UNIVERSITI PUTRA MALAYSIA

ACTIVATED CARBON-COATED COSMO BALL BIOMEDIA FOR WASTEWATER TREATMENT

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By

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Thesis submitted to the School of Graduate Studies,Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Current wastewater treatment systems, such as oxidation ponds, package systems, aerated lagoon, activated sludge, and various types of mechanical plants require long resident time to treat wastewater pollutants. With increasingly stringent discharge requirement, conventional WWTPs are no longer capable of producing effluent with the required quality. The aim of this study is to improve the surface of attached media in order to obtain a better removal efficiency in a biofilm reactor. Thus, the objectives of the study are to develop coating methods and characterize the coated surface to study the growth rate and mechanism of biofilm formation when a coated surface is used. This study also investigates the performance of activated carbon coated material with respect to organic and ammonia removal.

In this research, various coating methods were attempted and the conditions of granular activated carbon (GAC) on high-density polypropylene (HDPE) were evaluated in the effort to enhance the surface area of the HDPE for application in wastewater treatment.

In the biofilm study, a batch reactor with four AC coated Cosmo balls and four non-coated Cosmo balls were submerged in a 5-liter container filled with domestic wastewater. A laboratory-scale anoxic-aerobic reactor was installed at the Kolej 10 wastewater treatment plant and performance of the developed lab-scale reactor was evaluated under different conditions for coated and non-coated media.

The results of the experiments showed that the granular activated carbon coating with particle diameter from 100 to 800 μm were successfully deposited on the HDPE substrates used. The coatings deposited on the HDPE substrate produced high surface roughness of around 7 μm which is 10 fold higher than non-coated media. The surface area of the coated substrate is higher compared to that of the non-coated substrate due the BET of activated carbon of 426 m^2/g.

The formation of biofilm was clearly observed after Day 11 to show that the biofilm had covered 100% of coated area as opposed to only an estimated 70% of the non-coated area.

The highest removal of TP was achieved with coated Cosmo-Ball reactor which reached over 90% removal without the addition of any chemical, while only 54.6% TP was removed by the non-coated Cosmo-Ball. For organic removal, the coated Cosmo ball achieved 97.6% BOD, 92.2% COD and 98.3% TSS compared to non-coated Cosmo ball which gave 91% BOD, 87.8% COD and 92.47% TSS. Ammonia removal was significantly higher for coated Cosmo ball, 88.1% NH_3-N contrast to non-coated 69.2% only.

Two kinetic models namely Modified-Stover Kincannon and Grau were also studied to find kinetic parameters and the result showed that Modified-Stover Kincannon model can be recommended to be the best kinetic to use.

The result of this research also showed that, after coating, there is four (4)-fold increase in the surface area of the media compared to the non-coated media. This had resulted in a significant decrease of HRT from 6 to 3 hours, and it could also save up to 50% of the volume of aeration tank.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

BIOMEDIA BOLA COSMO BERSALUT KARBON DIAKTIFKAN UNTUK RAWATAN AIR BUANGAN

Oleh

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Sistem rawatan air buangan yang digunakan pada masa kini, seperti kolam pengoksidaan, sistem pakej, lagun berudara, enap cemar diaktifkan, dan pelbagai jenis loji mekanik, memerlukan masa tahanan yang panjang untuk merawat bahan cemar air buangan. Dengan keperluan buangan yang semakin ketat, loji rawatan air buangan konvensional kini tidak dapat lagi menghasilkan efluen yang menepati kualiti yang ditetapkan. Matlamat kajian ini adalah untuk menambah baik permukaan media lekat bagi mencapai kecekapan penyingkiran yang lebih baik dalam reaktor biofilem. Oleh itu, objektif kajian ini adalah untuk membangunkan kaedah salutan dan mencirikan permukaan bersalut bagi mengkaji kadar pertumbuhan dan mekanisme pembentukan biofilem apabila permukaan bersalut digunakan. Kajian ini turut menyiasat prestasi bahan bersalut karbon diaktifkan berhubung dengan penyingkiran bahan organik dan amonia.

Penyelidikan ini mencuba pelbagai kaedah salutan, dan turut menilai keadaan karbon diaktifkan berbutir (GAC) pada polipropilena ketumpatan tinggi (HDPE) dalam usaha untuk meningkatkan luas permukaan HDPE bagi penggunaan dalam rawatan air buangan. Dalam kajian biofilem, reaktor kelompok berisi empat bola Cosmo bersalut AC dan empat bola Cosmo tak bersalut ditenggelamkan dalam bekas lima liter yang diisi dengan air buangan domestik. Reaktor anoksik-aerobik skala makmal dipasang di loji rawatan air buangan Kolej 10 untuk dan prestasi reaktor skala makmal yang dibangunkan dinilai dalam keadaan berlainan untuk media bersalut dan tak bersalut.

Keputusan uji kaji menunjukkan yang salutan karbon diaktifkan berbutir dengan zarah berdiameter antara 100 dan 800 μm berjaya dienapkan pada substrat HDPE yang digunakan. Salutan yang diendapkan pada substrat HDPE menghasilkan kekasaran permukaan tinggi sekitar 7 μm, yang adalah 10 kali ganda lebih tinggi daripada media tak bersalut. Luas permukaan substrat bersalut adalah lebih tinggi berbanding dengan luas permukaan substrat tak bersalut disebabkan BET karbon diaktifkan sebanyak 426 m^2/g.

Pembentukan biofilem dapat dicerap dengan jelas selepas Hari 11 yang menunjukkan bahawa biofilem telah meliputi 100% kawasan bersalut berbanding hanya sekitar 70% kawasan tak bersalut.

Penyingkiran tertinggi TP dicapai apabila menggunakan reaktor bola Cosmo bersalut, yang mencapai lebih 90% penyingkiran tanpa penambahan sebarang bahan kimia, manakala bola Cosmo tak bersalut hanya menyingkirkan 54.6% TP. Bagi penyingkiran bahan organik, bola Cosmo bersalut mencatatkan penyingkiran 97.6% BOD, 92.2% COD, dan 98.3% TSS berbanding bola Cosmo tak bersalut yang mencatatkan penyingkiran 91% BOD, 87.8% COD, dan 92.47% TSS. Penyingkiran amnonia adalah jelas lebih tinggi bagi bola Cosmo bersalut, iaitu 88.1% NH_3-N, berbanding hanya 69.2% oleh bola Cosmo tak bersalut.

Dua model kinetik, iaitu Stover Kincannon diubah suai dan Grau, turut dikaji bagi menentukan parameter kinetik, dan keputusan menunjukkan bahawa model Stover-Kincannon diubah suai boleh disyorkan sebagai kinetik terbaik untuk digunakan. Keputusan penyelidikan ini juga menunjukkan bahawa, selepas penyalutan, terdapat peningkatan empat (4) kali ganda dalam luas permukaan media berbanding luas permukaan media tak bersalut. Ini mengurangkan HRT daripada 6 kepada 3 jam. Ia juga dapat menjimatkan sehingga 50% isi padu tangki pengudaraan.

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I certify that a Thesis Examination Committee has met on 26 October 2017 to conduct the final examination of Khaled Muftah Shahot on his thesis entitled "Activated Carbon-Coated Cosmo Ball Biomedia for Wastewater Treatment" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This is to confirm that:

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LIST OF ABBREVIATIONS

BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
TOC	Total Organic Carbon
NH_3-N	Ammonia Nitrogen
NO_2-N	Nitrite Nitrogen
NO_3-N	Nitrate Nitrogen
TKN	Total Kjehldahl Nitrogen
TS	Total Solids
TSS	Total Suspended Solids
TP	Total of Phosphate
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
SRT	Sludge Retention Time
ASP	Activated Sludge Process
OLR	Organic Loading Rate
F/M	Food to Microorganism Ratio
EPS	Extracellular Polymeric Substances
HRT	Hydraulic Retention Time
Qr	Recycling Flow Rate
PE	Population Equivalent
DO	Dissolved Oxygen
SEM	Scanning Electron Micrograph

BET	Brunauer-Emmett-Teller
EDX	Energy Dispersive X-ray
GAC	Granular Activated Carbon
AC	Activated Carbon
HDPE	High Density Polyethylene
TDS	Total Dissolved Solid
MBBR	Moving Bed Biofilm Reactor
MBR	Membrane Bioreactor
RBC	Rotating Biological Contactor
TF	Trickling Filter
PAC	Powdered Activated Carbon
NGS	Next Generation Sequencing

CHAPTER 1

INTRODUCTION

1.1 Background

Sewage can be defined as wastewater discharged from domestic sources such as homes, restaurant, industries, agricultural plants, etc. Wastewater contains substance such as human waste, food scraps, oil, soaps, and chemicals. Sewage generally flows through an extensive network of underground pipes to wastewater treatment plants where the polluted water is treated using various methods to remove pollutants. Feeding polluted water directly into rivers will result in water contamination.

The constituents of wastewater can be characterized based on its physical, chemical, and biological parameters. Physically, wastewater is usually characterized by a grey color and musty odor with 99.9% water and 0.1 % solids content. The solids are both suspended (30 %) and dissolved (70 %). The chemical constituents of wastewater are composed of organic and inorganic compounds as well as various gases. The organic components are composed of carbohydrates, proteins, fats, oils, grease and phenols, while inorganic components are composed of heavy metals, nitrogen, phosphorus, sulfur, chlorides, and toxic compounds. The biological constituents include various types of microorganisms, and those of concern are protista, plants and animals. The categories of protista which are most important in wastewater treatment are bacteria, fungi, protozoa, and algae (Hung, 2012).

The number of polluted rivers in Malaysia remains at 14 as in previous years, namely the Sg. Dondang, Sg. Juru, and Sg. Jejawi in Penang; Sg. Deralik and Sg. Raja Hitam in Perak; Sg. Kelang, Sg. Buloh and Sg. Sepang in Selangor; Sg. Tukang Batu, Sg. Pasir Gudang, Sg. Sedili Kecil, Sg. Kempas, Sg. Pontian Kechil, and Sg. Rambah in Johor (Shahot and Ekhmaj, 2012). Some of the rivers in Malaysia are heavily polluted with mean BOD levels almost six times the international standard. The higher level of BOD-related water pollution is due to residential pollution, followed by agriculture and industrial pollution. Of the 119 rivers monitored for wastewater pollution, 34 rivers recorded levels exceeding the standard level. They are nine rivers in Johor, seven in Selangor, six in Sarawak, three in Terengganu, two each in Melaka, Pahang, Perak and Sabah, and one in Negeri Sembilan (Shahot and Ekhmaj, 2012).

Water quality has been receiving a lot of attention throughout the world. People need water of the best quality for their daily lives. Therefore, water needs to be treated to make it safe for the consumption by humans and other living things. In addition, a better and compact wastewater treatment system is urgently needed. Both the cost and availability of land, combined with the implementation of secondary treatment standards, has given rise to the demands for wastewater treatment plants with small footprint which produce an effluent of high standard and at the same time fulfill the requirement for waste minimization (Leiknes and Odegaard, 2001). The last two decades have seen the emergence of new technologies in the form of oxidation ponds, aerated lagoons, package systems, and a various types of mechanical plants (Birima, 2008).

One treatment method uses activated carbon to remove wastewater pollutants. In 1965, for the first time, a full-scale advanced (tertiary) wastewater treatment plant which incorporated granular activated carbon (GAC) began operating in South Lake Tahoe, California. GAC beds as a unit process began to be commonly used in tertiary treatment system (Hendricks, 2006). GAC was utilized to reuse the effluent from municipal wastewater treatment plants for other purposes, for example industrial cooling water, irrigation of parks, etc. Adsorption with activated carbon (AC) has been successfully used for advanced (tertiary) treatment of municipal and industrial wastewater (Deegan, 2011; Areerachakul *et al.*, 2007**;** El-Sharkawy *et al.*, 2007; Awaleh and Soubaneh, 2014). Many studies have tested the use of activated carbon against inorganic pollutants such as cadmium (Ajmal *et al.*, 2006**;** Acharya *et al.*, 2009), lead, copper (Koutcheiko *et al.*, 2007; Kadirvelu *et al.*, 2001**),** methylene blue (Hassan and Elhadidy, 2017; Deng *et al.*, 2009), dissolved organic carbon (DOC) (Xing *et al.*, 2008), and organic pollutants such as phenol and its derivatives (Tseng *et al.*, 2003; Kumar *et al.*, 2007; Nouri and Haghseresht, 2004; Wu *et al.*, 2005).

Several studies have shown that aerobic microorganism lives mainly on the surface of carbon particle and relatively few of them live in carbon pores (Ha *et al.*, 2009**,** Sutton and Mishra, 1994**;** Joseph *et al.*, 2010). In 2009 Al-Jlil conducted a study to determine the reduction of chemical oxygen demand (COD) and biological oxygen demand (BOD) from domestic wastewater by using sedimentation, aeration, activated sludge, sand filter, and activated carbon. The mean maximum COD and BOD reduction was 92.1% and 97.6% respectively. Devi and Dahiya (2008) studied the reduction of COD and BOD in domestic wastewater through the use of mixed adsorbent carbon, (MAC), and commercial activated carbon, (CAC). Under optimum condition, maximum COD and BOD reduction of 95.8% and 97.4% respectively was achieved when using MAC and 99.0% and 99.5% respectively when using CAC. The results showed that MAC could be potentially beneficial in the removing COD and BOD from wastewater. Devi *et al*. (2008) assessed the reduction of COD and BOD in wastewater from a coffee processing plant by using activated carbon made from avocado peels. The maximum percentage of reduction in COD and BOD concentration under optimum operating conditions when using avocado peel carbon (APC) is 98.2% and 99.1%, respectively, and with CAC the reduction is 99% and 99.3%, respectively. As the adsorption capacity of APC is comparable with that of CAC in reducing COD and BOD concentration, it could be a promising technique for the treatment of domestic wastewater

Activated carbon is obtained from various sources such as saw dust, rice husk, wood, coconut shell, etc. These materials are cheap and readily available. The preparation of activated carbon is easy and fast, and can be done by placing carboneous source material in a tank without oxygen and pyrolysing them at a high temperature, usually between 600° and 900° C, to produce char (Mohamed, 2011). The source material is exposed to different chemicals, such as argon and nitrogen. The char is then blasted with steam at temperatures above 250° C, usually between 600° and 1200° C, to oxidize or "activate" them. This process removes all volatile compounds as layer after layer of carbon atoms are peeled off, thereby enlarging the internal pores and leaving behind a carbon skeleton. The internal surface area of a material is increased when the number of carbon atoms is reduced. After the process is completed, three pounds of source material usually yields one pound of activated carbon. This increases the surface area of the material and render them suitable for adsorption.

The use of activated carbon as a coat on plastic media such as Cosmo ball as a biofilm media has the potential to offer alternative design for compact treatment plants which are more effective than conventional wastewater treatment systems. Cosmo ball is a commercial media light in weight, floats in water, and is therefore easy to remove or clean whenever required, it has been proven to successfully remove acceptable levels of organic matter and nutrient. The main unique characteristics underlying these systems are the high porosity of activated carbon and its high surface area. These properties can increase the efficiency of the treatment processes through the adsorption of contaminants by activated carbon as well as by increasing the numbers of the bacteria which grow on their surfaces. The long residence time required to treat organic wastewater pollutants could give rise to economic problems with regard to the financial cost of constructing a large-volume basin as well as the large space required for its construction. Therefore, a new, rougher material is needed to improve the efficiency of wastewater treatment operationally by improving the area of solid retention and economically by reducing the large land area currently required to operate wastewater treatment plants.

1.2 Problem Statement

Presently, systems such as oxidation ponds, package systems, aerated lagoon, activated sludge and different types mechanical plants are used to treat polluted wastewater (Birima, 2008; Shahot and Ekhmaj, 2012). However, these sewage treatment plants (STPs) have various shortcomings. Amongst them are:

1. Commercial plastic media such as Cosmo ball has limited surface area, and this has limitation to its use when high biofilm growth rate are required.
2. The increase in flow and organic loading of wastewater due to increasing population require large STP to treat organic wastewater pollutants, this has given rise to economic problems (Yang *et al.*, 2012).
3. Conventional biological processes are designed to meet acceptable level of treatment standard. However, they typically do not remove ammonia and phosphorus to the extent required to protect receiving water (Birima, 2008).

Wastewater treatment facilities are increasingly required to implement processes which reduce effluent nutrient concentrations to safe levels (Naidoo and Olaniran, 2013). This could pose a challenge to wastewater treatment plants because it usually requires major process modifications to the plants, for example making a portion of the aeration basin anaerobic and anoxic, which reduce aerobic volume and limit nitrification capacity. Therefore, the present study is important and useful in reducing the large aeration size of STP by utilizing biofilm process such as activated carbon coated Cosmo balls that is capable of producing high quality effluents that comply with the effluent discharge standards.

The aim of this research is to develop an activated carbon coat on the surface of Cosmo balls for biofilm system in order to provide a large surface area for microbes to attach to. This would result in reduced retention time, hence making the plant more compact and effective compared to conventional systems. In addition, this plant produces effluent with excellent quality; meet the stringent requirements for discharge; retains suspended particles within the bioreactor, thereby significantly reducing plant footprint and produces less sludge.

1.3 Objectives of the Study

The following objectives have been identified for the successful completion of this research project.

1. To develop coating method on a biofilm media using granular activated carbon (GAC) and characterize the new coat surface.
2. To study the developmental growth rate of biofilm when surface is coated with GAC
3. To assess the performance of activated carbon coated material and the kinetic study with regard to organic removal at different loadings.
4. To compare the GAC coated Cosmo balls against non-coated Cosmo balls in removing ammonia nitrogen and evaluation of the mass balance.

1.4 Scope of the Study

To achieve the above mentioned objectives, the following tasks were undertaken:

1. The surface of Cosmo balls was coated using the (paint-spray-dry) technique since it is an easy way to produce activated carbon material which will be used as a media in attached growth process. After the Cosmo balls were coated several tests were conducted to determine the porosity, surface area (by using BET), and surface roughness. In addition, integrating tests were conducted on the coating to ensure that they adhere perfectly onto the surface of the balls.
2. Aerobic batch experiments were done in two 5000 mL beaker; one beaker was filled with four AC-coated Cosmo balls while another was filled with four non-coated carriers. Prior to conducting the experiments, the carriers were gently rinsed with deionized water to remove any compounds on the Cosmo ball. Approximately five litres of domestic wastewater were added to the beaker. Aeration was provided by an air diffuser installed at the bottom of the reactor. The degradation of BOD and COD were determined with time at a temperature of $26 \pm 2\ ^oC$, and to observe the formation and growth of bacteria on the carriers.
3. Prior to using the Cosmo balls to treat wastewater, a laboratory-scale reactor was successfully designed, constructed and installed at the Kolej 10 wastewater treatment plant close to the Faculty of Engineering, Universiti Putra Malaysia to facilitate the pumping of a continuous flow from the equalization tank to the reactor. The lab-scale reactor consists of a 700-litre equalization tank, 26 litter anoxic tank, a 90-littre aeration tank, and a settling tank. The reactor was operated under three different retention times of 6, 3, and 1.5 hours. In order to evaluate the performance of the non-coated and coated Cosmo balls under three influent flows of 60, 30 and 15 L/h, samples were taken from the inlet and outlet points and several parameters, namely COD, BOD_5, TSS, VSS, NO_3, NO_2, turbidity, pH ammonia nitrogen and total phosphate, were analyzed using standard methods to determine the performance of the plant in removing organic matters and nutrient.
4. Two kinetic models were studied namely Modified-Stover Kincannon and Grau model.
5. Mass balance was analysed with regard to BOD, COD, ammonia and TP at two hydraulic retention time 6 and 3 hours.

1.5 Thesis Layout

This thesis consists of five chapters. The introduction in Chapter 1 first gives the background of the study and the problem statement, and ends with stating the objectives and scope of the research. Chapter 2 covers literature review with the discussion focussing on coating methods, adhesion tests, and biofilm application in wastewater treatment with regard to removal of organic matters and nutrients. In Chapter 3, covers preparation of substrates, coating process, types of analytical equipment used, as well as analytical methods such as pH, BOD, COD, TSS, NH_3-N, NO_3, NO_2 TP and VSS were analysed in accordance with the Standard Methods for the Examination of Water and Wastewater. Chapter 4 presents the results and discussion on the coating and the performance of the system before and after coating was applied. Finally, Chapter 5 wraps up the thesis with a conclusion and recommendations for future work.

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