MULTI STAGE ANAEROBIC REACTOR FOR WASTEWATER TREATMENT

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MULTI STAGE ANAEROBIC REACTOR FOR WASTEWATER TREATMENT

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requirements for the award of the degree of
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DEDICATION

To Allah (SWT), my beloved mother and father, my darling husband and my precious daughters and son Anaz Sofea, Anaz Zafiera, Anaz Aryan Syah, and little baby Anaz Auni Aurora

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ABSTRACT

Staged reactors (two or more stages) in anaerobic digestion (AD) provide a solutions to meet wastewater requirements. Compared with other AD treatment options, staging can more effectively adapt to toxic slugs and improve its treatment efficiency by passing them in less time through the reactor system. In this study, the Multi Stage Anaerobic Reactor (MSAR) was developed and tested with various fundamental experiments, such as start-up performance, ability to withstand low hydraulic retention time (HRT) and high organic loading rate (OLR), generation of biomethane recovery, and ability to tolerate inhibitory compound (tetracycline and papermill wastewater). The start-up process was commenced with a synthetic wastewater (glucose) with an OLR of 0.18 kg COD/m^3/d at HRT of 4 d, and accomplished acclimatization and stable operation for the subsequent phase of the study. The effect of HRT on the process performance of the MSAR was investigated by gradually reducing the HRT from 4 d to 1 d. Results showed that not much difference in terms of COD removal performance. It was observed that when HRT was reduced from 4 d to 1 day, COD removal achieved was from 99.4% to 91.9%, signifying the stability of MSAR at low HRT. Meanwhile, volatile acid (VA) of the MSAR effluent fluctuated from 13 to 276 mg/L as HOAc, confirming no great variations in the profile. A variation ratio (VSS/TSS) from 0.2 to 0.92 in all stages was observed confirming no significant sludges wash out during this phase and the highest generation of methane observed at 0.0153 LCH4/g COD for HRT 4 d in stages. A different scenario was observed during the study of OLR effect on MSAR. The COD removal efficacy declined dramatically from 91% to 55%, 50%, 47% and 16%, when the OLR was increased from 0.79 kg COD/m^3/d to 1.05, 1.31, 1.57 and 1.84 kg COD/m^3/d, respectively. Meanwhile, the average of VA concentration escalated from 306 mg/L to 883, 1066, 1672 and 1581 mg/L as HOAc in the effluent stage (S4). The ratio of VSS/TSS in this study showed a value between 0.31-0.91 in stages. Concurrently, a decreased in methane generation was also observed at 0.0235, 0.0105, 0.0087, 0.0047 and 0.0045 L_{CH4}/g COD in stages. During the study on the effect of inhibitory substances to MSAR, tetracycline was greatly deteriorated the performance of the reactor. At 10 mg/L tetracycline concentration, the COD performance was dropped to 12.33% removal. At the same time, when the papermill wastewater tested, an average COD reduction of 75.8% was observed in this phase. The lethal effect of tetracycline significantly determines the abundance of VA result (2700 mg/L as HOAc), which leads to the inhibition of methane production in stages. An acceptable value of VA ($\leq$ 500 mg/L as HOAc) was observed during the effect of papermill wastewater confirming a potential tolerance to methanogenic microorganisms in this phase. Meanwhile, two theoretical methods for methanogen potential was carried out in this work. These methods use the COD characterization and elemental composition analysis corresponding to the affected parameters (HRT, OLR, tetracycline, and papermill wastewater) to predict methane production rates in stages. The COD characterization analysis observed the highest methane potential at 0.021 L_{CH4}/g COD for 3 d HRT, 0.06 L_{CH4}/g COD at OLR 0.785 kg COD/m^3/d, 0.054 L_{CH4}/g COD at 0.13 mg/L tetracycline concentration and 0.05 L_{CH4}/g COD at OLR of 0.785 kg COD/m^3/d papermill wastewater in stages, respectively. By elemental composition analysis, the highest methane observed was 0.25 L_{CH4}/g COD at 3 d HRT, 0.197 L_{CH4}/g COD at 1.571 kg COD/m^3/d and 0.253 L_{CH4}/g COD at 1.07 mg/L tetracycline concentration in the stages. Therefore, this study offers a potential solution for the use of MSAR to treat wastewater and contributes to the development of anaerobic reactor technology across Malaysian industries.

ABSTRAK

Rektor berangkai (dua atau lebih) untuk penguraian anarobik (PA) menyediakan resolusi dalam menambahbaik syarat rawatan air sisa. Berbanding dengan kaedah PA yang lain, cara ini lebih efektif untuk mengadaptasi sisa toksik dengan meneluskan sisa itu dalam masa yang lebih singkat dan seterusnya meningkatkan kadar cekap sistem. Dalam kajian ini, Rektor Anaerobik Berangkaian (RAB) telah dibangunkan dan diuji dengan kepelbagaian eksperimen asas seperti prestasi permulaan, keupayaan menampung masa tahanan hidraulik yang rendah (MTH) dan kadar bebanan organik (KBO) yang tinggi, potensi gas bio metana, kemampuan merawat komposisi sisa yang tercemar (air sisa tetracycline dan kilang kertas). Prestasi permulaan RAB telah diuji dengan air sisa sintetik (glukosa) dengan nilai KBO, 0.18 kg COD/m^3/hari dengan MTH 4 hari untuk penyesuaian persekitaran dan operasi yang stabil bagi fasa kajian selanjutnya. Keberkesan pengurangan MTH dari 4 ke 1 hari operasi yang dijalankan menunjukkan hanya sedikit pengurangan dalam permintaan oksigen kimia (POK) iaitu dari 99.4% ke 91.9% sekaligus menggambarkan kestabilan RAB terhadap MTH yang rendah. Asid yang meruap (AR) menunjukkan data turun-naik dari 13 ke 276 mg/L (HOAc), menggambarkan tiada variasi yang besar dalam profil hasil. Variasi nisbah pepejal terampai meruap (PTM) dan jumlah pepejal terampai (JPT) dicatat dari 0.2 ke 0.92 di dalam rektor sekaligus menunjukkan tiada enapcemar yang menjeluak keluar. Manakala gas metana yang diperhatikan adalah 0.0153 LCH4/g COD pada MTH 4 hari. Apabila KBO ditingkatkan dari 0.79 kg COD/m^3/hari kepada 1.05, 1.31, 1.57 and 1.84 kg COD/m^3/hari, kadar pengurangan POK menurun secara drastik dari 91% ke 55%, 50%, 47% dan 16%. Purata kepekatan AR adalah 306 mg/L HOAc dari efluen rektor (S4) pada nilai KBO 0.79 kg COD/m^3/hari, dan apabila KBO ditingkatkan kepada 1.05 kg COD/m^3/hari dan seterusnya, AR bertambah kepada 883, 1066, 1672 and 1581 mg/L HOAc. Pencirian enapcemar bagi nisbah PTM dan JPT pada fasa ini adalah diantara 0.31-0.91 di dalam rektor. Kesinambungan dari pertambahan KOB, nilai gas metana berkurangan dari 0.0235, 0.0105, 0.0087, 0.0047 dan 0.0045 $L_{CH4/}$gCOD. Seterusnya, kesan perencatan substrat dikaji terhadap prestasi sistem RAB. Pada kepekatan tetracycline 10 mg/L, sistem menunjukkan kemerosotan secara menyeluruh dan menurunkan POK secara drastik kepada 12.33% sahaja. Apabila sistem diuji dengan air sisa sebenar dari kilang kertas, nilai pengurangan POK dicatat sebanyak 75.8%. Kesan perentan akibat tetracycline menyebabkan peningkatan AR yang ketara (2700 mg/L HOAc) sekaligus menghalang potensi gas di dalam sistem. Namun begitu, kesan perencatan terhadap air sisa kilang kertas mencatatkan nilai AR yang dibenarkan (≤ 500 mg/L HOAc) sekaligus menjelaskan kemampuan mikroorganisma methanogenik bertoleransi terhadap air sisa tercemar di dalam fasa ini. Dua kaedah teori digunapakai dalam kajian ini iaitu kaedah pengkelasan POK dan komposisi elemen untuk jangkaan potensi gas metana terhadap kesan kesinambungan parameter yang diuji (MTH, KBO, tetracycline dan airsisa kilang). Dengan kaedah pengkelasan POK, nilai tertinggi bagi gas metana ialah 0.021 L_{CH4}/gCOD pada 3 hari MTH, 0.06 L_{CH4}/gCOD pada KBO 0.785 kg COD/m^3/hari, 0.054 L_{CH4}/gCOD pada 1.3 mg/L kepekatan tetracycline dan 0.05 L_{CH4}/gCOD dengan nilai KBO, 0.785 kg COD/m^3/hari dicatatkan. Dengan kaedah komposisi elemen, gas metana tertinggi adalah 0.25 L_{CH4}/gCOD pada 3 hari MTH, 0.197 L_{CH4}/gCOD terhasil pada KBO 1.571 kg COD/m^3/hari dan 0.253 L_{CH4}/gCOD terhasil pada 1.07 mg/L kepekatan tetracycline dengan KBO 0.785 kgCOD/m^3/hari dicatatkan. Kajian ini menyediakan solusi rawatan air sisa dengan menggunakan RAB dan seterusnya menyumbang kepada pembangunan teknologi anarobik kepada industri yang berpotensi di Malaysia.

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LIST OF ABBREVIATIONS

ABP	-	Anaerobic Biogas Potential
ABR	-	Anaerobic Baffled Rector
AD	-	Anaerobic Digestion
ADM1	-	Anaerobic Digestion Model 1
AF	-	Anaerobic Filter
AMBBR	-	Anaerobic Moving Bed Biofilm Reactor
AnMBR	-	Anaerobic Membrane Bioreactors
AOX	-	Adsorbable Organic Halides
APEO	-	Alkylphenol Polyethoxylates
BAM	-	Biogas Activity Monitoring
BMP	-	Bio-methane Potential
BOD	-	Biochemical Oxygen Demand
COD	-	Chemical Oxidation Demand
GC	-	Gas Chromatography
HRT	-	Hydraulic Retention Time
IPCC	-	Intergovernmental Panel on Climate Change
IR	-	Infrared Range
IWA	-	International Water Association
MSAR	-	Multi Stage Anaerobic Reactor
MSW	-	Municipal Solid Waste
OLR	-	Organic Loading Rate
PET	-	Polyethylene Terephthalate
POME	-	Palm Oil Mill Effluent
RT	-	Retention Time
SRB	-	Sulphate Reducing Bacteria
SRT	-	Solid Retention Time
SS	-	Suspended Solid
TC	-	Total Carbon
TOC	-	Total Organic Carbon
TS	-	Total Solid

TSAD	-	Three-Stage Anaerobic Digester
TSS	-	Total Suspended Solid
TVS	-	Total Volatile Solids
UAFB	-	Up-flow Anaerobic Fixed Bed
UASB	-	Upflow Anaerobic Sludge Blanket
UMAR	-	Up-flow Multistage Anaerobic Reactor
VA	-	Volatile Acid
VFA	-	Volatile Fatty Acid
VOC	-	Volatile Organic Carbon
VS	-	Volatile Solid
VSS	-	Volatile Suspended Rate
WWTP	-	Waste Water Treatment Plant

LIST OF SYMBOLS

a	-	Constant correction for the intermolecular forces
b	-	constant correction for molecular size
$C_{22}H_{24}O_8N_2$	-	Tetracycline
$C_6H_{12}O_6$	-	Glucose
$CaCO_3$	-	Calcium Carbonate
CH_4	-	Methane
CO_2	-	Carbon Dioxide
d	-	Day
g	-	gram
HCO_3^-	-	Bicarbonate Ion
HoAc	-	Organic Acid as acetic acid
K	-	Kelvin
kg	-	kilogram
L	-	Liter
mg	-	miligram
n	-	Number of mole
N_2	-	Nitrogen
NH_3N	-	Ammonical nitrogen
NH_4^+	-	Ammonium ions
P	-	Phosporous
P	-	Presssure
Q	-	Flow rate
R	-	Gas Constant
S_0	-	Influent Concentration
SO_4^{2-}	-	Sulphate Ion
T	-	Temperature
V	-	Volume

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The global issues concerning wastewater discharged are raising public worried particularly in terms of treatability and energy reclaimed. These problems have forced global and social policies to develop a process that can reduce discharge pollutants, as well as renewable energy recovery. It is strongly supported by the political decisions around the world as The United Nation predicted that by 2050, up to 77% of the global energy demands will be supplied from renewable resource (Serena *et al.,* 2016; Intergovernmental Panel on Climate Change (IPCC), 2011; Wang *et al.,* 2009; Bauer *et al.,* 2009). Thus, it will be necessary to mitigate a sustainable treatment facilities with higher treatment efficiencies as well as energy recovery to encounter both global demand challenges.

Despite this challenge, the anaerobic treatment of wastewater promises a great option for both disposal route and energy recovery (Hon *et al.,* 2013). Anaerobic digestion (AD) process biologically metabolises organic material (disposal) in the absence of oxygen and produces methane (CH_4, for energy recovery) at the end of process. The AD operates on either high-rate or low-rate system. For the high-rate system, AD retained biomass, while for the low-rate system, it occurs without biomass retention. Methane production in AD requires a diversity of archaea that participated in the degradation process. In AD process, it consists of four stages of degestion: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Gupta and Gupta, 2014).

The AD is a promising technology that offers substantial benefits of reducing pollution and compensating the dependency of fossil fuels by producing biogas or as by-product. It exploits biomass for potential energy source and generates an alternative energy pathway for clean energy source (Spyridon and Euverink, 2016).

Typically, energy recovery in AD process consists of 55-80% methane, 20-45% carbon dioxide, 0-3% nitrogen, 0-1% hydrogen, and 0-1% hydrogen sulfide (Bodius salam *et al.,* 2015; Gunaseelan, 1997). The composition of biogas are mainly depends on feed materials and biomass characteristic. Thus, poor quality of biomass and feed substrate will reduced the potential of energy recovery in the AD process (Yucong *et al.,* 2016; Pretel *et al.,* 2015).

The recognition of biogas benefits in AD, demands a good knowledge for controlling its phenomenon and behaviour especially for development of modern high rate and co-digestion system (Xavier Goux *et al.,* 2016; Chananchida Nathoa *et al., 2014*; Prajapati *et al.,* 2013). Yet, there are obstacles in achieving an efficient process for biogas generation in AD system as well as treatment efficiencies (Wendy *et al.,* 2013).

Over the years back, limitations have been reported in AD methodologies to the treatment effectiveness and energy reclaimed, especially at spontaneous biological reactions in the system. Despite many studies perform the feasibility of biogas generation through AD, only several have optimised the process. However, results of previous researches provided some resources to improve biogas generation. The improvement of AD process to date has enhanced the development of AD treatment process to encounter the previous inadequacy due to its low organic matter removal, long HRT, growth of excessive residual organic matter, large reactor volume requirements and low biogas generation (Gannoun *et al.,* 2009).

A wide variety of anaerobic systems have been developed to specifically treat different type of wastewater. There are many types of AD systems configuration, such as single, batch, continuous, and stages, where each system is designed for its specific requirement. Single-stage reactors utilise one reactor for both acidogenic and methanogenic phases. The designation is simple, as it requires low investment costs as well as easy to operate. On the other hand, the biogas recovery at this stage is lower (Verma, 2002). For batch application, it tends to operate under tightly controlled conditions inside the reactors. The system is the cheapest and requires low technology implementation. However, their major drawbacks are lower biogas yield, easy to clog, and consume large footprint (Vandevivere *et al.,* 2003). Meanwhile, staging configuration in AD improved stabilization in the system. The key idea behind these two or multi stage digesters is when digestion is separated into stages, it allow the optimum environmental conditions for each bacterial and archaeal group to perform. The separate function were designed to distribute organic load and to increase the contact time between the medium (sludge bed) and the feed particles to react efficiently.

Hypothetically, multi-stage configuration aims to separate the acidogen from methanogen phase, which enhances the performance of the reactor system (Marshall and Innocentia, 2016; Rafik *et al.,* 2015; Lei Sun *et al.,* 2012; Alkarimiah *et al.,* 2011). The distribution process promotes a superior performance at producing methane gas and bear to stand shock organic loading rate (OLR) at its earlier stage, allowing a more constant rate for methanogenic activity at the later stage (Hendroko *et al.,* 2014).

Therefore, in AD system, reactor configuration is crucial for a treatment at different type of wastewater characteristic. The slow-growing anaerobic in microbes in digestor require a longer sludge retention time (SRT) in the system while loading rates are primarily affected by the concentration of active biomass in AD reactors. Consequently, maintenance of high SRT is a major point of interest in practical application of AD process. High rate anaerobic treatment can be achieved by employing efficient biomass retention methods. In anaerobic reactors, in order to maintain higher biomass densities, SRT has to be in excess of hydraulic retention time (HRT); SRT>>HRT. High biomass densities also provide greater resistance to any inhibitory substance in the influent. Numbers of novel anaerobic reactor configurations have been developed for accomplish a higher treatment efficiency and consistency in AD system.

Anaerobic reactors have been used widely to treat different type wastewaters and may contain a varying degree of toxic or recalcitrant compounds. These inhibitory compound has been frequently detected in aquatic environment, threatening the ecological safety of wastestream influent (Weiwei *et al.,* 2017; Kara *et al.,* 2017). Inhibitory toxic compounds include pharmaceutical mixture, antibiotic residue, lignocellulose substances and halogenated organic compounds from pulp and papermill wastewater, waste from petroleum refining and tanning industries and other toxic substances that may be components of the influent wastestream, or byproducts will inhibit the metabolic activities of the digester microbial community.

Previous study found a large fraction of recalcitrant antibiotic compounds were discharged in unchanged and/or active forms to wastewater stream at concentrations from ng/L to mg/L (Zhang *et al.,* 2015; Wei *et al.,* 2011; Larsson *et al.,* 2007). A wide usage of antibiotic especially for medical intervention in treating infectious disease and in livestock growth promoter have been found all around the world and become serious environmental concern (Fetra *et al.,* 2020). Extensive rate on antibiotic consumption for human was increased by 39% between year 2000 and 2015 while 53% consumption for livestock was forecast in year 2013 and 2020 (Klein *et al.,* 2018; Van Boeckel *et al.,* 2017). Even though the concentrations on the detected antibiotics in wastewater are generally around μg/L levels, they can still impend adverse effect on drinking water safety and public health (Dongle *et al.,* 2018; Richardson and Ternes, 2011). The abusive consumption of antibiotics in livestock feed indicated high concentrations of antibiotics and their metabolites in manure, which were finally released into the wastewater stream. The persistent antibiotic residues in the environment may lead to the growth of antibiotic-resistant microbes and worse, the effect leads to threat to human health and ecosystem safety (Boxall *et al.,* 2003; Chee-Sanford *et al.,* 2001). In AD treatment, some antibiotics, with different specificities, have inhibitory effects on performance and decreasing methane potential by interfering with the activity of volatile acid-degrading bacteria, (Sanz *et al.,* 1996).

One of commonly used antibiotics was tetracycline, and it had been widely used due to broad spectrum antimicrobial activity and inexpensive for human and veterinary purposes (Zhang *et al.,* 2014; Shi *et al.* 2011). There is lack of knowledge involving degradation of tetracycline fate in wastewater treatment plan. A recent study suggested that anaerobic treatment appeared to be superior to other treatment opinions for tetracycline removal (Zhang *et al.,* 2018). Under anaerobic condition, previous evidence showed that tetracycline may affected up to 70-90% of organic removal (Wu *et al.,* 2011; Hu *et al.,* 2011). Biogas production corresponding to tetracycline concentration showed a reduction to 30% of methanogen inhibition at 9.8 mg/L (Arikan, 2008). The presence of a high Tetracycline could disrupt the balance of the AD system. The degradation of tetracycline may not be as active as its parent compounds, but may have an influence on the same concentration level as its parents constituent or even more harmful than its metabolites (Halling-Sorensen *et al.,* 2003). Furthermore, there is lack of knowledge involving degradation of tetracycline fate in wastewater treatment plan. A recent study suggested that anaerobic treatment appeared to be superior to other treatment opinions for tetracycline removal (Zhang *et al.*, 2018).

Other significant recalcitrant compound in water bodies are substances from pulp and papermill wastewater. During the operation of pulp and paper mill productivity, there are various of organic and inorganic recalcitrant substances that are presents in pulp and papermill residue such as halogenated organic compounds and lignocellulos substances (Kamali *et al.,* 2016). Effluent from pulp and paper mill industry can be originated from recycled paper mills wastewater, mechanical pulping effluents, black liquor and kraft bleaching (Driessen *et al.,* 1999). A different biological treatment options in AD was employed for treating pulp and paper mill effluent.

Among AD reactors, Upflow Anaerobic Sludge Blanket, (UASB) has become favorable treatment option adopted by pulp and paper industry since 1983, due to its advantages when compared with other AD types (Chong *et al.,* 2012). Other common high rate anaerobic reactors have been applied to treat pulp and paper mill wastewater was, anaerobic filters (AFs), and anaerobic membrane bioreactors (AnMBRs), respectively. Meanwhile, Anaerobic Baffled Rector, (ABR) shows a higher efficiency of 95% Chemical Oxygen Demand (COD) removal and 57% with biogas yield at 0.77 L/d (day 15) in treatment process (Haider *et al.,* 2014). By using UASB reactor in treating black liquor from a kraft pulp mill, treatment competency reached 80% of COD degradation (Buzzini & Pires, 2002).

Reactor (MSAR) was configured by the UASB and ABR designation, treatment efficiencies was expected to tolerate high and low organic loading rates, high methane potential, and can withstand with toxic or recalcitrant substances, significantly.

1.2 Problem Statement

During the early period of AD development, the single-stage anaerobic process was limit by the low rates of chemical oxygen demand (COD) removal, long HRT, accumulation of waste sludge, and requirement of large reactor volume. In single-stage of AD, the operating conditions are more or less suitable for all the reactions and no particular phase has been optimized. Single-stage of AD also require less capital cost and less maintenance, but generate lower gas production and organic conversion rate. There are no distribution of organic loading in single stage reactor and there are no physical separation between microbial communities (acidogen and methanogen) in reactor.

Among different types of single stage anaerobic reactors, the single stage UASB reactor embraces a successful applications in different wastewater types especially excellent in settling properties and high activity of granular sludge. However, when multiple stages of UASB was applied in treating similar wastewater, organic removal was improved, retention time was reduced as well as less reactor volume requirement. Besides, methane generation was better recovered as staging conceptual separates acidogenesis bacteria and methanogenis archaea in reactor. In the UASB reactors, the process involved an upward flow passes a thick layer (blanket) of suspended sludge with high biological activity and pose the greatest risk of washout possibilities in the biomass if the HRT is too low. Another popular AD treatment system was Anaerobic Baffled Reactor, ABR. This reactor type was a septic tank improvement with series of baffled implementation which can separate acidogenesis and methanogenesis longitudinally down the reactor, allowing the different bacterial groups to develop under most favorable conditions. The increased contact time with the active biomass (sludge) results in reactor will stabilized the sludge, resist to organic and hydraulic shock load, high BOD and COD reduction, low operating cost and long service life.

Dealing with the disadvantages of single-stage AD process, multiple stages of AD mode, (usually two or more reactors) has been designed in series to optimize the process and to enhance gas production. In AD process, methanogens archaea in system have long regeneration time compared to acidogens activity. Multiple stage served as time platform for methanogenis existence and will improved the performance of the reactor.

Different type of wastewater treated by AD system may contain a varying degree of toxic or recalcitrant compounds. AD is popular approaches to withstand those inhibitory compounds. In staging mode of operation, physical separation can optimize each process reaction for the breakdown of the organics and enhance more organic removal compared to single staging mode. A stage reactor can accommodate toxic slugs more efficiently by passing them in less time through the reactor system. This will result high treatment efficiency for recalcitrant substrates due to phase separation, which promotes favourable conditions for microbial populations involved in the degradation of recalcitrant compounds.

The proposed MSAR in this study is aimed at solving some problems previously encountered in single stage anaerobic treatment. The MSAR has been developed with different design approach with the combination of UASB and ABR reactor system. Staging mode of operation in MSAR is incorporated to represent a separate compartment for better removal of organic pollutants, ability to withstand the effect of recalcitrant compound, and for biogas (methane) potential recovery. Two different types of recalcitrant wastewater have been tested during the study. The antibiotic, Tetracycline has been selected as inhibitory compound due to wide application for human and veterinary purposes. Eventhough very small concentration was detected in water bodies, tetracycline antibiotic poses a great environmental influenced, threatening the ecological safety of wastestream influent. So far, little is known about the existence of antibiotics in water stream in Malaysia. The current study of antibiotic levels in the water samples from the Larut River have a significant contaminated with Tetracycline antibiotics (Low et al., 2020).

The other harmful substances that will disturb the stability of AD process was originated from pulp and paper mill industries. Paper mill wastewater contains complex organic substances such as halogenated organic compounds and lignocellulos substances which could not be treated completely using conventional treatment processes, e.g. aerobic process. Although numerous anaerobic reactors have been designed and used for treating paper mill wastewater in the past, very little evidence has disclosed a four-stage mode of anaerobic process for a better organic removal efficiencies. MSAR are projected to solve the problems of using single-stage reactor in removing pollutants at high organic loading rate and the potential for biogas reclamation, apart from being an effective solution to achieve compliance with law and legislation. In fact, toxic and recalcitrant wastewaters, which were previously believed to be unsuitable for anaerobic processes, are now potentially treated using via MSAR.

1.3 Aims and Objectives

The main aim of this study is to propose a new type of anaerobic reactor for wastewater treatment that incorporates the concept of staging mode of operation. In particular, the intention is to design and develop a new type of reactor that combines the UASB reactor and ABR. It is hoped that this new type of reactor can treat wastewater at high degree of organic removal, aside from accommodating to the effect of recalcitrant compound. Accordingly, this study has developed MSAR and tested the reactor with various fundamental experiments, such as start-up performance, ability to withstand low HRT and high OLR, ability to tolerate recalcitrant compound, and biomethane potential evaluation. Additionally, the reactor was also tested using real wastewater (paper mill) to verify if the reactor can be used for real application in the industry. The more specific objectives of this study are listed as follows: ”

i. To **define** the set-up of MSAR approaches at design , development and start-up performance.

ii. To **investigate** the effect of operational parameters (HRT and OLR) on the treatment performance of MSAR.

iii. To **investigate** the effect of recalcitrant compound inhibition for the treatment performance of tetracycline and real paper mill wastewater in MSAR.

iv. To **examine** the theoretical methane potential on the treatment performance of MSAR

1.4 Scope of Study

The main scope of this study is as follows:

i. The wastewater used in this study refers to a synthetic (glucose) prepared in laboratory, synthetic recalcitrant wastewater contain tetracycline (antibiotic) residue, and real paper mill wastewater.

ii. The two main operational parameters used in this study were HRTs and OLRs.

iii. The performance of MSAR system was based on COD removal efficiency, solid characteristic, pH level, volatile acid concentration and methane potential analysis.

iv. There are no microbial study during this experiment as reactor performance was evaluate by treatment efficiencies.

v. Alkalinity was maintained at 2000 mg/L as $CaCO_3$ in each phase of operation.

vi. Nutrient to feed ratio was maintained at (250:7:1) for (COD: N: P) elemental amount.

1.5 Significance of Study

AD treatment system continue to receive high levels of interest, proving to be a cost-effective way of degesting organic material to generate energy, as well as to achieve significant renewable energy in the future. Advancements in digester design, along with supportive regulations and incentives, continue to make AD a more feasible and practical solution for small- and large-scale waste management and environmental challenges. This study offers a potential solution for wastewater treatment system at an acceptable concentration and condition. Attempting to expand the stage reactor concept, this research addresses the latest development in anaerobic reactor technology across Malaysian industries and eventually developing a commercial reactor design that bear to treat at higher loading capacity and the threatening substances of recalcitrant wastewaters. Malaysia has placed vast initiatives in place, such as the National Renewable Energy Policy and Action Plan 2009 and the National Biomass Strategy 2020, to move towards greater renewable energy production through AD systems. With those being said, MSAR is one of the potential target treatment systems.

1.6 Thesis Organization

This thesis consists of nine chapters. In Chapter 1, the background of research, problem statement, research objectives, scope, and research significance are presented. In Chapter 2, a review of fundamental knowledge of AD processes, operational factors affecting AD, a review of anaerobic reactor technology, discussion on stage anaerobic reactor systems, treatment of various wastewaters using anaerobic stage reactor, the potential of AD process for removing recalcitrant compound, and biogas potential in AD are described. In Chapter 3, the methodology applied throughout the study is elaborated. The development of MSAR system and its configuration, wastewater characterization, crucial parameters for the experimental procedure, as well as depiction of experiments and analyses are given in Chapter 3. Next, Chapter 4 describes the design, the development, and the start-up of the novel MSAR. It also presents the detailed investigation on the start-up performance using synthetic wastewater (glucose). In Chapter 5, the effects of HRT and OLR on MSAR performance were investigated using synthetic wastewater (glucose). Chapter 6 is devoted to the effect of a recalcitrant compound on the MSAR. The capability of MSAR to withstand toxic concentration of Tetracycline was assessed. Moving on, Chapter 7 describes the treatment of another recalcitrant wastewater (paper mill) using MSAR. Chapter 8 Theoretical formula for methane potential is discussed in this chapter. Finally, Chapter 9 summarizes the outputs of this research and several recommendations for future study

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