

Berberine Finishing for Developing Antimicrobial Nylon 66 Fibers: % Exhaustion, Colorimetric Analysis, Antimicrobial Study, and Empirical Modeling

Young-A. Son,¹ Byung-Soon Kim,¹ K. Ravikumar,¹ Tae-Kyeong Kim²

¹Department of Organic Materials and Textile System Engineering, Chungnam National University, Daejeon 305-764, South Korea

²Department of Textile System Engineering, Kyungpook National University, Daegu 702-701, South Korea

Received 17 January 2006; accepted 17 August 2006

DOI 10.1002/app.25364

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Berberine, a natural cationic colorant was successfully employed onto nylon 66 fiber in this research. The effects of three important variables, namely pH, temperature, and liquor ratio were examined on the % exhaustion, color strengths, and color yields of the sample. It has been employed in antimicrobial finishing as a natural agent on nylon 66 due to its characteristics of cationic quaternary ammonium salt. Antimicrobial activity of the sample was studied against *Staphylococcus aureus* (ATCC 6538) and *Klebsiella pneumoniae* (ATCC 4352) according to test method KS K 0693-2001 and the corresponding berberine finished sample showed very effective antimicrobial functions showing

about 99.9% of bacterial reduction against above-mentioned two bacteria. The maximum % exhaustion, color strengths, and color yields were obtained at 98 °C, alkaline condition (pH 11) and lower liquor ratio (20 : 1). An appropriate predictable empirical models were also developed using Excel solver function incorporating interaction effects of all variables to predict the % exhaustion, color strength(K/S), and the satisfactory results ($R^2 > 0.99$) were obtained. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 1175–1182, 2007

Key words: berberine; nylon 66; % exhaustion; colorimetric analysis; empirical modeling

INTRODUCTION

Nylon 66 is a semi crystalline polyamide commonly used in fiber applications such as carpeting, clothing, and tire cord. Nylon 66's outstanding characteristic in the textile industry is its versatility. Nylon 66 is used both alone and in blends with other fibers, where its chief contributions are strength and abrasion resistance. Nylon washes easily, dries quickly, needs little pressing. It holds its shape well since it neither shrinks nor stretches.¹ Over the years, a number of studies are being carried on treatment property and % exhaustion on nylon 66. The enormous industrial significance of dyes and treatment has led to a substantial body of research related to the application of dyes to fiber.² The treatment efficiency of nylon fibers is enhanced due to the end groups —COOH and —NH₂, which exhibit polar and hydrophilic characteristics. Many ionic dyes were evaluated for corresponding treatment efficiency on nylon and a mechanism was proposed.³

The presence of terminal amino end groups in nylon fibers imparts substantivity towards anionic dyes, specifically acid dyes, direct dyes, and reactive dyes. The 1 : 2 premetallised acid dyes are generally being used when reasonable levels of wet fastness are required.⁴ However, even these treatments can cause some staining of adjacent fibers during laundering and repeated washings can result in loss of color. The most notable dyes for nylon 66 have probably been the water soluble dyes possessing electrophilic reactive groups⁴ as well as some disperse dyes⁵ and sulfur dyes⁶ have also been evaluated on nylon 66. Durable flame retardant finishing of nylon/cotton blend fabrics was carried out using hydroxyl-functional organophosphorus oligomer.⁷ Electron beam modification was done on cotton, cotton/polyester film, and nylon 6 by finishing with polyvinyl alcohol (PVA) and acrylic acid.⁸ Durable and rechargeable antimicrobial cotton was prepared by finishing the cellulose with polycarboxylic acid (PCA) using sodium perborate as an activating agent.⁹ By incorporating the halamine structures into nylon polymers, attempts were previously made to develop antimicrobial finished nylon fibers.¹⁰

A number of chemicals may be employed to impart antimicrobial finishing to textile goods. Those chemicals include inorganic salts, organometallics, iodophors (substances that slowly release iodine),

Correspondence to: Y. A. Son (yason@cnu.ac.kr).

Contract grant sponsor: Ministry of Commerce Industry and Energy, Korean Government.

phenols and thiophenols, onium salts, antibiotics, heterocyclics with anionic groups, nitro compounds, ureas and related compounds, formaldehyde derivatives, and amines.¹¹

In this context of functional finishing of nylon fiber, Berberine, a natural cationic colorant, isoquinoline alkaloid and an excellent natural antimicrobial agent is found in roots and rhizomes of the Amur Cork tree extract, Goldenseal *Coptis chinensis*, Oregon Grape (*Berberis aquifolium*), Barberry (*Berberis vulgaris*) Tree Turmeric (*Berberis aristata*), and Yerba mansa (*Anemopsis californica*)¹² was successfully tested in this research as a finishing agent. Berberine can be used as a dye as well as antimicrobial agent, known as natural yellow 18, being one of about 35 yellow dyes from natural sources whose measured UV spectrum is given in Figure 1. The absorption property of this berberine compound shows the yellow shades in the visible range of 400–440 nm. In our previous work,¹³ berberine was successfully applied to cellulose fibers, by providing anionic sites on the cellulose fibers through the reaction with synthesized anionic bridging agent and their antimicrobial ability was tested by the author. To apply this cationic berberine compound towards any fiber substrate, the anionic attraction sites are necessarily needed to impart electrostatic attraction forces. In this context, berberine compound can be applied to the nylon substrate having carboxylic groups within the fiber molecules. However, there appears to have been little, if any, reference in the open literature to the use of berberine on nylon 66 fiber. Nylon polymers have two different ends, amino and carboxylic groups, if they were polymerized properly. The carboxylic groups are able to react with cationic dyes i.e., berberine and thus can be utilized in durable functions.

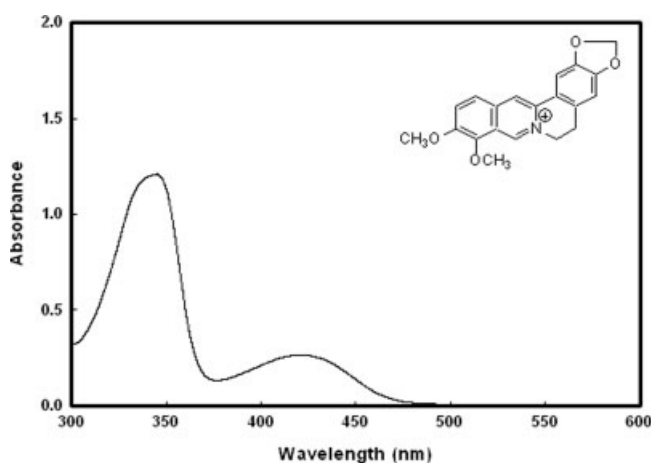


Figure 1 Molecular structure and UV absorption spectrum of berberine chloride.

This paper describes the coloration as well as antimicrobial ability of berberine on nylon 66 and the effects of three important variables i.e., pH, temperature, and liquor ratio on treatment. In addition, another new approach was made in this article to develop the mathematical correlations i.e., empirical models to predict the % exhaustion and K/S theoretically using the above-mentioned variables. Though the literatures were found, focusing on the effect of variables on % exhaustion and K/S values for different types of fibers, there have been only a very few, if any open reference to develop mathematical correlations to predict the behavior of the process theoretically. Thus, an effort was made successfully on this and the empirical models were developed to predict the % exhaustion and K/S values.

EXPERIMENTAL

Reagents and materials

All chemicals used were analytical grade and doubly distilled water was always used. Nylon 66 was purchased from Korea Apparel Testing and Research Institute (KATRI). Berberine chloride was purchased from Sigma.

Apparatus

A Hewlett-Packard UV-Vis spectrophotometer, Model HP8452 was used for measuring the absorbance, recording the normal and derivative spectra. Diano Color Formation System spectrophotometer interfaced to a PC was used for colorimetric analysis. A Corning model 220 pH meter was used for pH measurements.

Berberine finishing

Nylon fiber (warp 70f24, weft 140f48, 2 g) was treated with 2% owf of berberine chloride in a sealed, stainless steel dye pots of 120 cm³ capacity in a laboratory-scale infra red treatment machine (ACE-6000T). The treatment method used for the berberine (Natural Yellow 18, as berberine chloride, Sigma, 2% owf) is shown in Figure 2. The treatment was carried out at various temperatures (30–90°C), pH (3–12) and liquor ratio (20 : 1, 30 : 1, 50 : 1) by keeping any two variables constant. The pH was adjusted using 0.1N Na₂CO₃ and 0.1N CH₃COOH. At the end of treatment, the finished sample was removed, rinsed thoroughly in tap water. Then, it was dried in open air. Finally, the % exhaustion, color strengths, and color yields were measured.

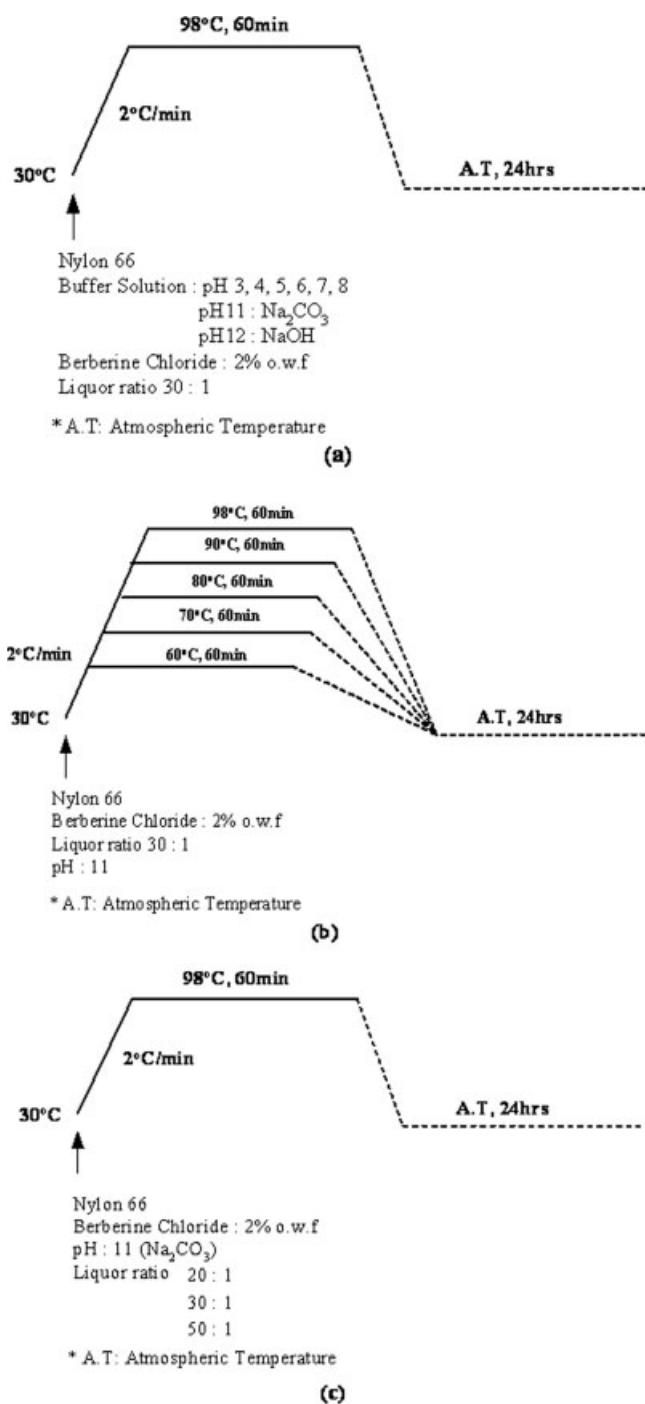


Figure 2 Treatment method (a) pH (b) Temperature (c) Liquor ratio.

% Exhaustion and color measurements

The exhaustion rate (%E) was calculated by the formula,

$$\% \text{ Exhaustion} = \left[\frac{D_o - D_t}{D_o} \right] \times 100 \quad (1)$$

where D_o and D_t is the quantity of berberine in the initial and final bath, respectively. Those values

were calibrated through absorbance measurement of original and exhausted bath by UV-Vis Spectrophotometer.

Colorimetric data of treatment of berberine were determined using a Diano Color Formation System spectrophotometer interfaced to a PC. Measurements were taken with the specular component of the light excluded and the UV component included, using illuminant D₆₅ and 10° standard observer. Each fiber was folded once to give two thicknesses and an average of four readings was taken each time. The L^* , a^* , b^* interceptions used in this system are based on the Commission International de E'clair (CIE) color triangle. The L^* value represents the dark-white axis; a^* , the green-red axis, b^* , the blue-yellow axis.

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, and liquor ratio. Root Mean Square Error (RMSE) is the important tool to validate the model equation for its prediction capacity.¹⁴ The RMSE is the distance, on average, of a data point from the fitted line, measured along a vertical line. If the value of the RMSE is zero, then the model is perfectly predicting the behavior of the system i.e., ideal model. The prediction capacity of the model thus decreases with respect to the corresponding value of the RMSE from zero. Thus, series of the equations varying the combinations of the variables like 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, thus the value of the model depends on the clarity and un-ambiguity of the relationships between the independent variables.

An empirical model follows the general form¹⁴:

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

where, Y is the dependent variable, β are the regression coefficients, x are independent data. RMSE was calculated using the following formula,¹⁴

$$\text{RMSE} = \sqrt{\frac{\sum_0^N (\text{Exp.} - \text{Pred.})^2}{N}} \quad (2)$$

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

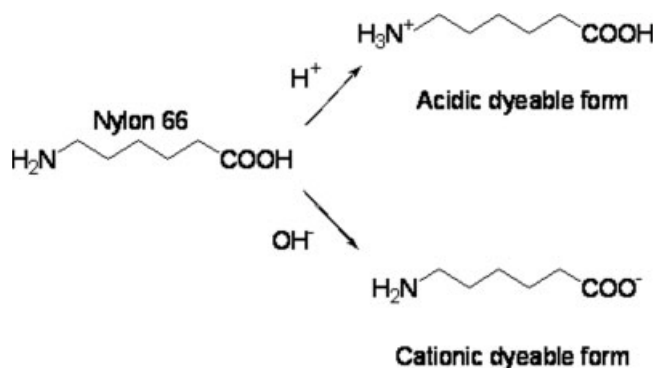


Figure 3 Dissociation scheme of nylon 66 at acidic and alkaline condition.

Antimicrobial study

The antimicrobial properties were quantitatively evaluated using *Staphylococcus aureus* ATCC 6538 and *Klebsiella pneumoniae* ATCC 4352, according to KS K 0693-2001 test method. The colonies of the both bacteria on the agar plate were counted. The reduction in numbers of the bacterium was calculated using the equation,

$$\text{Reduction rate (\%)} = \left[\frac{B - A}{B} \right] \times 100 \quad (3)$$

Where, B is the number of bacterial colonies at the initial stage (0 h) and A is the number of bacterial colonies after 18 h contact of finished fibers.

RESULTS AND DISCUSSIONS

Effect of pH on % exhaustion and color strength

Nylon fibers have two different end groups, amino and carboxylate, due to the polymerization reactions

employed in preparing the polymers. Both groups are chemically active, and the amino ends have been widely employed in the treatment of nylon fibers under acidic conditions. To promote ionic interaction between acid dyes with the nylon fibers, acidic conditions are very necessary to protonate amino groups of the nylon to their salt forms. Thus the resulting ionic interaction will lead to adsorption, exhaustion of acid dyes by the polymers, producing colored nylon fibers (Fig. 3). But, carboxylic acid ends of the nylon substrates have rarely been utilized in either treatment or chemical finishing of the nylon fibers. Ideally, a similar interaction between anionic carboxylic and cationic dyes such as berberine can be used in the finishing of nylon 66. Carboxylic acid groups will become more interactive with cationic groups under alkaline conditions, at which these groups can form carboxylate anions (Fig. 3). However, many cationic dyes are not stable at high pH, which might be one reason that such a reaction has not been employed in cationic treatment of nylon fibers. But, such ionic interaction can be a good access for durable antimicrobial agents, for example, berberine dyes are stable under pH variations. Therefore, antimicrobial finishing of polyamide fibers with berberine dyes is chemically feasible based on the above analysis.

To prove the feasibility of utilizing ionic interactions between carboxylic acids of nylon fibers and berberine, the treatment and colorimetric analysis of the berberine on the nylon 66 under different pH values were studied. The % exhaustion and colorimetric analysis were carried out at various pH conditions. The results of % exhaustion, color strength of the berberine are tabulated in Tables I and II. The graphical representation of the data is shown in Figures 4 and 5. As expected, both acidic and neutral

TABLE I
% Exhaustion of Berberine Chloride on Nylon 66 at Various Experimental Conditions

Variables	Temp. (°C)	pH	Liquor ratio	% Exhaustion of berberine		K/S	
				Exp.	Pred.	Exp.	Pred.
Effect of temp.	60	11	30 : 1	64.92	64.50	2.87	2.71
	70			65.4	65.68	2.86	2.95
	80			65.84	66.04	2.85	2.98
	90			66.3	65.78	2.83	2.79
	98			66.7	65.13	2.86	2.71
Effect of pH	98	3	30 : 1	0.0291	0.9811	0.20	0.05
		4		0.0461	0.1958	0.22	0.19
		5		2.69	2.7262	0.25	0.46
		6		6.864	7.7851	0.46	0.74
		7		12.578	14.980	0.98	1.05
		8		23.716	24.313	1.48	1.39
		11		69.23	65.131	2.44	2.50
12	76.56	83.011	2.45	2.91			
Effect of liquor ratio	98	11	20 : 1	72.14	72.15	2.97	2.99
			30 : 1	68.42	65.13	2.95	2.50
			50 : 1	65.59	65.59	2.89	2.90

TABLE II
Colorimetric Data of Berberine Chloride on Nylon 66 at Various Experimental Conditions

Variables	Temp. (°C)	pH	Liquor ratio	L^*	a^*	b^*	c^*	h	K/S	f_k
Effect of temp	60	11	30 : 1	79.80	-5.82	53.85	54.16	96.15	2.87	24.56
	70			80.15	-6.65	54.23	54.93	96.99	2.86	24.44
	80			79.91	-8.35	53.42	54.06	98.83	2.85	24.34
	90			80.41	-7.71	54.24	54.78	98.09	2.95	24.14
	98			81.37	-8.56	56.57	57.21	98.61	2.95	24.77
Effect of pH	98	3	30 : 1	87.65	-3.79	13.83	14.35	105.34	0.20	2.84
		4		87.23	-3.91	14.34	14.86	105.24	0.22	3.07
		5		86.43	-4.38	16.05	16.64	105.28	0.25	3.60
		6		84.65	-5.96	23.74	24.48	104.10	0.46	5.79
		7		80.65	-6.90	34.40	35.08	101.34	0.98	11.92
		8		79.27	-6.63	42.82	43.33	98.81	1.48	17.47
		11		74.86	-6.52	46.88	47.33	97.92	2.95	27.43
Effect of liquor ratio	98	11	20 : 1	77.32	-5.86	50.94	51.27	96.57	2.97	26.44
			30 : 1	79.18	-7.96	53.21	53.81	98.51	2.95	25.27
			50 : 1	79.48	-8.73	52.97	53.68	99.36	2.89	24.64

conditions yielded very poor % exhaustion of berberine on fiber. In contrast, the higher pH condition leads to a higher exhaustion since the berberine is more attractive to the negatively charged carboxylate groups under alkaline condition. Due to ionic interactions, berberine was quickly adsorbed and diffused into fibers. The higher exhaustion of berberine is expected in better antimicrobial functions on the berberine treated fibers.

The Kubelka–Munk theory¹⁵ models the spectrum of a colored body, based on a material dependent scattering and absorption function. The reflectivity was calculated by the model equation;

$$R_{\text{inf}} = \left(1 + K/S + (K^2/S^2 + 2K/S)^{1/2}\right)^{-1} \quad (4)$$

The theory has proven to be successful for a wide variety of materials and applications. Therefore, the

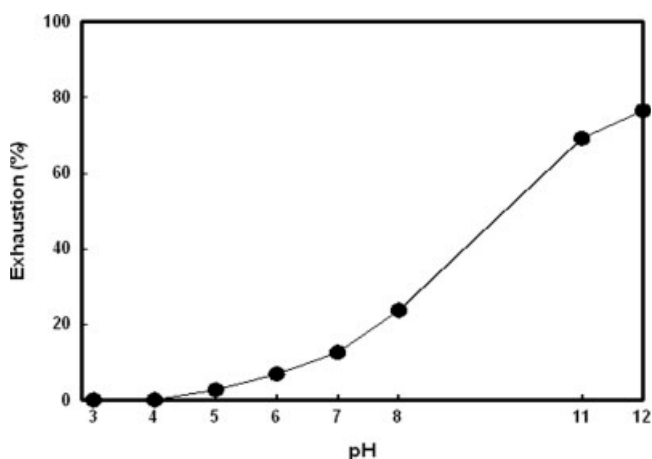


Figure 4 % Exhaustion of berberine on nylon 66 at various pH.

Kubelka–Munk theory is well suited for determining material properties from color measurements and Kubelka–Munk absorption coefficient K , and scattering coefficient S , were calculated by the solution of two conditions of the Kubelka–Munk theory. The color yield (f_k) and color strength (K/S) values of the nylon finished with berberine are given in the Table II at various pH. It is obvious from the results that both f_k and K/S increases with increasing pH and maximum values are achieved at the alkaline conditions as carboxylic groups of the nylon are more interactive with berberine at that condition. Figure 5 shows the graphical representation of the colorimetric reflectance of the nylon finished with berberine at various pH and it is just following the trend found in the literature. Absorption property of yellow color on the substrates represents corresponding absorption in ranges of 400–440 nm.

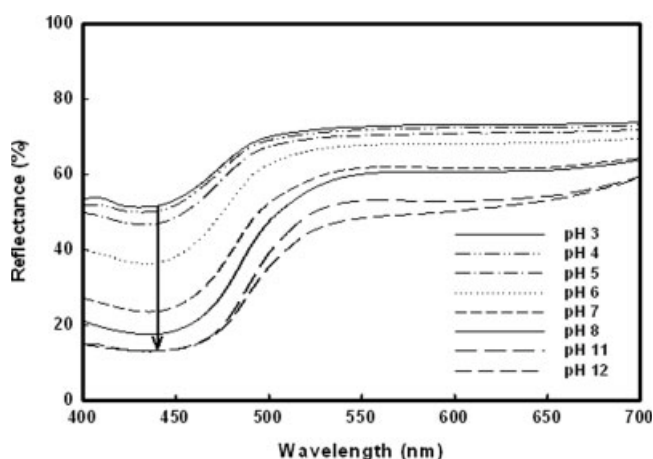


Figure 5 % Exhaustion of berberine on nylon 66 at various temperatures.

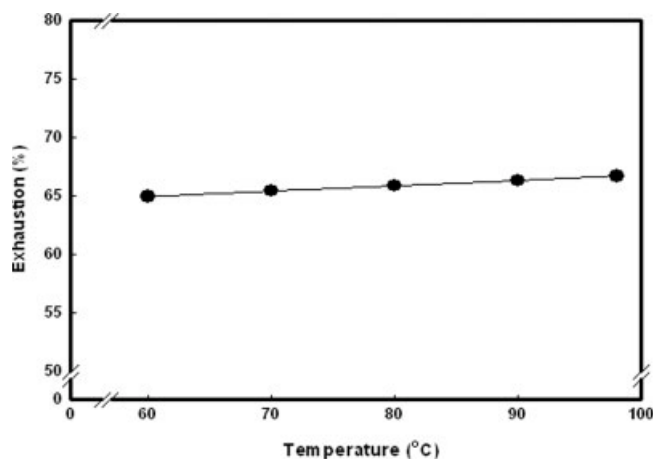


Figure 6 % Exhaustion of berberine on nylon 66 at various liquor ratios.

Effect of temperature on % exhaustion and color strength

The experiments were designed to evaluate the effect of temperature on % exhaustion and color strength after successfully proved that the berberine could be employed on nylon 66 under alkaline conditions (pH 11). The experiments were carried out at different temperatures (60–98°C) and the results were tabulated in Table I for % exhaustion and also graphically represented in Figure 6.

The experiments were started from 60°C i.e., glass transition temperature of nylon 66 as the author indicated in previous publication¹⁶ that significant exhaustion of the cationic salts did not occur until the finishing temperature was above the glass transition temperature (57°C). The data shows that the good % exhaustion was achieved by the ranges of the temperature from 60 to 98°C. Above the glass transition temperature, the amorphous regions of nylon fibers will provide more free volume to diffuse more berberine and as a result higher exhaustion of berberine occurs. In addition, a swelling effect resulted from the higher temperature and alkaline conditions should facilitate diffusion of berberine into nylon substrate. The similar kind of results was observed for ionic interaction of cationic antimicrobial salts with nylon fibers by the author.¹⁶ Color yield (f_k) and color strength (K/S) values were calculated using colorimetric analysis and it is observed from Table II that both of the values increase with increasing temperature as higher temperature leads to diffusion of more berberine dyes into nylon fiber. The colorimetric reflectance of the berberine treated nylon is shown in Figure 7 and it is observed that interpretation of the trend is difficult as the difference in reflectance is very tiny for the absorption range 400–440 nm. However, the increase in the value of the color yield (f_k) and color strength (K/S)

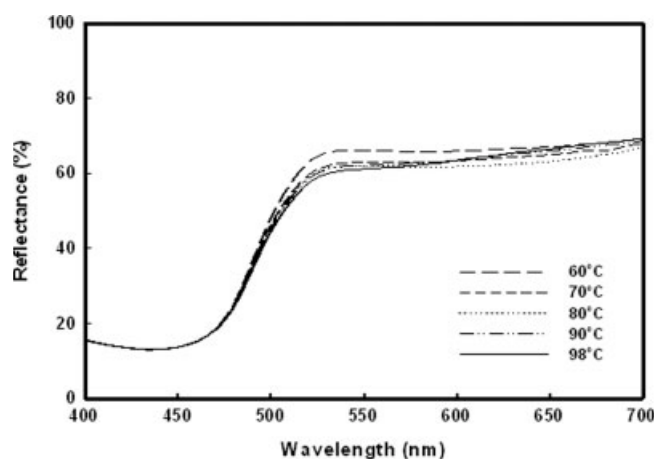


Figure 7 Colorimetric analysis of berberine on nylon 66 at various pH.

with increase in temperature was clearly found in the Table II.

Effect of liquor ratio on % exhaustion and color strength

Another important variable to be considered in the processes is liquor/fiber ratio. Selection of appropriate liquor ratio contributes to producing desired results. The experiments were carried out at the three different liquor ratios 20 : 1, 30 : 1, and 50 : 1 and the results are tabulated in Table I. Figure 8 shows the effects of liquid ratio on the % exhaustion. It was observed that lower liquor ratio resulted in higher % exhaustion and increase in liquor ratio resulted in decrease in % exhaustion. Maximum % exhaustion was achieved at the liquor/fiber ratio of 20 : 1.

This may be attributed to the fact that lowering the liquor ratio results in concentrated solution with maximum probable molecular collisions between nylon 66 and berberine and thus making more number

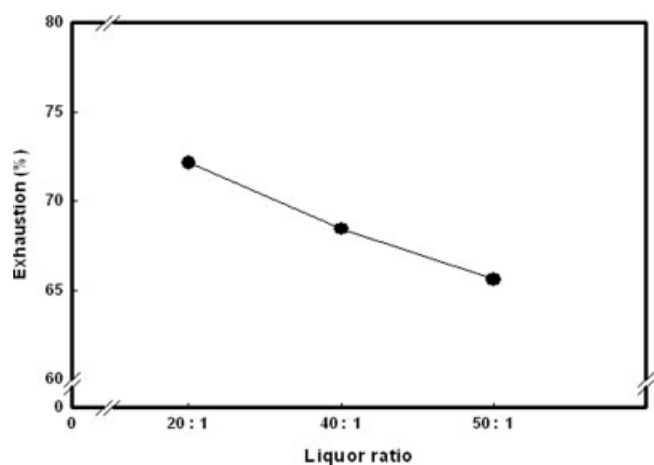


Figure 8 Colorimetric analysis of berberine on nylon 66 at various temperatures.

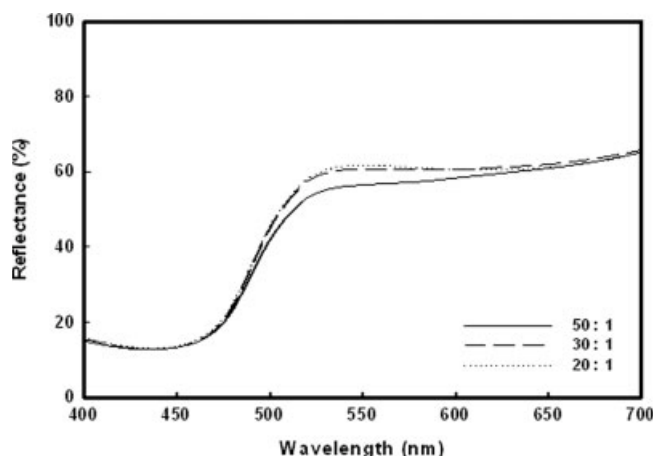


Figure 9 Colorimetric analysis of berberine on nylon 66 at various liquor ratios.

of berberine molecules to interact with fiber. Thus, the % exhaustion was increased. On the other hand, higher liquor ratio leads to more diluted state and less probable collision of the molecules and thus making less number of berberine molecules available for the reaction. Thus, the % exhaustion was decreased. The similar kinds of results were observed on the crosslinking of cotton fiber.¹⁷

Colorimetric analysis carried out on nylon 66 at three different liquor ratios is tabulated (Table II) and also graphically shown in Figure 9. The results obtained are so expected one and the interpretation from Figure 9 is difficult as very tiny difference is observed for the three different liquor ratio. However, Table II shows that the maximum color strength (K/S) and color yield (f_i) values were achieved at the minimum liquor ratio due to maximum possibility of molecular collision between berberine and nylon 66.

Empirical modeling

Empirical model is the functional form of the relationship between the dependent and independent variables and is found by direct examination of data related to the process. The model equations are highly useful to predict the behavior of the system.¹⁴ Using experimental results, the regression model equations (second order polynomial) relating the % exhaustion and the process variables were developed using Excel Solver function and are given in eqs. (5) and (6).

Polynomial regression equation for % Exhaustion
(RMSE = 0.238)

$$Y_1 = 0.987836 + (0.910991 X_1) + (0.575189 X_2) + (0.764022 X_3) - 0.061377 X_1 X_2 - (0.021368 X_2 X_3) + (0.421703 X_1 X_3) + (1.06,839 X_1^2) - (0.003097 X_2^2) + (0.024202 X_3^2) - (0.003762 X_1 X_2 X_3) \quad (5)$$

Polynomial regression equation for K/S
(RMSE = 0.2267)

$$Y_2 = 1.970713 + (0.817712 X_1) - (0.246113 X_2) + (0.534285 X_3) + (0.025409 X_1 X_2) + (0.004580 X_2 X_3) - (0.101363 X_1 X_3) + (0.010185 X_1^2) - 0.0010520 X_2^2 + (0.002305 X_3^2) - (0.00003039 X_1 X_2 X_3) \quad (6)$$

where, Y_1 is exhaustion rate (%), Y_2 is K/S , X_1 is pH, X_2 is temperature ($^{\circ}C$), and X_3 is liquor ratio.

The second order polynomial models thus developed are highly significant and adequate to represent the actual relationship between the response (% exhaustion and K/S) and the variables. The experimental and predicted values of the process are compared in Table I for both % exhaustion and K/S and shown in Figure 10 with high value of coefficient of determination ($R^2 = 0.9945$ for % exhaustion, $R^2 = 0.961$ for K/S). This implied that 99.45%, and 96.1% of the sample variation for % exhaustion, K/S respectively, were explained by the independent variables and this also means that the model did not explain only about 0.0055% and 0.039% of sample variation for % exhaustion and K/S respectively. Moreover, the RMSE for two eqs. (4) and (5) (RMSE = 0.238 for % exhaustion and RMSE = 0.2267 for K/S) were very less and showed the exact fit of the experimental data to the model.

Antimicrobial assesement

Organic compounds containing a structure of the quaternary ammonium salt strongly show antimicrobial functions. As shown in Figure 1, it contains a positively charged nitrogen atom in its chemical structure. Therefore, it is acceptable that berberine treatments could be utilized in functional finishing

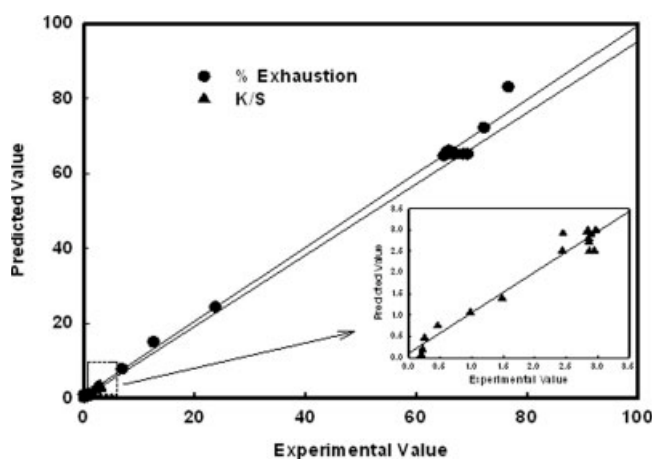


Figure 10 Experimental and predicted values of % exhaustion and K/S .

TABLE III
Bacterial Reduction (%) of Nylon 66 Using
Berberine Chloride

	Bacterial reduction of berberine treated nylon 66	
	<i>Staphylococcus aureus</i> (ATCC 6538)	<i>Klebsiella pneumoniae</i> (ATCC 4352)
0 h	2.5×10^4	2.0×10^4
18 h	<10	<10
Bacterial reduction (%)	99.9	99.9

process for antimicrobial purposes. This work herein discusses a useful approach of employing berberine colorant, a natural cationic quaternary ammonium salt, to achieve desired durable antimicrobial functions on nylon 66. To determine antimicrobial properties, the bacteria, namely *Staphylococcus aureus* and *Klebsiella pneumoniae* were used. The quantitative KS K 0693-2001 Test Method was employed to assess the antimicrobial activity by which the reduction in numbers of the bacteria was evaluated. The results are presented in Table III. It was observed that nylon treated with berberine displayed very effective antimicrobial activity showing 99.9% (~ 100%) of reduction in the number of bacteria.

CONCLUSIONS

Nylon 66 was successfully treated with berberine, a natural cationic yellow dyes as well as antimicrobial agent. The % exhaustion, colorimetric analysis and antimicrobial study were carried out and results were found to be very positive and encouraging. The berberine treatment provides very strong anti-

microbial functions showing 99.9% of reduction in the number of bacteria. Thus, based on the data obtained through experiments, it can be concluded that berberine finishing could be effective, very simple and practical to the nylon 66. The empirical models were successfully developed for predicting the % exhaustion and color strength (K/S) theoretically using the process variables pH, temperature and liquor ratio. The models were found to be exactly predicting the experimental values with high correlation coefficients.

References

- Burkinshaw, S. M. Chemical Principles of Synthetic Fiber Treatment; Chapman and Hall: London, 1995.
- Zollinger, H. Color Chemistry; VCH: New York, 1991.
- Gooding, J. J.; Compton, R. G.; Brennan, C. M.; Atherton, J. H. J Colloid Interface Sci 1996, 180, 601.
- Burkinshaw, S. M.; Gandhi, K. Dyes Pigments 1996, 32, 101.
- Carpiganano, R.; Savarino, P.; Barni, E.; Viscardi, G. Dyes Pigments 1989, 10, 23.
- Burkinshaw, S. M.; Chevli S. N.; Marfell, D. J. Dyes Pigments 2000, 45, 65.
- Yang, H.; Yang, C. Q. Polym Degrad Stab 2005, 88, 363.
- Ibrahim, M. S.; Salmawi, K. E.; Ibrahim, S. M. Appl Surf Sci 2005, 241, 309.
- Huang, L. K.; Sun, G. Ind Eng Chem Res 2003, 42, 5417.
- Lin, J.; Winkelman, C. E.; Worley, S. D.; Broughton, R. M.; Williams, J. F. J Appl Polym Sci 2001, 81, 943.
- Vigo, T. L. Handbook of Fabric Science and Technology Chemical Processing of Fabrics and Fabrics, Functional Finishes; Marcel Dekker: New York, 1983.
- Hill, D. J. Rev Prog Coloration 1997, 27, 18.
- Kim, T. K.; Yoon, S. H.; Son, Y. A. Dyes Pigments 2004, 60, 121.
- Ezekiel, M. Methods of Correlation Analysis; Wiley: New York, 1951.
- Goldman, J.; Goodall, R. R. J Chromatogr 1968, 32, 24.
- Son, Y. A.; Sun, G. J Appl Polym Sci 2003, 90, 2194.
- Hashem, M.; Refaie, R.; Hebeish, A. J Clean Prod 2005, 13, 947.