DYEING ADSORPTION KINETICS OF BETACYANIN PIGMENTS EXTRACTED FROM *HYLOCEREUS POLYRHIZUS* PEEL ONTO SPUN SILK AND ACRYLIC YARN

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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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LIST OF SYMBOLS

mg	milligram
g	Gram
kg	Kilogram
mL	miliLitre
L	Litre
nm	nanometer
mm	milimeter
cm	centimeter
°C	Degree celcius
K	kelvin
mg/g	Milligram per gram
mg/L	Milligram per litre
g/L	gram per litre
mL/min	Mililitre per minute
mg/min	Miligram per minute
g/L	Gram per litre
kJ/mol	Kilo joule per mole
w/v	Weight per volume
v/v	Volume per volume
h_i	The initial dye adsorption rate
K_1	Rate constant of pseudo first-order adsorption
K_2	Rate constant of pseudo second-order adsorption
q_e	The amount of dye adsorbed per gram yarn at equilibrium time
q_t	The amount of dye adsorbed per gram yarn at time t
t	time
min	minute
h	hour
A	exponential factor
E_a	Activation energy
R	Gas constant
T	Temperature

$\Delta H^{\#}$	Enthalpy of activation
$\Delta S^{\#}$	Entropy of activation
$\Delta H°$	Enthalpy change
$\Delta S°$	Entropy change
$\Delta G°$	Gibbs free energy
k_b	Boltzmann's constant
h	Plank's constant
Q	Adsorption capacity of the Langmuir isotherm
b	Langmuir constant
Q_f	Adsorption capacity of the Freundlich constant
C	concentration
C_e	Concentration of the dye left in the dyebath at equilibrium
C_o	Initial dye concentration
C_t	Dye concentration after dyeing time t
V	Volume
W	Weight of yarn used
A_T	Temkin isotherm equilibrium binding constant
b_T	Temkin isotherm constant
B	Constant related to heat of sorption
R^2	Coefficient of determination

LIST OF ABBREVIATIONS

MLR	Material to Liquid Ratio
SLR	Solid to Liquid Ratio
SEM	Scanning Electron Microscope
RSM	Response surface methodology
FFD	Full factorial design
FT-IR	Fourier Transform Infrared
SLR	Solid to Liquid Ratio
MLR	Material to Liquid Ratio
SSE	The sum of the square errors
RMSE	Residual root means square error
ANOVA	Analysis of Variance
OFAT	One Factor at One Time

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ABSTRAK

Sisa buangan daripada industri tekstil yang majoritinya mengandungi pewarna sintetik menyebabkan pencemaran kepada alam. Manakala pewarna semulajadi pula lebih mesra alam dan mudah terurai. Oleh itu, kajian dalam pengekstrakan pewarna semulajadi daripada tumbuhan telah dilakukan secara meluas kebelakangan ini dan menjadi focus utama dalam kertas kajian ini. Dalam penyelidikan ini, pewarna asli telah diekstrak daripada kulit buah naga di mana ia mengandungi pigmen betasianin. Proses pencelupan pigmen betasianin telah diuji ke atas benang sutera dan akrilik. Mekanisma penjerapan, penjerapan kinetik, parameter termodinamik dan pencelupan yang optimum telah ditentukan dalam kertas kerja ini. Kajian penjerapan pewarna semulajadi betasianin telah dijalankan dengan menggunakan model isoterma Langmuir, Freundlich dan Temkin. Isoterma Freundlich adalah model yang terbaik dan sepadan dengan data eksperimen, ia ditentukan dengan menggunakan ralat analisis. Kajian diteruskan untuk menentukan kinetik penjerapan dengan menggunakan model kinetik pseudo tertib pertama dan pseudo tertib kedua. Kemudian, nilai optimum bagi proses pencelupan benang ditentukan dengan kaedah FFD dan dioptimumkan dengan kaedah tindak balas permukaan (RSM). Proses penjerapan untuk pewarna ini adalah padan dengan model kinetik pseudo tertib kedua dengan kadar pemalar bagi benang sutera dan akrilik masing-masing ialah 0.128 g/min.mg dan 0.099 g/min.mg pada kepekatan awal 2.42 g/L dan suhu 30 °C. Faktor utama yang mempengaruhi proses penjerapan ialah pH, masa penjerapan dan kepekatan awal dan nilai yang optimum telah diperolehi iaitu pH 2, masa pencelupan selama 95 minit dan kepekatan pewarna sebanyak 110 g/L di mana peratusan pencelupan yang optimum selaras dengan faktor-faktor tersebut telah didapati pada 52.45%. Ini dapat dirumuskan bahawa penjerapan betasianin ke atas benang boleh berlaku dengan dua lapisan dan penjerapan dikawal sepenuhnya oleh proses kimia. Tenaga pengaktifan untuk proses penjerapan benang sutera ialah 55.7 kJ/mol dan akrilik adalah 44.5 kJ/mol. Daripada nilai parameter termodinamik yang telah ditentukan, ini menunjukkan bahawa penjerapan betasianin adalah spontan dan eksotermik.. Kesan mordan terhadap penjerapan pewarna betasianin juga telah dilakukan dan ia menunjukkan bahawa mordan berpotensi untuk meningkatkan mutu dan sifat pewarna semulajadi tersebut di atas benang. Kesimpulan keseluruhan daripada kajian ini ialah pewarna betasianin yang diekstrak dari kulit buah naga boleh digunakan sebagai pewarna semulajadi dan boleh digunakan untuk mewarna benang sutera dan akrilik.

ABSTRACT

Synthetic dyes that used in textile industry can caused considerable environmental pollution. While, natural dyes are known to be more environmental friendly. Therefore, the study on extraction of natural dyes from plants is currently become revival. In this present work, the natural dye was extracted from dragon fruit peel, also known as *Hylocereus polyrhizus*, which contains betacyanin pigments. The dyeing adsorption of red-violet betacyanin on the spun silk and acrylic yarn was investigated, where the adsorption mechanisms, adsorption kinetics and thermodynamic parameters as well as the optimum dyeing conditions were determined. The sample analysis was analyzed using UV-Vis spectroscopy in liquid phase. The experimental data were analyzed using Langmuir, Freundlich and Temkin isotherm models and the best-fitted isotherm model was then determined using error analysis. Further investigation on the adsorption kinetic was done using pseudo-first-order and pseudo second-order model to determine the rate constant. For the optimization of dyeing conditions, a systematic experimental design including One-factor-at-a-time (OFAT) and first order model of 2^5 full factorial designs (FFD) was used in the initial screening process to determine the significant variable factors. The dyeing conditions was optimized using RSM. The best fitting isotherm for both yarns was Freundlich isotherm, which is demonstrated by the highest values of coefficient of determination and was confirmed by three types of error analysis. The adsorption process was followed the pseudo-second-order kinetic model with the rate constant of 0.128 g/min.mg for spun silk and 0.099 g/min.mg at 30°C. By using the rate constant, the activation energies for the spun silk and acrylic yarn adsorption process can be calculated and it was established at 55.7kJ/mol and 44.5kJ/mol respectively. The results of FFD indicated that the pH, dyeing time and dye concentrations are the significant factors for the dyeing conditions with the value of 3, 90 minutes and 100 g/L respectively. These factors were further optimized by RSM and the optimum values obtained for the dyeing conditions were pH 2, 95 minutes dyeing time and dye concentrations of 110 g/L. Under these conditions, the optimum dye uptake at 52.45% was attained. It is confirmed that the overall process of betacyanin pigment adsorption is to be controlled by the chemical adsorption. The thermodynamic parameters such as Gibbs free energy, enthalpy, and entropy were also determined. It can be concluded that the betacyanin pigment adsorption was spontaneous, exothermic and the dye distribution on the surface-active site is randomness. The effect of mordant on the dye uptake was also carried out and it showed that mordants did improve the color properties. This study has demonstrated that the betacyanin pigment can be used as a natural dye and can be applied to the spun silk and acrylic yarn as a coloring agent.

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