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APPENDIX

APPENDIX A

JIS L 2310 - 1979 R

a. Tensile Strength

As recommended by the standard test method for the measurement of tensile strength, constant rate of traverse type yarn tensile tester was used by pulling the specimen under the initial load with the clamping distance of 50 cm at a rate of 30 ± 2 cm/min.

The specified initial load is equivalent to 1/30 gf(1/294 N) of the aggregate fineness of raw silk.

This standard condition shall be of category 2 specified in JIS Z 8703; i.e., temperature at 20 ± 2 °C and relative humidity of 65 ± 2%.

b. Coefficient of Variation of Tensile Strength

Owing to the variation in the test method, the numerical values obtained from the measurement of tensile strength should be subjected to statistical evaluation.

The coefficient of variation of such a measurement can be computed by the following formula.

$$\text{Cofficient of variation (\%)} = \frac{\sqrt{(x-\bar{x})^2/(n-1)}}{\bar{x}} \times 100$$

where

x = individual measurements

$\bar{x}$ = total mean

n = number of measurements

APPENDIX B

THE PREPARATION OF MANGANESE (III) ACETYLACETONATE

The preparation of manganese(III) acetylacetonate can be represented by the following two chemical equations.

$$MnCl_2 + 2\ HC_5H_7O_2 + 2\ NaC_2H_5O_2 \longrightarrow$$
$$Mn(C_5H_7O_2) + 2\ NaCl + 2\ HC_2H_3O_2$$

$$4Mn(C_5H_7O_2)_2 + KMnO_4 + 7\ HC_5H_7O_2 + HC_2\ H_3O_2 \longrightarrow$$
$$5Mn(C_5H_7C_2)_3 + KC_2H_3O_2 + 4H_2O$$

PROCEDURE

To a solution of 5.2 gm (0.026 mole) of manganese(II) chloride 4-hydrate and 13.6 gm (0.1 mole) of sodium acetate 3-hydrate in 200 ml of water, is added 20.0 gm (0.2 mole) of acetylacetone. To the resulting mixture, a solution of 1.04 gm (0.0066 mole) of potassium permanganate in 50 ml of water is slowly added at room temperature with stirring. After stirring for a few minutes, a solution of 13.6 gm (0.1 mole) of sodium acetate 3-hydrate in 50 ml of water is added in small amounts with stirring. The mixture is then heated on the hot plate for about 1.0 min.

and cooled to room temperature. The dark solid is filtered in a Buchner funnel. The product is washed with water and dried in vacuo over anhydrous calcium sulfate. The dried chelate is dissolved in 20 ml of warm benzene, the solution is then filtered, and the chelate is reprecipitated by cooling the solution into which 75 ml of petroleum ether is added. The recrystallized material is then dried at room temperature in vacuo over anhydrous calcium sulfate.

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APPENDIX C

STATISTICAL METHOD

Statistics is a tool applicable in research experiments to analyse the results from the summarizing of the data to the evaluation of the uncertainty of any statistical information drawn from them.

Correlation is a measure of the degree to which variables vary together. Correlation coefficient (r) is defined as

$$r = \frac{\Sigma(X - \bar{X}) (Y - \bar{Y})}{\sqrt{\Sigma (X - \bar{X})^2 - (Y - \bar{Y})^2}}$$

" r " lies between -1 and +1, that is, $-1 < r < +1$. The value +1 indicates a perfect linear correlation between the two variables in the same direction, and the values between 1 to 0 indicate the lesser correlations. In contrast to the above position value, the value -1 indicates perfect linear correlation between the two variables in the opposite direction.

A correlation coefficient tells us something about a joint relationship between variables. However, it doesn't indicate the influence of the independent variables affecting other dependent variables. There is another method called a regression analysis. A regression coefficient tells us that if we alter the value of the independent variable then we can expect the dependent variable to alter a certain amount on the average.

In this research, we also use the *multiple regression* method to estimate magnitude of an effect and calculate an interval within which the true value almost certainly lies. Such an interval is called a confidence interval.

The procedure of multiple regression is to fit the linear model to a given body of data ($Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \_\_\_\_\_ + \beta_p X_p + u$). Y and X represent dependent variables and independent variables respectively, β represents regression coefficient.

The hypothesis in the linear model can be tested by defining that if regression coefficients are zero, this implies that there is no linear relationship between the dependent variable and the set of independent variables.

The significant value (F-value) can be used for testing the null hypothesis, H_o ($\beta_1 = \beta_2 = \_\_\_\_ = \beta_p = 0$), in terms of the sample multiple correlation coefficient, R_p, that is:

$$F = \frac{R^2_p / p}{(1 - R^2_p) / (n - p - 1)}$$

degree of freedom, d.f. = p, n - p - 1

where

n = number of observations

p = number of independent variables

We usually expect the experimental data to lie between confidence interval 99% and confidence interval 95%.

If F - value calculated is larger than $F_{.01}$ - value (99 % confidence interval) in the analysis of variance (ANOVA) table, the null hypothesis is rejected. This implies that the independent variable is highly significant to the dependent variable.

If F - value is greater than $F_{.05}$ - value (95 % confidence interval) in the ANOVA - table, the null hypothesis is not rejected. This implies that the

independent variable is insignificant to the dependent variable.

If F - value is between $F_{.01}$ and $F_{.05}$ - value, this implies that the independent variable is significant to the dependent variable.

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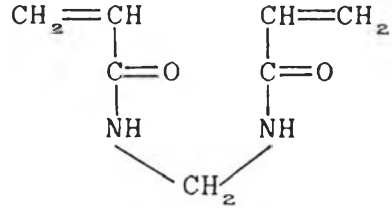
APPENDIX D

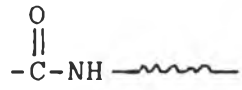
IR - SPECTRA

1. N,N'-METHYLENEBISACRYLAMIDE (N,N' -MBA)



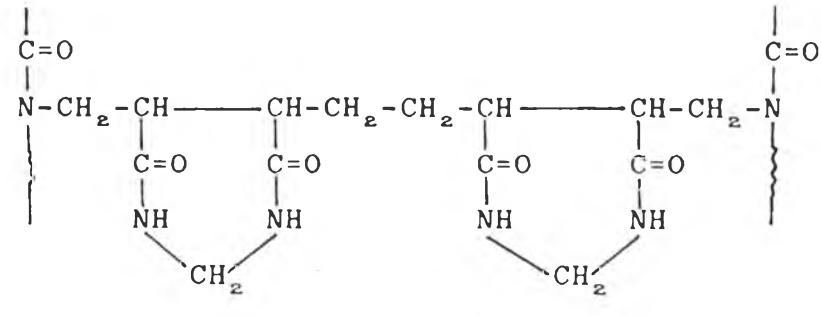
Group		Frequency range, cm^{-1}
1. C=O	νC=O	1950 - 1600
2. -C=C-H	νC=C	1670 - 1600
	δC-H	1420 - 1400
	νC-H	3100 - 3000
	δC-H	1000 - 670
	δ=C-H (out-of-plane)	1000 - 900
3. $-CH_2-$	$\nu_{as}CH_2$	2925
	$\nu_{s}CH_2$	2850
	δ CH_2 scissor	1470
	$\delta(CH_2)_n$ rock	725 - 720
4. -C-H	νCH	2890
	δCH	1340
5. C-N	νC-N	1235 - 1030
6. N-H	δN-H	1580 - 1490
	νN-H	3500 - 3300

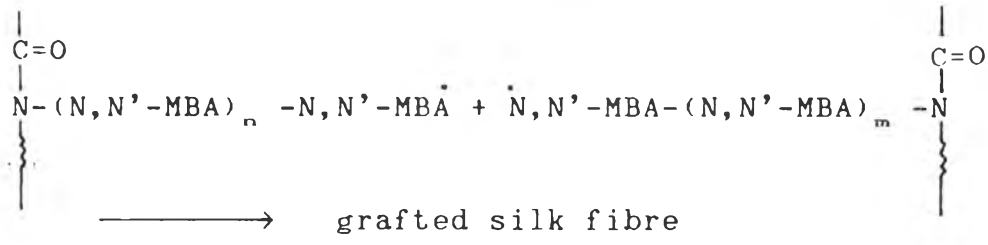
2. SILK FIBRES



Group		Frequency range, cm^{-1}
1. C-C		1440 - 1325
2. C=O	νC=O	1900 - 1600
3. C-N	νC-N	1235 - 1030 (aliphatic amines)
4. N-H	δN-H	1580 - 1490
	νN-H	3500 - 3300
5. 		1667, 1552

3. N,N'-MBA-GRAFTED SILK FIBRES





Group		Frequency range, cm^{-1}
1. C-C		1440 - 1325
2. C=O	νC=O	1950 - 1600
3. C-N	νC-N	1235 - 1030
4. $-CH_2-$	$\nu_{as}CH_2$	2925
	$\nu_{s}CH_2$	2850
	δ CH_2scissor	1470
	$\delta(CH_2)_n$rock	725 - 720
5. -C-H	νCH	2890
	δ CH	1340
6. =C-H	δ =C-H (out-of-plane)	1000-900
7. N-H	δN-H	1580 - 1490
	νN-H	3500 - 3300
8. $-\overset{O}{\overset{\|}{C}}-NR$		1667 - 1522

APPENDIX E

CALCULATION OF QUANTITY OF DYESTUFFS AND PERCENTAGE EXHAUSTION

E.1 Calculation of the Quantity of Dyestuff on the Silk Fibres

According to Section 3.5.4.3b, the absorption spectra of acid dyestuffs for the dyeing process are shown in Figures E.1a and E.1b.

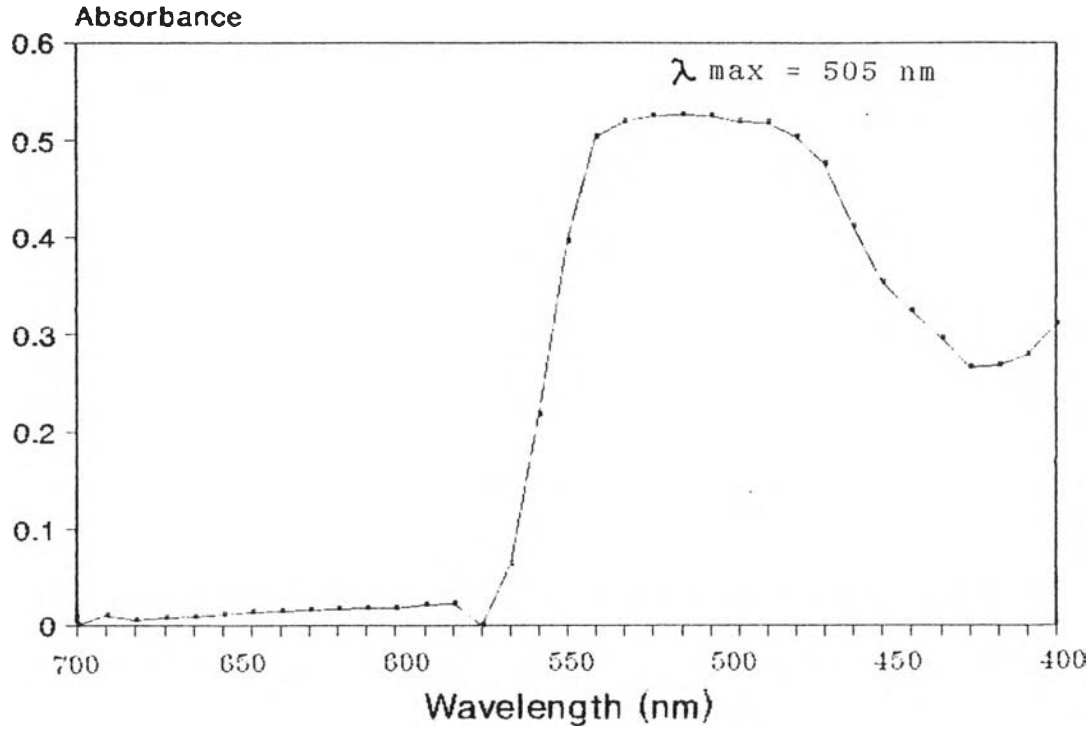


FIGURE E.1a Absorption spectrum of Supranol Fast Orange GSN 140% in 50% pyridine

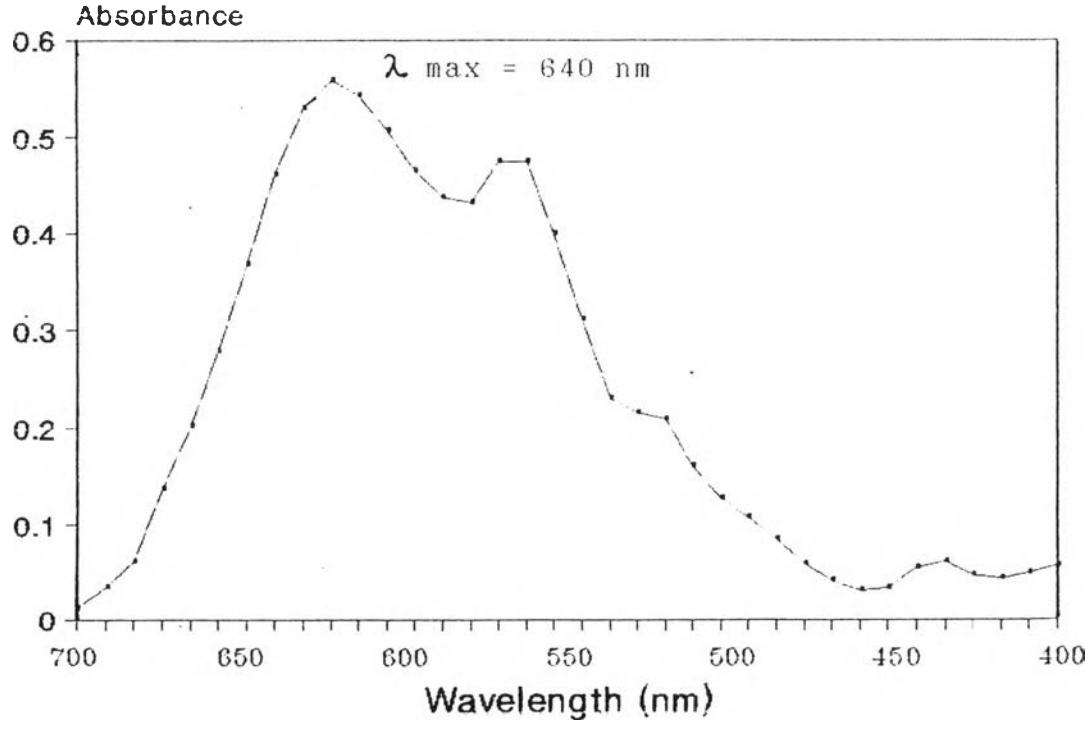


FIGURE E.1b Absorption spectrum of Kayacyl Sky Blue R in 50% pyridine

The concentration of the stock solution of acid dyestuff according to Section 3.5.4.3a is shown in Table E.1.

TABLE E.1 The concentrations of stock solution of acid dyestuffs and their λ max in 50% pyridine

Dyestuff	Concentration (mg/l)	λ max (nm)
Supranol Fast Orange GSN 140% (C.I. Acid Orange 53)	147.0	505
Kayacyl Sky Blue R (C.I. Acid Blue 62)	167.0	640

The data for calibration curve preparation according to Section 3.5.4.3b are summarized in Tables E.2a and E.2b.

TABLE E.2 Calibration curve of various dyestuffs

a. Supranol Fast Orange GSN 140%

Concentration (mg/l)	Absorbance
1.47	0.033
4.41	0.096
7.35	0.158
10.29	0.212
14.70	0.300
17.64	0.362
22.05	0.448
24.99	0.510
29.40	0.600

TABLE E.2 (continued)

b. Kayacyl Sky Blue R

Concentration (mg/l)	Absorbance
1.67	0.031
5.01	0.088
8.35	0.142
11.69	0.194
16.70	0.276
20.04	0.344
25.05	0.425
28.39	0.483
33.40	0.562

The calibration curves (absorbance - concentration curve) of the two dye solutions obtained from the calibration method (b) are shown in Figures E.2a and E.2b.

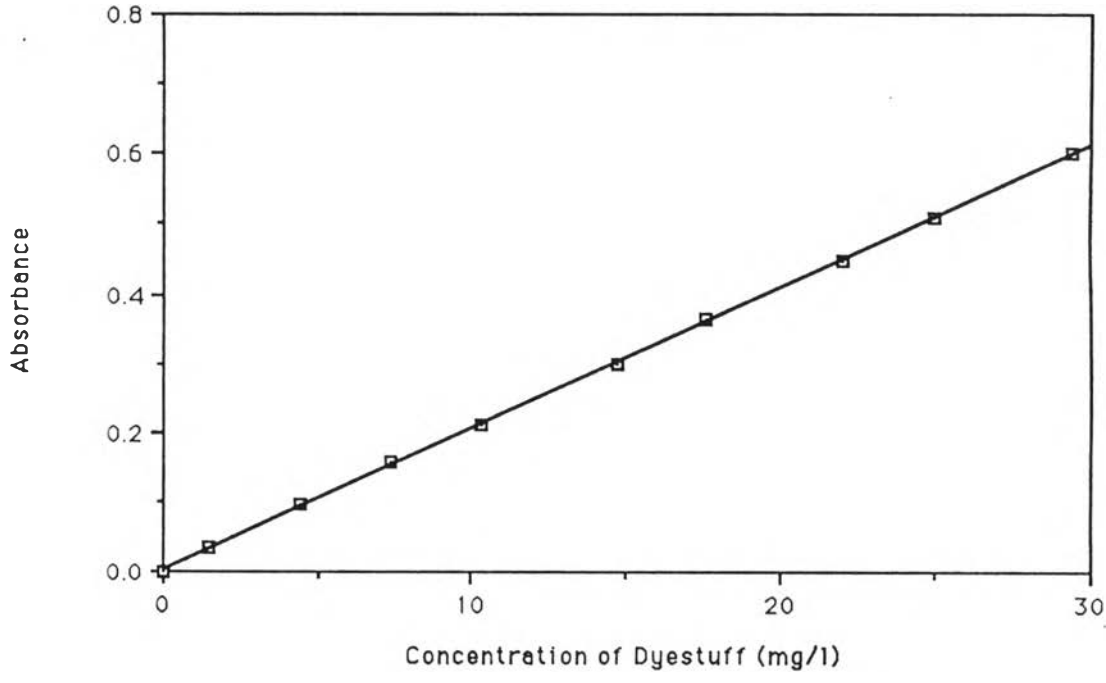


FIGURE E.2a Calibration curve of Supranol Fast Orange GSN 140% in 50% pyridine at λ max = 505 nm

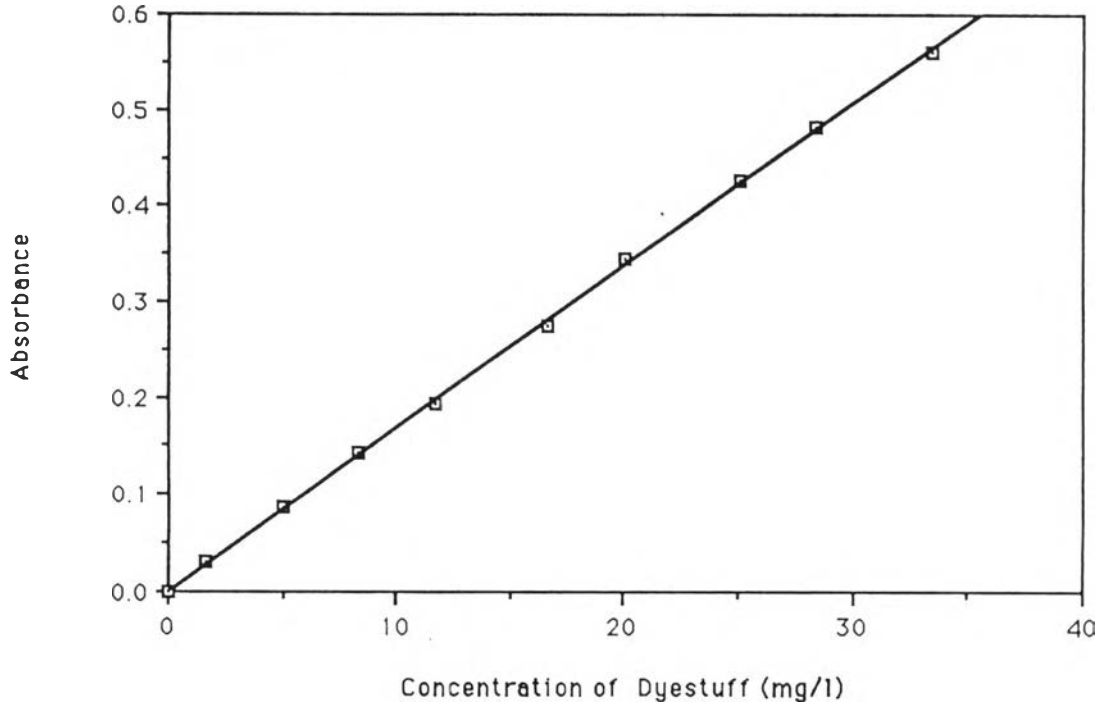


FIGURE E.2b Calibration curve of Kayacyl Sky Blue R in 50% pyridine at λ max = 640 nm

As shown in Figures E.2a and E.2b, the absorbance - concentration curve of each dye solution obtained from the calibration method (b) is a linear relationship, the equation of which is written as below:

$$A = \text{slope} \times C \quad \ldots\ldots \text{(E.1)}$$

then

$$C = \frac{A}{\text{slope}} \quad \text{mg/l} \quad \ldots\ldots \text{(E.2)}$$

where

C = concentrations of the dye solution in mg/l

A = absorbance of the dye solution at its maximum absorption.

$$\text{thus} \quad C = \frac{0.025A}{\text{slope}} \quad \text{mg/25 ml} \quad \ldots \text{(E.3)}$$

The calibration curve of Supranol Fast Orange GSN 140% shows the slope value of 0.020; thus, from equation (E.3), the C value is calculated as follows:

$$C = \frac{0.025A}{0.020} \quad \text{mg/25 ml} \quad \ldots \text{(E.4)}$$

Similarly, the calibration curve of Kayacyl Sky Blue R gives the slope value of 0.017, thus, from equation (E.3), the C value is:

$$C = \frac{0.025A}{0.017} \quad mg/25\ ml \quad \ldots (E.5)$$

According to Section 3.5.4.4, it should be noted that the quantity of the dyestuff on the dyed silk fibres could be determined by using equation (E.3). Therefore, the quantity of the dyestuff, based on 1 gm basis weight of the dyed silk fibres, is calculated by equation (E.6) as follows:

Quantity of the dyestuff fixed on the silk fibres (D_f)

$$= \frac{0.025A \times 1000}{\text{slope} \times 40} \quad \ldots\ldots\ldots\ldots (E.6)$$

Therefore, according to equations (E.4) - (E.6), the quantity of each dyestuff fixed on the silk fibres is calculated in equations (E.7) - (E.8).

Supranol Fast Orange GSN 140%

Quantity of the dyestuff fixed on the silk fibres (D_F)

$$= \frac{0.025A \times 1000}{0.020 \times 40}$$

$$= 31.25A \quad \ldots\ldots\ldots \text{(E.7)}$$

Kayacyl Sky Blue R

Quantity of the dyestuff fixed on the silk fibres (D_F)

$$= \frac{0.025A \times 1000}{0.017 \times 40}$$

$$= 36.76A \quad \ldots\ldots\ldots \text{(E.8)}$$

4.8.2 Calculation of Purity of the Dyestuff

"Absorbance" of the dyestuff solution from 3.5.4.5 = Ap

since $C = \frac{A}{\text{slope}}$ mg/l ..... (E.2)

$$\text{then} \quad C = \frac{Ap}{\text{slope} \times 100} \text{ mg/10ml} \quad \ldots\ldots\ldots \text{(E.9)}$$

Therefore, the dyed sample of 0.0100 gm contains the pure dyestuff with the following concentration:

$$= \frac{Ap \times 50}{\text{slope} \times 100 \times 2 \times 0.0100} \text{ mg} \ldots\ldots\ldots \text{(E.10)}$$

To report on a percentage basis, the 100 gm dyed sample should contain the following quantity of pure dyestuff:

$$= \frac{Ap \times 50}{\text{slope} \times 2 \times 0.0100} \text{ mg} \ldots\ldots\ldots \text{(E.11)}$$

For the determination of the amount of pure dyestuff deposited on the dyed silk fibres, one can use the following calculation:

$$\text{Percentage purity of dyestuff} = \frac{Ap \times 50}{\text{slope} \times 2 \times 10} \ldots \text{(E.12)}$$

According to Section 3.5.4.5 and equation (E.11), the Ap values of Supranol Fast Orange GSN 140% and of Kayacyl Sky Blue R are 0.650, 0.610 respectively. The percentage of purity of these dyestuffs is shown in Table E.3.

TABLE E.3 The percentage purity of the dyestuffs

Dyestuffs	Ap	Percentage purity of dyestuffs
Supranol Fast Orange GSN 140%	0.650	81.25
Kayacyl Sky Blue R	0.610	89.71

E.3 Calculation of percentage exhaustion of the solution

In this research, the silk fibres were dyed at 2% concentration of the dye solution and the corresponding percentage purity of the dyestuff is

$$\frac{Ap \times 50}{slope \times 2 \times 10}$$

Therefore,

$$\frac{\text{quantity of the dyestuff in the dyebath}}{\text{1 gm of the silk fibres}} = \frac{\text{purity of the dyestuff}}{5} \quad \ldots \text{(E.13)}$$

Based on the definition of dyeing, percentage exhaustion can be expressed as follows:

$$\text{percentage exhaustion} = \frac{\text{quantity of dyestuff fixed on silk fibres} \times 100}{\text{quantity of dyestuff in the dyebath}} \quad \ldots\ldots (E.14)$$

VITA

Miss Sireratana Charruchinda was born on May 11, 1959 in Bangkok. She graduated with a Bachelor Degree in Materials Science from Chulalongkorn University in 1980. She has worked in the Textile Chemistry Sub-division, Textile Industry Division, Department of Industrial Promotion, Ministry of Industry since 1984.