# ADSORPTION STUDY OF BETACYANIN ON FIBRE

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# ADSORPTION STUDY OF BETACYANIN ON FIBRE

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Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Biotechnology)

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JANUARY 2014

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# STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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# ***Dedication***

*This thesis is dedicated to my parents.*
*For their endless love, support and encouragement.*

# ACKNOWLEDGEMENT

First and foremost, thank you Waheguruji, for always being there for me as a guide and for helping me throughout the completion of this thesis.

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# ABSTRACT

The usage of synthetic dye in food and textile industries has generally shown some threatens to the environment and also human. Since then, natural dye has been introduced as an alternative and recently, research in extracting dye from natural fruit is being done in a large scale. In this research experiment, pitaya's waste was selected as a source for natural dye. However, since pitaya was has high viscosity as one of the properties, it has difficulty to fast on the cloth in textile industries. The ratio of pitaya waste and water is also not known to ensure maximum adsorption on the cloth. In order to tackle this problem, this research will be carried out by using mordant named aluminium sulphate to pretreat the fibres before the dyeing process to ensure the dye fasts on the fibre effectively. Experiments will be conducted to determine the adsorption level by using spectrophotometer and calculations for maximum adsorption will also be done using adsorption isotherm. This experiment was carried out by chemical mechanism. Chemical mechanism refers to solvent extraction using water to extract the dye from the fruit's peel, the usage of mordant on fabric and also the different concentration of the dye due to the ratio of pitaya waste and water. Three different factors that will be studied in this research, namely, the rate of adsorption on fibre using various dye concentrations, the effectiveness of adsorption on different types of fibre and the length of time required for the process of adsorption on the fibre. Basically, the adsorption process increases with the increase of dye concentration and cotton fabric has the highest adsorption effectiveness, followed by polyester fabric and finally chiffon fabric because of the different adsorption strength on the fibres. The equilibrium time required for the process of adsorption on the fibre is dependent on the ability of the fabric to adsorb betacyanin from pitaya waste onto the fibre.

# ABSTRAK

Penggunaan pewarna sintetik dalam industri makanan dan tekstil secara umumnya memberikan ancaman terhadap alam sekitar dan juga manusia. Oleh itu, pewarna semula jadi telah diperkenalkan sebagai alternatif kepada pewarna sintetik, dan baru-baru ini, penyelidikan dalam mengekstrak pewarna daripada buah-buahan semulajadi sedang dilakukan dalam skala yang besar. Dalam kajian ini, kulit buah naga telah dipilih sebagai sumber untuk pewarna semula jadi. Walau bagaimanapun, ekstrak kulit buah naga mempunyai kelikatan yang tinggi, merupakan salah satu sifat ianya sukar untuk menjerap pada kain dalam industri tekstil. Nisbah ekstrak kulit buah naga dan air juga tidak diketahui untuk memastikan penjerapan maksimum pada kain. Untuk menangani masalah ini, kajian ini telah dijalankan dengan menggunakan mordan aluminium sulfat untuk pra rawatan kain sebelum proses pencelupan bagi memastikan penjerapan pewarna ke atas kain dengan berkesan. Eksperimen ini dijalankan bagi menentukan tahap penjerapan dengan menggunakan spektrofotometer dan pengiraan penjerapan maksimum dengan menggunakan kaedah penjerapan isoterma. Eksperimen ini juga telah dijalankan menggunakan mekanisme kimia. Mekanisme kimia dalam kajian ini adalah merujuk kepada pelarut pengekstrakan menggunakan air bagi mengeluarkan pewarna daripada kulit buah naga, penggunaan mordan pada kain dan juga nisbah kulit buah naga dan air yang berbeza. Tiga faktor yang telah dikaji dalam kajian ini, iaitu, kadar penjerapan ke atas kain menggunakan pelbagai kepekatan pewarna, keberkesanan penjerapan pada jenis kain dan tempoh masa yang diperlukan untuk proses penjerapan ke atas kain. Pada asasnya, proses penjerapan bertambah dengan peningkatan kepekatan pewarna, dan kain kapas mempunyai keberkesanan jerapan yang tinggi, diikuti dengan kain poliester dan kain chiffon. Masa keseimbangan yang diperlukan untuk proses penjerapan ke atas kain adalah bergantung kepada keupayaan kain tersebut untuk menjerap pewarna dari kulit buah naga ke atas kain.

## TABLE OF CONTENTS

| | PAGE |
|---|---|
| **SUPERVISOR’S DECLARATION** | **IV** |
| **STUDENT’S DECLARATION** | **V** |
| **DEDICATION** | **VI** |
| **ACKNOWLEDGEMENT** | **VII** |
| **ABSTRACT** | **VIII** |
| **ABSTRAK** | **IX** |
| **TABLE OF CONTENTS** | **X** |
| **LIST OF FIGURES** | **XII** |
| **LIST OF TABLES** | **XIV** |
| **LIST OF ABBREVIATIONS** | **XV** |

**CHAP TER 1 INTRODUCTION**

| | | |
|---|---|---|
| 1.1 | Background of Study | 1 |
| 1.2 | Motivation | 1 |
| 1.3 | Problem Statement | 2 |
| 1.4 | Research Objective | 2 |
| 1.5 | Scope of Study | 3 |

**CHAPTER 2 LITERATURE REVIEW**

| | | |
|---|---|---|
| 2.1 | Introduction | 4 |
| 2.2 | Natural Dyes | 4 |
| 2.3 | Synthetic Dye and Natural Dye | 5 |
| 2.4 | Pitaya Fruit | 6 |
| 2.5 | Appearance of Pitaya Fruit | 8 |
| 2.6 | Species of Pitaya Fruit | 9 |
| 2.7 | Harvesting Period of Pitaya Fruit | 10 |
| 2.8 | Health Benefits of Pitaya Fruit | 11 |
| 2.9 | Nutritional Information of Dragon Fruit | 13 |
| 2.10 | Betacyanin Pigment | 13 |
| 2.11 | Pectin | 15 |
| 2.12 | Chemical and Physical Properties of Pectin | 16 |
| 2.13 | Viscosity of Pectin | 18 |
| 2.14 | Mordants | 19 |
| 2.15 | Types of Mordants | 20 |
| 2.16 | Aluminium Sulphate (Alum) | 22 |
| 2.17 | Cotton | 23 |
| 2.18 | Chiffon | 25 |
| 2.19 | Polyester | 27 |
| 2.20 | UV-Vis Spectroscopy | 29 |
| 2.21 | Adsorption Process | 33 |
| 2.22 | Langmuir Isotherm | 37 |
| 2.23 | Freundlich Isotherm | 38 |
| 2.24 | Fourier Transform Infrared (FTIR) | 39 |
| 2.25 | Scanning Electron Microscope (SEM) | 40 |

2.26 Desorption 41
2.27 Conclusion 41

**CHAPTER 3 METHODOLOGY**

3.1 Introduction 42
3.2 Material Description 43
3.3 Processes 43
3.4 Preparation of Natural Sample Dye 44
3.5 Process of Mordanting 47
3.6 Equilibrium Time 49
3.7 Process of Adsorption 50
3.8 Analysis of Adsorption by UV-Vis Spectrometer 52
3.9 Amount of Dye Adsorbed 54
3.10 Langmuir Isotherm 54
3.11 Freundlich Isotherm 54
3.12 Fourier Transform Infrared (FTIR) 55
3.13 Scanning Electron Microscope 55
3.14 Desorption Process 55
3.15 Statistical Analysis 56

**CHAPTER 4 RESULTS AND DISCUSSION**

4.1 Introduction 57
4.2 Equilibrium Time 58
4.3 Amount of Dye Adsorbed 63
4.4 Adsorption Process at Various Concentrations of Dye 65
4.5 Langmuir Isotherm 71
4.6 Freundlich Isotherm 73
4.7 Fourier Transform Infrared (FTIR) 76
4.8 Scanning Electron Microscope 81
4.9 Desorption Process 88

**CHAPTER 5 CONCLUSION AND RECOMMENDATION**

5.1 Conclusion 89
5.2 Recommendations 90

**REFERENCES 91**
**APPENDICES 96**

A.1 Raw Data 96
A.2 Statistical Analysis 102

## LIST OF FIGURES

| | | PAGE |
|---|---|---|
| Figure 2.1 | Synthetic dye used in textile industries | 5 |
| Figure 2.2 | Bad effect of using synthetic dye to the environment | 6 |
| Figure 2.3 | Pitaya Plant | 7 |
| Figure 2.4 | Flower of Pitaya Plant (Queen of the Night) | 8 |
| Figure 2.5 | Pitaya Fruit | 9 |
| Figure 2.6 | Pectin in Cell Wall | 15 |
| Figure 2.7 | Molecular Structure of Pectin | 17 |
| Figure 2.8 | The Concept of Viscosity | 19 |
| Figure 2.9 | Structure of Aluminium Sulphate (Alum) | 23 |
| Figure 2.10 | Cotton Tree | 24 |
| Figure 2.11 | Cotton Fibre | 25 |
| Figure 2.12 | Chiffon Fibre | 27 |
| Figure 2.13 | Polyester Fibre | 28 |
| Figure 2.14 | UV-Vis Spectrophotometer | 29 |
| Figure 2.15 | Schematic Diagram on How UV-Vis Spectrophotometer Works | 30 |
| Figure 2.16 | Relationship between Light absorption and Colour | 31 |
| Figure 2.17 | Determination of Langmuir Isotherm Constants | 37 |
| Figure 2.18 | Determination of Freundlich Isotherm Constants | 38 |
| Figure 2.19 | Schematic Diagram for FTIR | 39 |
| Figure 2.20 | Basic Construction of SEM | 40 |
| Figure 3.1 | Block Diagram showing the whole process of adsorption of dye onto the fibre and the analysis after that | 43 |
| Figure 3.2 | Process of Natural Dye Preparation | 44 |
| Figure 3.3 | The peel of Pitaya Fruit | 45 |
| Figure 3.4 | The pitaya peel being blend into small pieces | 45 |
| Figure 3.5 | The mixture of Pitaya peel with water being prepared to be centrifuged | 46 |
| Figure 3.6 | Pitaya extract after centrifugation process | 46 |
| Figure 3.7 | Process of Mordanting | 47 |
| Figure 3.8 | The fibres being soaked in the mordant solution | 48 |
| Figure 3.9 | The mordant solution being heated up to 82˚C using water bath | 48 |
| Figure 3.10 | Equilibrium time process | 49 |
| Figure 3.11 | Process of adsorption using various dye concentrations | 51 |
| Figure 3.12 | Different natural dye concentrations are being prepared for adsorption process | 52 |
| Figure 3.13 | Samples being prepared for UV-Vis analysis | 53 |
| Figure 3.14 | Desorption Process | 56 |
| Figure 4.1 | Betacyanin Content on Fibre at Various Time Intervals | 60 |
| Figure 4.2 | Fabric sample while they were left for drying | 61 |
| Figure 4.3 | Fabric sample after they were dried | 62 |
| Figure 4.4 | Amount of Dye adsorbed at different time intervals | 64 |
| Figure 4.5 | Betacyanin Content on Fibres at Various Dilution Factors | 66 |
| Figure 4.6 | Amount of Dye adsorbed at Various Dilution Factors | 68 |
| Figure 4.7 | Fibres while they were left for drying | 69 |
| Figure 4.8 | Fibres after they are dried | 70 |
| Figure 4.9 | Langmuir Isotherm on Adsorption of Dye on (a) Cotton, (b) | 72 |

Chiffon, (c) Polyester

Figure 4.10 Freundlich Isotherm for Adsorption of Dye on (a) Cotton, (b) Chiffon, (c) Polyester 74

Figure 4.11 FTIR images of (a) Untreated Cotton, (b) Treated Cotton, (c) Dyed Cotton 77

Figure 4.12 FTIR images of (a) Untreated Chiffon, (b) Treated Chiffon, (c) Dyed Chiffon 78

Figure 4.13 FTIR images of (a) Untreated Polyester, (b) Treated Polyester, (c) Dyed Polyester 79

Figure 4.14 FTIR analysis for Cotton, Chiffon and Polyester 80

Figure 4.15 SEM images of (a) Untreated Cotton, (b) Treated Cotton, (c) Dyed Cotton 82

Figure 4.16 SEM images of (a) Untreated Chiffon, (b) Treated Chiffon, (c) Dyed Chiffon 84

Figure 4.17 SEM images of (a) Untreated Polyester, (b) Treated Polyester, (c) Dyed Polyester 86

# LIST OF TABLES

| | | **PAGE** |
|---|---|---|
| Table 2.1 | Nutritional Information of Dragon Fruit | 13 |
| Table 2.2 | Freundlich and Langmuir Isotherm | 34 |
| Table 2.3 | Langmuir Isotherm | 36 |
| Table 2.4 | Freundlich Isotherm | 36 |
| Table 3.1 | Water and Dye ratio for Dilution Factor | 50 |
| Table 4.1 | Average Absorbance Reading at 15 minutes interval | 59 |
| Table 4.2 | Amount of Dye adsorbed at various time intervals | 64 |
| Table 4.3 | Average Initial Absorbance Reading at various dilution factors | 65 |
| Table 4.4 | Average Absorbance Reading after 90 minutes at various dilution factors | 66 |
| Table 4.5 | Amount of dye adsorbed (qe) | 67 |
| Table 4.6 | Langmuir and Freundlich Constants for Adsorption of Pitaya Dye on Cotton, Chiffon and Polyester | 75 |
| Table 4.7 | Betacyanin content in Water after Desorption Process | 88 |
| Table A.1.1 | Absorbance Reading at 15 minutes interval for 1st Trial | 96 |
| Table A.1.2 | Absorbance Reading at 15 minutes interval for 2nd Trial | 96 |
| Table A.1.3 | Betacyanin Content in the Dye solution after Adsorption Process | 97 |
| Table A.1.4 | Betacyanin Content on Fibre | 97 |
| Table A.1.5 | Initial absorbance readings at various dilution factors for 1st trial | 98 |
| Table A.1.6 | Absorbance reading after 90 minutes at various dilution factors for 1st trial | 98 |
| Table A.1.7 | Initial absorbance readings at various dilution factors for 2nd trial | 98 |
| Table A.1.8 | Absorbance reading after 90 minutes at various dilution factors for 2nd trial | 98 |
| Table A.1.9 | Initial Betacyanin Content at Various Dilution Factors | 99 |
| Table A.1.10 | Betacyanin Content at Various Dilution Factors after Adsorption Process | 99 |
| Table A.1.11 | Betacyanin Content on Fibre at Various Dilution Factors | 99 |
| Table A.1.12 | Langmuir Isotherm | 100 |
| Table A.1.13 | Freundlich Isotherm | 100 |
| Table A.1.14 | Final Absorbance Reading for 1st Trial (Desorption Process) | 101 |
| Table A.1.15 | Final Absorbance Reading for 2nd Trial (Desorption Process) | 101 |
| Table A.1.16 | Average Final Absorbance Reading (Desorption Process) | 101 |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| Io | intensity of the incident light |
| I | intensity of light transmitted through the sample solution |
| c | concentration of the solute in mol l-1 |
| l | path length of the sample in cm |
| ε | molar absorptivity |
| A | absorbance |
| DF | dilution factor |
| MW | molecular weight of betacyanin (550gmol$^{-1}$) |
| Є | molar extinction coefficient (60,000 L/mol cm in $H_2O$) |
| l | path length of cuvette (1cm) |
| BC | betacyanin content |
| $C_o$ | initial concentration of solution (g/m$^3$) |
| $C_e$ | equilibrium concentration of solution (g/m$^3$) |
| V | volume of solution (m$^3$) |
| W | weight of pitaya waste used (kg) |
| $q_e$ | dye bound per unit amount of adsorbent (mg/g) |
| $C_e$ | unbound dye concentration (in solution) (mg/L) |
| $q_{max}$ | saturation constant (mg/g) |
| $K_L$ | affinity constant for Langmuir isotherm (L/mg) |
| $K_f$ | freundlich adsorption constant |
| LSD | least significance difference |
| FTIR | Fourier Transform Infrared |
| SEM | Scanning Electron Microscope |
| ANOVA | Analysis of Variance |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Natural dyes are well known in producing extraordinary diversity of rich and colours that complement each other (Singh et al., 2012). Natural dyes can be used as a colouring agent mostly on textiles and food. It can also be used as a substitute of synthetic chemical as conventional dye. This is because synthetic dyes that are mostly used in the textile industries may cause pollution of water and give harmful effects to sensitive skin users. In this research, pitaya peel will be used as the source of natural dye. According to Jamilah et al. (2011), red pitaya (*Hylocereus polyrhizus*), which is a member of the family Cactaceae is known as dragon fruit or pitaberry locally. When this fruit has ripened, it has an attractive purple-red peel and the content within which is delicate and juicy with small black seeds well-dispersed. Pitaya is well known due to its health benefit claims such as reducing dyslipidemia. The pigment that is contained in pitaya peel is called betacyanin. . Because of the inherently poorer fastness properties of natural dyes compared to synthetic dyes, additional research on natural dyes and mordant agents is necessary to obtain the best colorfastness results. Many salts, called mordants, are used to help affix natural dyes to fabrics, called mordants, but the most common is aluminum sulfate (Erica, 2010).

## 1.2 Motivation

Synthetic dyes that are used in large scale nowadays are significant pollutants that cause environmental and health problem. Most of them contain a high content of toxic material and this constitutes a serious hazard for human and aquatic animals (Alver et al., 2012). To overcome the consequences of using synthetic dye, natural dye is used as an alternative in textile, food processing, and dye manufacturing industries. Pitaya waste, one of the many natural dyeing sources existing currently drew much attention of worldwide researchers, notonly because of their red-purple color as natural dye, but also for phenolic and flavonoids contents in pitaya waste which have potiential health benefits (Lee, 2009). According to Bulut et al. (2006), there are many methods of dye extraction from aqueous solution, however, adsorption is considered to be more reliable compared to other techniques because of its low cost, simplicity in design, viability and ability to treat dyes in more concentrated form. In this research, pitaya waste will be used as the adsorbate and fabric such as cotton, chiffon and polyester will be used as adsorbent.

## 1.3 Problem Statement

Pitaya waste is one of the many natural resources available to produce natural dye instead of using synthetic chemical as conventional dye which may be harmful to the environment. Enzymatic vice, the dye obtained from pitaya peel do not fast on the fabric for long and it can be easily washed away from the fabric. This may be due to the high level of viscosity of the dye which is probably caused by the presence of pectin in the dye. It is of no doubt that pitaya peel has the ability of producing a good quality natural dye, but the problem arises when we do not know the exact ratio of the concentration of pitaya waste and water to ensure maximum adsorption process on the fabric. There is also no proper calculation made available to determine the maximum adsorption rate on the dye.

## 1.4 Research Objectives

This study is carried out to study the Langmuir and Freundlich adsorption isotherm of betacyanin on various types of fibre.

## 1.5 Scope of Study

The scopes of study are:

a) Identify the length of time required for the process of adsorption on the fabric. The range of time is in the interval of 15 minutes for 120 minutes to determine the equilibrium time. Equilibrium time is important so that Langmuir and Freundlich isotherm can come into play for the analysis of adsorption isotherm.

b) Determine the rate of adsorption on fibre using various different dye concentrations. The ranges of the concentrations are from 0.93, 0.87, 0.80, 0.73, and 0.67 based on dilution factor in the form of ratio of stock solution and water. The rate of adsorption can be analysed by obtaining the optical density reading using UV-Vis Spectrophotometer. This can be done by calculating the betacyanin content using the equation as mentioned in the methodology by substituting the optical reading density obtained.

c) Analyse the effectiveness of adsorption on different types of fabric. The effectiveness of the adsorption can be determined by calculating the amount of dye that got adsorbed onto the different types of fibre at a given time. All the different fabrics will have different adsorption rate depending on the rate of fastness of each kind of cloth. The colour intensity of each kind of cloths will be different.

d) Carry out the qualitative analysis on the fibres that best adsorp the dye using Scanning Electron Microscope (SEM) and Fourier Transform Infrared (FTIR).

e) Determine whether the dye desorps after rinsing the fibre.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction

This section will discuss on the reviews of adsorption study of betacyanin on fibre. The review will be divided into a number of parts which includes the definition of natural dye, type of natural dye that will be used, mordants and adsorption isotherms.

## 2.2 Natural dyes

The use of natural dyes commercially in the textile and apparel industry has been increasing rapidly (Flint, 2008). Natural dyes can be anything that comes from natural resources, such as flowers, leaves, plants, bark, roots, shells, and mineral substances (Cardon, 2007). The color is often extracted from the natural source for dyeing process. There is a growing interest in the use of natural pigments for textile coloring because natural products are associated with quality and health promotion whereas synthetic pigments are usually assessed by consumers (Downham et al., 2000). Natural dyes from plant sources are receiving great interest from both textile manufacturers and consumers in the continuing replacement of synthetic dyes (Duhard *et al.,* 1997). Though the demand in textile industries is increasing, there is a problem occurred due to the lipid content in the dye which affects its stability, purity and integrity (Stintzing and Carle, 2004).

**Figure 2.1:** Synthetic dye used in textile industries

The natural dye extracts selected for this study is betacyanin pigment from pitaya fruit or most commonly known as dragon fruit. Pitaya fruit is selected for this research as it is widely used throughout the history and has good colorfast properties. In addition to that, dragon fruit can be easily obtained from anywhere. The particular type of pitaya fruit that will be used for this research is red pitaya.

## 2.3 Synthetic Dye and Natural Dye

Dyes are widely used in industries such as textiles, leather, paper, and plastics to colour their final products. Among them, the textile industry is one of the extensive dye users for the dyeing process of various fibre types. As such, wastewaters from the textile industry contain quite a number of other polluting matters, such as toxic organic residuals, acids, bases and inorganic matter, in addition to synthetic dyeing material. Some dyes are carcinogenic as they are being produced from dangerous chemicals, such as benzidine, metals and many more (Miodrag et al., 2010). Application of synthetic dyes can have bad affects on the health of the workers, and many dyes have been banned from use because they are possible carcinogens. Besides that, nearby rivers are really polluted that some villages, use river water as source of drinking water. The use of natural mordant dyes also can have an environmental impact when the more toxic chromium mordants are used. Other disadvantages of natural dyes include the greater quantity need to produce a desired depth of shade and inherently poorer fastness properties compared to synthetic dyes. However, the disadvantages of using natural dyes are not really appealing because it has lower environmental impact and human health effects than synthetic dyes (Flint, 2008).

The interest of using natural pigments for food coloring and also textile colouring is increasing because natural products are guaranteed with quality and health assurance whereas synthetic pigments are critically assessed and are not really favoured by consumers. Due to strong consumer demand for more natural product which assures more safety and health benefit, the trend towards replacement of synthetic dye by natural dye has been increasing although the cost of producing natural dye product is higher (Phebe et al., 2009).

**Figure 2.2:** Bad effect of using synthetic dye to the environment

## 2.4 Dragon Fruit

Since natural dye is preferred compared to synthetic dye in the industry, there are many colourful and characteristic fruits that are taken down to the industry for the process of extracting dye. One of the most preferred fruit among textile industries is called the Dragon fruit. Dragon fruit (*Hylocereus undatus*) or pitaya originated principally from the tropical and subtropical forest regions and currently occupies a growing nich in Europe’s exotic fruit market. This fruit has gained a lot of interest among the society because of its pleasant taste, nutritional value, exotic features and attractive colours (Belec et al., 2006). The red colour of pitaya fruit is attributed by betacyanins, which is a class of water-soluble, nitrogen containing vacoular pigments .The the yellow betaxanthins and the red-violet betacyanins belong to the betalain pigments, which are characteristics for plants of the order Caryophyllales. Betalains occur only in the plants from 10 families in the order Caryophyllales (Phebe et al., 2009). Almost 22% of the whole dragon fruit is covered by the pitaya peel.

The pitaya fruit grows best in dry tropical climate with average temperature of 21 – 29 degree celcius. However, it can also withstand temperature as high as 40 degree celcius and temperature as low as 0 degree celcius for short period of time. Therefore we can actually preserve the fruit for some time before consuming it. Pitaya fruit is best suited with rainfall ranging 600 – 1300mm with alternating rain and sunny seasons. Pitaya prefers more sunlight but however, it can also be destroyed if too much of sunlight is applied. Therefore, pitaya fruit really needs some shading. In addition to that, more organic matter and sand content in the soil improves the growth of pitaya fruit (Ong Boon Ping, 2011).

**Figure 2.3:** Pitaya Plant

## 2.5 Appearance of Pitaya Fruit

The pitaya fruit plant is a vine-like, segmented cactus with aerial roots. Because of its nature of being epiphyte, it holds on to cracks where there is a lot of organic materials to obtain its nutrients. Usually, the stem is three sided and occasionally 4 of 5 sided and it is lobed along the ridges which have small swelling and small thorns. From the swelling on the plant, large, fragrant, white petals with yellow centers flowers blooms which measures 1 foot long and 9 inches wide. These flowers only last one night where they bloom in the evening and wilt the very next morning. The flowers are really wonderful looking and because of that, they are given the name ‘moonflower’ or ‘Queen of the night’. Pitaya fruits are actually moderately large with weights ranging between 150-600g. The fruits have bright pink or yellow peel and purple or white flesh with many tiny black seed in them and these characteristics depends on the species of the Pitaya fruit (Ong Boon Ping, 2011).

**Figure 2.4:** Flower of Pitaya Plant (Queen of the night)

## 2.6 Species of Pitaya Fruit

Hylocereus undatus is a climbing cactus from the tropical rainforests of Central and northern South America. It is one species that has been used as a food source for many years now. It has already received worldwide recognition as a plant for the large, scented, night-blooming flowers. Its specialty is now spreading throughout the world for its fruit, especially in Israel, Vietnam and Australia. Other climbing cactus species grown for the edible fruit include Hylocereus polyrhizus and Selenicereus megalanthus. H. polyrhizus has red peel and also red flesh dotted with edible black seeds, while S. megalanthus, the pitaya amarillo or yellow pitaya, has yellow skin and clear to white flesh containing edible black seeds (Chang et al., 1997).

Columnar cacti, such as Cereus peruvianus are also grown for their edible fruit. The Opuntia species are well known due to their noxious weed status. Plants usually spread and grow rapidly from seeds and vegetative pieces. The potential for Hylocereus to become a weed is unknown at this stage (Luders et al., 2006).

**Figure 2.5:** Pitaya Fruit

## 2.7 Harvesting Period of Dragon Fruit

According to Francis et al. (2004), the normal harvesting and production season of pitaya is during the summer, from June to October. As mentioned earlier, three to five spherical buttons normally grows on the stem margins in which two to three of these may develop into flower buds in about 13 days. When anthesis happens, the light green, cylindrical flower buds reach approximately 11 inches after 16–17 days. The flowers open rapidly, starting at around 6:40– 7:00 p.m., and flowering is completed by about 10:00 p.m. At 2:00 a.m., with pollination completed, the flower begins to wilt. The flower petals tend to close completely by daybreak. Pitaya is usually pollinated by moths in the evening, and hand pollination can enhance fruit set and size. Most of the time, the blooming of pitaya flowers gets affected by temperature and light intensity. The flowers may open as early as 4:00 p.m. on a warm, cloudy day, while cool temperatures during off-seasons could slow flower wilting so it closes as late as 10:00 a.m.

Pitaya fruits are nonclimacteric. This means that they do not undergo rapid ripening change after maturity and they are sensitive to chilling injury. Fruits can be harvested 30 days after fruit set, but it is better to delay harvest, to perhaps as much as 50 days after fruit set so that more sweetness will be able to develop. With no noticeable change in sugar content, the fruit continues to grow in size until harvested. It was reported that fruits harvested 50 days after flowering are 50 percent heavier (average 17 oz) than at earlier harvests (30 days, average 12 oz) (Chang et al., 1997). Fruits can be stored in perforated plastic bags for 25–30 days at 40°F, but they will last less than 10 days at room temperature.

## 2.8 Health Benefits of Dragon Fruit

The content in the dragon fruit contributes to a lot of health benefits. One of them is the high amounts of unsaturated fats in relation to saturated fatty acids was associated with lower risk of coronary heart disease (Villalobos-Gutiérrez, 2012). Besides that, due to pitaya fruit’s high levels of linoleic acid, pitaya seed oil may help to get rid of rough skin and maintain the function of the body’s protective barrier (Affrin et al., 2009). The strong presence of antioxidants in pitaya may contribute by preventing diabetes and cancer, neutralize toxic substances such as heavy metals and reduce cholesterol and high blood pressure (Swarup et al., 2009). This is because pitaya has phenol, ascorbic acid, α-tocopherol, and anti-radical power components in it. The oligosaccharides of pitaya showed additional properties such as reduced caloric intake, constipation treatment and insulinaemia (Wichienchot et al., 2010).

Normally, dragon fruit has 1 g of fiber per 100 g serving. Fiber contributes health benefits such as lowering the risk of diabetes and heart disease. It also helps with weight management and in maintaining bowel integrity and health and is essential in lowering blood cholesterol levels and controlling blood sugar levels.

Dragon fruit is a rich source of antioxidants, which is important in fighting dangers from free radicals that occur naturally in the body. Free radicals has the tendency to damage cells and DNA and facilitate aging. Besides that, they also facilitate development of a number of health conditions, such as heart disease and cancer. Antioxidants can neutralize free radicals and reduce some of the damage they cause. The two types of antioxidants are primary, those produced by the body, and secondary, those found in foods, such as the dragon fruit.

Dragon fruit contains several important vitamins. It has Vitamin C, which helps improve the immune system and helps in healing cuts and bruises. It also contains many Vitamin B, usually Vitamin B1, which produces energy and metabolizes carbohydrates. Vitamin B2 helps in the normal thyroid function and metabolism and finally Vitamin B3 decreases bad cholesterol and helps rejuvenate the skin.Dragon fruit also contains B-sitosterol, which helps to reduce high blood pressure or hypertension.

Dragon fruit contains protein, which is essential to every cell in the body. Hair and nails mostly contains protein, and tissue requires protein for building and repair. Other than that, protein makes enzymes, hormones, bones, muscles, cartilage, skin and blood. Protein is a macro-nutrient, which means that the body requires a large amount because the body does not store it. Each 100 g serving of dragon fruit has 0.229 g of protein. Carotene on the other hand, prevents cancer and heart disease, boosts immunity and supports good vision, as mentioned in the UC-Berkeley Wellness Newsletter. Carotene is found in deeply colored fruits, such as dragon fruit. A 100 g serving of dragon fruit has 0.012 mg of carotene. (Greg, 2010)

## 2.9 Nutritional Information of Dragon fruit

The fleshy parts of raw pitaya consist of mostly water and carbohydrates, together with some protein and fat content. Pitayas contain slight amounts of iron, calcium phosphorus and other nutrients. The fatty acid compositions of two pitaya seed oils were determined as follows:

**Table 2.1:** Nutritional Information of Dragon Fruit

| | *Hylocereus polyrhizus* (Red-fleshed pitahaya) | *Hylocereus undatus* (White-fleshed pitahaya) |
|---|---|---|
| **Myristic acid** | 0.2% | 0.3% |
| **Palmitic acid** | 17.9% | 17.1% |
| **Stearic acid** | 5.49% | 4.37% |
| **Palmitoleic acid** | 0.91% | 0.61% |
| **Oleic acid** | 21.6% | 23.8% |
| **Cis-vaccenic acid** | 3.14% | 2.81% |
| **Linoleic acid** | 49.6% | 50.1% |
| **Linolenic acid** | 1.21% | 0.98% |

## 2.10 Betacyanin Pigment

Red-fleshed pitaya fruit is a potential fruit for betacyanins extraction. Nowadays, red beetroots (Beta vulgaris) are the main source of betacyanins which is now available in the concentrated and powder form. However, red beetroot contains geosmin and pyrazines that are responsible for the unpleasant peatiness of this crop as well as high nitrate concentrations associated with the formation of carcinogenic nitrosamines (Moβhammer et al., 2005). In contrast to red beetroot, red-fleshed pitaya fruit does not have this negative characterictics and it is not harmful. Therefore, betacyanins from red-fleshed pitaya fruit may be a potential source on top of red beetroots.

Pitaya fruit is a fast growing and developing fruit. Under the surrounding condition of Malaysia, the flesh of fruit turn from creamy white to full red-violet within 26-28 days after flower while peel took 1-2 days longer for the green colour changed to red. The fruit is ready for harvest once the peel has turned full red but not later than 35 days as fruit start to crack and split and thus the quality deteriorate. (Stintzing et al., 2002). In addition to that, there is little report on profiles and total contents of betacyanins in the peel and flesh as colour of fruit start turning into red. This information is useful in preparation for the emerging of peel and flesh pigment extraction industry. (Phebe et al., 2009)

Betalains (betacyanins) are a group of nitrogenous compounds responsible for the red and yellow colour of plants which belongs to the order Centrospermae. They are derivatives of betalamic acid. Betacyanins possess an indole-2-carboxylic acid moiety, which is N-linked to a pyridine dicarboxylic acid group, through an acetyl group (Daniel, 2006).

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Betacyanins usually exists as glycosides. More than 50 betacyanins are reported, most of which are glycosides of betanidin or isobetanidin. The red pigment Betanin is responsible for the colour in beetroots (Beta vulgaris). It is widely commercialised for colouring foodstuff, milk products and ice creams (Von et al., 1996). The red pigments from Phytolacca Americana and Amaranthus tricolor, have been legally used as food and wine colourant (Forni et al., 1983).

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## 2.11 Pectin

Pectin is a common name given to a class of polysaccharides of plant origin. They are found in the primary cell walls of all higher plants where they constitute approximately 22-35% of the dry mass. Pectic polysaccharides contribute to a number of important functions of cell walls, and of the plant. The pectic matrix resists compressive forces resting upon cell walls, determines porosity and contributes to ionic status. The pectic matrix also has important roles in the plant's defense mechanisms (Basic et al., 1988)

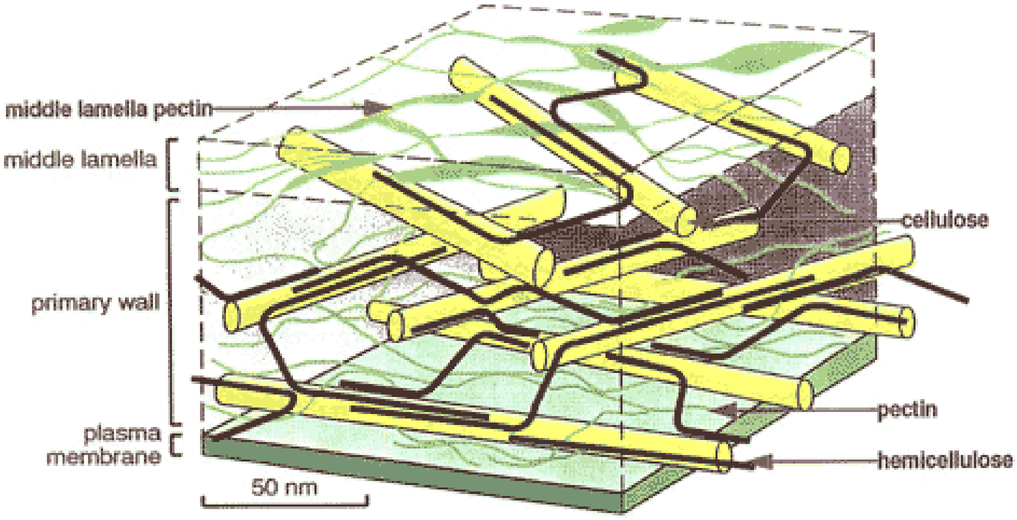


**Figure 2.6:** Pectin in cell wall

Approximately 5% of the pectin is cold water soluble, whereas 70% of the pectin is alcohol insoluble. Approximately 50% of the carboxyl groups of this fraction are methyl esterified. Hot water helps to solubilize a highly esperified pectin from the cell wall which represents 15% of the total amount of cell wall. In the remaining hot water insoluble pectin, only 30% of the pectic carboxyl groups are combined as esters.

The methyl ester group of pectin is supplied by the methyl group of methionine. Oat coleoptile section, form S-methylmethionine and methionine sulfoxide from methionine, whether they are intact or homogenates. Both of these compounds are also active as methyl donors for the formation of pectic esters. Indoleacetic acid increases both incorporation of the methyl of methionine into methyl ester moieties and the incorporation of glucose into galacturonic acid residues of water soluble pectins. This increase is a measure of an accelerated rate of pectin synthesis. Indoleacetic acid does not accelerate the rate of synthesis of the water insoluble pectins (Albersheim, 1959).

## 2.12 Chemical and Physical Properties of Pectin

The molecules of pectic substances are normally polymers of anhydrogalacturonic acid in the pyranose ring. These basic units are in the alpha form and are linked through carbons one and four in unbranched or also called as linear chains. The bond angle between the one and four carbons is actually such as to give the polymeric chain a screwlike configuration. Such polymers of anhydrogalacturonic acid are designated pectic acid to differentiate them from other forms of pectic substances. There is a possibility that pectic substances from certain plant sources have at least part of their galacturonic acid residues linked through carbons one and six, causing a branched molecule while the primary linkage of the molecule is through carbons one and four,. The number of anhydrogalacturonic acid residues, and consequently the chain length of the molecule are always different.

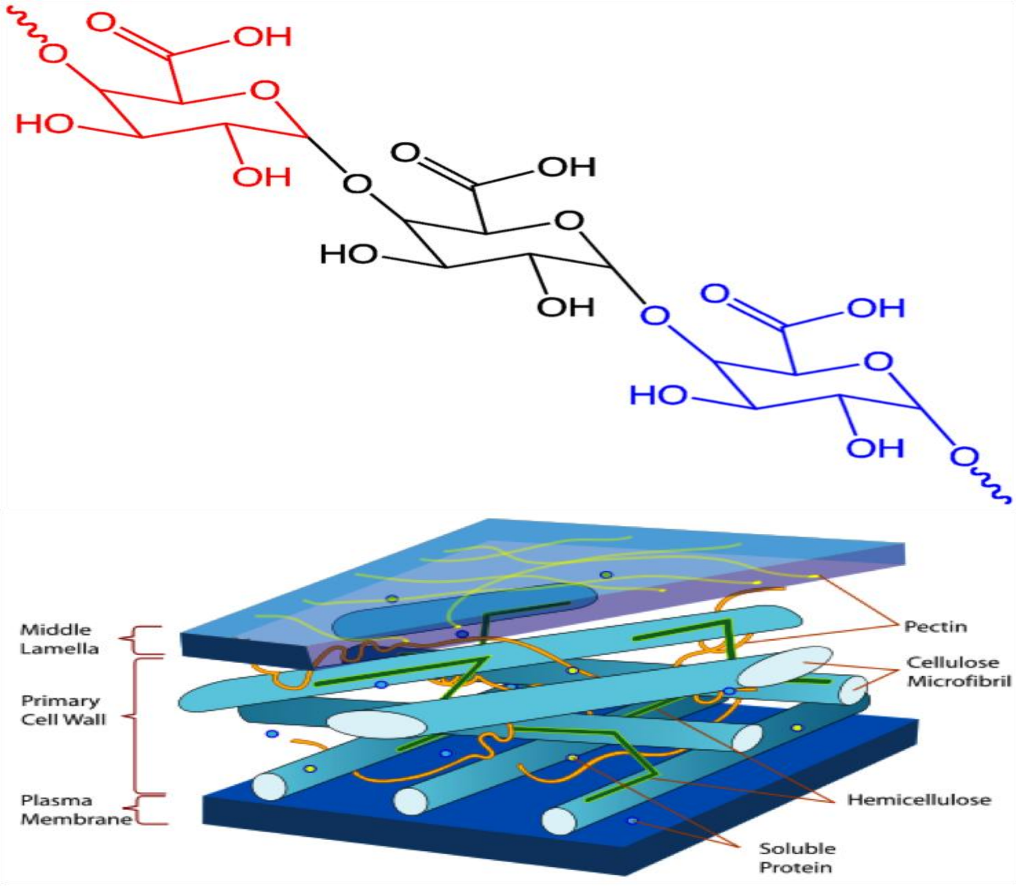


**Figure 2.7:** Molecular Structure of Pectin

Joseph and Havighorst, gave the molecular weight of pectin as 200, 000 to 300,000. Myers points out that the method used to determine the molecular weight affects the results. For example, end group analysis indicates a range of molecular weights from 2500 to 7500 while measurements of molecular weights by osmotic pressure indicates a range from 30,000 to 100, 000. In addition to being different greatly in size, the molecules of pectic substances vary also in the number of methyl groups which are esterified to the carboxyl groups along the chain. Such polymers of anhydrogalacturonic acid are called pectinic acid to distinguish them from the unmethylated polymers. Pectinic acids thus have fewer free carboxyl groups and a lower negative charge density than normal pectic acids. When over half of the carboxyl groups are esterified with methoxyl groups the resulting pectinic acids are designated as pectin. The methoxyl groups may be positioned in a regular or a random manner. Some of the properties exhibited by pectic substances are the result of the positioning of the methoxyl groups on the molecule as well as the number of methoxyl groups in the molecule.

The pectic substances are by definition colloidal in nature. The colloidal properties of the pectin molecule are due to the length of the polygalacturonic acid residue chains. There is no evidence as to the number of residues that is needed and must be linked to give polygalacturonic acid some colloidal properties. However, polymers of four units or less will probably be crystallized instead of colloidal. When pectic substances are subjected to hydrolysis there is an accompanying decrease in the colloidal properties.

Bonner mentioned that the colloidal characteristic of pectins is because they are hydrophilic colloids of high negative charge, a charge which varies with the number of free carboxyl groups. The viscosity of a pectinic acid solution decreases with progressive demethylation. It is now known that the viscosity is not exactly related to the methyl ester content but to the molecular size of the pectinic acid molecule. The viscosity of a pectinic acid solution is affected by the concentration of the pectin, changes in pH, changes in temperature of the solution (Bette, 1963).

## 2.13 Viscosity of Pectin

Pitaya peel contains considerable amount of pectin, betacyanin pigment and total dietary fibre. The peel had a good ratio of insoluble dietry fibre ratio to soluble dietry fibre ratio, IDF to SDF (3.8: 1.0). Hence, pitaya peel could be utilized as a good source of fibre, pectin and natural colorant (Jamilah et al., 2011). However, problem arises when the amount of pectin level is very high in the pitaya peel. This is because, high level of pectin causes high viscosity of the pitaya peel extract and this will cause the natural pitaya peel dye not to fast on fibre. Viscosity is a quantity that describes a fluid’s resistance to flow. Fluids resist the relative motion of immersed objects through them as well as to the motion of layers with differing velocities within them.

**Figure 2.8:** The concept of viscosity

## 2.14 Mordants

Since there is presence of pectin in the dye that will be extracted from pitaya peel, it is crucial to find out the proper way to ensure that the colour of the dye fasts on the fibre. One of the solutions is by mordanting the fibres so that the dye can be adsorp onto the fibre more efficiently.

The natural dyes having limited substantively for the fibre, require use of the mordant which enhances the fixation of the natural colorant on the fibre by the formation of the complex with the dye. Some of the important mordant used are alum, potassium dichromate, ferrous sulphate, copper sulphate, zinc sulphate, tannin and tannic acid. Although these metal mordants contribure to developing wide gamut of hues after complexing with the natural colouring compounds, most of these metals are toxic in nature and only in trace quantity their presence is found to be safe for the wearer.

The word mordant comes from the Latin word “mordere”, meaning “to bite”. A mordant is a chemical which can itself be fixed on the fibre and also forms a chemical bond with the natural colourants. It helps in absorption and fixation of natural dyes and also prevents bleeding and fading of colours i.e., improves the fastness properties of the dyed fibres. This complex may be formed by first applying the mordant and then dyeing (pre-mordanting process) or by simultaneous application of the dye and the mordant (meta-mordanting process) or by after treatment of the dyed material with the mordant (post-mordanting process).

## 2.15 Types of mordants

Mordants are important due to limitation on colour yield and poor fastness properties. Mordants will help to the increase natural dye uptake by textile fibres using certain chemicals. Different typesof mordants yield different colours even for the same natural dye. Therefore, final colour and colour fastness properties are not only dependant on the dye itself but are also determined by varying concentration and skillful manipulation of the mordants. Thus, a mordant is more important than the dye itself. The ideal type of mordant for bulk use should produce a great colour yield at low cost, without seriously affecting physical properties of fibre or fastness properties of the dyes. Also, the mordant should not cause any noxious effect during processing and the dyed textile material should not have any carcinogenic effect during use. Mordants can be classified into many different categories. They are as the following:

<u>Metallic mordants</u>

They are generally metal salts of aluminium, chromium, iron, copper and tin. The metallic mordants are of two types.

**a) Brightening mordants**

i. *Alum:* Among all types of alum, potash alum and aluminium sulphate is cheap, easily available and safe to use mordant. It usually produces pale versions of the prevailing dye colour in the plant.

ii. *Chrome (potassium dichromate):* It is also referred to as red chromate. It is relatively more expensive. However, Cr3+ or Cr6+ is considered to be harmful for human skin it contains heavy metal beyond a certain limit of its presence. Its use has been limited as per the norms of the eco-standards. The dichromate solution is light sensitive and therefore it changes colour under light exposure.

iii. *Tin (stannous chloride):* It gives brighter colours than any other mordant. However, they are oxidized on exposure to air and may impart a stiff hand to the fabric. Stannous chloride also causes higher loss of fabric tenacity (tensile strength) if applied beyond a certain concentrations.

**b) Dulling mordants**

i. *Copper (cupric sulphate):* Known as blue vitriol, it is readily soluble in water and easy to apply. It gives some special effects in shades, which otherwise cannot be obtained. However, copper beyond a certain limit is also under the eco-standard norms as heavy metals.

ii. *Iron (ferrous sulphate):* It is also known as green vitriol and is readily soluble in water. It is used for darkening /browning and blackening of the colours/ shades. It is easily available and one of the oldest mordants known. It is extensively used to get grey to black shades.

Tannins Mordant

The term ‘tanning agent’ is usually given to water-soluble cellulosic materials that predicates gelatin from solution. But all gelatin precipitation are not identified as tanning agent. Tannins are polyphenolic compounds having capacity of gelling under certain conditions. Among the tannins, myrobalan (harda) and galls/sumach are most important.

Oils type mordants

The example of oil type mordants are Vegetable oils or Turkey red oil (TRO). TRO as mordant is mainly used in the dyeing of deep red colour from madder. The main function of the TRO as oil mordant is to form a complex with alum when used as a main mordant. Sulphonated oil posses better binding-capacity than the natural oils. Oil mordanted samples exhibit superior fastness and hue.

In this research project, it has been decided that the mordant Aluminium Sulphate (Alum) will be used as a mordant to adsorp the dye from Pitaya peel onto the three different types of clothes. This is because Alum is easily available and it has no toxic properties, which makes it safe for customers to use the fabric. It is also the most common type of mordant used for dying process using natural dye.

## 2.16 Aluminium Sulphate (Alum)

Alum is not poisonous. However, it should be handled with care and not ingested. There are four different types of alum used in dying process They include aluminium sulphate, potassium aluminium sulphate (potash alum), ammonium aluminium sulphate (ammonium alum) and aluminium acetate. The different names refer to the different methods used in the processing of alum to refine it. In the early days of production, refining was necessary to make sure the alum was free from iron contamination and this was mainly done using either potassium or ammonia. Pure aluminium sulphate (without the potassium or ammonia) contains the highest proportion of aluminium of the 3 sulphates, but by only a small margin, and later in 19th century development, resulting from improved methods for removing the iron from alum shales. All the three aluminium sulphates mentioned above can be used for mordanting, although ammonium alum is less commonly used as a mordant. Aluminium acetate is used to mordant vegetable fibres and silk only.

The aluminium content of ammonium alum is slightly lower than that of the other two aluminium sulphates, but not sufficiently lower to make a significant difference. The difference between aluminium sulphate and aluminium acetate is that the former is processed using sulphuric acid and the latter using acetic acid. Alum acetate is usually used for mordanting vegetable fibres and silk only, not animal fibres such as wool, mohair etc. Aluminium sulphate is commonly used to dye most type of fabric, be it natural fibre and synthetic fibre.

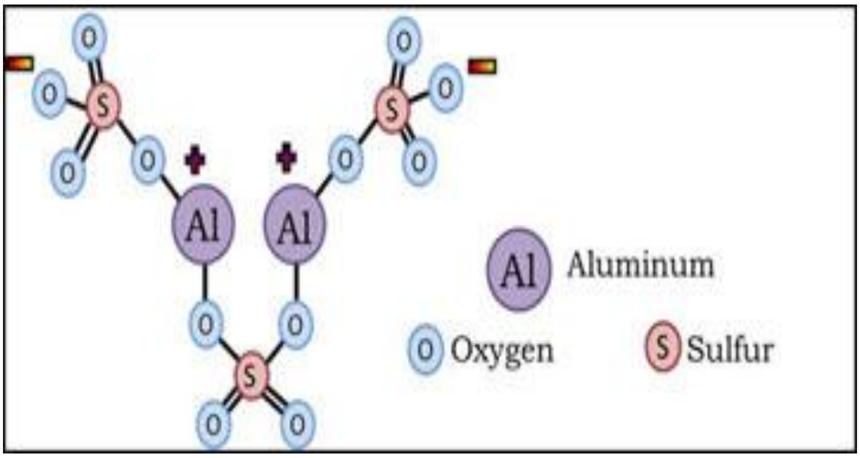


**Figure 2.9:** Structure of Aluminium Sulphate (Alum)

## 2.17 Cotton

Cotton is the collective name given to four species of plants in the genus *Gossypium*, *Gossypium hirsutum*,*Gossypium barbadense*, *Gossypium arboreum* and *Gossypium herbaceum* which are perennial shrubs in the family *Malvaceae*. They are grown for the fluffy fiber which protects the seeds of the plant. The plant species *G. hirsutum* comprises approximately 90% of world wide cotton production today (Rafiq, 1997). Generally, cotton trees have a main stem giving rise to several branches at the top. The leaves of the tree are spirally arranged on the branches, have long petioles and have 3–5 triangular lobes. The plant produces a single flower on each axillary branch which can be red-purple, yellow or white in color and forms a leathery, oval seed capsule, or 'boll' which is 2–6 cm long. Mature bolls usually split open to reveal the characteristic white cotton fibers and the seed. The cotton plant can reach heights of 1–1.5 m and is usually cultivated as an annual, surviving only one growing season. Cotton may also be referred to as tree cotton and its center of origin is unknown although the plant has diversified from Mexico, north-east Africa and Arabia and Australia (Franz et al., 2004).

**Figure 2.10:** Cotton Tree

Today, cotton is mainly used in the textile industry. According to Lawrence (1998), the fibers of the cotton plant are harvested and woven into fabric for the production of clothing, towels, bed sheets and many other textiles. Cotton fiber may also be used in the production of yarn and twine. The cotton seeds can be used to extract oil for use in the production of shortening or cooking oil and the manufacture of soaps and lubricants. The seed may be used as a feed for livestock. The fuzz produced as a byproduct of the ginning process can be used in the upholstery industry.

Cotton has many characteristics and properties. Some of them includes comfortable soft hand, good absorbency, resents colour, prints well, can be washed using machine, dry-cleanable, good strength, drapes well and easy to handle and sew (Tortora et al., 1997). Cotton has the ability to adsorp dye more effectively than the other type of fabrics.

**Figure 2.11:** Cotton Fabric

## 2.18 Chiffon

Chiffon is a textile made from cotton, silk, polyester, nylon or rayon. It is a combination of all these fabrics. It tends to be sheer, with a faint shimmer and a simple weave. This fabric strongly resembles closely woven netting is being held up to the light (Kadolph et al., 2007). Chiffon is generally used in evening and formal wear, since it drapes well and it is able to add a floaty look to gowns and dresses. Chiffon is a difficult fabric to work with, because of its slippery texture. Most fabric stores carry several forms in varying colors. The name comes from an Old French word for rag (Mary, 2013)

Silk chiffon is quite expensive, since it has a rich shimmer and slick texture. It is also quite strong for its weight. Because silk chiffon is made from a natural fiber, it tends to be more expensive than synthetics, and it also needs to be dry cleaned. Silk is also used to make crepe, a thicker and more opaque fabric that drapes and behaves like chiffon. Another natural fiber, cotton, is sometimes used to make chiffon, although the resulting fabric is more matte and less floaty (Mary, 2013).

Synthetic materials are most commonly used to make chiffon, since most synthetics take dye well, are quite cheap to make, and they tend to be sturdy. However, the fabric is still very delicate and therefore it should be hand washed with cold water only, and avoid running through a washing machine. Synthetics are also just as challenging to work with as silk, since they are also slippery (Kadolph et al., 2007). Although chiffon has the tendency to adsorp dye, it is not as effective as natural dye.

In formal wear, chiffon is often used as an overlay over more opaque fabrics. The fabric floats on top, adding shimmer and texture to the dress while keeping the wearer modest. The colour of the underlying fabric will also show slightly, so the chiffon is usually coordinated with the underlayer.

When sewing chiffon, many crafters layer tissue paper in between the two pieces being sewn together. The tissue paper helps keep the fabric together, with the rough surface of the tissue holding the chiffon in place while it is handled. After sewing, the tissue paper can be carefully taken out. Chiffon is also pinnable, as it will spring back, concealing pin marks. As a general rule, sewers should work slowly and steadily with this fabric, taking care not to run it through a sewing machine too quickly or it will bunch and gather (Mary, 2013). The texture of chiffon is such that it has large pores in between the fibres. The fibres are arranged far from each other.

**Figure 2.12:** Chiffon fabric

### 2.19 Polyester

Polyester is a term usually defined as “long-chain polymers chemically composed of at least 85% by weight of an ester and a dihydric alcohol and a terephthalic acid”. It also means the linking of several esters within the fibers. Reaction of alcohol with carboxylic acid results in the formation of esters (Kadolph et al., 1998).

Polyester can also be classified as saturated and unsaturated polyesters. Saturated polyesters refer to that family of polyesters in which the polyester backbones are saturated. They are thus not as reactive as unsaturated polyesters. Unsaturated polyesters refer to that family of polyesters in which the backbone consists of alkyl thermosetting resins characterized by vinyl unsaturation. They are mostly used in reinforced plastics (Geno, 2006). These are the most widely used and economical family of resins.

Chemists can now alter the size and shape of polyester fibers to look and feel more like natural fibers. Ultra thin microfibers can give polyester a smoother, softer feel than the polyester of twenty years ago. Polyester fabric also has the following characteristics. Polyester fabrics and fibers are extremely strong besides being very durable: resistant to most chemicals, stretching and shrinking, wrinkle resistant, mildew and abrasion resistant. Polyester is also hydrophobic in nature and quick drying. It can be used for insulation by manufacturing hollow fibers. Polyester retains its shape and hence is good for making outdoor clothing for harsh climates. Polyester is also easily washed and dried (Rosato et al., 2004). The mentioned characteristics were the common characterics of polyester.

The most popular and one of the earliest uses of polyester was to make polyester suits – all the rage in the 70s. Polyester clothes were very popular. Due to its strength and tenacity polyester was also used to make ropes in industries (Kadolph et al., 1998). PET bottles are today one of the most popular uses of polyester.

Taking care of polyester clothing is very easy and less time consuming. Polyester clothing can be machine washed and dried after that. Adding a fabric softener also generally helps. The fabric can be dried at low temperatures to get maximum usage from the clothing. Polyester does not require much ironing. If you need to, then iron warm (Geno, 2006). Polyester can be dry-cleaned with no hassles.

Polyester can also be easily dyed. However, since it is a synthetic fibre, it is not as efficient in adsorbing dyes as in the natural fibre family.

**Figure 2.13:** Polyester Fabric

## 2.20 UV VIS Spectroscopy

The equipment that will be used for the qualitative analysis in this research is UV-Vis Spectroscopy.

**Figure 2.14:** UV-Vis Spectrophotometer

Ultraviolet and visible spectrometers have been in general use for the last 35 years and over this period have become the most important analytical instrument in the modern day laboratory. In many applications other techniques could be employed but none rival UV-Visible spectrometry for its simplicity, versatility, speed, accuracy and cost-effectiveness (Skoog et al., 2007). In this research, UV- Vis spectrophotometer will be used to determine the amount of natural pitaya dye adsorbed onto 3 different types of fibre. The figure below illustrates how UV-Vis spectrophotometer works.

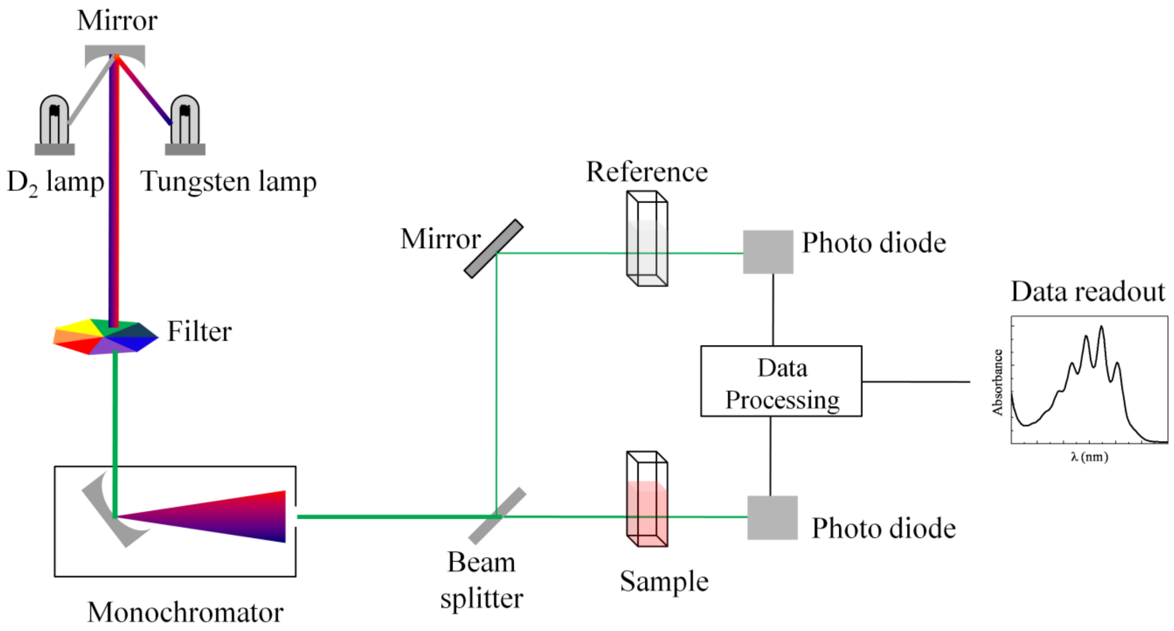
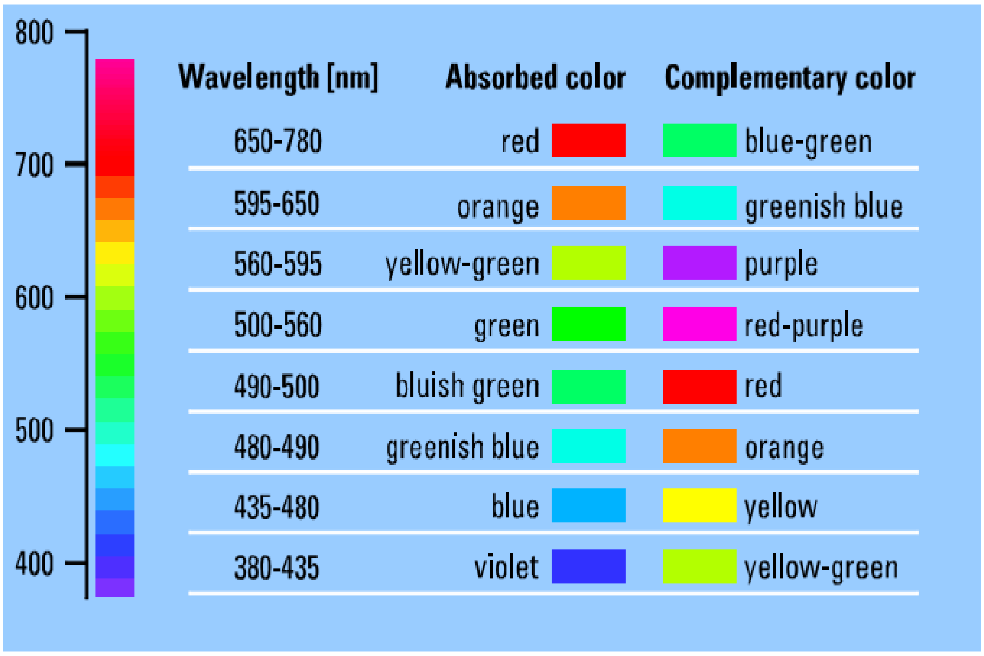


**Figure 2.15:** Schematic diagram on how UV-Vis Spectrophotometer works

When white light falls upon a sample, the light may be reflected totally, which means that the substance appears white or the light may be totally absorbed, in which case the substance will appear black. However, if only a portion of the light is absorbed and the balance is reflected, the colour of the sample is determined by the reflected light. Therefore, if violet is absorbed, the sample appears yellow-green and if yellow is absorbed, the sample appears blue. The colours are therefore described as complementary. However, many substances which appear colourless do have absorption spectra. In this instance, the absorption will take place in the infra-red or ultraviolet and not in the visible region (Hirayama, 1967). The figure below illustrates the relationship between light absorption and color.



| Wavelength [nm] | Absorbed color | Complementary color |
|---|---|---|
| 650-780 | red | blue-green |
| 595-650 | orange | greenish blue |
| 560-595 | yellow-green | purple |
| 500-560 | green | red-purple |
| 490-500 | bluish green | red |
| 480-490 | greenish blue | orange |
| 435-480 | blue | yellow |
| 380-435 | violet | yellow-green |

**Figure 2.16:** Relationship between light absorption and colour

In this research, the wavelength that is used to examine the adsorption of dye onto the cloth is 560nm. It falls at the range where red-purple colour is observed but green colour is absorbed.

A significant relationship exists between the colour of a substance and its electronic structure. A molecule or ion will exhibit absorption in the visible or ultraviolet region when radiation causes an electronic transition within its structure. Therefore, the absorption of light by a sample in the ultraviolet or visible region is accompanied by a change in the electronic state of the molecules in the sample (Hirayama, 1967). The energy supplied by the light will promote electrons from their ground state orbitals to higher energy which is the excited state orbitals.

The greater the number of molecules that absorb light of a given wavelength, the greater the extent of light absorption and higher the peak intensity in absorption spectrum (Friedel et al., 1951). This is why when the concentration of the sample of dye is high, the resulting adsorption will also be high. If there are only a few molecules that absorb radiation, the total absorption of energy is less and consequently lower intensity peak is observed. This makes the basis of Beer-Lambert Law which states that the fraction of incident radiation absorbed is proportional to the number of absorbing molecules in its path (Sadtler, 1971).

When the radiation passes through a solution, the amount of light absorbed or transmitted is an exponential function of the molecular concentration of the solute and also a function of length of the path of radiation through the sample (Skoog et al., 2007). Therefore,

$$\text{Log } I_o / I = \varepsilon \, c \, l$$

Where,
$I_o$ = Intensity of the incident light
$I$ = Intensity of light transmitted through the sample solution
$c$ = concentration of the solute in mol $l^{-1}$
$l$ = path length of the sample in cm
$\varepsilon$ = molar absorptivity or the molar extinction coefficient of the substance whose light absorption is under investigation. It is a constant and is a characteristic of a given absorbing species (molecule or ion) in a particular solvent at a particular wavelength. $\varepsilon$ is numerically equal to the absorbance of a solution of unit molar concentration ($c = 1$) in a cell of unit length ( $l = 1$) and its units are liters.moles-1 . cm-1. However, it is a practice among organic chemists to omit the units (Sadtler, 1979).

The ratio $I / I_o$ is known as transmittance T and the logarithm of the inverse ratio $I_o / I$ is known as the absorbance A. Therefore,

$$- \text{Log } I / I_o = - \log T = \varepsilon \, c \, l$$
$$\text{and Log } I_o / I = A = \varepsilon \, c \, l$$
$$\text{or } A = \varepsilon \, c \, l$$

For presenting the absorption characteristics of a spectrum, the positions of peaks are reported as $\lambda$max (in nm) values and the absorptivity is expressed in parenthesis (Sadtler, 1979).

## 2.21 Adsorption Process

Adsorption is a process which is widely used in the removal of contaminants from wastewaters. The design and efficient operation of adsorption processes require equilibrium adsorption data for use in kinetic and mass transfer models. These models can then be used to predict the performance of the adsorption contact processes under a range of operating conditions (Allen et al., 2002). The equilibrium adsorption data usually were described by means of Langmuir or Freundlich equation. It could be used for comparison of adsorption behaviour for tested adsorbents or for various experimental conditions (Klimiuk et al., 2006). The following are the literature reviews about Langmuir and Freundlich isotherm and list of adsorbent and adsorbate involved in each journals.

From the Tables 2.2, 2.3 and 2.4 displayed below for the different types of adsorption isotherm equations, we can see that Langmuir isotherm is one of the most widely used equation to determine the trend of an adsorption system and the maximum adsorption that occurs when the surface is covered with a monolayer adsorbate. Freundlich isotherm is famous for heterogeneous surface energy which is applicable for this research.

The concept of adsorption process is well established according to these previous studies of adsorption process. In this literature review, there are about 13 journals that studies about comparison of Freundlich and Langmuir isotherm, 3 journals that study only about Langmuir isotherm and 1 journal that study about Freundlich isotherm.

Among these papers, there are no studies on cloth as adsorbent and natural dye as adsorbate. Most of the journals only studied about synthetic dye. All these journals also only showed on how to remove dyes from the solute. None of them discussed about adsorption of dye on solid material. The reason why I choose comparison between Langmuir and Freundlich isotherm is to be able to compare which isotherm is suitable for my research. Besides that, both these isotherms are common adsorption method and easy to be understood.

**Table 2.2:** Freundlich and Langmuir Isotherm

| Title and Process | Authors |
|---|---|
| Freundlich and Langmuir adsorption isotherms and kinetics for the removal of Tartrazine from aqueous solutions using Hen Feathers<br>**Adsorbent: Hen feathers**<br>**Adsorbate: Tartrazine** | Mittal et al. (2006) |
| Adsorption isotherm models for basic dye adsorption by peat in single and binary component systems<br>**Adsorbent: Moss Peat**<br>**Adsorbate: Basic red 22, basic blue 3, basic yellow 21** | Allen et al. (2004) |
| Anionic dye removal from aqueous solutions using modified zeolite: Adsorption Kinetics and Isotherm studies<br>**Adsorbent: Zeolite(minerals of Heulandite type)**<br>**Adsorbate: Azo dyes(RR-239 and RB-250)** | Alver et al. (2012) |
| Equilibrium and kinetics studies for adsorption of direct blue 71 aqueous solution by wheat shells<br>**Adsorbent: Wheat Shells**<br>**Adsorbate: Direct Blue 71** | Bulut et al. (2006) |
| Comparison of optimised isotherm models for basic dye adsorption by kudzu<br>**Adsorbent: Dried Kudzu**<br>**Adsorbate: Basic Yellow 21, Basic Red 22** | Allen et al. (2002) |
| Equilibrium and kinetic modeling of adsorption of reactive dye on cross-linked chitosan beads<br>**Adsorbent: Chitosan Beads**<br>**Adsorbate: Reactive Red 189 Dye** | Chiou et al. (2002) |
| Sorption of dye from aqueous solution by peat<br>**Adsorbent: Peat**<br>**Adsorbate: Basic Blue 69, Acid Blue 25** | Ho et al. (1998) |
| Equilibrium Studies for Acid Dye Adsorption onto Chitosan<br>**Adsorbent: Chitosan**<br>**Adsorbate: Acid Green 25, Acid** | Wong et al. (2003) |

| | |
|---|---|
| **Orange 10, Acid Orange 12, Acid Red 18, Acid Red 73** | |
| The Effectiveness of Surfactants Adsorption onto Chitin and Dye-Modified Chitin<br>**Adsorbent: Chitin**<br>**Adsorbate: anionic (Borutosol KRN, Siarczanol N-2) and nonionic (Rokafenol N-8) surfactants** | Klimuik et al. (2005) |
| Adsorption from Aqueous Solution onto Natural and Acid Activated Bentonite<br>**Adsorbent: Acid Activated Bentonite**<br>**Adsorbate: Methylene Blue** | Laila et al. (2012) |
| Adsorption of Dye by Using the Solid Waste from Leather Industry as an Adsorbent<br>**Adsorbent: Leather Waste**<br>**Adsorbate: Acid Red 131 Dye** | Jitendra et al. (2013) |
| A comparative evaluation for adsorption of dye on Neem Bark and Mango Bark Powder<br>**Adsorbent: Neem Bark and Mango Bark**<br>**Adsorbate: Malachite Green** | Ruchi et al. (2010) |
| Batch Equilibrium Adsorption of Reactive Dye onto Natural Biopolymer<br>**Adsorbent: Chitosan**<br>**Adsorbate: Verofix Red** | Gurusamy et al. (1997) |

**Table 2.3:** Langmuir Isotherm

| Title and Process | Authors |
|---|---|
| Langmuir Isotherm Models applied to the Multicomponent Sorption of Acid Dyes from Effluent onto Activated Carbon<br>**Adsorbent: Activated Carbon T ype F400**<br>**Adsorbate: Acid Red 114(AR114), Acid Blue 80(AB80), Acid Yellow(AY117)** | Keith et al. (2000) |
| Kinetic models for the sorption of dye from aqueous solution by wood<br>**Adsorbent: Wood Sawdust**<br>**Adsorbate: Basic Blue 69, Acid Blue 25** | Ho et al. (1998) |
| Decolorization of a textile vat dye by adsorption on waste ash<br>**Adsorbent: Ash from burning brown coal**<br>**Adsorbate: Vat dye Ostanthren Blue GCD (C.I. Vat Blue 14)** | Miodrag et al. (2010) |

**Table 2.4:** Freundlich Isotherm

| Title and Process | Authors |
|---|---|
| Kinetic and Isotherm modelling of adsorption of dyes onto Rusk Husk Carbon<br>**Adsorbent: Rusk Husk Carbon**<br>**Adsorbate: Crystal Violet, Direct Orange, Magenta dye** | Verma et al. (2010) |

## 2.22 Langmuir Isotherm

Langmuir isotherm described by the adsorbate-adsorbent systems in which the extent of adsorbate coverage is dependent on one molecular layer at or before a relative pressure of unity is reached. Although the isotherm is usually used for the description of chemisorption, the equation also obeyed at moderately low coverages by a number of systems, and can be used to describe the behaviour of binary adsorbate systems. The equation for Langmuir Isotherm is as follows: (Ghosh, 2006)

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max}K_L C_e} \tag{2.1}$$

Where,

$q_e$ = Dye bound per unit amount of adsorbent (mg/g)

$C_e$ = Unbound dye concentration (in solution) (mg/L)

$q_{max}$ = saturation constant (mg/g)

$K_L$ = Affinity constant for Langmuir isotherm (L/mg)

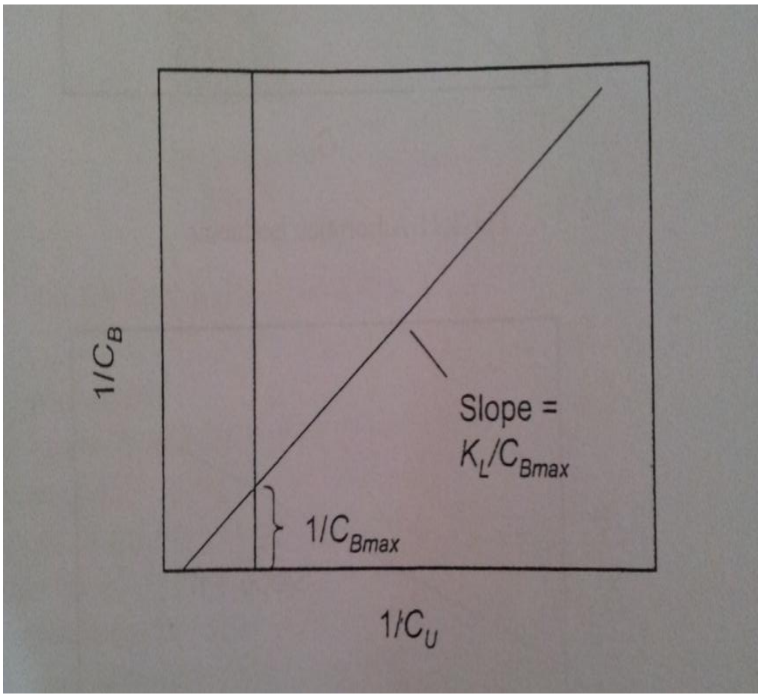


**Figure 2.17:** Determination of Langmuir Isotherm constants

## 2.23 Freundlich Isotherm

Freundlich isotherm is also an adsorption isotherm, in which there is a curve relating the concentration of a solute on the surface of adsorbent to the concentration of solute which is present in liquid in which it is in contact. Freundlich isotherm of adsorption is famous for heterogeneous surface energy which is applicable for this research. The equation formulated is as follows: (Ghosh, 2006)

$$\log q_e = logK_f + \frac{1}{n} logC_e \tag{2.2}$$

Where,

$q_e$ = Solute bound per unit amount of adsorbent (mg/g)

$C_e$ = Unbound solute concentration (in solution) (mg/L)

$K_f$ = Freundlich adsorption constant

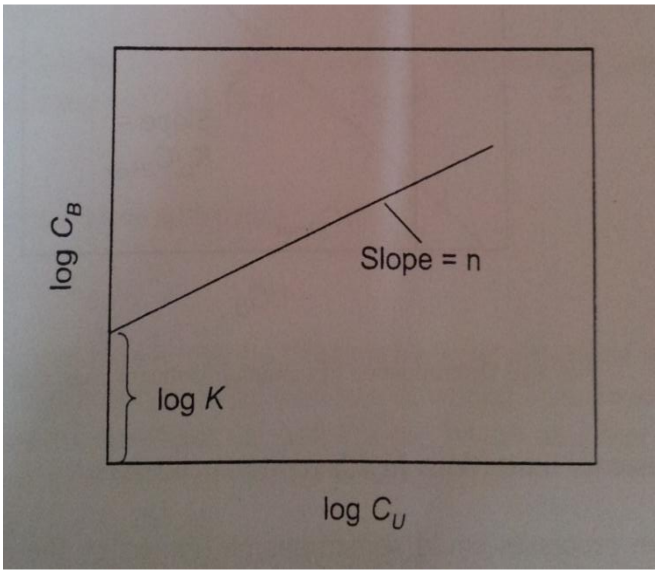


**Figure 2.18:** Determination of Freundlich Isotherm constants

## 2.24 Fourier Transform Infrared

FT-IR stands for Fourier Transform Infrared. This is the most preferred method of infrared spectroscopy. In infrared spectroscopy, infrared radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is transmitted through the sample. The resulting spectrum indicates the molecular absorption and transmission, creating a molecular fingerprint of the sample. No two unique molecular structures produce the same infrared spectrum like a fingerprint. This makes infrared spectroscopy very useful for several types of analysis (Nikolet, 2001).

FTIR helps to provide us with the following informations:

- It can identify unknown materials
- It can determine the quality or consistency of a sample
- It can determine the amount of components in a mixture

Similar to SEM, the fabrics will also undergo FTIR testing before the fabric is being pretreated using mordant, after pretreatment using mordant and after dyeing.

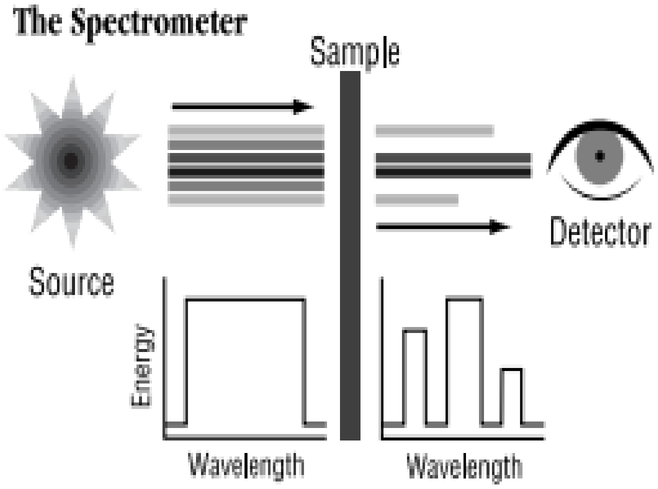


**Figure 2.19:** Schematic Diagram for FTIR

## 2.25 Scanning Electron Microscope

SEM stands for scanning electron microscope. The scanning electron microscope is a microscope that uses electrons instead of light to form an image. Since their development in the early 1950's, scanning electron microscopes have developed new areas of study in the medical and physical science communities. The scanning electron microcope has allowed researchers to examine a much bigger variety of specimens. By using a focussed beam of electrons, the SEM reveals the details and complexity inaccessible by light microscopy. SEM can magnify an object from about 10 times up to 300,000 times (McMullen, 2006). A scale bar is often provided on an SEM image. From this the actual size of structures in the image can be calculated.

The scanning electron microscope has many advantages over normal traditional microscopes. The SEM has a vast field, which allows more of a specimen to be in focus at one time. The SEM also has quite a high resolution, so closely spaced specimens can be magnified at much higher levels (Smith et al., 1955). The researcher has much more control in the degree of magnification because the SEM uses electromagnets rather than lenses. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

The surface morphology of fibres will be examined using SEM in this research. The surface morphology includes before pretreatment using mordant, after pretreatment using mordant and after dyeing.

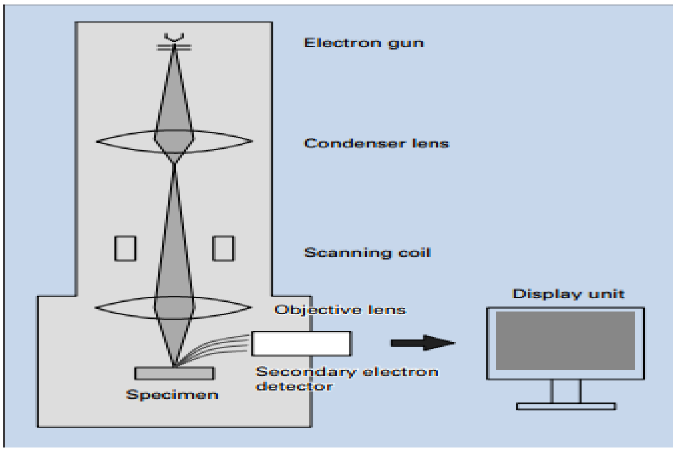


**Figure 2.20:** Basic Construction of SEM

## 2.26 Desorption

Desorption is a process whereby a substance is released from or through a surface. This process is the opposite of sorption which is either adsorption or absorption. This occurs in a system being in the state of sorption equilibrium between bulk phase (fluid, i.e. gas or liquid solution) and an adsorbing surface which in most cases is solid. When the concentration of substance in the bulk phase is lowered, some of the sorbed substance changes to the bulk state.

After adsorption, the adsorbed chemical will remain on the substrate nearly, provided the temperature remains low. However, if the temperature rises, so does the likelihood of desorption occurring (Somorjai et al., 2010).

In this research, after conducting all the experiments of adsorption process, there will also be a simple procedure to determine whether there is desorption of the dye occurring after the drying process.

## 2.27 Conclusion

Based on the above literature review, most of the findings are only related to adsorption process of synthetic dyes as adsorbate onto many different adsorbent such as hen feathers, moss peat, dried kudzu and many more. The adsorption process of natural dyes onto adsorbents is not available probably because no one has ever carried out such research. Through this research, we can actually identify the efficiency of adsorption of natural dye onto adsorbent which is fibre for this case. This research may become the source for many more upcoming researches on the adsorption of different natural dyes on adsorbent.

# CHAPTER 3

# METHODOLOGY

## 3.1 Introduction

This chapter will discuss the method used for the dye extraction from pitaya waste, the process of mordanting of the fabric, and the process of dye adsorption onto the fabric.

## 3.2 Material Description

The Pitaya fruits were obtained from a fruit stall in Taman Tas. The raw material was peeled to get its waste, which was then blended and stored at -20$^{o}$C not more than 2 days (48 hours) so that blend material is kept fresh and to avoid the colour from fading because of oxidation process. For adsorption process, a mordant which is aluminium sulphate (Alum) was bought from Sigma Aldrich as well. The fibre namely cotton, chiffon and polyester were bought from Kamdar. The fibres were soaked in the mordant so that the adsorption process on the fibre will be smoother later on.

## 3.3 Processes

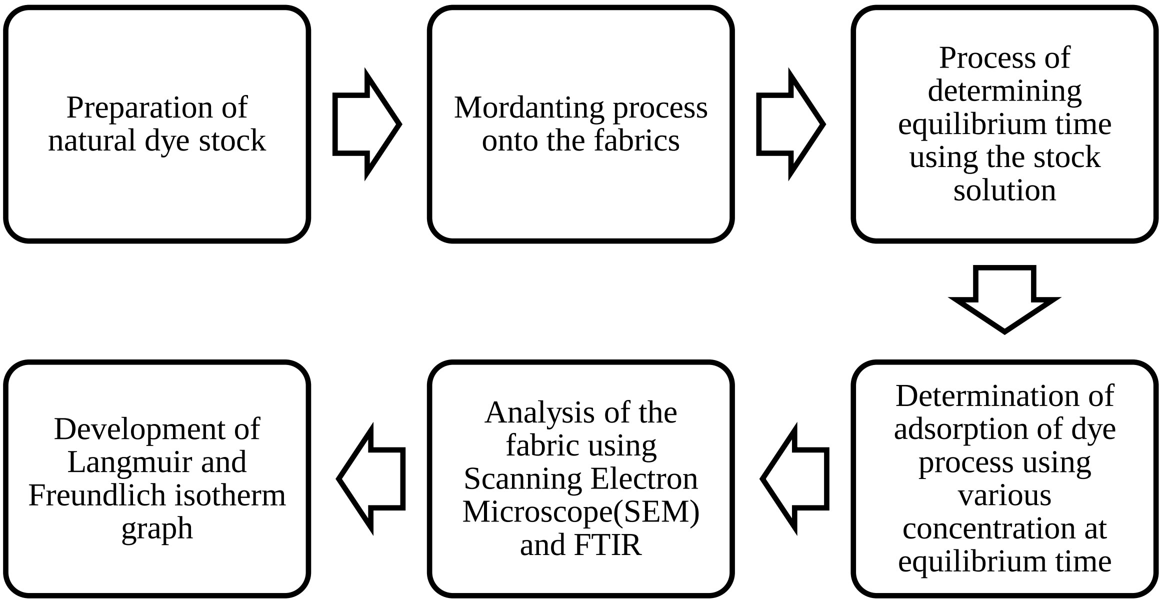


**Figure 3.1:** Block Diagram showing the whole process of adsorption of dye onto the fibre and the analysis after that

## 3.4 Preparation of Natural Sample Dye

Natural dye was prepared by using water extraction. The purpose of using water extraction is to make sure that the colour is safe from the chemical and harmful substance. Pitaya peel was first cleaned and the unnecessary part of the pitaya peel was cut. The pitaya peel was then grinded into small pieces, about 1mm, to increase the surface area. Then, the natural dye was extracted by applying ratio 1:3 corresponding to the ratio of 1 g of raw material to 3 g of water. The mixture was then centrifuged at 4 $^{o}$C, 10000rpm for 15 minutes (Mohana, 2012). The mixture was filtered to collect the supernatant dye. The mixture was centrifuged for the second time at 4 $^{o}$C , 12000rpm for 10 minutes to ensure that there is no more sediment or precipitate present in the dye solution. Finally, filtration of the supernatant was done by using stainless steel filter fabric (0.3 mm mesh) (Bechtold et al., 2003). The resulting extract was used for the further experiment. Figures 3.3, 3.4, 3.5 and 3.6 shows the processes of obtaining the pitaya peel extract in the lab.

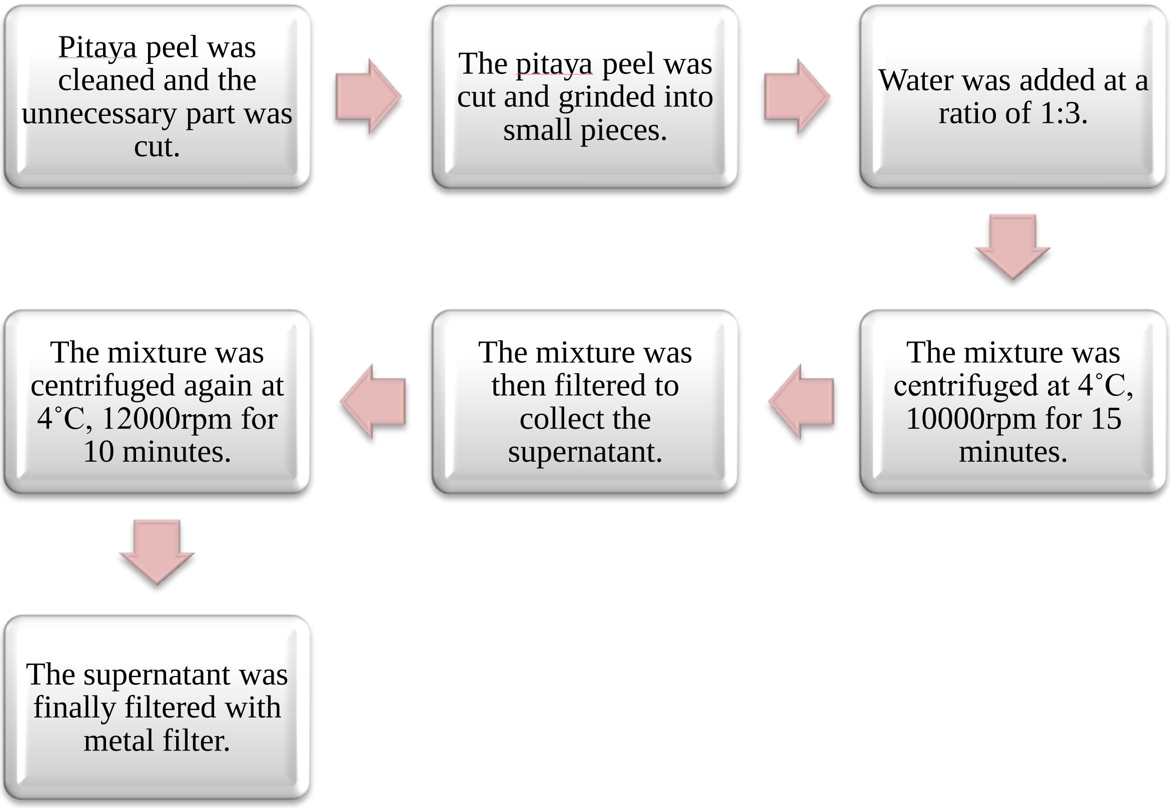


**Figure 3.2:** Process of Natural Dye Preparation

**Figure 3.3:** The peel of Pitaya Fruit

**Figure 3.4:** The pitaya peel being blend into small pieces

**Figure 3.5:** The mixture of Pitaya peel with water being prepared to be centrifuged

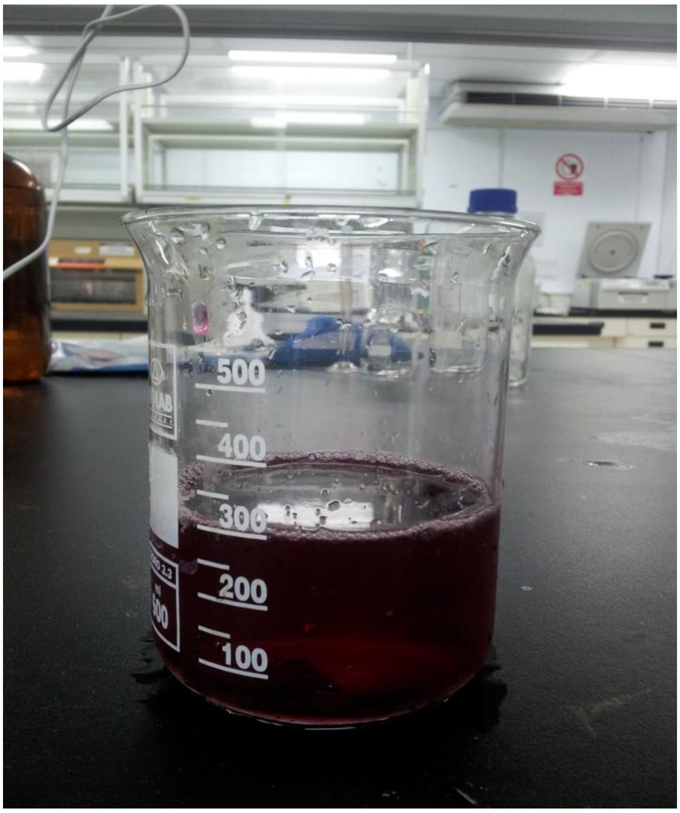


**Figure 3.6:** Pitaya extract after centrifugation process

## 3.5 Process of Mordanting

The three types of fibres that are used in this experiment (cotton, chiffon, polyester) are first subjected to thorough washing with detergent and then rinsed with distilled water. They were then soaked in a 30% hydrogen peroxide solution to remove unwanted dirt on the cloth. After that, they were soaked in 25% aluminium sulphate (Alum) solution which acts as a mordant. The mordant solution is heated up to 82˚C and then left to cool for 45 minutes. The fibres were then removed from the mordant solution and rinsed well with water. (Dean, 1994)

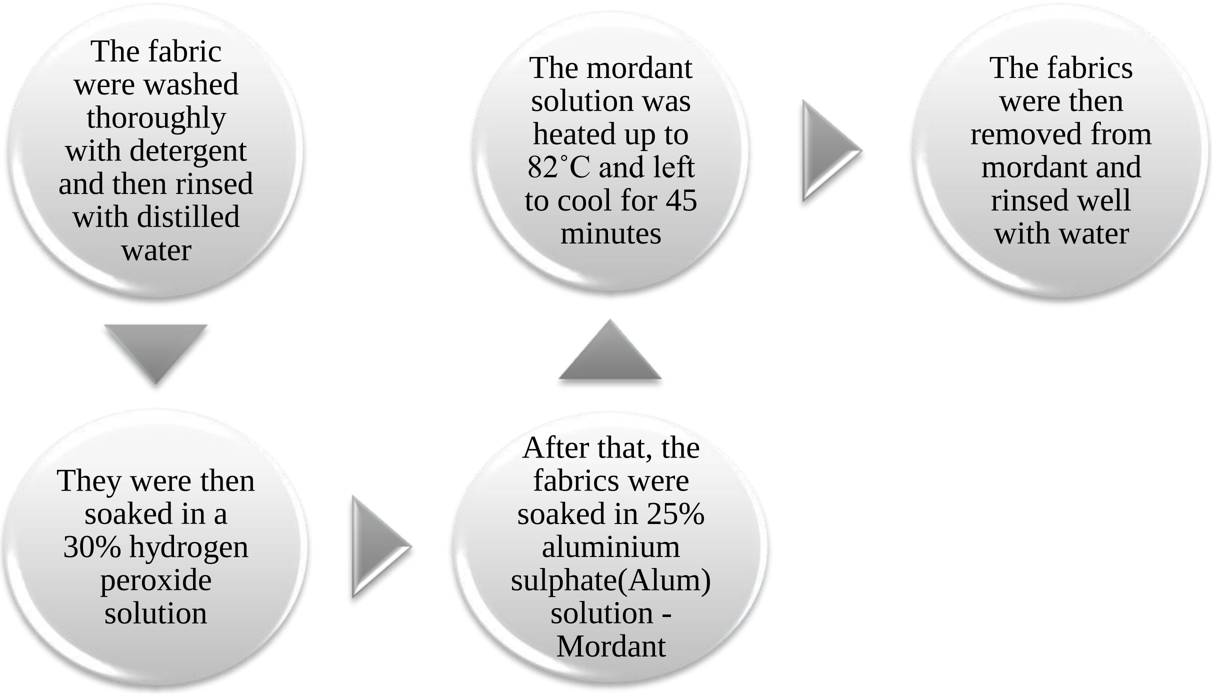


**Figure 3.7:** Process of Mordanting

**Figure 3.8:** The fibres being soaked in the mordant solution

**Figure 3.9:** The mordant solution being heated up to 82˚C using water bath

## 3.6 Equilibrium Time

The experiment to determine the time required for the adsorption to reach equilibrium can be performed by batch technique in room temperature (25°C). The test tubes were filled with 30 ml of the stock dye solution and the 3 different types of fabrics namely cotton, chiffon and polyester will be put into the test tube and then agitated in incubator shaker KS4000i at 100rpm at room temperature. The liquid samples will be taken out at 15 minutes interval for 120 minutes and the concentration of dye adsorbed are analyzed using UV-Vis Spectrophotometer. (Azlina et al., 2010)

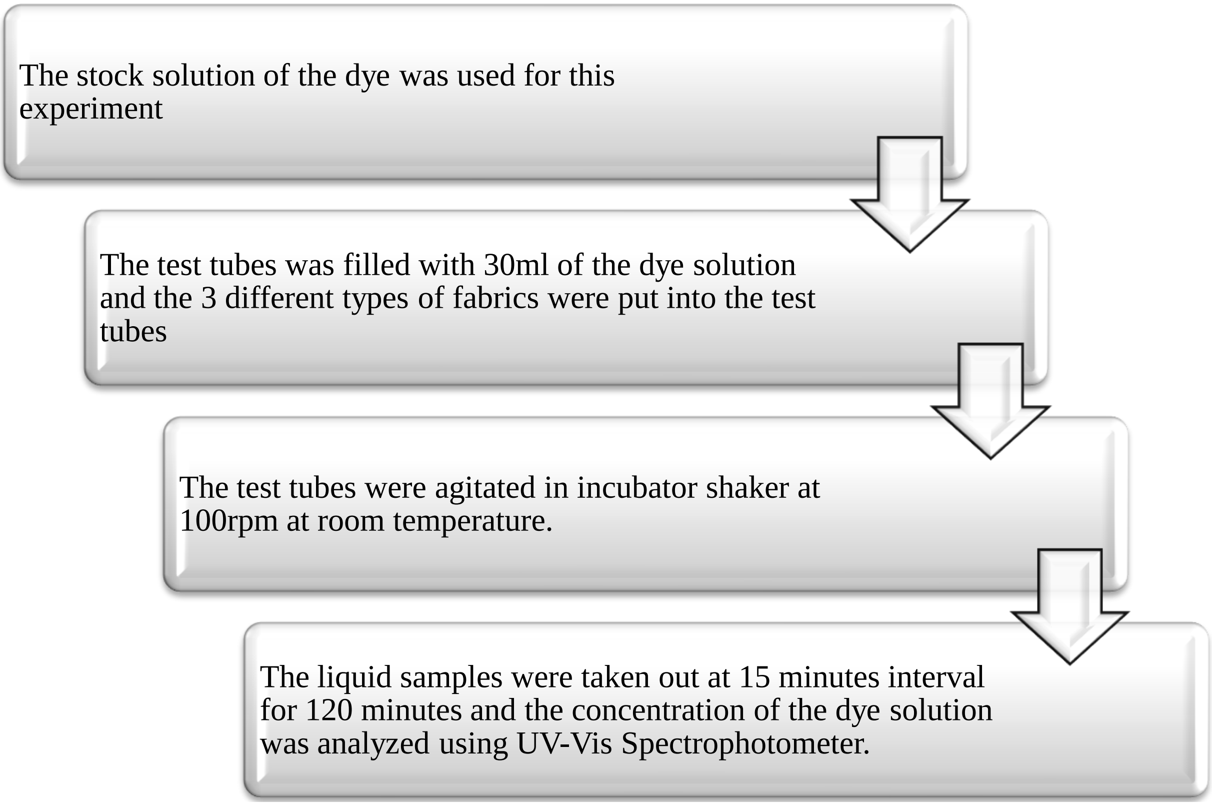


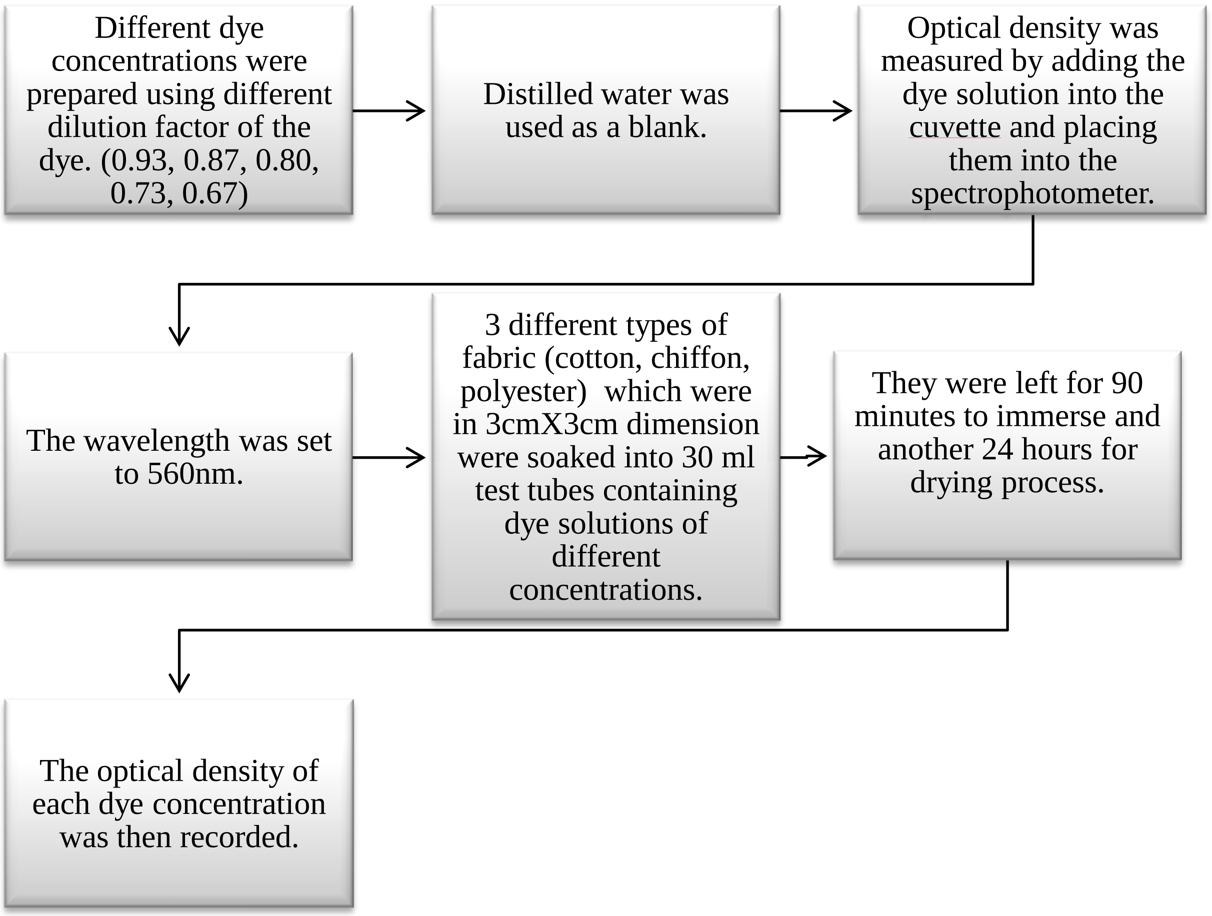
**Figure 3.10:** Equilibrium time process

## 3.7 Process of Adsorption

Different dye concentration solutions are prepared using different dilution factor of the dye. The dilution factors are 0.93, 0.87, 0.80, 0.73 and 0.67 respectively. Distilled water is used as a blank in this analysis. The optical density of the solution is then measured by adding the dye solution into the cuvette using micropipette and then placing the cuvette into the spectrophotometer. The wavelength is set to 560nm. The optical density for each dye concentrations is then recorded. Three different types of fabric, namely, cotton, silk and chiffon of 3cm X 3 cm dimension were soaked into a 30 ml test tubes containing dye solution of different concentrations. They are left for 90 minutes to immense as that is the equilibrium time for dye solution and another 24 hours for drying process.

**Table 3.1:** Water and Dye ratio for Dilution Factor

| **Dilution Factor** | **Water (mL)** | **Dye (mL)** |
|---|---|---|
| **0.93** | 2 | 28 |
| **0.87** | 4 | 26 |
| **0.80** | 6 | 24 |
| **0.73** | 8 | 22 |
| **0.67** | 10 | 20 |



Different dye concentrations were prepared using different dilution factor of the dye. (0.93, 0.87, 0.80, 0.73, 0.67)

Distilled water was used as a blank.

Optical density was measured by adding the dye solution into the cuvette and placing them into the spectrophotometer.

The wavelength was set to 560nm.

3 different types of fabric (cotton, chiffon, polyester) which were in 3cmX3cm dimension were soaked into 30 ml test tubes containing dye solutions of different concentrations.

They were left for 90 minutes to immerse and another 24 hours for drying process.

The optical density of each dye concentration was then recorded.

**Figure 3.11:** Process of adsorption using various dye concentrations

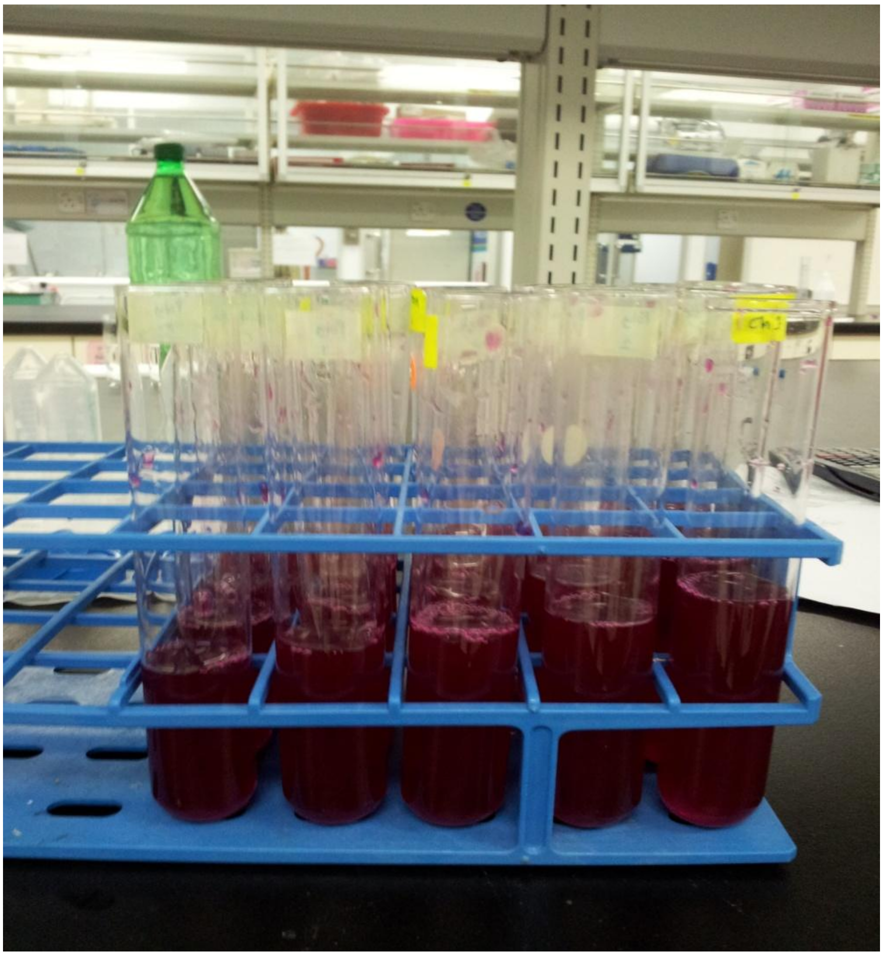


**Figure 3.12:** Different natural dye concentrations are being prepared for adsorption process

### 3.8 Analysis of Adsorption by UV-Vis Spectrometer

After 90 minutes, the fabric is removed from the test tubes and then the dye solution remaining in the test tubes will undergo the same process of determining the optical density of the solution using spectrophotometer. After obtaining the optical density readings before and after the adsorption process, the values has to be introduced to the equation mentioned below to calculate the total betacyanin content from the pitaya peel that has been adsorbed by the fabrics (Jamilah et al., 2011).

$$BC\ (mg\ L^{-1}) = \frac{A \times MW \times 1000 \times DF}{\in \times l} \tag{3.1}$$

Where:

A = Absorbance

DF = Dilution factor

MW = Molecular weight of betacyanin ($550 gmol^{-1}$)

Є = Molar extinction coefficient (60,000 L/mol cm in $H_2O$)

l = Path length of cuvette (1cm)

The difference in betacyanin content can be determined using the following formula:

BC(before adsorption) – BC(after adsorption) = Betacyanin content on fibre

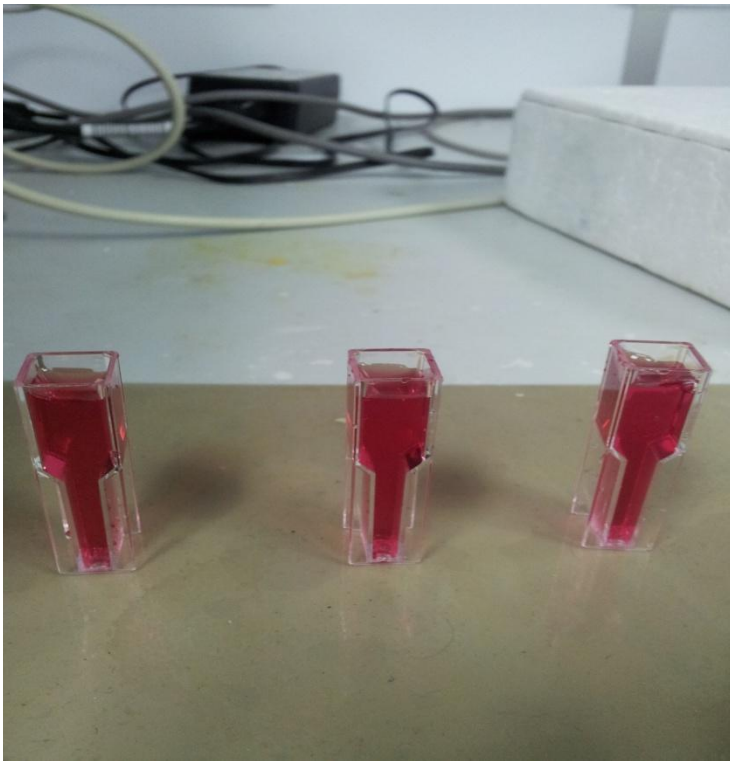


**Figure 3.13:** Samples being prepared for UV-Vis analysis

## 3.9 Amount of dye adsorbed

The amount of dye adsorbed can be calculated using the equation that follows; (Chiou et al., 2002)

$$q_e = \frac{(C_o - C_e)V}{W} \tag{3.2}$$

Where:

$C_o$ = Initial concentration of solution (g/m$^3$)

$C_e$ = Equilibrium concentration of solution (g/m$^3$)

V = Volume of solution (m$^3$)

W = Weight of fibre used (kg)

## 3.10 Langmuir Isotherm

Langmuir isotherm is one of the most widely used equation to determine the behaviour of an adsorption system and the maximum adsorption that occurs when the surface is covered with a monolayer adsorbate. The equation formulated is as follows (Ghosh, 2006):

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max} K_L C_e} \tag{3.4}$$

Where,

$q_e$ = Dye bound per unit amount of adsorbent (mg/g)

$C_e$ = Unbound dye concentration (in solution) (mg/L)

$q_{max}$ = saturation constant (mg/g)

$K_L$ = Affinity constant for Langmuir isotherm (L/mg)

## 3.11 Freundlich Isotherm

Freundlich isotherm of adsorption is famous for heterogeneous surface energy which is applicable for this research. The equation formulated is as follows:

$$\log q_e = log K_f + \frac{1}{n} log C_e \qquad \text{eq. (3.5)}$$

Where,

$q_e$ = Solute bound per unit amount of adsorbent (mg/g)

$C_e$ = Unbound solute concentration (in solution) (mg/L)

$K_f$ = Freundlich adsorption constant

## 3.12 Fourier Transform Infrared Spectroscopy (FTIR)

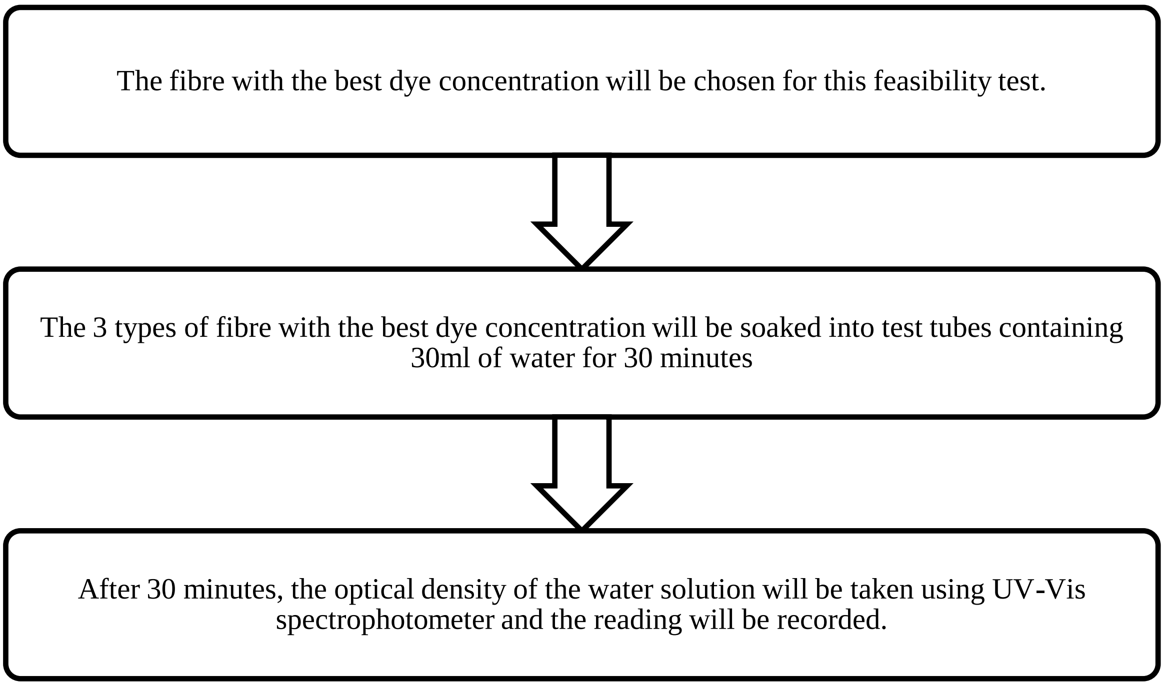
Fourier Transform Infrared Spectroscopy (FTIR) is used to determine the functional group of the fibres. The fibres chosen for this analysis are the fibres that adsorb the most dye at the best dilution factor. The best dilution factor of untreated, treated and dyed fibres (cotton, chiffon and polyester) are sent for this analysis.

## 3.13 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) is used to determine the surface morphology of the fibre at desired magnification. Similar to FTIR, the fibres chosen for this analysis are the fibres that adsorb the most dye at the best dilution factor. The best dilution factor of untreated, treated and dyed fibres (cotton, chiffon and polyester) are sent for this analysis.

## 3.14 Desorption Process

To ensure the feasibility of the dye adsorption experiment, it is necessary to determine whether the dye solution on the fibre undergoes desorption when soaked in the water. Therefore, the fibre with the best concentration of dye that is adsorbed onto the fibre will undergo this feasibility test. The 3 type fibres with the best dye concentration were soaked in 30ml of water in test tubes and after 30minutes, the optical density of the water solution is taken and the reading is recorded.



The fibre with the best dye concentration will be chosen for this feasibility test.

The 3 types of fibre with the best dye concentration will be soaked into test tubes containing 30ml of water for 30 minutes

After 30 minutes, the optical density of the water solution will be taken using UV-Vis spectrophotometer and the reading will be recorded.

**Figure 3.14:** Desorption Process

### 3.15 Statistical Analysis

Analysis of variance (ANOVA) was performed by using the Data Analysis tools in Microsoft Excel 2010, and a least significant difference (LSD) test was used to compare the means with a confidence interval of 95%.

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 Introduction

This chapter will discuss the results obtained from (a) fabric characterization, (b) adsorption of pitaya dye on fabric, (c) equilibrium time of process of dyeing, (d) Langmuir isotherm, (e) Freundlich isotherm, (f) FTIR analysis, (g) SEM analysis and (h) desorption process.

## 4.2 Equilibrium Time

After 15 minutes interval, the fabric is removed from the beaker and then the dye solution remaining in the beaker undergo the process of determining the optical density of the solution using spectrophotometer. After obtaining the optical density readings before and after the adsorption process, the values was put into the equation 4.1 mentioned below to calculate the total betacyanin content from the pitaya peel that has been adsorbed by the fabrics (Jamilah et al., 2011).

$$BC\ (mg\ L^{-1}) = \frac{A \times MW \times 1000 \times DF}{\epsilon \times l} \tag{4.1}$$

Where:

A = Absorbance

DF = Dilution factor (1.0)

MW = Molecular weight of betacyanin (550gmol$^{-1}$)

Є = Molar extinction coefficient (60,000 L/mol cm in $H_2O$)

l = Path length of cuvette (1cm)

The difference in betacyanin content can be determined using the following formula:

$$\text{BC(before adsorption)} - \text{BC(after adsorption)} = \text{Betacyanin content on fibre} \tag{4.2}$$

The dilution factor is 1.0 for equilibrium time because the standard stock dye is used. There is no dilution being done to the extracted dye solution. Below is the absorbance reading obtained for two trials. The average will be taken to calculate the betacyanin content on fibre.

The value from Table 4.1 is inserted into the equation 4.1 and 4.2 to determine the betacyanin content available in the dye solution. The betacyanin content that is adsorbed onto the 3 types of fibre, namely cotton, chiffon and polyester can be determined by subtracting the betacyanin content in the dye solution before the adsorption process to the betacyanin content in the dye solution after the adsorption process at different time inteval. The graph on betacyanin content on the fibre is then plotted to analyse the trend of the adsorption on different types of fibre at different time interval.

**Average Reading**

**Initial Absorbance Reading: 2.3565**

**Table 4.1:** Average Absorbance Reading at 15 minutes interval

| Minute | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **15** | 2.326 | 2.341 | 2.336 |
| **30** | 2.298 | 2.321 | 2.316 |
| **45** | 2.287 | 2.315 | 2.305 |
| **60** | 2.275 | 2.299 | 2.290 |
| **75** | 2.262 | 2.293 | 2.288 |
| **90** | 2.256 | 2.288 | 2.283 |
| **105** | 2.257 | 2.288 | 2.283 |
| **120** | 2.257 | 2.287 | 2.284 |

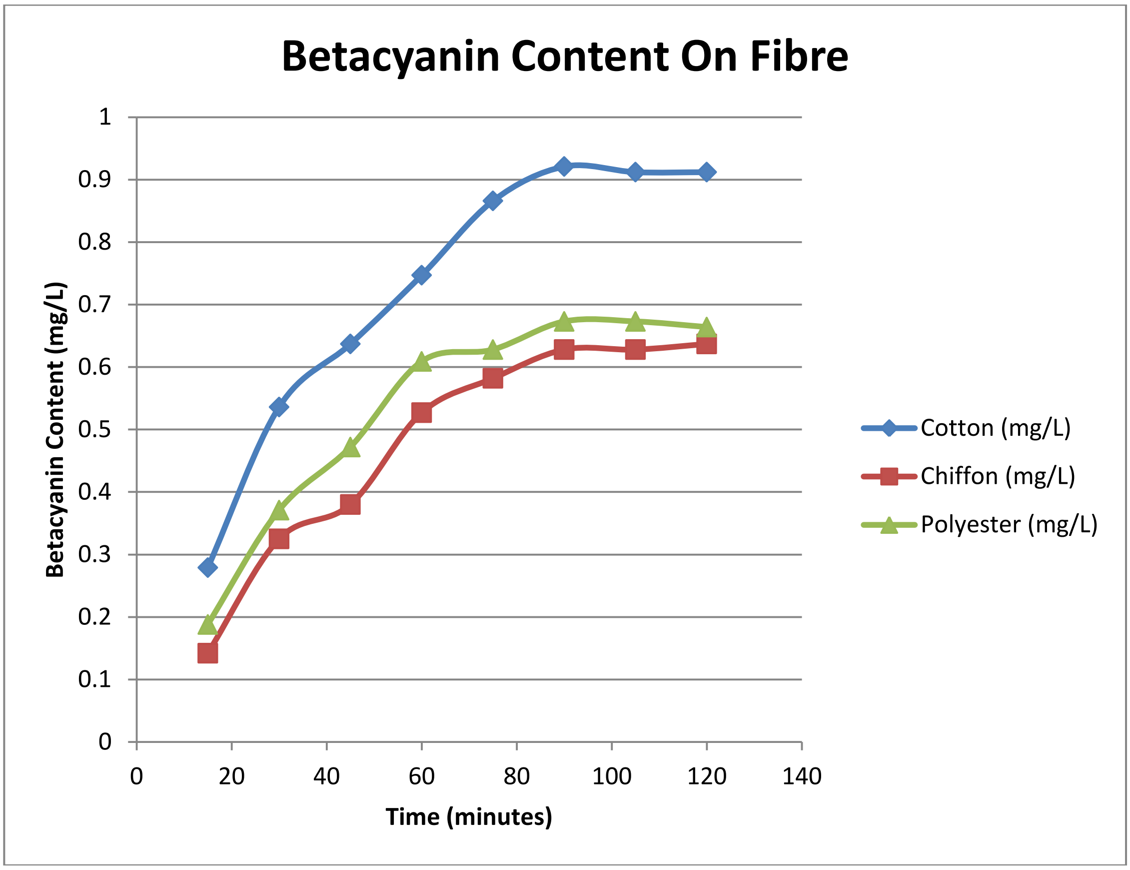


**Figure 4.1:** Betacyanin Content on Fibre at Various Time Intervals

The graph on betacyanin content on fibre at various time intervals is shown in Figure 4.1. According to the graph, we can see that the amount of dye being adsorbed increased drastically until about 90 minutes. After 90 minutes, the betacyanin content on the fibres has started to be constant. This shows that the equilibrium time for the betacyanin dye adsorption is achieved at 90 minutes.

From the trend of the graph, it is also noticed that the betacyanin content is the highest in cotton, followed by polyester and then chiffon. The trend is such because cotton has the highest strength to adsorb the colour pigment. According to Emily (2009), natural fibre has the tendency to adsorb dye more effectively than the synthetic fibres. Since cotton is a natural fibre, it would bond the best with the dye overall. There is not much difference in the strength of adsorption of the natural dye in polyester and chiffon. Polyester however has better adsorption strength since there is presence of more betacyanin pigment in it. Therefore it can adsorb colour better than chiffon.

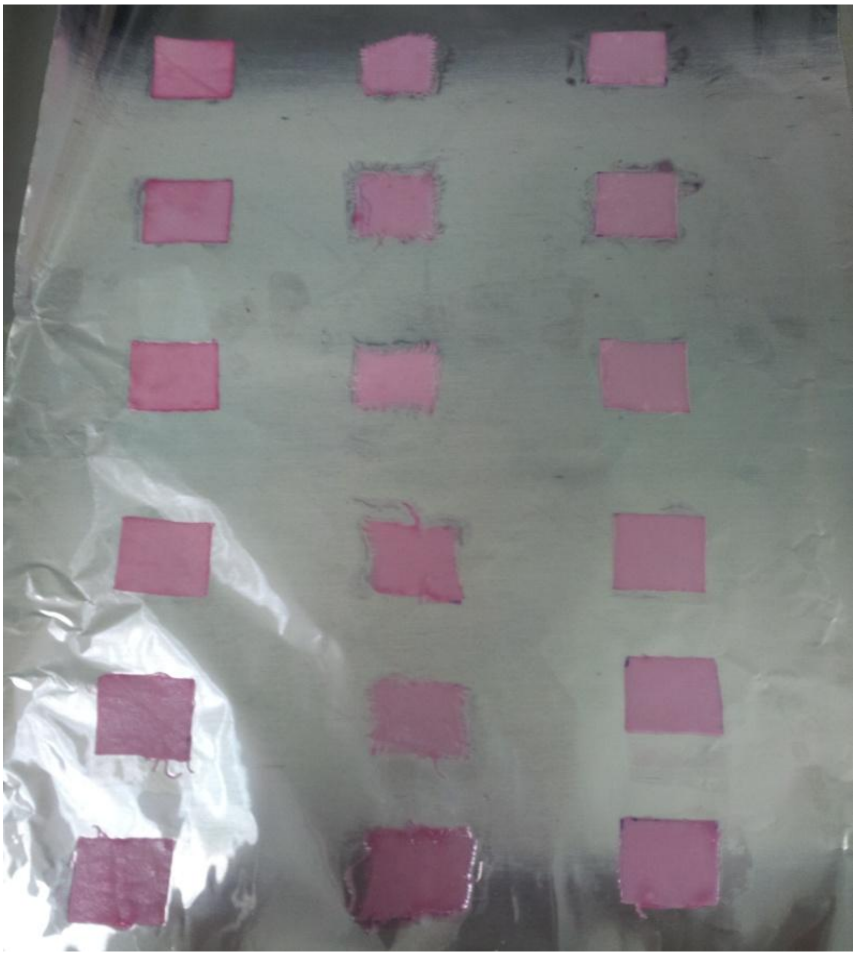


**Figure 4.2:** Fabric sample while they were left for drying

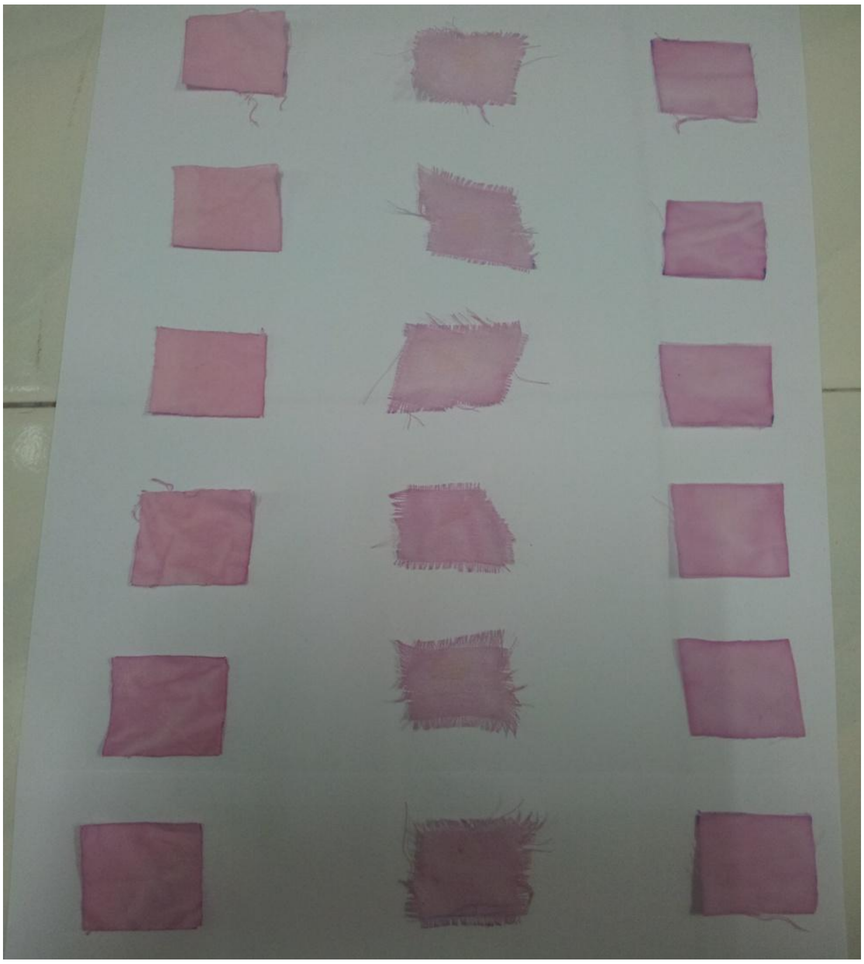


**Figure 4.3:** Fabric sample after they were dried

Figure 4.2 and Figure 4.3 the fabric samples of cotton, chiffon and polyester before and after being dried after the dyeing process. The fibres were removed from the dye solution after every 15 minutes and left for drying while the optical density of the dye solution is taken to identify the betacyanin content left in the solution.

From the figures, the intensity of the colour seems to be increasing as the fabric is left in the dye solution for longer period of time. This means that the fabric is able to adsorb more betacyanin pigment when the time of contact between the fabric and dye is increased. However, dyeing process using natural can be very sensitive as it is sensitive to heat, light exposition and also oxygen (Teixeira et al., 2007). Therefore, careful precautions must be taken throughout the dyeing process. The pitaya dye can change colour if the temperature exceeds 30˚C based on observation.

The exposure of the natural dye to light must be minimized throughout the dyeing process. Exposure to light can also cause the colour of the natural dye to fade away. Therefore, throughout the dyeing process, the test tubes are covered with aluminium foil to prevent light from penetrating into the dye solution.

## 4.3 Amount of Dye Adsorbed

The amount of dye adsorbed onto the fabric in equilibrium can be calculated using the following formula (Aboul-Fetouh et al., 2010):-

$$q_e = \frac{(C_o - C_e)V}{W} \tag{4.3}$$

Where:

$C_o$ = Initial concentration of solution (mg/L)

$C_e$ = Equilibrium concentration of solution (mg/L)

V = Volume of solution (L)

W = Weight of adsorbent cotton, chiffon and polyester (g)

Since the adsorption reached equilibrium on the 90th minute, the concentration of dye in the solution at 90minute will be used as $C_e$ for all the three different types of fabric. The volume of solution (V) is 0.03L as the experiment is done using test tubes containing 30ml of dye solution. The weight cotton, chiffon and polyester fibre used in 30ml of the dye solution is 0.1170g, 0.1005g and 0.0468 respectively. The calculated amounts of dye adsorbed are tabulated as follows:

**Table 4.2:** Amount of Dye adsorbed at various time intervals

| Minute | Cotton (mg/g) | Chiffon (mg/g) | Polyester (mg/g) |
|---|---|---|---|
| **15** | 0.0715 | 0.0910 | 0.0561 |
| **30** | 0.1374 | 0.2083 | 0.1107 |
| **45** | 0.1633 | 0.2436 | 0.1409 |
| **60** | 0.1915 | 0.3378 | 0.1818 |
| **75** | 0.2221 | 0.3731 | 0.1875 |
| **90** | 0.2362 | 0.4026 | 0.2009 |
| **105** | 0.2338 | 0.4026 | 0.2009 |
| **120** | 0.2338 | 0.4083 | 0.1982 |

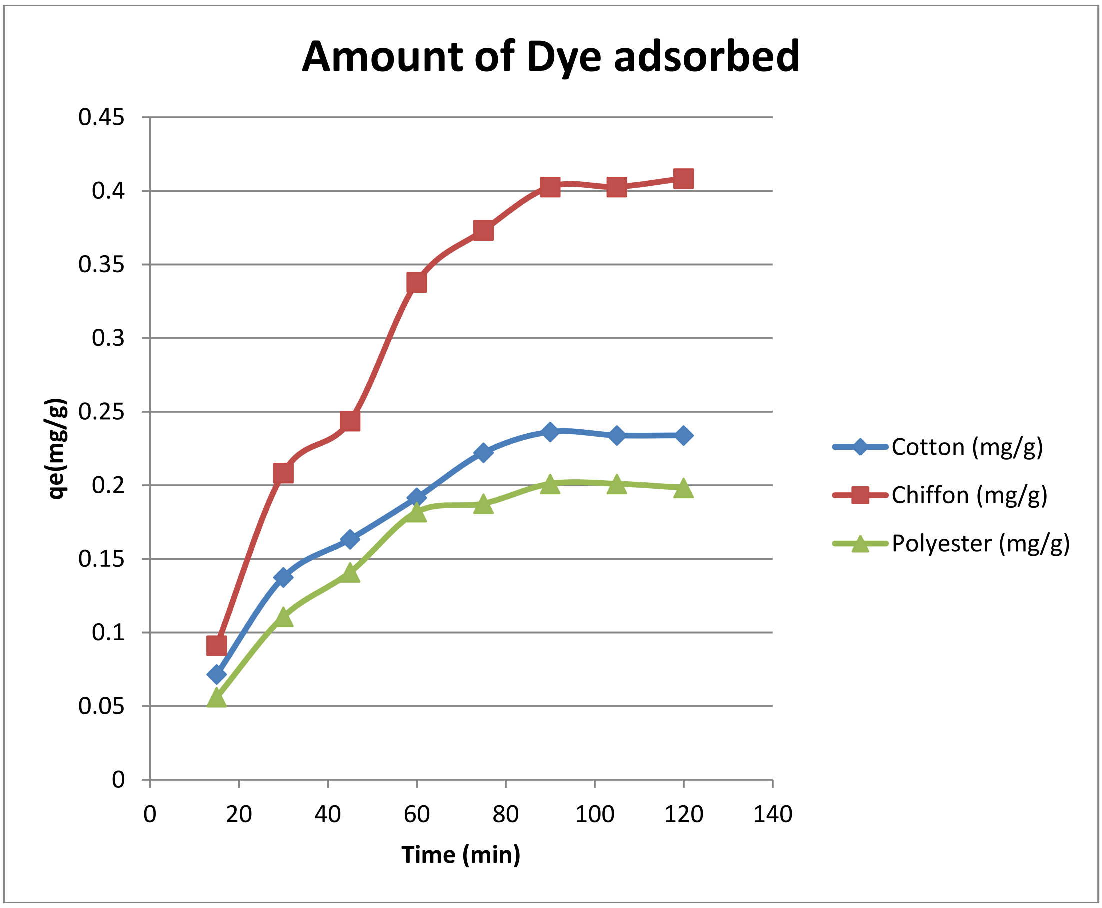


**Figure 4.4:** Amount of Dye adsorbed at different time intervals

From Figure 4.4, we can determine the exact amount of dye that had been adsorbed onto cotton, chiffon and polyester fabrics. The trend of the data above is similar to the betacyanin content on the fibre data earlier. This means that, we can also see that equilibrium time is reached at the minute 90 for dye adsorption using pitaya waste. Since the equilibrium is reached at 90 minutes, the experiment on the adsorption of dye at different concentration ratios will be done for 90 minutes as well.

Besides that, it is also noticed that chiffon has the highest amount of dye adsorbed, followed by cotton and then polyester. This is rather alarming because cotton theoretically has highest adsorption tendency as it is a natural fibre (Emily, 2009). When further analysis was done, it is determined that the reason why the amount of dye adsorbed in chiffon is higher compared to cotton is because the weight of 3cm X 3cm dimension of chiffon is much lower than cotton. Therefore, even though the betacyanin pigment content is higher in cotton fibre than chiffon fibre, the amount of dye adsorbed in cotton is lower than chiffon as the weight of chiffon is taken into consideration. Chiffon is light-weighed with a large pores compared to cotton which have tiny pores. The dye must have been stuck in between the large pores of the chiffon fabric when the reading is taken. However, after the drying process, cotton still has the highest colour intensity of the dye, followed by polyester and then chiffon.

## 4.4 Adsorption Process at Various Concentrations of Dye

The process of adsorption at various concentration of betacyanin is done on the fibres to determine the concentration of betacyanin dye with the best adsorption result. The initial and final average absorbance reading at various dilution factors are tabulated in Table 4.3 and Table 4.4 respectively.

**Table 4.3:** Average Initial Absorbance Reading at various dilution factors

| Dilution Factor | Initial Reading |
|---|---|
| **0.93** | 2.572 |
| **0.87** | 2.470 |
| **0.80** | 2.428 |
| **0.73** | 2.332 |
| **0.67** | 2.300 |

**Table 4.4:** Average Absorbance Reading after 90 minutes at various dilution factors

| Dilution Factor | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **0.93** | 2.342 | 2.457 | 2.410 |
| **0.87** | 2.272 | 2.355 | 2.326 |
| **0.80** | 2.263 | 2.363 | 2.300 |
| **0.73** | 2.190 | 2.277 | 2.237 |
| **0.67** | 2.185 | 2.261 | 2.218 |

The values from Table 4.3 and Table 4.4 are inserted into equation 4.1 and 4.2 to determine the betacyanin content available in the dye solution. The betacyanin content that is adsorbed onto the 3 types of fibre, namely cotton, chiffon and polyester can be determined by subtracting the betacyanin content in the dye solution before the adsorption process to the betacyanin content in the dye solution after the adsorption process at various dilution factors. The graph on betacyanin content on the fibre is then plotted to analyse the trend of the adsorption on different types of fibre at different dilution factors.

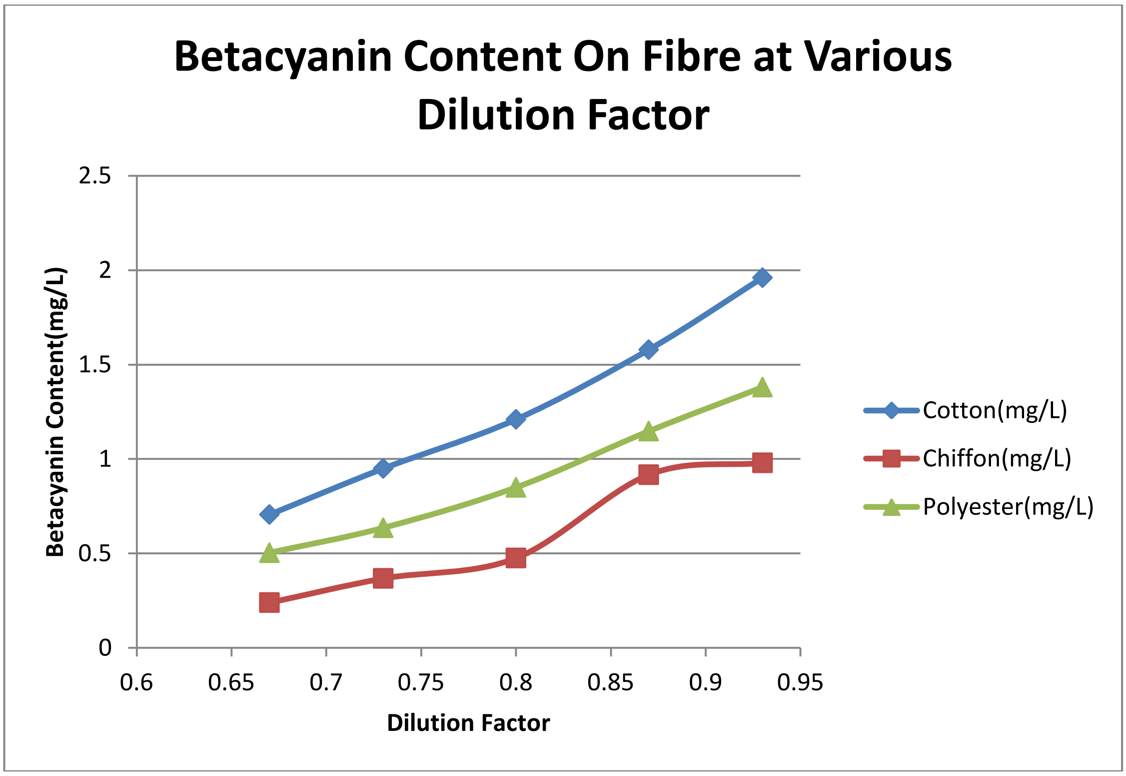


**Figure 4.5:** Betacyanin Content on Fibres at Various Dilution Factors

Figure 4.5 illustrates the betacyanin content on fibre at various dilution factors. Based on the graph, an increasing trend of betacyanin content can be seen as the dilution factor increases. This is because, as the dilution factor increases, the dye solution is getting more concentrated with betacyanin content. Therefore, it can get adsorbed into all the 3 types of fibres effectively at higher dilution factor.

Other than that, the trend of betacyanin content is also similar with the experiment on equilibrium time, whereby, cotton has the highest betacyanin content, followed by polyester and then chiffon. Again, the trend is such because cotton has the highest strength to adsorb the colour pigment. Natural fabric has the tendency to adsorb dye more effectively than the synthetic fibres (Emily, 2009). Since cotton is a natural fibre, it would bond the best with the dye overall. There is not much difference in the strength of adsorption of the natural dye in polyester and chiffon. Polyester however has better adsorption strength since there is presence of more betacyanin pigment in it. Therefore it can adsorb colour better than chiffon. Chiffon shows a sudden increase in betacyanin content at dilution factor 0.80 to 0.87. This is because chiffon adsorbs the betacyanin content best at dilution factor of 0.87.

Table 4.5 shows the amount of dye adsorbed onto cotton, chiffon and polyester. The weight cotton, chiffon and polyester fibre used in 30ml of the dye solution is 0.1170g, 0.1005g and 0.0468 respectively. The graph of amount of dye adsorbed versus dilution factor was then plotted.

**Table 4.5:** Amount of dye adsorbed ($q_e$)

| **Dilution Factor** | **Cotton(mg/g)** | **Chiffon(mg/g)** | **Polyester(mg/g)** |
|---|---|---|---|
| **0.93** | 0.5026 | 0.6282 | 0.4122 |
| **0.87** | 0.4049 | 0.5878 | 0.3427 |
| **0.80** | 0.3103 | 0.3051 | 0.2800 |
| **0.73** | 0.2436 | 0.2359 | 0.1899 |
| **0.67** | 0.1810 | 0.1538 | 0.1504 |

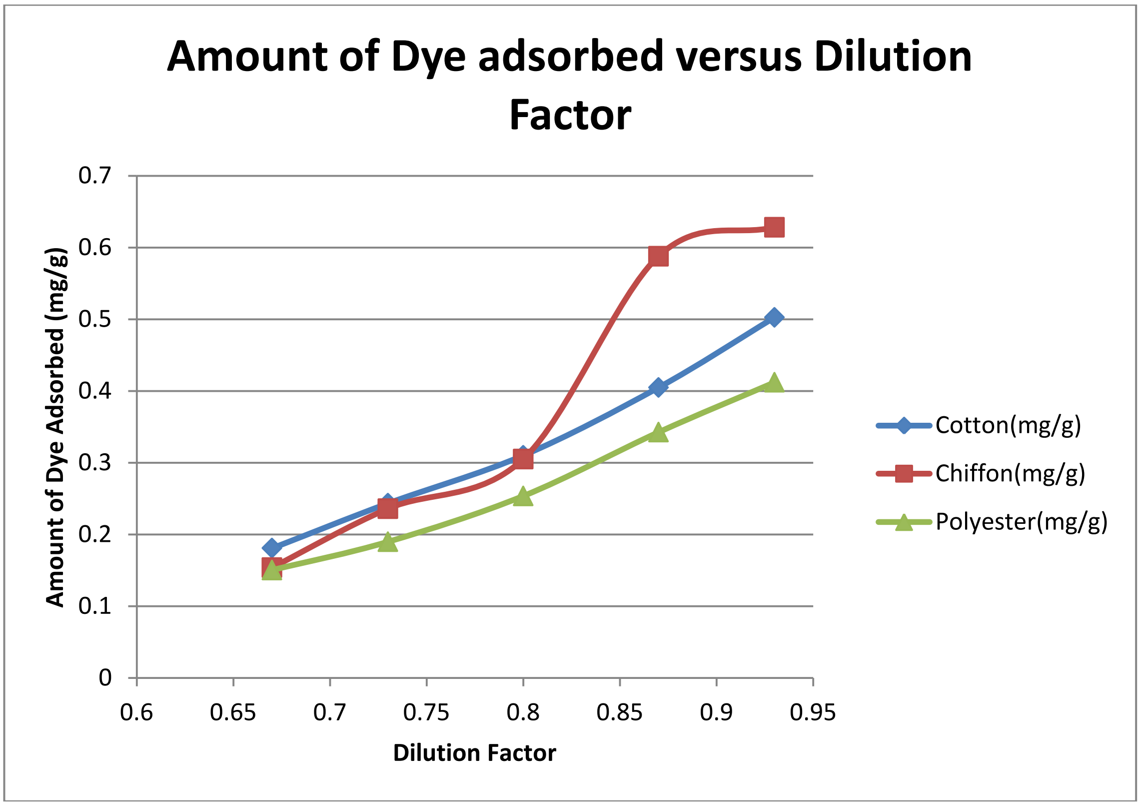


**Figure 4.6:** Amount of Dye adsorbed at Various Dilution Factors

According to Figure 4.6, we can determine the exact amount of dye that had been adsorbed onto cotton, chiffon and polyester fabrics at various dilution factors. The trend of the data above varies a little to the betacyanin content on the fibre data earlier.

It is noticed that cotton has the highest amount of dye adsorbed from the dilution factor of 0.67 to 0.80, followed by cotton and then polyester. The trend then changed with chiffon having the highest amount of dye adsorbed followed by cotton and then polyester at dilution factor 0.87 and 0.93. The trend at high dilution factor is rather alarming because cotton theoretically has highest adsorption tendency as it is a natural fibre (Emily, 2009). When further analysis was done, it is determined that the reason why the amount of dye adsorbed in chiffon is higher compared to cotton is because the weight of 3cm X 3cm dimension of chiffon is much lower than cotton. Therefore, even though the betacyanin pigment content is higher in cotton fibre than chiffon fibre, the amount of dye adsorbed in cotton is lower than chiffon as the weight of chiffon is taken into consideration. Chiffon is light-weighed with a large pores compared to cotton which have tiny pores. The dye must have been stuck in between the large pores of the chiffon fibre when the reading is taken. However, after the drying process, cotton still has the highest colour intensity of the dye, followed by polyester and then chiffon.

**Figure 4.7:** Fibres while they were left for drying

**Figure 4.8:** Fibres after they are dried

Figure 4.7 and Figure 4.8 shows the images of the fibres (cotton, chiffon and polyester) before and after the drying process. The sequence the fibres are such that the fibre that has adsorbed the highest dilution factor of the dye is placed on the top until the lowest dilution factor at the bottom. It is very clear that the fibres with the highest dilution factor of dye being adsorbed have the highest colour intensity. This is because the dye is concentrated with betacyanin pigment at the highest dilution factor and as the dilution factor lowers, the colour intensity decreases as the dye solution gets more diluted.

Cotton has the highest dye intensity followed by polyester and then chiffon. This is again due to the highest adsorption capacity of cotton compared to polyester and chiffon.

## 4.5 Langmuir Isotherm

Langmuir isotherm is one of the most widely used equation to determine the behaviour of an adsorption system and the maximum adsorption that occurs when the surface is covered with a monolayer adsorbate. The equation formulated is as follows (Ghosh, 2006):

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max} K_L C_e} \qquad (4.4)$$

Where,

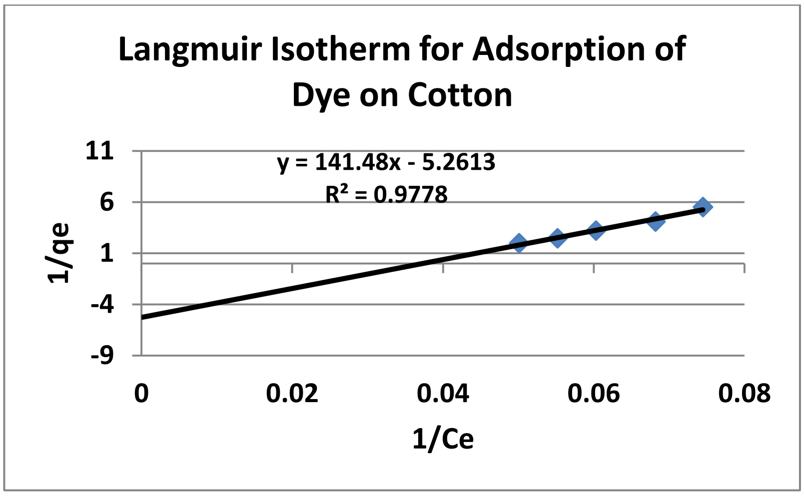
$q_e$ = Dye bound per unit amount of adsorbent (mg/g)

$C_e$ = Unbound dye concentration (in solution) (mg/L)

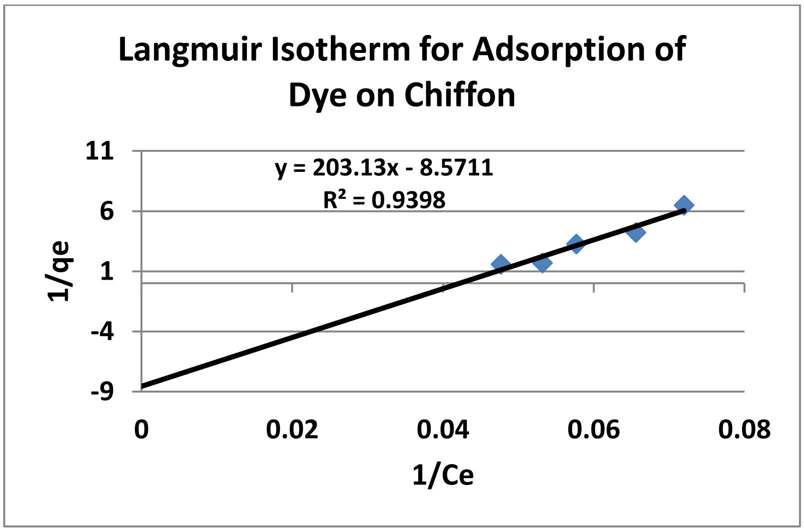
$q_{max}$ = saturation constant (mg/g)

$K_L$ = Affinity constant for Langmuir isotherm (L/mg)

Figures 4.9 (a), (b) and (c) shows Langmuir isotherm on the adsorption of betacyanin on cotton, chiffon and polyester at various dilution factors. The Langmuir constant $K_L$ and $q_{max}$ are calculated using the slope and intercept of the line, obtained from the plot of $1/q_e$ versus $1/C_e$. According to Hamid et al. (2007), the Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies with no transmigration of adsorbate in the plane surface. Once a site is filled, no further sorption can take place at that site. This indicates that the surface reaches a saturation point where the maximum adsorption of the surface will be achieved.



**(a)**



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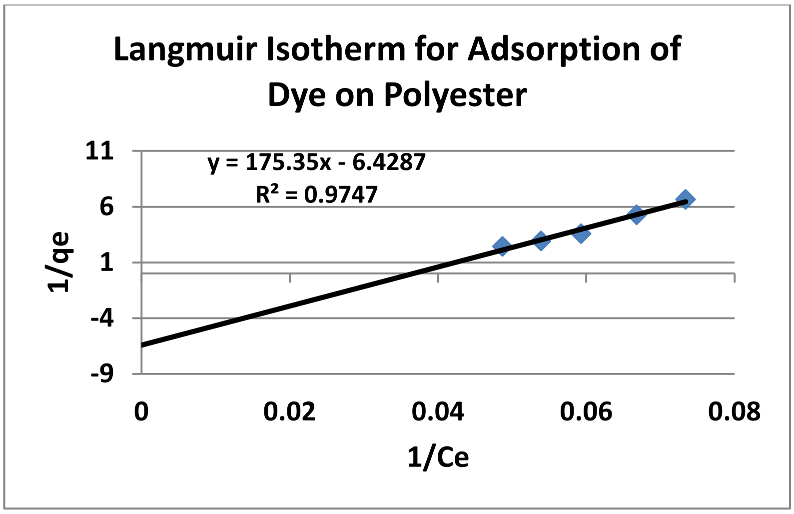


**(c)**

**Figure 4.9:** Langmuir Isotherm on Adsorption of Dye on (a) Cotton, (b) Chiffon, (c) Polyester

## 4.6 Freundlich Isotherm

Freundlich isotherm of adsorption is famous for heterogeneous surface energy which is applicable for this research. The equation formulated is as follows (Ghosh, 2006):

$$\log q_e = logK_f + \frac{1}{n} logC_e \tag{4.5}$$

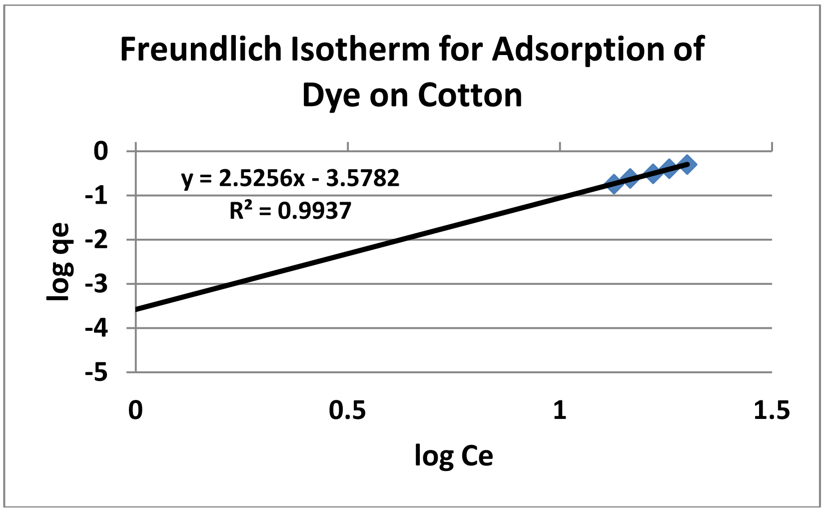
Where,

$q_e$ = Solute bound per unit amount of adsorbent (mg/g)

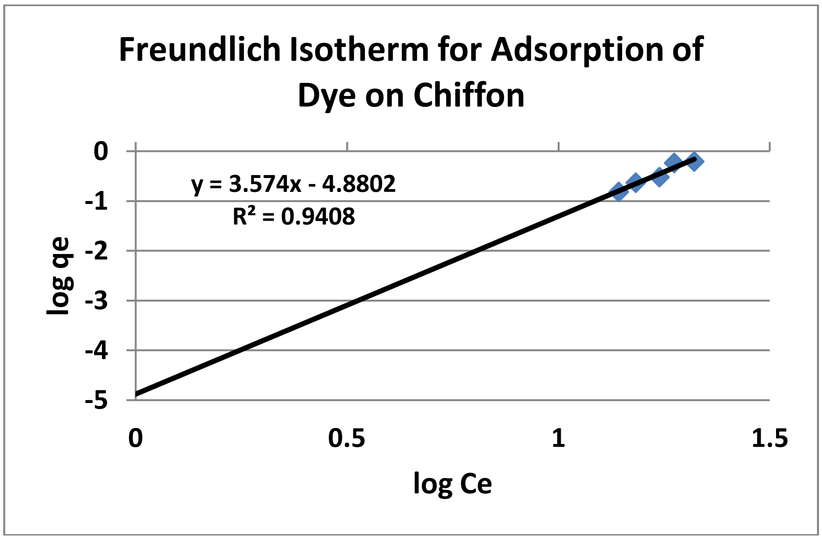
$C_e$ = Unbound solute concentration (in solution) (mg/L)

$K_f$ = Freundlich adsorption constant

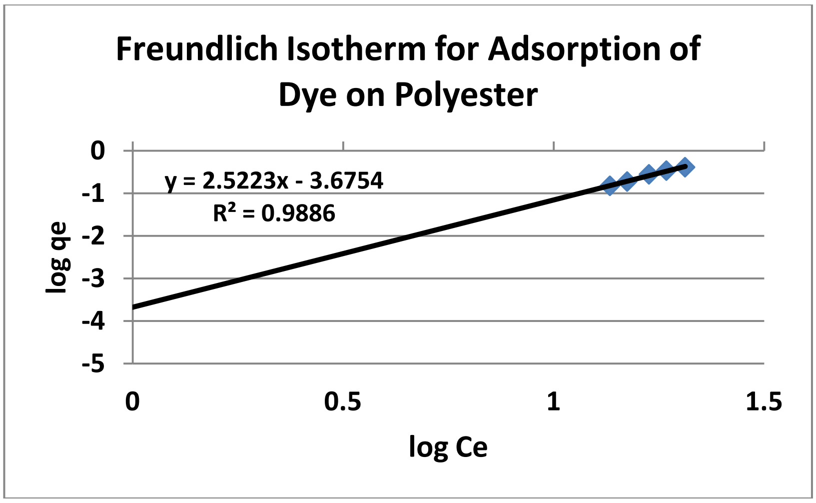
Figures 4.10 (a), (b) and (c) shows the Freundlich isotherm for adsorption of betacyanin on cotton, chiffon and polyester at various dilution factors. Freundlich isotherm constants $K_F$ and *n* are determined from the slope and intercept of the line obtained from the plot of log $q_e$ versus log $C_{e.}$ Freundlich isotherm has a benefit because it is a binding model that can provide accommodation and quantify the heterogeneity and it is extremely valid in determining the heterogeneity (Gregory et al., 2005). The Freundlich equation is usually measured to be purely empirical in nature but it has been used widely to designate the adsorption of dye on adsorbent. A number of investigators indicated that the Freundlich adsorption isotherm describe adsorption data in better way than by the Langmuir equation (Sidhu et al., 2004).



(a)



(b)



(c)

**Figure 4.10:** Freundlich Isotherm for Adsorption of Dye on (a) Cotton, (b) Chiffon, (c) Polyester

The isotherm constants and the correlation coefficient ($R^2$) are listed in Table 4.6. The correlation coefficient, $R^2$ values, which are a measure of goodness-of-fit, show that both the Langmuir and Freundlich isotherm models can describe the adsorption data well for the adsorption of betacyanin on all three type of fibres. However, negative values for Langmuir isotherm constants for the adsorption of betacyanin on cotton, chiffon and polyester are obtained. This indicates the inadequacy of the isotherm model to explain the adsorption process, since these constants helps to indicate the surface binding energy and monolayer coverage. Thus, the adsorption of betacyanin on fibres can be very well described and fitted by Freundlich isotherm model. Similar finding was also reported by Wu et al. (2001). Freundlich $K_F$ value indicates the ratio of the amount of $K_F$ in the solid phase to the amount of $K_F$ solution. According to Shayan and Davey (1978), it is proven that a higher $K_F$ value has more adsorption capacity than having a lesser $K_F$ value. Cotton has the highest $K_F$ value which concludes that it has higher adsorption capacity compared to polyester and chiffon. The value of indicates the degree of nonlinearity between solution concentration and adsorption. The intensity of adsorption is an indication of the bond energies between dye and adsorbent, and the possibility of slight chemisorptions rather than physisorption (Arivoli et al., 2006). The values of *n* obtained for cotton, chiffon and polyester are less than one, indicating the physisorption is much more favourable.

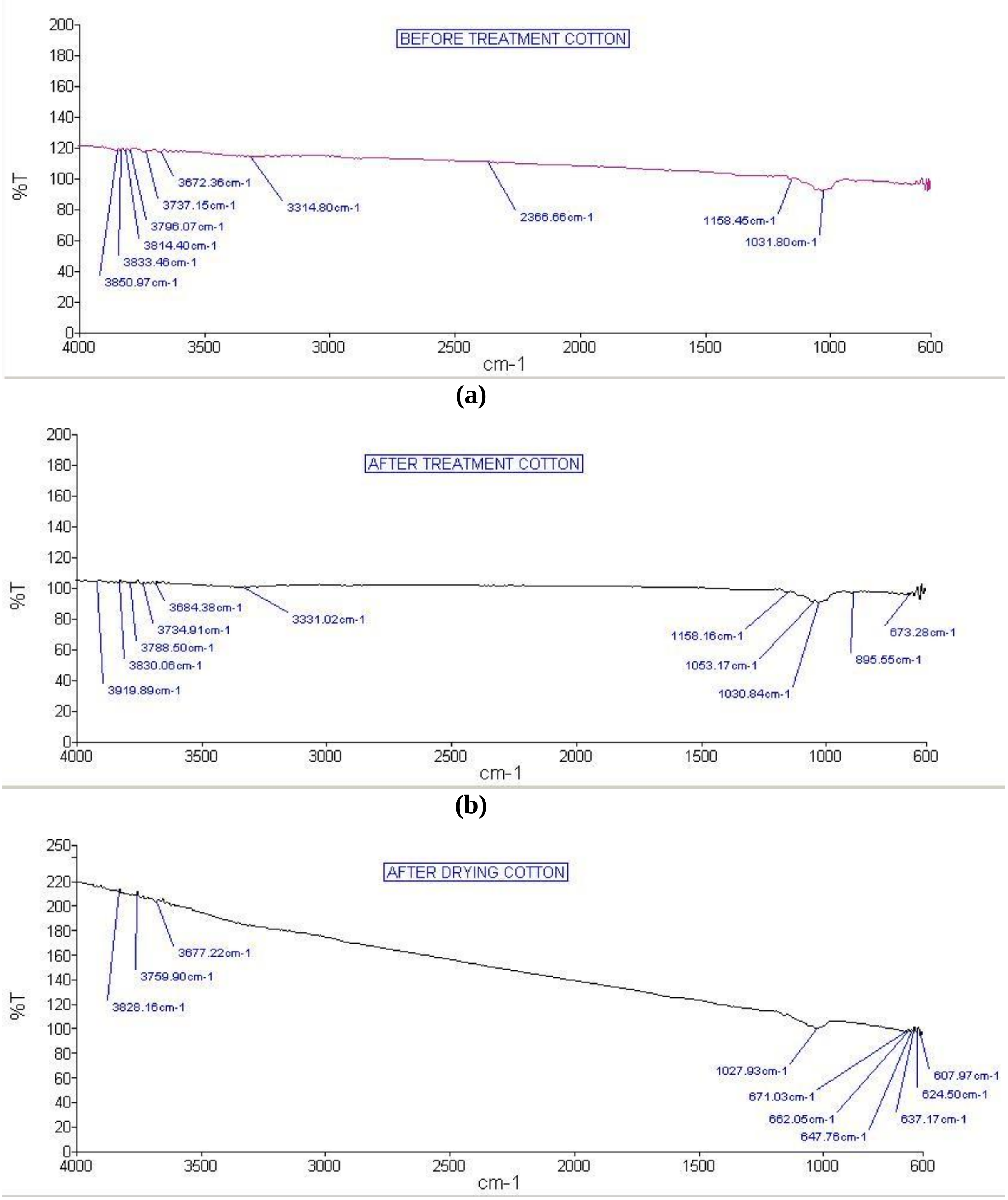
**Table 4.6:** Langmuir and Freundlich Constants for Adsorption of Pitaya Dye on Cotton, Chiffon and Polyester

| | **Langmuir Constant** | | | **Freundlich Constant** | | |
|---|---|---|---|---|---|---|
| **Fabric** | **$q_m$(mg/g)** | **$K_L$(L/mg)** | **$R^2$** | **$K_F$(L/mg)** | **n** | **$R^2$** |
| **Cotton** | -0.1901 | -0.0372 | 0.9778 | 0.0003 | 0.3959 | 0.9937 |
| **Chiffon** | -0.1167 | -0.0422 | 0.7147 | 0.00001 | 0.2798 | 0.9408 |
| **Polyester** | -0.1556 | -0.0367 | 0.9140 | 0.0002 | 0.3695 | 0.9886 |

It can be observed that Freundlich isotherm shows a better fit of the adsorption data compared to Langmuir isotherm. The $R^2$ value obtained for both isotherms showed that both model may be applicable in demonstrating betacyanin adsorption on cotton, chiffon and polyester fibres. However, due to the negative values obtained for Langmuir constants, the data obtained does not fit the monolayer adsorption theorem. Therefore, from the result obtained, it can be said that Freundlich isotherm is more fitted with the data. From these data also, cotton can be chosen as the best adsorbent as it has the highest $R^2$ value and has the highest adsorption capacity compared to polyester and followed by chiffon.

## 4.7 Fourier Transform Infrared Spectroscopy (FTIR)

The formation of functional group in the untreated, treated and dyed cotton, chiffon and polyester samples was conducted using FTIR spectroscopic analysis. The FTIR spectrums of the untreated, treated and dyed cotton, chiffon and polyester possess the same trend of spectrum respectively. This is because all the samples came from the same functional group in cotton, chiffon and polyester. Figure 4.11, Figure 4.12 and Figure 4.13 show the spectrum of untreated, treated and dyed fibre samples.



**(a)**

**(b)**

**(c)**

**Figure 4.11:** FTIR images of (a) Untreated Cotton, (b) Treated Cotton, (c) Dyed Cotton

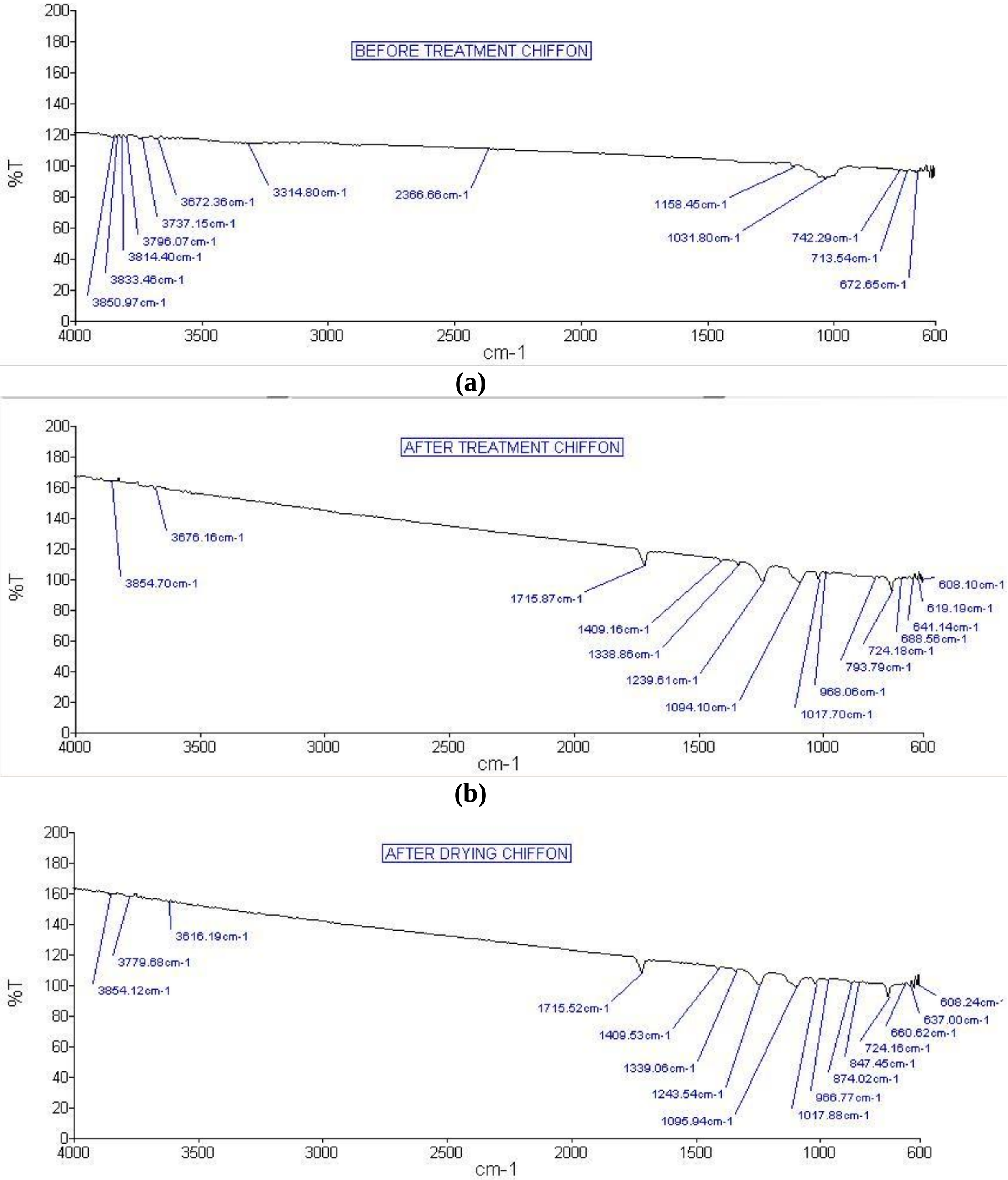
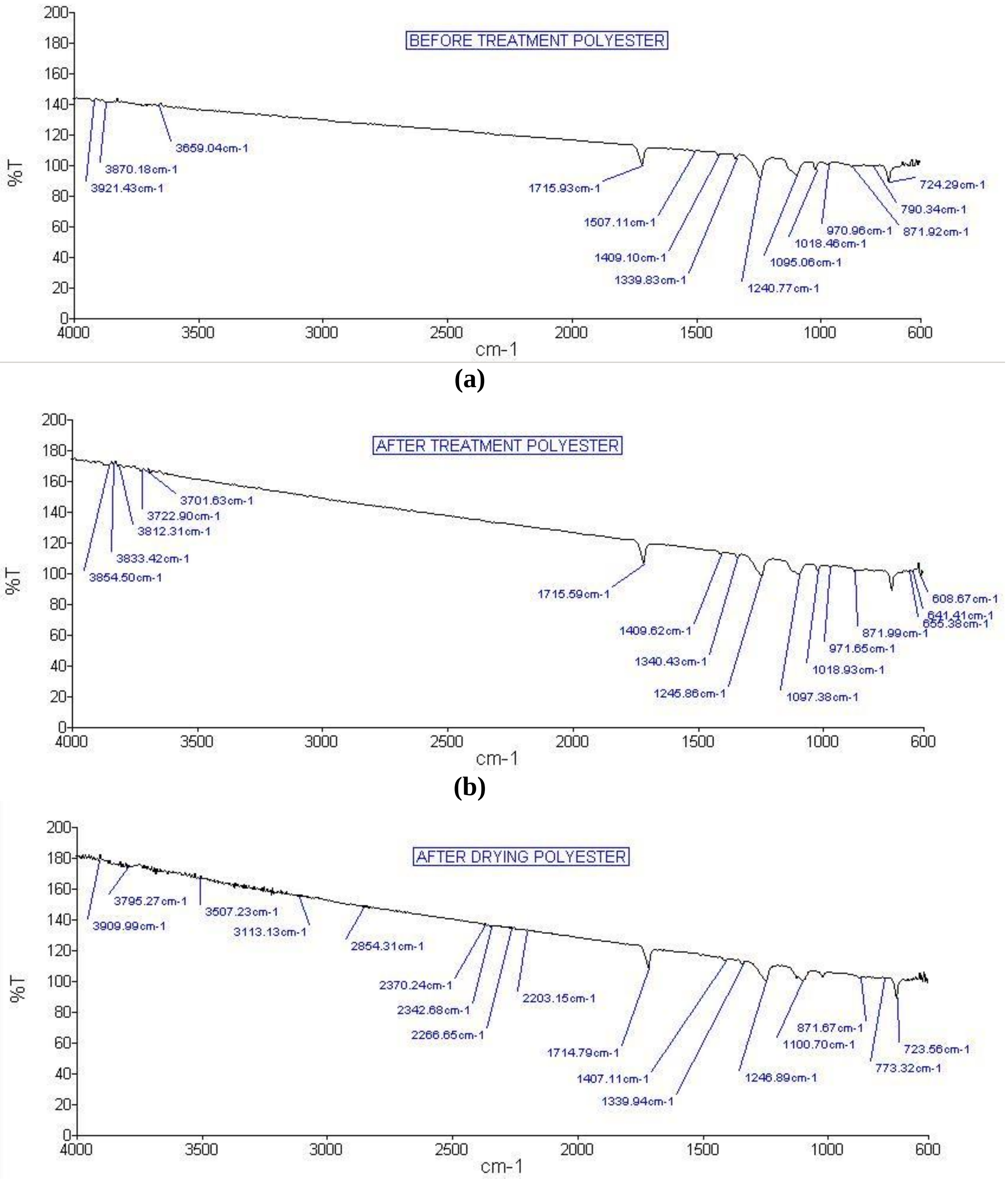


**Figure 4.12:** FTIR images of (a) Untreated Chiffon, (b) Treated Chiffon, (c) Dyed Chiffon



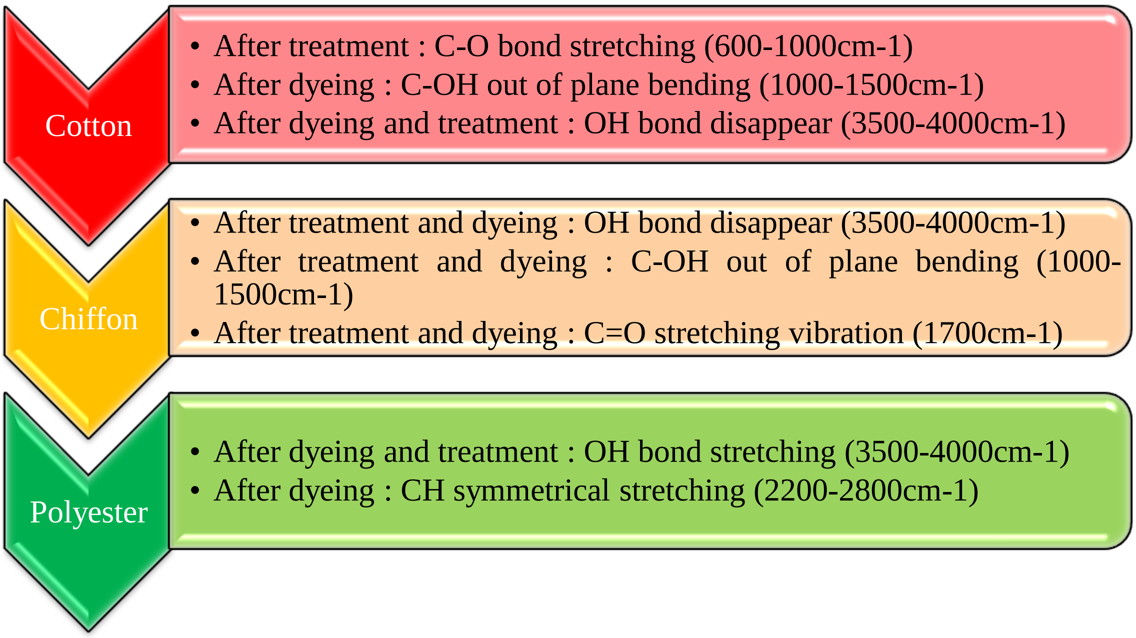
**(a)**

**(b)**

**(c)**

**Figure 4.13:** FTIR images of (a) Untreated Polyester, (b) Treated Polyester, (c) Dyed Polyester

The FTIR spectrum of cotton, chiffon and polyester before treatment after mordant treatment and after dyeing is shown in Figures 4.11, 4.12 and 4.13 respectively. FTIR was recorded to assess structural change if any made in the fiber of the alteration of existing functional groups as a consequence of pretreatment using mordant and process of dyeing. It was found, from the spectra, that the patterns are almost identical for both treated and untreated samples without any additional peaks. However, on comparing the sample of dyed fibres and samples of treated fibre, the samples of dyes fibres caused a slight shift in the position of the peak to a higher wave number than the fibres treated with the mordant Alum due to its higher polarity index. The extent of shift was found to be dependent on solvent pretreatment time.



Cotton

- After treatment : C-O bond stretching (600-1000cm-1)
- After dyeing : C-OH out of plane bending (1000-1500cm-1)
- After dyeing and treatment : OH bond disappear (3500-4000cm-1)

Chiffon

- After treatment and dyeing : OH bond disappear (3500-4000cm-1)
- After treatment and dyeing : C-OH out of plane bending (1000-1500cm-1)
- After treatment and dyeing : C=O stretching vibration (1700cm-1)

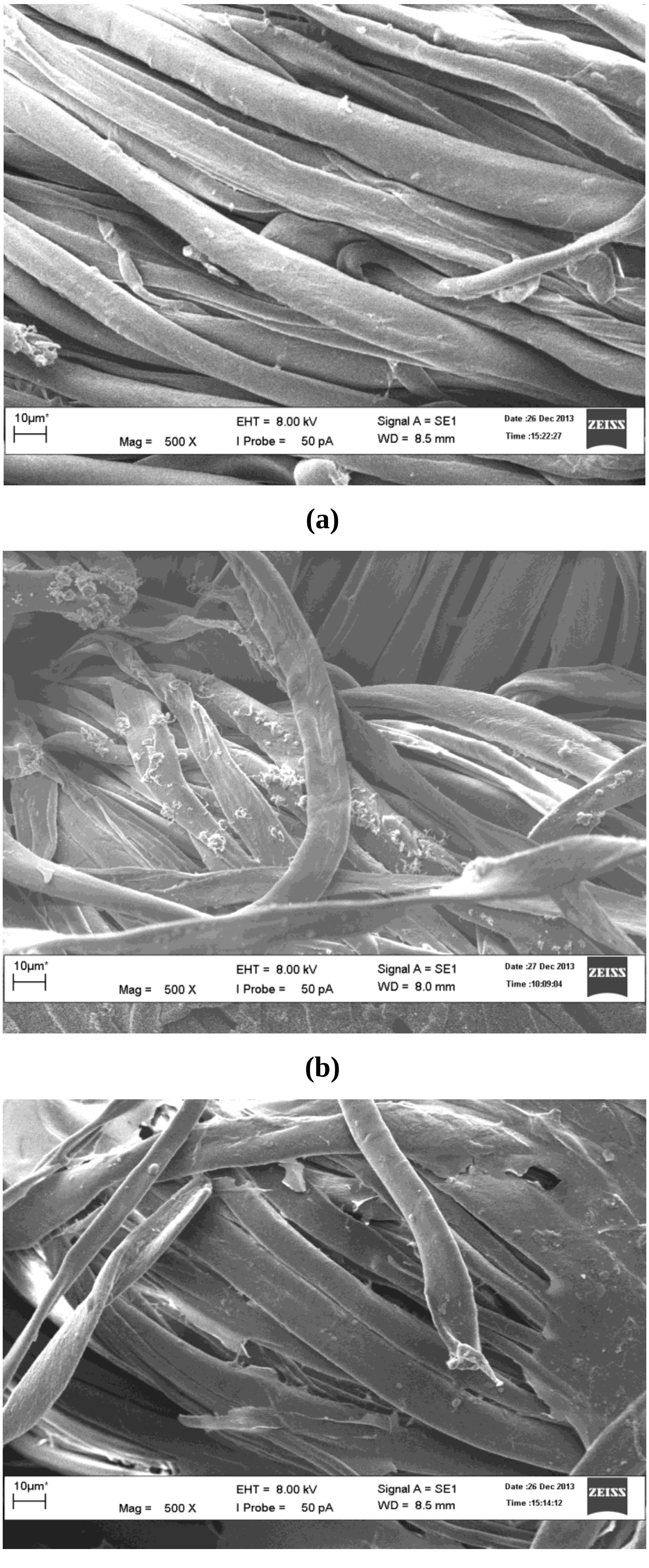
Polyester

- After dyeing and treatment : OH bond stretching (3500-4000cm-1)
- After dyeing : CH symmetrical stretching (2200-2800cm-1)

**Figure 4.14:** FTIR Analysis

## 4.8 Scanning Electron Microscope (SEM) Analysis

Scanning electron micrographs of untreated, treated and dyed cotton, chiffon and polyester fibres are presented in Figures 4.15, 4.16 and 4.17 respectively. The untreated samples exhibit smooth surface texture. In the treated samples, it appears that the mordant attached itself to almost the entire surface of the fibre compared to untreated samples. When the fibres are dyed, the fibres seem to be interconnected by the dye and it looks like a sticky gum attracting the fibres together. The observed results resemble those of earlier reports available in the literature (Muralidharan et al., 2004; Rajendran et al., 1996).



**(a)**

**(b)**

**(c)**

**Figure 4.15:** SEM images of (a) Untreated Cotton, (b) Treated Cotton, (c) Dyed Cotton

The surface morphologies of untreated, treated and dyed cotton are shown in Figure 4.15 (a), (b) and (c) respectively. The morphology of untreated cotton is smooth, whereas when the cotton is treated with Aluminium Sulphate (Alum) mordant, there are quite a number of particles attaching themselves on the surface of the fibre. They act to attract the dye solution onto the fibre. In short, they act as a binding agent. When the cotton fibre is treated with dye, the fibres seem to have attached to each other. This is due to the mordant being the binding agent which caused the dye to be adsorbed onto the surface of the cloth and caused such morphology on the fibre.

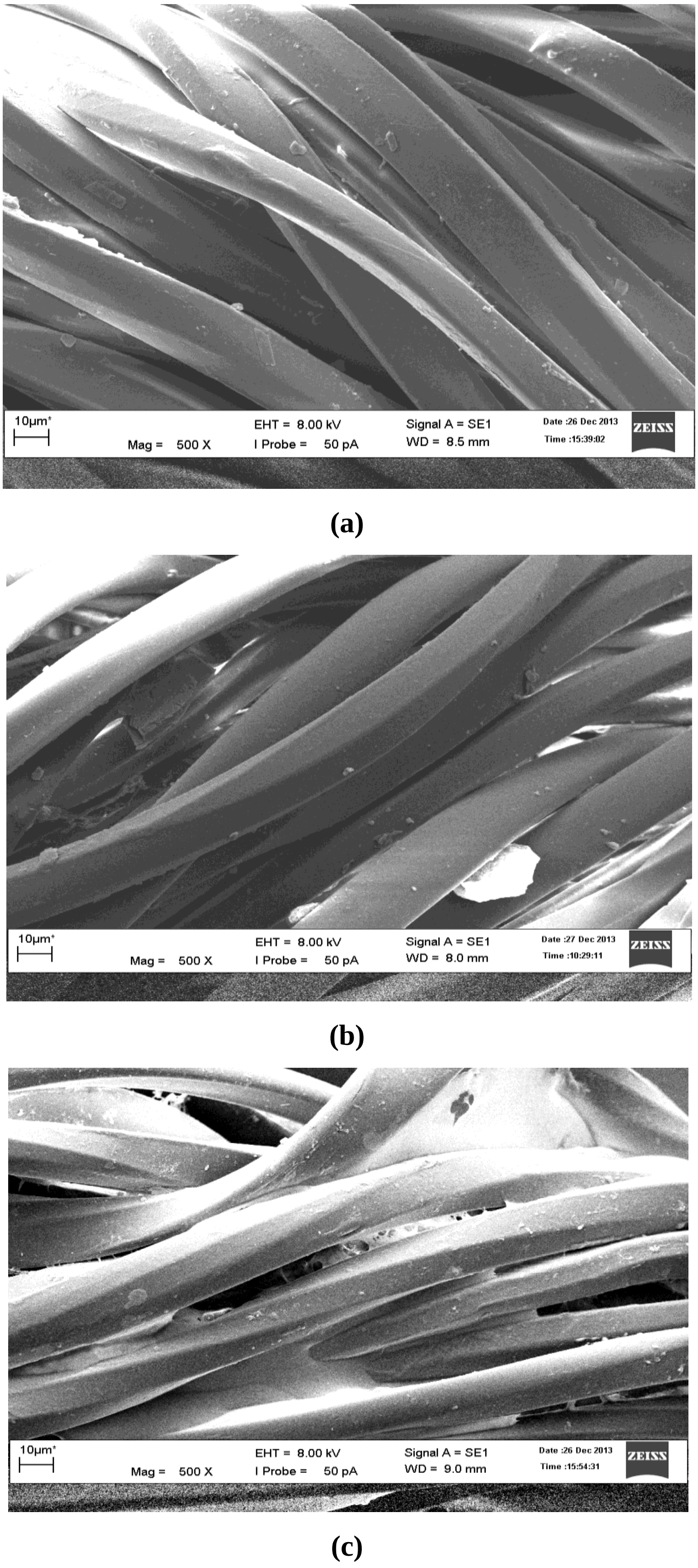
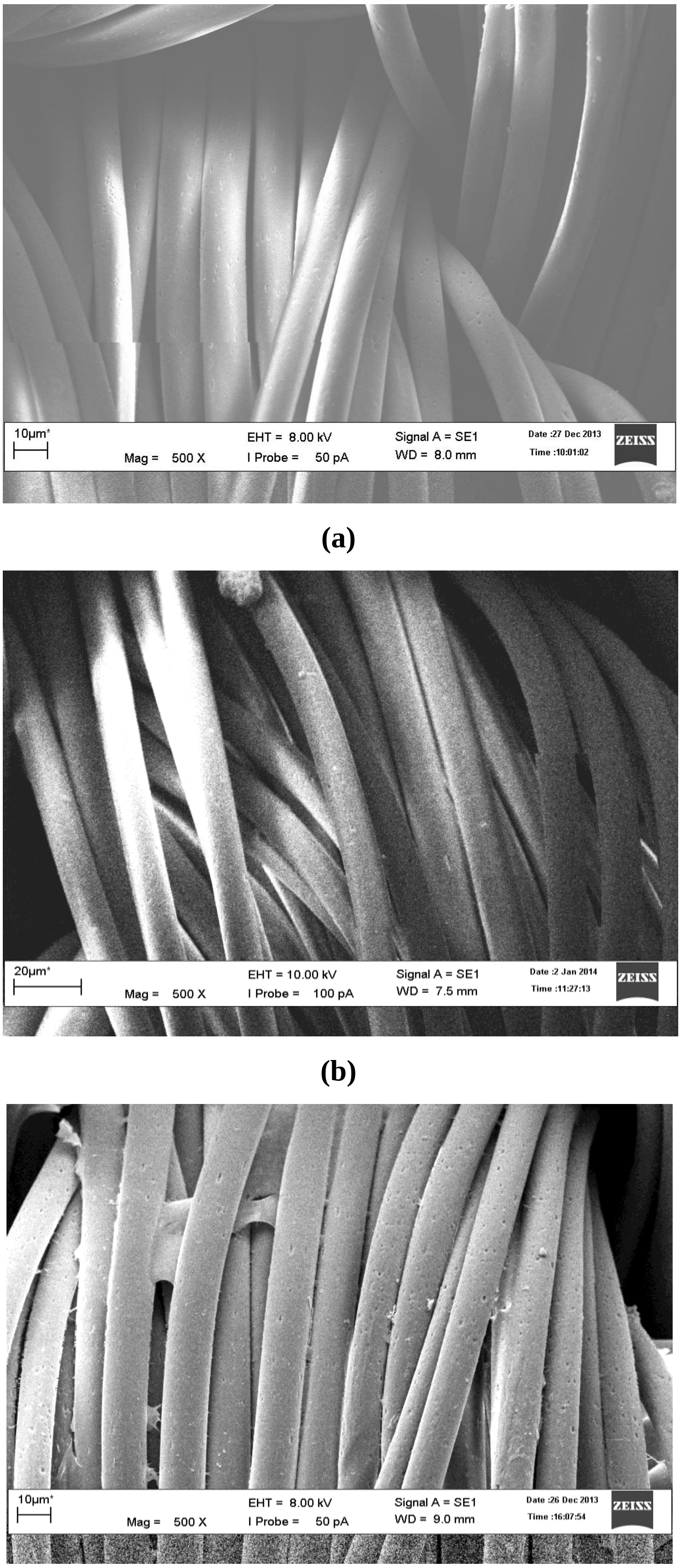


**Figure 4.16:** SEM images of (a) Untreated Chiffon, (b) Treated Chiffon, (c) Dyed Chiffon

The surface morphologies of untreated, treated and dyed chiffon are illustrated in Figure 4.16 (a), (b) and (c) respectively. Untreated chiffon showed an acceptable smooth surface. However, there is only a little difference between the untreated and treated chiffon fibre because there are only a few particles of Alum (mordant) visible. This may be due to the inability of the mordant solution to attach itself onto the surface of the fibre. As a result of that, there is a little binding agent on the surface of the fibre which makes the process of adsorption inefficient. The surface morphology of the dyed chiffon shows the fibres being interconnected by the dye solution that was attracted by the mordant particles.



**(a)**

**(b)**

**(c)**

**Figure 4.17:** SEM images of (a) Untreated Polyester, (b) Treated Polyester, (c) Dyed Polyester

The surface morphologies of untreated, treated and dyed polyester fibres are shown in Figure 4.17 (a), (b) and (c) respectively. Untreated polyester shows a very smooth surface morphology. Treated polyester however shows a not so smooth morphology of the fibre due to the presence of the mordant particles. These mordant particles then cause the dye solution to get attracted to the surface of the fibre and cause the fibre to be interconnected to each other as shown in Figure 4.17 (c).

When compared, cotton fibre seemed to have the most mordant particle attached to the surface of fibre, followed by polyester and then cotton. This is because the suface of the cotton fibre is more favourable for the mordant solution to get attracted compared to polyester and chiffon. Cotton fibre naturally has the highest tendancy of adsorption as it is a natural fibre. Therefore, more dye gets adsorbed onto the surface of cotton fibre, followed by polyester and chiffon (synthetic fibres).

## 4.9 Desorption Process

To ensure the feasibility of the dye adsorption experiment, it is necessary to determine whether the dye solution on the fibre undergoes desorption when soaked in the water. Therefore, the fibre with the best concentration of dye that is adsorbed onto the fibre had undergone this feasibility test. The 3 type fibres with the best dye concentration (dilution factor) which is 0.93 were soaked in 30ml of water in test tubes and after 30minutes, the optical density of the water solution is taken and the reading is recorded.

Betacyanin content desorbed was calculated using equation 4.1. The amount of betacyanin content in water can be calculated by subtracting the amount of betacyanin content in water after desorption with betacyanin content in water before desorption. Table 4.7 shows the betacyanin content in water after desorption process.

**Table 4.7:** Betacyanin content in Water after Desorption Process

| Type of Fibre | Betacyanin Content (mg/L) |
|---|---|
| **Cotton** | 0.077 |
| **Chiffon** | 0.111 |
| **Polyester** | 0.094 |

From the figures in Table 4.7, it can be seen that cotton fibre has the highest betacyanin content being desorbed into 30ml of water solution in 30 minutes, followed by chiffon and then polyester. This is because cotton has the highest ability to retain the dye on the cloth compared to polyester and chiffon. Chiffon however has to lowest tendency to retain the betacyanin dye onto the cloth. Despite all of that, the rate of desorption is considered low as the amount of betacyanin content in the water solution is not very significant. Therefore, this experiment is feasible.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

## 5.1 Conclusion

This project focuses on the adsorption study of betacyanin on fibre. The equilibrium time for adsorption process of betacyanin on cotton, chiffon and fibre was determined to be 90 minutes. The best dilution factor for maximum adsorption of betacyanin on fibre was determined to be 0.93 with ratio of 28mL of betacyanin dye and 2 mL of water. The best adsorption process was proven to be on cotton, followed by polyester and then chiffon. This is because of the high adsorption capacity present on cotton fibre after the process of mordanting using Alum. The best adsorption isotherm that describes and fits the data well for adsorption of betacyanin on fibre was Freundlich isotherm. Cotton also has the lowest desorption of the dye followed by polyester and then chiffon.

## 5.2 Recommendations

To further improve on the research project, the adsorption analysis can be done by using multiple types of mordants and determine which mordant assists best in the process of dye adsorption. Besides that, the research on minimizing desorption of dye from the fibre can also be done to ensure that there is no much of colour loss when washing the fibre. Other than that, experiments on determining the possible way to retain the colour of dye can also be done because natural dye has the tendency of fading in terms of colour.

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# APPENDICES

## A.1 Raw Data

### Equilibrium Time

Trial 1

Initial Absorbance Reading: 2.420

**Table A.1.1:** Absorbance Reading at 15 minutes interval for 1st Trial

| Minute | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **15** | 2.372 | 2.398 | 2.387 |
| **30** | 2.340 | 2.373 | 2.366 |
| **45** | 2.331 | 2.369 | 2.357 |
| **60** | 2.315 | 2.349 | 2.333 |
| **75** | 2.298 | 2.334 | 2.342 |
| **90** | 2.292 | 2.329 | 2.338 |
| **105** | 2.291 | 2.330 | 2.336 |
| **120** | 2.293 | 2.329 | 2.337 |

Trial 2

Initial Absorbance Reading: 2.293

**Table A.1.2:** Absorbance Reading at 15 minutes interval for 2nd Trial

| Minute | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **15** | 2.279 | 2.283 | 2.285 |
| **30** | 2.256 | 2.269 | 2.266 |
| **45** | 2.243 | 2.260 | 2.252 |
| **60** | 2.234 | 2.249 | 2.246 |
| **75** | 2.226 | 2.252 | 2.234 |
| **90** | 2.220 | 2.247 | 2.230 |
| **105** | 2.222 | 2.245 | 2.230 |
| **120** | 2.221 | 2.245 | 2.230 |

Betacyanin Content

Before Adsorption: 21.601 mg/L

**Table A.1.3:** Betacyanin Content in the Dye solution after Adsorption Process

| Minute | Cotton (mg/L) | Chiffon (mg/L) | Polyester (mg/L) |
|---|---|---|---|
| **15** | 21.322 | 21.459 | 21.413 |
| **30** | 21.065 | 21.276 | 21.230 |
| **45** | 20.964 | 21.221 | 21.129 |
| **60** | 20.854 | 21.074 | 20.992 |
| **75** | 20.735 | 21.019 | 20.973 |
| **90** | 20.680 | 20.973 | 20.928 |
| **105** | 20.689 | 20.973 | 20.928 |
| **120** | 20.689 | 20.964 | 20.937 |

**Table A.1.4:** Betacyanin Content on Fibre

| Minute | Cotton (mg/L) | Chiffon (mg/L) | Polyester (mg/L) |
|---|---|---|---|
| **15** | 0.279 | 0.142 | 0.188 |
| **30** | 0.536 | 0.325 | 0.371 |
| **45** | 0.637 | 0.380 | 0.472 |
| **60** | 0.747 | 0.527 | 0.609 |
| **75** | 0.866 | 0.582 | 0.628 |
| **90** | 0.921 | 0.628 | 0.673 |
| **105** | 0.912 | 0.628 | 0.673 |
| **120** | 0.912 | 0.637 | 0.664 |

**Adsorption Process at Various Concentration of Natural Dye**

Wavelength: 560nm

Trial 1

**Table A.1.5:** Initial absorbance readings at various dilution factors for 1st trial

| Dilution Factor | Initial Reading |
|---|---|
| **0.93** | 2.512 |
| **0.87** | 2.387 |
| **0.80** | 2.357 |
| **0.73** | 2.229 |
| **0.67** | 2.201 |

**Table A.1.6:** Absorbance reading after 90 minutes at various dilution factors for 1st trial

| Dilution Factor | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **0.93** | 2.252 | 2.372 | 2.332 |
| **0.87** | 2.161 | 2.227 | 2.227 |
| **0.80** | 2.167 | 2.277 | 2.219 |
| **0.73** | 2.056 | 2.159 | 2.129 |
| **0.67** | 2.051 | 2.151 | 2.111 |

Trial 2

**Table A.1.7:** Initial absorbance readings at various dilution factors for 2nd trial

| Dilution Factor | Initial Reading |
|---|---|
| **0.93** | 2.632 |
| **0.87** | 2.552 |
| **0.80** | 2.498 |
| **0.73** | 2.434 |
| **0.67** | 2.399 |

**Table A.1.8:** Absorbance reading after 90 minutes at various dilution factors for 2nd trial

| Dilution Factor | Cotton | Chiffon | Polyester |
|---|---|---|---|
| **0.93** | 2.432 | 2.542 | 2.487 |
| **0.87** | 2.382 | 2.482 | 2.425 |
| **0.80** | 2.358 | 2.448 | 2.380 |
| **0.73** | 2.324 | 2.394 | 2.345 |
| **0.67** | 2.319 | 2.371 | 2.325 |

Betacyanin Content

**Table A.1.9:** Initial Betacyanin Content at Various Dilution Factors

| Dilution Factor | Initial Betacyanin content(mg/L) |
|---|---|
| **0.93** | 21.926 |
| **0.87** | 19.698 |
| **0.80** | 17.805 |
| **0.73** | 15.605 |
| **0.67** | 14.126 |

Betacyanin Content after adsorption ($C_e$)

**Table A.1.10:** Betacyanin Content at Various Dilution Factors after Adsorption Process

| Dilution Factor | Cotton (mg/L) | Chiffon (mg/L) | Polyester(mg/L) |
|---|---|---|---|
| **0.93** | 19.966 | 20.946 | 20.545 |
| **0.87** | 18.119 | 18.781 | 18.550 |
| **0.80** | 16.595 | 17.329 | 16.867 |
| **0.73** | 14.655 | 15.237 | 14.969 |
| **0.67** | 13.420 | 13.886 | 13.622 |

**Table A.1.11:** Betacyanin Content on Fibre at Various Dilution Factors

| Dilution Factor | Cotton(mg/L) | Chiffon(mg/L) | Polyester(mg/L) |
|---|---|---|---|
| **0.93** | 1.960 | 0.980 | 1.381 |
| **0.87** | 1.579 | 0.917 | 1.148 |
| **0.80** | 1.210 | 0.476 | 0.938 |
| **0.73** | 0.950 | 0.368 | 0.636 |
| **0.67** | 0.706 | 0.240 | 0.504 |

## Langmuir Isotherm

**Table A.1.12:** Langmuir Isotherm

| | $1/C_e$ | | | $1/q_e$ | | |
|---|---|---|---|---|---|---|
| **Dilution Factor** | **Cotton (mg/L)** | **Chiffon (mg/L)** | **Polyester (mg/L)** | **Cotton (mg/g)** | **Chiffon (mg/g)** | **Polyester (mg/g)** |
| **0.93** | 0.0501 | 0.0477 | 0.0487 | 1.9897 | 1.5918 | 2.4260 |
| **0.87** | 0.0552 | 0.0532 | 0.0539 | 2.4697 | 1.7013 | 2.9180 |
| **0.80** | 0.0603 | 0.0577 | 0.0593 | 3.2227 | 3.2776 | 3.5714 |
| **0.73** | 0.0682 | 0.0656 | 0.0668 | 4.1051 | 4.2391 | 5.2659 |
| **0.67** | 0.0745 | 0.0720 | 0.0734 | 5.5249 | 6.5020 | 6.6489 |

## Freundlich Isotherm

**Table A.1.13:** Freundlich Isotherm

| | Log $C_e$ | | | Log $q_e$ | | |
|---|---|---|---|---|---|---|
| **Dilution Factor** | **Cotton (mg/L)** | **Chiffon (mg/L)** | **Polyester (mg/L)** | **Cotton (mg/g)** | **Chiffon (mg/g)** | **Polyester (mg/g)** |
| **0.93** | 1.3003 | 1.3211 | 1.3127 | -0.2988 | -0.2019 | -0.3849 |
| **0.87** | 1.2581 | 1.2737 | 1.2683 | -0.3927 | -0.2308 | -0.4651 |
| **0.80** | 1.2200 | 1.2388 | 1.2270 | -0.5082 | -0.5156 | -0.5528 |
| **0.73** | 1.1660 | 1.1829 | 1.1752 | -0.6133 | -0.6273 | -0.7215 |
| **0.67** | 1.1278 | 1.1426 | 1.1342 | -0.7423 | -0.8130 | -0.8228 |

**Desorption Process**

Trial 1

Initial Absorbance reading: 0.000

**Table A.1.14:** Final Absorbance Reading for 1st Trial (Desorption Process)

| Type of Fibre | Final Absorbance Reading |
|---|---|
| **Cotton** | 0.009 |
| **Chiffon** | 0.013 |
| **Polyester** | 0.011 |

Trial 2

Initial Absorbance reading: 0.000

**Table A.1.15:** Final Absorbance Reading for 2nd Trial (Desorption Process)

| Type of Fibre | Final Absorbance Reading |
|---|---|
| **Cotton** | 0.008 |
| **Chiffon** | 0.013 |
| **Polyester** | 0.010 |

Average Reading

Initial Absorbance reading: 0.000

**Table A.1.16:** Average Final Absorbance Reading (Desorption Process)

| Type of Fibre | Final Absorbance Reading |
|---|---|
| **Cotton** | 0.009 |
| **Chiffon** | 0.013 |
| **Polyester** | 0.011 |

## A.2 Statistical Analysis

### Betacyanin Content on Fibre

Anova: Single Factor

SUMMARY

| *Groups* | *Count* | *Sum* | *Average* | *Variance* |
|---|---|---|---|---|
| Column 1 | 8 | 5.81 | 0.72625 | 0.053013 |
| Column 2 | 8 | 3.849 | 0.481125 | 0.032761 |
| Column 3 | 8 | 4.278 | 0.53475 | 0.031447 |

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* |
|---|---|---|---|---|---|---|
| Between Groups | 0.265691 | 2 | 0.132846 | 3.399887 | 0.052586 | 3.4668 |
| Within Groups | 0.820544 | 21 | 0.039074 | | | |
| | | | | | | |
| Total | 1.086235 | 23 | | | | |

### For Langmuir Isotherm

Anova: Single Factor

SUMMARY

| *Groups* | *Count* | *Sum* | *Average* | *Variance* |
|---|---|---|---|---|
| Column 1 | 5 | 0.3083 | 0.06166 | 9.62E-05 |
| Column 2 | 5 | 0.2962 | 0.05924 | 9.38E-05 |
| Column 3 | 5 | 0.3021 | 0.06042 | 9.76E-05 |
| Column 4 | 5 | 17.3121 | 3.46242 | 1.969681 |
| Column 5 | 5 | 17.3118 | 3.46236 | 4.1193 |
| Column 6 | 5 | 20.8302 | 4.16604 | 3.078306 |

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* |
|---|---|---|---|---|---|---|
| Between Groups | 100.8314 | 7 | 14.40449 | 8.641837 | 4.2E-05 | 2.463774 |
| Within Groups | 36.6703 | 22 | 1.666832 | | | |
| | | | | | | |
| Total | 137.5017 | 29 | | | | |

## For Freundlich Isotherm

Anova: Single Factor

SUMMARY

| *Groups* | *Count* | *Sum* | *Average* | *Variance* |
|---|---|---|---|---|
| Column 1 | 5 | 6.0722 | 1.21444 | 0.00479 |
| Column 2 | 5 | 6.1591 | 1.23182 | 0.005032 |
| Column 3 | 5 | 6.1174 | 1.22348 | 0.005071 |
| Column 4 | 5 | -2.5553 | -0.51106 | 0.030749 |
| Column 5 | 5 | -2.3886 | -0.47772 | 0.068317 |
| Column 6 | 5 | -2.9471 | -0.58942 | 0.032634 |

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* |
|---|---|---|---|---|---|---|
| Between Groups | 22.98437 | 7 | 3.283481 | 123.1922 | 3.71E-16 | 2.463774 |
| Within Groups | 0.586373 | 22 | 0.026653 | | | |
| | | | | | | |
| Total | 23.57074 | 29 | | | | |