limited researches are available regarding treatment of hair dye compounds in bathroom greywater. Bessegato *et al*.,(2018) revealed that the hair dye wastewater has low conductivity (164.0 ± 1.4 µS), pH 7.31 ± 0.12 and high levels of TOC (169.4 ± 0.72 mg/L), COD (370 ± 2 mg/L), colour (347.3 ± 6.1 mg PtCo/L) and turbidity (64.83 ± 2.5 NTU).

The hair dye compounds in the bathroom greywater are alike which is found in the original products or secondary products are produced due to partial degradation after combining with greywater (Noman *et al*., 2019). Despite the fact that hair dyeing process occurs occasionally in a single house, a huge quantity of greywater is being generated during washing and rinsing phases. According to Chong *et al*. (2015), the concentration of dye in household greywater ranges from 1-10ppm. It should be noted that HDBGW is more polluted and lethal than regular bathroom greywater produced on daily basis which will end at local wastewater treatment plant (Grčić *et al*., 2015). Thus, it is clear that the HDBGW grants an adequate amount and its treatment needs to be explored distinctly. Evidently, the treatment of HDBGW is a great environmental and public health risks. However, they are not receiving sufficient consideration within scientific community (Bessegato *et al*., 2018).

Hair dyes belong to the group of aryl amines (Handa *et al*., 2012). Synthetic hair dyes are widely used in the cosmetic industry worldwide. Hair dyes are available as semi-permanent, permanent and temporary. Oxidative or permanent dyes are most frequently used due to its ease of application, stability, high diversity of coloration, versatility, long permanence effect and represent about 80% of hair dyes available in the market (Hudari *et al*., 2014; Guerra *et al*., 2017). Permanent hair dyes are not coloured or slightly coloured compounds but coloured complexes are created when they are mixed in with an oxidant such as hydrogen peroxide and ammonia (as alkaline) through oxidative condensation between a coupler and intermediate. These coloured complexes will penetrate the hair's cortex giving the preferred shade (Guerra *et al*., 2017). Permanent hair dyes are hardly washed off through shampooing (Kim *et al*., 2016). Hair coloration by permanent hair dyes necessitates three key parts. First is the primary intermediate, an o- or p-substituted (hydroxy or amino) aromatic amine such as p-phenylenediamine and p-aminophenol. The second part is the coupler which is an aromatic compound with electron-donating groups positioned meta to each other such as resorcinol, m-phenylenediamines and naphthols (Zanoni *et al*., 2018). Couplers are categorized into three components based on the colour obtained in the fibre with the primary intermediates: blue, yellow-green and red (Zanoni *et al*., 2014). The third component is the oxidant (hydrogen peroxide) which oxidizes the primary intermediates in the presence of ammonia and lightens the natural hair colour. However, lower level of bleaching can reached by a "no- ammonia" alkalizer, usually monoethanolamine (Morel and Christie, 2011).

Semi-permanent dyes are being used directly on the hair without demanding colour development by oxidative chemical reactions. They are often di- or trisubstituted with amino, derivatives of nitrobenzene, hydroxyl and alkoxy electron donor groups (Rollison *et al*., 2006). Meanwhile, temporary hair dyes are developed with combinations of the non-oxidative hair dyes which can be categorized into acid dyes, basic dyes and nitro dyes. Semi-permanent dyes are removable in 4 to 12 shampooing whereas temporary dyes can simply removable in one shampoo rinse (Chisvert *et al*., 2018). This is because smaller molecules of semi-permanent hair dyes effortlessly penetrate into the hair's cortex but drawn-out easily in following washes meanwhile larger molecules of temporary hair dyes are incapable to penetrate into the hair's cortex and hence washed out easily in single wash. These both types of dyes are depending on van der Waals forces for adhesion; therefore, they do not need chemical reactions to transmit colour (Kim *et al*., 2016). Table 2.2 shows the types of hair dyes and ingredients in hair dye formulations.

Table 2.2: Types of hair dyes and ingredients in hair dye formulations (Habib and Ali, 2020)

Hair dye type	Ingredients
Temporary	Azo derivatives Azine derivatives Thiazine derivatives Indoamines Triphenylmethane
Semi-permanent	Nitroanilines Nitrophenylenediamines Nitroaminophenols Azo derivatives Anthraquinone
Permanent	Para-phenylenediamine Para-toluylenediamine Substituted para-diamines Ortho-or para-aminophenols

2.1.2 Toxicity of hair dye compounds

The releases of synthetic dyes into the environment are poisonous, carcinogenic and mutagenic to living organism and even can kill aquatic organisms (Zhao *et al*., 2018). High volume of dye wastes is detrimental to aquatic life in the sea, rivers and lakes as it prevents the penetration of light and disrupt the biological processes in water bodies. Twenty-two hair dyes have been banned since 2006 for consumer safety (Zanoni *et al*., 2014). Dyes are classified as harmful to humans and environment as they reductively cleave and form aromatic amines which are highly carcinogenic when in contact with saliva, sweat and gastric juices and has potential to accumulate in food chains (Chequer *et al*., 2011; Lebechi *et al*., 2018). Besides that, hair dye products contain many chemicals which can cause allergic reactions such as redness, sores, itching, burning sensation, and discomfort for some people. Skin irritation can appear around neck, forehead scalp, eyelids and ears. These allergic reactions and skin irritation may not happen instantly after coloration processes but noticeable after a few hours or even a day later (Kim *et al.*, 2016).

Cardona *et al*., (2015) have stated that BR51 are used to formulate the black colour dyes which are capable of causing cytotoxic and genotoxic effects in human cells even at very low concentration such as these used for hair coloration processes as detected in the vitro experiments. Garrigue *et al*., (2006) reported that hair dyes are mutagenic and carcinogenic *in vitro* meanwhile Genina *et al*., (2002) revealed that hair dyes compounds able to penetrate the human skin. Moreover, Liu *et al*., (2019) showed that women who prolonged use of hair dyes have a great risk of ovarian cancer and non-Hodgkin's lymphoma and multiple myeloma. Maiti *et al*., (2016) have studied the effect of dyes on *Escherichia coli*, *Allium cepa* bulbs, human red blood and white blood cells. Abe *et al*. (2018) observed that exposure of BR51 dyes decrease the energy consumed of zebra fish embryos for their locomotor activity. Luna *et al*., (2014) proved that azo dyes are toxic to *Daphnia similis* while Jong *et al*.,(2016) have observed toxicity in *Hydra attenuate*. These findings confirmed that hair dyes were cytotoxic and cause mutagenic effect on living organisms regardless of microbes, plant and animal.

2.1.3 Artificial / Synthetic greywater

Artificial or synthetic greywaters is produced by mixing various chemical products used by households and/or chemicals identified to exist in real greywater. Hence, the water quality of generated greywater is controlled by these chemical products (Abed *et al*., 2017). Artificial greywater (AGW) is frequently produced for research studies as real greywater (RGW) due to the problem with the long-term storage as RGW degrades and changes its composition (Thompson *et al*., 2017). AGW need to be produced that matches with the RGW in terms of physicochemical parameters and representative commercial personal care products (PCPs) such as shampoo, hair conditioner, face cleansing and shower gel, toothpaste, laundry detergent and fabric softener that contain wide range of chemicals and constituents such as emulsifiers, colouring agents and preservatives (Tsoumachidou *et al.*, 2017). The formulated AGW should match the RGW to ensure effectiveness in treating RGW (Thompson *et al*., 2017). Various AGW recipes have been reported in the literature (Table 2.3).

However, in this study, AGW was formulated according to Wurochekke, (2017) who produced AGW recipe based on the typical bathroom products.

Table 2.3: Artificial greywater recipe by previous researchers

Ingredients/Compounds	Nghiem *et al.* (2006)	Saumya et *al.* (2014)	Chrispim and Nolasco (2016)	Eslami *et al.* (2017)
Humic acid	20 mg/L	-	-	-
Kaolin 50	50 mg/L	-	-	-
Cellulose	50 mg/L	-	-	-
Calcium chloride	0.5 mM	-	-	-
Sodium chloride	10 mM	-	-	-
Sodium bicarbonate	1mM	-	0.0094 g/L	25 mg/L
Shampoo	-	10g/L	0.1007 g/L	720 mg/L
Body lotion	-	-	0.159 g/L	-
Toothpaste	-	10g/L	0.016 g/L	32.5 mg/L
Deodorant	-	-	0.0106 g/L	10 mg/L
Conditioner	-	-	0.1113 g/L	-
Soap	-	10g/L	0.1219 g/L	-
Laundry Detergent	-	-	0.188ml/L	150 mg/L
Softener	-	-	0.0986 g/L	-
Lactic acid	-	-	0.0168 g/L	-
Sodium sulfate	-	-	0.0188 g/L	35 mg/L
Sodium hydrogen phosphate	-	-	0.0188 g/L	39 mg/L
Shower gel	-	10g/L	-	-
Shaving cream	-	10g/L	-	-
Moisturising cream	-	10g/L	-	10-15 mg/L
Make up	-	10g/L	-	-
Make up remover	-	10g/L	-	-
Secondary effluent	-	-	-	20ml/L
Boric acid	-	-	-	1.4mg/L
Glucose	-	-	-	28mg/L
Clay (Unimin)	-	-	-	50 mg/L
Vegetable oil	-	-	-	7 mg/L

2.2 Greywater treatment technologies

The improper discharge of greywater from households influence the water supply. Furthermore, untreated greywater can form puddles and small pools that create a suitable environment for proliferation of breeding insects, pests and pathogenic bacteria (Mohamed *et al*., 2014). The precise selection of the most proper technology relies on the socio-economic factors, cost of water, scale of operation and end use of the water (Gisi *et al*., 2016). Greywater treatment methods include physical system such as filtration, screening and ultra-filtration (UF) membranes. The chemical systems include ion exchange resins and coagulation/flocculation while the biological systems include activated sludge systems, constructed wetlands, rotating biological reactor (RBC), aerobic and anaerobic bio-filters and membrane bioreactor (MBR). Other bio-based system includes bio-rotor and submerged aerated filters, sequencing batch reactor (SBR) and bio-rolls as presented in Figure 2.2 (Barişçi and Turkay, 2016; Prajapati *et al*., 2019). In this section the physical, biological and chemical treatment used for the greywater treatment are reviewed.

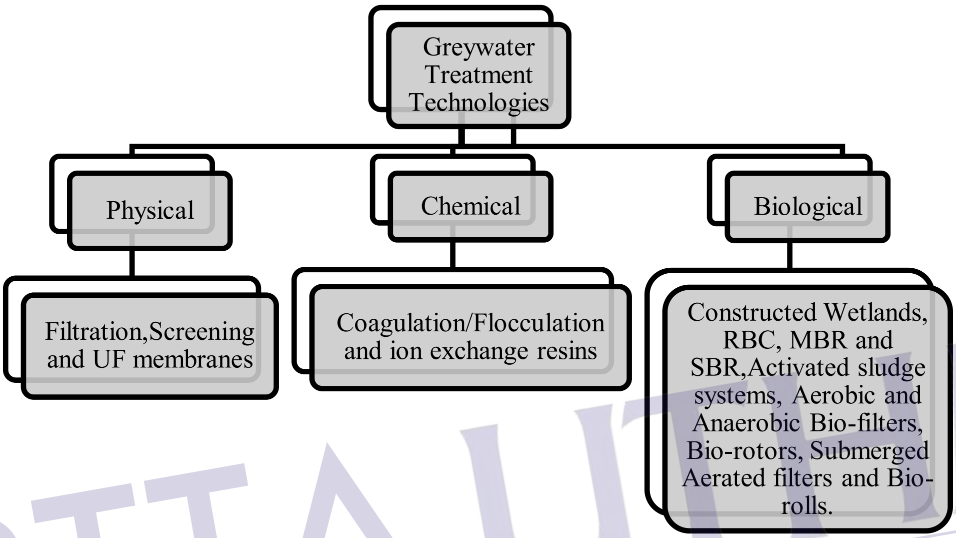

Figure 2.2: Greywater treatment technologies

2.2.1 Physical treatment of greywater

Physical treatment systems are most common methods used for the greywater treatment especially with low organic strength (Antonopoulou *et al*., 2013). However, the methods should be followed by the disinfection process to improve the microbiological quality of the treated greywater (Barişçi and Turkay, 2016). Mohamed *et al*., (2018) conducted filtration of BGW by ceramic waste and observed the reduction of COD, TSS, TN and turbidity was 38.8%, 58.47%, 66.66% and 88.3% respectively. The lower percentage removal of COD and TSS by the physical treatment process necessitate the further treatment.

Physicochemical treatments such as sedimentation tank, sand filtration column and granular activated carbon column have been investigated for greywater treatment. Noutsopoulos *et al*. (2017) conducted a research using physicochemical treatments such as sedimentation tank, sand filtration column, and granular activated carbon column. The study recorded 78.8% and 94.93 of turbidity and COD removal, respectively. However, high operational cost of the system is needed due to the frequent replacement of the activated carbon.

2.2.2 Biological treatment of greywater

Biological treatment systems are efficient for treating greywater in the wastewater treatment plant. However, many XOCs have been detected in the treated greywater by biological treatment underlines the need for more advanced treatment options (Tsoumachidou *et al*., 2017). For instance, anaerobic treatment is not suitable for removing of XOCs from greywater due to the additional requirement for removing the organisms from the treated wastes. Moreover, this technique has low efficient for reducing COD due to the occurrence of high concentrations of XOCs (Noman *et al*., 2019). The potential for constructed wetlands (CWs) in developing countries for greywater treatment is described as massive. These systems utilize wetland plants, soils and associated microorganisms to remove contaminants from wastewater (Ghaitidak and Yadav, 2013). CWs are economically and energetically efficient way of treating greywater if assessed on a case-by-case basis (Arden and Ma, 2018). However, the CWs need huge areas of land, complex construction and operation. Hence, it is not applicable in tight urban areas due to high space demand. CWs performances are influenced by different factors such as greywater characteristics, climatic conditions, substrate materials and native plant species (Fowdar *et al*., 2017). These factors are considered as the main biological components of CWs which directly or indirectly transform the procedures of primary pollutant removal over time (Gorgoglione and Torretta, 2018). Moreover, CWs occasionally have low nitrogen removal efficiencies because of low oxygen transfer or limited de-nitrification due to low amounts of available organics, especially under high nitrogen loading rates (Wang *et al*., 2020). However, in terms of treatment performance and operation and management costs (O&M), CWs is considered as the most environmentally friendly and cost-effective technology although they require a large space (Gisi *et al*., 2016). Abed *et al*., (2017) revealed that TSS and turbidity values dropped by using scale floating treatment wetlands significantly ($p < 0.05$). However, no significant differences ($p > 0.05$) in the removal of BOD_5 were noted. Saumya *et al*., (2014) assessed a CW system for synthetic greywater treatment and revealed that COD, BOD_5

and turbidity removal was 39.7%, 70% 92.1 % respectively. Uddin *et al*., (2016) revealed that a maximum removal rate was achieved for COD (100%), ammonium ion (NH_4^+) (99%), nitrite ion (NO_2^-) (97%), TSS (97%), phosphate ion (PO_4^-) (87%) and *E. coli* (98%) using CW.

Membrane bioreactors (MBRs) have exhibited an efficiency for COD and TSS but not nutrients reduction (Liu *et al.*, 2015). The most widely used treatment methods are sequencing batch reactors (SBRs), MBRs and biologically aerated filters, which have relatively high potential to produce higher greywater quality compared to the traditional processes such as the primary and secondary processes. However, the energy consumption and capital cost of these methods are relatively high (Jabornig and Favero, 2013 ; Wurochekke *et al*., 2016). Chrispim and Nolasco, (2016) used a moving bed biofilm reactor (MBBR) to improve the efficiencies for organic matter degradation and nutrient removal. The removal of BOD and COD were 59% and 70%, respectively. However, the phosphorus removal was low. Fountoulaks *et al*. (2016) indicated that the high cost of MBR is due to the membrane fouling, accompanied by higher requirements for aeration and consequently, energy demand. Over 50% of the energy consumption in MBRs is expended on air scouring to prevent the fouling of the membranes.

2.2.3 Chemical treatment of greywater

Coagulation-flocculation is one of the chemical treatment methods of greywater. These methods encompass charge neutralisation and floc formation which causes fast settling of the colloidal and suspended particles (Ghaitidak and Yadav, 2015). Pidou *et al*. (2008) used coagulation and ion exchange resin techniques to treat greywater. Coagulation with aluminium salt reduced COD, turbidity, TN and PO_4^{3-} from 791 to 287 mg/L, 46.6 to 4.28 NTU, 18 to 15.7 mg/L and 1.66 to 0.09 mg/L respectively. Meanwhile, by ion exchange resin decreased COD to 272 mg/L, turbidity to 8.14 NTU, TN to 15.3 mg/L and PO_4^{3-} to 0.91 mg/L, respectively. However, the coagulation and ion exchange resin have insignificant effects in removing TN and PO_4^{3-}. Ghaitidak and Yadav (2015) conducted coagulation/flocculation using alum and achieved turbidity, BOD and *E. coli* removal of 88%, 53-77% and 95-99% respectively.

On the other hand, the photocatalytic treatment under artificial and solar illumination showed the best results for improving greywater quality. The studies revealed that the photocatalytic treatment had reduced dissolved organic carbon (DOC) by 72% after 210 min (Tsoumachidou *et al*., 2017). Moreover, the decrease in toxicity, phytotoxicity and biodegradability of the simulated wastewater was observed after solar-induced photocatalytic treatment. Figure 2.3 shows the number of publications on photocatalytic treatment of wastewater (except greywater) by zinc oxide (ZnO) nanoparticles (NPs). It appeared that number of publications on photocatalytic process using ZnO NPs increases over the years. It can be noted that the photocatalytic treatment of the wastewater with using ZnO NPs have received high attention between 2017 and 2019 due its high performance of ZnO NPs in degrading harmful compounds by photocatalytic degradation. However, there were only one journal published in the 2009 and 2013, 2016 and 2017 and two journals in 2016 on the application of photocatalytic treatment for bathroom greywater. Furthermore, there were no journals published on photocatalytic degradation for hair dye compounds in bathroom greywater by using ZnO NPs. Hence, more research is required on treatment of bathroom greywater containing hair dye using ZnO NPs.

Figure 2.3: Publication on photocatalytic degradation treatment of other wastewater and bathroom greywater (source: http://www.sciencedirect.com, 2019, searches terms 'Photocatalytic degradation' and 'bathroom greywater' within these results)

The current technologies used for treating greywater are illustrated in Table 2.4. From all the previous studies, it can be noticed that stacked multi-layer reactors with passive aeration and particle trapping of greywater from the old public bathhouse achieved good removals for turbidity, TSS, COD and microbiological parameters (Prajapati *et al*., 2019). The multi-layer sandwich construction of filter material further reduces the risk of clogging, as sediment is accumulated in a different layer from where water is flowing, allowing for long-term degradation of multiple cycles. The suggested this investigation can turn light greywater into water that is clear and largely free of bulk contaminants, and with a marked reduction in microbial parameters can be reused for high-level purposes, including personal hygiene. However, it was suggested that this method needs further development before its use can be promoted on a large scale, including adequate removal of TN and TP, the addition of a final disinfection step, testing over longer periods than five weeks, optimization of reactor design for maximum water production rate, and a scheme for operation and maintenance. Besides, the evapotranspiration-constructed wetland combined system achieved good removal efficiency of COD, turbidity, total dissolved solids, TS and TSS (Filho *et al*. 2018). The plants presented in the units and the anaerobic digestion tank improve hydraulic and volumetric efficiency, decrease short-circuiting and improve mixing conditions in the system. It is observed that rainfall enables salt elimination, thus increasing evapotranspiration (ET), which promotes effluent reduction and enables the system to have zero discharge when reuse is unfeasible. Moreover, the period with higher evapotranspiration rates enabled a reduction of approximately 32% of the inlet volume, indicating the possible use of the proposed configuration as a zero dis- charge system, by combining several units, if reuse is undesirable or unfeasible.

Ramprasad *et al*. (2017) used Green roof-top water recycling system (GROW) constructed wetland to treat greywater generated from students' hostels showed the best removal efficiencies for COD, BOD_5, FC, TN, TP, TSS, nitrate-nitrogen (NO_3-N), sodium do-decyl sulphate (SDS), propylene glycol and trimethyl amine. It was found that the removal rate was high during summer season compared to other seasons. Also, the removal efficiency was more at higher hydraulic retention time. The promising results from this study may increase the applicability of GROW systems as a robust, cost-effective and reliable green roof systems in India and other tropical countries.

The type of treatment required for greywater depends on the standards limits needed for the disposal or reuse of greywater where it differs from one country to other countries and the economic status of the country. Despite various treatment technologies by in many of developing countries, most of the researchers have only focused on reducing the basic parameters such COD, BOD, turbidity, TDS, TSS, TN and TP. There are very few studies focusing on XOCs removal especially hair dye compounds from the greywater. Few studies such as by Grčić *et al*., (2015) conducted photocatalytic degradation of greywater loaded with hair colorants using TiO_2-coated textile fibers coupled flocculation with chitosan exhibited considerable removal efficiency of COD (90.67%), BOD_5 (76.88%), turbidity (95.52%), TOC (80.33%). Many countries did not execute the legislations for these parameters. Hence, more studies are needed to investigate the removal of hair dye compounds in greywater.

Table 2.4: Review of different technologies used to treat greywater

No	Location	Technology	Greywater source	Removal Efficiency	References
1	Nigeria	Batch-flow free water surface constructed wetland	Campus	COD (81%), TN (85%), TK (82%), TP (10%), pH (0.2%), TSS (81%), Zn (91%), Al (81%), Mg (94%), and Fe (90%)	Raphael *et al*. (2020)
2	Denmark	Stacked multi-layer reactors with passive aeration and particle trapping	Old public bathhouse	Turbidity (95%), TSS (94%), COD (87%), and microbiological parameters (55- 98%)	Prajapati *et al*. (2019)
3	Brazil	Hydraulic and hydrological aspects of an evapotranspiration-constructed wetland combined system	Household	COD (82%), turbidity (91%), TDS (90%), TS (92%), TSS (91%)	Filho *et al*. (2018)
4	Malaysia	Ceramic Waste Filter	Bathroom greywater in village area in Batu Pahat, Malaysia	COD (38.8%), TN (66.66%), TSS (58.47%), turbidity (88.31%),	Mohamed *et al*. (2018)
5	Australia	Biofiltration systems (living wall system)	Synthetic greywater mix	BOD (>90%), TSS (>80%) TN (>80%), TOC (>70%) DOC (70-95%)	Fowdar *et al*. (2017)
6	Morocco	Horizontal sub-surface flow Constructed wetland	Washbasins in the schoolboys	COD (89%), BOD_5 (87%) TN (42%), TP (50%) turbidity (88%)	Laaffat *et al*. (2017)
7	Norway	Biofilter system	Cottages and small households	COD (64%), BOD_5 (61%) TN (85%), TP (88%) TSS (75%)	Moges *et al*. (2017)
8	India	Green Roof-Top Water Recycling System (GROW) constructed wetland	Students hostels IIT Madras campus, Chennai	COD (92.5%), BOD_5 (90.8%), FC (91.4%), TN (91.7%), TP (87.9%), TSS (91.6%), NO_3–N (83.6%), SDS (85.7%), Propylene glycol (93.4%), Trimethyl amine 88.9%.	Ramprasad *et al*. (2017)

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