BIOSORPTION OF METHYLENE BLUE DYE USING SEAWEED BIOMASS

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We hereby declare that we have checked this thesis and in our 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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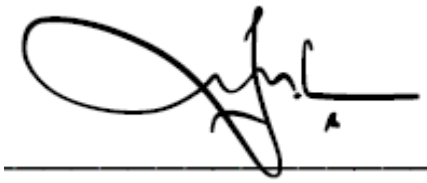

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti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

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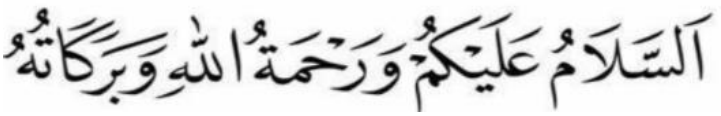

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ABSTRAK

Pewarna azo telah digunakan secara meluas dalam industri tekstil kerana pewarna tersebut mudah diproses, menawarkan kos yang murah, mudah digunapakai, berdaya tahan tinggi dan tersedia dalam pelbagai warna. Terkini, penyelidikan terhadap sistem rawatan air sisa berwarna dari industri yang bercirikan alam sekitar dan ekonomi menjadi perhatian utama penyelidik. Oleh itu, objektif kajian ini adalah untuk mengenalpasti potensi rumpai laut Malaysia untuk dijadikan penjerap mesra alam kepada pewarna azo, *methylene blue* (MB). Saringan lima jenis spesis rumpai laut liar daripada kumpulan merah, hijau dan coklat dinilai dari segi keupayaan maksimum penjerapan(q_{max}) dan afiniti(*b*) dan proses pra-rawatan fizikal dan kimia. Ciri-ciri rumpai laut yang berpotensi kemudian diselidik menggunakan spektrofotometer inframerah *fourier transform (FT-IR),* pengimbas mikroskopi electron dengan analisis tenaga X-Ray *dispersive (SEM-EDX),* analisis termogravimetrik *(TGA)*, *Brunauer-Emmett-Teller (BET)* dan titik zero cas (pH_{pzc}). Eksperimen penjerapan menggunakan mod kelompok telah dijalankan menggunakan kaedah *satu-faktor-per-masa* (OFAT) bagi mengkaji kesan parameter operasi seperti masa dedahan (5- 360min), pH (2–11), dos penjerap (0.2 – 1.2 g/L), kepekatan pewarna (50 – 200 mg/L) dan suhu (30-50°C). Seterusnya, kajian pengoptimuman dijalankan menggunakan *Response Surface Methodology-Central Composite Design* (RSM-CCD). Sejumlah 20 sampel dimatrikkan untuk membina model kuadratik. Kajian penyahpisiran dilakukakan bagi mengkaji potensi guna semula bahan penjerab. Isoterm tidak linear seperti isoterm keseimbangan (model dua parameter dan model tiga parameter) dan isotherm kinetik dilakukan untuk menganalisis mekanisma penjerapan. Seterusnya, verifikasi terhadap model dijalankan menggunakan *Artificial Neural Networks* (ANN) untuk menilai kejituan ramalan ke atas rawatan pengasingan pewarna MB. Keputusan kajian menunjukkan masa keseimbangan untuk semua jenis spesis rumpai laut dicapai dalam tempoh 60-80 min pada suhu 27°C. Pada kepekatan rendah (<1000 mgSL), *E. spinosum* daripada spesis rumpai laut merah menunjukkan potensi paling tinggi dalam keupayaan penjerapan, q_{max} dan *b*. Disebabkan proses pra-rawatan tidak menggalakkan peningkatan keupayaan penjerapan, *E.spinosum* asli digunakan untuk keseluruhan aktiviti kajian. Proses pengoptimunan menggunakan model statistik RSM-CCD menunjukkan kapasiti maksimum penjerapan boleh dicapai sebanyak 399mg/g (>95%) pada masa dedahan 60min, pH 6.9-7.1, dos penjerap sebanyak 0.72 g/L dan kepekatan MB sebanyak 300 mg/L. Setelah empat kitaran berturutan, kadar penjerapan pewarna MB menurun dari 94.5% kepada 48.5%, dan kadar penyahpisiran menurun dari 51.5% kepada 23.4%. Data eksperimen mematuhi isoterm mengikut urutan seperti berikut: Toth > Sips =Hill = Breunor-Sotolongo > Freundlich > Redlich-Peterson > Koble-Corrigan > Langmuir > Tempkin, Dubidin-Radushkeevich. Manakala, data kinetik mematuhi model kinetik pseudo-peringkat kedua. Akhir sekali, verifikasi model menggunakan ANN menunjukkan ANN (R^2=0.9994, adj-R^2=0.9916, MSE=0.19, RMSE=0.4391, MAPE=0.087 and AARE=0.001) memberi kejituan ramalan yang tepat. Kesimpulannya, rumpai laut merah *E.spinosum*, terbukti mempunyai potensi yang baik sebagai alternatif penjerab semulajadi terutamanya untuk menyingkirkan pewarna MB.

ABSTRACT

Azo dye has been extensively used in textile dyeing processes due to its simplicity in production, cost effectiveness, ease of application, durability, and availability in various colours.. At present, investigation on treatment system that promoting environmental and economic sustainability for the remove dyes from industries have received numerous attentions among researchers. Therefore, this study is aimed to investigate the potential of Malaysian seaweed to be used as a biosorbent for the removal of azo-dye, methylene blue (MB) in aqueous solutions. The screening of five indigenous species was based on their maximum biosorption capacity (q_{max}) and affinity (*b*) and the effect of pre-treatment. Characterisation of potential seaweed was determined by fourier transform infrared spectrophotometer (FT-IR), Scanning electron microscopy with energy dispersive X-Ray analysis (SEM-EDX), thermogravimetric analysis (TGA), Brunauer-Emmett-Teller (BET), and points zero charge (pH_{pzc}). The effect of various operational parameters such as contact time (5–360 min), pH (2–11), biosorbent dosage (0.2–1.2 g/L), initial concentration (50–200 mg/L) and temperature (30–50°C) on biosorption was investigated further using batch mode study under One-factor-at-time (OFAT) approach. Upon optimisation, the experimental design for the biosorption process was carried out via Response Surface Methodology-Central Composite Design (RSM-CCD). A total of 20 runs were carried out to generate a quadratic model. A desorption study was performed to investigate the reusability of *E.spinosum*. Non-linear models of equilibrium isotherm (consisting of two-parameter models and three-parameter models) and kinetic isotherm were applied to analyse the biosorption mechanism. Model verification using Artificial Neural Networks (ANN) was adopted for an accurate prediction of dye removal. The results reveal that the equilibrium time for all seaweed species can be achieved within 60–80 min at 27°C. At lower MB initial concentrations (< 1000 mg/L), *E. spinosum* from red seaweed had the highest q_{max} and b. The pre-treatment process did not enhance the uptake capacity of *E. spinosum*, raw *E.spinosum* was used for the entire experiment. From optimisation through statistical model, it was observed that the maximum uptake capacity of 399 mg/g (> 95%) is obtained at the equilibrium time of 60 min, pH solution of 6.9–7.1, dosage of 0.72 g/L, and initial dye concentration of 300 g/L. Experimental data complied with the following equilibrium isotherms: Toth > Sips = Hill = Brouers-Sotolongo > Freundlich> Redlich-Peterson > Koble-Corrigan > Langmuir > Temkin, Dubidin-Radushkevich. The kinetic data, however, were better fitted to the pseudo-second-order kinetic model. After four consecutive biosorption/desorption cycles, the MB dye biosorption efficiency decreased from 94.5% to 48.5%, and the dye desorption efficiency decreased from 51.5% to 23.4%. Finally, model verification using ANN demonstrated that the ANN model (R^2 = 0.9994, adj-R^2 = 0.9916, MSE = 0.19, RMSE = 0.4391, MAPE = 0.087, and AARE = 0.001) is able to provide an accurate prediction. As a conclusion, red seaweed of *E.spinosum* was found to have great potential as an alternative natural occurring biosorbent specifically for MB dye removal.

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