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〈Research Paper〉

Development of Cationic Dyeable Polyamide Substrates by Pretreatment with Synthetic Tanning Agent: Statistical Optimization and Analysis

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Abstract— Design of experiments (DoE) concept was successfully applied to determine the optimum processing conditions that yield maximum % exhaustion for berberine interaction with synthetic tanning agent pretreated polyamide substrates. The potential of synthetic tanning agent to provide anionic sites on the polyamide for berberine interaction which is cationic in nature was tested to increase the % exhaustion of berberine in this article. Experiments were designed according to Central Composite Rotatable Design (CCRD). The three factors for synthetic tanning agent pretreatment and two factors for berberine interaction each at five different levels, including central and axial points were considered. Experiments were conducted in a laboratory scale infra-red treatment instrument according to CCRD. For each response, second order polynomial models were developed using multiple linear regression analysis incorporating linear, interactions and squared effects of all variables and then optimized. The significance of the mathematical model developed was ascertained using Excel regression (solver) analysis module. Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted models. The response surfaces and contour maps showing the interaction of process variables were constructed. Applying Monte Carlo simulation, response surface and contour plots, optimum operating conditions were found and at this optimum point, % exhaustion of 81% and 74% respectively for synthetic tanning agent pretreatment and berberine interaction were observed and subsequently the results were experimentally investigated.

Keywords: *design of experiments, optimization, ANOVA, polyamide, pretreatment, berberine chloride, synthetic tanning agent*

1. Introduction

Polyamide (Nylon 6,6) is derived from 1,6-hexamethylene diamine and adipic acid. It is a semi crystalline thermoplastic polymer used for numerous engineering applications. Polyamide contains a mixture of chains that have only amines, only acid groups, or a combination of the two at their ends. The special properties of this synthetic fiber have remarkable applications in the textile industries for broad usage[1]. Berberine chloride, a natural antimicrobial agent has been successfully tested with cellulose and nylon fibers in our previous studies[2,3]. Our purpose in this study is to investigate the exhaustion properties of polyamide fiber in the process of interaction of a cationic dye i.e., berberine where the fibers were previously treated with synthetic tanning agent. A detailed study was carried out by Burkinshaw and group to develop metal free tannic acid-based aftertreatment for nylon 6,6[4]. A study was previously carried out by Jimenez for absorption and surface free energy of leacril fibers dyeing with cationic dye, pretreated by tannic acid[5]. The basic idea of this research is to provide large number of anionic sites on the polyamide substrates through pretreatment of the polyamide with synthetic tanning agent to produce cationic dyeable polyamide substrates.

Pretreatment of fibers with various chemical

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agents to modify its characteristics is not a novel concept in textile finishing. But, the use of statistical concept to design and perform the skillful experiments is relatively a new approach. In this context, Response surface methodology (RSM), an important tool in process and product improvement is applied in the textile finishing[6].

RSM is a collection of experimental design and optimization techniques that enables the experimenter to establish the relationship between the response and the independent variables. RSM is typically used for mapping a response surface over a particular region of interest, optimizing the response, or for selecting operating conditions to achieve target specifications. This method is highly functional for studying the surface properties of textiles in the processes of adsorption of surfactants and dyes onto these substrates in textile finishings.

The design procedure of response surface methodology is as follows[6]:

(i) Designing a series of experiments for adequate and reliable measurement of the response of interest.

(ii) Developing a mathematical model of the second order response surface with the best fittings.

(iii) Finding the optimal set of experimental parameters that produce a maximum or minimum value of response.

(iv) Representing the direct and interactive effects of process parameters through two and three dimensional plots.

Thus, in this research article, experiments were designed by incorporating all important process variables namely pH, temperature and concentration using Statistical Design Software i.e., Minitab 14 (PA, USA). Experimental design allows a large number of factors to be screened simultaneously to determine which of them has a significant effect on % exhaustion. A polynomial regression response model shows the relationship of each factor towards the response as well as the interactions among the factors. Those factors can be optimized to give the maximum response (% exhaustion) with a relatively lower number of experiments. In this context, a new approach using statistically designed experiments for the interaction of the berberine onto polyamide which is pretreated with synthetic tanning agent was discussed in detail. The corresponding interactions among the variables were studied and optimized using central composite rotatable design.

2. Experimental Methods

2.1 Reagents and materials

All chemicals used were of analytical grade and doubly distilled water was always used. Polyamide was purchased from Korea Apparel Testing and Research Institute (KATRI). Synthetic tanning agent (Matexil FAAN 25) was kindly provided by ICI-Woobang Co. and berberine chloride was purchased from Aldrich Chemical Co.

2.2 Apparatus

A Hewlett Packard UV-Vis spectrophotometer, Model HP8452 was used for measuring the absorbance and recording the normal and derivative spectra. A Corning model 220 pH meter was used for pH measurements.

2.3 Factorial experimental design

Preliminary experiments were carried out to screen the appropriate variables to fix the experimental domain. Temperature, pH and concentration were chosen as independent variables and the % exhaustion as dependent output response variable. Independent variables, experimental ranges and levels for synthetic tanning agent pretreatment and berberine interactions are given in Table 1 and Table 2 respectively. Centre values for factors were repeated for error estimation. The formulated design matrix, shown in Table 3 for synthetic tanning agent is a response surface central composite rotatable design consisting of 20 sets of uncoded conditions. It comprises a full replication of 2^3 (=8) factorial design plus 6 center points and 6 star points. All the variables at the intermediate level (0) constitute the center points and the combinations of each of the variables at either it's lowest (-1) level or highest (-1) leve 1

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Table 1. Experimental ranges and levels of variables for % exhaustion of pretreatment of polyamide with synthetic tanning agent

Independent variables	Range and level				
	-α	-1	0	1	+α
pH (X_1)	7.9	9	10.5	12	13
Temperature (°C, X_2)	66.60	70	75	80	83.4
Concentration (% omf, X_3)	0.97	2	3.5	5	6.02

Table 2. Experimental ranges and levels of variables for % exhaustion of berberine interaction with pretreated polyamide

Independent variables	Range and level				
	-α	-1	0	1	+α
pH (X_1)	8.3	9	10.5	12	12.6
Temperature (°C, X_2)	55.85	60	75	80	84.14

Table 3. Central Composite Rotatable Design (CCRD) for polyamide pretreatment with synthetic tanning agent

S.No.	pH	Temperature (°C)	Concentration (% omf)	% Exhaustion (Exp.)	% Exhaustion (Pred.)
1	9.0000	70.0000	2.00000	48.77	48.92
2	12.0000	70.0000	2.00000	52.54	52.77
3	9.0000	80.0000	2.00000	72.54	71.47
4	12.0000	80.0000	2.00000	74.56	75.17
5	9.0000	70.0000	5.00000	43.25	42.52
6	12.0000	70.0000	5.00000	44.15	44.97
7	9.0000	80.0000	5.00000	64.25	63.88
8	12.0000	80.0000	5.00000	66.15	66.18
9	7.9773	75.0000	3.50000	46.35	47.56
10	13.0227	75.0000	3.50000	53.75	52.73
11	10.5000	66.5910	3.50000	30.87	30.60
12	10.5000	83.4090	3.50000	66.98	67.40
13	10.5000	75.0000	0.97731	80.12	80.15
14	10.5000	75.0000	6.02269	67.12	67.21
15	10.5000	75.0000	3.50000	47.34	47.30
16	10.5000	75.0000	3.50000	47.34	47.30
17	10.5000	75.0000	3.50000	47.34	47.30
18	10.5000	75.0000	3.50000	47.34	47.30
19	10.5000	75.0000	3.50000	47.34	47.30
20	10.5000	75.0000	3.50000	47.34	47.30

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Table 4. Central Composite Rotatable Design (CCRD) for berberine interaction with pretreated polyamide

S.No.	pH	Temperature (°C)	% Exhaustion	
			(Exp.)	(Pred.)
1	9.0000	60.0000	69.25	67.77
2	12.0000	60.0000	71.80	71.89
3	9.0000	80.0000	73.00	72.13
4	12.0000	80.0000	74.00	72.75
5	8.3787	70.0000	68.00	68.71
6	12.6213	70.0000	72.00	72.06
7	10.5000	55.8579	70.00	70.04
8	10.5000	84.1421	73.00	73.73
9	10.5000	70.0000	71.00	71.32
10	10.5000	70.0000	71.00	71.32
11	10.5000	70.0000	71.00	71.32
12	10.5000	70.0000	71.00	71.32
13	10.5000	70.0000	71.00	71.32

with the other variables at the intermediate levels constitute the star points. Thus, 20 experimental runs allowed the estimation of the linear, quadratic and two-way interactive effects of the process variables on the % exhaustion. In the case of berberine treatment, the design matrix shown in Table 4 is a response surface central composite design consisting of 13 sets of coded conditions. It comprises a full replication of 2^2 (=4) factorial design plus 5 center points and 4 star points. All the variables at intermediate levels (0) constitute the center points and combinations of each of the variables at either its lowest (-1) level or highest (-1) level with the other variables at the intermediate levels constitute the star points. Thus, 13 experimental runs allowed estimation of the linear, quadratic and two-way interactive effects of the process variables on % exhaustion. The results of experimental design were studied and interpreted by MINITAB 14 (PA, USA) statistical software to estimate the response of the dependent variable (% exhaustion).

2.4 Polyamide substrates treatment

Polyamide substrates (warp 70f24, weft 140f48, 2g) were first pretreated with synthetic tanning agent separately, in a sealed stainless steel dye pots of 120 cm^3 capacities. These were analysed in a laboratory scale infra-red treatment instrument (ACE-6000T, Korea) and then treated with berberine in a separate experiments. Experiments were performed according to response surface central composite rotatable design given in Table 2 and Table 3 for synthetic tanning agent pretreatment and berberine interaction respectively. The pH was adjusted using 0.1N Na_2CO_3 and 0.1N CH_3COOH. At the end of the experiments, polyamide sample was removed, rinsed thoroughly in tap water and dried in open air. The exhaustion rate (%E) was then calculated using the formula,

$$\%E = \frac{[D_o - D_t]}{D_o} \times 100 \quad (1)$$

where and is the quantity of synthetic tanning agent and berberine for the respective experiments in the initial and final bath respectively.

2.5 Empirical modeling

Empirical model i.e., second order polynomial regression equations were developed using Excel Solver function to predict the % exhaustion, relating the process variables i.e., pH, temperature, concentration for polyamide pretreatment and pH, temperature for berberine interactions. RMSE (Root Mean Square Error) is the important tool to validate the model equation for its prediction capacity[7)]. The RMSE is the distance, on average, of a data point from the fitted line, measured along a vertical line. If the value of RMSE is zero, then the model is perfectly predicting the behaviour of the system i.e., ideal model. The predicting capacity of the model thus decreases with respect to the corresponding value of RMSE from zero. So, a series of equations by varying the combinations of the variables like linear, interactions effects and squared effects were run using solver function so as to get the least value of the RMSE. The goodness of fit is a measure of how well the model fits the data. Model is only developed with a sample, and the value of the model depends on the clarity and un-ambiguity

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of the relationships between the independent variables.

The behaviour of the system was explained by the following empirical model[7] :

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \quad (2)$$

Where, is the dependent variable, are the regression coefficients, are independent data. Root Mean Square Error (RMSE) was calculated using the following formula[7],

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{0}^{N} (\mathrm{Exp.} - \mathrm{Pred.})^2}{N}} \quad (3)$$

Where, Exp. is the experimental value, Pred. is the predicted value from model equations and N is the total number of experiments.

2.6 Data analysis

MINITAB 14 (PA, USA) was used for statistical analysis of the experimental data. The quality of fit of the polynomial model equation was expressed by the coefficient of determination R2 and its statistical significance analyzed by Fisher's F-test and Student's t-test (Analysis of Variance (ANOVA)). The model was simplified by dropping terms which were not statistically significant ($p > 0.05$) by analysis of variance (ANOVA). The lack of fit test was used to determine whether the constructed models were adequate to describe the observed data. If Fisher's F-test for the model is significant at the 5% level (i.e., <0.05) there is evidence that the model has some power to explain the variation in the response. The level of significance was given as values of P less than 0.0001. A differential calculation (Monte Carlo Optimization) was then employed for evaluating the optimum point.

3. Results and discussion

Designs of experiments (DoE) concepts were successfully applied to perform the skillful experiments and optimize the operational variables for maximizing the (%) exhaustion. The basic idea of the present study is to pretreat polyamide substrates with synthetic tanning agent. This was done to impart anionic sites and thereby increasing the % exhaustion of beberine colorant which is cationic in nature.

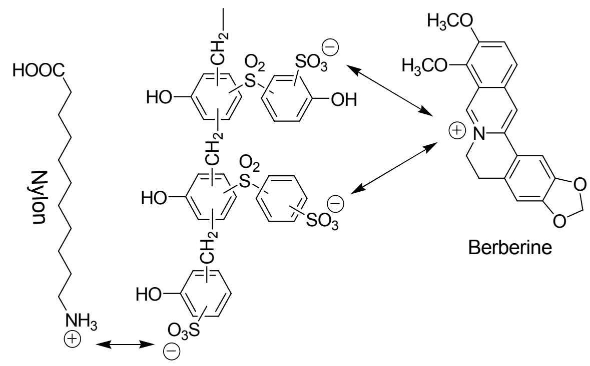

Fig. 1. Reaction mechanism of the polyamide pretreatment with synthetic tanning agent and berberine interaction with the pretreated polyamide.

The reaction mechanism of the synthetic tanning agent pretreatment with polyamide and then subsequent berberine interaction is given in Fig. 1. At first, experiments were designed to pretreat the polyamide with synthetic tanning agent. The range and levels of experimental conditions were chosen based on the screening experiments.

3.1 Main effect of variables on synthetic tanning agent pretreatment

The pH, temperature and concentration of the synthetic tanning agent were chosen as independent variables and % exhaustion as dependent variable. The pH range studied was between 7.9 and 13, temperature was 66.6 and 83.4°C and concentration was 0.97 and 6.02 % omf. The experiments were performed as per the CCRD design given in the design matrix. The main effects of the independent variables on % exhaustion were plotted using the experimental data and is given in Fig. 2. From the Fig. 2., it was observed that alkaline condition, higher temperature and lower concentration were found to be suitable process conditions for maximum % exhaustion. The maximum % exhaustion was found to be at pH 12 which shows that the

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interaction between hydroxyl groups of the synthetic tanning agent and amino groups of the polyamide is more at the prevailing alkaline conditions. Higher temperatures lead to higher % exhaustion i.e., above the glass transition temperature, the amorphous regions of polyamide provided more free volume to interact more hydroxyl groups of the synthetic tanning agent. As a result of this, higher exhaustion of synthetic tanning agent occurred. In addition, a swelling effect resulted from the higher temperature and alkaline conditions should facilitate interaction of synthetic tanning agent into polyamide substrate.

Interaction of synthetic tanning agent with polyamide depends on the concentration of synthetic tanning agent in the finishing bath. The concentration of synthetic tanning agent directly affects the uptake rates on polyamide due to interactions between hydroxyl groups of the synthetic tanning agent and polyamide. From the main effects plot (Fig. 2.), it was observed that lower concentration resulted in higher % exhaustion and increase in concentration resulted in decrease in % exhaustion. Maximum % exhaustion was achieved at the concentration of 0.977 % omf for synthetic tanning agent. This may be attributed to the fact that polyamides have limited amino end groups. Thus excess amount of synthetic tanning agent in the finishing bath could not produce a higher exhaustion than the maximum amount of the end groups that could react with hydroxyl groups. Instead, it plays a negative role in decreasing exhaustion of synthetic tanning agent into polyamide.

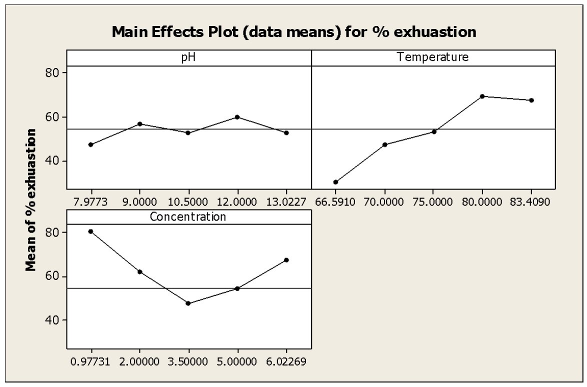

Fig. 2. Main effects plot of variables on % exhaustion of synthetic tanning agent with polyamide.

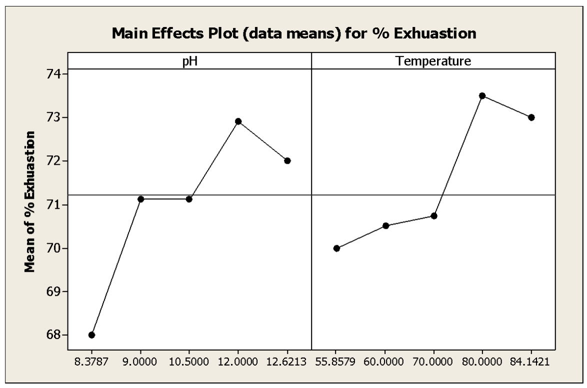

Fig. 3. Main effects plot of variables on % exhaustion of berberine interactions with pretreated polyamide.

3.2 Main effect of variables on berberine interaction with pretreated polyamide

The pH and concentration of berberine were chosen as important variables to design experiments in order to study the % exhaustion of berberine interaction. The main effects of the variables were plotted and shown in Fig. 3. It is observed that alkaline condition and higher temperature lead to maximum % exhaustion which is the same trend as the pretreatment results. From the plot, it is observed that maximum interaction occurred at pH 12 and 80°C. At alkaline condition, ionization of the sulphonates groups in the synthetic tanning agent leads to anionic sites so that berberine which is cationic in nature interacts more and then % exhaustion is high at the alkaline condition. The high diffusion movement of the berberine molecules at higher temperature leads to higher % exhaustion.

3.3 Model fitting and optimization

Using the experimental results, regression model equation (second order polynomial) relating % exhaustion and variables were developed for both pretreatment experiments and berberine interactions. The empirical model equations is given in equation (4) and (5),

Polynomial regression equation for % exhaustion of pretreatment of polyamide with synthetic tanning agent

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$$Y = -96.96 - 7.4668\,X_1 - 1.2464\,X_2 - 26.9543\,X_3 + 0.4761\,(X_1X_2) + 0.0249\,(X_1X_3) + 4.2436\,(X_2X_3) - 0.0099\,(X_1X_1) - 0.1672\,(X_2X_2) - 0.0472\,(X_3X_3) \quad (R^2 = 0.9985) \qquad (4)$$

Polynomial regression equation for % exhaustion of berberine interactions with synthetic tanning agent pretreated polyamide

$$Y = 49.7009 + 3.6381\,X_1 - 0.1396\,X_2 - 0.0833\,(X_1X_2) + 0.0031\,(X_1X_1) - 0.0167\,(X_2X_2) \quad (R^2 = 0.9985) \qquad (5)$$

The coefficient of determination, R^2, is defined as the ratio of explained variation to total variation and is a measure of the degree of fit. It is also the proportion of variability in response variables, which is accounted by the regression analysis. The empirical model fits the actual data well when R^2 approaches unity. The dependent variables in the model will be less relevant in explaining the behaviour of variation, when the value of the R^2 is small[7]. The empirical model developed for the experimental data have high coefficient of determination values (R^2=0.9985 for pretreatment, R^2=0.9646 for berberine interaction). This implies that 99.85% and 96.46% of sample variation is explained by independent variables and also means that the model did not explain only about 0.0015% and 0.0354% of sample variation for polyamide pretreatment and berberine interactions respectively. The predicted values (using model equations) were compared with experimental results and are shown in Fig. 4.

Although numerous studies on the effects of variables on % exhaustion of polyamide have been reported in the literature, no attempt has been made to optimize them using statistical optimization methods. But, in this work, the model equation (4) and (5) was optimized using multistage Monte-Carlo optimization technique[8]. The optimum values of the process variables were first obtained in coded units and then converted to uncoded i.e., real units by using the equation (6).

For statistical calculations, the variables were coded as according to the following relationship,

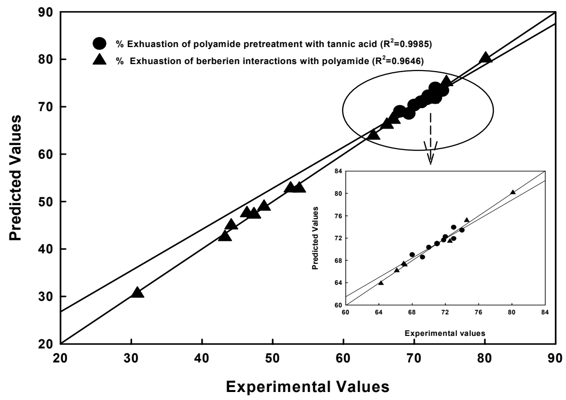

Fig. 4. Experimental and Predicted values of % exhaustion of synthetic tanning agent pretreatment and berberine interaction.

$$x_i = \frac{X_i - X_0}{\delta X} \qquad (6)$$

The optimum values of the process variables for maximum % exhaustion are shown in Table 5. These results closely agree with those obtained by response surface analysis i.e., graphically by surface and contour plots. This confirms that the design of experiments concept could be effectively used to optimize the process variables in complex processes using the statistical design of experiments. The surface plot models are dependent and useful for establishing desirable response values along with operating conditions. A surface plot displays a three-dimensional view that may provide a clear picture of the response surface. The coordinates of the central point within the highest surface levels in each of these figures will correspond to the optimum values of the respective constituents. The stationary point or central point is the point at which the slope of the contour is zero in all directions. The coordinates of the central point within the highest contour levels in each of these figures will correspond to the optimum values of the respective constituents. The maximum predicted yield is indicated by the surface confined in the smallest curve of the contour diagram.

The surface plots shown in Fig. 5. show the relative effects of any two variables when the remaining one variable is kept constant for all combinations.

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Table 5. Optimum values of the variables for maximum % exhaustion of pretreatment of polyamide with synthetic tanning agent and berberine interaction with retreated polyamide

Parameter	Optimum Conditions	
	Synthetic tanning agent pretreatment	Berberine interactions
pH	10.68	10.68
Temperature (oC)	76	76
Concentration (% omf)	0.9958	---

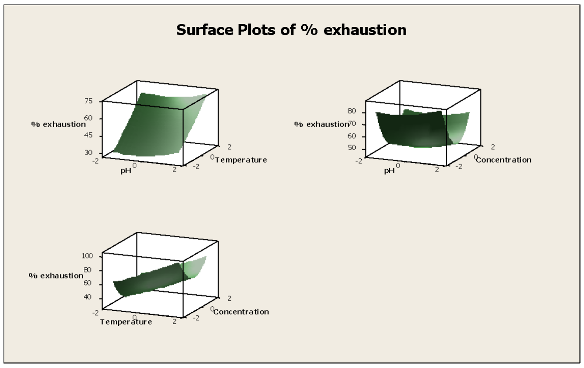

Fig. 5. Response surface plot of synthetic tanning agent pretreatment with polyamide.

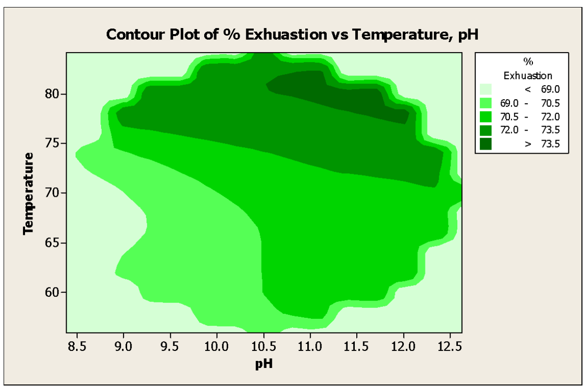

Fig. 6. Response contour plot of berberine interaction with pretreated polyamide.

The contour plots shown in Fig. 6. show the relative effects of pH and temperature. The response surface and contour plots of mutual interactions among the variables were found to be similar types of trends, found in the literature[9]. The optimum values drawn from these figures are in close agreement with those obtained by optimizing the regression model equation (4) and (5) using Monte-Carlo techniques. The optimum values were also obtained by running the optimization program with MINITAB-13 within the experimental range investigated.

This confirms that the design of experiments could be effectively used to optimize the process variables using statistical design of experiments concept. The optimum conditions obtained by the theoretical analysis were experimentally investigated and the highly feasible results were obtained. At this optimum point, % exhaustion of 81% and 74% respectively for synthetic tanning agent pretreatment and berberine interaction were observed.

3.4 Statistical analysis

From the statistical parameter estimates, it can be determined which variable contributes most to the prediction model, there by allowing the researcher to focus on the variables that are most important to the product acceptance. Apart from linear effect of the variables for the exhaustion, the design of experiments gives an insight into quadratic and interaction effects of the variables. These analyses were done by means of Fisher's F-test and Student's t-test. The student's t-test was used to determine the significance of the regression coefficients of variables. The P-values were used as a tool to check the significance of variables, which in turn may indicate patterns of the interactions among the variables. In general, larger the magnitude of t and smaller the value of P, the more significant is the corresponding coefficient[10]. The regression coefficient, t and P values for all linear, quadratic and interaction effects of the variables are given in Table 6 and Table 7 for synthetic tanning agent pretreatment and berberine interactions respectively.

For the pretreatment of the polyamide substrates with synthetic tanning agent, it was observed that coefficients for the linear effect of pH (P=0.001) and concentration (P=0.000) was considered to be significant compare to the temperature (P=0.058). In the interaction effects, pH:concentration (P=0.013) and temp:concentration (P=0.017) were considered to be significant. In the case of squared effects, all the three variables i.e., pH (P=0.000), temp (P=0.000) and concentration (P=0.000) was considered to be highly significant. For the berberine interactions, out of all variables, only constant (P=0.000)

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Table 6. Estimated regression coefficients and corresponding t and P values for polyamide pretreatment

Term	Coefficients	SE coefficients	t	P
Constant	-96.96	25.7658	3.763	0.004
X1	-7.4668	1.5224	-5.023	0.001
X2	-1.2464	0.5835	-2.136	0.058
X3	-26.9543	1.3983	-19.276	0.000
X1X2	0.4761	0.0165	-0.598	0.563
X1X3	0.0249	0.0551	-3.036	0.013
X2X3	4.2436	0.0165	-2.859	0.017
X1X1	0.0099	0.0410	11.607	0.000
X2X2	-0.1672	0.0037	6.743	0.000
X3X3	0.0472	0.0410	103.433	0.000

Table 7. Estimated regression coefficients and corresponding t and P values for berberine interaction with pretreated polyamide

Term	Coefficients	SE coefficients	t	P
Constant	53.71	20.57	2.611	0.035
X1	4.2964	2.3764	2.362	0.114
X2	-0.3932	0.3565	-1.103	0.306
X1X1	-0.0819	0.0935	-0.877	0.410
X2X2	0.0052	0.0021	2.690	0.031
X1X2	-0.0258	0.0190	-1.356	0.217

and squared effects of temperature (P=0.031) were considered to be significant. The significance of this quadratic and squared effects among the variables would have been lost if the experiments were performed by conventional or traditional approach.

The statistical significance of the ratio of mean square variation due to regression and mean square residual error was tested using Analysis of Variance (ANOVA). ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with specific sources of variation for the purpose of testing hypotheses on the parameters of the model[10]. According to the ANOVA, which is shown in Table 8 and Table 9 synthetic tanning agent pretreatment and berberine interactions respectively, the FStatistics values for all regressions were higher. The large value of FStatistics indicates that most of the variation in the response can be explained by the regression model equation. The associated P-value is used to estimate whether Statistics is large enough to indicate statistical significance. A P-value lower than 0.0001 (i.e., α = 0.0001, or 99.99% confidence) indicates

Table 8. ANOVA for % exhaustion of polyamide pretreatment with synthetic tanning agent

Source	Degree of freedom (DF)	Sum of squares (SS)	Mean square (MS)	Fstatistics	P
Regression	9	3230.60	358.956	2923.83	0.000
Linear	3	1913.11	15.844	129.06	0.000
Square	3	1315.31	438.437	3571.23	0.000
Interaction	3	2.18	0.726	5.92	0.014
Residual Error	10	1.23	0.123		
Lack of fit	5	1.23	1.23		
Pure error	5	0.00	0.00		
Total	19	3231.83			

Table 9. ANOVA for % exhaustion of berberine interaction with pretreated polyamide

Source	Degree of freedom (DF)	Sum of squares (SS)	Mean square (MS)	Fstatistics	P
Regression	5	26.9310	5.38620	5.58	0.050
Linear	2	23.5820	1.12283	3.44	0.091
Square	2	2.7483	1.37417	4.21	0.063
Interaction	1	0.6006	0.60063	1.84	0.217
Residual Error	7	2.2854	1.37417		
Lack of fit	3	2.2854	0.76181		
Pure error	4	0.00	0.00		
Total	13	31.0380			

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that the model is considered to be statistically significant[10]. The ANOVA table also shows a term for residual error, which measures the amount of variation in the response data left unexplained by the model. The form of the model chosen to explain the relationship between the factors and the response is correct.

The Statistics values of 2923.83 for polyamide pretreatment and 5.58 for berberine interactions are greater than tabulated F9,10 values, F5,7 values respectively indicate that the fitted model exhibits no lack of fit at the confidence level.

ANOVA shows P value of 0.000 for polyamide pretreatment and 0.050 for berberine interactions which indicates that the second-order polynomial model (Equation 4 and 5) was highly significant and adequate to represent the actual relationship between the response (% exhaustion) and the variables, with a very high coefficient of determination (R^2=0.9985 for pretreatment, R^2=0.9646 for berberine interaction).

4. Conclusions

An efficient approach for determining the optimum process variables for polyamide pretreatment and berberine interactions, with the pretreated polyamide by using second-order response surface model is presented and investigated experimentally.

The significant level of both the main effects and interaction are observed by analysis of variance (ANOVA) approach.

Based on the statistical analysis, the results have provided much valuable information on the relationship between important controlling factors such as pH, temperature and concentration and % exhaustion properties.

The factorial prediction model for both polyamide pretreatment and berberine interactions was established and optimized.

At this optimum point, % exhaustion of 81% and 74% respectively for synthetic tanning agent pretreatment and berberine interaction were observed and subsequently the results were experimentally investigated.

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