

EFFECT OF DIFFERENT EXTRACTION
CONDITIONS ON THE PRODUCTION OF
ANTHRAQUINONE

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Master of Science

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LIST OF SYMBOLS

C	Concentration of solution
Calc	Calculated
C_e	Equilibrium concentration of solution
C_o	Initial concentration of solution
CO ₂	Carbon dioxide
Exp	Experiment
K_L	Adsorption equilibrium constant
K_F	Freundlich constant
ϵ	Absorbance
ϵ	Dielectric constant
μ	Dipole
I	Length
q_e	The amount of adsorption at equilibrium concentration of the solution
q_{max}	Maximum adsorption capacity reflecting a complete monolayer
V	Volume of solution
W	Mass of dry adsorbent used

LIST OF ABBREVIATIONS

AB7	Acid blue 7
AG25	Acid green 25
Alum	Aluminium
ANOVA	Analysis of variance
ARE	Average relative error
BET	Brunauer-Emmett-Teller
CCD	Central composite design
DSSC	Dye-sensitized solar cells
EU	European union
FTIR	Fourier transform infrared
HHP	High hydrostatic pressure
IR	Infrared
LSR	Liquid solid ratio
MAE	Microwave assisted extraction
MG	Malachite green
MO	Methyl orange
OFAT	One factor at a time
RMSE	Residual root mean square
RSM	Response surface methodology
SEM	Scanning electron microscope
SLR	Solid liquid ratio
SSE	Sum of the square of the roots
UAE	Ultrasonic assisted extraction
UV-Vis	Ultraviolet visible

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ABSTRAK

Produk semulajadi telah digunakan sejak turun temurun dan baru-baru ini, saintis telah mula menunjukkan minat terhadap *Morinda citrifolia* dan produknya. Manfaatnya diketahui dimana akar tumbuhan ini dilaporkan menjadi sumber baik sebatian penting, yang dipanggil anthraquinones. Anthraquinone diperolehi secara meluas untuk menghasilkan pewarna kuning atau merah. Walau bagaimanapun, pengekstrakan teknikal dan kaedah pencelupan sebenar pewarna anthraquinone masih belum diterokai. Ditambah pula, pewarna semulajadi selalunya mempunyai hasil warna rendah dan sifat keupayaan warna untuk kekal yang lemah kerana warna tersebut biasanya dikeluarkan dari serat semasa proses mencuci. Kajian ini dijalankan bagi mengkaji kesan nisbah pepejal cecair (SLR) (1: 100 hingga 5: 100), masa pengekstrakan (1 hingga 10 jam), pH (1 hingga 11) dan jenis pelarut (etanol, aseton dan asetonitril) kepada kepekatan anthraquinone menggunakan kaedah pengekstrakan pelarut. Kaedah pengekstrakan pelarut didapati sebagai kaedah pengekstrakan yang paling mudah yang juga dapat meningkatkan hasil warna dan sifat keupayaan warna untuk kekal pada serat. Keadaan paling baik ekstrak anthraquinone telah ditentukan dengan menggunakan Kaedah Permukaan Respons (RSM). Selepas itu, ekstrak anthraquinone dianalisis dan dicirikan menggunakan Ultraungu-Nampak Spektroskopi (UV-Vis Spectroscopy) dan Spektrometer Infra-merah (FTIR). Kajian penjerapan telah dijalankan bagi tiga faktor yang berbeza, iaitu, kadar penjerapan ke atas benang menggunakan pelbagai kepekatan awal pewarna (0.105 kepada 0.117 g/L), keberkesanan penjerapan pada tiga jenis benang (sutera, sutera poliester dan kapas poliester) dan tempoh masa (15 hingga 120 minit) yang diperlukan untuk proses penjerapan pada benang. Kepekatan tertinggi anthraquinone boleh diperolehi dengan menggunakan aseton pada 4:100, 2 jam, dan pH 7 masing-masing bagi SLR, masa pengekstrakan, dan pH. Walau bagaimana pun, kondisi paling baik apabila menggunakan RSM bagi SLR dan masa pengekstrakan masing-masing adalah 4:100 dan 1 jam 30 minit. Penghasilan anthraquinone dalam kajian ini meningkat kira-kira 66.67%; 0.003 g/g (0.102 g/L) berbanding dengan kajian terdahulu; hanya menghasilkan 0.001 g/g anthraquinone (Vázquez et al., 2014). Perbandingan ini membuktikan pengekstrakan pelarut yang digunakan dalam kajian ini adalah sesuai untuk dipraktikkan untuk kajian lanjut. Malah, Vázquez et al. (2014) menggunakan dua jenis pelarut; benzena dan etil asetat, dan memerlukan masa pengekstrakan yang lebih lama (16 jam). Hasil anthraquinone yang berlainan mungkin disebabkan oleh perbezaan jenis bahan mentah, kaedah pengekstrakan dan pelarut yang digunakan. Masa keseimbangan dan kepekatan awal yang sesuai bagi proses penjerapan anthraquinone ialah 60 minit dan 0.117 g/L. Sutera didapati mempunyai keberkesanan penjerapan yang tertinggi, diikuti oleh sutera poliester dan kapas poliester. Susunan persamaan isoterma yang selari dengan data ialah isoterma Langmuir > isoterma Freundlich. Secara kesimpulan, kajian ini mencadangkan bahawa akar pokok mengkudu boleh menghasilkan warna coklat kemerahan yang berpotensi dalam industri tekstil memandangkan keadaan terbaik proses pencelupan pewarna anthraquinone telah ditentukan. Anthraquinone bertindak sebagai alternatif yang mesra alam setara dengan sintetik untuk menjadikan kehidupan yang lebih hijau. Anthraquinone yang diekstrak dalam kajian ini memberi produk bernilai tinggi kepada industri tekstil seperti 'Tenun Diraja Pahang' untuk pengkomersialan.

ABSTRACT

Natural products have been used for generations and recently, the scientists have begun to show interest in *Morinda citrifolia* and its products. Its benefits become known where the roots of these plants were reported to be a good source of important compounds, called anthraquinones. Anthraquinone are widely acquired in nature to produce yellow or red dyes. However, the actual technical extraction and dyeing method of anthraquinone dyes are still unexplored. Moreover, the natural dyes are often having low exhaustion color and poor fastness properties as its color is usually removed from the fibre during washing. This research was carried out to study the effects of types of solvent (ethanol, acetone, acetonitrile), solid liquid ratio (SLR) (1:100 to 5:100), extraction time (1 to 10 hour) and pH (1 to 11) on the concentration of anthraquinone using solvent extraction method. The solvent extraction method are found as a simplest extraction method which are also can improved color yield and the fastness properties of the fibres. The best condition of anthraquinone extract was determined using Response Surface Methodology (RSM). The anthraquinone extract was analysed and characterized using UV-Vis Spectrophotometer and Fourier Transform Infrared Spectroscopy (FTIR) respectively. The adsorption study was then conducted as three different factors, namely, the rate of adsorption on fibre using various initial dye concentrations (0.105 to 0.117 g/L), the effectiveness of adsorption on different types of fibre (spun silk, polyester spun silk and polyester cotton) and the duration of time (15 to 120 minutes) required for the process of adsorption on the fibre. The highest concentration of anthraquinone can be obtained by using acetone at the ratio of 4:100, 2 hours, and pH 7 of SLR, extraction times, and pH, respectively. However, the best condition for anthraquinone extraction using RSM for SLR and extraction times was 4:100 and 1 hour 30 minutes respectively. Production of anthraquinone in this study increased about 66.67 % as; 0.003 g/g (0.102 g/L) compared to previous study; 0.001 g/g of anthraquinone production yield (Vázquez et al., 2014). This comparison proved the solvent extraction used in this study was applicable for further study. In fact, Vázquez et al. (2014) use two types of solvents; benzene and ethyl acetate, and require longer extraction time (16 hours). The different anthraquinone yield might be because of different types of raw material, extraction method and solvent used. The equilibrium time and initial dye concentration fitted for adsorption process of anthraquinone was determined to be 60 minutes and 0.117 g/L, respectively. Spun silk fibre was found has the highest adsorption effectiveness, followed by polyester spun silk and polyester cotton fibre. The order of isotherm equations followed by the present data is Langmuir > Freundlich isotherm. As for conclusion, the study suggested that the roots of *Morinda citrifolia* can yield a reddish brown colour that has potential in the textile industries since the best conditions of dyeing process of anthraquinone dye were determined. Anthraquinone served as an eco-friendly alternative to their synthetic equivalents to make a greener life. The anthraquinone extracted in this study give a high value product to the textile industry like 'Tenun Diraja Pahang' for commercialization.

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