

**DEVELOPMENT OF MEMBRANE SUPPORT
FOR LEVULINIC ACID EXTRACTION USING
SUPPORTED LIQUID MEMBRANE PROCESS**

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DOCTOR OF PHILOSOPHY

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ABSTRAK

Permintaan asid levulinic (LA) dan derivatifnya yang berharga semakin meningkat dalam pelbagai aplikasi. Pada masa kini, LA daripada biojisim telah mendapat perhatian disebabkan keimbangan pengurangan bahan api fosil, peningkatan harga minyak dan permasalahan alam sekitar. Namun, cabaran utama dalam penapisan bio adalah untuk mengasingkan LA daripada produk biojisim. Penyelidikan ini bertujuan untuk membangunkan kaedah baru untuk memisahkan LA dari produk biojisim dengan menggunakan membran cecair yang disokong (SLM). Pada peringkat awal kajian, formulasi fasa membran cecair organik untuk pengekstrakan LA telah dibuat dan parameter operasi dalam lembaran rata SLM (FSSLM) telah dioptimumkan dengan menggunakan membran kepingan rata hibrid graphene/polietilsulfona (PES) sebagai penyokong. Seterusnya, membran sokongan PES diubahsuai menggunakan pelbagai jenis pengisi and agen pembentuk liang. Membran dicirikan dari segi morfologi, keliangan, hidrofobisiti membran, dan kekuatan mekanikal. Formulasi larutan dop yang diubahsuai telah digunakan untuk penyiasatan fabrikasi gentian berongga (HF) PES selanjutnya. Parameter fabrikasi HF seperti cecair lubang, jurang udara dan kelembapan relatif dalam teknik putaran kering-basah telah disiasat. Seterusnya, tiga faktor operasi gentian berongga membran cecair yang disokong (HFSLM) seperti kepekatan tri-n-otlamina (TOA), sodium hidroksida (NaOH), dan LA disaring menggunakan reka bentuk faktor penuh. Kajian kinetik telah dijalankan, dan model telah dibangunkan. Akhirnya, kecekapan teknik SLM dalam pemisahan LA dari larutan biojisim pelepas kelapa sawit (OPF) diuji dengan menggunakan serat berongga yang dioptimumkan pada keadaan operasi SLM yang optimum. Hasilnya, 89.2% LA telah diekstrak melalui FSSLM dari larutan 10 g/L LA dengan menggunakan formulasi membran cecair dari 0.3 M TOA di dalam pelarut 2-etyl-1-heksanol dengan 0.5 M NaOH sebagai agen pelucutan. SLM telah dijalankan dengan kadar aliran suapan dan pelucutan pada 75 ml/min. Sokongan membran lembaran rata terbaik untuk pengekstrakan LA dibuat menggunakan 0.1 jisim% graphene dan polietilena glikol 200 sebagai bahan pengisi dan pembentuk liang masing-masing. Ia mempunyai permukaan hidrofobik dengan sudut kontak 98 °, keliangan tinggi 87.1% dan kekuatan tegangan 1032.9 kPa. Ia menghasilkan pengekstrakan LA tertinggi iaitu 89.2%. Gentian terbaik dengan permukaan hidrofobik, 94.1 °, keliangan 77.57%, dan tegangan tegangan 1524.7 kPa dibuat dengan menggunakan 60% v/v dimetilasetamida sebagai cecair lubang, jurang udara 6 cm dan pada kelembapan relatif 86%. Ia menghasilkan pengekstrakan LA tertinggi iaitu 72.2%. Seterusnya, data saringan digunakan sebagai titik tengah (0.5 M TOA, 0.7 5M NaOH, dan 10 g/L LA) untuk mengoptimumkan pengekstrakan LA melalui Metodologi Permukaan Respons. Keadaan optimum dicapai pada 0.32 M TOA, 0.77 M NaOH, dan 10.08 g/L LA menggunakan reka bentuk berpusatkan wajah. Hasil pengekstrakan LA meningkat selepas proses pengoptimuman kepada nilai 74.82%. Berdasarkan kajian kinetik, langkah pengawalan kadar dalam pengekstrakan ini adalah penyebaran LA di seluruh lapisan filem antara fasa suapan dan organik. Pengangkutan LA terbukti menjadi penyebaran tulen, dan penyebaran mengawal pengangkutan LA. Model fluks penyebaran yang baru dikembangkan dianggap mencukupi untuk pengekstrakan LA melalui HFSLM. Hasilnya, 63.62% LA telah diekstrak, dan 40.44% LA diperoleh dari larutan biojisim OPF yang mengandungi 10.05 g/L LA. Berdasarkan kajian ini, kaedah SLM ini sesuai digunakan untuk menghilangkan perencat, LA, dari hidrolisat biomas sebelum penapaian dan juga terbukti sebagai kaedah yang berkesan untuk memisahkan LA dari larutan biomas berair dan OPF berbanding dengan beberapa proses pemisahan lain.

ABSTRACT

Levulinic acid (LA) and its valuable derivatives are in growing demand in various applications. Nowadays, LA from biomass gained significant attention due to concerns over fossil fuel depletion, increasing oil price, and environmental problems. However, the primary challenge in biorefinery is the separation of LA from biomass products. This research aimed to develop a new method for separating LA from biomass products using a supported liquid membrane (SLM). In the initial stage of the study, the organic liquid membrane (LM) phase formulation for LA extraction was established and the operating flat sheet SLM (FSSLM) parameters were optimized using a hybrid graphene/polyethersulfone (PES) flat membrane as the support. Then, the PES membrane supports were modified using various hydrophobic fillers and pore forming agents. The membrane were characterized in terms of morphology, porosity, membrane hydrophobicity, and mechanical strength. The modified dope solution formulation was used for further PES hollow fiber (HF) spinning investigation. In HF spinning, the fabrication parameters such as bore liquid, air gap and relative humidity in the dry-wet spinning technique were investigated. Later, three hollow fiber supported liquid membrane (HFSLM) operating factors such as tri-n-octylamine (TOA), sodium hydroxide (NaOH), and LA were screened using the full-factorial design (FFD) and optimized using face-centered design (CCF). The kinetic study was conducted, and a model was developed. At last, the efficiency of the SLM technique in LA separation from oil palm frond (OPF) biomass solution was tested using optimized HF at the optimal SLM operation conditions. As a result, 89.2% of LA was extracted via FSSLM from 10g/L of LA solution using a LM formulation of 0.3 M TOA in 2-ethyl-1-hexanol with 0.5 M of NaOH as stripping agent at both feed and stripping flowrate of 75 ml/min. The best flat sheet membrane support was fabricated using 0.1 wt.% graphene and polyethylene glycol 200 as filler and pore-forming agents, respectively. It had a hydrophobic surface with a contact angle of 98°, porosity of 87.1% and tensile stress of 1032.9 kPa. Moreover, it resulted in the highest LA extraction of 89.2%. Furthermore, the best fiber with a hydrophobic surface of 94.1°, 77.57% of porosity, and tensile stress of 1524.7 kPa were spun using 60% v/v dimethylacetamide as the bore fluid, an air gap of 6 cm and relative humidity of 86%. It yielded the highest LA extraction of 72.2%. Moreover, the screening using FFD results were used as a center point (0.5 M TOA, 0.75 M NaOH, and 10 g/L of LA) to optimize the LA extraction in Response Surface Methodology. The optimal conditions were 0.32 M TOA, 0.77 M NaOH, and 10.08 g/L LA using a CCF. The LA extraction yield was increased after the optimization to a value of 74.82%. Based on the kinetic study, the rate-controlling step in this investigation is LA diffusion across the film layer between the feed and organic phases. The LA transport was proven to be a pure diffusion, and the diffusion controls the LA transport. A newly developed diffusion flux model is considered adequate for LA extraction through HFSLM. The developed methods successfully extracted 63.62% of LA and recovered 40.44% of LA from an OPF biomass solution containing 10.05 g/L LA. Based on this study, SLM method can be used to remove inhibitor, LA, from biomass hydrolysate before fermentation and also proved to be an effective method to separate LA from aqueous biomass solution and OPF compared to some other separation processes.

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