ENHANCING ANAEROBIC DIGESTION OF PALM OIL MILL EFFLUENT (POME) BY ADDITION OF CaO DERIVED FROM WASTE COCKLE SHELL AS A BUFFERING SOLUTION IN AN UPFLOW ANAEROBIC SLUDGE BLANKET (UASB) REACTOR

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pelbagai kaedah telah dikaji untuk merawat efluen kilang sawit (POME). Walaubagaimanapun, tidak semua kaedah rawatan itu sesuai dan cekap dalam merawat POME. Reaktor selimut bakteria anaerobik aliran menaik (UASB) boleh digunakan untuk merawat air sisa berkepekatan tinggi seperti POME dengan kecekapan penyingkiran permintaan oksigen kimia (COD) yang lebih tinggi serta kadar pengeluaran metana (CH_4) yang baik boleh dicapai. Dalam pencernaan anaerobik, pH yang optima perlu dikekalkan untuk memastikan proses anaerobik mengurai bahan organik secara berkesan. Potensi menggunakan bahan buangan sebagai penyelesaian penimbal atau agen peneutralan yang sesuai boleh membawa kepada pendekatan yang murah dan berdaya maju daripada segi ekonomi. Sehingga kini, masih kurang kajian membincangkan potensi penggunaan kulit kerang buangan sebagai agen peneutralan rawatan POME. Oleh itu, kajian ini telah dilakukan untuk menilai kesan penambahan larutan penimbal yang mengandungi kalsium oksida (CaO) yang dihasilkan daripada sisa kulit kerang terhadap keberkesanan reaktor UASB merawat POME secara berterusan. Secara keseluruhan, terdapat 4 fasa yang terlibat dalam mencapai objektif utama: i) mengkaji hubungkait antara parameter operasi pengkalsinan dan hasil CaO ii) mengkaji kesan penambahan kulit kerang terkalsin (CCS) ke atas penghasilan biometana dan kebolehrawatan anaerobik air sisa buatan dan POME iii) kajian kinetik dan kajian mikrob telah dijalankan ke atas reaktor UASB yang merawat POME dengan penambahan CCS. iv) menilai pengeluaran biohidrogen yang dihasilkan melalui proses pembaharuan kering (DR) yang menggunakan biometana yang dihasilkan daripada pencernaan anaerobik POME dalam reaktor berterusan UASB. Setelah parameter operasi pengkalsinan telah mencapai hasil CaO tertinggi (pada suhu 900°C, hasil CaO adalah 97.57%), CCS dilarutkan di dalam air untuk mendapatkan larutan CaO berkepekatan diantara 1 hingga 15 g/L sebelum digunakan sebagai larutan penimbal dalam pencernaan anaerobik POME. Kesan penambahan CCS yang berbeza-beza kepekatan di dalam pencernaan anaerobik POME dalam reaktor berterusan UASB yang beroperasi pada kadar pemuatan organik (OLR) yang pelbagai dari 0.3 hingga 13.4 g COD/L.h, masa pengekalan hidraulik (HRT) diantara 1 hingga 6 hari dan pada suhu yang berbeza diantara 28 hingga 38°C dengan suapan i) air sisa buatan mengandungi COD dengan kepekatan diantara 1,665 hingga 3,416 mg/L ii) POME dengan kepekatan COD diantara 1,030 hingga 40,460 mg/L telah dinilaikan. Kesan penambahan CCS berbanding Na_2CO_3 komersial juga telah ditentukan. Hasil kajian menunjukkan penambahan CCS semasa pencernaan anaerobik air sisa buatan menujukkan pengeluaran COD sehingga 92.35% berbanding sodium karbonat, (Na_2CO_3) komersial (79.36%). Sebagai perbandingan dengan rawatan air sisa buatan, penggunaan CCS dalam rawatan POME telah meningkatkan penyingkiran COD pada kira-kira 6% (98% berbanding 92%) dan menggalakkan pengeluaran berterusan biogas diantara 14 dan 22 L/d pada OLR sebanyak 7 hingga 14 g COD/L.hari. Untuk air sisa buatan, hanya sedikit penghasilan biogas direkodkan. Parameter biokinetik yang dinilai bagi reaktor UASB dalam rawatan POME dengan penambahan CCS adalah hasil pertumbuhan (Y_G) iaitu 3.906 g VSS/g $COD_{removed}$.d; pereputan biojisim tertentu (b), 0.233/d; kadar pertumbuhan biojisim khusus (μ_{maks}), 1.861/d; pemalar tepu (K_s), 3.459 g-COD/L. Dengan menggunakan teknik pengklonan 16S ribosomal asid ribonukleik (16S rRNA), genus *methanosarcina* dan *methanosaeta* adalah metanogen yang dominan yang ditemui di dalam reaktor UASB bagi kedua-dua jenis substrat. Secara keseluruhan, populasi mikrob (bakteria dan arkea) di dalam POME sebagai substrat adalah lebih

pelbagai berbanding di dalam air sisa buatan kerana kewujudan yang banyak populasi mikrob di dalam POME mentah. Kajian juga merekodkan proses DR menukar biogas kepada 83% hidrogen dan 100% CH_4 telah ditukar. Kesimpulannya, penggunaan CCS sebagai larutan penimbal telah meningkatkan pencernaan anaerobik POME di dalam reaktor berterusan UASB dari segi kecekapan pengeluaran COD dan kadar penghasilan biogas. Parameter biokinetik yang dinilai boleh digunakan untuk meramalkan prestasi pencernaan anaerobik POME yang menggunakan reaktor UASB. Potensi menggunakan teknik biologi molekul untuk menyediakan profil terperinci struktur komuniti mikrob akan meningkatkan pengoptimuman proses rawatan dan akhirnya menghasilkan kualiti efluen yang boleh diterima.

ABSTRACT

Various methods have been studied for the treatment of Palm Oil Mill Effluent (POME). However, not all of the treatment methods are suitable and efficient in POME treatment. The Upflow Anaerobic Sludge Blanket (UASB) reactor could be utilised to treat a high-strength wastewater such as POME and a higher Chemical Oxygen Demand (COD) removal efficiency and a good methane production rate could be achieved. In the anaerobic digestion of POME, optimum pH should be maintained to ensure the anaerobic process degrades organic matter effectively. The potential of utilizing waste into suitable buffering solutions or neutralization agent could lead to an inexpensive and economically viable approach. To date, there is still a lack of research discussing the potential of using waste cockle shells as a neutralization agent for POME treatment. Thus, this study is designed to evaluate the impact of adding a buffering solution containing calcium oxide (CaO) derived from waste cockle shells on the performance of the continuous UASB reactor treating POME. Overall, four phases were involved in achieving the main objective; i) establishing the correlation between calcination's operating parameters of waste cockle shells and CaO yield. ii) study the impact of adding calcined cockle shells (CCS) on biomethane production and anaerobic treatability of synthetic wastewater and POME .iii) a kinetic and microbial study was conducted on the UASB reactor treating POME with the addition of CCS. iv) evaluating the biohydrogen production via a dry reforming (DR) process fed with biomethane produced from anaerobic digestion of POME in the continuous UASB reactor. Once the calcination's operating parameters of obtaining the CCS containing the highest CaO (temperature 900°C, 97.57% CaO) were established, the CCS was dissolved in water (between 1 and 15 g/L concentrations) before the utilization as a buffering solution in the anaerobic digestion of POME. The impact of adding CCS in various concentrations during the anaerobic digestion of POME in the UASB reactor operated at various organic loading rate (OLR) (0.3 to 13.4 g COD/L.day), hydraulic retention time (HRT) (1 to 6 day) and different temperature (28 and 38ºC) fed with i) synthetic wastewater containing chemical oxygen demand (COD) concentrations between 1,665 and 3,416 mg/L, ii) POME with COD concentrations of 1030 and 40,460 mg/L was evaluated. The impact of adding CCS in comparison with commercial Na_2CO_3 was also determined. The result indicated that adding CCS during the anaerobic digestion of synthetic wastewater has enhanced the COD removal up to 92.35% compared with commercial Na_2CO_3 (79.36%). In comparison with the UASB reactor treating synthetic wastewater, the utilization of CCS in treating POME has improved the COD removal at about 6% (98% compared to 92%) and promoted the continuous production of biogas between 14 and 22 L/d at OLR of 7 to 14 g COD/L.day. As for synthetic wastewater, minimal biogas production was recorded. The biokinetic parameters evaluated on the UASB reactor treating POME with the addition of CCS were the growth yield (Y_G), 3.906 g VSS/g $COD_{removed}$.d; the specific biomass decay (*b*), 0.233 d^{-1}; the specific biomass growth rate (μ_{max}), 1.861 d^{-1}; and the saturation constant (K_s), 3.459 g-COD/L. Using the 16S ribosomal ribonucleic acid (rRNA) cloning technique, the genus *methanosarcina* and *methanosaeta* were dominant methanogens found in both feeding schemes in the UASB reactor. Overall, the microbial population (Bacteria and Archaea) in POME as the substrate is more diverse than synthetic wastewater due to the abundance of microorganism population in raw POME, which was used as a substrate. The result also shows that the dry reforming converted biogas into 83 % of H_2, and 100% CH_4 conversion was recorded. In

conclusion, using CCS as a buffering solution has proven to improve the anaerobic digestion of POME in a continuous UASB reactor in terms of COD removal efficiency and the biogas production rate. The evaluated biokinetic parameters could be utilized for predicting the performance of the anaerobic digestion of POME using the UASB reactor. The potential of using molecular biology techniques to provide a detailed profile of the microbial community structure will enhance the optimization of treatment processes and eventually produce acceptable effluent quality.

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