

**DEVELOPMENT OF POLYVINYLDENE
FLUORIDE-BENTONITE MEMBRANE IN
MEMBRANE DISTILLATION SYSTEM FOR
TREATMENT OF PALM OIL MILL EFFLUENT**

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I hereby declare that the work in this thesis is based on my original work except for quotation 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

Nanozarah tanah liat kebiasaannya digabungkan dalam membran polimer untuk memperbaiki sifat fizikokimia membran yang dihasilkan. Walau bagaimanapun, penyelidikan mengenai aplikasi membran nanokomposit untuk rawatan efluen industri adalah masih terhad. Matlamat kajian ini adalah untuk membangunkan membran nanokomposit gentian geronggang polivinilidin florida (PVDF) yang digabungkan dengan bentonit untuk rawatan efluen kilang kelapa sawit (POME) menggunakan sistem penyulingan bermembran secara langsung (DCMD). Dalam kerja ini, larutan terdiri daripada 12% berat PVDF yang diadun dengan enam muatan bentonit yang berbeza (0.25 % berat, 0.30 % berat, 0.40 % berat, 0.50 % berat, 0.75 % berat, dan 1.00 % berat) telah disediakan. Membran-membran gentian geronggang tersebut kemudiannya disediakan melalui kaedah basah-jet penyongsangan fasa dan dicirikan menggunakan mikroskop elektron pengimbas (SEM) dengan sinar-X penyebaran tenaga (EDX), spektroskopi inframerah jelmaan Fourier (FTIR), analisis termogravimetri (TGA), kalorimeter pengimbasan pembeza (DSC), tekanan kemasukan cecair (LEP), keliangan membran, sudut sesentuh, pembelauan sinar-X (XRD) dan mikroskop daya atom (AFM). Sebelum ujian, membran diuji terlebih dahulu menggunakan air ternyahion untuk melihat kebocoran. Untuk aplikasi sebenar, sampel POME yang diambil dari kolam anaerobik digunakan dalam kajian ini. Fluks resapan tertinggi diperolehi oleh membran PVDF-0.50% berat bentonit diikuti oleh membran PVDF-0.30 % berat bentonit iaitu sebanyak $3.62 \pm 1.25 \text{ kg/m}^2.\text{hr}$ dan $3.45 \pm 0.51 \text{ kg/m}^2.\text{hr}$ masing-masing semasa rawatan POME. Bagi kecekapan penyingkiran, lebih 99% daripada jumlah pepejal terlarut (TDS), keperluan oksigen kimia (COD), nitrogen nitrat, warna dan kekeruhan telah dikeluarkan daripada efluen. Membran PVDF-0.30 % berat bentonit berprestasi lebih baik daripada membran PVDF-0.50 % berat bentonit dalam penyingkiran TDS. Membrane PVDF-0.30% bentonit telah dipilih untuk ujian lanjutan dengan POME mentah dan kajian jangka panjang kerana membran tersebut adalah paling hidrofobik ($94.77 \pm 0.13^\circ$) berbanding dengan membran lain. Semasa ujian POME mentah, fluks resapan turun kepada $1.41 \pm 0.62 \text{ kg/m}^2.\text{jam}$ berbanding $3.45 \pm 0.51 \text{ kg/m}^2.\text{jam}$ apabila membran tersebut diuji dengan POME dari kolam anaerobik. Pengurangan fluks boleh dikaitkan dengan POME mentah yang mengandungi kepekatan tinggi pepejal terampai, minyak dan gris yang meningkatkan kemungkinan masalah kekotoran. Untuk menentukan kestabilan membran dan kecenderungan kekotoran, proses DCMD jangka panjang telah dijalankan sehingga 72 jam. Diperhatikan bahawa fluks resapan berubah-ubah sepanjang eksperimen dan kemudian stabil pada akhir eksperimen dengan fluks purata ialah $3.342 \pm 1.26 \text{ kg/m}^2.\text{hr}$. Kecekapan penyingkiran melebihi 95% telah dilaporkan untuk semua parameter kualiti air. Keputusan menunjukkan bahawa membran PVDF-0.30 % berat bentonit boleh memperoleh fluks resapan yang tinggi dan menghasilkan resapan berkualiti tinggi yang bebas daripada kekotoran semasa kajian jangka panjang. Kajian ini menyerlahkan peningkatan sifat dan prestasi membran apabila bentonit dimasukkan ke dalam membran polimer.

ABSTRACT

Clay nanoparticles are commonly incorporated in polymeric membranes to improve the physicochemical properties of the fabricated membrane. However, research on the application of the nanocomposite membrane for industrial effluent treatment remains limited. The aim of this study is to develop polyvinylidene fluoride (PVDF) incorporated bentonite hollow fiber nanocomposite membranes for palm oil mill effluent (POME) treatment using direct contact membrane distillation (DCMD) system. In this work, solutions consisted of 12 wt% PVDF blended with six different bentonite loadings (0.25 wt%, 0.30 wt%, 0.40 wt%, 0.50 wt%, 0.75 wt% and 1.00 wt%) were prepared. The hollow fiber membranes were then fabricated via jet-wet phase inversion method and characterized using scanning electron microscope (SEM) and energy dispersive x-ray (EDX), fourier transform infrared (FTIR), thermal gravimetric analysis (TGA), differential scanning calorimetry (DSC), liquid entry pressure (LEP), membrane porosity, contact angle, X-ray diffraction (XRD) and atomic force microscope (AFM). Prior to testing, the membranes were first tested using deionized water to observe leakage. For the actual application, POME samples collected from an anaerobic pond were used in this study. The highest permeate flux was obtained by PVDF-0.50 wt% bentonite membrane followed by PVDF-0.30 wt% bentonite membrane which was $3.62 \pm 1.25 \text{ kg/m}^2.\text{hr}$ and $3.45 \pm 0.51 \text{ kg/m}^2.\text{hr}$ respectively during the POME treatment. For removal efficiencies, over 99% of total dissolved solids (TDS), chemical oxygen demand (COD), nitrate nitrogen, color, and turbidity were removed from the effluent. The PVDF-0.30 wt% bentonite membrane performed better than the PVDF-0.50 wt% bentonite membrane in TDS rejection. The PVDF-0.30 wt% bentonite membrane was chosen for further testing with raw POME and long-term studies because the membrane is the most hydrophobic ($94.77 \pm 0.13^\circ$) in comparison to other membranes. During the raw POME test, the permeate flux drops to $1.41 \pm 0.62 \text{ kg/m}^2.\text{hr}$ compared to $3.45 \pm 0.51 \text{ kg/m}^2.\text{hr}$ when the membrane was tested with POME from an anaerobic pond. The flux reduction can be attributed to the raw POME contains a high concentration of suspended solids, oil and grease which increased the likelihood of fouling problem. To determine the membrane stability and fouling propensity, a long-term DCMD process was conducted for up to 72 hours. It was observed that the permeate flux fluctuated throughout the experiment and then stabilized at the end of the experiment with the average flux is $3.342 \pm 1.26 \text{ kg/m}^2.\text{hr}$. Removal efficiencies of over 95% were reported for all water quality parameters. The results showed that the PVDF-0.30% bentonite membrane could obtain a high permeate flux and produce a high quality permeate that is independent of the fouling during the long-term study. This study highlighted the improvement in membrane properties and performance when bentonite was incorporated into the polymeric membrane.

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