Screening of Factors Influencing Dyeing Uptake Exhaustion of Betacyanin Extracted onto Spun Silk using Fractional Factorial Design

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ABSTRACT

Dyeing uptake exhaustion of betacyaninn extract onto spun silk using batch process was carried out. In order to determine the most influence to the dye uptake percentage, the 2^5 full factorial design was used. The five factors was examined, namely temperature, dyeing time, dye bath concentration, salt concentration and the initial pH of the solution. Based on the ANOVA, it was verified that dyeing time, dye bath concentration and pH variables had an influence on the maximum dye uptake percentage at the level of 5% probability. The main effect was found to be determined as pH ($P<0.001$), dye bath ($P<0.0001$) and dyeing time ($P<0.008$). Temperature and salt concentration was determined as a secondary effect.

Keywords: adsorption, betacyanin, dragon fruit, FFD, Introduction, silk

1.0 Introduction

Dragon fruit or pitaya/ pitahaya, and its scientific name *Hylocereus polyrhizus*, is one of the tropical fruits in the Cactaceae or the cactus family[1]. Dragon fruit gets its name from its outer skin which has ‘scales’ or bracts on the surface that makes the fruit look like a dragon [2]. The flesh of this fruit is translucent dark-red in color and rich in nutrients and minerals such as vitamin B1, vitamin B2, vitamin B3 and vitamin C, protein, fat, carbohydrate, flavonoid and also much valued antioxidant properties. The peels mostly come from the dragon fruit juice processing industry which normally ends up as wastes. The peels, which are vibrant red violet in color, have the potential to replace synthetic dyes. Natural colorants from plant sources have received considerable attention from both food manufacturers and consumers as possible alternatives to synthetic colorants [3]. In this regard, the natural colorant from the dragon fruit peel may be used in the cosmetic and pharmaceutical industries. Similarly, it can also be arguably used in the textile industry as opposed to the use of the synthetic red color. As such, the objective of this study was to investigate the optimal dyeing conditions for spun silk using betacyanin as a natural dye. The dyeing conditions such as dye-bath concentration, pH of the solution, the salt concentration, the dyeing temperature and the dyeing time were investigated using full factorial design. The full factorial design is widely used to investigate the effects of experimental factors and the interaction between the factors [4]. The advantages of factorial experiment include reduced number of experiments, increased possibilities to evaluate the interaction among the variables and simultaneous investigation of the effects of all the variables of interest [5].

2.0 Materials and Methods

2.1 Materials

Two main materials were used in this study, namely, spun silk and dragon fruits. Spun silk was purchased from the Pusat Tenun, Kuantan, Pahang while the dragon fruits were bought from a farm near Universiti Malaysia Pahang where the experiments took place.

2.2 Betacyanin Extract

The betacyanin was extracted from the dragon fruit peel using acidified water extraction with ratio of 1:99 [6]. The peel was first cut and blended. Then, 10g of macerated dragon fruit peel was later mixed with 50mL of acidified water with the temperature of 45°C for 30minutes. the solution was filtered and centrifudged [3]

2.3 Adsorbance measurement

The ultraviolet/visible (UV-Vis) absorption measurement was recorded for the determination of adsorption (%) on *Hitachi U-1800 Spectrophotometer* in the wavelength 538 nm. The adsorbance of dye solution was recorded before and after dyeing the silk and acrylic yarn at an average of three measurements. The amount adsorbed was calculated [7] by using Equation 1:

$$\%\ \text{Dye uptake Exhaustion} = \frac{\text{Adsorbance before dyeing} - \text{Adsorbance after dyeing}}{\text{Adsorbance before dyeing}} \times 100 \qquad (1)$$

2.4 The full factorial design

By using a randomization technique, a total of 35 sets of experiment with four central points was employed for screening. The factorial design matrix and dye uptake percentage measured in each factorial experiment are shown in Table 1 with low and high levels specified as (-1) and (+1). There is five variables have been selected, namely temperature (45°C - 75°C), dyeing time (60 min – 90 min), initial pH (1-4), salt concentration (0.3 g/L – 0.9 g/L) and dye-bath concentration (60 g/L – 100 g/L).

3.0 Results and Discussion

Factors that influenced the dye uptake percentage were evaluated by using factorial plots in terms of main effects, normal probability plots and ANOVA. The ANOVA and P-value significant levels were used to check the significance of the effects on the dye uptake percentage.

3.1 ANOVA

Fisher's statistical test for ANOVA was employed for the determination of significant variables. Table 1 shows the results of ANOVA of dye uptake percentage during the dyeing process of extracted betacyanin onto the spun silk. Based on the value of *F*-ratio, the larger magnitude of *F*−value and corresponding the smaller of the 'Prob>F' value indicated the corresponding model and individual coefficient were more significant. From the results presented in Table 1, *F*-value and *P*-value of the model were found 76.82 and <0.0001, respectively. Hence, it can be said that the estimated model fitted the experimental data adequately. The coefficient of *R*-squared (R^2) was 0.9709 which indicated 97.09% variability in dye uptake percentage well explained by the model. A high value of R^2 would be most desirable as it indicated that there was little variation around the average prediction of the model. The value of adjusted determination coefficient 'Adj *R*-squared' (Adj R^2) was 0.9583 (95.83%) and predicted *R*-squared (Pred R^2) was 0.9364 (93.64%). Therefore, it can be assumed that the model provided good prediction for average outcomes because of the following: the ANOVA showed that the results were significant; the model's lack of fit was insignificant; the values of adjusted and predicted *R*-squared (R^2) were within 0.2 of each other; and, the residuals fitted well [11].

From the ANOVA results, the dye uptake percentage onto the spun silk yarn may be expressed using the following equation (2):

$$E\ \% = 43.23 - 1.98 * A + 8.06 * B - 20.69 * C - 1.22 * D - 3.27 * E - 1.84 * A * C - 3.06 * A * E - 2.15 * B * D - 1.15 * B * E - 1.18 * C * D \quad (2)$$

This function described how the experimental variables and their interactions influenced the dye adsorption [12]. The initial pH (*C*) of the solution had the greatest effect on the dye uptake percentage, followed by the dyeing time (*B*), dye-bath concentration, interaction of temperature and dye concentration (*A*-*E*), interaction of pH and salt concentration (*C*-*D*), salt concentration (*D*), temperature-salt concentration interaction (*A*-*D*), temperature (*A*) and lastly interaction of temperature and dyeing time (*A*-*B*). The positive values of these effects revealed that an increase in these parameters led to an increase in the dye uptake. Conversely, negative values of the effects decreased the response [4]. The equation enabled the prediction on the dyeing uptake as a function of temperature, dyeing time, pH, salt concentration and dye-bath concentration.

Table 1: ANOVA for 2^5 full factorial design; response: dye uptake exhaustion percentage (%)

Source	Sum of squares	DF	Mean squares	F-value	Prob > F	
Model	17981.56	10	1798.16	76.82	<0.0001	Significant
A	42.87	1	42.87	1.83	0.1891	
B	2426.61	1	2426.61	103.66	<0.0001	
C	14559.86	1	14559.86	621.98	<0.0001	
D	111.01	1	111.01	4.74	0.0399	
E	344.53	1	344.53	14.72	0.0008	
AB	39.87	1	39.87	1.70	0.2048	
AD	63.90	1	63.90	2.73	0.2048	
AE	192.96	1	192.96	8.24	0.0086	
BC	41.00	1	41.00	1.75	0.1987	
CD	158.95	1	158.95	6.79	0.0158	
Residual	572.72	23	24.90			
Lack of Fit	531.92	21	25.33	7.82	0.1194	Not significant
Pure	6.48	2	3.24			

Error				
Cor Total	19647.69	34		
Standard deviation	4.84		R^2	0.9709
Mean	40.44		Adjusted R^2	0.9583
			Predicted R^2	0.9364

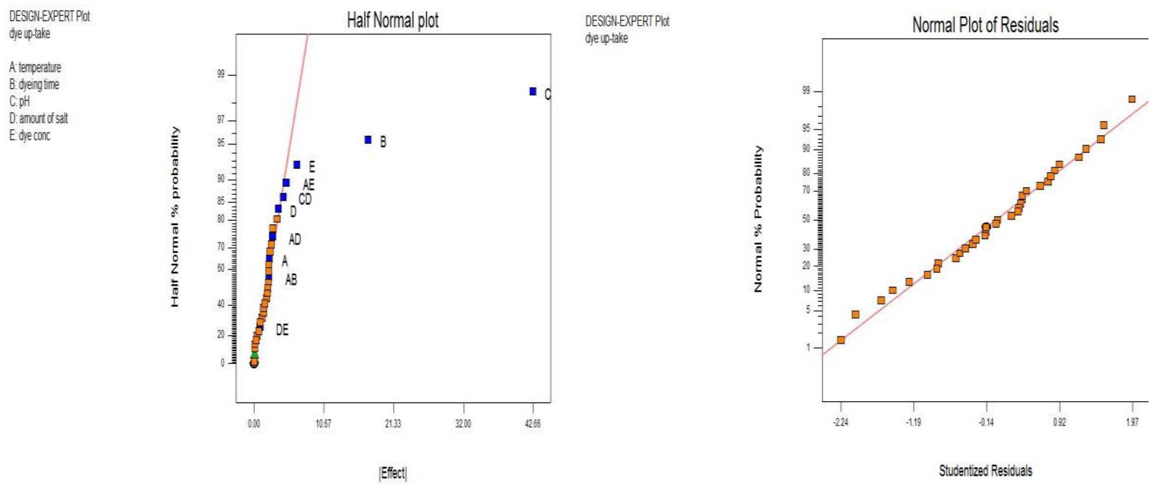
3.2 The main effects

In order to determine the most significant factor in the dyeing process of betacyanin extract onto the spun silk yarn, the main effects analysis was performed. For this study, the analysis function from Design Expert was used. As indicated from Design Expert, pH (*C*) had the highest percentage of contribution by 74.10%. This was consistent with the finding of a past study which had revealed that the pH of the dye solution played an important factor in the dyeing process [10]. The effect of dye bath pH may be attributed to the correlation between the dye structure, the fibers used and dye stability [13].

Another factor that showed high percentage of contribution was dyeing time (*B*) with 12.35%. The next highest contributor was dye concentration (*E*) by a 1.75% contribution. Other factors such as salt concentration (*D*) and dyeing temperature (*A*) were found to have made less contribution with 0.56% and 0.22% respectively. Temperature gave a minor contribution to the dyeing process because betacyanin pigment retention was decreased as the temperature increased [14]. In further experiments, the primary factors selected were pH, dyeing time and dye concentration while salt concentration and dyeing temperature were selected as secondary factors.

3.3 Normal probability plots

The significance of the effects and interactions of factors on a response was further analysed and compared. This is graphically illustrated in the half normal plot as shown in Figure 1(a). The half normal plot displays the absolute value of all effects, positive and negative. Instead of putting negative effects to the left and positive effects to right, all the significant effects are placed on the right side of the origin, in order to compare their relative magnitudes. This graphical tool can help to estimate which factors were important and which were not important. The results in the half normal plot represented the important and influential factors which were consistent with the ANOVA analysis result. As shown in Figure 1(a), variables *C*, *B* and *E* are significant as they are located far from the fitted line. The fitted line indicates that the effect were zero if the points fell on the line. The distribution of residual values, defined as the difference between the predicted and the observed value, was determined. Figure 1(b) illustrates the normal probability plot of residual values for dye uptake percentage. It shows that the set of observed values closely followed the theoretical distribution. The experimental points were satisfactorily aligned indicating a normal distribution. It showed that the selected model sufficiently described the observed data.



(a) (b)

Figure 1: (a) The half normal plot for 2^5 full factorial design (b) normal probability of studentized residuals on dye uptake exhaustion percentage

4.0 Conclusion

A statistical design of the experiments combined with techniques of regression was applied in optimizing the conditions of maximum dyeing uptake of extracted betacyanin onto the spun silk. The initial pH of the dye-bath solution affected the greatest influence on the amount of betacyanin adsorbed onto the spun silk. From the ANOVA, the variables found to have significantly contributed to the maximum dyeing uptake were dyeing time, pH and dye-bath concentration. It was also observed that a high coefficient of determining value (R^2) of 0.9709 ensured a satisfactory fit of the second-order polynomial regression model with the experimental data.

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