CHAPTER III

EXPERIMENTAL

3.1 Materials

Materials used in this research as tabulated in Table 3.1 are obtained from various sources.

Table 3.1 Materials and source.

Materials	Trade name	Source of suppliers
- Eucalyptus leaves and Eucalyptus bark	-	Kasetsart University
- Filter papers No.41	-	Rungsup Chemical Co., Ltd.
- Silk Fabric	-	Sumet Labtest Co., Ltd.
- Cotton Fabric	-	Sumet Labtest Co., Ltd.
- Multifiber adjacent fabric (D.W. type)	Multifiber	Sumet Labtest Co., Ltd.
- Cotton rubbing cloth	Cotton lawn	Sumet Labtest Co., Ltd.
- Blue wool standard No.1 – No.8	Blue wool fabric	Sumet Labtest Co., Ltd.
- Distilled water.	-	Intertek Testing Services (Thailand) Co., Ltd
- Ethanol	Ethyl alcohol	Italma (Thailand) Co., Ltd.
- Methanol	Methyl alcohol	Italma (Thailand) Co., Ltd.
- Potassium aluminium sulphate	$AlKO_8S_2 \bullet 12H_2O$	Italma (Thailand) Co., Ltd.
- Copper sulphate	$CuSO_4 \bullet 5H_2O$	Italma (Thailand) Co., Ltd.
- Stannous chloride	$SnCl_2 \bullet 2H_2O$	Italma (Thailand) Co., Ltd.
- Iron (II) sulphate	$FeSO_4 \bullet 7H_2O$	Italma (Thailand) Co., Ltd.
- Non-Ionic soap	Metapon X-80	Ilin Enterprise Co., Ltd.
- Acetic acid	CH_3COOH	Lab Scan Asia Co., Ltd.
- Sodium carbonate	Na_2CO_3	Italma (Thailand) Co., Ltd.

3.2 Apparatus

3.2.1 Soxhlet Extractor: Witeg Germany.
3.2.2 Soxhlet air bath heating: Model BE4 250
3.2.3 Rotary evaporator: Model V800 & V805 BUCHI
3.2.4 Analytical balance: BP 221S Sartorius
3.2.5 Oven: Binder RD115
3.2.6 Launder-Ometer: Model LEF ATLAS
3.2.7 Perspiration tester: James H. Heal, Halifax, England
3.2.8 Xenon Fade-Ometer: Model Ci3000 ATLAS
3.2.9 A.T.C.C. Crock meter: model CM1 ATLAS
3.2.10 Spectrophotometer: Spectraflash SF600 Datacolor
3.2.11 Cabinet light box: Model CAC150 Verivide
3.2.12 Grey scale for assessing change: SDC Standard method ISO 105-A03:1993
3.2.13 Grey scale for assessing staining: SDC Standard methods ISO 105-A02:1993
3.2.14 Blender
3.2.15 Desiccator
3.2.16 pH meter: MP220 Mettler Toledo

3.3 Experimental process

3.3.1 Dye extraction from leaves and bark

The fresh eucalyptus leaves were baked in oven at 100°C, 48 hours and crumble by blender. The eucalyptus bark were cut into small pieces by blender. These two kinds of crumble were the raw material for dye extraction

3.3.1.1 Find the suitable temperature for dye extraction

The crumble of eucalyptus leaves and bark were separately extracted in the soxhlet extractor, which varied the temperature at 50°C, 60°C, 70°C, 80°C, 90°C and 100°C, the time was constant variable at 3 hours. The liquor ratio of material /

solvent was 1:40. Three types of solvent were used; distilled water, methanol and ethanol, respectively as shown in Table 3.2. The results of extraction in each material varied by time and solvent were calculated to the percentage of total solid.

The percentage of total solid was calculated by following method.

a) 100 ml. Solution from the extraction was put into the rotary evaporator until all solvent was evaporated and remained the solid material.
b) The solid material was baked in oven at 100°C, for 4 hours.
c) The baked solid material was brought in to the desiccator at 30 minutes
d) Weighing the solid material and calculated the percentage of total solid

Table 3.2 Condition of dye extraction from eucalyptus leaves and bark by varying temperature for dye extraction

Eucalyptus	Solvent	Temperature (°C)						Time (Hours)	Liquor ratio
Leaves	Distilled water	50	60	70	80	90	100	3	1:40
	Methanol								
	Ethanol								
Bark	Distilled water								
	Methanol								
	Ethanol								

3.3.1.2 Find the suitable time for dye extraction

5 grams crumble of leaves and bark of eucalyptus were extracted separately in soxhlet extractor, varying time from 1 hour, 2 hours and 3 hours respectively at 100°C, the ratio of material / solvent was 1:40. The types of solvent were used water, ethanol and methanol (Table 3.3). Follow with the process to calculate the percentage total solid as 3.3.1.1

Table 3.3 Condition of dye extraction from eucalyptus leaves and bark by varying time for dye extraction

Eucalyptus	Solvent	Temperature (^{o}C)	Time (Hours)			Liquor ratio
Leaves	Distilled water	100	1	2	3	1:40
	Methanol					
	Ethanol					
Bark	Distilled water					
	Methanol					
	Ethanol					

3.3.1.3 Find the suitable liquor ratio of material and solvent for dye extraction

20 grams (for L:R = 1:10), 10 grams (for L:R = 1:20), 6.6 grams (for L:R = 1:30) and 5 grams (for L:R = 1:40) of crumble leaves and bark of eucalyptus were extracted separately in soxhlet extractor, varying the liquor ratio (L:R) from 1:10, 1:20, 1:30 and 1:40 respectively at 100°C for 1 hour. Three types of solvent were used, water, ethanol and methanol as seen in Table 3.4. Following the process to calculate the percentage total solid as 3.3.1.1

Table 3.4 Condition of dye extraction from eucalyptus leaves and bark by varying liquor ratio of material and solvent for dye extraction

Eucalyptus	Solvent	Temperature (^{o}C)	Time (Hours)	Liquor ratio			
Leaves	Distilled water	100	1	1:10	1:20	1:30	1:40
	Methanol						
	Ethanol						
Bark	Distilled water						
	Methanol						
	Ethanol						

3.3.2 Find the optimal condition for dyeing on silk and cotton fabrics

The dye of eucalyptus leaves and bark using solvent was distilled water at 100°C for 1 hour. The liquor ratio of material/solvent is 1:40

3.3.2.1 Find the optimal temperature condition for dyeing on silk and cotton fabrics

Cotton and silk fabric were dyed with the extracted leaves and bark eucalyptus dye in launder O-Meter machine, vary temperature from 30°C,40°C, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C sequentially, at 60 minutes, the liquor ratio of dye solution was 1:50 as seen in Figure 3.1. The dyeing result was measured to the ΔL*, Δa*, Δb* and K/S value by spectrophotometer.

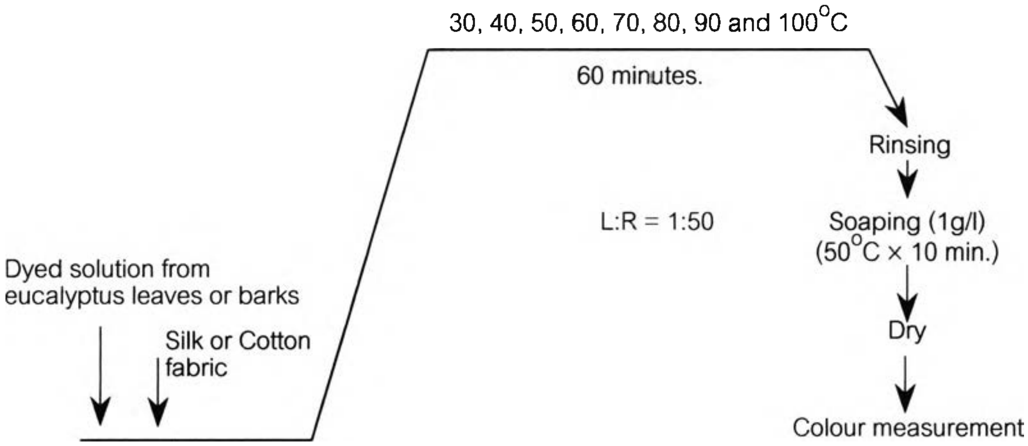

Figure 3.1 Diagram for dyeing on silk and cotton fabrics (Temperature parameter)

3.3.2.2 Find the optimal time condition for cyeing on silk and cotton fabric

The process of experiment in 3.3.2.1 was follow but specific time from 10, 20, 30, 40, 50 and 60 minutes respectively, at 60°C for cotton fabric and 90°C for silk fabric, the liquor ratio of dye solution was 1:50 as seen in Figure 3.2. The dyeing result was measured to the ΔL*, Δa*, Δb* and K/S value by spectrophotometer.

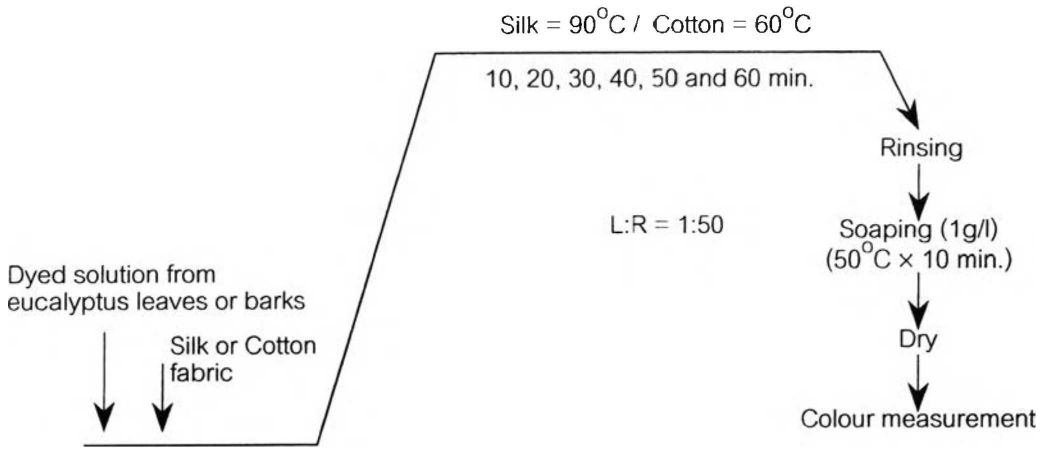

Figure 3.2 Diagram for dyeing on silk and cotton fabrics

(Time parameter)

3.3.2.3 Find the optimal liquor ratio condition for dyeing on silk and cotton fabrics

Follow the process of experiment in 3.3.2.1 by varying the liquor ratio from 1:10, 1:20, 1:30, 1:40 and 1:50 sequentially at 60°C for cotton fabric and 90°C for silk fabric, for time 40 minutes as seen in Figure 3.3. The dyeing result was measured to the ΔL*, Δa*, Δb* and K/S value by spectophoto meter

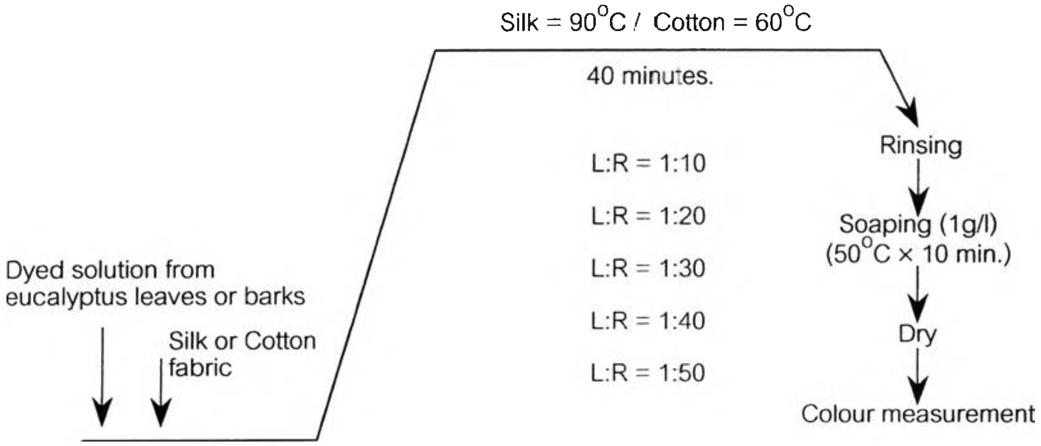

Figure 3.3 Diagram for dyeing on silk and cotton fabrics

(Liquor ratio parameter)

3.3.2.4 Find the optimal pH of dye solution condition for dyeing on silk and cotton fabrics

The silk and cotton fabric, were dyed in launder O-Meter machine, with the dyed solution was extracted from the eucalyptus leaves and bark, varying the pH value as 4, 5, 6 and 7 respectively for silk dyeing, dyed solution with pH 7, 8, 9 and 10 respectively for cotton dyeing. The constant variable are the temperature of dyeing as 90°C for silk fabric and 60°C for cotton fabric for 40 minutes. The liquor ratio was 1:30, the result of dyeing was measured the K/S value by using spectophotometer.

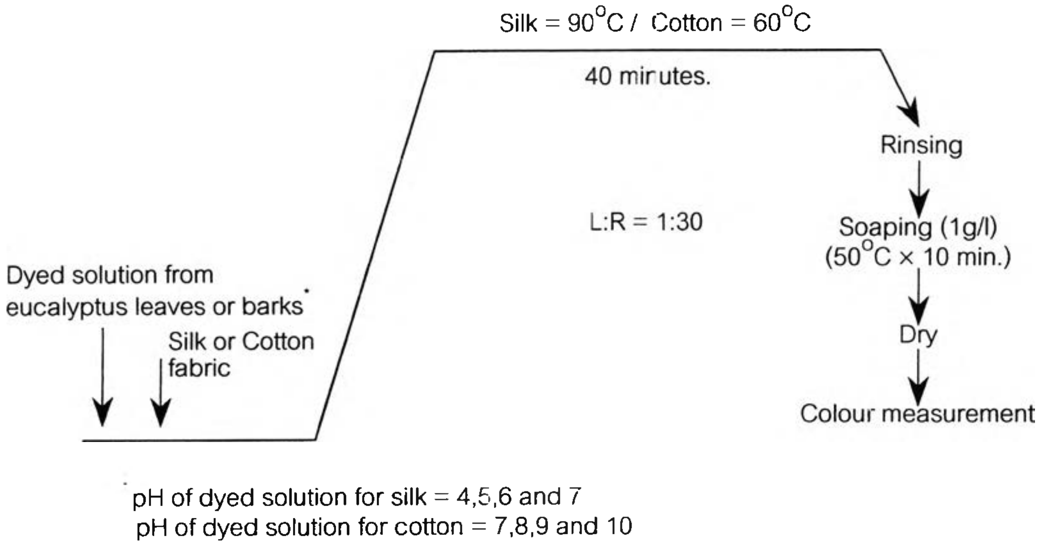

Figure 3.4 Diagram for dyeing on silk and cotton fabrics

(pH value of dye solution parameter)

3.3.3 Find the optimal condition for pre-mordant on silk and cotton fabrics

The optimal condition for mordant application on silk and cotton was investigated which in pre-mordant process varied the mordant substance as following, Potassium aluminium sulphate, Copper sulphate, Ferrous sulphate and stannous chloride, and the suitable time, temperature and quantity of mordant substance which performed the highly effective result was determined. After the silk and cotton fabric which treated in pre-mordant process, passed through the dyeing process at, 60°C, duration 40 min, L:R 1:30, They were measured for K/S value by spectrophotometer.

3.3.3.1 Find the optimal temperature condition for pre-mordant on silk and cotton fabrics

The silk and cotton fabric for premordant process was prepared by varying the temperature as: 30°C, 40°C, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C sequently, the constant variable was the time which fixed at 50 minutes. The quantity of mordant substance was 0.1% on weight fabric, then passed thought dyeing process, when finished, measured the K/S value by spectrophotometer (Figure 3.5).

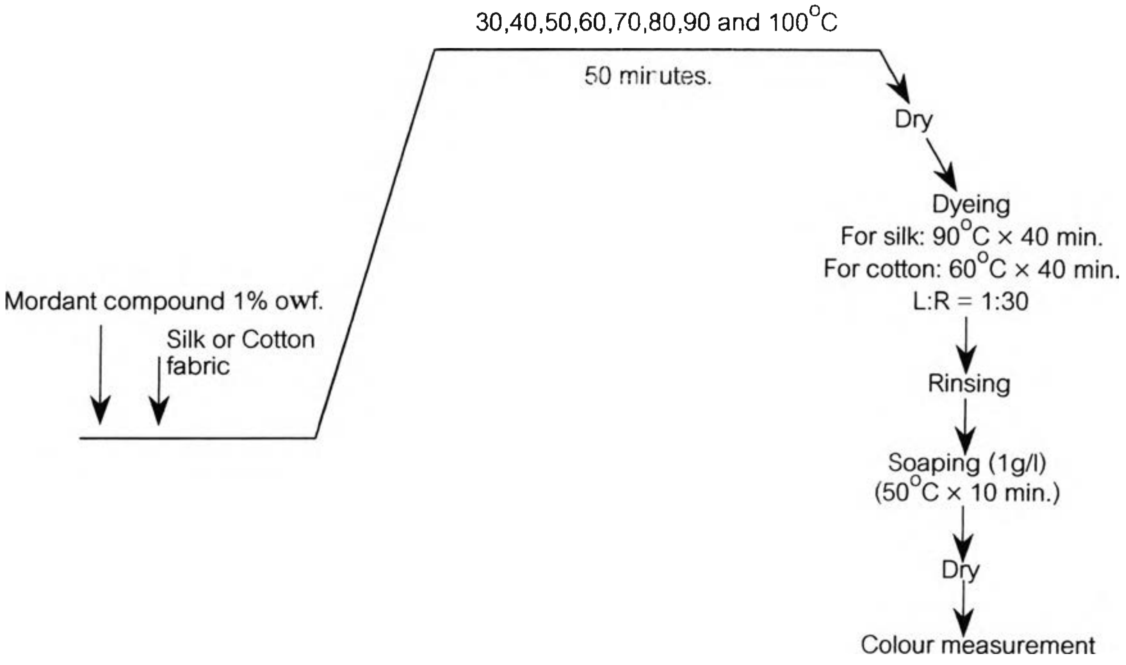

Figure 3.5 Diagram for pre-mordant on silk and cotton fabrics (Temperature parameter)

3.3.3.2 Find the optimal time condition for pre-mordant on silk and cotton fabrics

The silk and cotton fabric for premordant process was prepared by varying the time: 10 min., 20 min., 30 min., 40 min. and 50 min. The constant variable was temperature, which fixed at 30^{0}C. The quantity of mordant substance was 0.1% on weight fabric. The pre-mordanted fabric passed through the dyeing process, when finished, measured the K/S value by spectrophotometer (figure 3.6).

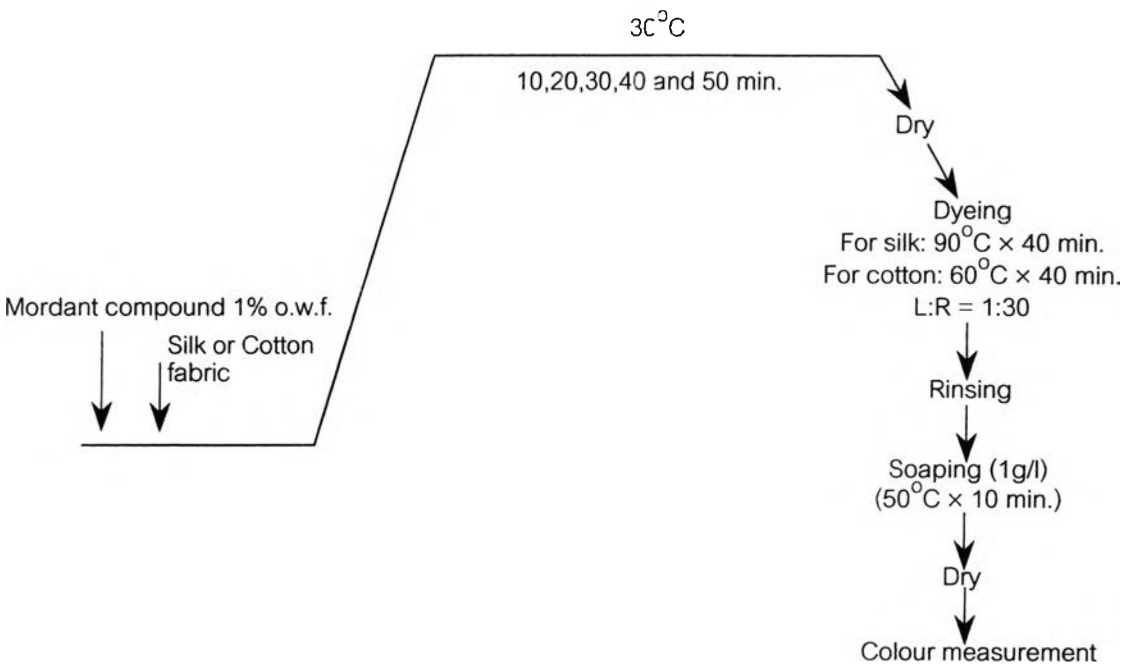

Figure 3.6 Diagram for mordant on silk and cotton fabrics

(Time parameter)

3.3.3.3 Find the optimal quantity condition for pre-mordant on silk and cotton fabric

The silk and cotton fabric for pre-mordant process was prepared by varying the quantity of mordant substance: 0.1%, 0.5%, 1.0%, 1.5% and 2.0% on weight fabric sequently, the constant variable was temperature at 30^{0}C and time at 10 min. The pre-mordanted fabric passed though the dyeing process, when finished, measured the K/S value by spectrophotometer (Figure 3.7).

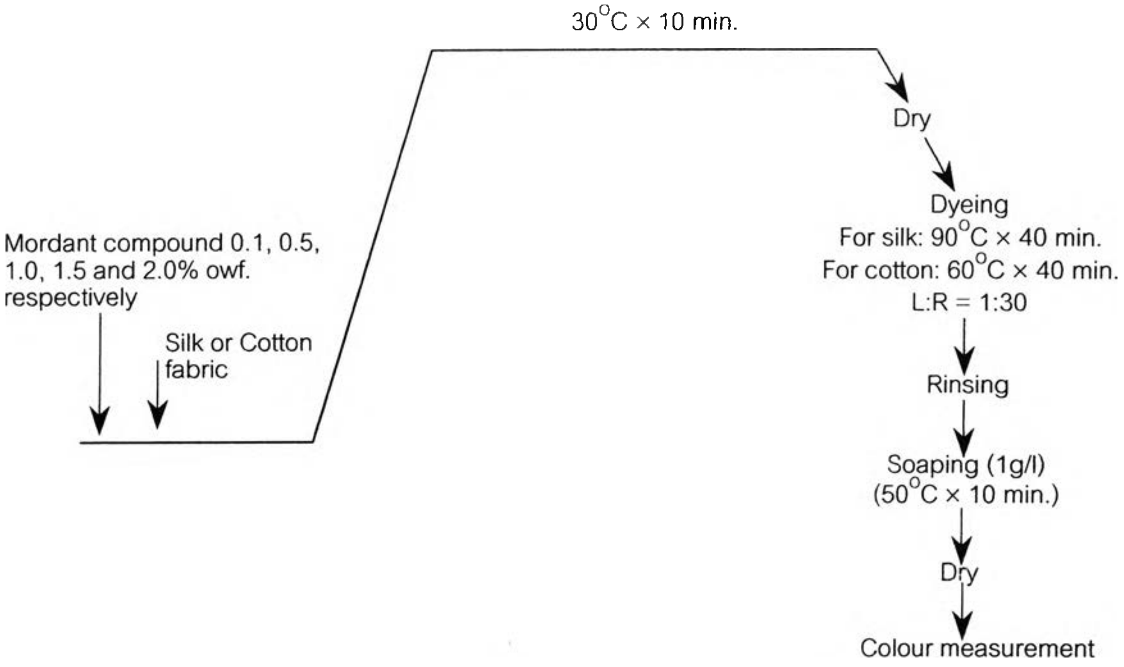

Figure 3.7 Diagram for pre-mordant on silk and cotton fabrics
(Quantity of mordant compound parameter)

3.3.4 Suitable condition for silk and cotton dyeing from eucalyptus leaves and bark

3.3.4.1 Suitable condition for pre-mordant

In pre-mordant process, each substance, used as mordant on silk and cotton fabric must follow this condition (Figure 3.8)

- Temperature of pre-mordant = 30°C
- Time of pre-mordant = 10 minutes
- Quantity of mordant compound = 0.1% on weight fabric
- Mordant compound: Potassium aluminium sulphate, Copper sulphate, Ferrous sulphate, Stannous chloride.

The pre-mordanted fabric was passed through the dyeing process in the suitable condition on next step.

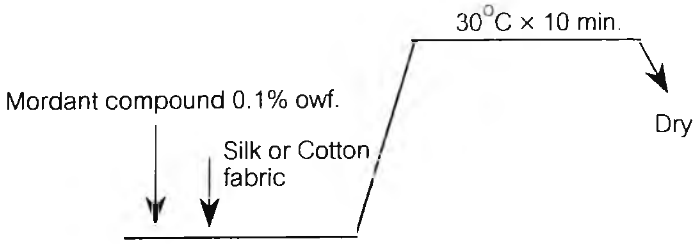

Figure 3.8 Diagram of suitable condition for pre-mordant

3.3.4.2 Suitable condition for silk and cotton for dyeing

The pre-mordanted silk and cotton fabric were passed through the dyeing process as following conditions (Figure 3.9)

- Temperature of dyeing: For silk = 90°C and cotton: 60°C
- Time of dyeing: 40 minutes
- Liquor ratio of dyeing solution: 1:30
- pH of dyeing solution: pH 4 for silk and no adjustment for cotton

The dyed fabric was measured the K/S value bind colour fastness sequently.

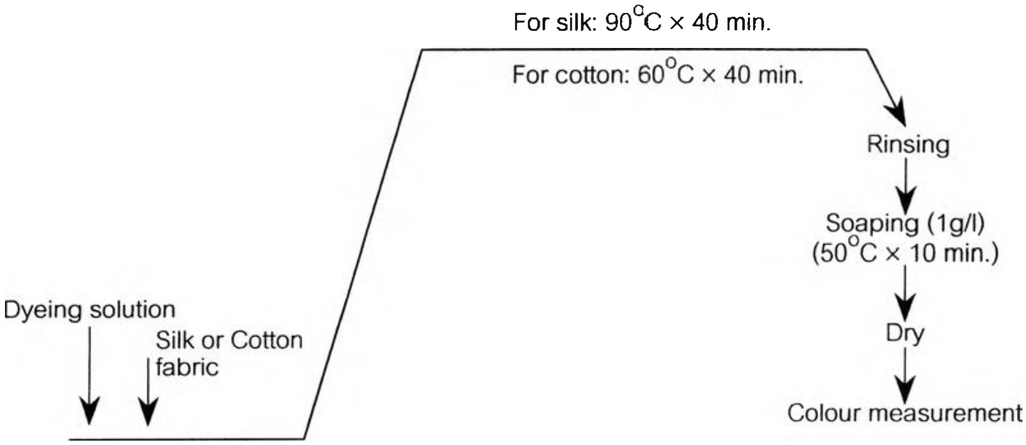

Figure 3.9 Diagram of suitable condition for dyeing

3.3.5 Colour fastness test

With regard to the dyestuff properties of the dyeing agent, the ability of mordanting agent to fix on a fiber is the important requirement. Obviously, this relates to the colour fastness properties, which normally test according to ISO standard test. These tests including colour fastness to light, colour fastness to washing, colour fastness to water colour fastness to perspiration, and colour fastness to rubbing. The test result were expressed in grey scale terms instead of the k/S value. According to the standard grey scale for color change and grey scale for colour staining [21], [22]. Sets of grey scales, examples of which are shown in Figure 3.10 and Figure 3.11. The fastness grades are given in Table 3.5 and Table 3 6

Table 3.5 Grey scale terms for colour change

Grade	Colour fastness
5	Excellent (no change)
4	Good (Slightly change)
3	Fair (noticeably change)
2	Poor (considerably change)
1	Very poor (much change)

Table 3.6 Grey scale terms for colour staining

Grade	Colour fast
5	Excellent (no staining)
4	Good (Slightly staining)
3	Fair (noticeably staining)
2	Poor (considerably staining)
1	Very poor (much staining)

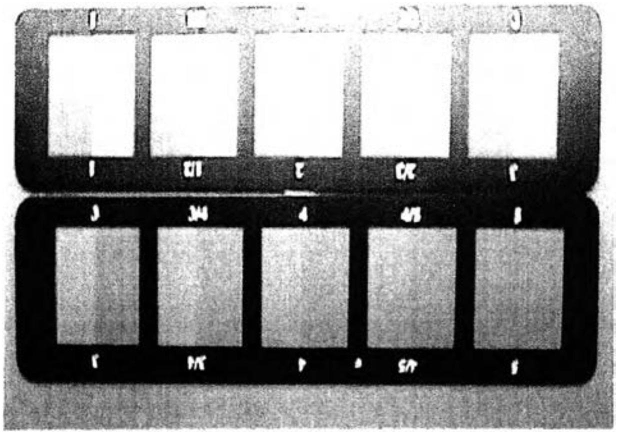

Figure 3.10 Grey scale for colour change

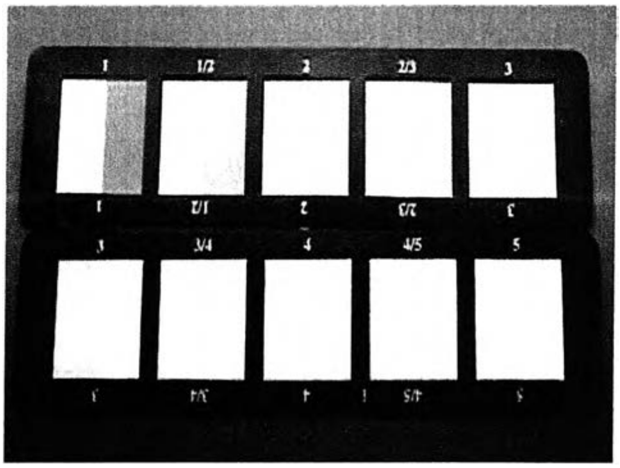

Figure 3.11 Grey scale for colour staining

3.3.5.1 Sample preparation

Fabric was usually tested in the form of a composite specimen (10 cm. x 4 cm.), made up of the test specimen placed in contact with undyed fabric, usually in the form of multifiber strip, of the same size. The purpose of the undyed fabric is to measure the staining effect of any dye that has been lost from the test fabric.

There are two types of multifiber adjacent fabric, one with wool which was type DW and one without wool which was given in Table 3.7 [23].

Table 3.7 Multifiber strip

Multifiber DW	Multifiber TV
Secondary acetate	Triacetate
Bleached cotton	Bleached cotton
Polyamide	Polyamide
Polyester	Polyester
Acrylic	Acrylic
Wool	Viscose

3.3.5.2 Colour fastness to light (ISO 105-B02: 1994) [24]

This test measures the resistance to fading of dyed textiles when exposed to daylight. The test was of importance to the dyestuff manufacturer, the dyer and the retailer. The xenon arc light was used for these test.

(i) Light sources: The xenon arc was a much more intense source of light which has a very similar spectral content to that of daylight so that the test is speeded up considerably. Because of the large amount of heat generated by the lamp on efficient heat filter to be placed between the lamp and specimen and the temperature monitored. This was in addition to a glass filter as above to remove to ultra-violet radiation [23]. A fade O-meter, an example of which was shown in Figure 3.12

Figure 3.12 Fade-Ometer machine for colour fastness to light test

(ii) Reference standards: The essence of the test was to expose the sample under test to the light source together with eight blue wool reference standards. The sample and blue wool standards were partly covered so that some of the material fades and some was left unfaded. A rating was given to the sample which was the number of the reference standard which shows a similar visual contrast between exposed and unexposed portions as the specimen. This means that the specimen will be given a grade between one (poor light fastness) and eight (highly resistant to fading). If the result was in between two blue wool standards, it is given as for example 3 - 4. There was bule wool reference standard in used these test were identified by the numerical designation 1 to 8. They range from 1 (very low light fastness) to 8 (very high light fastness).

(iii) Recommended procedure: The sample under test and a set of blue wool reference standards were arranged on a suitable backing as shown in Figure 3.13. The middle third of the strips was covered with opaque card (A). The assembly was then exposed to light until the specimen just shows a change in shade (4 – 5 on the grey scale). The number of the standard showing a similar change is noted.

The exposure was continued until the contrast in the specimen was equal to grey scale 4, at which point a second segment of the specimen and standard was covered with another pieced of opaque card (B). The exposure is again continued until the contrast between the exposed and unexposed parts of the specimen is equal to grey scale 3, at which point the exposure was terminated.

When the cards are removed the specimen and standards will show two areas that have been exposed for different lengths of time together with an unexposed area. The specimen was given the rating of the standard which shows similar changes. If the exposed areas have different ratings then the overall rating was the mean of the two ratings.

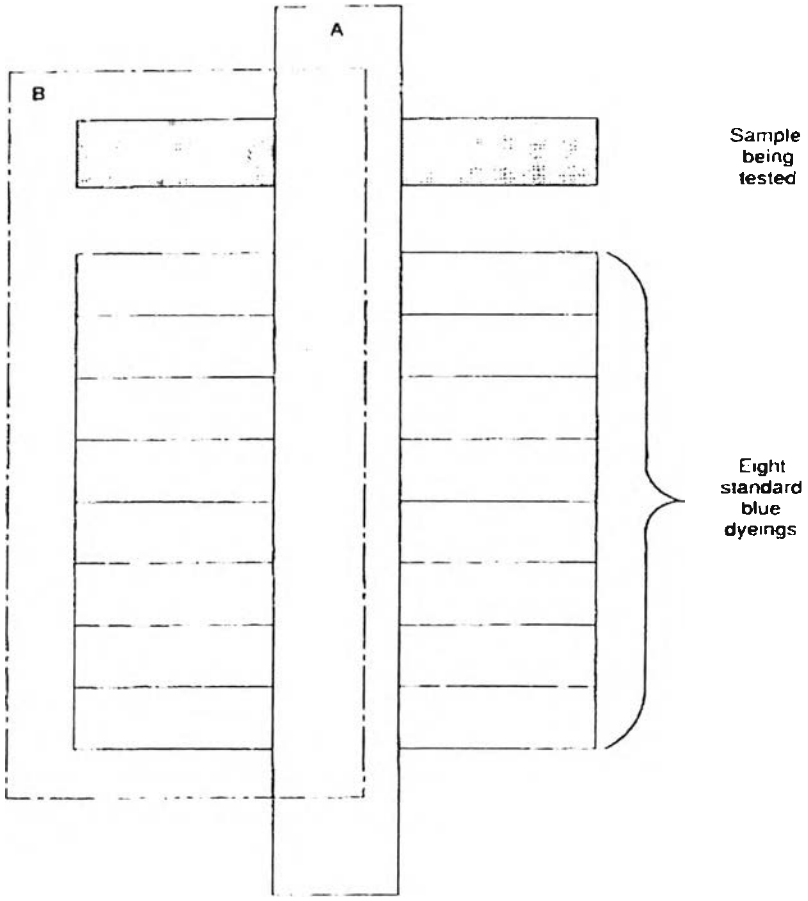

Figure 3.13 Sample for light fastness test

3.3.5.3 Colour fastness to washing (ISO 105-C06 A1S: 1994) [25]

The specimen of the fabric was put in contact with multifiber adjacent fabric. The specimen was treated in an aqueous solution containing 4 g/l of ECE detergent at 40°C for 30 minutes. After that, the colour fastness property of the tested sample was evaluated using the grey scale for colour chang and grey scale for colour staining. The launder O-meter, an example of which was shown in Figure 3.14.

Figure 3.14 Launder-Ometer machine for colour fastness to washing

3.3.5.4 Colour fastness to water (ISO 105-E01: 1994) [26]

The specimen of the fabric in contact with multifiber adjacent fabric was immersed in distilled water, drained and placed between two places under a pressure of 12.5 kPa. Place the test device containing the composite specimen in the oven for 4 hours at 37°C. The specimen and the multifiber adjacent fabric were dried. The change in colour of the specimen and the staining of the multifiber adjacent fabric were assessed with the grey scales. A perspiration tester, an example of which was shown in Figure 3.15, consists of a stainless steel frame constructed to hold a number of glass or acrylic plates each measuring 60 mm. x 115 mm. The samples of side 40 mm. X 100 mm. are each placed separately between a pair of these plates in order to keep them moist. A mass of 5 kg was then placed on top of the apparatus so as to apply a pressure of 12.5 kPa to each specimen. The perspiration tester was so constructed that when the mass was removed the specimens remain under pressure.

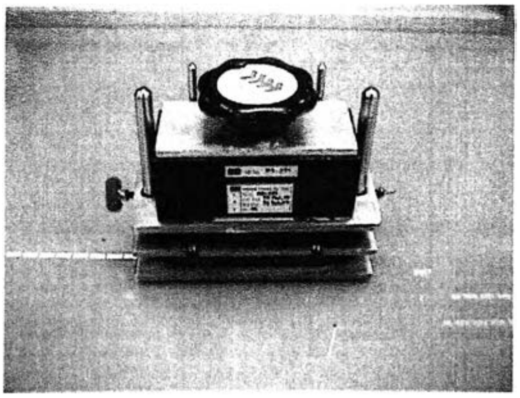

Figure 3.15 Perspiration tester machine for colour fastness to water test

3.3.5.5 Colour fastness to perspiration (ISO 105-E04: 1994) [27]

The specimens of the fabric in contact with multifiber adjacent fabrics were treated in acid solution and alkaline solution containing histidine, drained and placed between two plates under pressure of 12.5 kPa. Place the test device (Figure 3.15) containing the composite specimen in the oven for 4 hours at 37°C. The specimens and the multifiber adjacent fabrics were dried separately. The change in colour of each specimen and the staining of the multifiber adjacent fabrics were assessed with grey scales.

(i) Solutions

1. Alkaline solution, freshly prepared, containing 0.5 g histidine monohydrochloride monohydrate, 5 g sodium chloride and 2.5 g disodium hydrogen orthophosphate (Na_2HPO_4 $2H_2O$) per litre, brought to pH 8.0 with 0.1 N sodium hydroxide.
2. Acid solution, freshly prepared, containing 0.5 g histidine monolydro chloride monohydrate, 5 g sodium chloride and 2.2 g sodium dihydrogen orthophosphate (NaH_2PO_4 $2H_2O$) per litre, brought to pH 5.5 with 0.1 N sodium hydroxide.

(ii) Procedure

Thoroughly wet one composite specimen in the solution at pH 8 (solution 1 above) at a liquor ratio of 50:1 and allow it to remain in the solution for 30 min. at room temperature. Wipe excess liquid off the specimen between two glass rods and place the specimen between two plates of the perspiration tester, with a pressure of 12.5 kPa. Repeat with the other composite specimen in the acid solution using a separate perspiration tester. Place the perspiration tester in an oven at 37°C for 4 hrs. and then removed specimen, open out allow to dry. Both specimens were then assessed for colour change of the test fabric and staining of the multifiber fabric.

3.3.5.6 Colour fastness to rubbing (ISO 105-X12: 2001) [28]

The specimens of the fabric was rubbed with a dry rubbing cloth and with a wet rubbing cloth. For dry rubbing, With the dry rubbing cloth flat in place over the end of the finger of crock meter (Figure. 3.16), rub to end fro in a straight line along a track 10 cm. long on the dry specimen, 10 times to and fro in 10 seconds with a drawnward force of a Newton. For wet rubbing, repeat the test described in dry rubbing with a fresh dry specimen and with a rubbing cloth that has been wetted with water, using take-up of about 95-100%. After rubbing, dry the cloth at room temperature. The staining of the rubbing cloths is assessed with the grey scale.

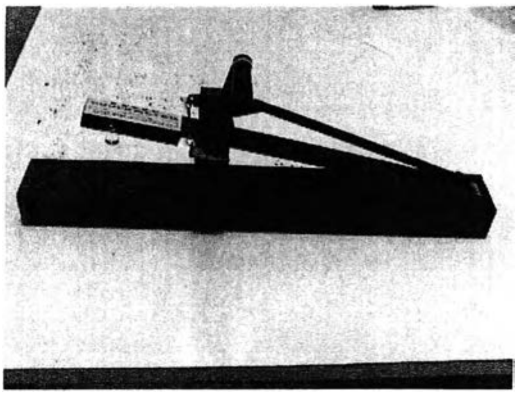

Figure 3.16 Crock meter machine for colour fastness to rubbing test