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HYDRODYNAMIC CAVITATION USING ORIFICE PLATE CONFIGURATIONS AND ARRANGEMENTS FOR TERTIARY TREATMENT OF PALM OIL MILL EFFLUENT

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
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…to my lovely wife Shafiah Dolhakim
daughter Sarah Noor Liyana
parents Jusoh Jaafar and Kamariah Che Soh
thank you for your du'a, support and patience all this while…

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ABSTRACT

Hydrodynamic Cavitation (HC) is one of Advanced Oxidation Processes (AOPs), which generates and utilises hydroxyl radicals ($HO^{\cdot}$) as its oxidising agent. It has been studied for different applications to treat pharmaceuticals waste, seawater and microalgae, where much effort has been conducted to enhance its performance such as using pH, aeration and hydrogen peroxide (H_2O_2). However, the production of $HO^{\cdot}$ using multiple-plate combination has not yet been studied. The use of pH, aeration and H_2O_2 has proven to give significant improvement for $HO^{\cdot}$ formation, but these have not being studied previously using multiple-plate combination. The use of HC as a tertiary treatment for POME has not being reported before. Therefore, in this study, the enhancement of the HC has been investigated using double and triple orifice plate configurations and arrangements. The best system was then tested on biologically treated palm oil mill effluent (BT-POME). As the colour of POME is difficult to remove the performance of ponding treatment system was evaluated to understand the causes of colour in POME. The experiments were conducted in a lab-scale HC system, treating 10 L samples for reaction time ranging from 30 to 180 min. The effect of pH (2-7), aeration (2-10 L/min) and H_2O_2 dosing (50-200 mg/L) were explored. The performance of the HC system was based on iodine liberation, and removal of colour and chemical oxygen demand (COD). The byproducts of BT-POME degradation was identified. Additionally, the performance of an existing ponding system treating POME was assessed and the relationship between colour and few selected parameters were studied. Within the range of the experimental conditions used in this study, the HC orifice plate configurations and arrangements were found to have significant effects on $HO^{\cdot}$ generation. The iodine liberation for both double and triple plate were higher than that of a single plate. The $HO^{\cdot}$ generation was also affected by the arrangement and the distance between the plates; arrangement plate of P3P2 with 10 cm distance gave the highest iodine liberation (1296 mg/L). The performance of HC was enhanced under the effect of pH, H_2O_2 and aeration as compared to HC alone. For the conventional ponding treatment system, the anaerobic pond played the most significant role in treating POME with removal up to 97%. Among the pollutants analysed, colour has strong relationship with phenolics, tannin, lignin and carotene, indicating the roles of these compounds in causing colour of POME. The degradation of BT-POME by the HC system was not encouraging as only up to 14.7% of colour was removed, with lower removal of COD. The addition of H_2O_2 and aeration have significant effect in removing COD, while pH and addition of H_2O_2 have significant effect on colour removal. The degradation of BT-POME, particularly phenolics and tannin/lignin was found to form catechol and ρ-benzoquinone as by-products. The study showed another approach in improving HC system performance but further work is required before the system can be applied in treating BT-POME effectively.

ABSTRAK

Hydrodynamic Cavitation (HC) adalah salah satu Proses Pengoksidaan Lanjutan (AOPs) yang menghasilkan dan menggunakan radikal hidroksil ($HO^{\cdot}$) sebagai agen pengoksidaan. Ia telah dikaji untuk pelbagai kegunaan dalam merawat sisa farmaseutikal, air laut dan mikroalga, dan banyak usaha telah dijalankan untuk meningkatkan prestasinya seperti penggunaan pH, pengudaraan dan hidrogen peroksida (H_2O_2). Walau bagaimanapun, penghasilan $HO^{\cdot}$ menggunakan beberapa plat berliang belum pernah dijalankan. Penggunaan pH, pengudaraan dan H_2O_2 telah terbukti memberikan peningkatan yang bererti kepada pembentukan $HO^{\cdot}$, tetapi tidak pernah dijalankan meggunakan gabungan beberapa plat. Penggunaan HC sebagai rawatan tertier untuk POME tidak pernah dilaporkan sebelum ini. Oleh itu, dalam kajian ini, peningkatan HC telah dikaji menggunakan susunan dan konfigurasi dua dan tiga plat berliang. Sistem yang terbaik kemudiannya diuji pada air sisa kilang minyak sawit yang telah terawat secara biologi (BT-POME). Disebabkan warna POME adalah sukar untuk disingkirkan prestasi sistem kolam rawatan telah dinilai untuk memahami penyumbang kepada warna POME. Ujikaji telah dijalankan di dalam sistem HC berskala makmal, merawat 10 L sampel dengan masa tindakbalas dari 30 hingga 180 minit. Kesan pH (2-7), pengudaraan (2-10 L/min) dan dos H_2O_2 (50-200 mg/L) telah dikaji. Prestasi sistem HC adalah berdasarkan kepada penghasilan iodin, dan penyingkiran permintaan oksigen kimia (COD) serta warna. Hasil sampingan penguraian BT-POME telah dikenalpasti. Sebagai tambahan, prestasi sistem kolam rawatan POME sedia ada telah dinilai dan kaitan diantara warna dan beberapa parameter pilihan telah dikaji. Dalam julat keadaan eksperimen yang digunakan dalam kajian ini, susunan dan konfigurasi plat berliang HC didapati mempunyai kesan yang signifikan kepada penghasilan $HO^{\cdot}$. Penghasilan iodin bagi dua dan tiga plat adalah lebih tinggi berbanding plat tunggal. Penghasilan $HO^{\cdot}$ juga dipengaruhi oleh susunan dan jarak diantara plat; susunan plat P3P2 dengan jarak 10 sm menghasilkan iodin tertinggi (1296 mg/L). Prestasi HC dipertingkatkan menggunakan pH, H_2O_2 dan pengudaraan berbanding HC sahaja. Bagi sistem rawatan kolam konvensional, kolam anaerobik berperanan penting dalam merawat POME dengan penyingkiran sebanyak 97%. Dikalangan bahan pencemar yang dianalisis, warna berhubung kuat dengan fenol, tanin/lignin dan karotena, menunjukkan sebatian ini berperanan menghasilkan warna dalam POME. Penguraian BT-POME oleh sistem HC adalah tidak memberangsangkan sekadar 14.7% penyingkiran warna dengan catatan penyingkiran COD yang rendah. Penambahan H_2O_2 dan pengudaraan memberi kesan yang bererti kepada penyingkiran COD, sementara pH dan penambahan H_2O_2 memberi kesan yang bererti kepada penyingkiran warna. Penguraian BT-POME, khususnya fenol dan tanin, lignin didapati menghasilkan katekol dan ρ-benzokuinon sebagai hasil sampingan. Kajian ini menunjukkan pendekatan yang lain dalam meningkatkan prestasi sistem HC tetapi kajian lanjutan diperlukan sebelum sistem ini boleh digunakan merawat BT-POME dengan berkesan.

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LIST OF ABBREVIATIONS

AOPs	-	Advanced oxidation processes
Ar	-	Argon
AC	-	Acoustic cavitation
AF	-	Anaerobic filter
AHR	-	Anaerobic hybrid reactor
ABF	-	Anaerobic baffled filter
ADF	-	Anaerobic downflow filter
ABSR	-	Anaerobic bench scale reactor
ADMI	-	American Dye Manufacturing Index
AN	-	Ammoniacal nitrogen
AC	-	Activated carbon
BOD	-	Biological oxygen demand
BT-POME	-	Biological treated palm oil mill effluent
COD	-	Chemical oxygen demand
FBR	-	Fluidised bed reactor
HC	-	Hydrodynamic cavitation
H_2O_2	-	Hydrogen peroxide
HRT	-	Hydraulic retention time
HCl	-	Hydrochloric acid
KI	-	Potassium iodide
MLSS	-	Mixed liquor suspended solids
MWCO	-	Molecular weight cut-off
MBR	-	Membrane bioreactor
MTBE	-	Methyl-tert-butyl ether
O&G	-	Oil & grease
OLR	-	Organic loading rate

POME	-	Palm oil mill effluent
P1	-	Plate 1
P2	-	Plate 2
P3	-	Plate 3
P4	-	Plate 4
P5	-	Plate 5
P6	-	Plate 6
RO4	-	Reactive orange 4
RBC	-	Rotating biological contactor
RR2	-	Reactive Red 2
TOC	-	Total organic carbon
TSS	-	Total suspended solids
TP	-	Total phosphorus
TN	-	Total nitrogen
UASB	-	Up-flow anaerobic sludge blanket
UF	-	Ultrafiltration
US	-	Ultrasound
VSS	-	Volatile suspended solids

LIST OF SYMBOLS

A	-	Total weight of flask
B	-	Tare weight of flask
Cv	-	Cavitation number
d_h	-	Diameter of the orifice
g	-	Gravity acceleration
H	-	Pressure head
H	-	Hydrogen atoms
$HO^{\cdot}$	-	Hydroxyl radical
I_3^-	-	Iodine molecules
Na_2SO_4	-	Sodium sulphate
N_2	-	Nitrogen
NaOH	-	Sodium hydroxide
n	-	Number of holes of orifice plate
O_2	-	Oxygen
P_2	-	Downstream pressure of liquid
P_v	-	Vapour pressure of liquid
Q	-	Flow rate in the main line
$R^{\cdot}$	-	Alkyl radical
RH	-	Organic substrate
ROH	-	Hydroxylated adduct-radical
$ROO^{\cdot}$	-	Peroxyl radical
ROOH	-	Hydroperoxides
t	-	Time of operation
v	-	Velocity of liquid
v_o	-	Velocity at the orifice
v_p	-	Fluid velocity of inlet pipe

α	-	Total perimeter of orifices/total area of orifices
β	-	Total area of orifices/cross sectional area of pipe
ρ	-	Density of the liquid

CHAPTER 1

INTRODUCTION

1.1 Preamble

Advanced oxidation processes (AOPs) are treatment technologies that use free radicals, particularly hydroxyl radicals ($HO^{\cdot}$) as a medium to attack and degrade organic substances. The $HO^{\cdot}$ can be formed in AOPs under photochemical and non-photochemical procedures (Quiroz *et al.,* 2011). The photochemicals consist of photo-fenton, heterogeneous photocatalysis, UV/H_2O_2 and UV/O_3. The non-photochemicals consist of alkaline media ozonation, O_3/H_2O_2, fenton and fenton-like reactions, electrochemical oxidation, cavitation and sub/super critical water.

AOPs offer several advantages against other processes such as easy operation, high efficiency, less production of residuals and toxic intermediates in the wastewater treatment (Jawale *et al.,* 2014). The formation of $HO^{\cdot}$ and other free radicals such as $H^{\cdot}$, $HO_2^{\cdot}$ and H_2O_2 contain powerful oxidizing potential which is able to degrade target contaminants in aqueous solutions (Zhang *et al.,* 2014). However, among free radicals released, only hydroxyl radical ($HO^{\cdot}$) is of particular interest because of their high oxidation capability. Its oxidizing capacity is up to 10^9 $M^{-1}\cdot s^{-1}$ stronger than ozone with second order reaction rate constant in the range of $10^6 - 10^9$ $M^{-1}\cdot s^{-1}$ (Aris, 2008).

The formation of $HO^{\cdot}$ oxidise target pollutant molecules and decompose them non-selectively to less harmful substances, leading to the ultimate mineralization products of carbon dioxide (CO_2) and water (H_2O) (Cheng *et al.,* 2016). In these process, the $HO^{\cdot}$ involves three possible mechanisms for pollutant decomposition in contaminated water which are electron transfer, radical addition and hydrogen abstraction (Mehrjouei *et al.*, 2015).

One of techniques that can be utilised to generate $HO^{\cdot}$ is cavitation. The collapse of microbubbles or cavities in cavitation can be violent enough to produce radicals. Cavitation consists of four techniques, namely acoustic, hydrodynamic, optic and particle cavitation (Gogate, 2010). Compared to other cavitation techniques, hydrodynamic cavitation (HC) is a better alternative technique to generate $HO^{\cdot}$ (Parsa *et al.*, 2013). In recent years, HC has gained considerable importance as it is a relatively benign technique with respect to economic and environmental considerations. The use of HC is preferred to generate $HO^{\cdot}$ because of less energy consumption (Arrojo and Benito, 2008; Arrojo *et al.*, 2007; Ambulgekar *et al.*, 2004; Gogate, 2002; Kumar *et al.*, 2000), simpler reaction device (Gogate and Kabadi, 2009; Ambulgekar *et al.*, 2004; Gogate, 2002), lower maintenance cost and more convenient operation (Zhang *et al.*, 2014).

The HC process involves the formation, growth and subsequent collapse of cavities that occur at small intervals of time and emit large amount of energy at several locations in a very small reactor. The $HO^{\cdot}$ is produced from the cavitation activities as it is induced by the passage of liquid through simple mechanical constrictions (orifice plate) under controlled conditions (Jadhav *et al.*, 2013). At the vena-contracta of the constriction, the pressure of the liquid is reduced lower than the vapour pressure of the liquid as it passes through the mechanical constriction at the operating temperature, hence producing a large amount of cavities. During system operation, the reduced pressure is recovered by the flow of liquid ending up at the downstream section of the constriction resulting in the subsequent collapse of the generation of cavities and the release of millions of radicals (Moholkar *et al.*, 1999).

Hydrodynamic cavitation has offered considerable promise in wastewater treatment applications due to its ability to generate $HO^{\cdot}$ in situ and the ease of operation. The successful degradation of organic pollutants using HC involved two main mechanisms which are the reaction of $HO^{\cdot}$ with the pollutants and thermal decomposition of the volatile pollutant molecule entrapped inside the cavity (Jawale *et al.,* 2014). Wang *et al.* (2009) have studied on rhodamine B degradation using swirling jet-induced cavitation combined with H_2O_2. Zhang *et al.* (2009) have investigated the degradation of C.I. Acid Orange 7 using ultrasound enhanced heterogeneous Fenton-like process. Both techniques are acceptable with the performance of rhodamine B degradation is up to 99.2% removal, while 56% of COD removal and 90% of colour removal have been removed from C.I. Acid Orange 7.

The application of HC throughout the past few decades had never been reported on the treatment of biological treated palm oil mill effluent (BT-POME). The tertiary treatment of BT-POME, however, was reported in other techniques including ultrafiltration (UF) membrane (Idris *et al.,* 2010), UV-responsive ZnO photocatalyst (Ng and Cheng, 2016) and fenton's oxidation (Aris *et al.,* 2008). The degradation of BT-POME is approximately in the ranged of 64% - 82.4% of COD removal and the colour removal is increased as high as 92.4% when using fenton's oxidation. The performance of AOPs and other techniques responded to the pollutants of BT-POME, HC method therefore could be adopted as an alternative approach for tertiary treatment of BT-POME considering of its achievement in degrading pollutants in other wastewaters.

1.2 Problem Statement

The generation of $HO^{\cdot}$ using cavitation technique has been conducted using HC reactor. The most preferred cavitation technique is the use of the orifice plate as constriction device, which operate individually in circulation closed loop reactor (Balasundaram and Harrison, 2006; Ambulgekar *et al.,* 2004; Sivakumar and Pandit, 2002; Vichare *et al.,* 2000). Several studies have been conducted to improve the capacity of HC in generating the radicals. Wang *et al.* (2015) and Ghayal *et al.* (2013) have studied the performance of multiple orifice in a single plate to generate the radicals. Additional constriction of venturi within multi-hole orifice plates have been to extend the degradation of Rhodamine B has been reported by Mishra and Gogate (2010), while Chakinala *et al.* (2008) have used chloroalkanes as additives in improving the performance of HC. Similarly, Ambulgekar *et al.* (2004) and Wu *et al.* (2015) studied the effect of aqueous potassium permanganate and hydrogen peroxide, respectively in enhancing the efficacy of cavitation.

To date, study on the $HO^{\cdot}$ production using multiple-plate combination considering the number and arrangement of plates has not yet been published. The use of multiple-plate using HC operational mode is expected to enhance the production of huge amounts of cavities as well as the possibility of collapsing cavities enabling the formation of more $HO^{\cdot}$. In addition, although pH, aeration and hydrogen peroxide has proven to generate more $HO^{\cdot}$ in cavitation process (Li *et al.*, 2015; Gogate and Katekhaye, 2012; Gore *et al.*, 2014) it has never been studied on multiple-plate combination. The formation of radicals is expected to sufficiently degrade organic pollutants in wastewater treatment.

The most common and conventional method to treat POME is ponding system. Its performance in treating POME was previously evaluated; however, the performance characteristics in relation to colour of the POME has never been reported.

Colour removal for POME treatment is still an unsolved problem. While the use of techniques such as membrane separation and carbon adsorption has been reported, their applications are still remote due to unattractive cost. HC could provide another alternative in dealing with the problem. Therefore, this study focuses on the production of $HO^{\cdot}$ using HC reactor with the novel multiple-plate combinations that are anticipated to accelerate the formation of $HO^{\cdot}$. This technique is then tested for further removal of COD and colour of BT-POME.

1.3 Objectives of the Study

The aim of this study is to generate $HO^{\cdot}$ using HC reactor with multiple-plate combinations under appropriate conditions. The formation of $HO^{\cdot}$ observed in potassium iodide solution (KI solution) is compare their performance with BT-POME. Special attention is directed to the selected conditions for the formation of $HO^{\cdot}$ based on iodine liberation in KI solution and the best conditions for the degradation of BT-POME especially on COD and colour removal are selected.

The detail objectives of this study are as follows:

i. To evaluate the effect of HC plate configurations and arrangements in terms of orifice characteristics and number of plate on the generation of the $HO^{\cdot}$.

ii. To determine the effect of pH, H_2O_2 dosing and aeration on the performance of HC.

iii. To investigate the performance of the existing POME's pond treatment system and to relate the performance characteristics in determining the colour causing compounds.

iv. To assess the performance of the HC process in treating BT-POME with respect to COD and colour removal, colour causing compounds, and by-products formation and degradation.

1.4 Scope of the Study

This study involves the design, fabrication and application of a 10-litre laboratory-scale of HC reactor. The design and operation of the reactor were based on the system developed by Vichare *et al.* (2000). In order to enhance the formation of $HO^{\cdot}$, the cavitation chamber was modified to suit multiple-plate combinations.

The experimental works were conducted separately for observing the generation of $HO^{\cdot}$ and treatment of BT-POME. The performance in generating $HO^{\cdot}$ was observed initially in KI solution and later implemented on BT-POME. The BT-POME sample was obtained from the discharge point of the treatment ponds at Felda Bukit Besar palm oil mill. The POME in previously analysed on its characteristics from six sources of raw POME and treatment ponds. The optimised HC was later tested for treating BT-POME. Six plates with different configurations were used in this study. The plates were arranged in sequence for single-, double- and triple-plate with the distance between plates were 10cm and 20 cm. The effects of pH, hydrogen peroxide (H_2O_2) and aeration in accelerating the formation of $HO^{\cdot}$ was also investigated. The statistical approach using Excel (Microsoft), Minitab v17 (Minitab) and SPSS (IBM) for an in-depth study of the parameters involved.

1.5 Significance of the Study

The application of HC to generate $HO^{\cdot}$ employing orifice plate have been extensively reported (Braeutigam *et al.,* 2010; Balasundaram and Harrison, 2006; Kanthale *et al.,* 2005; Gogate and Pandit, 2000; Moholkar and Pandit, 1997). In addition, HC has been studied extensively for the improvement in terms of the generation of $HO^{\cdot}$ (Gogate and Patil, 2014; Gore *et al.,* 2014; Wu *et al.,* 2012; Franke *et al.,* 2011; Pradhan and Gogate, 2010; Chakinala *et al.,* 2009; Jyoti and Pandit, 2003). However, the use of multiple-plate combinations appears to be missing in the experimental study. The significance of this study are, therefore, listed as follows;

i. This study provides design and technical procedural inputs of a lab-scale HC system which was not covered in the literature. It was modified specifically for the multiple-plate combinations and to explore some other aspects of HC.

ii. The study verified the advantages of multiple-plate combinations as compared to single plate to enhance the formation of $HO^{\cdot}$. The generation of $HO^{\cdot}$ was determined based on iodine liberation.

iii. The present study provides a better understanding of the factors that affect colour of the POME. It provides a statistical relationship describing how the colour is related with other factors.

iv. This study determines the viability of HC as tertiary treatment of BT-POME under the current conditions involved. In order to verify the performance of $HO^{\cdot}$ generated, the reduction of COD and colour were quantified during the operation under similar conditions.

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