INVESTIGATION OF HIGH VOLUME BID MATERIALS AS POTENTIAL COLOURANTS AND FINISH CHEMICALS FOR FIBROUS SUBSTRATES

BY

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The dissertation was submitted to the Department of Textile and Clothing Technology of the University of Moratuwa, Sri Lanka in partial fulfillment of the requirement for the Degree of Doctor of Philosophy

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Abstract

The modern consumer (1990 onwards) is aware of the toxic chemical residues on textiles/garments (resulting from dyes and chemicals used) which can have carcinogenic/ dermatological and allergic effects on the wearer, especially because textiles are in contact with human skin for 24 hours of the day. The second aspect deals with the 'pollution' (air/water) at each of all stages in production of textiles. The third concern is about the 'ecological' problems during disposal (of garbage / on incineration).

The aim of this research is to show feasibility of producing high quality natural dyes from plants, creating new opportunities for both farmers and the fabric / garment industry in line with the current consumer trends towards eco-friendly natural products. The direct national benefit is shown. Environmental and economical factors too need to be considered to make this viable in the long run.

Investigation of the traditional dyeing techniques and dye producing plants with special reference to Sri Lanka, and development of natural dyes and investigation of their suitability as textile dyes were the two major objectives of this research study.

Research investigations based on the comprehensive analysis of 10 best dye yielding plants which have been chosen from 47 dye yielding plants in Sri Lanka are presented. The available raw material spectrum had been reviewed. The ten (10) selected species are Kothala Himbutu *(Salacia reticulata)*, Weniwal *tCoscinium fenestratum)*, Rambutan *(Nephelium lappaceum)*, Mangus *tGarcinia mangostana)*, Big onion skin *(Allium cepa)* , Marigold *(Tegetus erecta)*, Tea *(Camellia sinensis)*, Jak *(Artocarpus heterophyllus)*, Walmadata *(Rubia cordifolia)* and Turmeric *(Curcuma domestica)*. Some of the above plant extracts have not been used before in textile dyeing.

Environmental performance was another aspect of the research. Results from effluent characteristics of best dyeing solutions reveal significant reduction in pollution potential. The concept of ready to use dye concentrates is also presented.

Dedication

to

My

Parents, husband and children
Who contributed in their own way

With

Love and Gratitude

'The Path to Knowledge is Awareness'

DECLARATION

I Samudrika Wijayapala , hereby certify that the work described in this dissertation was carried out by me in the Departments of Textile and Clothing Technology and Chemical and Process Engineering of the University of Moratuwa , Sri Lanka and Indian Institute of Technology , Kanpur , India between January 2004 and January 2010. This research project was carried out in partial fulfillment of the requirement for the degree of Doctor of Philosophy. This dissertation is the result of my own work and includes nothing which is the outcome of work done in collaboration, except where otherwise stated. Neither this thesis nor any part thereof has ever been submitted for any degree at this or any other University.

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We certify the statement above is true to best of our knowledge and that the dissertation is ready for submission

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"Authorship of any sort is a fantastic indulgence of the ego. It is well no doubt, to reflect on how much one owes to others- J.K.Galbraith"

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ABBREVIATIONS

AATCC	American Association of Textile Chemists and Colourists
AD	*Anno Domini* – After Death
BASF	Badische Anilin und Soda Fabrik *(German chemical products company)*
BC	Before Christ
BOD	Biochemical Oxygen Demand
BMICH	Bandaranayake Memorial International Conference Hall
CD	Compact Disk
CI	Colour Index
CIELAB	*Commission Internationale d'Eclairage*
COD	Chemical Oxygen Demand
DNA	Deoxyribo Nucleic Acid
ESCAP	Economic and Social Commission for Asia and the Pacific
FTIR	Fourier Transform Infra Red spectroscopy
ICI	Imperial Chemical Industries
ICP	Inductively Coupled Plasma Optical Emission Spectrophotometer
IR	Infra-red
ISO	International Standard Organisation
K/S	Relationship between Absorption and Scattering Spectrum
LF	Light Fastness
MLR	Material to Liquor Ratio
MT	Metric tonnes
NA	Not Applicable
ND	Not Detected
owf	On Weight of Fabric
RTDC	Ready to Dye Concentrate
RF	Rubbing Fastness
TA	Tannic acid
UV-Vis	Ultra Violet Visible Spectroscopy
WF	Wash Fastness
WHO	World Health Organisation

Chapter One

INTRODUCTION AND OVERVIEW

1.1 The need for natural dyes

"The Sri Lankan garment industry is trying to differentiate itself from the low cost competition in international markets by positioning itself as an 'ethical manufacturing designation'. The industry has already launched an international campaign called "Garments without Guilt". The campaign is trying to raise awareness internationally about Sri Lanka's much higher labour and environmental standards, compared to other lower cost, garment manufacturing countries. In 2007, earnings from garment exports reached to US$ 3.2 billion. The industry also claims it has reduced its import component and that domestic value addition now stands at 50 % compared to 25 % in the 1970's. The industry remains the largest employer in manufacturing, providing direct employment to an estimated 270,000 people mainly young women from rural parts of the country" (FT, 2008, Oct.).

Textile and garment industry is an important economic activity with more than 40% contribution to the Sri Lankan economy. Industrial exports expanded by 10% in 2007, largely supported by a marked increase in the earnings from exports of garments and textiles. Exports of garments and textiles continue to be the largest source of foreign exchange earnings for Sri Lanka (Central Bank, 2007). Dyes are an important process requirement for which the country spends about 8.5 mn US$ annually. Almost all dyes presently used for textile dyeing are of synthetic origin which is imported to the country. However, in Sri Lanka historically, textile dyeing has been through natural dyes.

Mother nature supplies an abundance of brilliant colours readily available to anyone's enjoyment. Dye materials (such as, onion skins, marigold flowers, and black tea etc.,) can be easily found in our backyard and in our kitchens. Natural dyes comprise of dyes and pigments that are obtained from animal and vegetable matter without chemical processing.

Natural dyes are extracted from roots, barks, flowers, leaves, stems, berries, lichens, mushrooms, vegetables, animals and minerals. Their colour fastness is achieved by using a mordant such as a metal in powder form like copper, iron or tin or with natural mordants such as vinegar and salt.

Natural dyes in vogue during ancient days were indigo for dark blue / light blue, pomegranate rind for yellow/brown/green, lac for scarlet / crimson / purple, jak fruit heartwood for yellow / green, manjistha root for rust red, myrobalan (Aralu) for khaki / green / black (Internet, 1).

With the discovery of the first chemical dye in the mid nineteenth century, natural dyes were slowly superseded. Until this time both the dyer and the printer had been completely dependant on natural dyes.

Although dyeing with natural dyes has not received the due attention of scientists as well as of industrialists, recently the textile industry is being confronted more with enquiries on the theme of "dyeing with natural dyes". The use of natural colours that should be assumed to have given way to the synthetic dyes over 140 years ago is today once again a matter of topical interest due to the following reasons (Internet, 2).

(a) Within the textile manufacturing chain, wet processing is clearly identified as having a potential adverse effect on the environment. In fact one of the major problems threatening the textile industry today is environmental pollution, arising out of the wet processing of the textiles. The production of synthetic dyes involves many violent reactions, which are conducted at high temperature and pressure, using much hazardous petroleum based primary chemicals as well as the production of hazardous intermediates. It is high time that the over utilisation of synthetic dyestuffs should be thought about in the context of health of the people and environment. In this regard some European countries have introduced a ban on certain azo dyes, which have been found to be carcinogenic. It is also noted to realize that about 2/3 or more of all synthetic dyes are azo and many more of them may be listed as carcinogenic in time to come. In this context, the day that the importance of natural dyes as possible alternatives to at least some of the synthetic dyes would not be very far. Further the recent realization that many intermediates and chemicals used in production of synthetic dyes are toxic and thus hazardous to human health as well as to the environment, has led to the revival of interest in the potentially non-toxic, biodegradable and eco-friendly natural dyes.

(b) The lower possibility of allergenic reactions to the consumer by textile materials dyed with natural dyes together with its possibility of producing unique and fascinating colours which are not achievable with synthetic dyes are two other important factors for the revival of natural dye usage regardless of its high cost and other disadvantages.

(c) Although synthetic dyes have now superseded the natural dyes in the industrial field, certain natural dyes derived from plants are still of some commercial importance in developing countries. One advantage in the use of natural dyes is that various tints and shades may be obtained from a single dye by combining it with different mordants.

(d) Some natural dyes such as indigo from *Indigofera sp.* are softer and more permanent colour than synthetic dyes such as aniline. Eg. The famous Persian rugs and carpets in which natural dyes are used.

(e) Natural dyes come out as a good alternative to the environmental pollution arising from synthetic dyes but also provide low toxicity and allergic reactions while giving unique and fascinating colours which are not achievable from synthetic dyes.

Earlier, understanding of dyeing techniques and their applications was empirical and was not backed by scientific reasoning and therefore natural dyeing had developed essentially as a folk art. However in recent times the dyeing technique is interpreted on sound scientific principles, where the interaction between the dye and the substrate is well understood. This has developed an interest on natural dyeing techniques, which were earlier disregarded by the high paced world (Internet, 3).

1.2 Present situation and justification of the study

Sri Lankan textile sector is a vibrant and a dynamic industry sector. With 300 garment industries at present in Sri Lanka, dyeing is an important process activity in this industry segment.

With the revival of natural dye usage in the world as a solution to the enhanced environmental pollution arising out of the wet processing of textile industry, an upsurge in interest in natural dyes has been manifested in many areas including reconstruction of ancient and traditional dyeing technologies and chemical characterisation of colourants in flora and fauna.

Therefore the use of waste materials as a resource for dye extraction would be beneficial to the environment in many ways. In an overall view, such a natural dyeing application provides eco friendly dyestuffs and dyed textile products leading to the conservation of environmental and human health. Such an application also may minimise the environmental pollution arising in the synthesis and usage of synthetic dyes. Another advantage of using a natural dye obtained from a bio resource categorised as a 'waste', is that it reduces the accumulation of waste materials in the environment by reuse of them leading to a cleaner and healthier environment while saving the cost of handling and discharging the wastes (Internet, 4).

1.3 Objectives

The present research was undertaken with the view to study the role of natural dyes for textile industries. The emphasis was on newer dyeing sources, mordant study and newer dyeing techniques.

Therefore the objectives of this study are,

(a) To investigate the natural dye producing plants in the world and those which are indigenous to Sri Lanka.

(b) To study different techniques of natural dyeing available in the world and to investigate the traditional dyeing techniques practiced in Sri Lanka and their current status.

(c) To select plant materials which go as waste but still contains a dye material in relatively large quantity.

(d) To select a method and develop new natural dyes which is ecologically friendly and with less health hazards from a selected plant material.

(e) To investigate its suitability as a textile dye and to indicate pathways for large scale exploitation to support the local textile industry.

(f) To investigate the role of ultrasonic and conventional dyeing processes. These high-energy releasing devices used for dyeing are advancements over the conventional heating method.

(g) To emphasize on making ready- to- use newer natural dyes for commercial use. These plant extracts are otherwise sticky masses, not very easy to handle and store.

The investigation of the traditional dyeing techniques and dye producing plants was reached within the following scope.

(a) A detailed literature survey was carried out to investigate the history of textile colouration with the intention of discovering the origin of natural dyeing practices in early history.

(b) A survey through literature was performed to reveal the gradual progression path of natural dyeing techniques and the reasons for decline in their usage which has led to almost complete exclusion of them from commercial practice in the world with specific reference to Sri Lanka.

(c) A list of indigenous dye producing plants of Sri Lanka was prepared by narrowing down the list of dye producing plants found in different literature, which were used throughout the world history to the levels of Asia, South Asia (especially India) and finally in Sri Lanka.

(d) The investigation of traditional dyeing techniques involving natural dyes in Sri Lanka and their usage and current status, was carried out through a literature survey together with interviewing selected persons knowledgeable in the field.

1.4 Scope and overview of the research work into natural dyes

The scope of this part of the study was to extract or develop new natural dye yielding materials from available plant sources and to investigate their suitability as textile dyes, based on the colour fastness properties. Although it is mainly based on experimentation, the selection of suitable plant sources as raw materials for the extraction or development of dye yielding bio-materials were based mainly on the literature survey. The sources of raw materials were selected by trial experiments conducted on the plant sources selected from the literature survey.

High emphasis was given to the selection of waste materials as sources of extraction or development as potential textile dye yielding bio-materials.

The aim of this work is to show the feasibility of producing high quality natural dyes creating new opportunities for both farmers and the fabric / garment industry –in line with the current consumer trends towards eco-friendly natural products. In this research 'environmental and economical' factors too need to be considered to make the study viable.

The thesis titled "INVESTIGATION OF HIGH VOLUME BIO MATERIALS FOR POTENTIAL COLOURANTS AND FINISH CHEMICALS" has been systematized and compiled into five chapters each dealing with the specific aspects of dyes, extraction and dyeing of fabrics.

Chapter One the **INTRODUCTION AND OVERVIEW** , defines introduction to natural dyes , general features of natural dyes , their sources from where these natural dye can be obtained , why it is important to revert back to use of natural dyes and the increased interest in natural dyes world wide and justification , scope and objective of the research.

Chapter Two reports a comprehensive **LITERATURE REVIEW** containing 180 references on historical background of dyes, sources of various natural dyes, constitutional aspects of colourants, classification, chemistry of natural colour and the fastness properties of dyed fabric and some important natural dyes. This also presents a review of dyeing practices in Sri Lanka and information regarding the synthetic dyes.

Chapter Three focuses on the experimental techniques used during the study or **METHODOLOGY.** This chapter includes selection of dyes, extraction of dyes using different methods, and the instruments used for the extraction and structure confirmation etc. This chapter also includes the specification of the fabric and method of dyeing fabrics, evaluation of eco-friendly properties of dyes extracted and the preparation of cloth for dyeing and evaluation of best possible condition of dyeing and fastness properties of dyed fabrics.

Chapter Four reports the **RESULTS AND DISCUSSION** of the research investigation. This section is divided into two sections. First section deals with the optimization of best dyeing conditions and interpretation of the results obtained from 47 dye yielding bio materials available in Sri Lanka. Second section interprets the selection of best suitable 10 bio materials from the above 47. This chapter includes the colour catalogue of swatches generated from the 47 bio materials in Sri Lanka and detailed analysis of selected best 10 possible colour yielding bio-materials.

Chapter Five constitutes the **CONCLUSIONS AND RECOMMENDATIONS** drawn from all the experimental and theoretical studies of this research. Finally suggestions are made for further research and development of natural dyes. The steps towards developing a local natural dye based industry are also outlined.

Chapter Two

HISTORY AND BACKGROUND

2.1 History of colouration

Adding colour to textile has been considered literally as adding colours to life. The application of colour to textiles has been known for thousands of years. Colour and pattern provide appeal for clothing and home textiles, and indicate use and quality. Colour in textiles makes an important contribution to the quality of life, reflecting personal preferences and sense of identity. In most cultures the colours of costumes worn for specific events have particular significance, indicating, for example, rank or role is considered to indicate both social status and standing (Smith and Block, 1982).

The ability of natural dyes to colour textiles has been known since ancient times. The earliest written record of the use of natural dyes was found in China dated 2600 BC. Chemical tests of red fabrics found in the tomb of King Tutankhamen in Egypt show the presence of alizarin, a pigment extracted from madder. Alexander the Great mentioned having found robes purple in colour dating to 541 BC in the royal treasury when he conquered Susa, the Persian capital. Kermes (from the Kermes insect) is identified in the bible book of Exodus, where references are made to scarlet coloured linen. By the 4th century AD, dyes such as woad, madder, weld, brazilwood, and indigo and a dark reddish-purple were known. Brazil was named for the wood found there (Pardeshi and Paul, 2002).

As early as 600 BC geometric designs were woven into wall hangings and rugs. Piece dyeing or application of dyes to the entire piece of cloth was practiced using natural dyes, two thousand years ago (Smith and Block, 1982). In Egypt, indigo has been found on cloth dating back to the fifth dynasty (2500 BC). Kermes was the first red dye used by the primitive man (Pardeshi and Paul, 2002). It was used by the Hebrews and is mentioned in the Old Testament. Henna is used even before 2500 BC. Saffron is mentioned in the bible. Promegranate was used as early as 2000 BC in Mesopotamia and 1500 BC in Egypt. Orchil, a violet dye, was discovered in Florence in 1300 AD and for hundred years or so Florence supplied all the Orchil to the rest of Europe (Pradeshi and Paul, 2002). The progression from drawings on cave walls to the vast array of coloured objects of the modern world has been a long, slow process, fuelled by an innate desire for beauty and variety, and powered by a growing knowledge of why some materials have the capacity to lend colour to other materials (Internet, 5).

Ancient Egyptian hieroglyphs contain a thorough description of the extraction of natural dyes and their application in dyeing (Rys and Zollinger, 1972). The basis for most of the dyeing methods used until the nineteenth centaury was established by the ancient Egyptians, who developed the application of plant extracts often in association with mordanting. Further developments extending over many thousand years led to rather complicated dyeing processes and high quality dyeings. Among these the following deserve special mention: Indigo, which was obtained both from dyers woad, indigenous to Europe, and from Indigofera tinctoria, a native plant of Asia; ancient purple, which was extracted from a gland of the purple snail by the process developed by the Phoenicians; Alizarin, on which Turkey red is hased, which was obtained from madder campeachi wood extract exported from Africa (Rys and Zollinger, 1972).

Figure 2.1 Peruvian textiles

Peruvian Textile of the coast of Peru, some of the best examples of textiles from the Pre-incan period have survived, buried in desert tombs, especially on the Paracas Peninsula, 2,500-year-old textiles have been perfectly preserved and reveal extraordinary technical expertise. Most are made of wool or cotton and are decorated with geometric patterns that sometimes represent animals and human figures (Fig. 2.1). These materials were often coloured with mineral and vegetable dyes (Smith and Block, 1982).

Some animal sources of dyes were shield scales or cochineal insects found on cacti in North and South America, and kermes, a scale insect found on oak trees near the Mediterranean; both produced reds and pinks. Purple was made from a mollusk and clothing made from it was so expensive only the royal family could afford it. It was extracted from a small gastropod mollusk found in all seas or from a crustacean called a Trumpet Shell or Purple Fish, found near tyre on the Mediterranean coast. Their body secreted a deep purple fluid which was harvested by cracking the shell and digging out a vein located near the shellfish head with a small pointed utensil. The mucus-like contents of the veins were then mixed together and spread on silk or linen. Estimates are that it took 8,500 shellfish to produce one gram of the dye, hence the fact this dye was worth more than its weight in gold (Pardeshi and Paul, 2002).

By the 15th century, dyes from insects, such as cochineal and Kermes, were becoming more common. By the 17th century, dyeing cloth "in the wood" was introduced in England: logwood, fustic, etc Indigo began to be grown in England, and Cudbear, a natural dye prepared from a variety of lichens, is patented. Another natural dye, Quercitron, from the inner bark of the North American oak, was patented in 1775.

Figure 2.2 Textile dyeing in Morocco

Textile Dyeing in Morocco is done in concrete or clay vats with techniques that have been used for centuries (Fig.2.2). The material may be treated again with another chemical so that its colour will not run or fade (Rys and Zollinger, 1972).

Natural colours are used not only in dyeing textiles but also in cosmetics. The use of cosmetics is worldwide and dates from the remotest antiquity. Although it is generally believed that cosmetics as they are now known originated in the Far East, the study of non-industrial cultures indicates the use of cosmetics in every part of the world. The war paint of Native Americans, the tattooing and scarification practiced by many people (Maori of New Zealand and numerous African cultures, for instance), and the use of woad (a plant dye used by ancient Britons to paint their bodies blue) are all forms of cosmetic used for psychological intimidation of the enemy as well as adornment. Egyptian women also developed the art of decorating the eyes by applying dark green colour to the lower lid and by blackening the lashes and the upper lid with kohl, preparation made from antimony or soot (Smith and Block, 1982).

The first uses of paint were entirely decorative. Thus, paint without a binder, consisting of iron oxide, was used for cave paintings about the 15 BC. In Asia, several pigments made from ores, prepared mixtures, and organic compounds were known about 6000 BC. Indigo, a pigment extracted from the indigo plant, was known to the ancient Egyptians, Greeks, and Romans, and to the Inca. Gum arabic, egg white, gelatin, and beeswax were the first vehicles used for these pigments (Pardeshi and Paul, 2002).

Until the late 19th century natural dyes were used for colouring weaving yarns. Information about dyeing practices before the 16th century is limited, but it is known that all dye matter came from mineral pigments, plant materials, or from animals and insects. Mineral pigments such as ochre (yellow or red), limestone or lime (white), manganese (black), cinnabar (red), blue azurite, green malachite, lead oxide (red), and lapis lazuli (blue) were probably the first materials used for dyeing. Vegetable dyes came from leaves, roots, bark, and, occasionally, fruit or flowers of plants. Woad, a plant of the cabbage family, and indigo, a bush from the pea family, were both used for blue dye. Four vegetable yellows were especially important i.e. saffron, safflower, weld, and fustic. Madder and redwoods had been used since ancient times for oranges, and browns and blacks came from logwood, betel nuts, walnut and butternut hulls, and a gum resin called cutch (Pardeshi and Paul, 2002).

2.1.1 Water colour painting

Water colour paints are produced by binding dry powdered pigment mixed with gum arabic, a gum, obtained from acacia trees, that solidifies through evaporation but which is soluble in water. Solid water colour can then be dissolved in water and applied to paper with a brush. Although water colour is a relatively modern type of paint, various water-based paints related to water colour have been used throughout recorded history. Ancient Egyptian painted papyrus scrolls may be considered the first water colours, and early oriental ink drawings are forms of monochrome water colour. In medieval Europe, water-soluble pigments bound with a thickener derived from eggs were used in illuminated manuscripts (Bhattacharya , Doshi , and Sahasrabudhe , 1998).

2.1.2 Tempera painting

Tempera Painting, method of painting in which the pigment is dissolved in water and "tempered", or mixed into a paste, with egg, casein, gum, or glycerine solution. The process of painting in tempera is the oldest method of painting known; the wall paintings of ancient Egypt and Babylonia, and of the Mycenaean period in Greece, were probably executed in tempera with a medium of egg yolk (Bhattacharya , Doshi and Sahasrabudhe , 1998). The use of tempera subsequently became widespread throughout Europe and reached its height in Italy.

2.1.3 Fresco painting

Fresco painting flourished in India. Famous examples include frescoes in Ajanta caves, painted between 200 BC and 650 AD, and at Ellora, dating from the late 8th century. Painting on plaster walls was also an ancient art in China; examples dating from the 5th century onwards, and depicting scenes from the life of the Buddha, survive in the Cave of the Thousand Buddhas, at Tun-huang. Ajanta Caves, group of about 20 caves carved out of the sides of a steep ravine near the village of Ajanta, in central India. These historic wall paintings, dating from between 200 BC and 650 AD, traces the development of painting styles during that time. Most of the wall paintings are based on the jataka stories or on events in the life of the Buddha (Internet, 6).

2.1.4 Oil painting

Traditionally, oil painting proceeds in stages. First the design is sketched on the ground in pencil or charcoal, or paint diluted with turpentine. Then broad areas of colour are filled in with thin paint. They are successively refined and corrected in thicker paint to which oil and varnish are added. The paint is usually applied with brushes made from stiff hog bristle, although softer brushes of badger or sable hair may be used. The process may require only a few sessions or extend over months or even years (Internet, 7).

2.2 Evolution of synthetic dyes

Synthesis of dyes created the demise of natural dyes at this stage. Today however practically all dyes are synthetic. They are prepared from aromatic compounds, for which then the only available source was coal tar. Hence the name coal tar dyes. W H Perkins made the first synthetic dye (mauve) in 1856. Mauve was an aniline dye. Perkin found a dye that would always produce a uniform shade and he pointed the way to other synthetic colors, thus revolutionizing the world of both dye making and fashion (Internet, 7).

As early as 1740, experiments had shown the possibility of synthesizing dyes. Naturally occurring indigo was treated with concentrated sulphuric acid by bath to produce a product, which was marketed as Saxe Blue (Carr, 1995). Picric acid which was obtained by Woulfe in 1771 by treating indigo with nitric acid was subsequently occasionally used for dyeing silk yellow but did not attain any significance. For this reason really incorrectly William H. Perkin, not Woulfe, was given credit for having produced the first synthetic organic dye (Rys and Zollinger., 1972). The initial attempts of this nature to produce synthetic dyes had no systematic basis for two reasons; firstly due to the general belief that only living organisms or God could prepare organic materials and secondly due to the insufficiency of chemical knowledge to base anything other than random excursions into chemical synthesis (Carr, 1995).

In 1856, English chemist, William H.Perkin, succeeded in obtaining by oxidation of a mixture of aniline bases, not quinine, which he had hoped for , but a violet cationic dye which he called Mauveine ; initially the yield was poor. If Perkin is to be regarded as the founder of the synthetic dye industry this is correct in the sense that, on the one hand, with the primitive means then at his disposal he was able to prepare a relatively pure and technically interesting product and, on the other hand, was able to develop its synthesis so that it could be used in large scale production (Rys and Zollinger, 1972).

The brilliant violet hue of Mauveine on silk immediately attracted much attention and stimulated other chemists to carry out similar experiments. In this way, in 1859, E. Verguin in Lyon discovered fuchsine, whilst the discovery of diazo compounds by P. Griess in England laid the foundation for the development of currently largest class of synthetic dyes, namely the azo compounds. The first true azo dye, bismarck brown, was developed by Martius in 1863 (Rys and Zollinger, 1972).

Using the discoveries of the William W. Perkins, BASF in Germany became one of the first companies to manufacture dyes from coal tar. Its specialty was the bright bluish-purple known as indigo. The attraction of BASF's process lay in the fact that it took coal tar, a byproduct of gas distillation, and transformed it into something that replaced a more expensive and unreliable organic substance. BASF's synthetic dyes were less expensive, brighter, and easier to use than organic dyes. Profits from these dyes were used to finance BASF's diversification into inorganic chemicals later in the century as well as new production facilities across the river in Ludwigshafen. This site still houses one of the largest chemical process industries in the world and textile dyes played a major role in these developments (Internet, 7).

In England ICI (Imperial Chemical Industries) now taken over by Akzo Nobel was another major company who worked with textile dyes. ICI first came out with the fibre reactive dye Procion, in 1956 which was a major success. The huge success of ICI's first Procion dye brought about a real revolution in the dye industry. All the major dye manufacturers began research programs to develop other types of reactive dyes. Today the leader is DyStar a company that was formed with units from BASF, Ciba etc. DyStar is the world's leading supplier of textile dyes. The company has by far the broadest product range on the market, covering almost all fibers and quality specifications (Internet, 8).

The ban on Azo dyes which originated from Germany changed the situation for synthetic dyes. The environmental issues and health aspects (carcinogenic, allergenic and poisonous issues) have slowed down the demand for all types of synthetic dyes. Today the switch over to natural dyes or the desire to go back to nature is providing natural dyes another opportunity (Internet, 8).

The developments in the knowledge of synthetic organic chemistry (leading to the preparation of new dyes, the discovery of new reactive groups, etc.), of the reaction mechanisms (leading to the optimisation of manufacturing processes) and of the techniques of application has ultimately led to the commercial availability of thousands of synthetic dyestuffs world over today.

The world wide synthetic dyestuff usage based on dye classes and the annual consumption of the cellulose dyes are shown in Tables 2.1 and 2.2 which give a general idea about the high quantities of chemicals released to the environment annually.

Table 2.1 World dyestuff usage (Holme, 2002)

Type of Dye	MT	% of Total Dye Usage
Sulphur	90	19
Direct	74	16
Vat	36	8
Reactive	60	13
Azoic	28	6
Acid	55	12
1:2 Premetallised	15	3
1:1 Premetallised	2	<1
Chrome	10	2
Disperse	85	18
Total	467	100

Table 2.2 Estimated annual global consumption of cellulosic dyes (Holme, 2002)

Type of dye	Usage per Annum (MT)		
	1988[a]	1992	2004
Sulphur	90000	70000	70000
Direct	74000	60000	68000
Vat	36000	21000	22000
Indigo	12000	12000	12000
Azoic	28000	18000	13000
Reactive	60000	109000	178000
Total	300000	290000	354000

[a] Not include China, India and Eastern Europe

2.3 Environmental aspects of synthetic dyes

Dyes are part of peoples' everyday colourful lives, and consumer goods like textiles often come directly into contact with human skin. Azo colourants are the most important class of synthetic dyes and pigments representing 60 % to 80 % of all organic colourants.

Not all azo dyes are harmful to human health, however some azo dyes under reductive conditions generate aromatic amines and these dyes are carcinogenic. The European Union, the USA and other developed countries have already banned the use of these carcinogenic dyes. In the USA another type of dye called vat dyes is no longer manufactured because of environment concern (WHO, 1987).

The effects of textile dyes and chemicals on human health are a serious health and environmental policy issue, especially in the newly industrializing and developing world. Because the coloured materials can be visualized by the naked eye, they have received more attention for treatment by the industry.

In recent years, the accumulated literature has revealed many adverse health effects of contaminated surface, ground water and soil from dyes and associated auxiliaries. Table 2.3 depicts dyes/chemicals used in the textile industry along with a treatment difficulty and pollution category that is in a range of 1 to 5, with 1 being the least harmful and 5 being the most harmful (Cooper, 1990).

Table 2.3 Pollution potential of some of the dyes and chemicals used in the textile industry

General chemical type	Difficulty of treatment	Pollution category
Alkali, Mineral acids, Natural salts and oxidizing agents	Relatively harmless inorganic pollutants	1
Starch sizes, Vegetable oils, Fats and waxes,Bio-degradable surfactants, Organic acids and reducing agents	Readily bio-degradable Moderate to High	2
Dyes and fluorescent brighteners, Fibres and polymeric impourities, Polyacrylate sizes, Synthetic polymer finishes, silicon	Dyes and polymers difficult to biodegrade.	3
Wool grease, PVA sizes, Starch ethers and esters, Mineral oils, surfactants resistance to biodegration and anionic & Non ionic softners	Difficult to bio-degrade Moderate BOD	4
Formaldehyde and N- methylol reactants, Chlorinated solvents & carriers, Cationic retarders & softners, biocides, sequestering agents, heavy metal salts.	Unsuitable for conventional biological treatment Negligible BOD	5

2.4 Textile colourants

Textile colourants impart colour that is not easily altered, to textile materials by becoming an integral part of it. Colouration of textiles improves the aesthetic quality and adds value to the product. Textile colouration is done by dyeing and / or printing on substrates such as fibre, yarn, fabric and garments. In dyeing, the dye is applied evenly by dipping fabric, fibre, yarn or garment in the dye solution. Printing is done on yarns, fabrics and garments by localised application of a dye paste on textiles. The textile colourants can be either natural or synthetic based on their origin. Synthetic colourants can be classified into two major types; namely, dyes and pigments while natural colourants mainly consist of dyes.

Dyeing requires specific conditions so that the colour can penetrate the fibres: the majority of dyes are naturally suited to particular fibres and are rejected by others. The other very important component of the dyeing process is the mordant. This is mainly used in natural dye applications to improve fastness properties. It is an intermediary agent, combining with certain natural dyes to bind the colouring matter to the fibre. Different mordants yield different colours in the same dye bath; while vegetable dyes yield warm, subtle colours, their density and colour fastness are determined by varying concentrations and skilful manipulation of the mordants. The matter used for dyes and for mordants is the most crucial aspect in determining the colour as well as the effects on health and environment. Consciousness about the environment and the health has led to an evaluation of the matter used for dyeing and for mordants (Internet, 8).

A textile dye should have the following properties:

1. Must be absorbed by the substrate
2. Give satisfactory colour yield
3. Economical in its application
4. Satisfactory fastness
5. Be non toxic
6. Be environmentally friendly

2.5 Chemical basis of textile colouration

Prior to the sixteenth century dyeing was a secret and a closely guarded technology. For example, the production in Turkey of the famous Adrianopolis or Turkey red by the application of the extraction of the madder root to cotton, mordanted with alum was a secret which was retained for 250 years, (Carr, 1995). All dyeing recipes of this age were the result of tedious experimentation carried out by the dyers without any chemical knowledge. However, with the expansion of trade and travel that accompanied the European renaissance and, more particularly, the development of the printing press, there was an explosion of knowledge and information, which exposed much of the mystery to the light of day. Many works were published in that period detailing dyeing and colouring procedures for different textiles, largely for domestic use. Of special importance, in that it was directed towards the industrial dyer, was The Plictho of Giovanventura Rosetti, published in 1548, which records, in addition to many dyeing recipes and methods of plant and other extracts, procedures for the preparation of many important chemicals such as hydrochloric acid (Carr, 1995). With the expansion of knowledge and great increases in the volume of production that followed this period came the demand for more reliable quality control, and as a consequence the demand for greater understanding, and the beginnings of scientific investigation into the phenomena involved.

Dufay de Cisternay, who is regarded as the founder of the modern dyeing theory, published the first scientific account of dyeing processes based on physical and chemical ideas (Carr, 1995). The work of a succession of chemists following Dufay, made way to the current knowledge to account for the properties of textile colourants, mordants and other assistants involved in textile industry, and to the concepts of dyeing available today such as the conception that dyeing is a physical binding process on a molecular scale (Carr, 1995). With the advancements in science and technology, many concepts have been brought forward to explain the process of textile dyeing to the molecular level. Some of the major concepts are being explained below.

(a) The colouring agents (dyes and pigments) should contain chromophores, which are capable of selective absorption of frequencies from the visible spectrum of radiation and as a result, are responsible of its own colour as well as of the substrate.

(b) In any dyeing process regardless of the type of dye applied, there are three major stages involved namely, adsorption on the fibre surface (take up or exhaustion), diffusion into the fibre and fixation or anchoring to the dyeing sites of the fibre. Adsorption is affected by factors like electro potential forces, temperature and agitation where as diffusion depends on molecular size, porosity and dye concentration at the interface.

(c) The fixation or anchoring involves one or more of the following principles (Shenai, 1994).

Attachment of the dye in the fibre by certain physical forces like Van der Waals forces, hydrogen bonds and electrostatic interaction

Mechanical trapping of the insoluble dye in the fibre

Dissolution of the dye in the fibre

Chemical reaction of the dye molecule with the fibre to form covalent bonds

2.6 Classification of synthetic textile colourants

Studies in the analysis of natural colourants in textile are a fascinating subject, which started as early as 1930. One of the first chemists who analysed natural dyestuffs was the French Chemist Pfister who used a microchemical analysis in which the result was achieved hy colour reactions with different chemicals. The method was highly time consuming and not very reliable. Abraham's and Edelstein reported a method for extraction and identification of various natural dyes viz. alizarine, indigo, 6, 6 dibrommoindigo, carminic acid (extracted from cochineal), yellow dye saffron, etc., using infrared special analysis. They also reported that the method could be extended to analyse dyes in ancient dyed cotton, linens and silks.

There are several ways for classification of dyes. It should be noted that each class of dye has a very unique chemistry, structure and particular way of bonding. While some dyes can react chemically with the substrates forming strong bonds in the process, others can be held by physical forces. Some of the prominent ways of classification are given here under.

(a) Organic/Inorganic

(b) Natural/Synthetic

(c) By area and method of application

(d) Chemical classification- based on the nature of their respective chromophores

(e) By nature of the electronic excitation (i.e, energy transfer colourants, absorption colorants and fluorescent colourants).

(f) According to the dyeing methods

- Anionic (for Protein fibre)
- Direct (Cellulose)
- Disperse (Polyamide fibres)

However the most popular classification is the one that is advocated by the US International Trade Commission. This system classifies dyes into 12 types (Internet, 9).

Table 2.4 Classification of dyes according to the dyeing methods

Group	Application
Direct	Cotton, cellulosic and blended fibres
Vat dyes	Cotton, cellulosic and blended fibres
Sulphur	Cotton, cellulosic fibre
Organic pigments	Cotton, cellulosic, blended fabric, paper
Reactive	Cellulosic fibre and fabric
Disperse dyes	Synthetic fibres
Acid dyes	Wool, silk, paper, synthetic fibres, leather
Azoic	Printing inks and pigments
Basic	Silk, wool, cotton

Textile colourants can be classified into two major classes namely; dyes and pigments. Both dyes and pigments are also used to colour surfaces and substances other than textile materials; the difference between the two classes is in the methods and techniques used for colouring the substrates. Pigments are finely ground colour particles dispersed through a carrying base and take effect by using spread over the surface. Dyes are water-soluble colours or they can be converted into water-soluble compounds and under the prerequisite conditions for the class of dye, the colour is absorbed by the textile fibres (Green, 1972). In textile industry dyes are classified by both chemical type and method of utilization. A general classification of colouring materials is given in Figure 2.3.

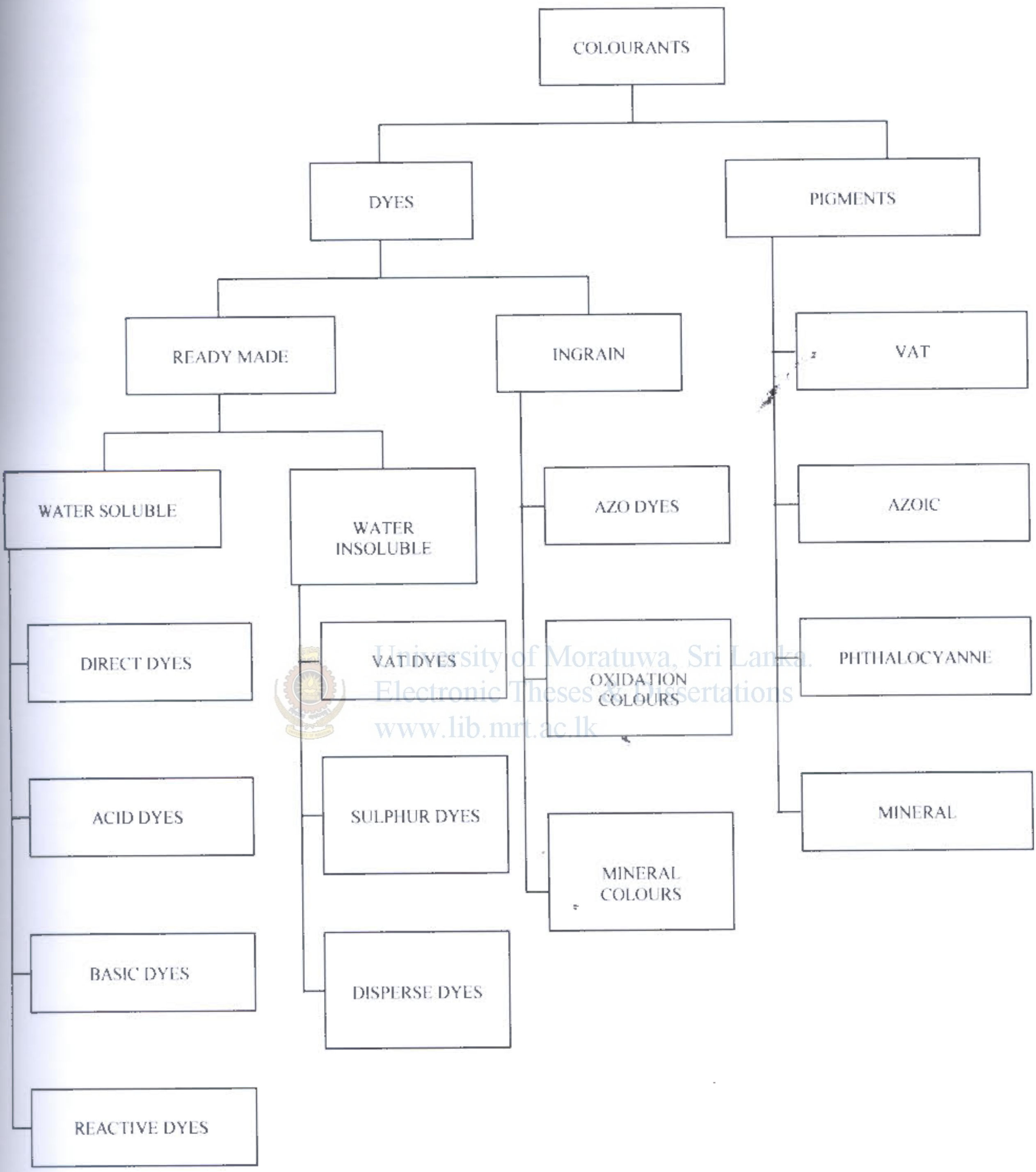

Figure 2.3 Classification of textile colourants (Shenai, 1994)

2.7 Natural dyes

With the discovery of the first chemical dye in the mid nineteenth century, natural dyestuffs were slowly superseded. Until this time both the dyer and the printer had been completely dependant on the natural dyestuffs (Green, 1972).

The available literature puts the number of plants (Gulrajani *et al.*, 1992) species yielding dyes to be around 300, but chemistry and availability of only few plants have been investigated so far. A survey of indigenous flora should be made in order to determine the availability of dye yielding materials like fruits, flowers, leaves and barks from Sri Lankan forests rather than the use of tree plants where the dye is extracted from roots. For this, different plant parts (Chatopadhyay *et al.*, 1997) may have to be scanned for their dye contents. Promotion of use of renewable species of dye bearing plants with their growth of natural dye production, especially growth of plant species, which simultaneously offer other marketable products, should be given priority. A choice of flora yielding dyes more than one important product, of which one is a dye would be of agronomical benefits (Chavan, 1995; Jeyakodi, 1996).

The chemical screening of plant material of the Sri Lankan forests particularly those which are reported to be vegetable wastes of forests is thus a necessity. Particularly the herbs and shrubs may be studied in detail with regard to their agronomical practices, chemical modification and purification of dyes. Another aspect of sourcing is to select plants, which offer more than one product, for example, a resin is extracted from lac and the residue then yields the dye. Many essential oil producing flowers (Prayag, 1994) give perfumes and subsequently dye can be extracted from the remains such as tegetus, such activities should be encouraged. However natural origin may not necessarily be always guaranteed freedom from toxicity. The migratory or residual component from processing aids, auxiliaries and mordant may at times lead to handling hazards. The natural dyes need to be standardized.

However the standardization of natural dyes (Vineet and Bharti, 1998) is a difficult proposition as the percentage of the colouring compounds in a given plant changes from place to place, soil to soil, variety to variety and even plant to plant. A method to standardize (Ishrat , 1993) natural dye in available powder or paste from which a known measure of its standard depth on a given fabric is obtained, needs to be developed. However for this it is important to identify:

(a) Safe natural dyes

(b) Use of safe mordants

(c) Eventually aim for ecofriendly textiles.

(d) Marketable natural dyes-powder, concentrate or paste.

Ecofriendly textile fibres which when transformed to garments should at no stage of its processing, printing, colouring and production cause any environmental pollution, in the true sense (Glovev and Piersce, 1993). It is the pollution control, which has diverted the attention of users towards nature. Both the government and people are getting aware of this problem. The use of natural dyes is not new; it's just that there is a revival of trends in advanced scientific technology.

Although about fifteen natural dyes are being used by the department of textiles industries, Sri Lanka, they are being marketed in crude form. Purification, colour fastness, colour variety with mordants and stability have not been still explored. There is always a continuous need to explore newer natural dyes for getting full gamut of colours.

With the revival of interest in natural dyes and ecofriendly textiles, a great deal of interest and potential lies in this area. (Anon, 1998; Howes, 1953). Alternate methods of isolation, collection, and standardization need to be developed. Prima facie, natural dyes are more expensive than synthetic dyes. However this handicap can be overcome by using wastelands be used for the cultivation of plants, which are dye sources. This would enable bulk production of the dye subsequently, by lowering the production cost. A great deal of planned chemical research is called for understanding the nature of active chromophoric substance, which in turn should give clues for the chromophoric activities. New mordants could be chelated to show fastness to light and washing properties. It is with this intention that the explorations of newer plant species for natural dyes were tried.

There are numerous plants, the extracts from which are capable of colouring wool, silk, cotton and linen. For various reasons such as dyeing behaviour, fastness properties or their biological availability, only a few have found application as dyeing agents (Pardeshi and Paul, 2002). Tannin is closely related to dyestuffs and generally occurs in plants as an excreta in the bark and other parts, which may be either employed direct or used for extracting tannin in a concentrated form (Munidasa, 1988). There is no doubt that the use of natural dyes on a commercial scale is gradually increasing. There is no difficulty in accepting the challenge of retailer selling merchandise dyed or printed with natural dyes (Horrocks, 1996).

There are very few natural dyestuffs, which have a natural affinity to textile fibres and become permanent without a preliminary treatment. Dyes with this property are being described as "substantive". In the majority of cases the dyestuff will colour the cloth only when assisted by a chemical compound, a mordant. The mordants used in dyeing are metallic salts, although some are acidic (Horrocks, 1996).

2.8 Classification of natural dyes

Natural dyes can be broadly classified in various ways, out of which the earliest one being according to the alphabetical order. Later with the developments in chemical knowledge classifications were based on chemical structure. Besides these methods of classification, natural dyes have been listed on the basis of their botanical names, common names, origin, application and colour.

2.8.1 Classification based on origin

Natural colouring matters are broadly classified into four categories based on their origin:

(a) Vegetable origin

The colouring matter is derived from root, leaf, bark, trunk or fruit of plants

(b) Animal origin

Lac, cochineal and kermes have been the principal dye yielding insects.

Cochineal

A good example is cochineal, which is a brilliant red dye produced from insects living on cactus plants. Cochineal, red dye derived from the dried bodies of female scale insects, dactylopius coccus. The properties of the cochineal bug were discovered by pre-Columbian Indians who would dry the females in the sun, and then ground the dried bodies to produce a rich, rich red powder. When mixed with water, the powder produced a deep, vibrant red coloring. Cochineal is still harvested today on the Canary Islands.

Lac

Lac, resinous substance secreted by the lac insect on the twigs and young branches of certain trees. The lac insect, Kerria lacca, is of the Coccoidea super family (scale insect). Females insert their long proboscises into the bark of the twigs or branches, drawing their foodstuff from the sap. They exude a secretion that accrues and coalesces, forming hard, resinous layers that completely cover their bodies. The ovaries contain a crimson fluid called lac dye, resembling cochineal.

Crude lac, known as stick-lac, consists of the resin, the encrusted insects, lac dye, and twigs. When crushed and washed free of the dye, twigs, and insects, it becomes granular and is known as seed-lac or grained lac. After melting and further purification, the resulting lac resin is solidified into thin layers or flakes that constitute commercial shellac. Shellac varies in colour from yellow to deep orange.

(c) Mineral origin

Various inorganic metal salts and metal oxides. Ocher is a dye obtained from an impure earthy ore of iron or ferruginous clay, usually red (hematite) or yellow (limonite). In addition to being the principal ore of iron, hematite is a constituent of a number of abrasives and pigments.

(d) Lichen origin

Used to extract dyes such as litmus, orchil, etc (McGrath, 1977; Macmillan, 1943).

Apart from these four origins, which give natural dyes essentially from natural sources, there are two other origins of natural dyes, which are not necessarily natural origins of them (Vankar, 2002). They are,

(1) Chemical synthesis

This involves synthesis of dyes with molecular structures identical to those of natural dyes (Vankar, 2002).

(2) Tissue or cell culture by DNA Transfer Biotechnology

Certain fungi such as *Drechslera* and *Trichoderma* produce anthraquinone derivatives as secondary metabolites. As anthraquinones are a very important class of dyes, exploiting the fungi would be advantageous over their chemical synthesis. If genetic modifications can be achieved, it is possible to develop fungi that produce substituted anthraquinones. It is possible that these compounds may be engineered by genetic modification. The two most eco-friendly approaches to natural dye manufacturing are:

- By enriching the natural sources by efficient cultivation techniques and extraction techniques.
- Bio-technological methods to obtain safer natural structural entities in an inherent natural way (Vankar, 2002).

2.8.2 Classification based on chemical nature

Natural colouring substances can be classified into seven categories according to their chemical nature (Pardeshi and Paul, 2002; Vankar, 2002).

(a) Indigoids: This is perhaps the most important group of natural dyes. The dye is extracted from woad and *Indigofera tinctorial* plants.

(b) Anthraquinones: Some of the most important red dyes are based on Anthraquinone structure. They are obtained both from plants as well as from insects or animals. These dyes are characterized by light fastness ratings.

(c) Alpha-hydroxy-Naphthoquinones: The most prominent member of this class of dyes is henna obtained from the leaves of *Lawsonia inermis*. Another similar type of dye is extracted from the shell of unripe walnuts.

(d) Flavones: Most of the yellow colours are derivatives of hydroxyl or methoxy substituted flavones or isoflavones.

(e) Dihydropyrans: These are the principal colouring bodies of logwood and are the historically most important natural dyes for dark shades on silk, wool and cotton. Substituted dihydropyrans like hematin and its leuco form, hematoxylin are closely related in chemical structure to the flavones

(f) Anthocyanidins: The naturally occurring members of this class include carajurin, obtained from the leaves of Bignonia chica and Awobanin. It dyes silk in blue shade.

(g) Carotenoids: The class name carotene is derived from the orange pigment found in carrots. The prominent natural dyes based on carotenoid structure are annatto and saffron.

2.8.3 Classification based on application

Natural dyes can also be classified according to the basis of its application. Here the natural dyes are classified into two groups, namely, substantive and adjective dyes. Substantive dyes have natural affinity to textile fibres and therefore do not need any treatments to fix the dye to the fibre (e.g. indigo, orchil, turmeric, etc.). The adjective dyes have no natural affinity to the textile material and hence are capable of dyeing material only after mordanting the material with metallic salts or with the addition of such a salt to the dye bath. The substantive dyes can be further classified as direct (turmeric, safflower on cotton; safflower on wool and silk), acid (saffron on wool and silk) and basic (berberine on silk and wool) (Pardeshi and Paul, 2002).

2.8.4 Classification based on colour

Natural dyes can also be classified based on the colours they produce. Various natural dyes could present all the colours of the visible spectrum (Vankar, 2002). On this basis they are broadly categorised into two, namely, monogenetic and polygenetic (Pardeshi and Paul, 2002). The monogenetic type of dyes produce only one colour irrespective of the mordant applied; where as, the polygenetic dyes produce different colours depending on the mordant employed e.g. alizarin, logwood, cochineal and fustic (Humel, 1888). In the colour index dyes are classified according to the chemical constitution as well as the major application classes of sixteen. Within an application class dyes are arranged according to hue. Natural dyes form a separate section in the Colour Index (CI). In this section the dyes are arranged hue wise. The dyes of each hue are given in Table 2.3. Some dyes produce more than one hue and treating with different mordants can alter the hue of a particular dye. If the dye is of plant origin, the colour may vary depending on the soil properties, part of the plant, season of harvesting, cultivation practices, etc.

Table 2.5 Number of natural dyes for different hues (Pardeshi and Paul, 2002)

C.I.Natural	No of Dyes	Percentage
Yellow	28	30.4
Orange	6	6.5
Red	32	34.8
Blue	3	3.3
Green	5	5.5
Brown	6	6.5

2.8.5 Sources of natural dyes on the basis of colour

Normally natural dyes (Mell, 1977) are extracted from the roots, stems, leaves, flowers, fruits of various plants, dried bodies of certain insects and minerals. Some plants may have more than one colour depending upon which part of the plant one uses. The hue shade of the colour a plant produces will vary according to time of the year it is picked, how it was grown, soil content etc. Minerals in the water used in a dye bath can also alter the colour. Some natural dyes contain natural mordants.

(a) Red colour

Unlike the wide abundance of yellow colour in nature, most red dyes are hidden in roots or bark of plants camouflaged in the bodies of dull grey insects. Although, sources are limited, they occur in large groups in a single plant. Cocheneal is an important red colour and it is the brightest of all the available natural red dyes. Manjistha (Goel, 1997), Sappan and Kusumbar are among the vegetable sources that give red colour. Lac (Suri *et al.*, 2000) and kermiz are among the animal sources, which also give red colour.

Table 2.6 Global sources of natural red dyes (Bhawna, 2001)

Name	**Botanical name**	**Parts used**	**Mordants**
Safflower	*Carthamus tinctorious*	Flower	N/A
Alkanate	*Morinda citrifolia*	Root, Bark	Alum
Blood root	*Sanguinaria canadensis*	Root	Alum
Anchusa	*Anchusa tinctoria*	Root	Alum
Ladys bed straw	*Galium verum*	Root	N/A
Cochineal	*Insect*	Dried body	Alum/Tin

(b) Blue colour

Commonly there are three natural blue colour dyes that is natural indigo (Elters, 1996), suphonated natural indigo and the flowers of the Japanese (Tsuykusa) used mainly for making some special kind of papers.

Table 2.7 Global sources of natural blue dyes (Bhawna, 2001)

Name	**Botanical Name**	**Parts Used**	**Mordants**
Water Lily	*Nymphaea alba*	Rhysomes	Iron
Woad	*Isatis tictoria*	Leaves	N/A
Sunt Berry	*Acacia nilotica*	Seed Pods	N/A
Piret	*Ligustrum*	Mature berries after frost	Alum and Iron

(c) Black colour

Black colour can be obtained from harda, custard apple etc.,

Table 2.8 Global sources of natural black dyes (Bhawna, 2001)

Name	**Botanical Name**	**Parts Used**	**Mordants**
Babla	*Acacia Arabia*	Bark	Alum
Alder	*Alnus gultinosa*	Bark	Iron
Rofblamala	*Laranthus pentapetulus*	Leaves	Iron
Custard Apple	*Amona reticulate*	Fruit	N/A
Harda	*Terminalia chebula*	Fruit	Iron

(d) Yellow colour

Yellow is the loveliest and perhaps the most abundant of all hues in nature. The numbers of plants, which yield yellow dyes, are much higher than those yielding other colours (Shivakumar *et al.*, 2001) marigold (Gurumalle, Raja and Prabu, 1998) Turmeric (Saxena, Vardarajan and Nachane, 2001) , Teak (Patel *et al.*, 1999), pomegranate (Ansari, Thakur and Joshi, 1999) and heart wood part of jak fruit (Nanda and Patra, 1999) give yellow colour.

Table 2.9 Global sources of natural yellow dyes (Vandana, 2000)

Name	**Botanical Name**	**Parts Used**	**Mordants**
Turmeric	*Curcuma Longa*	Ground tubers	N/A
Golden rod	*Solidago grandis*	Flower	Alum
Agrimony	*Agrimonia eupatoria*	Leaves, Stem	Chrome
Horsetail	*Equisetum genus*	Leaves,stalk	Tin
Fustic	*Chlorophora tinctoria*	Wood	Alum
Harda	*Teminalia chebula*	Fruit	Alum
Pomegranate rind	*Punica granatum*	Fruit	Alum
Black Berry	*Rubus fructicosa*	Old stem and leaves	N/A
Weld	*Reseda luteola*	Bark	Alum
Chir	*Pinus roxburghii*	Whole plant	N/A
Ling Leather	*Calluna vulgaris*	Tops of flowering plant	Alum,Iron
Hazel	*Corylus avellana*	Mature Catkins	Alum
Corn Marigold	*Chrysanthemum*	Flowering tops	Alum

(e) Green colour

Green colour is hard to get directly from natural source; normally it is obtained by mixing indigo and yellow however cannas and lily give nice florescent green.

Table 2.10 Global sources of natural green dyes (Bhawna, 2001).

Name	**Botanical Name**	**Parts Used**	**Mordants**
Lilly	*Convallaria majalis*	Leaves and Stalk	Ferrous sulphate
Stinging	*Urtica diocia*	Leaves	Alum
Wild St.John's wort	*Hypericum perforatum*	Whole plant except root	Alum or no mordant

(f) Brown colour

Similarly brown colour can be obtained from cutch, sumach and eucalyptus.

Table 2.11 Global sources of natural brown dyes (Bhawna, 2001)

Name	**Botanical Name**	**Parts Used**	**Mordants**
Sumach	*Rhus species*	Berries	Alum
Cutch	*Catechu*	Wood	Iron
Auch	*Mordina tinctoria*	Leaves	N/A
Marigold	*Tegutus species*	Flowers	Chrome
Black berries	*Rubus fructicosus*	Berries	Iron
Lodh	*Symplocos reacemosa*	Bark	N/A

(g) Orange/Peach colour

Orange/ Peach colour can be obtained from Dahlia and annatto (Gulrajani *et al*., 1992) using different mordants.

Table 2.12 Global sources of natural orange/peach dyes (Bhawna, 2001).

Name	**Botanical Name**	**Parts Used**
Babla	*Acacia Arabia*	Bark
Alder	*Alnus gultinosa*	Bark
Rofblamala	*Laranthus pentapetulus*	Leaves
Custard Apple	*Amona reticulata*	Fruit
Harda	*Terminalia chebula*	Fruit

2.9 Extraction methods of natural dyes

Vegetable dyestuffs owe their origin to the presence of small quantities of certain chemical substances secreted in the plant tissues, which are extracted by processes of fermentation, boiling or chemical treatment (Macmillan, 1943). Almost all plant extracts used as dyes or dye intermediates were extracted by means of boiling and the only reported exceptions been the indigo dye extracted from woad and indigo plants by fermentation process (Green, 1972). Mainly natural dyes are extracted in two different methods, boiling and fermentation. Out of these plant colours are almost always extracted by boiling where the two notable exceptions being woad and indigo, which are processed by fermentation. Even in boiling, plant parts such as fibrous roots and barks should be steeped in water first; the depth of shade is dependent on the length of time they are left steeping (Green, 1972).

Before the introduction of indigo into Western Europe in the sixteenth century, woad (*Isatis tinctoria*) yielded the only blue colour used and was cultivated specifically for the pigment it contained. Indigo dye is obtained from a shrub (*Indigofera tinctoria*), a native of India, although it grows widely elsewhere, except in Europe. The colouring matter is found only in the leaves, which are cut and fermented in tepid water (Green, 1972).

To produce the dye indigo from *indigofera* plants, fermentation is carried out in large masonry tanks. The green crop is placed in these tanks and weighted down with planks. Water is laid on so as to cover the planks, which is subjected to a process of fermentation and churning. Fermentation is allowed to go on for 12-16 hours and stopped when the leaves become a pale colour. The liquid is run off by means of a tap at the bottom of the tank, into a second tank or cistern, and is kept constantly agitated by wading coolies beating with paddles, or by a mechanical contrivance, for 2-3 hours, after which the indigo settles in the bottom in the form of hluish mud. This after draining off the water, is put into hags, which are hung to dry, and afterwards cut into cubes about 3 inches square, stamped, and further dried (Macmillan, 1943).

2.10 Natural dyeing

2.10.1 Advantages of natural dyes

Today dyeing is a complex, specialized science. The market is becoming interested in changing to natural products. Customers more aware of environmental issues are now demanding natural products, naturally sourced. If a fashion company introduces a new line of clothes produced with a natural fibre, the naturally sourced dye is needed to complete the green label. Natural dyes can offer not only a rich and varied source of dyestuff, but also the possibility of an income through sustainable harvest of the dye plants. Also, they have a far superior aesthetic quality, which is much more pleasing to the eye. Nature provides a wealth of plants which will yield their colours for the purpose of dyeing. New ways of dyeing the wool and silk with the natural dyes are fairly cheap compared to the artificial, chemical dyes. The raw materials for production of natural dyes are abundantly available (Internet, 10).

While working with the natural dyes, if 1.0 kg of fiber, directly spent expenses include the preparation of the dyeing products, expenses on drying and threshing them, the expenses on washing and dyeing, and the expenses on the very process of dyeing. The total sum of the expenses compared to the total sum of the expenses on working with the artificial and chemical dyes, i.e. buying the artificial, chemical, color changing chemical elements and the process of dyeing with those substances, is much less.

2.11 Mordants and mordanting

Natural dyes are either substantive, needing no mordant, or adjective requiring one. The majority of natural dyes need a chemical in the form of metal salt to create affinity between the fibre and the pigment. These chemicals are known as mordants. Dyeing with these mordants is called as 'mordanting' (Shenai, 1994). Mordants are considered as an intergral part of the natural dyeing process by the dyers of natural dyes. This is an anomaly, which continues to be pertetuated by different authors and practitioners of natural dyeing (Chattopadhyay *et al.*, 1997).

A mordant is a substance used to set dyes on fabrics by forming an insoluble compound with the dye. It may be used for dyeing fabrics, or for intensifying stains in cell or tissue preparations. A mordant is either inherently colloidal or produces colloids and can be either acidic or alkaline. The mordant has affinity for both fibre and the dye. Thus those dyes, which do not have any affinity for a fibre, can be applied by using mordants. Thus improves the staining ability of any dye along with increase in fastness properties. Mordants form an insoluble compound within the fibre. The mordant dye includes those differing widely from colou (Anon, 1998).

A close look at the chemical structures of the natural dyes isolated would show that these dyes are capable of forming complexes with metals or not. Many natural dyes have good affinity for the fibre; however their uptake as well as hue can be further modified by pre-treatment or post treatment with the metal salts called mordants. Mordants are classified in three classes:

(a) Tannins and Tannic acid

(b) Metal salts or metallic mordants

(c) Oils or oil mordants

2.11.1 Tannins and tannic acid

The term tannin was introduced by Seguin in 1796 to describe the substances present in number of vegetable extracts which are responsible for converting purifying animal skins in to the stable product by tanning process. Dyeing with natural dyes; tannins play a very important role. Among the naturally occurring mordants are the tannins. It improves the affinity of fibres towards different dyes. With different natural dyes it gives different shades like yellow, brown, grey and black.

Pre-treatment with tannic acid followed by metal salt treatment to cotton introduces additional hydroxyl and carboxyl groups in fibre. These groups by themselves can only increase the dye uptake. A subsequent treatment of the tannin treated cotton with metal salts such as alum introduces the aluminium ions in the fibre. The tannin treated cotton at the hydroxyl or carboxyl groups absorb these ions, either by forming metal – complex or metal salts. These metal ions then provide sites for the mordant dyes. Hence introducing metal ions in the fibre either directly or as tannin metal complexes can increase the affinity of cotton towards mordant dyes. It is apparent that the tannins by themselves do not act as mordants but tannin-metal salt combination can only act as a mordant for the natural dyes (Anon, 1998).

The stability of the tannin-fibre bond depends on the pH, ionic strength and metal chelators. The vegetable tannin may be divided structurally into two distinct classes depending on the type of phenolic group involved and the way they are joined together. Catechin (a powerful, water soluable polyphenol and antioxidant that is easily oxidized) was first isolated 140 years ago by Runge from the tannins of *Acacia catechtu*. *Acacia catechu* is a deciduous tree of 25m height and is native to Sri Lanka, Burma and India.The colouring component obtained from this tree is popularly known as 'Cutch', which produces copper red colours on cotton, wool and silk having very good washing and light fastness (Vandana, 2002).

2.11.2 Application of tannins

Cotton has very low affinity for most of the natural dyes. The tannins play an important role in cotton dyeing and are largely used for preparing cotton, so as to enable it to retain colouring matter permanently. Tannic acid is the best "tannin" for the mordanting of cotton since it is the purest of all, and does not contain the natural impurities, which are partly ineffective, partly injurious to mordanting and dyeing. Tannic acid is extensively used in the dyeing of the light and brilliant shades. For dark shades extracts of gallnut, sumach and myrobalan are largely employed. For the light shades 2-5 % (owf) tannic acid is used, while for dark shades 5-10 % tannic acid is required (Vandana, 2002).

2.11.3 Metal mordants

It is the general opinion that the metallic salts, because of their corrosive nature, make the textile rough, opened their pores, and made them more receptive to the colouring matters. A mordant is now regarded as a chemical which can allow certain dyes with no affinity for the fibre to be fixed, where the dyes are capable of being directly used and the mordants help to produce faster shades by forming an insoluble compound of mordant and dye stuff within the fibre (Internet, 11).

Besides metallic salts, tannins and oils are also used as mordants. Generally cotton is mordanted with these mordants. These mordants impart affinity for basic dyes. Cotton on further treatment with tannic acid can absorb all the types of metallic mordants. Further the metallic mordants form complex with the carboxylic groups of tannic acid. The cloth thus treated can be dyed with the mordant dyes easily and successfully (Gulrajani *et al.*, 1992).

The common mordants are Alum (Potassium aluminium sulphate), Copper (Copper sulphate), Chrome (Potassium dichromate), Tin (Stannous chloride), Iron (Ferrous sulphate) and Tannic Acid. These are used in various combinations with assistants for mordanting wool, cotton, linen and silk. Potasium aluminium sulphate , commonly called alum. Alum is a white powder that is safe to have and easy to use. Alum produces bright shades and gives relatively good light-fastness. If used in excess, alum will make wool feel sticky, so it is recommended that one measures accurately. If you use an aluminum pan it will contrihute to the brightness of the colour, but will not guarantee the colour fastness.

- Iron - Ferrous sulphate is a greenish powder that dissolves to make a rusty-coloured liquid. You can also simmer dyes in a cast-iron pot or use rusty nails or iron shavings. Iron produces dark, dull colours that are fast. Iron used in excess on wool will weaken the fibers and causes yarns and fabrics to wear out prematurely.
- Copper - Use of copper sulfate gives a beautiful blue colour when dissolved in water. Copper darkens colors and gives a greenish cast. It provides good colour-fastness and is not as hard on fibers as iron. A solid copper pot will make an excellent copper mordant.
- Tin - Stannous chloride is a white powder that will dissolve into a clear solution. It brightens colours, sometimes producing a remarkable "unnatural" effect. Tin provides good fastness, but can make wool feel brittle and rough. It is best to use alum as the primary mordant and just a pinch of tin for brightness.
- Chrome - Bright orange crystals known as potassium dichromate make a bright orange solution. If exposed to light, this solution becomes unstahle, so it should be kept with a lid on the container and not exposed to light. Chrome in any form is toxic, so treat this mordant with respect and caution. Chrome gives good bright colors that are very fast and it gives wools a soft texture (Internet, 12).

2.11.4 Oil mordants

Oil mordants are used mainly in the dyeing of turkey red colour from maddar (*Rubia cordifolia*). The main function of the oil mordant is to form a complex with alum used as the main mordant. Since alum is soluhle in water and does not have affinity for cotton it is easily washed out from the treated fabric. The naturally occuring oils contain fatty acids such as palmitic, stearic, oleic, ricinlic etc, and their glycerides. The sulfonated oils, which possess better metal binding capacity than the natural oils due to the presence of sulfonatic acid group binds metal forming a complex with the mordant dye to give superior fastness and hue.

2.11.5 Mordanting

In the application of the dyes, different techniques of mordanting and post-treatment were used to improve colour fastness properties. As a result, a broad set of variations in the dyeing recipes is given in the literature, and an optimization of the dyeing conditions with regard to the type of natural dye is quite common. The numerous variations of plant sources and dyeing processes proposed in the literature make an introduction of natural dyeing into full-scale technical dyeing processes rather difficult.

The chemistry of dyes to fibres is complex; it involves direct bonding, H-bonds, hydrophobic interactions. Mordants to this effect increase binding of dye to fabric by forming a chemical bridge. The mordant has affinity to both fibre and the dye. Thus those dyes, which do not have affinity for a fibre, can be applied by using mordants. Thus impoves the staining ability of any dye along with increase in fastness properties. Mordant forms an insoluble compound of the dye within the fibre.

2.12 Fastness properties of natural dyes

It is a fundamental requirement that coloured textile should withstand the conditions encountered during processing, following colouration and during their subsequent useful life (Venkataraman, 1978; Stevenns, 1979; Taylor, 1986; Gulrajani *et al.*, 2001; Seerangarajan , 2001). When a coloured textile is subjected to particular conditions, e.g. light or washing, one or more of several things may happen. As far as the colour of the material is concerned there may be alteration in all three. A red material may become paler, yellowish and duller. Further under certain conditions, adjacent white material may become coloured and coloured material may acquire new colour due to the transfer of dye from the original dyed material.

The colour fastness of coloured textile is therefore, defined as its resistance to these changes when subjected to a particular set of conditions. It follows that colour fastness must be specified in terms of the changes expressed in terms of their magnuitude. Fastness properties are divided into two classes: (Vandana, 2002).

(a) Fastness properties of natural dyes.

(b) Fastness properties of dyed material.

In early times clothing was infrequently washed and the fading of colours on clothing was accepted as inevitable. A study of the older literature shows that early in the history, man was aware of the fleeting nature of natural dyes available to him and was perpetually making efforts to improve the fastness properties of these dyes. Pliny , writing in the first centaury AD, records in great detail a method by which the

Egyptians smeared white cloth with a series of 'colourless drugs' (mordants) and plunged the whole into boiling dye bath. After the dyeing was completed, the cloth was multi coloured, the variations in hue being dependant on drugs that had been placed. There is evidence that Egyptians had learned this technique of mordanting from India. Thus as far as first century, man was using mordants to improve the fastness of his dyeing and for shade developments (Vandana, 2002).

Although mordanting and certain after treatments improved fastness, the inherent instability of the chromophores of the natural colouring matters resulted in low fastness to washing and light. Old textiles dyed with natural dyes have acquired an overall brownish hue. Greens produced by over dyeing indigo with a natural yellow dye inevitably fade to a bluer hue because of the higher light fastness of the indigo component. These effects are readily observed in old tapestries.

In recent as well as in classical studies it has been reported that most natural dyes have poor light stability, and hence the colours in museum textiles are often different from their original colours. It has also been observed that some natural dyes undergo marked changes in hue on washing, shown to be attributed due to even small amounts of alkali in washing mixture, highlighting the necessity of knowing the pH of alkaline solutions used for cleaning of textiles with natural dyes.

The poor wash fastness of many natural dyes is mainly attributed to following factors:

(a) Weak dye-fibre bonds between the natural dye and the fibre.
(b) Change in hue due to breaking of the natural dye-metal complex during washing.
(c) Ionisation of the natural dyes during alkaline washing. Since most of the natural dyes have hydroxyl groups which get ionized under alkaline conditions, many fabrics dyed with natural dyes under acidic conditions change the colour on washing with alkaline detergents or soap.

Wash fastness of some of the natural dyes can be improved with the post treatment with alum or dye fixing agent resulting in the formation of a dye-fibre complex or a cross link between dye and fibre, respectively.

The factors affecting light fastness of dyed materials such as the nature of dye, fibre and state of dye inside the fibre, also, in general, have a similar effect on the wash fastness properties but the physically determined wash fastness properties are relatively simpler and less challenging than that of light fading.

The affinity of dye for fibre molecules reduces the rate of absorption and desorption from the fibre. The dye-fibre attractive forces tend to keep the dye molecules and retard their diffusion along the pores of the fibre. The superior wash fastness of metal-complex dyes is due to the ability of dye molecules to associate into large aggregates in the fibre, which have low absorption rates and not because of the additional forces of attraction between wool and metal ions (Vandana, 2002).

2.13 Environmental aspects of natural dyeing

Although dyes are derived from nature, the metallic salts used as mordants for better dye fixation on textile and to improve fastness, are not always eco-friendly (Gill, 1991). Health hazards as well as the environment friendly behavior of natural dyes has been investigated (Ali, 1993). Very little work has been carried to assess the toxicity of natural dyes. Only one or two have been identified as posing potential problems (Chavan, 1995).

Eco-friendliness of natural dyes is done by assessing the eco-parameters viz. toxic heavy metals, pesticides, formaldehyde, pentachlorophenol, azo-dyes based on carcinogenic amines or banned amines etc by analyzing the dye extract.

The mordant that are used for fixation and development of color on textiles are mainly Alum (Potassium aluminium sulphate), Tin (Stannous chloride and Stannic chloride), Iron (Ferrous sulphate), Chromium (Potassium dichromate) and Copper (Copper sulphate). Out of these, copper and chrome are red listed and have been restricted to some stipulated limits by various eco-labels. On the basis of analysis of some natural dyes like Katha, Jakwood, turmeric and indigo show the presence of arsenic, lead, mercury, copper and chromium less than 0.2 ppm which is much below the stipulated limit except for chromium. This shows that the natural metal contaminants in the dyes are very low and so can be used safely. But the concentrations of mordants used in dyeing are sometimes very high. Therefore optimization of mordants is necessary.

Contamination of natural dyes and fibers by chorine-based pesticides may occur during the growing of plant from soil or during storage. The presence of any of the banned amines in natural dyes is ruled out because most of the natural dyes, whose structures are known as, based on quinines, flavonoids anthraquinonies, alkaloids, napthaquinone etc. and not based on azo-linkages (Bhattacharya , Doshi and Sahasrabudhe , 1998).

Moreover, while working with synthetic dyes the other perspective of this problem should also be taken into consideration, and that is the additional expenses for buying the cleaning equipment and making it work constantly in order to provide the safety and protection measures. Building the cleaning stuff and making it constantly working require a lot of big investments. This increases the cost of the fiber dyed with the artificial and chemical dyes. The third aspect of the issue is that the frequently used artificial and chemical dyes from the ecological point of view are harmful for people and the environment. This is because within the chemical dyes the elements like "ant" acid, sulphuric acid and the various combinations of manganese with the iron, aluminium, copper, chromium and of others are used (Internet, 13).

Tyrian Purple is obtained from a family of carnivorous shellfish, the most common of which is found in the Mediterranean, that is *Murex brandaris*. Enormous numbers of shellfish were necessary to yield a very small quantity of dye. If the dye were to be used for all present blue dyeings, let alone in mixtures with other natural dyes, it would result in some 200 square miles of land being deep in shells (Horrocks, 1996). Therefore dyes from vegetable sources, including roots, as well as dyes of animal origin do not offer any easier environmental alternatives. Glover and Pierce have stated that in dyeing of wool, assuming an average depth of 1.7 %, some 43000 tones of synthetic dyes are used. To replace the synthetic dyes with natural dyes, 15 million tonnes of fresh plant would be required. In the cotton sector, the analogous figures are even more dramatic. Taking the average depth of colour as 2 %, about 400 000 tones of synthetic dyes are used. To replace the synthetic dyes with natural dyes the weight of fresh plants needed to extract the natural dyes would be 176 million tones and to grow that amount of vegetable matter for subsequent dye extraction, at least 30% of the world's agricultural land would be required (Horrocks, 1996).

2.14 Comparison of environmental and safety aspects of natural and synthetic dyes

The following can be presented as a summary from reviewing the environmental aspects of synthetic vs. natural dyes

Natural:

- use of renewable resources
- lack of toxicity during production and reduction of work hazard
- full biodegradation and reduction of the environmental impact

- lack of toxicity of the end products

Synthetic:

- consumption of non-renewable resources such as oil and by-products
- work hazard during production
- high environmental impact during production and waste disposal
- danger of allergies (dermatitis by contact) for consumers

2.15 Natural dyes and dyeing practices in Sri Lanka

In Sri Lanka the practice of natural dyes goes back in history and it was practiced locally as a heritage of certain families or casts. The distribution of these families and the traditional dyeing practices carried out were based on the natural distribution and availability of dye producing plants through out the island. The dyeing was not restricted to dyeing textile materials but it was also used for religious wall paintings, handicrafts such as masks, mats and other such products, drums and coir based goods. Certain villages were and still are world famous for their natural dyed products such as handlooms, mats and masks, for example Dumbara mats and Ambalangoda masks, are still considered world class products (Internet, 14).

The basic dyes were always obtainable from certain specific plants naturally available in different regions of the country and the dyeing recipes and technology used depend on the methodology inherited by a particular community. As a result, the same natural dye based on the same plant material will be extracted and dyed using different methodologies in different local communities (Coomaraswamy, 1908). Some of the famous dyes, their extraction and dyeing techniques are explained below:

(a) Blue dyes

The world popular blue dye, indigo, which was obtained from indigo plant (Nil awariya / *Indigofera tinctoria*) and marketed in large scale by Java and India a century ago is also known and used in Sri Lanka (Tilakasiri, 1994).

(b) Red dyes

Patangi (*Caesalpinia sappan*) whose wood, boiled in pieces, yielded red dye valued for dyeing wood, rush and calico, was profusely used and quantities exported in the 1920s (Tilakasiri, 1994). Annatto (*Bixa orellana*) shrub naturalised in Sri Lanka, bearing large clusters of crimson capule like fruits containing seeds with crimson covering yielding a crimson dye of commercial value (Macmillan, 1943; Thilakasiri, 1994). Nearly hundred years ago, the plant was cultivated in Matale. The common shoe flower (*Hibiscus rosasinensis*) offered a red dye for use as a local colouring in cookery (Munidasa , 1988, Tilakasiri, 1994). Mannar also yields in abundance the Chaya (*Oldenlandia umbellata / Hedyotis umbellata*) root, which was once exported to Europe for the sake of its brilliant red dye (Tennent, 1860).

(c) Yellow dyes

Flowers of Gaskela (Flame of the Forest / *Butea monosperma*) are used to produce a yellow or orange red dye (Munidasa, 1988 ; Tilakasiri, 1994). Jak (Kos / *Artocarpus heterophyllus)* heartwood yields a yellow dye used for dyeing mats and especially robes of Buddhist monks, etc. (Macmillan, 1943; Munidasa, 1988, Tilakasiri, 1994). Rata Goraka (*Garcinia xanthochymus*) used for cloth dyeing, Dodan Kaha or Weli Kaha *(Memecylon capitellatum*) used for colouring mats and Sepalika (*Nyctanthes Arbor tristis*) used for dyeing cotton are also some of the plants used locally to produce yellow dyes (Macmillan *et a.l*, 1943 ; Tilakasiri, 1994).

(d) Brown dyes

Divi Divi (*Caesalpinia coriaria*), which is a native plant of Central America and introduced into Sri Lanka in about 1834, is used for tanning using the extracts from its brown colour pods (Tilakasiri, 1994). Ranawara (*Tanner's cassia / Cassia auriculata*) a quick growing large shrub native to Sri Lanka is well known for its tanning ability of the bark (Munidasa, 1988 ; Tilakasiri, 1994).

Some of the plants used in Ayurvedic prescriptions such as fruit juice of Sen Kottan, (*Terminalia cattappa*) Medicinal nelli (*Phyllanthus emblica*), aralu (*Teminalia chebula*) and bulu (*Terminalia belerica*) nuts also possess strong dyeing and tanning qualities, which have been known to the ancients (Tilakasiri, 1994). Apart from plants, lichens, which grow on them, are other sources of natural dyes. For example Orchella (*Rocella montaguei*), a pale, greenish gray lichen was formerly exported from Sri Lanka for the extraction of the dyes such as litmus, orchil, etc. (Macmillan *et al*, 1943). In Kandyan paintings yellow was prepared from gamboges from the Gokatu (*Garcinia morella*) tree, blue from Indigo leaves and black from lamp black prepared by using Jak milk (*Artocarpus integrifolius*), Kekuna (*Canarium zeylanicum*) oil and rosin (Hal Dummala) from the Hal (*Vateria acuminata*). Grass used for weaving mats is dyed red obtained from Patangi (*Caesalpinia sappan*) and yellow from Kaha (*Bixa orellana*) or saffron. Niyanda (*Sanseviemrnia Zelanica*) fibre is dyed red with Patangi

(*Caesalpinia sappan*), yellow with a decoction of Weniwal (*Coscinium fenestratum*)and black with gallnuts Aralu (*Teminalia chebula*) and Bulu (*Terminalia belerica*) (Coomaraswamy, 1908).

Two different extraction methods of Patangi (*Caesalpinia Sappan*) were reported from Niriella and Welimada (Coomaraswamy, 1908). At Niriella (Sabaragamuwa area) the method takes four days to complete the extraction and dyeing of grass. On the first day two handfuls of Korakaha (*Memecylon umbellatum*) leaves are pounded in a mortar, squeezed out in water by hand, and the resulting liquid resembling Pea soup is strained; two handfuls of Patangi (*Caesalpinia Sappan*) chips are added and the whole left to stand. On the second day the solution has become red. The Patangi (*Caesalpinia Sappan*) chips are removed, pounded, and replaced, and the whole boiled with the Indi kola which is to be dyed, tied up in little sheaves. The pot is allowed to cool and left till next day. On the third day leaves of Bombu (*Symplocos spicata*) leaves, Hin Bovitiya (*Osbeckia octandra*) and Korakaha (*Memecylon umbellatum*), pieces of Kebella (*Aporosa lindleyana*), and a handful of yellow wood chips called Ahu (*Morinda tinctoria*), together with a small bundle of roots of Ratmul (*Knoxia platycarpa*) are pounded and added to the solution, in which the Indi leaves remain. The whole is boiled and allowed to cool and stand till next day when the leaves are removed and dried after which they are ready for use (Coomaraswamy, 1908). At Welimada, for a pound of Galehe (another grass used for mats, *C.corymbosus)* dyeing, two pounds of Korakaha or Velikaha (*Memecylon umbellatum*), half a pound of Kiribatmul (root of *Knoxia platycarpa*), two pounds Patangi (*Caesalpinia Sappan*) chips, green saffron and lime are added to four pots of pure water. They are boiled for three days with a strong fire in the morning, slow fire during the day, and again strong fire in the evening and slow fire during the night. The pot is not removed from the hearth during the three days, after which the dyed grass can be removed and dried in the shade (Coomaraswamy, 1908). The colour thus produced by Patangi (*Caesalpinia Sappan*) is a fine fast red, which does indeed fade slowly, but lasts as long as the mat is likely to, which is more than said of the aniline dyes, which have entirely replaced Patangi (*Caesalpinia Sappan*) in many districts (Internet, 15).

It may be mentioned that apart from the very few traditional dyeing applications, which function to date such as mat dyeing in Dumbara, the only natural dyeing technique practiced in the island is the robe dyeing of Buddhist monks. The yellow dye of priests robes is obtained from the wood of Jak (*Artocarpus heteropyllus*) (Munidasa, 1988 ; Coomaraswamy, 1908 ; Tilakasiri, 1994). Small chips of Jak wood are boiled for a day in pure water and the cloth is soaked in this extraction. The extraction is sometimes boiled with Bomboo (*Symplocos spicata*) leaves or the cloth previously soaked in a boiled extraction of Bomboo (*Symplocos spicata*) leaves (Internet, 16). In any case the jak (*Artocarpus heteropyllus*) wood extraction is strained and left to cool before soaking the cloth (Internet, 17). The dyeing is repeated at least four times to get a better colour yield. The yellow dye is not permanent but can be easily renewed. The use of Sepalika (*Nyctanthes arbortristis*) flower extract followed by this first dyeing gives a brighter colour (Coomaraswamy, 1908).

There are also many other plants and shrubs used for the purpose of dyeing and tanning on a small scale, but their specific properties and characteristics have never been studied scientifically. They were also not commercially exploited since the production was meant for a small populace, but still show the effectiveness of their usage for the specific design and form of the article (Tilakasiri, 1994). In the literature survey of natural dye practices in Sri Lanka details of natural dye were found under several areas. Pigments and dyes were used in dyeing cotton yarn (Robe dyeing), painting murals, mat weaving, masks, Batik, body painting and in lacquer work etc. All these were made using various resins extracted from plant matter, insect's secretions, and from clays. Although these arts are not commonly practiced today according to their early manner with the use of natural dyes, people in some parts of Sri Lanka still use the early procedures and materials. The best examples are weavers of "Talagune" and "Maduramunai" (Internet, 17-19).

In painting and dyeing, predominant colours used were yellow, red, white, black, blue and green and they were made as follows (Horrocks, 1996):

Yellow: Made from the secretions made by cutting the bark of Gokatu (*Garcinia merella*) tree.

White: Made using the type of butter clay (Kirimati) known as makul clay. This clay mainly came from makul caves of Maturata and hence also known also as Maturata makul.

Black: It was made first by grinding koholle *(Artocarpus integrifolius)* (dried milk extracted from Jak fruit), resin of Hal (*Oryza sativa*) tree in Kekuna *(Canarium* *zeylancium)* oil and mixing it with old rags. The mixture is then burned in a closed vessel. The deposits on the lid were used as the pigment.

Red: Made by grinding the sadilingam *(Cinna bar)* found in caves in Meemure area and dissolving it in water and warm with juice of Ratmal (*Ixora coccinea. L*) bark.

Blue: Blue was rarely used in Anuradhapura and Polonnaruwa , even in Kandyan period murals. It was made extracting the juice of Nilavariya (*Indigofera tinctorea*) leaves.

Green: It was made by mixing yellow and blue. The lighter shades were made by adding white to dark colours.

Gold: The gold colour also used in some paintings and was made by grinding the mineral hiriyal (Opriment in lump) or ran hiriyal (scaly Opriment) and baking. Another recipe gives the following description for making colours for wider application.

Equal parts of hiriyal and ran hiriyal was taken and ground with juice of Milla (*Vitex altissima. L.*) bark, Gammalu (*Pterocarpus Marsupium, Rox. b.)* bark and Gokatu (*Garcinia morella, Desr.)* bark and it is roasted with black badama (varnish composition). Badulla (*Semecarpus gardneri, Thw.*) milk, Keppetiya lakada *(Gardinia latifolia)* and Hal-dummala (resin from tree *Vateria acuminate, Hayne*) and old jak milk *(Artocarpus integrifolius)* in equal parts are mixed and ground these together and warm.

Yellow (badama): Gokatu *(Garceinia Morella*) juice hiriyal (Opiment in lump) and ratukaha (*Bixa orellana. L*) is taken in equal parts collected old jak milk (*Artocarpus integrifolius*) and ground with dorana (*Dipterocarpus glandulosus*) oil and applied it and polished with Cheeta's tooth gold coloured badama (Varnish). Equal parts of hiriyal (Opiment in lump), gokatu *(Garceinia morella*) milk, quick silver, alum, sal-ammoniac borax and white lead (sudu iyan) are taken and ground in dorana (*Dipterocarpus grandulosus*) oil apply to any surface and polished it with Cheeta's tooth.

To a limited degree, Sri Lanka has used extracts from mangrove plants as valuable sources of dyes (Horrocks, 1996).

2.16 History of dyeing practices

Records have been found for paintings in wooden surfaces and textiles in the Sri Lankan history. Paintings on wooden surfaces are found on the ceilings of religious buildings, manuscript covers, screens, boxes used to store sacred objects and votive tablets. Godapitiya Rajamaha Vihara and Kadurugahamaditta Gangarama Vihara posses two large wooden boxes with the Dhahamsonda Jataka painted on both.

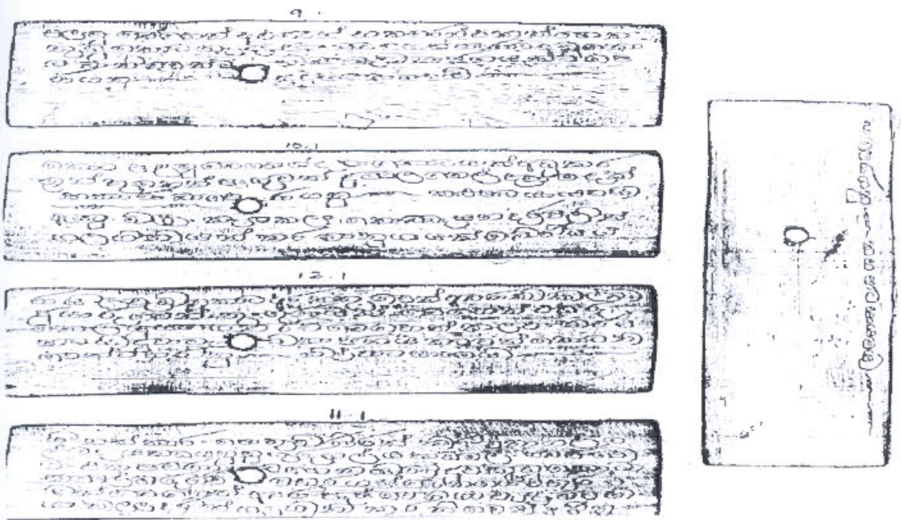

Figure 2.4 Puskola or Ola book (Sinhalese and Sanskrit palm leaf manuscripts which have inestimable archeological, cultural and scientific values) from Ambakke Muhandiram (Madya kaleena Sinhala Kala, pp 179)

There are numerous literary references to the practice of painting on cloth in Sri Lanka from as early as the second century BC to the nineteenth century. The great chronicle Mahavamsa has it that as King Dutthagamani lay dying, Saddhatissa, having covered the half finished Maha Thupa with white cloth over a wooden frame, commanded painters to make on it a vedika duly and rows of filled vases likewise and the row with the five finger ornament'. Due to the non-durability of the material, only the eighteenth century examples are extent today. In terms of iconographic tradition, compositional arrangements, figurative forms, treatment of design and colour schemes, the surviving examples of petikada bear a relationship to the temple murals of the Kandyan style.

Petikada may provide the clue to a missing link in the records of the Buddhist paintings of Sri Lanka between the thirteenth and the eighteenth century. The stylistic origins of Kandyan painting which proliferated in the eighteenth century are shrouded in mystery, as it is diametrically opposed in style to the pre-thirteenth century classical paints available. In the absence of any immediate temple painting tradition to follow, the Kandyan painters may have been inspired by the paintings on cloth (Internet, 20).

2.16.1 Mural painting

Most of our colour paintings can be seen at Buddhist temples. There are certain other traditional colour paintings, but the overall amount of those paintings are negligible when comparing with temple paintings. However the majority of painting designs can be seen in traditional paintings rather than temple paintings.

The paintings of Sri Lanka can be sub-categorized as follows:

Paintings on the walls of temples, Dewala and on the roofs of cave temples such as Dambulla and Degaldoruwa.

(a) Painting on ceilings, wooden doors and wooden towers.

(b) Paintings on javelins, walking sticks and sticks for flags.

(c) Paintings of pottery industry.

(d) Paintings on upholstery and certain flags.

(e) Paintings on "Bali" (Falk dance) & Thoran (decorated with lanterns, lights and special displays of lights).

It was an essential task of an artist at that time to know about the preparation and usage of different colours in ancient paint industry.

Some of the colors were prepared as follows.

Yellow

(a) from "*Gokatu* tree" *(Garceinia morella)*

(b) from "*Hiriyal*" (Opiment in lump)

Hiriyal was used for pottery and furniture paintings. As it was difficult to find the "*Gokatu trees*" *(Garceinia morella)*, *Hiriyal* (Opiment in lump)was widely used for paintings at that time.

Red

(a) from "*Sadilingam*" *(Cinna bar)*

(b) from "*Gurugal*" (Kapok found in river beds)

(c) A dark red colour was obtained using Sadilingam *(Cinna bar)*. The special feature of this colour was its durability. The red color prepared from Gurugal (Kapok found in river beds) was fairly brownish red and it was so called as "Guru". This colour was widely used to show the sharp points of the drawings. It was also used for pottery painting and Bali (Falk dance) & Thoran (decorated with lanterns, lights and special displays of lights) which did not last for a long time.

Blue

This colour was prepared using the juice of "*Awariya* tree" (*Indigofera tinctorea*). The colour was not the basic blue color and it was rather a greenish blue. The colour was very rarely used in the ancient paintings. Generally the ancient people did not recognize any difference between blue and green. In present time also people are calling the both colours as blue. For some paintings of "Vishnu", (God of Sinhala Buddhists) instead of blue it was used a dark ashy colour prepared by mixing black and white.

Green (Pachcha) -Tattos

This colour was prepared by mixing blue and yellow colours. The term "Pachcha" (tattos) is locally used for this green colour and it was related to the Gem industry, where there is a particular type of gem with this colour and so calling "Pachcha" (tattos). For Bali & Thoran, this colour was prepared using "*Ranawara" (Gassia auriculata)* and "*Kehipittan*" *(Cyclea barmanni)* juice. The amount used in the ancient paintings is fairly low.

Black

This was a stable colour. The colour was prepared by burning a mix of "*Haldummala*" (*Vateria accuminata hyne*), "*Kekuna oil*" *(Canarium zeylancium)*, jak tree milk and wear off cloths. The prepared colour was locally called "Kaluanduna" (A relatively unstable black colour was obtained by mixing the oil with burned coconut shell).

White

This was prepared using clay called "*Makulu*" (type of butter clay) which is being used for ceiling painting in present days.

White or Black colour was added accordingly, whenever it was needed to lighten or darken a particular colour. Ramha (orange) colour was prepared by mixing yellow and red. The Ramba (orange) colour are seeing in the ancient paintings is not the actual Ramba colour. It was so seen, because the pure yellow color in the paintings had darkened a bit to Ramba (orange) colour, after applying some varnish on those original paintings. Even when mixing yellow colour with "*Dorana oil*" (*Dipterocarpus glandulosus*) the Ramba colour (Orange colour) can be obtained.

Grinding, mixing and preparation of paints were basically done by the students who were learning about the paintings at that time. The raw materials were well ground and then mixed with the sticky solvents. Sticky juice from cashew (*Anacardium occidentale)* and Wood apple (*Feronia limonia*) trees were used as the solvent.

There is not much evidence of ancient paintings on clothes. But it is mentioned in "*Mahawamsa*" (A book consisting of historical background of the Sri Lankan nation written in sanskrit) that during the second and thirteenth centuries there were certain types of paintings in Sri Lanka. There were two types of cloth paintings at that time. Those are dyeing and water colour painting. In the present time these dyeing techniques are extensively used in the textile industry. Those ancient cloth paintings were used to depict the life story of Lord Buddha and famous "*550 Jathaka katha*" (Ancient stories consisting of various incidences of Budha's life). The painted clothes were used to decorate the "*Pirith Mandapa*" (A special place decorated to chant Buddhist religious verses) and "*Darma Shala*" (A place where Buddhist gather to hear Buddha's teachings by a priest). Some of these old cloth paintings can be seen at National Museum, Colombo (Madya kaleena Chithra Kalawa, paintings of Medieval stage).

2.16.2 Apsara paintings

The most famous features of the Sigiriya complex are the fifth-century paintings found in a depression on the rock face more than 100 metres above ground level. The Sigiriya paintings have heen the focus of considerable interest and attention in both ancient and modern times.

Most of the ancient graffiti are notes or poems referring to the Sigiriya Maidens. The long history of Buddhist painting in Sri Lanka falls into two clearly identifiable periods: the Classical and the Kandyan. The Classical period can be dated from the existing records to a period from the fifth to the twelfth or the thirteenth century; and the Kandyan period from the eighteenth to the nineteenth century. Fresco paintings of the beautiful damsels interpreted by historians as apsaras (celestial nymphs) executed on lime plaster on a pocket of western face of the fifth century rock fortress of King Kassapa (478-496) at Sigiriya represent the earliest datable, the best preserved and the most outstanding examples of the classical style (Internet, 20). The colours used for these paintings were obtained from natural sources like Sadalingam *(Cinna bar)* from

(Green colour), Hiriyal (Orpiment in lump) from soil (yellow colour), Nil aweriya (*Indigofera tinctoria*) (blue colour) etc.,

Figure 2.5 Apsara paintings at Sigiriya

2.16.3 The caves and paintings

The Dambulla temple is composed of five caves which have heen converted into shrine rooms. Within these rooms is housed a collection of one hundred and fifty statues of the Buddha and several more of devotees.

In its conventions particularly in the decorative designs and representation of trees and creepers. The paintings are executed in brilliant colour schemes, where yellow and red are predominant (Internet, 20). The Dambulla dapata reads that some caves of Dambulla were painted on the orders of King Senarath (1604-1635 AD). This would mean that they were executed by the Kandyan artists of the 17^{th} century. During the period of Kirthi Sri Rajasinghe the paintings were renovated and over-painted again. Paintings in cave no. 4 belong wholly to the new Sinhala School of painting which flourished in the Kandyan provinces after the 17^{th} century (Internet, 21).

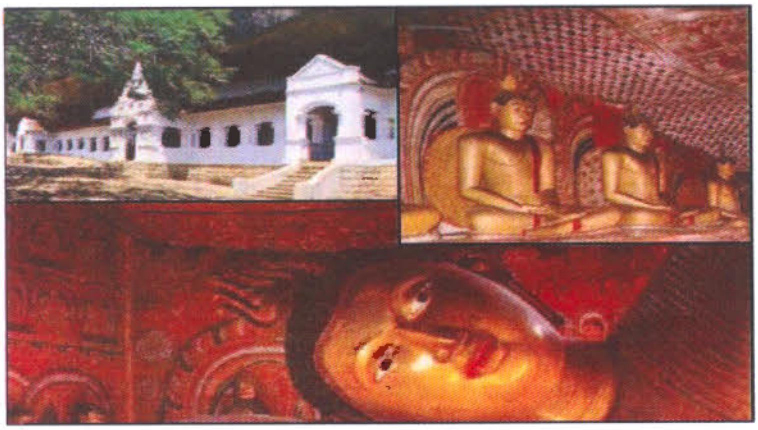

Figure 2.6 Cave paintings at Dambulla temple

2.16.4 Robe dyeing

Formerly monks had to make and dye their own robes. Modern monks are so well supported by lay devotees that they have forgotten their humble roots. To remind them of their gratitude to their benefactors, monks are required to go through the tedious process of making and dyeing their own robes at least once.

Natural dyes were prescribed by the Buddha to match the allowable shades of robe colour. The production of such dyes is time-consuming and many of them fade or run after a few washings. It is educational for a monk to learn how to make such natural dyes and use them to dye his robes. However, for practical reasons we can use chemically dyed cloth to make robes and re-dye them with self-made dye. Thai forest monks use jak fruit pith while some Burmese monks use ficus bark or shells of ironwood fruits to make robe-dye. Mahogany barks had been used by some monks in Sri Lanka to make their dye for robes (Internet, 22).

The Katina robe was at first sewn by the monks themselves with the cloth offered by buddhist people. The cloth was cut up to specifications and sewn according to a set pattern and dyed in water with some pieces of jak wood, giving the yellow dye. Dyeing a robe was extremely difficult because they had to boil the bark of the tree to get the colour wanted (Internet, 23).

2.16.5 Mat weaving

This is done as a handicraft and mats come with beautiful inter woven patterns, ratas (motifs), floral designs and with figures of trees, birds etc. The reed Hawen Pan *(Cyperes dehiscens)*, Galehe Pan *(C.corymbosus)*, Tunhiriya *(Colubrina asiatica)*, Dunukaiya *(Pandanus foetidus)*, Indi *(Phoenix zeylanica)* are used for mats. The mats made with galehe and wetakeyia are more robust for wear and tear. These reeds are cut and dried on ground in shade and in sun. The Palmirah (*Borassus flabelliformis)* and Talpat *(Corypha umbraculifera)* leaves are used for large mats (magal), which are mainly used for drying paddy, cover the floors and walls. To have motifs the reed (pan) is dyed with traditional pigments made by coloured clays and vegetable extracts. The red colour for mat weaving is obtained by boiling chopped patengi (*Caesalpinia Sappan*) stems with korakaha *(Memecylon umbellatum)* leaves (Internet, 23).

The famous Dumbara mats as name suggests were woven in Dumbara valley in the suburbs of Kandy. It is made not by hand but with a very primitive type of a loom.

This setup, similar to the loom used to weave clothes was normally installed in the courtyard of the house, produced a (special sized) mat with traditional motifs. The bird, elephant, tree, lion are the common patterns used in the Dumbara mats, and they are used as wall hangings, seat covers etc. These mats were sought by high Sri Lankan society few years back. With the increasing trend for machine made goods the demand for arts and crafts have tended to decline.

Today, there is an increasing worldwide trend for eco friendly goods as people gradually tend to go back to nature. Such a development will have a future for indigenous crafts such as the weaving of Dumbara mats which is unique to Sri Lanka. A unique Sri Lankan handicraft in the form of a wall hanging mainly produced in a village call Henawala in the Dumbara valley of Kandy (Internet, 23).

The Dumbara mats were made also from the Niyanda (*Sansevieria zeylanica*). This is no longer prevalent due to the scarcity of the plant and insufficient fiber for large-scale manufacture (Coomaraswamy, 1908). It appears that the shift from Niyanda (*Sansevieria zeylanica*) to hana *(Furcraea gigantean)* was beginning to take place. At present weavers purchase leaves from growers of some 30-40 miles away. The leaves are scraped against along with a sharp implement and this removes the fleshy part of the leaf leaving behind the fiber. Then the fibres are dried in the sun before they are dyed. The fiber is then dyed with natural dyes obtained from plants such as patangi (*Caesalpinia sappan*) which yields a red dye, weniwal (*Coscinium fenestratum*) which gives a yellow dye, katarolu (*Clitorea terneata*) which gives a purplish dye and bulu (*Terminalia belerica*) which yields a black dye when combined with mordarnts.

The Dumbara mats produced here are however strictly speaking not mats in the conventional sense, as they are not meant to sit, but rather in the form of wall hangings. These wall hangings come in a variety of colours and designs with motifs of flowers and various fauna and flora. (Internet, 24).



Figure 2.7 Dumbara mats

2.16.6 Masks

The legacy of centuries of master craftsmanship, ingenuity and inbred skill, these goods are turned out using age-old techniques, tools and natural indigenous material, mainly in the homes of craftsmen or occasionally at rural craft centers. A marked degree of regional specializations exists, based on the availability of raw materials as well as other factors such as royal patronage in the past and the demand for products.

Figure 2.8 Masks painting in Sri Lanka

To make brushes for painting, the fiber of the roots of the Vetake tree (*Pandanus tectorious*), hairs of animals, Alliadu (a mixture of resin powder and oil) stones or fossilized matters, skins, flowers and leaves of trees were used. To mix colours, tree oil called Dorana *(Dipterocarpus grandulosus)* oil and resin were used. To provide a durable coat, a mixture of resin powder of dummala (resin), tree oil, dorana oil *(Dipterocarpus glandulosus)* and clay (Alliyadu) was applied. This surface was smootherized with the spathe of the bread-fruit flower (*Del saveran)* Then painting started. Usually yellow was applied as a basic colour, and then the detailed painting was done. To protect the colours, a mixture of resin powder and tree oil (Valichchia) was applied on the mask.

2.16.7 Batiks

There are several countries known for their batik creations, starting with India where it originated. After that it moved to Indonesia, Malaysia, Sri Lanka, Thailand and the West. The history of Indian batik can be traced as far back as 2000 years. Indians were conversant with the resist method of printing designs on cotton fabrics long before any other nation had even tried it. Rice starch and wax were initially used for printing on fabrics.

It consists of applying a design to the surface of the cloth by using melted wax. The material is then dipped in cool vegetable dye; the portions protected by the wax do not receive the dye, and when the wax is removed in hot water the previously covered areas display a light pattern on the colored ground.

India has always been noted for its cotton and dyes. The indigo blue, which is the basic color for batik, is one of the earliest dyes. In the past, batik was considered as a fitting occupation for aristocratic ladies whose delicately painted designs based on bird and flower motifs were a sign of cultivation and refinement just as fine needlework was for European ladies of similar position. The beauty of batik lies in its simplicity and the fact that one need not he an artist to achieve results. Some of the best effects in batik are often achieved by chance.

2.16.8 Lacquer work

The lacquer work is a traditional craft especially in Kandyan areas done by carpenters, or the arrow makers. The products ranges from sesath handles, beeralu fanlight and window uprights, heppu (betel receptors), boxes, Puskola book (Sinhalese and Sanskrit palm leaf manuscripts which have inestimable archeological, cultural and scientific values) covers, incense containers, dragger handles and even axe and spear handles etc. There are two types of items the turned ones and the hand applied ones.

The raw material used are coloured lacquer, which comes from the resin of two types obtained from insects in Keppitiya (*Croton aromaticus*) or kon (*Schleicheria trijuga*) lakada plants. The other variety green comes from telakiriya (*Tachardia conchiferata*). The craft is carried out in Hapuvida Matale, Paleekanda in Balangoda and in Patha Dumbara and in Angalmaduwa near Tangalle. The twigs having the lac insects are sun dried and the resin is removed and after winnowing put inside a clean cloth and heated over fire. The melted resin that oozes out are collected by wrapping on two sticks and drawn to fibre. The heating and drawing is repeated until the resin clear brown resin is obtained then this is mixed with powdered for basic colours red by adding (vermilion - sadilingam), yellow (orpiment-hiriyal), black (as in painting) and green (to yellow).

The "*niyapotu work*" (Nail painting) is done by finger nail by wrapping the finely drawn fiber/thread over the article held over the cinders. A heated Tal leaf is rubbed over to flatten and to polish the design. In lathe work similar to the ivory bench but the article is turned by a bow string. The coloured lacquer is applied one by one by holding it to the turning artifact and followed by holding a grove shaped split over the lacquer. The heat generated by friction melts the lacquer and spread it evenly over the desired area to be coloured. The other colours too are applied in the same manner. Once the application of colour is complete the Tal leaf is applied lightly to the turning object to smoothen the lacquered surface and to polish it.

The village call Pallehapuwita of Matale district is famous for lac work. Lac is a form of wax obtained from a species of insect in the past. This wax mixed with colours is applied on wooden objects with the help of a hand driven lathe.

Nowadays imported wax called shellac is used instead of Lac obtained from insects and produce Powder bowls, vases, walking sticks, jewelry boxes etc.,

Figure 2.9 Laquor painted items

2.16.9 Body painting

Natural clays and pigments made from a great variety of plants and minerals are often mixed with vegetable oils and animal fat to make body paint. These include red and yellow ochre (iron rich clay), red cam wood, cinnabar, gold dust, many roots, fruits and flowers, cedar bark, white kaolin, chalk, and temporary skin dyes made from indigo and henna leaves. People all over the world adorn the living and also treat the dead with body paint.

The veddha people in Sri Lanka apply ground plant leaves on their bodies for their protection from sunlight as well as from poisonous plant types in the forest, when they are going for hunting (Internet, 24).

2.16.10 Hair dyeing

Hair is one of the easiest and most obvious parts of the body subject to change, and combing and washing hair is part of everyday grooming in most cultures. Styles of combing, braiding, parting, and wrapping hair can signify status and gender, age and ritual status, or membership in a certain group. Henna and shoe flowers are used to dye hair.

There are two common sources of brown, henna from a plant, and a dye leached from certain tree wood. Henna (*Hawsonia interims*) comes from a small shrub which grows in warm climates, especially in India and in North Africa. Almost all of the plant above ground can be used; the stem leaves and flowers. As the moisture varies, so does the deepness of the color. After grinding to powder, it is mixed with hot water to form a paste. This is then painted on the skin as a design, or added to the dye pot as a dye. It can be allowed to dry and stored as cakes of dye, and is suitable as trade commodity. Many variations can be obtained by mixing with other plant leaves, as from coffee shrub, lemon tree, etc. However, the shade tends to become lighter with the mixing, which is why the darker the shade the more valuable. The henna found in Africa tends to black and is called black henna.

The crushed leaves of the henna plant, when mixed with other natural ingredients, yields a thick, fragrant paste used for painting hands and feet. The olive green, dried henna powder, once mixed with such ingredients as black tea and coffee, cloves and tamarind, turns dark. Once the paste is applied on the skin, it is allowed to dry, sometimes overnight. The dried henna is scrapped off the skin resulting in a maroon-red stain. Henna has traditionally also been used for hair conditioning and dyeing, skin antiseptic and tonic, and as cloth and leather dye. The use of henna goes far beyond use as a dye. It has many superstitious attachments, and many medicinal uses, real or imaginary. The use of henna can be traced back 5000 years, and its origin is lost to antiquity. When used as an art form to adorn the body, the art is called mehandi; this is becoming popular in the United States recent years (Internet, 24).

Figure 2.10 Body paintings by Henna

In Sri Lanka henna is called namely by maruthani. The art form of henna (maruthani) varies from region to region. Varying designs have a different meaning for members of each culture, such as good health, fertility, wisdom, protection and spiritual enlightenment. While Arabic henna designs are usually large, floral patterns on the hands and feet, maruthani involves fine, thin lines for lacy, floral and paisley patterns covering entire hands, forearms, feet and shins. However in Sri Lanka the use of henna is limited. Availability of maruthani in our country is less and can be obtained in some areas such as North and Western provinces.

2.17 Hand Loom weaving

2.17.1 The Moor weavers of Marudamunai

The Moorish town of Marudamunai in the Ampara district has a tradition of weaving said to go back to Kandyan times. The Moors here, it is believed, were settled in the days of King Senarath for purposes of weaving and dyeing cloth, a tradition that continues to this day. Although these expert Nesavalar or weavers formerly manufactured garments such as somans and sirwals which are no longer used, they now largely produce sarees and sarongs to meet the demands of today's market.

The garments produced then were of cotton and included sarongs for men and camboys and somans for the womenfolk, besides other sundry items such as shawls and towels. They used natural dyes such as turmeric and kadakai for their products, a practice which however died out sometime ago with the introduction of synthetic dyes. It was only recently that natural dyes were reintroduced into the area and these are now largely used for dyeing fabric which has much demand among foreigners. The weavers here made camboys which were widely worn by Sinhalese women and known as kambaya.

Figure 2.11 A woman at the loom

Marudamunai also manufactured a garment known as soman which was similarly worn by the Moor women of the east, though they were far more expensive than the kambayas. These somans too were made of cotton, though sometimes silk was used for the border.

The natural dyes used to dye fabric in Marudamunai are made by combining plant matter with mordants. For instance, Walmadata and soda ash gives a pink colour while tea and potassium permanganate yields a brown colour. Aralu, alum and alzarin gives an orange brown colour while tea and ferrous sulphate yields a silvery black colour. A combination of aralu and potassium permanganate gives a dark black colour. Marathondi which is widely used by Muslim women to decorate their hands and feet is also used to dye fabric and gives a light red colour. Natural dyed fabric is especially in demand by foreigners and consequently fetches a high price (Internet, 24).

2.17.2 The cotton weavers of Talagune

The weaving of home made cotton items which were flourishing earlier in the Kandy District, today it is confined to only one solitary village, Talagune in the Udu Dumbara area. About 10 to 12 families' resident here for generations have produced eye-cating traditional motives. The items produced include wall hangings, cushion covers, bags and other utility items. Cotton weavers of Talagune are the sole custodians of an ancient craft. Little is known that the weaving of traditional homemade cottons once widely prevalent in the Kandyan vicinity. For generations weavers turned out beautiful cottons adorned with traditional motifs known as Dumbara rataa captivate the hearts of not only of Sri Lankans but also foreigners. Wall hangings cushion covers and bags are the items that are made according to age-old methods. Younger generations have acquired this skill and they are moving towards modern and colorful designs which are likely to have demand in the days to come.

The weavers of Talagune trace their origins to Rakshagoda, the very place where the legendary Kuveni who espoused Prince Vijaya lived. According to historical Mahavamsa Kuveni when first seen by one of Vijaya's followers was spinning like a woman hermit and it is only natural to suppose that the art of weaving should somehow be connected to her. A thriving industry is believed to have flourished here before it gradually spread to the other areas in the days of the Sinhala kings. Now the art is only surviving Talagune.

In the work of Mediaeval Sinhalese Art observed nearly a hundred years ago that the weaving of homespun cotton cloth once universal in the Kandyan provinces was only done at Talagune, and perhaps occasionally near Vellassa in Uva and not long ago in Balangoda. The industry still flourishes a little; two families have looms, and a number of the village lasses are skilled in spinning (Internet, 24).

Although the villagers today procure their cotton from outside sources, it appears that in the olden days they grew their own cotton trees at *'Kappili Tenne Hena'* the village not far from where these families reside clearly proves this.

Dyeing of the thread too seems to have been done by the villagers using natural dyes obtained from the bark and leaves of trees. The patterns include the designs from fauna & these designs have undergone some modification with time. The loom used to weave the final product somewhat resembles the old looms used in cotton weaving, though it is a much simpler device. The weaving itself appears to he a time-consuming process and needs a lot of skill and patience (Internet, 24).

The colours are strictly in keeping with tradition and are not very varied, being either red, orange or black and white.

The large scale textile sector has taken synthetic dyes as the basis for their operation. There is only a small segment of operation that relies on natural dyes for their textile colourations.

The review presented was carried out to determine the rich heritage of Sri Lanka associated with natural dyes. It appears that there had been many practices and rich outputs hased on these practices.

Currently small scale natural dyeing practices are being carried out by the Department of Textile Industries situated in Katubedda and Rajagiriya. To enhance this area of natural dyeing department is being carrying out several workshops, seminars on this area. Some products from Sri Lankan natural dye industry is demonstrated in Figures 2.12 - 2.15 (Natural dyeing plant at Rajagiriya, Colombo, Sri Lanka).

Figure 2.12 Natural dyed yarns

Figure 2.13 Natural dyed woven saree

Figure 2.14 Industrial scale natural dyeing

Figure 2.15 Natural dye kitchen

Figure 2.16 Spread of activities involving, natural dyes in Sri Lanka

2.18 Recent developments of natural dyes

The use of natural dyes has increased substantially during the last couple of years. These dyes are being mainly used by:

(a) Traditional dyers and printers

(b) Academic Institutes and Research Associations/Laboratories

(c) Industry

(d) Designers

(e) Hobby groups

(f) Non-government organizations (NGO's)

(g) Museums

The use of natural dyes, particularly as a hobby, has continued in spite of the advent of synthetic dyes. Of late, this activity has increased considerably particularly in US where many organization have been formed to discuss about these dyes and organize workshops and training programmes on regular basis. A quarterly journal 'Turkey Red Journal' covers information about the workshops and the activities of various hobby groups. The easy accessibility of internet has also given impetus to this activity, where various individuals exchange notes almost on daily basis. Many companies have come out with Hobby Kits for beginners. In India, Alps Industries have come out with a large range of do-it-yourself' kits for others. Table 2.13 summerises some companies selling natural dyes in USA.

Table 2.13 US companies selling natural dyes through internet

1.	Jane's Fiber Works http://greene.xtn.net/-fiber/dyes.html
2.	The Mannings Catalog http://the-mannings.com
3.	Carol Leigh's Hillcreek Fiber Studio http://www.hillcreek.com
4.	Mawia Handprints http://www.maiwa.com
5.	Louet Sales http://www.cybertap.com
6.	La Lana Wools http://www.taaosfiber.com

2.19 Estimates of dye requirements

The demand consumption potential for natural dyes will be small in the initial years being related to a select segment of handloom products. Thus natural dyes are unlikely to threaten the market for synthetic dyes in the foreseeable future. With novelty and variety of shades, natural dyes would indeed complement the synthetic dyes hopefully open up new potentials.

The consumption of synthetic dyes has been estimated to be close to one million tones per year. As per the report of the German ministry of food, agriculture and forestry about 90,000 tones of natural dyes can be produced every year. The production of natural dyes requires agricultural land. It is estimated that about 250-500 million acres of land will be required to produce about 100 million tones of plant material needed to produce one million tones of natural dyes. This land requirement corresponds to 10-20 % of the area cultivated for grain throughout the world.

Allegro natural dyes company has set a target to replace about 1 % of the synthetic dyes worldwide. According to their calculations, they shall require 0.02 % of the total arable crop land. USA is one of the major importers of natural dyes. The total import of these dyes, which is about 3.5 MT per year, works out to be 0.4 % of the synthetic dyes. Imports of natural dyes of EU countries were 5300 tones for the year 1995. This accounts for 0.53 % of the synthetic dyes. Besides this in many countries the dyes are produced for local consumption, which may also, be about 1 MT. From the foregoing it is apparent that the present requirements of the natural dyes are about 10 MT, which is equivalent to 1 % of the world synthetic dyes consumption (Internet. 24).

Chapter Three

STUDY METHODOLOGY

3.1 Materials and Methods

3.1.1 Literature review

Comprehensive literature review on the field of interest in this project (i.e. natural dyes) was carried out and presented in chapter Two. It included the early use of natural dyes in the world, Asia and Sri Lanka, the source of obtaining natural dyes and methods of extracting dyes. It also included the new trends in the global market towards natural dyes.

Although there are three main sources of getting natural dyes viz plants, animals and minerals.

3.1.2 Robe dyeing

After investigating all the current dyeing practices, robe dyeing was selected for the study due to its importance and its continuity across many parts of the country. The in depth study is expected to demonstrate and yield issues facing this type of practices.

After several discussions with the temple priest, the traditional robe dyeing procedure was carried out in a temple situated in Kandy (Gallangolla Temple). Several discussions with the temple priest were carried out. A white cotton robe was dyed with the assistance of the temple priest.

The steps of a traditional robe dyeing process are;

(a) The robe was first prepared by stitching previously cut standard white fabric pieces.

(b) The dye solution was prepared by boiling chopped jak *(A. heterophyllus)* bark until the required colour (dark yellow) appeared in the solution.

(c) Extracted solution was strained and used for dyeing.

(d) Then the robe was soaked in the solution of jak *(A. heterophyllus)* extract (Figure 3.1a & 3.1b). For the required time (about 2 hr) until the required shade appeared.

(e) Dyed robe was washed in clean water and dried under sunlight.

Laboratory trials were done on this process to confirm the process.

a Pandu Oruwa b

Figure 3.1 a, b Dyeing of the robe and squeezing

Laboratory scale operation of the above procedure was carried to ensure better results and to overcome drawbacks of traditional robe dyeing which could give more reproducibility.

Prior to dyeing, the robe was soaked in the Bomboo (*Symplocos spicata*) solution which was prepared by boiling 1.0 kg of Bomboo (*Symplocos spicata*) chips in 8 l of water for 2 hrs to remove impurities, then squeezed and left in the shade for few minutes to dry.

2.0 kg of matured jak bark chips were dried in the laboratory oven at 37 °C until the oven dry weight was obtained. Then these chips were ground by using industrial grinding machine and sieved to get finely ground particles (355 μm). 1.0 kg of the powder was added to 4 l of water and boiled while stirring for one hour. This was followed by an addition of another 4 l to the same container to get maximum extraction of dye particles (MLR used was 1:8). Then extracted dye solution was filtered out by using a vacuum filtration unit.

5.0 g of fabric pretreated with extract of Bomboo (*Symplocos spicata*) bark was dyed in a bath containing 30 g/l Sodium chloride salt and 250 ml of the extracted dye solution at 80 °C for 30 min.

The dye bath was prepared with jak wood bark chips. Prepared dye solution was put in to the "Pandu Oruwa" (Small pot having a shape of a boat - Figure 3.1 a,b). The folded robe was turned side by side to ensure uniform absorptivity of dye. It was kept for few days (2-3 days) until the robe gets the optimum dye absorption.The robe was then washed, rinsed and the excess water squeezed out and dried.

3.1.3 Selection of dye yielding bio-materials for natural dye extraction

Comprehensive literature survey was carried out to investigate the natural dye producing plants in the world and those which are indigenous to Sri Lanka. Initial selection of plants from the overall list of bio materials was carried out. After a comprehensive literature survey, about 90 natural dye giving plants were identified.

Studies were done to evaluate sources of natural dyes available in South Asia including Sri Lanka (Taylor, 1986; Mell, 1929; Munidasa, 1988; Tilakasiri, 1994). Starting with more than 50 different plant parts which could be used as raw materials for dyestuff extraction, a selection was performed with regard to the following requirements:

(a) Production of the plant material in sufficient amounts with modern agricultural methods including simple extraction methods to obtain the dyestuffs.

(b) Formation of suitable classes of dyes which are, in their applicability, comparable to the classes of synthetic dyes in use at the present.

Plant raw materials which go as waste but still contained dye materials in relatively large quantity were selected. Some new natural dyeing methods were selected which are ecologically friendly and developed with less health hazards for a selected plant material to investigate its suitability as a textile dye.

3.2 Dyeing tests and quality criteria

The applicability of plant dyes for industrial purposes makes high demands on the quality of the product, especially with respect to the transportability and shelf life of the dyestuff as well as to the standardisation of high quality dyestuff and the reproducibility of the dyeing results.

In order to establish a resource-efficient and economically viable product line, mainly residual material from the food and wood-working industry were used as resources. The dye stuff product was assessed according to the following criteria:

(a) Availability of raw material at the lowest costs possible,

(b) Possibility of making ready –to –dye concentrates,

(c) Transportability and shelf life, in order to guarantee supra-regional supply,

(d) Manageability at operational level,

(e) Material is ready to compost without further treatment and can be used for other purposes,

(f) Use of watery plant- extracts is possible, use of solvent and chemicals is not intended,

(g) Good fastness of the dyed product,

(h) Applicability to the dyeing of protein fibre (wool), cellulose fibre (linen, cotton) and

(i) Dyestuff which does not need mordant is preferred, if mordant is necessary, copper and aluminium mordant or bio mordant to be used.

Sri Lanka is at an advantageous position since the country holds a rich reservoir of natural raw materials. Different parts (leaves, bark, seed, flowers, roots and woods etc.,) of a considerable number of plants have been reported to yield dyes, however a large number of them are hitherto unexplored. The number of possible plant sources was reduced by rigorous selection considering the main aspects given in Table 3.1. A general overview of the dyestuff production and dyeing step is given in Figure 3.2. These form the basis for analysis.

Table 3.1 The main requirements for a basic set of natural dyes

Agricultural demands	Requirements defined by a technical dye house
Reasonable Requirements for Production and harvesting of the plant materials	Simple and rapid dyeing process, no intermediate drying steps.
Easy handling and storage of the raw materials	One-bath dyeing
High dyestuff content	Broad range of shades formed by a basic set of brilliant dyes, including dark shades (black)
Easy extraction with water	Applicability in dyeing machines in use today
	Easy correction of deviations in colour depth and shade, Acceptable fastness properties Observance of existing waste water limits. No use of mordants based upon Cu, Sn, or Cr salts
	Bio-degradability of dyes in wastewater treatments
	Consumption of eco friendly chemicals

(e) Material is ready to compost without further treatment and can be used for other purposes,

(f) Use of watery plant- extracts is possible, use of solvent and chemicals is not intended,

(g) Good fastness of the dyed product,

(h) Applicability to the dyeing of protein fibre (wool), cellulose fibre (linen, cotton) and

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	Observance of existing waste water limits.
	No use of mordants based upon Cu, Sn, or Cr salts
	Bio-degradability of dyes in wastewater treatments
	Consumption of eco friendly chemicals

Agricultural products of plant material

Easy to harvest and handle
Simple storage

Hot water extraction

No chemicals added
No organic solvents

Residual plant material for further use as animal feed or soil conditioner

Aqueous dye stuff solution
(Formation of a deliverable solid or liquid product)

Dyeing Process

One bath dyeing
Technical requirements

Wastewater containing minimum chemical load
(Released to wastewater treatment plant)

Dyed product

Broad range of shades
Acceptable fastness properties

Figure 3.2 Dyestuff extraction and dyeing step.

3.3 Selection of fabric materials to be dyed

The characterization of fabric was carried out at Physical Testing Laboratories of the Department of Textiles and Clothing Technology, University of Moratuwa, Sri Lanka.

3.3.1 Macroscopic features:

Molecular structures, length, cross section were determined by using laboratory microscope (Make: AJAY OPTK, Model: CM/L – 9010250, India).

3.3.2 Physical properties:

Tenacity (g/den), Stretch and elasticity were measured by using Universal Testing Machine (Make: Instron , Model : 4465 , USA)

Moisture Regain - The fabric samples were conditioned in the standard atmosphere of 65% Relative Humidity and 27 ± 2 °C for four hours until the oven dry mass was obtained. Moisture regain was calculated using following equation.

$$MR = \frac{IM - ODM}{ODM} \qquad \text{----------------------------} \qquad (3\text{-}1)$$

Where,	MR	-	Moisture Regain
	IM	-	Initial Mass at standard atmosphere
	ODM	-	Oven Dry Mass

Specific gravity of fabrics and yarns was calculated by using a hydrometer.

3.4 Preparation of cloth for dyeing (fabric pre- treatment)

(a) Cotton fabrics

Grey cloth, as it comes from the loom stage, is unattractive and contains natural as well as added impurities, which hinders the successful operation of dyeing by reducing the absorption capacity of the fabric, hence it is necessary to make the fabric water absorbent, by making the fabric free from any natural as well as added impurities in order to achieve a successful dyeing process.

Preparation of the cotton cloth was carried out by using the following steps.

- Desizing
- Scouring
- Bleaching
- Mercerisation

Desizing:

Gray cotton cloth was impregnated in 5 % Genencore GC 2X desizing agent with 10 % alkaline (NaOH) solution at 80 °C for one hour with material to liquor ratio of 1: 50. Desizing was done at pH 6.5 - 8.0 for 1 hr.

Scouring:

Scouring was done to remove natural and added oil and waxes present in the desized fabric. The desized fabric was treated with 4 % Sodium hydroxide and 0.5 % detergent solution at 95 °C for 1 hour with material to liquor ratio of 1: 50.

Bleaching:

The scoured fabric was treated with 35 % Hydrogen peroxide solution at 90 °C for 1 hr keeping the material to liquor ratio at 1:50 to remove natural colouring matter.

Mercerisation:

Desized, scoured, bleached fabric was subjected to mercerization at low temperature under tension. 20 % NaOH , 52 sec. under tension at 20 °C.

5 g samples of each prepared fabric pieces were dyed individually with 47 selected extracts of bio-materials.

(b) Silk fabrics

The munga silk of 45.0 g/m^2 fabric was scoured with solution containing 0.5 g/l sodium carbonate and 2.0 g/l non-ionic detergent (Labolene) solution at 40 - 45 °C for 30 min, keeping the material to liquor ratio at 1: 50. The scoured material was thoroughly washed with tap water and dried at room temperature. The dried scoured material was then soaked in clean water for 30 min prior to dyeing or mordanting. Degumming was carried out by treating 5.0 g sample of silk with saturated hot soap solution at pH 10-12 for 45 min to remove natural gum present in silk.

(c) Wool yarns

The cleaned wool yarn of 60.0 g sample was scoured with solution containing 2.0 g/l non-ionic detergent (Labolene) solution at 30-35 °C for 30 min, keeping the material to liquor ratio at 1:50. The scoured material was thoroughly washed with tap water and dried at room temperature. The scoured material was soaked in clean water for 30 min prior to dyeing or mordanting.

Silk and wool were directly premordanted with metal salts; no tannic acid treatment is required for these fibres.

3.5 Extraction of colour yielding parts from the bio-materials

Following sequence of processes was followed to obtain colour yielding parts from the bio- materials.

(a) Drying
(b) Grinding
(c) Sieving
(d) Extraction
(e) Filtration

3.5.1 Drying

Figure 3.3 Laboratory drying oven

Most of the plant materials were dried in a laboratory oven (Make: SDL Atlas , Model : DP61, UK) at 37 °C until all the moisture was evaporated from the bio-material and a constant mass was obtained (Oven Dry Mass).

3.5.2 Grinding

Figure 3.4 Industrial grinding Machine

Dried materials were powdered to fine particles to obtain maximum extraction of colouring material from the bio-materials. This was carried out in industrial type grinding machine with 6000 rpm. (Make: Hauser, Model: S45-400, UK).

3.5.3 Sieving

Figure 3.5 Sieve analyser

Ground raw materials were subjected to sieve analyser (Make: Cilas, Model: 1190, UK) to obtain fine sand of uniform particle size (355 μm) of the raw materials.

3.6 Extraction of colourants:

The dye yielding components of the bio materials were extracted by the following three methodologies:

(a) Aqueous extraction

(b) Solvent extraction

(c) Sonicator extraction

3.6.1 Aqueous extraction

Raw materials (2.0 kg) were subjected to grinding and sieving. Ground and sieved raw materials (1.0 kg) were soaked over night. The MLR (Material to Liquor Ratio) of extraction bath was 1:8. For 1.0 kg of raw material initially 4 l of distilled water was added and plants parts were boiled. After about 1 hr another 4 l of water was added to the same extraction to ensure maximum extraction of dye yielding parts from the raw material. Extraction was carried out until the volume of bath reduced to 1 l for 2 hrs. After that it was left to cool down. This can be considered as concentrated natural dye and was used for dyeing of fabric samples. Figure 3.6 shows the extracted dye solution of *R. cordifolia* (Walmadata).

Table 3.2 Experimental liquor volumes

Wt. of raw material (kg)	Liquor volume (ml)	Final liquor volume (Concentrated) (ml)
1	4000 + 4000	1000

Figure 3.6 Aqueous extraction of *R. cordifolia* (Walmadata)

This aqueous extraction procedure was repeated for all the selected 47 natural dye giving bio-materials.

3.6.2 Solvent extraction

Sometimes colourants, present in natural sources, did not get extracted into the aqueous medium. In such a case, soxhlet was used to extract the natural colourant in the organic solvents. Mainly, the solvent used was methanol.

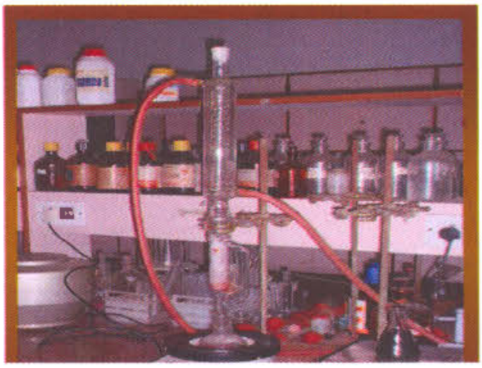


Figure 3.7 Solvent extraction unit

Colouring matters were extracted by using solvent extraction method. Bio-materials were cut into pieces, dried and were refluxed in soxhlet with methanol till it discharged the colour. The extraction process was carried out for 4-6 hours. This method was used for extraction of colouring matter from the finally selected ten samples.

3.6.3 Sonicator extraction

Figure 3.8 Sonicator

The extraction of colouring matter from bio-materials was carried out by using the sonicator. 100g of finely ground raw material was added to the sonicator bath. The sonicator used was of 20 kHz frequency and 150 W, (Make: Julabo, Model: 30, India). When the bath is irradiated with high energy, ultrasonic cavitation occurs which releases considerable amount of energy due to collapsing of the bubbles. This increases with the surface tension at the bubble interface and decreases with the vapour pressure of the liquid. Since the aqueous extraction bath has water, which has comparatively high surface tension, it is a very effective medium for extraction of maximum amount of colouring part.

3.7 Filtration

Figure 3.9 Vacuum filtration unit

The insoluble residue was separated by sedimentation and filtration through a stainless steel filter fabric (0.3 mm mesh). The extracted dye samples were cooled down and were subjected to filtration to get rid of fine solid particles to prevent deposition on the fabrics. The filtration was carried out by using a vacuum filtration unit (Make: Millipore, Model: HAWP04700, Thaiwan). The resulting extract was used for the dyeing of fabrics.

3.8 Mordanting

Three basic methods of mordanting are in vogue on yarns / fabrics. The use of different mordants changes the colour of a dyestuff and enhances the colour stability. Dyeing would depend upon the type of mordanting used. The three methods used for mordanting are:

(a) Pre-mordanting:

The cotton fabric was treated with 4 % (owf) solution of tannic acid prepared in water. The fabric was dipped in tannic acid solution for at least 4-5 hours and covered to avoid patchy stains on the fabric, squeezed and dried. In this method the yarn/fabric was mordanted in the first stage and then dyed in the second stage. An aqueous solution was prepared by dissolving required amount of suitable mordant in water. The yarn/fabric was entered and boiled for 30 to 45 min in the mordanted solution. The yarn/fabric was dyed in the prepared dye bath.

(b) Simultaneous Mordanting:

In this method the mordant and the dye were applied simultaneously in the same bath. The yarn/fabric was dipped in the extracted dye liquor and boiled for 15 min. The required amount of mordant was added to the extracted dye solution and stirred well and boiled for 30 to 45 min. Then the fabric was washed, rinsed and dried.

(c) Post-mordanting:

In this method the fabric was first dyed and then mordanted. The dye solution was prepared. The yarn/fabric was dyed in the dye solution. The aqueous solution was prepared by adding 5% of suitable mordant. The dyed material in the mordanting liquor was boiled for 30 to 45 min.

3.8.1 Selection of mordants

Two types of mordants were used to enhance the performance properties of dyed materials.

3.8.2 Synthetic mordants

Synthetic mordants are chemical substances which fulfill the above purposes. Some synthetic mordants were selected to use with plant colourant extracts are Ferrous sulphate, Potassium dichromate etc,.

3.8.3 Natural mordants

These are natural substances which give above mentioned properties. The natural mordants used were Aralu (*Terminalia chebula*) and Sepalika (*Nyctanthes arbor-tristis*).

3.9 Dyeing under different conditions

During the dyestuff selection, a one-bath dyeing process with the addition of the mordant into the dye bath was investigated to serve as the general dyeing procedure instead of a two-bath dyeing step with separated mordanting. The selection of a one-bath dyeing procedure was made with regard to the demands of the textile dyers, who would reject a two bath dyeing process with the arguments of handling, time consumption, and risks of lower reproducibility. The possibility for a variation in colour depth and shade with use of dyestuff mixtures and mixed mordants was found.

Initially extracted colourants were used to dye mercerized cotton fabric at 40 °C, 60 °C, and 80 °C. From above temperatures the best performing temperature was selected. Then the salt concentrations were varied. (i.e.10.0 g/l ,20.0 g/l ,30.0 g/l). From these salt concentrations best possible salt concentration was selected based on the better dye uptake and fastness properties. With the optimization of above parameters, pH of the individual dye baths was varied. i.e. pH <7 , pH =7, pH >7. pH of these dye baths were measured by using a pH metre (Make : Hanna: Model : H1 8314, Portugal). pH of the aqueous dye baths were determined by directly inserting pH meter into the dye bath while for methanolic extracted dye baths , the methanol was evaporated and extracted dye was dissolved in water. pH was measured in this aqueous solution. By using the above optimum conditions, samples were generated for all (47) selected bio-materials. The temperatures, salt concentrations and pH conditions followed are illustrated in the Figure 3.10.

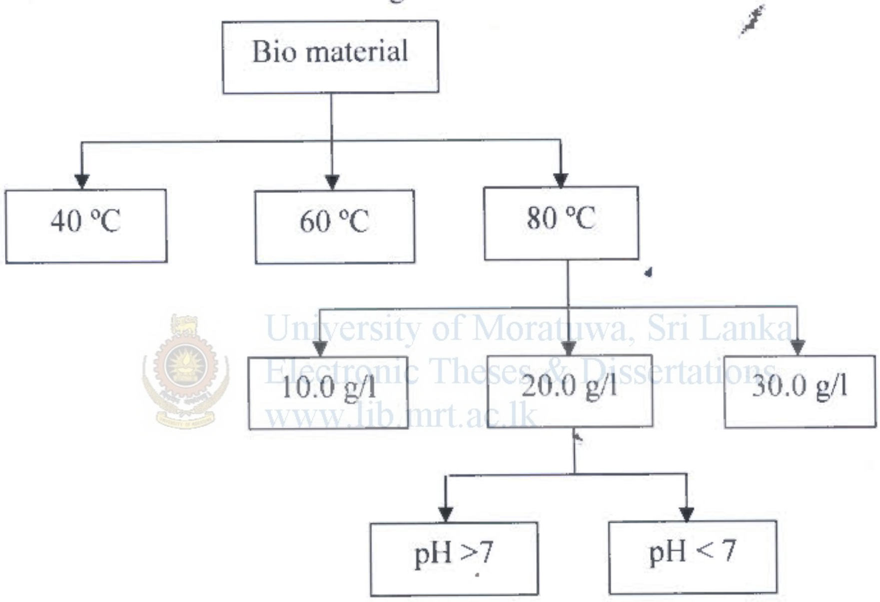


Figure 3.10 Selection of optimum conditions for dyeing

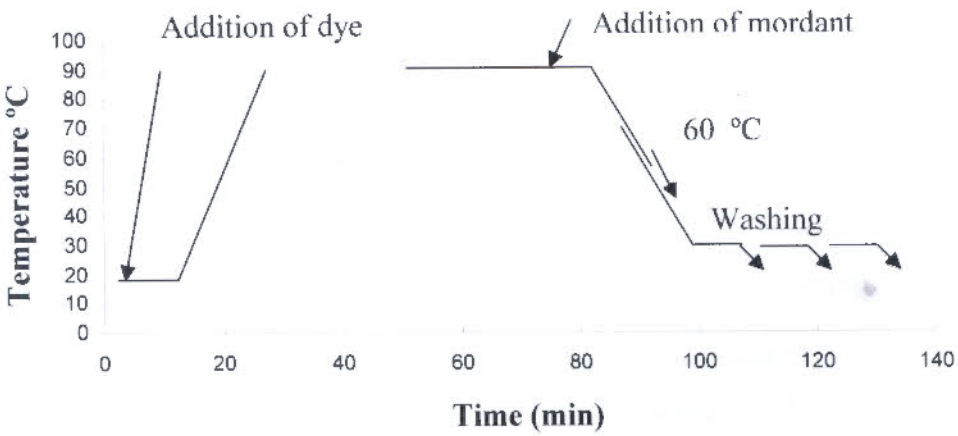


Figure 3.11 Temperature time diagram for dyeing process

The dyeing was performed on cellulosic material by the exhaustion method using a MLR of 1:20 (For 1.0 g of fabrics/yarn 20 ml of liquor) with cellulosic material.

Bleached wool yarn (10.0 g) was used as a protein fiber substrate with M.L.R. 1:20 at 50 °C for one hour. The dyeing trials were performed in a sample dyeing machine according to the temperature time diagram given in Figure 3.11.

3.10 Techniques used for dyeing

Two techniques of dyeing were used to compare the shades of dyed fabrics or yarns.

(a) Conventional dyeing

(b) Sonicator dyeing.

3.10.1 Conventional dyeing

Conventional dyeing of substrates were carried out at 95 °C for one hour in the sample dyeing machine (Make: Colour Pet 12, Model: 12 LMP, Japan) in the laboratories of the Department of Textile and Clothing Technology, University of Moratuwa, Sri Lanka.

Figure 3.12 Conventional dyeing in sample dyeing machine

3.10.2 Sonicator dyeing

Sonicator dyeing was carried out in the facility for Ecological and Analytical (FEAT) laboratories in Indian Institute of Technology, Kanpur, India. The same equipment (Make SR05: Model: Julabo, India) used for extraction was used for dyeing.

In sonicator dyeing 250 ml of extracted dye was added at the beginning to the dye bath. In all the dyeing processes, tap water was used. In sonicator dyeing, extracted dye was taken into sonicator bath and the treated fabric was dipped in for one hour at

40 °C. Dyed fabric was dipped in 4 % sodium chloride solution for one hour and then fabric was washed with tap water and dried.

3.11 Evaluation of performance properties

Evaluation of performance was carried out in accordance with the standard methods. Evaluation of fastness properties was done by measuring washing, light, rubbing and perspiration fastness values using Wash wheel, Microscal, Crock meter and Perspirometer respectively. The dyed samples were tested according to standard test methods as given in the Table 3.3.

Table 3.3 Standard reference numbers for fastness testing

Test parameter	Standard reference number
Colour fastness to Washing	ISO - 05 - C01 05
Colour fastness to Light	ISO - 105 - B02
Colour fastness to Rubbing	ISO - 105 -X12
Colour fastness to Perspiration	ISO - 105 - E04

All the testing of the fastness properties of the dyed fabrics were carried out at the wet Processing laboratory of the Department of Textile and Clothing Technology, University of Moratuwa.

3.11.1 Colour fastness to washing

For colour fastness to washing, ISO105 C 01 05, wash fastness ratings from 1 (fading) to 5 (excellent fastness) were used (Table 3.4).

3.11.2 Colour fastness to rubbing

This test method assesses the resistance of the colour of textile materials to rubbing off in the dry state or in the presence of moisture or solvent. Such rubbing of colour may result in fading or streaking, and/or staining of other materials.

For colour fastness to rubbing, ISO 105 X 12, rub fastness ratings from 1 (fading) to 5 (excellent fastness) were used, similar to wash fastness ratings (Table 3.4).

Table 3.4 Wash fastness (WF) and Rub fastness (RF) ratings

Rating No.	Description
1	severely fading
2	Poor fastness
3	Medium fastness
4	good fastness
5	Excellent fastness

3.11.3 Colour fastness to light

For colour fastness to light, ISO -105 – B 02, a Microscal to measure resistance to fading using a laboratory apparatus (Microscal; James H. Heal, UK) was used under the following conditions: light-exposure system featuring an air-cooled Microscal discharge lamp simulating outdoor global radiation; irradiation on sample level λ 300–400 and 400–700 nm; test chamber temperature: 25 °C; and relative humidity 65 %. A light fastness rating from 1 (severely fading) to 8 (excellent fastness) was made by comparing the resistance to fading of each sample to that of eight different blue tones.

Table 3.5 Light fastness (LF) ratings

Rating No.	Description
1	Severely fading
2	Fading
3	Medium fastness
4	Quite good fastness
5	Good fastness
6	Very good fastness
7	Exceptional fastness
8	Excellent fastness

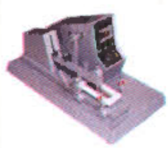
3.11.4 Colour fastness to perspiration

This assessment specifies a method for determining the resistance of the colour of textiles of all kinds and in all forms to the action of human perspiration. The fastness of colour when subjected to perspiration is a constant problem for manufacturers of clothing. To evaluate this phenomena colour fastness to perspiration, ISO 105 E04 was used. Perspiration fastness ratings from 1 (fading) to 5 (excellent fastness) were used, similar to wash fastness ratings and rub fastness ratings given in the Table 3.4.

3.12 Equipment used for performance analysis

Evaluation of fastness properties was done by measuring washing, light, rubbing and perspiration fastness values using following equipment given in the Table 3.6.

Table 3.6 Equipment used for performance evaluation

Test parameter	Equipment	Pictorial view	Manuf.	Make Model Year of Manf.
Colour fastness to Washing	Wash wheel		James H. Heal, UK	Thermolab,UK (2001)
Colour fastness to Rubbing	Crockmeter		James H. Heal, UK	Ravindra Eng.,India (2003)
Colour fastness to Light	Microscal		James H. Heal, UK	Microscal,UK (2000)
Colour fastness to Perspiration	Perspirometer		James H. Heal, Uk	Sashmira ,UK (1989)
Trace elements and Heavy metals	Atomic Absorption Unit		Perkin Elmer	Perkin Elmer, USA (2004)

3.13 Measurements and analysis

3.13.1 Colour measurements

The relative colour strength of dyed fabrics expressed as *K/S* was measured by the light reflectance technique using the Kubelka–Munk equation (3.2). The mathematical basis for all colour matching software is the Kubelka–Munk series of equations. These equations state that for opaque samples such as textile materials, the ratio of total light absorbed and scattered by a mixture of dyes is equal to the sum of the ratios of light absorbed and scattered by the dyes measured separately. The reflectance of dyed fabrics was measured on a Premier Colourscan.

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \qquad \text{--------------------------} \qquad (3\text{-}2)$$

where R - Decimal fraction of the reflectance of dyed fabric.

K - Absorption of characteristic of light

S - Scattering characteristic of light

3.13.2 Evaluation of parameters related to colour matching system

The reflectance of dyed fabrics was measured on a Premier Colourscan.

C.I.E. is for "Commission Internationale de l'Eclairage ", which in English is the "International Commission on Illumination". The C.I.E. system is used for colour specification. It describes all the colours visible to the human eye.

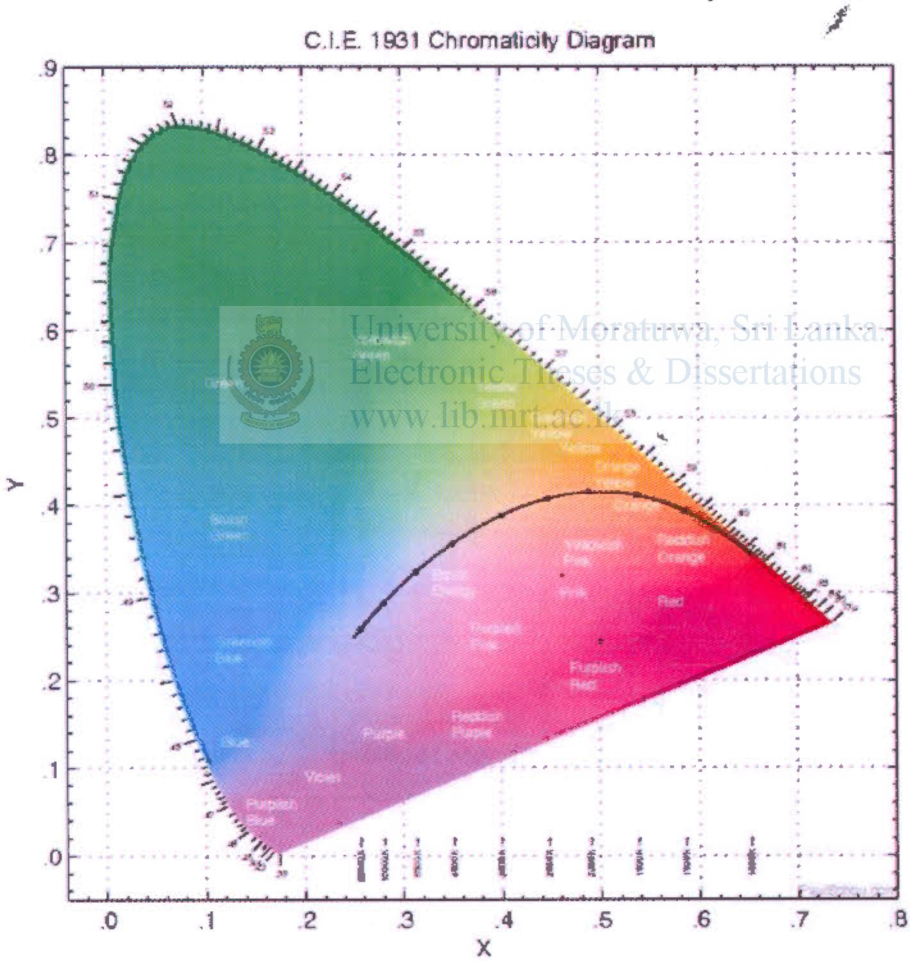


Figure 3.13 CIE Lab colour coordinate system

L* represents lightness value, the higher the lightness value represent lower the colour yield. a* and b* represent the tone of the colour, positive values of a* and b* represent redder and yellower tones while negative shows greener and bluer tones. C* represents chroma or purity of colour. h represent hue (shade) of colour.

The CIE Lab values were also recorded for all dyed samples along with controlled sample.

The CIE Lab values of the dyeings were measured with a tristimulus colourimeter. The colours are given in CIE lab coordinates, L* corresponding to the brightness (100 = white, 0 = black), a* to the red–green coordinate (positive sign = red, negative sign = green) and b* to the yellow–blue coordinate (positive sign = yellow, negative sign = blue).

3.13.3 Measurement of dye exhaustion

The mixtures of extracted dye were subjected to characterization through various techniques. The UV spectra for any dye extract give the typical absorbance values of the colourant which is specific. The extracted dye was diluted and dissolved in a suitable solvent system (water) and scanned through UV-Visible spectrophotometer. Absorbance measurements were done to identify characteristic of spectra.

3.14 Equipment used for analysis

Table 3.7 gives details of analytical equipments used for measurements and analysis.

Table 3.7 Equipment used in the performance analysis of dyed materials

Test parameter	Equipment	Picture
Characterisation of extracted dye	UV –Visible Spectroscopy (2001) (Make : Perkin Elmer : Model 295 spectrophotometer, USA)	
Colour Measurements	Premier Colour Scan (1999) (Make :Perkin Elmer, Model : SS6200A, India)	

3.15 Economic consideration

Ten selected plants were subjected to evaluate the economic feasibility of these natural dyes. Standard depth calculations enabled in determining required quantities for standard depth of shades.

Standard Depth Calculations were carried out according to the American Association of Textile Chemists and Colorists AATCC Evaluation Procedure 4 (Standard Depth Scales for Depth Determination).

Using the maximum dye yielding parameters, mercerized cotton samples were dyed with aqueous extraction of natural dyes until the standard depth shades were obtained. Finally the amount and cost of dye material needed to achieve standard depth was calculated.

3.16 Preparation of Ready – to –Dye Concentrates

The extracted colour yielding parts of the bio-materials were used for this purpose. The extracts obtained from section 3.6.1 were taken as Ready –to –Dye Concentrates (RTDC) Extract. Methyl paraban and Sodium benzoate were added to these dye solutions as preservatives. The efficiency of their chemicals as RTDC extract preservatives were also tested as shelf life observations.

3.17 Market potential in Sri Lanka

Apart from the examination of the technical and economic feasibility of the use of plant dyes in the textile industry; in the course of the project, market research was conducted in order to be able to give marketing recommendations. According to the findings of the survey, naturally dyed textiles should not be advertised by using the term "eco-textiles" because consumers associate negative emotions with this term (e.g. baggy clothes). Furthermore, they tend to assume that the textiles are not wash- and lightfast and have a limited sample board. However, successful communication strategies stress the exclusiveness, skin compatibility and naturalness of the product. Similar prejudices with regard to natural dyes can be found on the side of the dyeing industry as well. The entrepreneurs doubt that the use of natural dyes is possible in industrial plants. They argue that plant dyes are not reproducible and that good fastnesses and appealing colours are only achievable by the application of poisonous mordant.

A questionnaire (Annexure A) was prepared to evaluate the market potential of products from natural dyeing industry in Sri Lanka.

3.17.1 Analysis of questionnaire

The questionnaire was used with participants to several natural dye exhibitions and workshops. Following exhibitions which were held in Sri Lanka during the last couple of years were visited and comments and observations recorded.

(a) Art gallery Exhibition in Sri Lanka (year 2004)

(b) Art Gallery exhibition (year 2005)

(b) Exhibition at BMICH (Year 2005)

(c) Exhibition at BMICH (Year 2006)

(d) Exhibition at BMICH (Year 2007)

3.18 Evaluation of environmental impact

It is important to evaluate the natural dyes according to the environmental performance. Environmental Impact of the extracted natural dyes and the dyed fabric was assessed by testing for toxic heavy metals or trace elements.

Significance of environmental impacts was determined by analyzing for trace elements and heavy metals present in selected natural dye yielding plants. These data were analysed using Atomic Absorption Unit in the Atomic Energy Authority of Sri Lanka.

Toxic heavy metals content in the dye and the dyed fabric were determined by using Inductively Couple Plasma Optical Emission Spectrophotometer (ICP). For analysis, 1000 ppm solution 0.1 g sample digested in conc. Hydrochloric acid and made up to 100 ml by adding distilled water was used. The upper limits of trace elements detected were 10.0 mg/l.

3.19 A colour catalogue

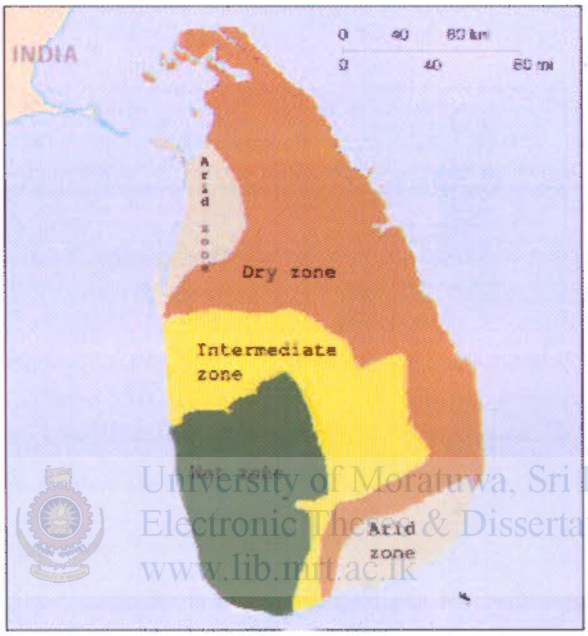
Results from the entire basic study were collected on to a resource compendium. The dyed results could be observed in the CD compilation attached to the thesis as supplementary material.

Chapter Four

RESULTS AND DISCUSSION

4.1 Indigenous dyes and dyeing methods

After carrying out the comprehensive literature survey on resources, following results were obtained in relation to the availability of natural dye sources in Sri Lanka (Figure 4.1).



Plant	Botanical Name	Zone wise area				
		Wet	Intermediate	Dry	Arid	Other
Beetroot	*Beta vulgaris*	■	■	■		
Henna	*Lawsonia inermis*				■	
Hora	*Dipterocarpus zeylanicus*			■		
Madder	*Rubia coordifolia*				■	
Jak	*Artocarpus heteropphyllus*	■	■	■		
Kadol	*Avicennia officinalis*				■	Coastal Area
Kone	*Schleichera oleosa oken.*			■		
Nilawariya	*Indigofera tinctoria*	■	■	■		Paddy fields
Patangi	*Caesalpinia sappan*				■	
Ranawara	*Cassia auriculata*			■	■	
Red onion	*Allium cepa*			■		Jaffna
Sepalika	*Nyctanthes arbor-tristis L.*		■	■		
Tea	*Camelia sinensis*	■	■			
Teak	*Tectona grandis*		■	■		
Turmeric	*Curcuma domestica*	■		■		
Tropical Almond	*Terminalia catappa*	■	■	■	■	
Vatake	*Acacia nilotica*				■	Coastal Area
Weniwel	*Consinium fenestratum*	■				

Figure 4.1 Availability of Natural dye sources - Regional Distribution

4.1.1 Traditional robe dyeing process

Figure 4.2 shows the developed (according to the scientific conditions) process flow diagram for traditional robe dyeing process.

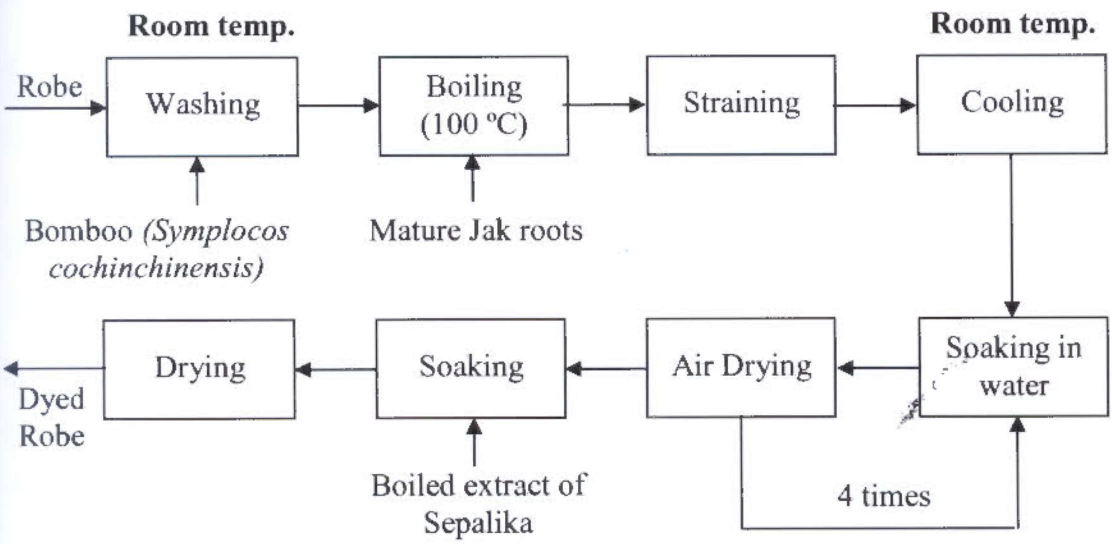


Figure 4.2 Developed process flow diagram for traditional robe dyeing

It was observed that the traditional technique continues to date. The objective of this part of the study was to study the shortcomings of the use of traditional dyeing techniques. Observed short comings in the traditional method were:

(a) Fabric pre-treatment was not carried out for better absorptivity of dyes.

(b) Dye bath concentration was not uniform

(c) Materials to liquor ratios were not properly selected.

(d) No definite quantity of salt addition.

(e) All the raw materials used were according to the traditional knowledge and practice. No up to date knowledge was applied.

(f) Adequate after treatments were not carried out.

(g) No quality control other than perceptive acceptance.

(1) **(2)** **(3)**

Figure 4.3 Traditional robe dyed fabric samples in the laboratory

Vithout Bomboo & Nuga (*Ficus benghalensis*), (2) With Bomboo & without Nuga, (3) With Nuga & without Bomboo

Above samples illustrate the shades given by the traditional method of robe dyeing carried out in the laboratory to compare the results with modern scientific conditions. This investigation was done to modify the traditional robe dyeing method with the scientific methods.

4.2 Investigation of dye yielding bio-materials for natural dye extraction

About 90 dye yielding plants in Sri Lanka were found as a result of literature survey (Annexure B). From these 90 plants, 47 bio-materials were selected for dyeing trials. Laboratory trials were carried out on these samples to determine the suitability of these bio-materials for commercial applications.

A colour catalogue consisting of the above 47 samples are prepared as a supplementary material given in the CD. After investigation of all 47 bio-materials for possibility of dyeing an evaluation matrix was prepared.

Selected Bio-materials, and their parts used for colouring matter extraction, the evaluation matrix for selection of best dye giving bio-materials and criteria for evaluation matrix are presented in the Table 4.7.

4.3 Selection of fabric material to be dyed

For the screening of 47 natural dye yielding plants, mercerized cotton fabric samples from the local industry were used in the laboratories of University of Moratuwa. Three types of fabric materials were used with the ten best selected bio-materials in the testing laboratories of Indian Institute of Technology, Kanpur, India. The selected fabric chacterisations are as follows:

4.3.1 Characteristics of cotton fabric

Physical parameters and properties of cotton fabric selected for dyeing is presented in Table 4.1.

Table 4.1 Characteristics of cotton fabric

Parameter	Properties
Molecular Structure	Cellulose
Macroscopic features	
Length	0.3 to 5.5 cm
Cross Section	Kidney shaped
Physical properties	
Tenacity (gf/den)	3.0-5.0 (dry), 3.6-6.0 (wet)
Stretch and elasticity	3-7 % elongation at break
Moisture Regain	8.5 %
Specific Gravity	1.54

4.3.2 Characteristics of silk fabric

Physical characteristics of silk fabrics are presented below.

Table 4.2 Characteristics of silk fabric

Parameter	Properties
Molecular Structure	Extended Protein (fibroin)
Macroscopic features	
Length	400 to 700 m
Cross Section	Triangular cross section
Physical properties	
Tenacity (gf/den)	2.4 to 5.1
Stretch and elasticity	15 % elongation at break. 90 % recovery at 2 % elongation
Moisture Regain	11 %
Specific Gravity	1.25

4.3.3 Characteristics of wool yarn

Physical characteristics of wool yarn are presented below.

Table 4.3 Characteristics of wool yarn

Parameter	Properties
Molecular Structure	Helical Protein (Keratin)
Macroscopic features	
Length	2 to 50 cm
Cross Section	Elliptical
Physical properties	
Tenacity (gf/den)	1 to 2
Stretch and elasticity	35 % elongation at break. 100 % recovery at 2 % elongation
Moisture Regain	13.6 to 16 %
Specific Gravity	1.30 – 1.32

4.4 Extraction of colourants from the bio-materials

For the maximum extraction of dye yielding matter from the bio-materials several steps were followed.

4.4.1 Grinding & Sieving

Different sizes of mesh were selected to get fine particles from the ground raw materials to ensure uniform extraction. Sieve analysis data for 25 samples out of 47 (which can be dried and ground) are attached in Annexure C.

Table 4.4 Sieve analysis data for *N. lappacium* sample

Mesh size	Retained wt.	Cumulative wt.	% Cumulative wt.
355	50	50	2.5
250	200	250	12.5
150	700	950	47.5
0	1050	2000	100

4.5 Extraction of colourants

Figure 4.4 Finely ground particles and their aqueous extracts

All 47 selected samples were subjected to aqueous extraction. 47 different extracted solutions were realized. Figure 4.4 presents a collection of few extracted solutions from 47 colour extracts.

Both conventional dyeing and sonicator dyeing of aqueous extracts were carried out to compare the best dye yielding conditions.

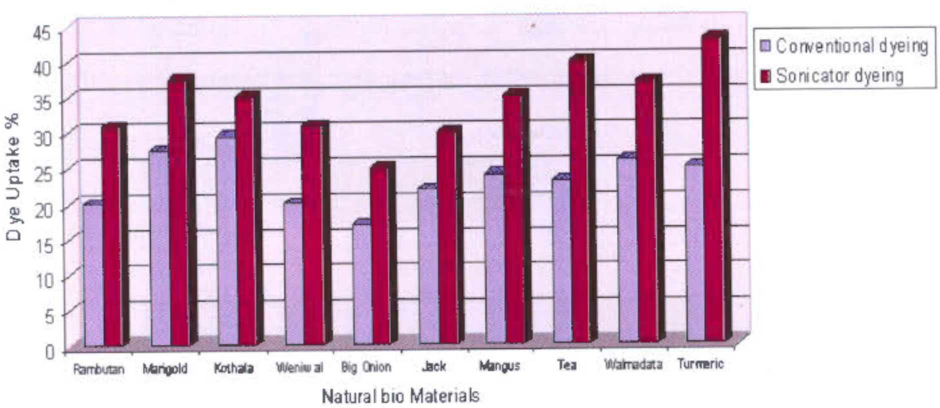


Figure 4.5 Dye uptake for bio-materials by different dyeing methods

Table 4.5 Dye uptake values for different dyeing techniques

Local name	Botanical name	% dye uptake in conventional dyeing	% dye uptake in sonicator dyeing	% improvement in dyeing
Rambutan	*N. lappaceum*	20	31	55
Marigold	*T. erecta*	27.5	37.5	36
Kothala	*S. reticulate*	29.5	35	17
Weniwelgata	*C. fenestratum*	20	31	55
Big onion	*A. cepa*	17	25	47
Jak	*A. heterophyllus*	22	30	36
Mangus	*G. mangostana*	24	35	37.5
Tea	*C. sinensis*	23	40	74
Walmadata	*R. cordifolia*	26	37	42
Turmeric	*C. domestica valet*	26	44	69

From the above graph it can be concluded that about 47 % of average improvement of dyeing can be achieved by sonicator dyeing.

4.6 Optimization of dyeing conditions

To obtain the maximum absorptivity of dyes by fabrics, dyeing parameters should be optimized. The important dyeing parameters considered in this study are; temperature of the dye bath, salt concentration, time taken and pH of the dye bath. As the initial assessment optimum dyeing temperature at 80 °C was selected. At this optimum temperature, i.e. by keeping the temperature constant at 80 °C, the optimum salt concentration was given as 20.0 g/l. By keeping above all two parameters constant optimum pH was selected. It showed pH < 7 was the suitable range. The sequence of optimum conditions obtained was illustrated in the Figure 4.6.

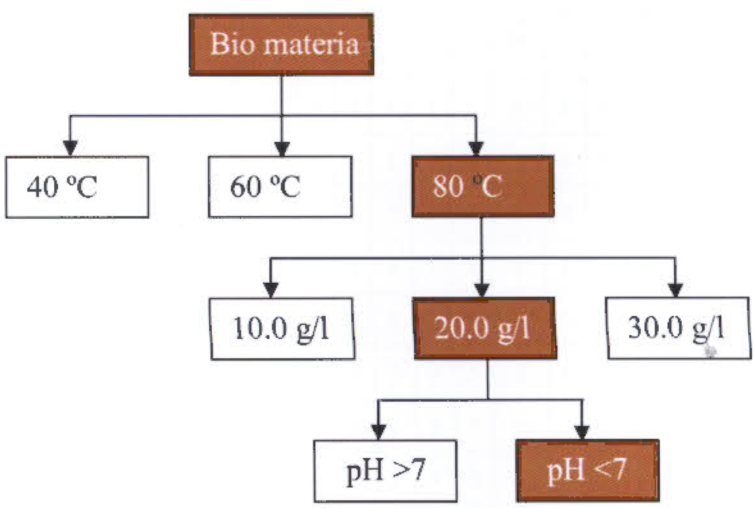


Figure 4.6 Dyeing conditions

4.7 Mordanting

By using two types of natural mordants Aralu *(Terminalia chebula)* & Sepalika *(Nyctanthus arbo-tristis)* and one synthetic mordants (Copper sulphate) all 47 samples were dyed with simultaneous and post mordanting methods. The Colour catalogue prepared with different conditions presented in the CD as a supplementary material. A colour catalogue is presented in this manner to save space as well as for facilitating data comparisons later.

4.8 Evaluation of fastness properties

Fastness properties are very important. Fastness properties of all dyed fabric samples were tested against colour fastness to Washing, Rubbing, Perspiration and Light. Results are tabulated in evaluation matrix (Table 4.6).

4.9 Selection of dye yielding plants

Initial experiments were carried out on 47 types of bio-materials. However, it is important to concentrate on a few selected bio-materials and prioritize from this list. An evaluation matrix was developed and used for prioritizing. The matrix was formed under five areas. Evaluation matrix was generated to select the best bio-materials. The generated matrix is shown in Table 4.6.

Table 4.6 Characteristics of selected natural dyes in Sri Lanka

Bio- Material	Botanical names	Part Used	Raw Material			Process Requirement					Fixation				Application			Environmental Aspects			Total
			Availability	Ease of Extr.	Yield	Temperature	Time	Salt	pH	MLR	Wash Fastness	Rubbing	Light	Perspiration	Ease of application	Levelness	Shade	COD	Trace elements	Waste or N'waste	
Walmadata	*Rubia cordifolia*	Root	10	10	10	4	4	3	3	5	9	8	8	8	10	7	9	10	0	1	119
Madan Pothu	*Syzygium cumini*	Stem	8	8	7	4	4	3	3	5	7	6	6	3	8	5	4	9	0	2	92
Venival	*Coscinium fenestratum*	Stem	10	10	10	4	4	3	3	5	9	8	7	4	10	8	9	10	0	1	115
Turmeric	*Curcuma domestica valet*	Rhysome	10	10	10	4	4	3	3	5	9	8	5	9	10	10	10	9	0	2	121
Kothala Himbutu	*Salacia reticulata*	Bark	10	10	10	4	4	3	3	5	9	7	7	7	10	7	8	10	0	2	116
Delum (Skin)	*Punica granatum*	Fruit Skin	8	8	8	4	4	3	3	5	8	6	7	6	8	7	8	8	0	8	109
Rath Handun	*Pterocarpus santalinus*	Stem	9	9	10	4	4	3	3	5	10	7	6	6	10	7	7	9	0	2	111
Ranawara	*Cassia auriculata*	Flowers	8	7	7	4	4	3	3	5	6	6	5	5	9	4	3	9	0	1	89
Aralu	*Terminalia chebula*	Bark	9	9	9	4	4	3	3	5	9	7	7	7	9	5	6	9	0	1	106

Table 4.6 Contd…

Bulu	*Terminalia belerica*	Bark	9	9	9	4	4	3	3	5	9	6	4	5	9	6	5	9	0	1	100
Munamal Pothu	*Mimusops elengi*	stem	8	8	8	4	4	3	3	5	8	6	5	4	8	3	6	9	0	1	93
Jak	*Artocarpus heterophyllus*	Saw Dust	10	10	10	4	4	3	3	5	10	9	9	9	10	9	9	10	0	10	134
Mangustene	*Garcinia mangostana*	Fruit Skin	10	10	10	4	4	3	3	5	10	9	9	9	10	9	9	10	0	10	134
Rambutan (Yellow)	*Nephelium lappaceum*	Fruit Skin	10	10	10	4	4	3	3	5	10	9	9	9	10	9	9	10	0	10	134
Rambutan (Red)	*Nephelium lappaceum*	Fruit Skin	10	10	10	4	4	3	3	5	10	8	8	8	10	9	9	10	0	10	131
Merygold (Orange)-1	*Tegetes erecta*	Petals	10	10	10	4	4	3	3	5	10	8	7	8	10	8	8	10	0	10	128
Big Onion	*Allium cepa*	Skin	10	10	10	4	4	3	3	5	10	9	9	9	10	9	9	10	0	10	134
Red onion	*Allium rubrum*	Skin	10	10	7	4	4	3	3	5	7	8	8	8	10	8	7	10	0	10	122
Wada	*Hibiscus rosa-sinensis*	Flowers	10	10	2	4	4	3	3	5	1	2	3	3	2	1	3	2	0	10	68
Tea	*Camellia sinensis*	Used Leaves	10	10	10	4	4	3	3	5	10	9	9	[illegible]	10	9	9	10	0	10	134
Katarolu	*Clitoria ternatea*	Flowers	8	9	8	4	4	3	3	5	0	1	2	2	0	3	2	6	0	9	69
Kuppamaniya	*Allium porrum*	Leaves	8	2	3	4	4	3	3	5	0	3	2	1	0	1	3	5	0	8	55
Kopi Dalu (Boiled)	*Coffea arabica*	Leaves	8	2	2	4	4	3	3	5	2	4	3	2	0	3	4	8	0	7	64
Kopi Dalu (Crushed)	*Coffea arabica*	Leaves	8	9	9	4	4	3	3	5	7	3	2	3	6	5	3	8	0	7	89
Kopi Powder	*Coffea Arabica*	Seeds	8	9	9	4	4	3	3	5	8	6	4	5	8	4	6	7	0	0	93
Kottamba	*Terminalia catappa*	Ripped Leaves	9	9	9	4	4	3	3	5	6	5	6	6	8	7	7	8	0	9	108
Devadara	*Erythroxylumm onogynum*	Stem & bark	1	2	1	4	4	3	3	5	1	1	1	1	0	1	1	6	0	0	35
Beet root	*Beta vulgaris*	Stem	7	9	9	4	4	3	3	5	0	1	1	1	0	1	1	7	0	0	56
Hik	*Lannea coromandelica*	Stem	3	8	9	4	4	3	3	5	8	6	6	6	9	5	6	8	0	1	94
Kumbuk	*Terminalia arjuna*	Stem	3	8	9	4	4	3	3	5	8	5	4	5	9	6	7	8	0	1	92
Mahogani	*Swietenia macrophylla*	Stem	2	8	9	4	4	3	3	5	6	4	3	2	9	6	7	8	0	9	92
Ehala	*Cassia fistula*	Bark	5	6	6	4	4	3	3	5	5	2	3	2	6	4	5	7	0	6	76
Thekka	*Tectona grandis*	Leaves	1	8	9	4	4	3	3	5	3	4	5	3	7	6	7	6	0	1	79
Pangiri mana	*Cymbopogon nardus*	Leaves	2	3	4	4	4	3	3	5	1	2	1	2	6	1	2	7	0	8	58
Beetle nut (Crushed)	*Piper betle*	Leaves	7	9	9	4	4	3	3	5	5	3	2	4	8	3	2	7	0	1	79
Beetle nut (Boiled)	*Piper betle*	Leaves	7	3	2	4	4	3	3	5	2	3	2	1	1	1	3	7	0	1	52
Eucalyptus	*Eucalyptus resinifera*	Stem	1	2	2	4	4	3	3	5	2	1	2	3	3	2	3	7	0	1	48
Marathondi (boiled)	*Lawsonia inermis*	Leaves	1	2	2	4	4	3	3	5	3	8	6	4	2	4	6	7	0	0	64
Marathondi (Crushed)	*Lawsonia inermis*	Leaves	1	9	9	4	4	3	3	5	8	8	6	4	8	8	7	7	0	0	94
Sera	*Cymbopogon citrates*	Leaves	2	3	4	4	4	3	3	5	3	2	3	2	5	1	2	7	0	5	58
Nuga	*Ficus altissima var ferugsonii*	Stem and bark	2	3	4	4	4	3	3	5	5	2	3	2	5	3	2	7	0	1	58
Beli	*Aegle marmelos*	Fruit	2	5	8	4	4	3	3	5	6	3	2	3	6	5	7	7	0	1	74
Ratu kaha	*Bixa Orellana*	Seed coat	2	9	9	4	4	3	3	5	9	5	6	7	9	8	8	7	0	1	99

Table 4.6 Contd…

Iramusu	*Hemidesmus indicus*	Bark and stem	5	7	7	4	4	3	3	5	4	1	2	4	7	3	2	7	0	1	69
King coconut	*Eugenia bracteata*	Husk	10	8	8	4	4	3	3	5	9	8	3	4	5	3	5	8	0	10	100
Sepalika	*Nyctanthes arbor-tristis*	Flowers	10	10	10	4	4	3	3	5	7	6	6	5	9	9	9	7	0	10	117
Areconut	*Areca catechu*	seed	10	6	6	4	4	3	3	5	8	7	6	4	8	8	5	7	0	10	104
Areconut + Betlenut	*Areca catechu + Piper betle*	N/A	10	9	9	4	4	3	3	5	9	8	7	5	7	8	9	8	0	6	114
Kesel Muwa	*Musa sapientum*	Muwa	10	8	8	4	4	3	3	5	9	7	7	7	8	7	8	8	0	2	108
Ahu	*Morinda citrifolia*	Bark	6	7	6	4	4	3	3	5	7	6	7	6	8	7	8	7	0	5	99
Ahu	*Morinda citrifolia*	Root	6	7	6	4	4	3	3	5	7	6	7	6	8	7	8	7	0	5	99
Gammalu	*Pterocarpus marsupium*	Milk	5	8	8	4	4	3	3	5	5	6	7	4	9	8	9	7	0	1	96
Kohomba	*Azadirachta Indica*	Bark	10	10	10	4	4	3	3	5	7	6	5	4	8	7	8	7	0	2	103

4.10 Evaluation matrix and tested samples in the laboratory

Following parameters were selected for the evaluation of dye yielding plants having the most potential characteristics as a colouring matter for textile substrates. The scale used is 1-10.

(a) Raw material

In the case of raw material selection the following aspects were considered

Availability	Plant materials for dye extraction should be frequently available in sufficient quantities. After carrying out a market survey conclusions were drawn. Eg. If it is readily available and a waste material rating given are 10. If it is a medicinal plant, rating given is less than this rating.
Ease of Extraction	The extraction procedure should also be easy and convenient and supportive in scaling up. If by simple water extraction it is not possible the rating given is in between 1-5. If the extraction can be done by simple extraction methods rating 8-10 is given.
Yield	The yield obtained from the extraction should be high. Such high yields results in realizing the quantity through a smaller resource base. Therefore if dark colours are obtained the rating given is in between 8-10. If the colour of extraction bath obtained is pale the rating given is in between 1-5.

(b) Process requirements

Temperature — There should be an optimum extraction temperature. If it is at alow temperature, rating given is in between 6-10. If it is at elevated temperatures rating given is 1-5.

Time — Ease of extraction means lower processing time. If the lower processing time is required rating given is 8-10. If the processing time is considerably high rating given is 1-5.

Salt — Minimum amount of salt concentration should be used. If it is a low amount, rating given is in between 8-10. If the considerable amount of salt is used, rating falls in between1-5.

pH — pH should be adjusted to the optimum extraction conditions. For some plants better extraction can be taken under acidic medium while for other plants the medium can be alkaline or neutral. In that case rating depends on the conditions applied.

MLR — Material to liquor ratios should also be in optimum range. If dyeing can be done with low liquor ratios (5-20), that condition is better and hence a higher rating is given.i.e. 8-10. If high liquor ratios (50-100) are used, a low rating is given i.e. 1-5.

(c) Fixation

If the dye fixation is in the acceptable range, following ratings should be achieved in relation to quality of fabrics.

Wash Fastness — Fastness to washing should be in between 4-4/5. For these, rating is 10.

Rubbing — Fastness to rubbing should be in between 4-4/5.For these, rating is 10.

Light — Fastness to light should be more than 5. For these, rating is 10.

Perspiration — Fastness to perspiration should be in between 4-4/5. For these, rating is 10.

(d) Application

Ease of application — If it is easy to apply on to the fabrics and absorption is optimum, higher rating is given i.e. 8-10. If the application of dyes is complicated rating given is at low range. i.e. from 1-5.

Levelness — If the levelness is satisfactory rating given was 8-10 poor levelness is obtained rating given is less. i.e. 1-5.

Shade — If brighter shades are obtained the rating given is 8-10. If pale shades are obtained, the rating given is 1-5 depending on the appearance.

(e) Environmental aspects

Three aspects were considered under environmental consideration. They are Chemical Oxygen Demand (COD), Presence of heavy metals and the source of raw materials.

Chemical Oxygen Demand	If COD is less high ratings are given. If COD is higher a lower rating is given
Presence of heavy metals	If low content of heavy metals are present, high ratings are given; if the heavy metal content is high a rating is low.
Source of raw material (Waste or non waste)	If the raw material is a waste material, high rating is given i.e. in between 8-10. If it is a non - waste material rating given is lower i.e., 1-5.

When selecting dye yielding bio-materials for dyeing of textile substrates waste materials were considered to be more useful. For example Jak sawdust, Rumbutan skin, Mangus skin, Marigold flowers, Tea waste etc.

4.11 List of the plant materials selected

Above mentioned parameters were evaluated by inserting data into an evaluation matrix. Prioritization was given only to ten (10) bio-materials for further studies. These ten bio-materials were subjected to the same experimental studies but in greater detail in a repetitive manner.

Table 4.7 Selected bio- materials for detailed studies

Local name	**Part used**	**Botanical name**	**Total score**
Big Onion	Skin	*Allium cepa*	134
Jak	Saw dust	*Artocarpus heterophyllus*	134
Kothala Himbutu	Bark	*Salacia reticulate*	116
Mangustene	Fruit skin	*Garcinia mangostana*	134
Marygold (Orange)	Petals	*Tegetus erecta*	128
Rambutan (Yellow)	Fruit skin	*Nephelium lappaceum*	134
Tea	Used leaves	*Camellia sinensis*	134
Turmeric	Rhysome	*Curcuma domestica valet*	121
Venival	Stem	*Coscinium fenestratum*	115
Walmadata	Root and stem	*Rubia cordifolia*	119

4.12 Detailed analysis of ten selected resource streams

The following observations were made from the investigations on screening, extraction and characterization of dye yielding bio-materials in detail. For the selection of ten best possible dye yielding bio materials all 47 samples were subjected to aqueous extraction. After selecting these ten bio-materials, methanolic extraction was used to get better dye yield for further investigation.

(a) Optimisation of dyeing and extraction parameters

In the case of aqueous extraction optimum conditions were obtained by trial and error methods. The optimum parameters for dyeing for all the ten samples are as follows unless stated otherwise.

Dye extraction Temperature	100 °C
Dye extraction Time	1 h
pH of the extract	4-9
Dyeing Time	1 h
Dyeing Temperature	80 °C
Mordanting Time	1 h
Material to Liquor Ratio	1: 50

(b) Colour measurements: K/S was measured for cotton, silk fabrics and wool yarns by using Premier Colourscan. K/S values vs. wavelength graphs show change in K/S value for different mordants for a particular fabric. Also the scanned graphs are given for 400 – 700 nm range of wavelengths and as far as the Y axis is concerned it may not be depicting the actual value and just to keep in scale (These graphs and corosponding K/S values generate and display on the computer screen for particular fabrics). The corresponding tables (for cotton, silk and wool) illustrate only λ_{max} values (wavelength at which the absorbance is maximum) attained in a particular wavelength.

4.12.1 Rambutan (*Nephelium lappaceum)*

Botanical Name	:	*Nephelium lappaceum*
Family	:	Sapindacea
Common Names English	:	Rambutan
Source	:	Dried skins were collected from home gardens and fruit sellers
Availability	:	May - August

a b

Figure 4.7 a, b Raw *N. lappaceum* fruit and dried pericarps

(a) Characterization

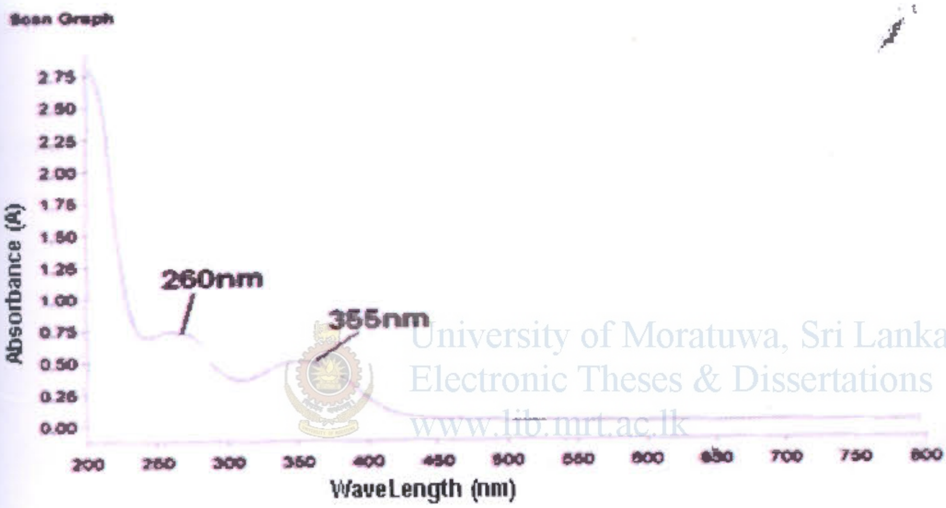


Figure 4.8 UV-Vis spectrum of methanolic extract of *N. lappaceum*

Visible spectrum of *N. lappaceum* extract: The methanolic extract from *N. lappaceum* shows peak at 260 nm and 355 nm in the above diagram.

UV/Vis absorption is not, however, a specific test for any given mixture of extracts. The nature of the solvent, the pH of the solution, temperature, high electrolyte concentrations, and the presence of interfering substances can influence the absorption spectra of the mixture, as can variations in slit width (effective band width) in the spectrophotometer.

(b) Colour measurements : K/S values for cotton, silk fabrics and wool yarns are shown in Figures 4.9 - 4.11 and CIELab values are shown in Tables 4.8- 4.10.

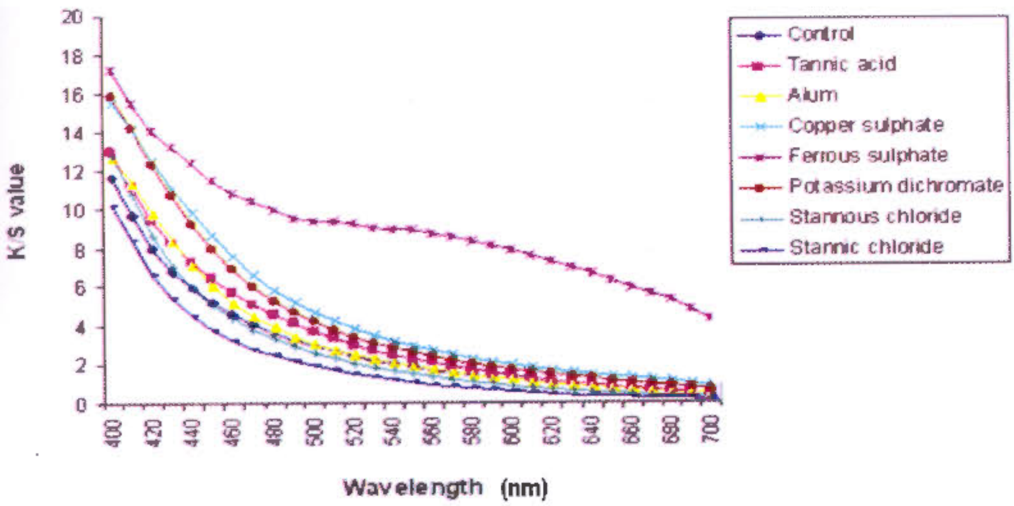


Figure 4.9 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *N. lappaceum*.

Table 4.8 Characteristics for cotton fabrics dyed with methanolic extract of *N.lappaceum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	50.265	8.79	26.61	28.02	71.68	55.66
	Control + Tan. acid	50.314	9.76	27.01	28.72	70.09	68.21
	Alum	51.800	8.44	29.56	30.74	74.03	62.42
	Copper sulphate	50.306	7.29	26.87	27.84	74.78	89.98
	Ferrous sulphate	43.126	1.44	7.30	7.44	78.82	170.11
	Pot. Dichromate	50.887	7.28	27.82	28.76	75.30	83.44
	Stannous chloride	52.850	10.64	31.68	33.42	71.41	52.01
	Stannic chloride	53.694	11.80	33.33	35.36	70.46	68.81

By considering the above graphs the sequence of order of K/S values are Fe > Cu > K > Al > Sn (II) > Sn (IV) in cotton for *N. lappaceum,* and results show the absorption of colour by cotton fabric was enhanced when using metal mordants. For cotton fabrics best mordant is Ferrous sulphate having a maximum K/S value of 170.11.

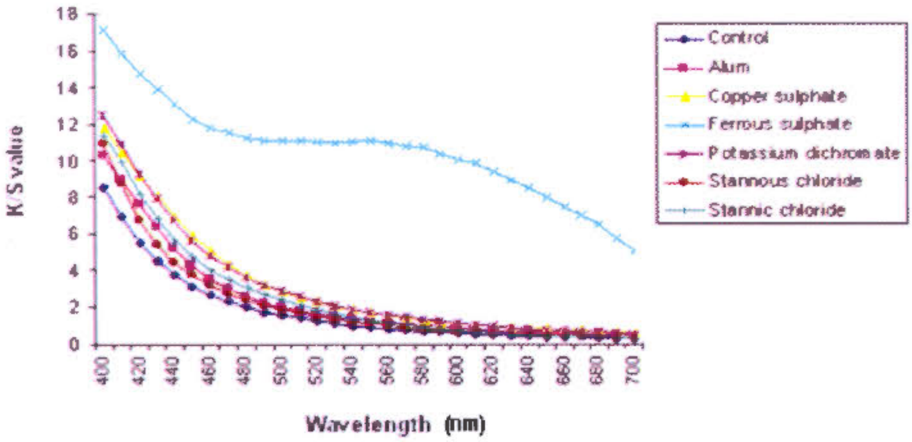


Figure 4.10 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *N. lappaceum.*

Table 4.9 Characteristics for silk fabrics dyed with methanolic extract of *N. lappaceum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	60.097	6.24	30.61	31.24	78.44	32.96
	Alum	59.769	4.38	29.52	29.85	81.51	45.26
	Copper sulphate	59.629	5.68	30.25	30.78	79.32	59.51
	Ferrous sulphate	50.203	1.28	4.03	4.23	72.32	197.44
	Pot. dichromate	59.250	5.83	29.17	29.74	78.65	59.35
	Stannous chloride	60.046	6.47	30.40	31.08	77.93	39.88
	Stannic chloride	60.978	8.94	32.90	34.10	74.77	48.19

From the above graphs the order of K/S values is as following: Fe > Cu > K > Sn (IV) > Al > Sn (II) in silk for metanolic extract of *N. lappaceum*, and can be concluded the absorption of colour by silk fabric was enhanced when using metal mordants. The optimum K/S value for this combination is 197.44.

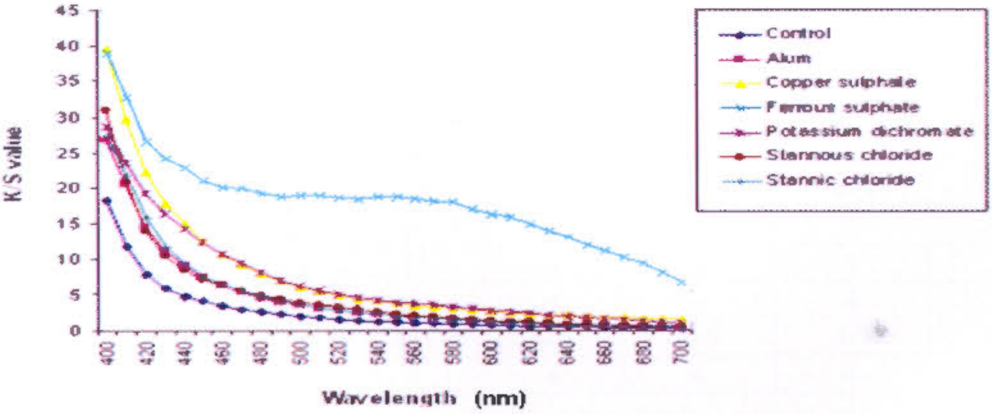


Figure 4.11 Change in K/S values with different mordants for wool yarns after dyeing with methanolic extract of *N. lappaceum*

Table 4.10 Characteristics for wool yarns dyed with metanolic extract of *N. lappaceum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	57.567	8.23	32.88	33.89	75.91	43.26
	Alum	57.633	8.70	33.03	34.16	75.21	79.39
	Copper sulphate	55.814	4.88	29.60	30.00	80.60	133.54
	Ferrous sulphate	46.34	2.19	5.06	5.52	6658	335.45
	Pot. Dichromate	54.605	5.21	27.83	28.32	79.34	130.17
	Stannous chloride	56.154	9.91	30.16	31.74	71.78	80.30
	Stannic chloride	58.300	9.51	34.18	35.48	74.43	80.94

The order of K/S values is: Fe > Cu > K > Sn (IV) > Sn (II) > Al in wool for methanolic extract of *N. lappaceum*. The optimum K/S value 335.45 was obtained when using ferrous sulphate as metal mordant for wool yarn.

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison.

(c) Fastness properties of dyed fabric

The fastness properties of dyed fabrics from the methanolic extract of *N. lappaceum* are as follows:

Table 4.11 Fastness properties of dyed cotton, silk fabrics and wool yarns under conventional conditions of metal modanting with methanolic extract of *N. lappaceum*.

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Control)	3-4	3	3	3	3	3-4
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Wool (Control)	3	3	3	3	3	3
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5
Silk (Control)	3	3	3	3	3	3
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness , Per – Perspiration fastness., Rub – Rubbing fastness.

From the above results overall fastness properties of *N. lappaceum* dyed fabrics are in acceptable range. Hence this type of dyes can be considered as suitable for dyeing of textile substrates.

The dyeing of cotton fabric with metal mordant by the natural dye *N. lappaceum*, shows that by this process very good results of even dyeing are obtained. The dye uptake in case of cotton dyeing ranges from 53-64 %, for silk 55-70 % and wool 57-69 % with different mordants. The effectiveness of metal mordant of *N. lappaceum* showed better dye uptake appears to be an improved process resulting in good dye adherence which results in good fastness properties.

Dye shades given by different fabric samples after dyeing with methanolic extract of *N. lappaceum* are shown in Figure 4.12.

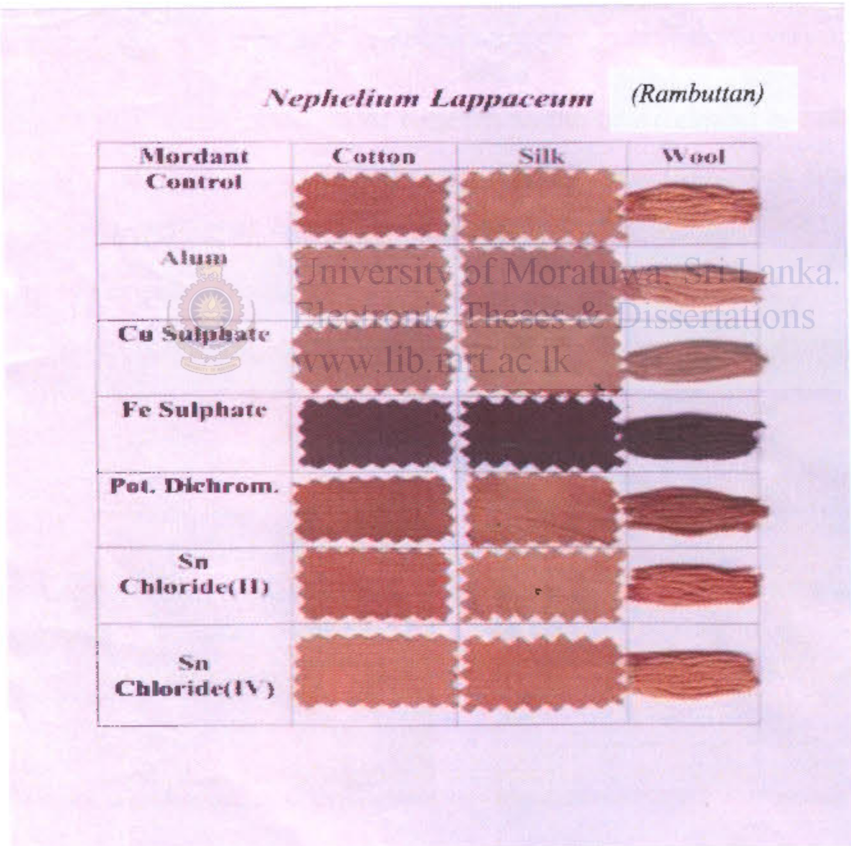


Figure 4.12 Fabric samples dyed with methanolic extract of *N. lappaceum* with different mordants

These different colourful shades can be generated by using methanolic extract of *N. lappaceum* to dye textile substrates. For example dark brown was obtained when using

using Ferrous sulphate as metal mordant for cotton, silk and wool yarns as shown in Figure 4.12.

(d) Characterization of environmental impact

Trace elements levels in the dye, and Chemical Oxygen Demand (COD) of methanolic extract of *N. lappaceum* and fabric dyed with *N. lappaceum* extract in mg/l are tabulated in Table 4.12.

Table 4.12 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.12	ND	0.03	ND	ND	ND	0.4	0.14
Dyed fabric (mg/l)	0.02	0.12	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	237 -300								

ND – Not detected up to 10mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from a textile dyeing operation involving *N. lappaceum* extract.

4.12.2 Marigold *(Tegetus erecta)*

a b

Figure 4.13 a, b Fresh *T.erecta* and dried petals

Botanical name - *Tegetus erecta*

Family - Asteraceae

Common name - Marigold

Source - Cultivated throughout Sri Lanka, from home gardens and from flower sellers, From religious places

(a) Characterization

Visible spectrum of *T. erecta*: The yellow coloured methanolic extract of *T.erecta* shows peak at 430 nm.

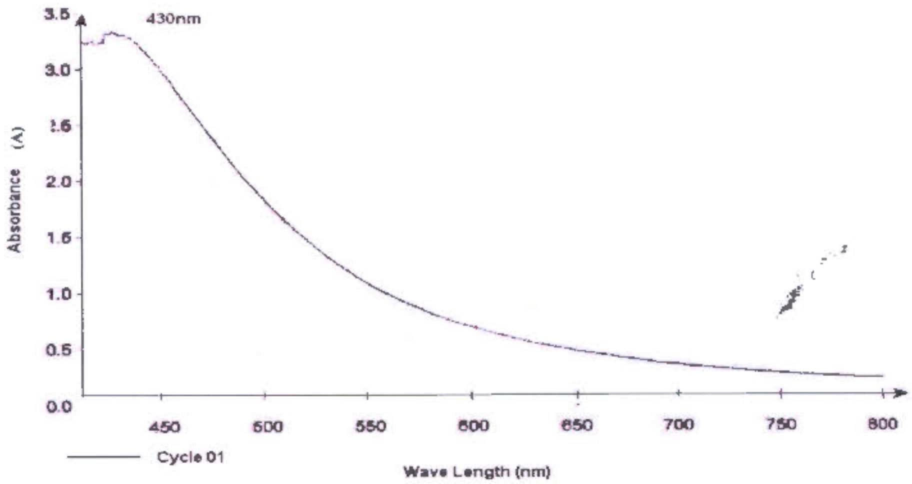


Figure 4.14 UV- Vis spectrum of methanolic extract of *T.erecta*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.15 – 4.17 and CIE lab values are shown in Tables 4.13 – 4.15.

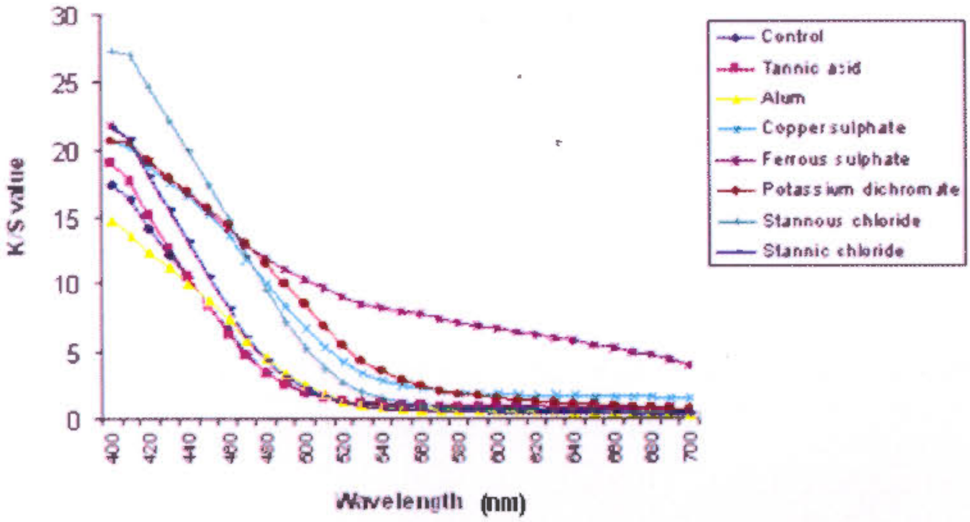


Figure 4.15 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *T.erecta*

Table 4.13 Characteristics for cotton fabric dyed with methanolic extract of *T.erecta*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control (Without mordant)	56.62	-0.88	44.06	44.07	--	72.64
	Tannic acid and dye	55.97	-2.07	42.30	42.35	2.23	74.86
	Alum	59.45	5.56	51.79	52.09	10.44	79.98
	Copper sulphate	52.16	6.44	39.43	39.95	9.49	131.65
	Stannous chloride	62.84	11.09	58.50	59.54	19.75	136.70
	Ferrous sulphate	41.68	2.09	15.25	15.39	32.59	187.77
	Pot. Dichromate	52.45	14.64	40.47	43.04	16.47	138.84
	Stannic chloride	63.56	4.01	56.87	57.01	15.36	86.01

The order of K/S values is Fe > K > Sn (II) > Cu > Sn (IV) > Al in cotton for methanolic extract of *T. erecta,* the best metal mordant for cotton fabric is ferrous sulphate having best K/S value of 187.77.

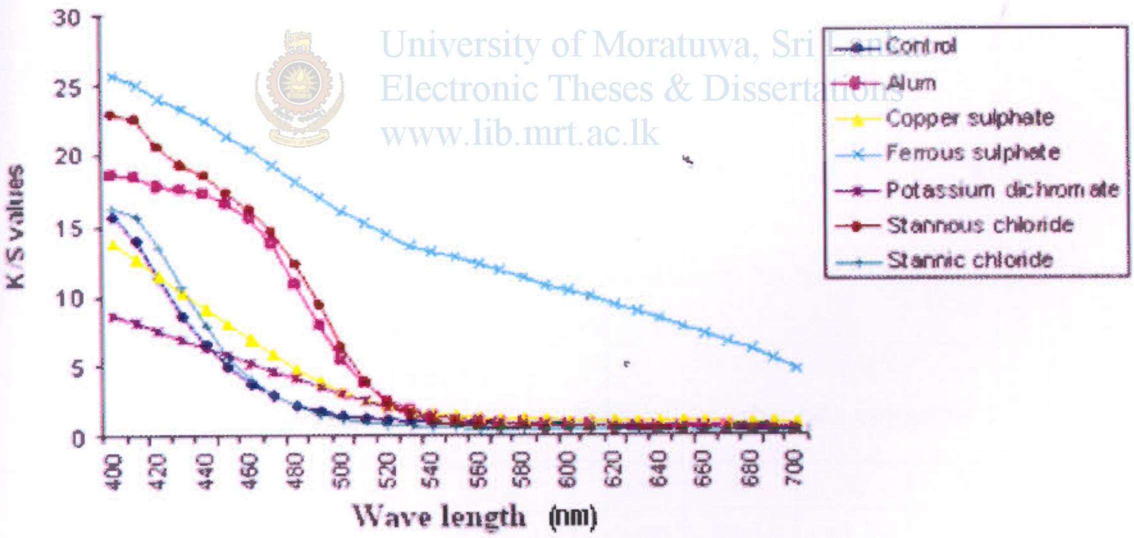


Figure 4.16 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *T. erecta*

Table 4.14 Characteristics for silk fabric dyed with methanolic extract of *T. erecta*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	57.35	-3.91	34.70	34.93	--	52.04
	Alum	64.64	7.38	55.13	55.62	24.45	126.07
	Copper sulphate	56.38	2.59	37.56	37.65	7.18	72.44
	Stannous chloride	69.06	10.62	63.29	64.18	34.14	132.91
	Ferrous sulphate	45.64	3.77	13.10	13.63	25.74	271.63
	Pot. dichromate	55.23	9.81	36.42	37.72	14.00	53.34
	Stannic chloride	64.99	-1.48	48.21	48.23	15.70	54.01

The best metal mordant for silk fabric is Ferrous sulphate having best K/S value of 271.63.

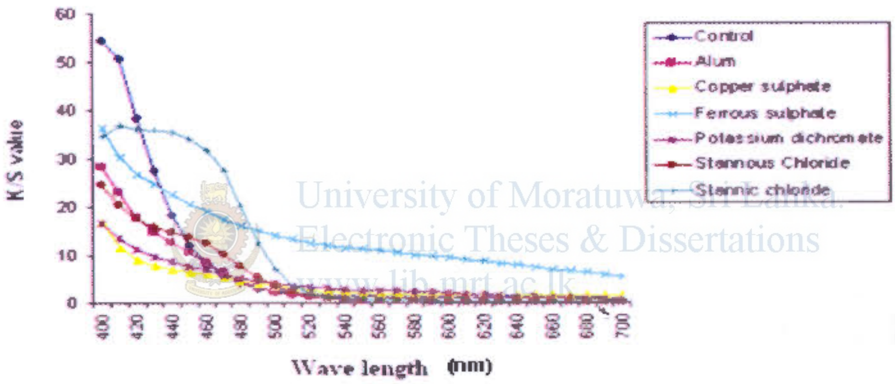


Figure 4.17 Change in K/S values with different mordants for wool yarn after dyeing with methanolic extract of *T.erecta*

Table 4.15 Characteristics for wool yarn dyed with methanolic extract of *T. erecta*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	52.14	-2.00	43.34	43.38	--	132.50
	Alum	58.03	10.80	60.83	61.78	22.47	84.88
	Copper sulphate	37.18	2.03	23.82	23.90	24.92	167.08
	Stannous chloride	59.31	12.80	65.44	66.68	27.55	102.12
	Ferrous sulphate	32.85	2.45	13.46	13.68	35.84	256.54
	Pot. dichromate	36.90	5.55	22.96	23.62	26.54	181.18
	Stannic chloride	67.98	13.50	80.56	81.69	43.33	234.06

The order of K/S values is: Fe > Sn (IV) > Sn (II) > Al > K > Cu in wool for *T. erecta.* The absorption of colour by wool yarn was enhanced when using Ferrous sulphate as the metal mordant. Fe (II) provides best chelation having optimum K/S value of 256.54.

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison.

(c) Fastness properties of dyed fabrics

Fastness properties of metal mordanted samples for the conventional dyeing conditions are shown in Table 4.16.

Table 4.16 Fastness properties of dyed cotton, silk fabrics & wool yarn under conventional dyeing of different metal mordanting with methanolic extract of *T.erecta.*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Without mordant)	2-3	2	2	2-3	2-3	2
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk (Control)	2	2	2-3	2-3	2	2
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (Control)	3-4	3-4	3	3	3	3-4
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness, Per – Perspiration fastness, Rub – Rubbing fastness.

The colourimetric data obtained from dyed fabrics and yarn which had been pre treated with tannic acid/metal ions mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pre treatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to controlled samples. It also increased the colour strength and flattened the shade of the dyeing. The two stage dyeing of cotton fabric with metal mordant by the natural dye *T.erecta* shows that by this process very good results of even dyeing are obtained. Most of the natural dye extract have poor affinity for cotton fibers, but their fastness is enhanced by metal mordants, which forms an insoluble complex.

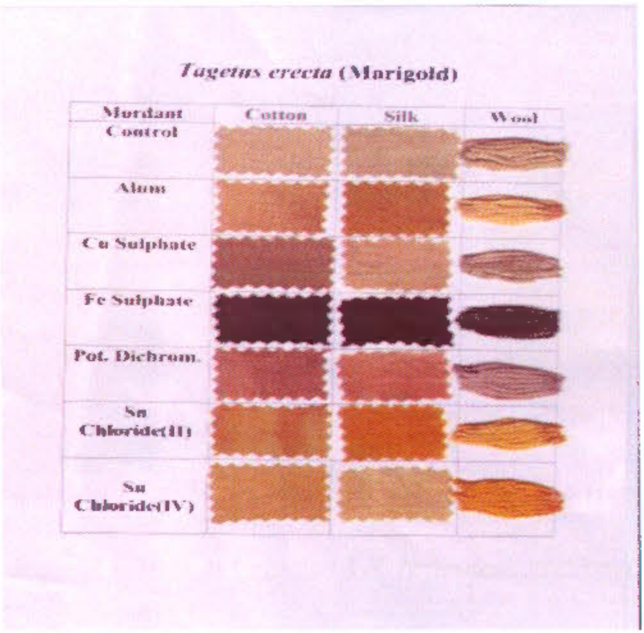


Figure 4.18 Fabric samples dyed with methanolic extract of *T. erecta* with different mordants

Above colourful samples show the possibility of producing fascinating colours from *T.erecta* flowers to textile substrates.

(d) Characterization of environmental impact

Trace elements levels in the dye, and Chemical Oxygen Demand (COD) of methanolic extract of *T. erecta* and fabric dyed with *T. erecta* extract in mg/l are tabulated in Table 4.17.

Table 4.17 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.12	ND	0.03	ND	ND	ND	0.4	0.14
Dyed fabric (mg/l)	0.02	0.12	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	200 -210								

ND – Not Detected up to 10mg/l.

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *T. erecta*. The effluent COD value is below that of the recommended value of CEA for discharge standards applicable to inland surface water.

4.12.3 Kothala Himbutu *(Salacia reticulata)*

a

b

Figure 4.19 a, b Leaves of *S. reticulata* and bark

Botanical name	: *Salacia reticulata*
Family	: Hipocrataceae
Common name	: Koranti
Key applications	: Antidiabetic
Sources	: Medicinal retailers
Part used	: Stem and root extracts

(a) Characterization

Visible spectrum of *S. reticulata*: The methanolic extract from *S. reticulata* shows peaks at 210 nm and 280 nm.

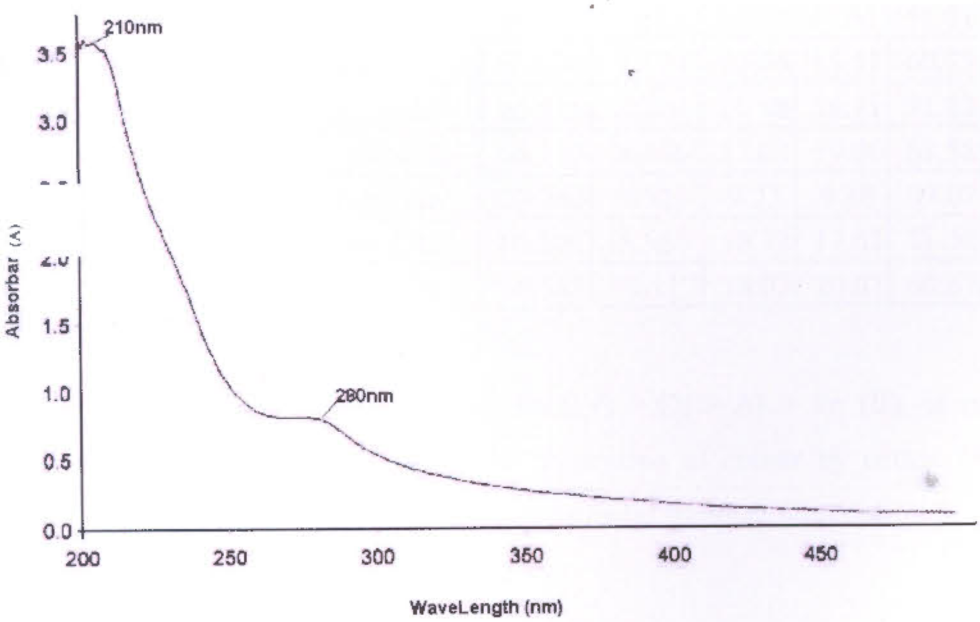


Figure 4.20 UV-Vis spectrum of methanolic extract of *S. reticulata*

(b) Colour measurements:

K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.21-4.23 and CIE lab values are shown in Tables 4.18 - 4.20.

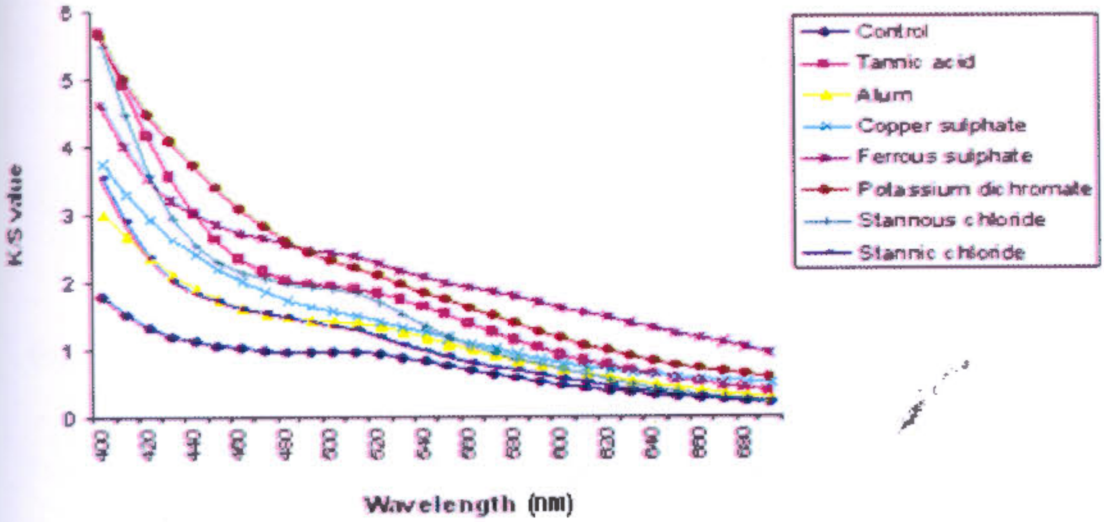


Figure 4.21 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *S. reticulata*

Table 4.18 Characteristics for cotton fabric dyed with methanolic extract of *S. reticulata*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	64.034	9.011	10.80	14.07	50.15	14.15
	Tannic acid +Dye	66.026	7.181	15.86	17.41	65.61	33.04
	Alum	65.005	7.579	13.54	15.51	60.73	22.12
	Copper sulphate	65.825	5.204	15.88	16.71	71.83	26.25
	Stannous chloride	66.117	8.446	17.02	19.00	63.58	20.61
	Ferrous sulphate	63.766	3.525	9.23	9.88	69.07	39.66
	Pot. Dichromate	66.101	5.580	16.72	17.63	71.52	39.77
	Stannic chloride	66.588	10.117	18.02	20.67	60.67	28.10

The order of K/S values is: K > Fe > Sn (IV) > Cu > Al > Sn (II) in cotton for methanolic extract of *S. reticulata,* the absorption of colour by cotton fabric was enhanced when using Potasium dichromate as metal mordant with optimum K/S value of 39.77 with corresponding CEI Lab values.

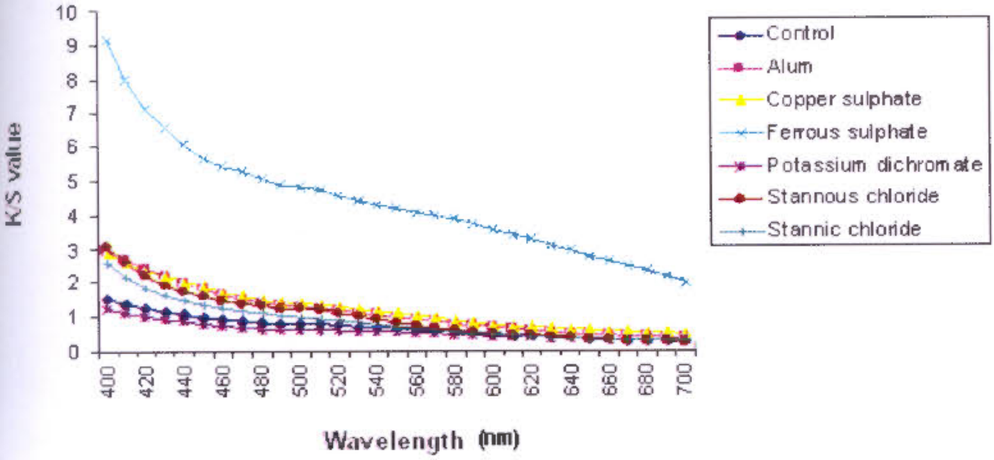


Figure 4.22 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *S. reticulata*

Table 4.19 Characteristics for silk fabric dyed with methanolic extract of *S. reticulata*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	65.600	5.132	11.12	12.25	65.21	12.86
	Alum	66.888	4.904	15.07	15.84	71.95	22.16
	Copper sulphate	66.315	4.63	13.40	14.18	70.89	23.23
	Stannous chloride	67.179	5.257	16.03	16.87	71.81	16.21
	Ferrous sulphate	64.832	2.172	7.97	8.26	74.74	81.93
	Pot. dichromate	65.307	2.250	9.65	9.91	76.84	16.87
	Stannic chloride	67.439	7.254	16.95	18.44	66.80	19.12

The order of K/S values is : Fe > Cu > Al > Sn (IV) > Sn (II) > K in silk for *S. reticulata* , the absorption of colour by silk fabric was enhanced when using ferrous sulphate as metal mordants with optimum K/S value of 81.93 and corresponding CEI Lab values.

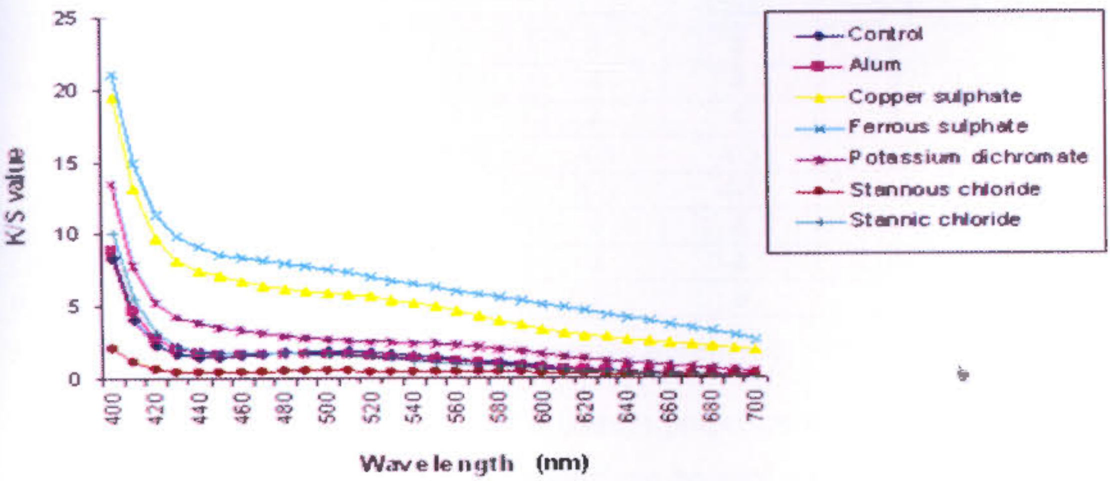


Figure 4.23 Change in K/S values with different mordants for wool yarns after dyeing with methanolic extract of *S. reticulata*

Table 4.20 Characteristics for wool yarn dyed with methanolic extract of *S. reticulata*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	54.971	13.864	7.87	15.94	29.58	25.58
	Alum	56.354	10.622	12.02	16.04	48.53	25.67
	Copper sulphate	55.999	6.616	11.72	13.46	60.53	97.67
	Stannous chloride	57.747	11.470	15.89	19.60	54.16	25.45
	Ferrous sulphate	55.334	3.836	9.38	10.13	67.73	125.28
	Pot. Dichromate	56.586	5.864	12.76	14.05	65.30	49.27
	Stannic chloride	53.730	6.209	2.82	6.81	24.41	28.98

The order of K/S values is as following: Fe > Cu > K > Al > Sn (II) > Sn (IV) in wool for methanolic extraction of *S. reticulata,* the absorption of colour by wool yarn was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 125.28.

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to cotton, wool and silk fabrics. Control samples without mordant were also prepared for comparison.

(c) Fastness properties of dyed fabrics

Fastness properties of dyed samples with methanolic extract of *S. reticulata* are shown in Table-4.21.

Table 4.21 Fastness properties of dyed cotton, silk fabrics and wool yarn metal mordanting with methanolic extract of *S.reticulata*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Silk (Alum)	4	4	4	4	4	4
Wool (Alum)	4	4	4	4	4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness, Per – Perspiration fastness, Rub – Rubbing fastness.

From the above results it can be shown a fastness properties are in the acceptable range. Therefore methanolic extract of *S. reticulata* can be used as dye for dyeing of textile substrates.

Figure 4.24 Fabric samples dyed with methanolic extract of *S. reticulata* with different mordants

A wide range of fascinating colours can be obtained from *S. reticulata* with different mordants to dye textile substrates (e.g. Dark brown colour can be obtained when using Ferrous sulphate as a metal mordant).

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of *S. reticulata* and fabric dyed with *S. reticulata* extract in mg/l are tabulated in Table 4.22.

Table 4.22 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	ND	ND	0.03	ND	ND	ND	0.23	0.13
Dyed fabric (mg/l)	0.02	ND	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	175 -260								

ND – Not detected up to 10 mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from a dyeing operation involving *S. reticulata*.

4.12.4 Weniwelgata (*Coscinium Fenestratum*)

a

b

Figure 4.25 a, b *C. fenestratum* plant and dried bark

(a) Characterization

Visible spectrum of *C. fenestratum*: The methanolic extrac from *C. fenestratum* shows peak at 432 nm.

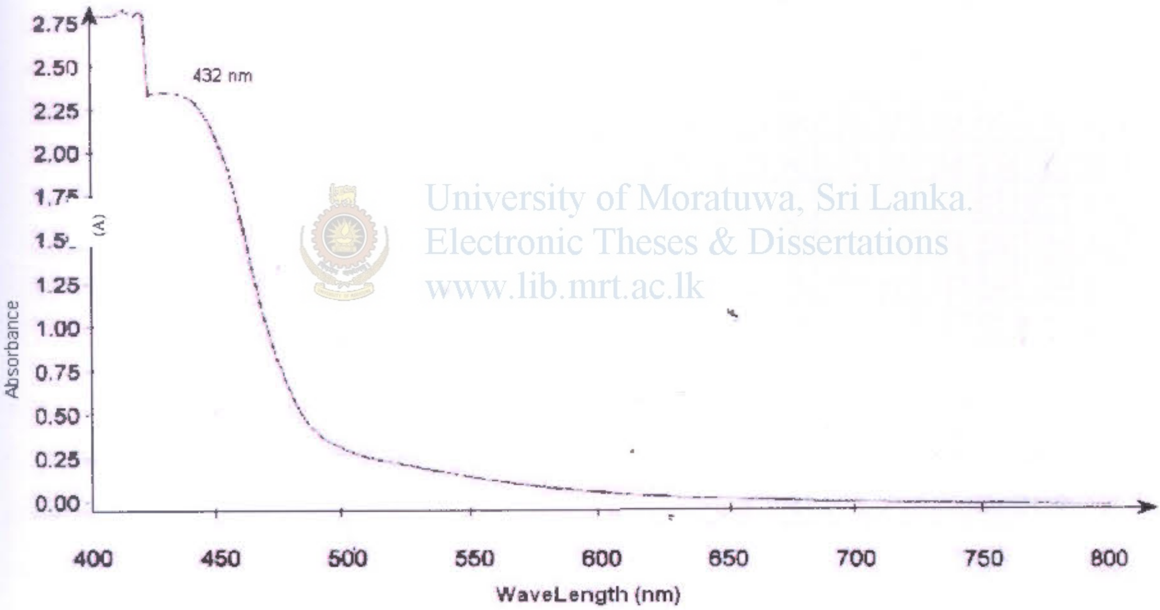


Figure 4.26 UV-Vis spectrum of methanolic extract of *C. fenestratum*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.27- 4.29 and CIE lab values are shown in Tables 4.23 - 4.25.

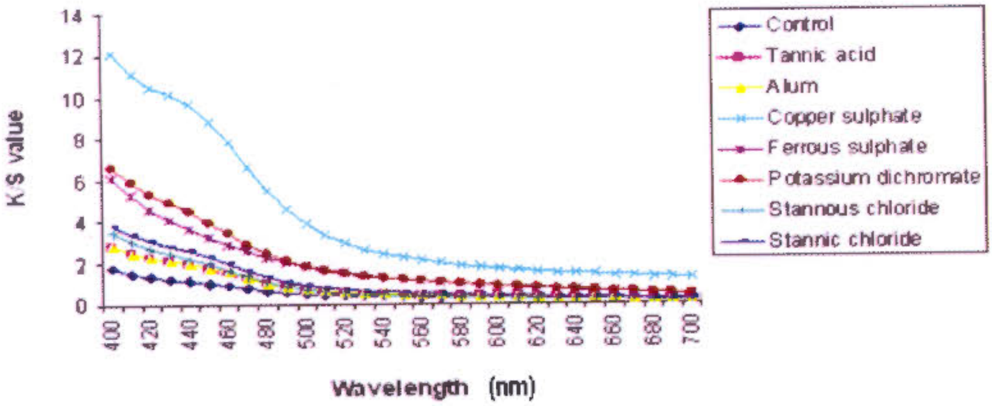
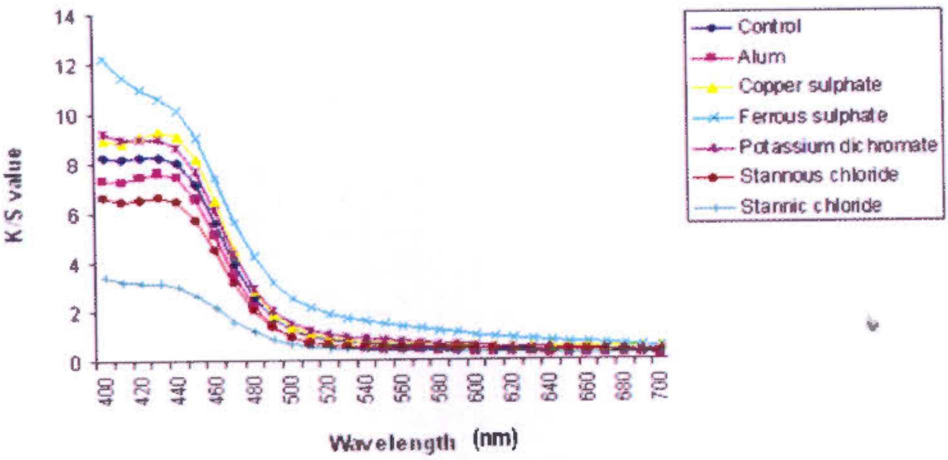


Figure 4.27 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of C. *fenestratum*

Table 4.23 Characteristics for cotton fabric dyed with methanolic extract of *C. fenestratum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	72.64	-0.004	21.97	21.97	90.04	10.19
	Tannic acid +Dye	74.29	-0.49	26.08	26.08	91.13	16.33
	Alum	74.28	-0.46	26.06	26.06	91.06	16.40
	Copper sulphate	74.01	0.55	25.94	25.94	88.73	81.86
	Stannous chloride	76.29	0.06	30.50	30.50	89.84	17.18
	Ferrous sulphate	72.14	2.71	20.86	21.04	82.56	34.92
	Pot. dichromate	73.39	0.90	23.91	23.92	87.80	39.23
	Stannic chloride	74.94	0.97	27.71	27.73	87.95	21.38

The order of change in K/S values is : Cu > K > Fe > Sn (IV) > Sn (II) > Al in cotton for *C. fenestratum* and also the absorption of colour by cotton fabric was enhanced when using Copper sulphate as the metal mordant and optimum K/S value given is 81.86 with corresponding CIE Lab values.



Figur 4.28 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *C. fenesttratum*

Table 4.24 Characteristics for silk fabric dyed with methanolic extract of *C. fenestratum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	62.99	-0.69	48.52	48.53	90.85	51.74
	Alum	65.19	-1.35	52.12	52.14	91.52	56.11
	Copper sulphate	62.89	-2.99	48.31	48.31	93.58	58.74
	Stannous chloride	62.51	-2.90	47.52	47.61	93.52	61.72
	Ferrous sulphate	57.41	2.97	38.09	39.09	85.60	73.68
	Pot. Dichromate	61.34	0.25	45.73	45.73	89.64	57.40
	Stannic chloride	57.26	-1.95	38.24	38.29	92.96	71.75

The order of K/S values is : Fe > Cu > K > Al > Sn (II) > Sn (IV) in silk for methanolic extract of *C. fenestratum*, the absorption of colour by silk fabric was enhanced when using Ferrous sulphate as the metal mordant with maximum K/S value of 73.68. Fe (II) provides best chelation.

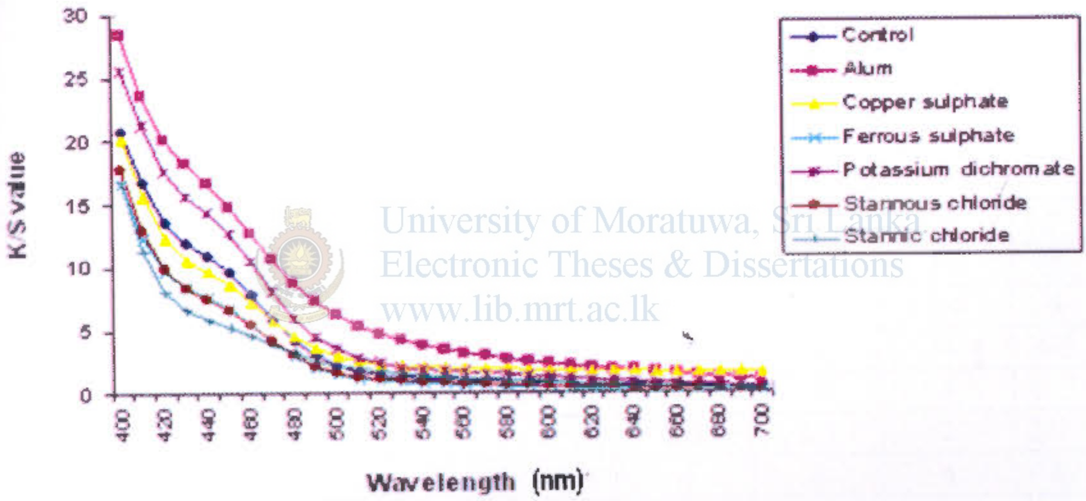


Figure 4.29 Change in K/S values with different mordants for wool yarn after dyeing with methanolic extract of *C. fenestratum*

Table 4.25 Characteristics for wool yarn dyed with methanolic extract of *C. fenestratum*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	56.45	3.87	46.52	46.68	85.21	76.98
	Alum	48.77	7.32	33.21	34.01	77.54	137.16
	Copper sulphate	47.95	-2.29	30.17	30.26	94.38	81.36
	Stannous chloride	55.79	4.83	45.34	45.60	83.88	84.94
	Ferrous sulphate	57.39	5.46	48.09	48.40	83.47	93.67
	Pot. Dichromate	54.07	4.87	42.61	42.88	83.43	104.88
	Stannic chloride	48.92	8.21	32.90	33.91	75.95	80.56

The order of K/S values is: Al > K > Cu > Sn (II) Fe > Sn (IV) in wool for methanolic extract of *C. fenestratum*, the optimum absorption of colour by wool yarn was enhanced when using alum as metal mordant having K/S value of 137.16.

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison. Therefore, in pre mordanting technique, the dyed fabrics were mordanted with stannic chloride, stannous chloride, ferrous sulphate, copper sulphate, potassium dichromate and alum.

(c) Fastness properties of dyed fabrics

Fastness properties of dyed fabrics with different metal mordants are shown in Table 4.26.

Table 4.26 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional dyeing with different metal modanting with methanolic extract of *C. fenestratum*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Control)	2-3	2	2	2-3	2-3	2
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk (Control)	2	2	2-3	2-3	2	2
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (Control)	3-4	3-4	3	3	3	3-4
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness, Per – Perspiration fastness, Rub – Rubbing fastness.

The colorimetric data obtained from dyed fabrics and yarn which had been pre-treated with tannic acid/metal mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pre-treatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to control samples. It also increased the colour strength and flattened the shade of the dyeings.

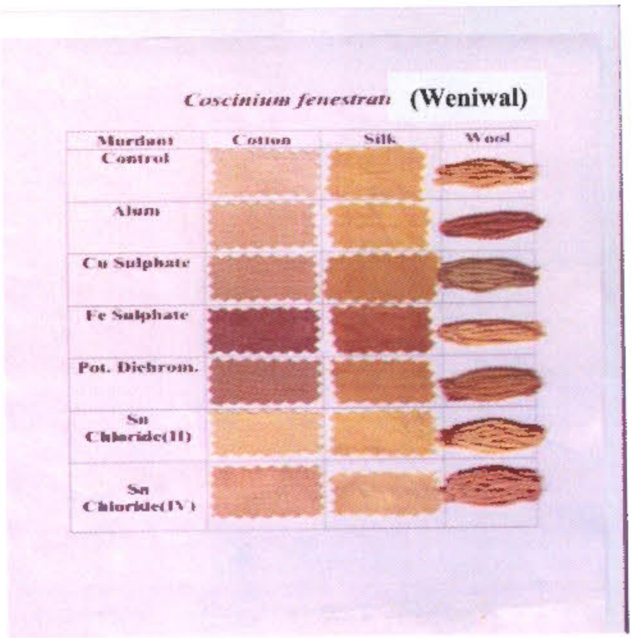


Figure 4.30 Fabric samples dyed with methanolic extract of methanolic extract of *C. fenestratum* with different mordants.

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of *C. fenestratum* and fabric dyed with *C. fenestratum* extract in mg/l are tabulated in Table 4.27.

Table 4.27 Characterization of e nvironmental impact

Trace elements (mg/l)	Cu	Zn	Cd	Co	Pb	As	Hg	Ni	Cr
In the dye (mg/l)	ND	0.12	ND	0.03	ND	ND	ND	0.06	0.04
Dyed fabric (mg/l)	ND	ND	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	145 -260								

ND – Not detected up to 10 mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *C. fenestratum* extract.

4.12.5 Big onion *(Allium cepa)*

Common name : Big Onion

Botanical Name : *Allium cepa*

Family : Alliaceae

Part used : Dried skin

Source : Domestic Kitchens, Markets and restaurants.

a b

Figure 4.31 a , b *A. cepa* bulbs and skin

(a) Characterization

Ultra – violet visible spectrum of Onion skin extract: The methanolic extract of *A. cepa* shows a peak at 280 nm.

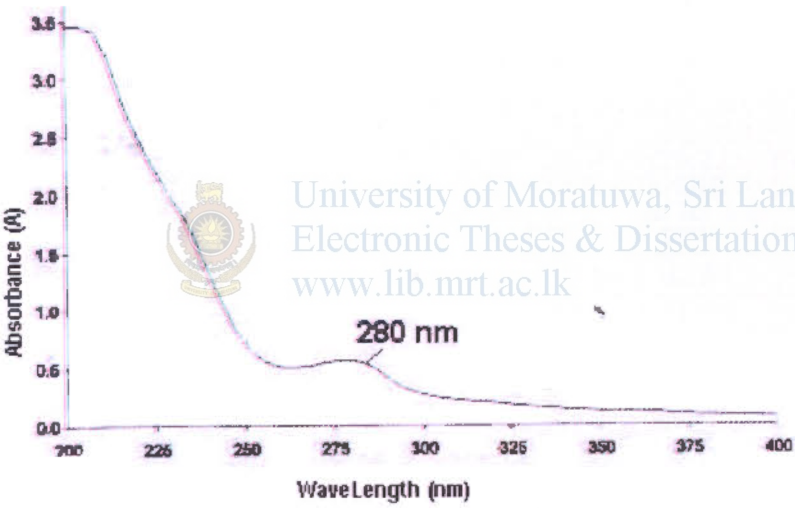


Figure 4.32 UV-Vis spectrum of methanolic extract of *A. cepa*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.33 – 4.35 and CIE lab values are shown in Tables 4.28 - 4.30.

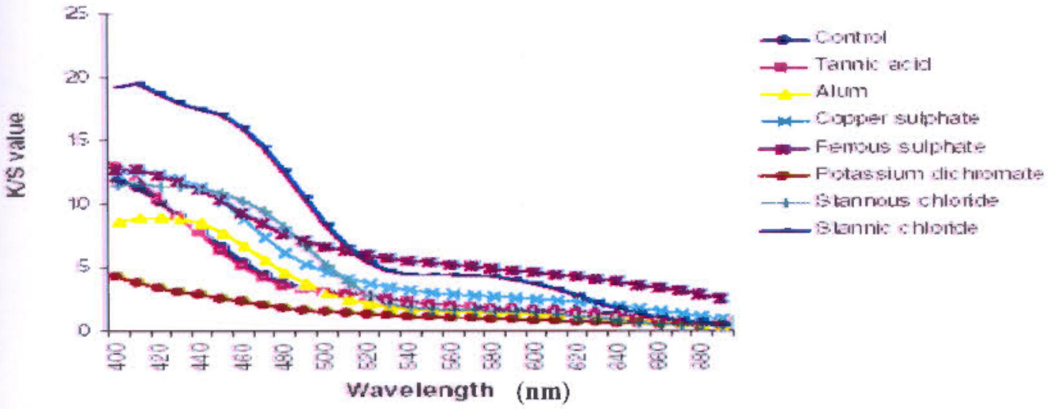


Figure 4.33 Change in K/S values with different mordants for cotton fabricafter dyeing with methanolic extract of *A. cepa*

Table 4.28 Characteristics for cotton fabrics dyed with methanolic extract of *A.cepa*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	47.08	3.58	25.20	25.45	81.86	69.10
	Control+ Tan	46.948	4.11	24.60	24.94	80.46	78.51
	Alum	50.461	4.16	34.10	34.36	83.00	70.20
	Copper sulphate	47.100	2.59	26.60	26.72	84.39	98.66
	Ferrous sulphate	42.243	1.90	14.72	14.84	82.59	124.25
	Pot. dichromate	44.315	5.20	20.25	20.91	75.55	78.78
	Stanous chloride	52.703	7.83	39.84	40.60	78.84	92.08
	Stannic chloride	48.406	6.73	30.52	31.25	77.52	158.97

The order of K/S values is Sn (IV) > Fe > Cu > Sn (II) > Al > K in cotton. For the absorption of colour by cotton fabric was enhanced when using Stannic chloride as metal mordant with optimum K/S value of 158.97 at corresponding CEI Lab values.

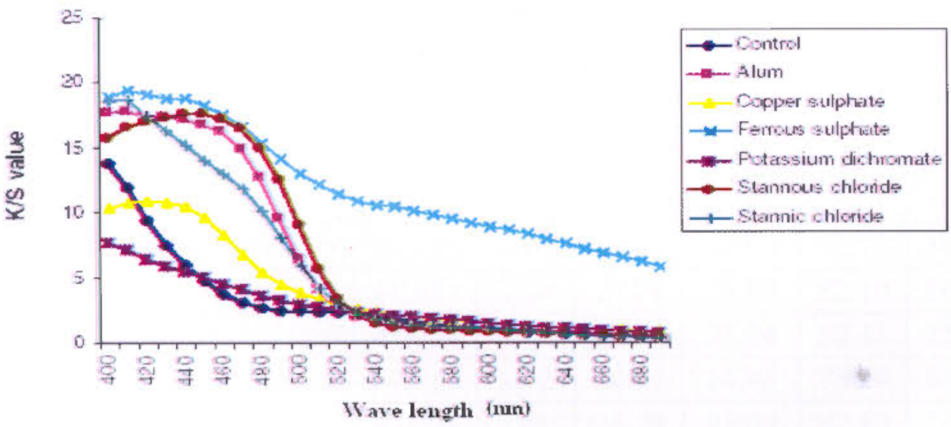


Figure 4.34 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of A. *cepa*

Table 4.29 Characteristics for silk fabrics dyed with methanolic extract of *A.cepa*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	51.93	10.2	26.37	28.28	68.76	53.18
	Tannic Acid +	63.24	4.11	24.60	24.94	80.46	68.51
	Alum	53.82	6.70	53.45	53.87	82.82	130.70
	Copper sulphate	45.33	5.78	33.54	34.03	80.19	83.35
	Ferrous sulphate	48.57	2.14	13.09	13.26	80.60	227.11
	Pot. dichromate	64.90	5.52	21.61	22.30	75.62	55.13
	Stanous chloride	61.29	11.3	57.05	58.16	78.75	136.11
	Stannic chloride	48.40	6.73	49.31	50.32	78.46	116.16

The order of K/S values is: Fe > Sn (II) > Al > Sn (IV) > Cu > K in silk for methanolic extract of *A.cepa,* the absorption of colour by silk fabric was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 227.11 with orresponding CIE Lab values.

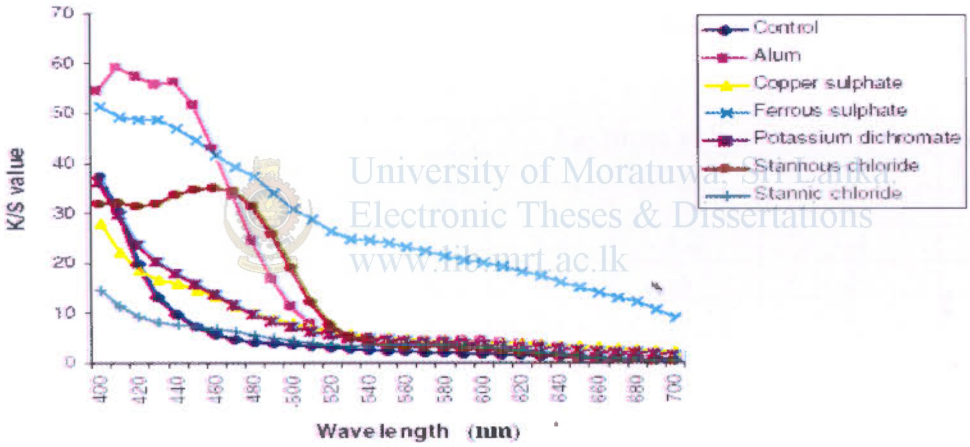


Figure 4.35 Change in K/S values with different mordants for wool yarns after dyeing with methanolic extract of *A. cepa*

Table 4.30 Characteristics for wool yarn dyed with methanolic extract of *A.cepa*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	45.523	5.18	26.58	27.08	78.93	91.14
	Control+Tan	55.056	-1.49	48.98	49.00	91.78	381.20
	Alum	41.634	3.08	23.04	23.24	82.10	161.85
	Copper sulphate	41.634	3.08	23.04	23.24	82.33	152.03
	Ferrous sulphate	37.916	2.92	14.16	14.46	78.29	539.51
	Pot. dichromate	43.699	3.69	26.79	27.04	82.10	161.85
	Stanous chloride	53.842	13.1	49.57	51.29	75.07	277.85
	Stannic chloride	39.132	3.89	16.67	17.12	76.80	96.77

The order of K/S values is: Fe > Al > Sn (II) > K > Cu > Sn (IV) in wool for methanolic extract of *A.cepa*, the absorption of colour by wool yarn was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 539.51 with orresponding CIE Lab values.

Effect of mordanting conditions

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison.

(c) Fastness properties of dyed fabrics

Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional dyeing of metal modanting with methanolic extract of *A.cepa* are shown in Table 4.31.

Table 4.31 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional conditions of metal modanting with methanolic extract of *A.cepa*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidi}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Control)	3-4	3	3	3	3	3-4
Cotton (Alum)	4	4	3-4	3-4	3-4	4
Cotton (FeSO4)	4-5	4	4	4	4	4-5
Cotton (CuSO4)	4	4	4	4	4	4
Wool (Control)	3	3	3	3	3	3
Wool (Alum)	4	4	4	4	4	4
Wool (FeSO4)	5	4-5	4-5	4-5	4-5	5
Wool (CuSO4)	4-5	4	4	4	4	4-5
Silk (Control)	3	3	3	3	3	3
Silk (Alum)	4	4	4	4	4	4
Silk (FeSO4)	5	4-5	4-5	4-5	4-5	4-5
Silk (CuSO4)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness , Per – Perspiration fastness, Rub – Rubbing fastness

The colourimetric data obtained from dyed fabrics and yarn which had been pre-treated with tannic acid/metal mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pre-treatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to controlled samples. It also increased the colour strength and flattened the shade of the dyeings.

controlled samples. It also increased the colour strength and flattened the shade of the dyeings.

Figure 4.36 Fabric samples dyed with methanolic extract of *A. cepa* with different mordants.

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of *A. cepa* and fabric dyed with *A. cepa* extract in mg/l are tabulated in Table 4.32.

Table 4.32 Characterisation of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.12	ND	0.03	ND	ND	ND	0.43	0.14
Dyed fabric (mg/l)	0.02	0.12	ND	ND	ND	ND	ND	ND	ND
COD of the extract	154 -300								

ND – Not detected up to 10 mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *A.cepa*.

4.12.6 Mangustene *(Garcinia Mangostana)*

a b

Figure 4.37 a,b Fresh fruit of *G. Mangostana* and dried pericarp

Kingdom	Plantae
Division	Magnoliophyta
Family	Clusiaceae
Genus	*Garcinia*
Species	*G. mangostana*
Source	Domestic and sellers

(a) Characterization

Spectral analysis of the dye extract: Methanolic extract was also prepared to record UV-Visible spectrum as shown in Figure 4.38. Peaks are shown at 210 nm and 280 nm.

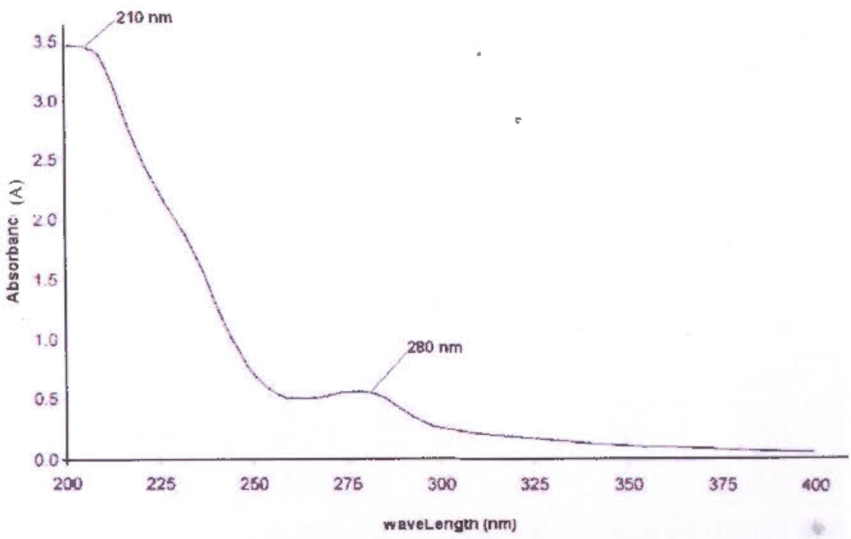


Figure 4.38 UV-Vis spectrum of methanolic extract of *G.mangostana*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.39 – 4.41 and CIE lab values are shown in Tables 4.33 - 4.35.

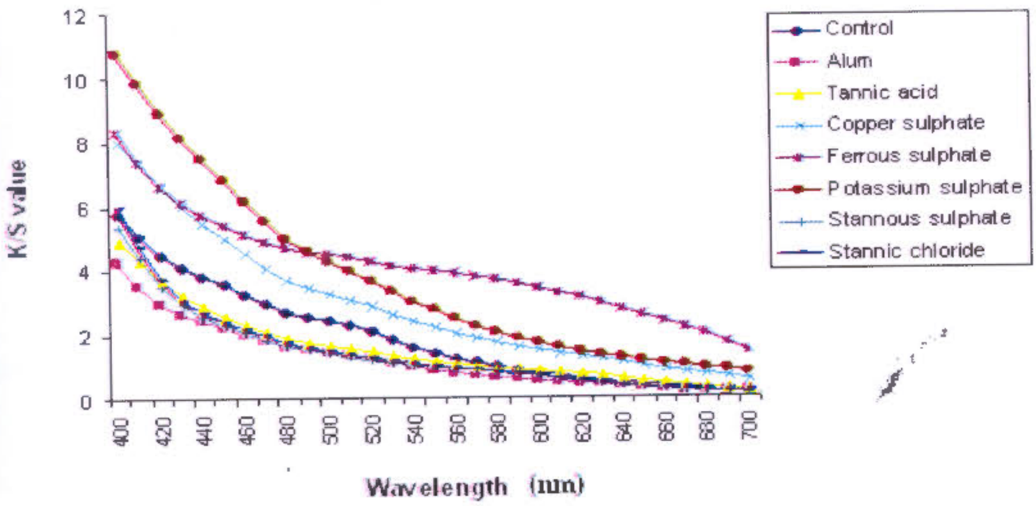


Figure 4.39 Change in K/S values for cotton samples after dyeing with methanolic extract of *G. mangostana*

The best values are obtained with ferrous sulphate (K/S value of 77.70) and the order of reactivity is Fe> Cr > Cu > Al >Sn (IV) > Sn (II) for cotton samples as shown in Figure 4.38.

Table 4.33 Characteristics for cotton fabric dyed with methanolic extract of *G.mangostana*

Pre-mordanting	L*	a*	b*	C	H	K/S
Control	54.817	13.25	24.94	28.24	--	37.61
Alum	53.338	5.51	20.30	21.04	9.14	39.65
Ferrous sulphate	49.259	2.60	8.40	8.79	20.43	77.70
Stannous chloride	54.287	7.17	22.47	23.52	6.57	46.05
Copper sulphate	53.399	7.18	20.98	21.17	7.38	55.98
Pot. dichromate	54.347	8.62	23.52	25.05	4.85	73.37
Stannic chloride	54.567	8.10	23.21	24.59	5.43	47.19

The best values are obtained with ferrous sulphate (K/S value of 93.32) and the order of reactivity is Fe> Cr > Cu > Al >Sn (IV) > Sn (II) for silk samples as shown in Figure 4.39.

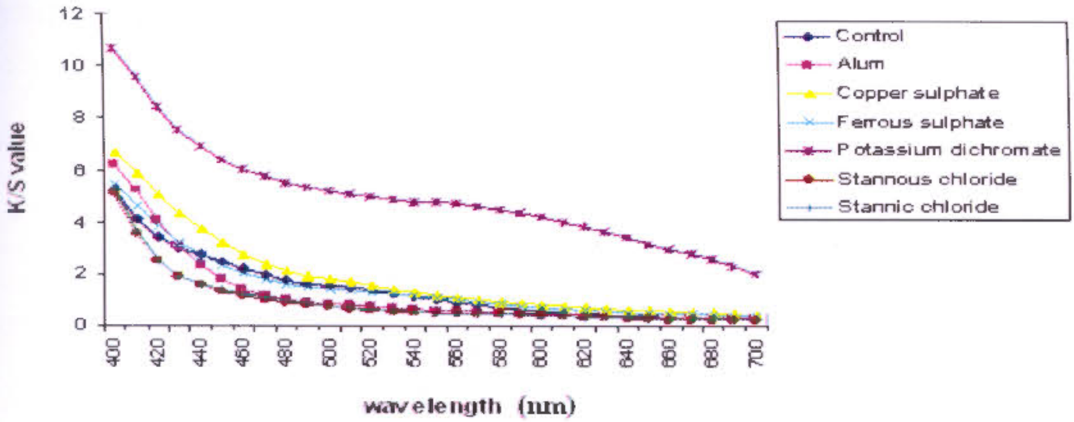


Figure 4.40 Change in K/S values for silk samples after dyeing with methanolic extract of *G. mangostana*.

The best values are obtained with ferrous sulphate (K/S value of 129.57) and the order of reactivity is Fe> Cu > Cr >> Al >Sn (IV) > Sn (II) for wool samples as shown in Figure 4.39.

Table 4.34 Characteristics for silk fabric dyed with methanolic extract of *G.mangostana*

Pre-mordanting	L*	a*	b*	C	H	K/S
Control	59.974	10.14	24.03	26.09	---	26.31
Alum	62.732	1.02	26.49	26.51	9.84	30.34
Ferrous sulphate	54.775	1.62	8.48	8.64	18.47	93.32
Stannous chloride	60.860	1.76	23.92	23.96	8.43	35.48
Copper sulphate	59.211	6.01	21.04	21.88	5.16	27.19
Pot. dichromate	60.659	5.16	24.87	25.40	5.09	34.52
Stannic chloride	61.155	2.37	25.11	25.22	7.93	45.79

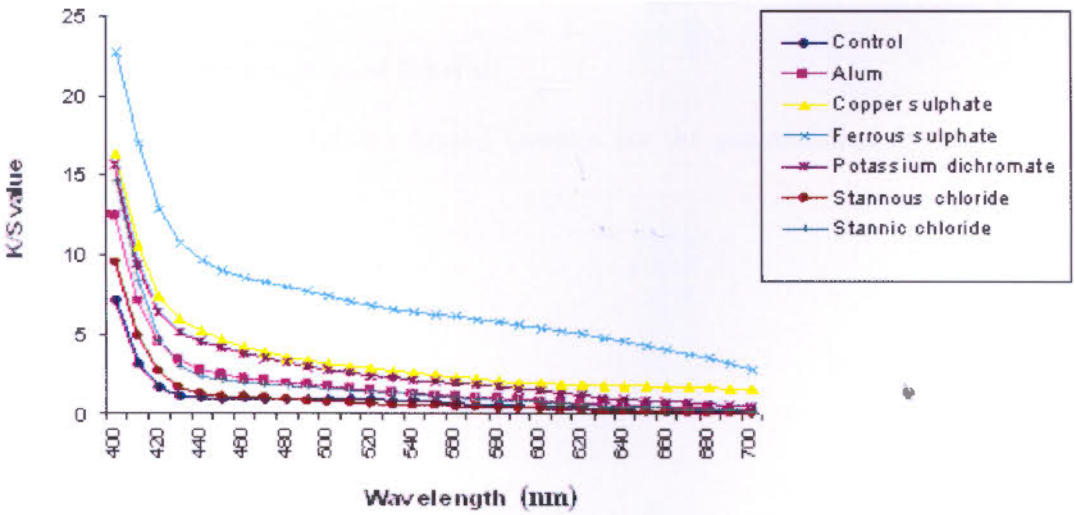


Figure 4.41 Change in K/S values with different mordants for wool yarns after dyeing with methanolic extract of *G.mangostana*

The best values are obtained with ferrous sulphate and the order of reactivity is Fe> Cu > Cr >> Al >Sn (IV) > Sn (II) for wool samples as shown in Figure 4.43.

Table 4.35 Characteristics for wool yarns dyed with methanolic extract of *G. mangostana*

Pre-mordanting	L*	a*	b*	C	H	K/S
Control	64.914	10.28	11.87	15.71	--	14.28
Alum	67.309	5.20	18.63	19.35	8.79	31.58
Ferrous sulphate	64.021	1.55	9.64	9.77	9.05	129.57
Stannous chloride	68.967	5.73	21.35	22.11	11.27	15.25
Copper sulphate	66.03	1.38	15.45	15.51	9.65	59.47
Potassium dichromate	66.615	4.75	17.57	18.20	8.12	50.51
Stannic chloride	67.543	6.59	18.31	19.46	7.87	28.65

Different mordants are used in 1-2 % keeping in mind the toxicity factor of some mordants. Varied hues of colour can be obtained from pre mordanting the cotton, silk and wool yarn with $FeSO_4$, $SnCl_2$, $CuSO_4$, $SnCl_4$, $K_2Cr_2O_7$ and alum were dyed by methanolic extract of pericarp of *G.mangostana* as shown in the Figure 4.48, the different mordants not only cause difference in hue colour and significant changes in K/S values but also L* values and brightness index values. The cotton samples were pre-treated with tannic acid before mordanting. The colour strengths, K/S has been found to be very good in dyed samples.

(c) Fastness properties of dyed fabrics:

Fastness properties of metal mordanted samples for the conventional dyeing conditions are shown in Table 4.36.

Table 4.36 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional dyeing with different metal modanting with methanolic extract of *G. mangostana*

Fabric (Mordant)	**Fastness values**					
	WF	**Per_{acidic}**	**Per_{basic}**	**Rub_{dry}**	**Rub_{wet}**	**LF**
Cotton (Control)	2-3	3	3	2-3	2-3	2
Cotton (Alum)	4	4	3	3-4	3-4	3
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	3
Silk (Control)	2	2	2-3	2-3	2	2
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (Control)	3-4	3-4	3	3	3	3-4
Wool (Alum)	4	4	4	4	4	3
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4
Wool ($CuSO_4$)	4-5	4	4	4	4	4

WF = wash fastness, LF = light fastness , Per – Perspiration fastness, Rub – Rubbing fastness.

The fastness properties have also been evaluated and were found to be well above the acceptable limits.

Figure 4.42 Fabric samples dyed with methanolic extract of *G.mangostana* with different mordants

Fabric samples dyed with methanolic extract of *G. mangostana* with different mordants show attractive shades for dyeing textile substrates.

(d) Characterization of environmental impacts

Trace elements in the dye, and Chemical Oxygen Demand (COD) of methanolic extract of G. *mangostana* and fabric dyed with *G. mangostana* extract in mg/l are tabulated in Table 4.37.

Table 4.37 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.01	ND	0.03	ND	0.04	ND	ND	ND
Dyed fabric (mg/l)	0.02	0.10	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	180 -250								

ND – Not detected up to 10mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *G.mangostana.*

4.12.7 Jak fruit (*Artocarpus heterophyllus*)

a

b

Figure 4.43 a, b *A. heterophyllus* plant and saw dust

Class : *Magnoliopsida*
Order : Rosales
Family : Moraceae
Genus : *Artocarpus*
Source : Saw dust from saw mills at Moratuwa area

(a) Characterization

Visible spectrum of *A. heterophyllus* bark extract: The crude methanolic extract from *A. heterophyllus bark* showed peaks at 260 nm and 350 nm. Most of the yellow dyes show similar visible spectrum.

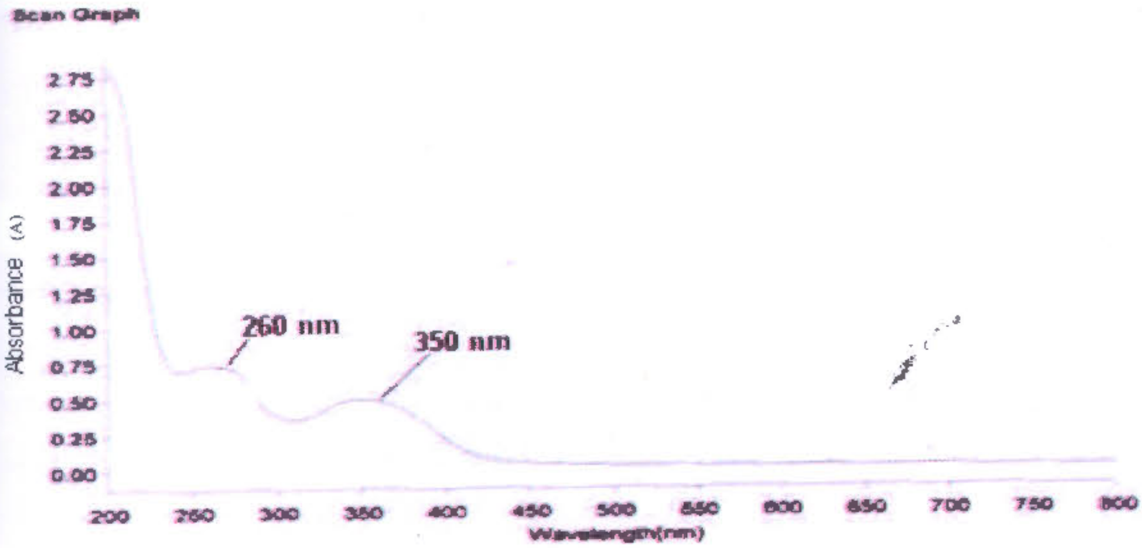


Figure 4.44 UV-Vis spectrum of methanolic extract of *A. heterophyllus* bark

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.45- 4.47 and CIE lab values are shown in Tables 4.38- 4.40.

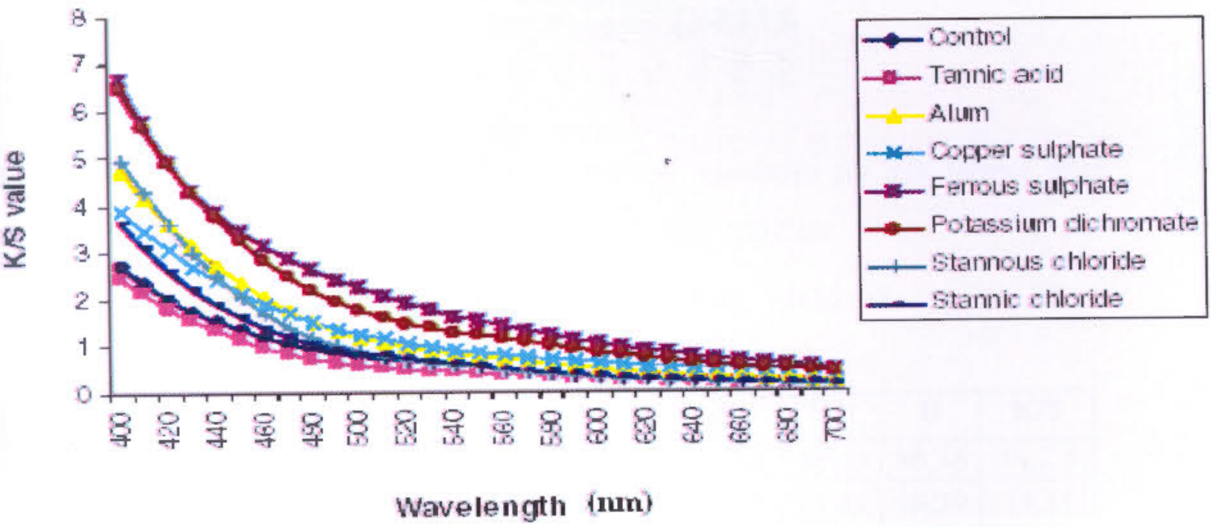


Figure 4.45 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *A. heterophyllus*

Table 4.38 Characteristics for cotton fabric dyed with methanolic extract of *A.heterophyllus*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	67.68	5.08	21.84	22.42	76.86	14.65
	Control+TanAcid	68.23	2.36	22.53	22.65	83.97	17.41
	Alum	69.31	4.13	25.67	26.00	80.82	24.18
	Copper sulphate	67.33	2.65	20.93	21.10	82.73	23.49
	Ferrous sulphate	66.77	4.79	19.74	20.31	76.32	39.52
	Pot. Dichromate	68.07	3.04	22.61	22.81	82.31	35.22
	Stanous chloride	71.56	4.04	30.01	30.28	82.28	20.33
	Stannic chloride	69.01	5.44	24.78	25.37	77.57	17.04

The order of K/S values is as following: Fe > Cr > Al > Cu > Sn (II) > Sn (IV) in cotton for methanolic extract of *A. heterophyllus*, the absorption of colour by cotton fabric was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 39.52 with corresponding CIE Lab values.

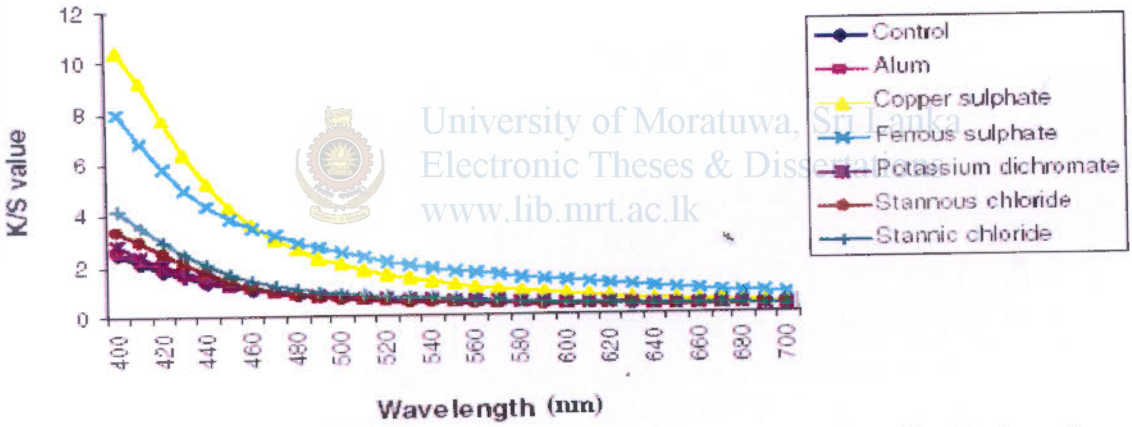


Figure 4.46 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *A. heterophyllus*

Table 4.39 Characteristics for silk fabric dyed with methanolic extract of *A. heterophyllus*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	66.18	0.93	16.66	16.69	86.76	14.2 5
	Alum	67.30	0.56	19.48	19.49	88.29	14.35
	Copper sulphate	70.77	2.86	27.75	27.90	84.08	44.85
	Ferrous sulphate	66.65	2.50	18.41	18.58	82.21	45.62
	Pot. Dichromate	66.69	0.61	17.84	17.85	88.00	15.58
	Stanous chloride	68.99	-1.01	22.96	22.98	92.57	15.84
	Stannic chloride	68.42	1.49	22.00	22.05	86.07	19.00

The order of K/S values is as following: Fe > Cu > Sn (IV) > Sn (II) > Cr > Al in silk for *A. heterophyllus*, the absorption of colour by silk fabric was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 45.62 with corresponding CIE Lab values.

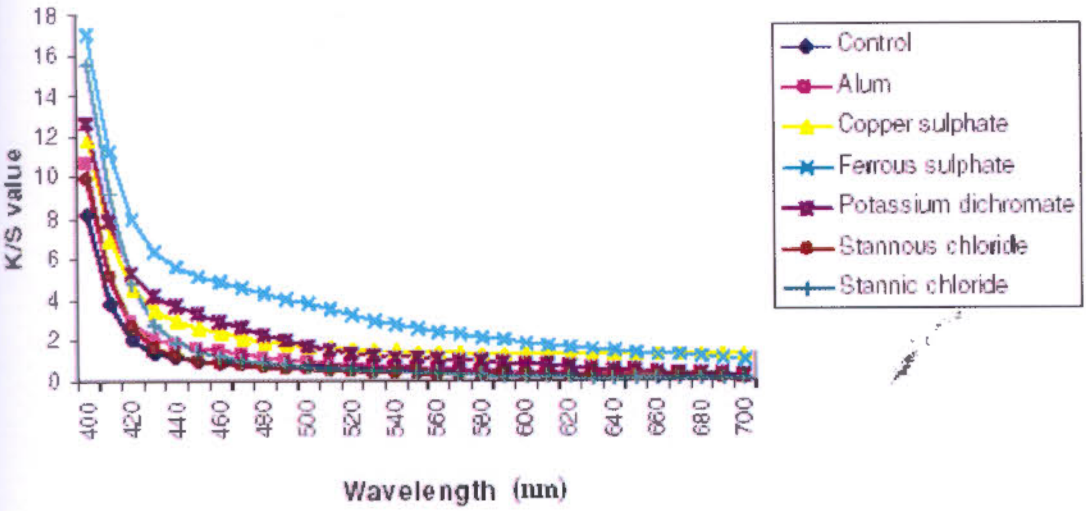


Figure 4.47 Change in K/S values with different mordants for wool yarn after dyeing with methanolic extract of *A. heterophyllus*

Table 4.40 Characteristics for wool yarn dyed with methanolic extract of *A.heterophyllus*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-Mordanting	Control	68.66	5.05	17.58	18.29	73.93	13.09
	Alum	70.03	4.22	22.16	22.56	79.17	19.13
	CopperSulphate	67.34	-2.25	14.51	14.68	98.86	34.83
	Ferrous Sulphate	67.51	4.23	17.05	17.56	76.03	64.15
	Pot. Dichromate	70.46	2.93	24.26	24.44	83.07	35.71
	StanousChloride	72.40	2.18	24.87	24.97	84.94	13.87
	Stannic Chloride	75.59	4.58	30.11	30.45	81.31	18.56

The order of K/S values is as following: Fe > Cr > Cu > Al > Sn (IV) > Sn (II) in wool for methanolic extract of *A. heterophyllus*, the absorption of colour by wool yarn was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 64.15 with corresponding CIE Lab values. Fe (II) provides hest chelation in all the three cases.

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison. Therefore, in pre mordanting technique,

(c) Fastness properties of dyed fabrics

The fastness properties of fabrics dyed with and without metal salts are shown in Table 4.41.

Table 4.41 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional conditions of metal modanting with methanolic extract of *A. heterophyllus*

Fabric (Mordant)	**Fastness values**					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Ruh_{wet}	LF
Cotton (control)	3-4	3	3	3	3	3-4
Cotton (Alum)	4	4	3-4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk (control)	3	3	3	3	3	3
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (control)	3	3	3	3	3	3
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4 -5
Wool ($CuSO_4$)	4-5	4	4	4	4	4 -5

WF = wash fastness, LF = light fastness , Per – Perspiration fastness, Rub – Rubbing fastness.

The colorimetric data obtained from dyed fabrics and yarn which had been pre treated with tannic acid/metal mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pre treatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to control samples. It also increased the colour strength and flattened the shade of the dyeings.

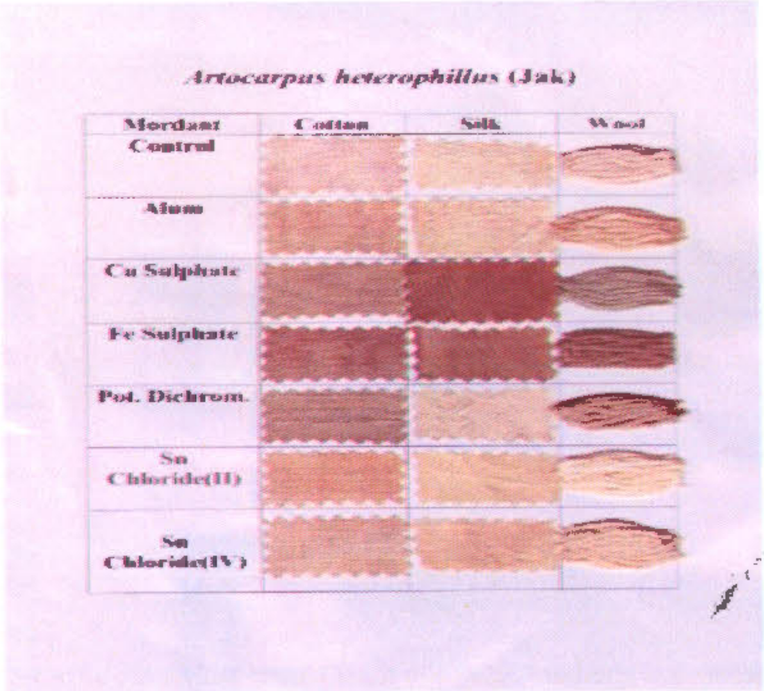


Figure 4.48 Fabric samples dyed with methanolic extract of *A. heterophyllus* with different mordants.

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of *A. heteropyllus* and fabric dyed with *A. heterophyllus* extract in mg/l are tabulated in Table 4.42.

Table 4.42 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.04	ND	0.03	ND	0.04	ND	0.03	0.04
Dyed fabric (mg/l)	0.02	0.03	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	149 -200								

ND – Not detected up to 10mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *A. heterophyllus.*

4.12.8 Tea *(Camelia sinensis)*

a b

Figure 4.49 a, b Fresh tea leaves and dried leaves

Botanical name	*Camellia sinensis*
Family	Theaceae
Source	Waste tea leaves from domestic kitchens and restaurants

(a) Preparation and optimization of aqueous extract of *C. sinensis*

The dried used *C. sinensis* leaves were found to give colour in hot water very easily by aqueous extraction. Increasing the quantity of leaves from 2.0 g to 20.0 g per 100 ml water boiled for 1hour is accompanied with increase in colour strength and depth in colour hue.

(b) Characterization

Methanolic extract was also prepared to record UV-Visible spectrum as shown in Figures 4.50. This spectrum indicates that *C.sinensis* extract shows peaks at 280 nm and 355 nm.

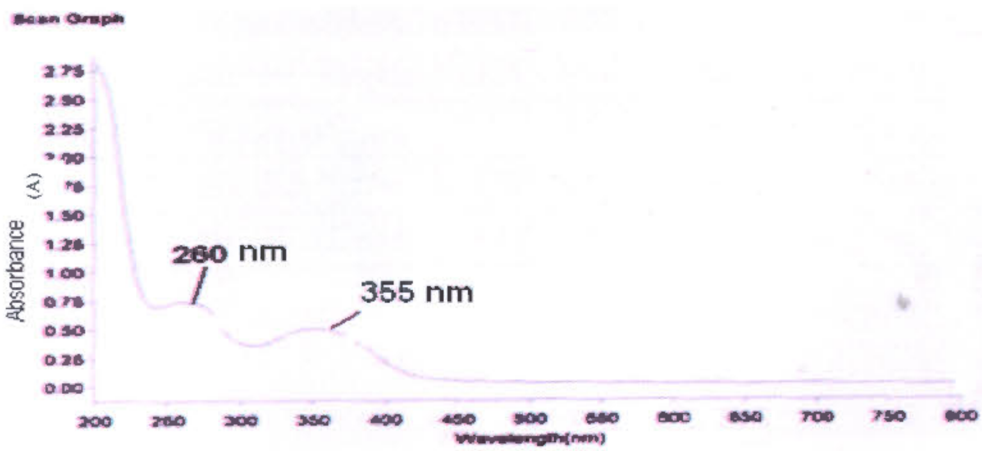


Figure 4.50 UV-Vis spectrum of methanolic extract of *C. sinensis*

(c) Colour measurements : K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.51- 4.53 and CIE lab values are shown in Tables 4.43-4.45.

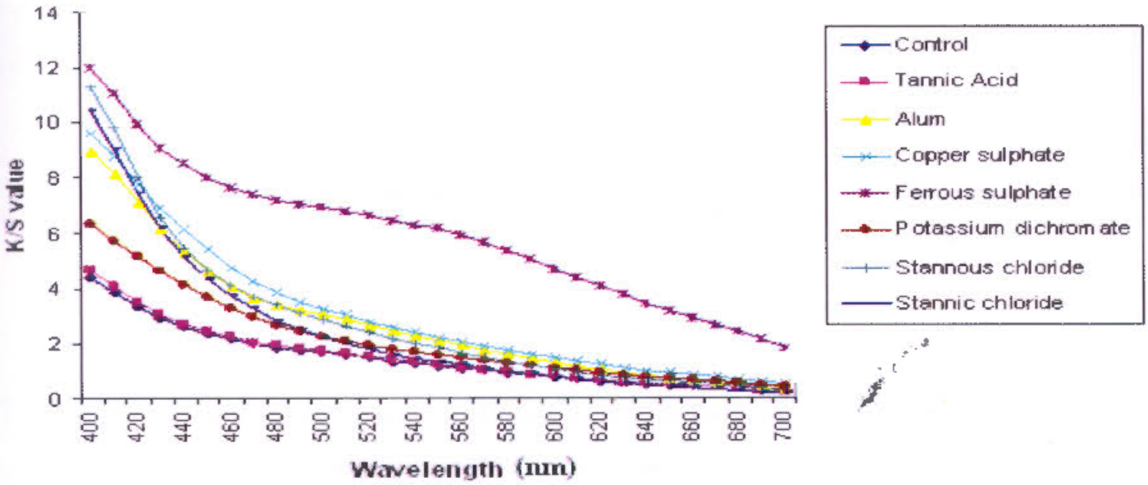


Figure 4.51 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *C. sinensis*

The best values are obtained with ferrous sulphate (K/S value of 34.92) and the order of reactivity is Fe> K > Cu > Al > Sn (IV) > Sn (II) for cotton samples as shown in Figure 4.51. The cotton samples were pre treated with tannic acid before mordanting.

Table 4.43 Characteristics for cotton fabric dyed with methanolic extract of *C.sinensis*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	72.64	-0.004	21.97	21.97	90.04	10.19
	Tannic acid +Dye	74.29	-0.49	26.08	26.08	91.13	16.33
	Alum	74.28	-0.46	26.06	26.06	91.06	16.40
	Copper sulphate	74.01	0.55	25.94	25.94	88.73	81.86
	Stannous chloride	76.29	0.06	30.50	30.50	89.84	17.18
	Ferrous sulphate	72.14	2.71	20.86	21.04	82.56	34.92
	Pot. dichromate	73.39	0.90	23.91	23.92	87.80	39.23
	Stannic chloride	74.94	0.97	27.71	27.73	87.95	21.38

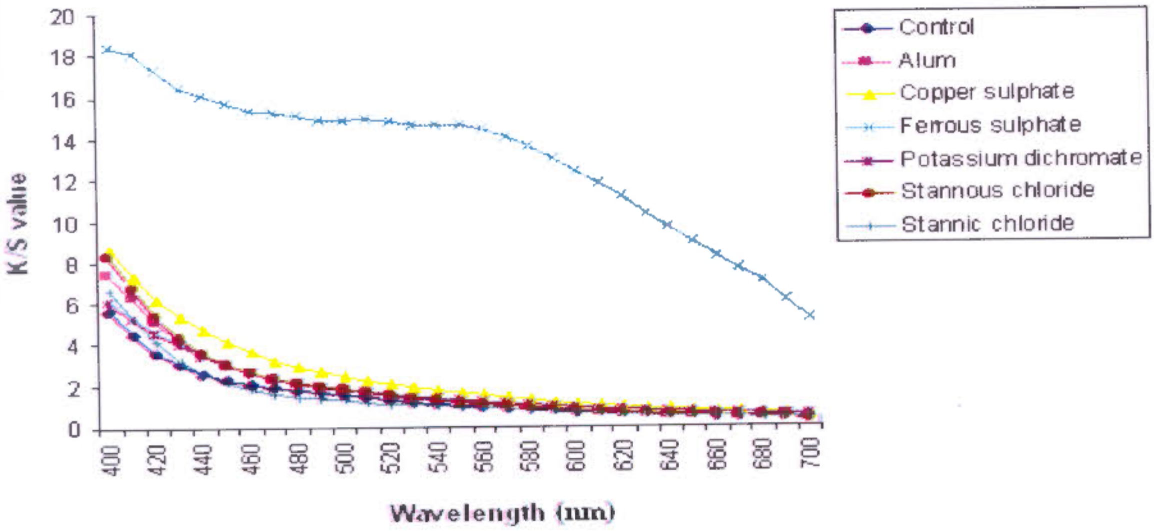


Figure 4.52 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *C. sinensis*

The best values are obtained with ferrous sulphate (K/S value of 73.68) and the order of reactivity is Fe> Cu > Cr > Al > Sn (IV) > Sn (II) for silk samples as shown in Figure 4.52.

Table 4.44 Characteristics for silk fabric dyed with methanolic extract of *C.sinensis*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	62.99	-0.69	48.52	48.53	90.85	51.74
	Alum	65.19	-1.35	52.12	52.14	91.52	46.11
	Copper sulphate	62.89	-2.99	48.31	48.31	93.58	58.74
	Stannous chloride	62.51	-2.90	47.52	47.61	93.52	41.72
	Ferrous sulphate	57.41	2.97	38.09	39.09	85.60	73.68
	Pot. dichromate	61.34	0.25	45.73	45.73	89.64	57.40
	Stannic chloride	57.26	-1.95	38.24	38.29	92.96	21.75

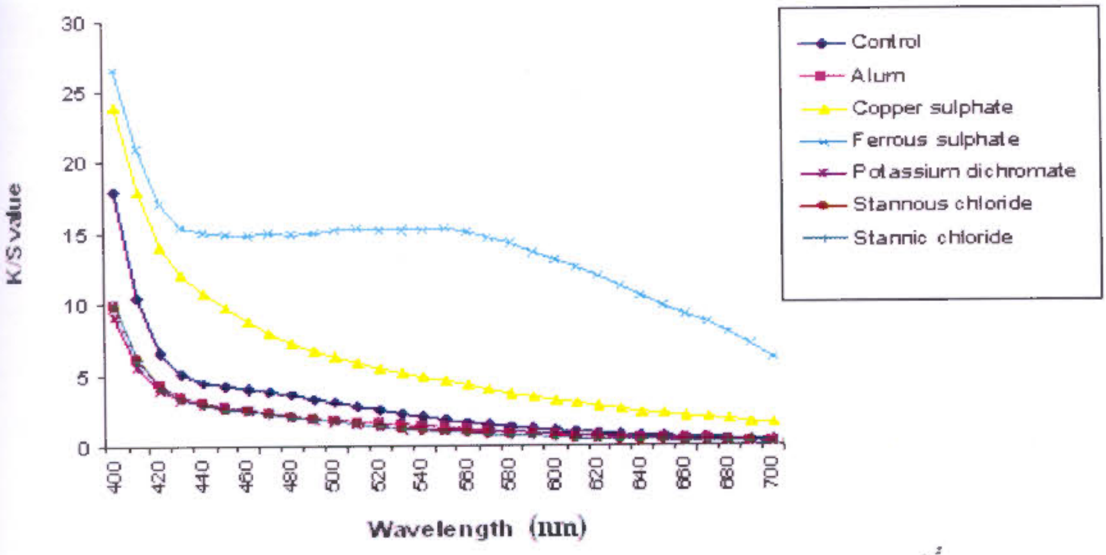


Figure 4.53 Change in K/S values with different mordants for wool fabrics after dyeing with methanolic extract of *C. sinensis*

The best values are obtained with ferrous sulphate (K/S value of 58.67) and the order of reactivity is Fe> Cu > Cr > Al > Sn (IV) > Sn (II) for wool yarns as shown in Figure 4.53.

Table 4.45 Characteristics for wool yarn dyed with methanolic extract of *C.sinensis*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	56.45	3.87	46.52	46.68	85.21	76.98
	Alum	48.77	7.32	33.21	34.01	77.54	37.16
	Copper sulphate	47.95	-2.29	30.17	30.26	94.38	81.36
	Stannous chloride	55.79	4.83	45.34	45.60	83.88	54.94
	Ferrous sulphate	57.39	5.46	48.09	48.40	83.47	58.67
	Pot. Dichromate	54.07	4.87	42.61	42.88	83.43	104.88
	Stannic chloride	48.92	8.21	32.90	33.91	75.95	50.56

Optimization of mordants with K/S and colour hue changes

Different mordants are used in 1-2 % keeping in mind the toxicity factor of some mordants and in line with the objectives of this study. Varied hues of colour can be obtained from pre mordanting the cotton, silk and wool yarn with $FeSO_4$, $SnCl_2$, $CuSO_4$, $SnCl_4$, $K_2Cr_2O_7$ and alum were dyed by methanolic extract of leaves of *C. sinensis* as shown in the Figure 4.53, the different mordants not only cause difference depth of shade also increased (i.e. K/S of 0.9860 increased to 2.1746). Similar results were obtained for the samples dyed in the presence of mordants using different dyeing techniques.

This could be attributed to the population effect of dye molecules present in the dye bath at higher concentrations. It was observed that, from the three dyeing techniques used for dyeing cotton with methanolic extract of *C. sinensis*, the post mordanting method showed a higher depth of shade, as well as colour values, compared with the fabrics dyed using the other two methods.

This may be due to the greater complex-forming ability of the metal ions with the dye molecules in this technique. *C. sinensis* has a very good substantivity for cotton. Thus, in the post-mordanting method, the dye is adsorbed onto the fibre, followed by the formation of an insoluble complex with metal ions.

(d) Fastness properties of dyed fabrics

The fastness properties of fabrics dyed with and without metal salts are shown in Table 4.46.

Table 4.46 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional dyeing with different metal modantings with methanolic extract of *C. sinensis*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (control)	4	2	2	2-3	2-3	4
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk (control)	2	2	2-3	2-3	2	2
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (control)	3-4	3-4	3	3	3	3-4
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness , Per – Perspiration fastness., Rub – Rubbing fastness.

Figure 4.54 Fabric samples dyed with methanolic extract of *C. sinensis* with different mordants.

The control sample shows comparatively good light and wash fastness properties, which were further improved with mordanting. Thus, ferrous sulphate and copper sulphate and showed the most marked effect on the light fastness of dyed cotton samples, irrespective of the method of mordanting. In the case of samples dyed by the simultaneous-mordanting and post-mordanting techniques, higher light fastness ratings were observed as compared with the pre-mordanting method of dyeing, irrespective of the mordant used. This could be attrihuted to the more rigid complex formation of the dye with a mordant, possibly due to better penetration of the dye molecules in the fibre matrix during meta- and post mordanting techniques.

(e) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of metbanolic extract of C. *sinensis* and fabric dyed with *C. sinensis* extract in mg/l are tabulated in Table 4.47.

Table 4.47 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.12	ND	0.03	ND	0.04	ND	0.43	0.14
Dyed fabric (mg/l)	0.02	0.12	ND	ND	ND	ND	ND	ND	ND
COD of the extract	270 -300								

ND – Not detected up to 10mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *C.sinensis.*

4.12.9 Walmadata *(Rubia cordifolia)*

a b

Figure 4.55 a, b *R. cordifolia* plant and dried chips

Local Name	Walmadata
Botanical Name	*Rubia cordifolia*
Colour Extracted	Reddish orange
Parts Used	The whole plant, mainly from roots, stems and leaves
Source	Traditional medicinal shops.
Part Used	Dried roots and stem

(a) Characterization

Visible spectrum of *R. cordifolia* extract: The methanolic extract from *R.cordifolia* shows peak at 400 nm.

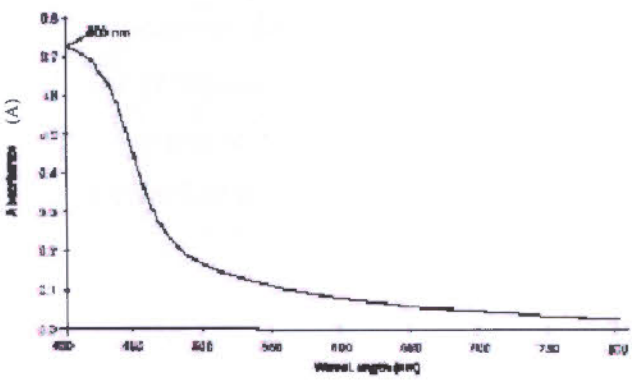


Figure 4.56 UV-Vis spectrum of methanolic extract of *R. cordifolia*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.57- 4.59 and CIE lab values are shown in Tables 4.48- 4.50.

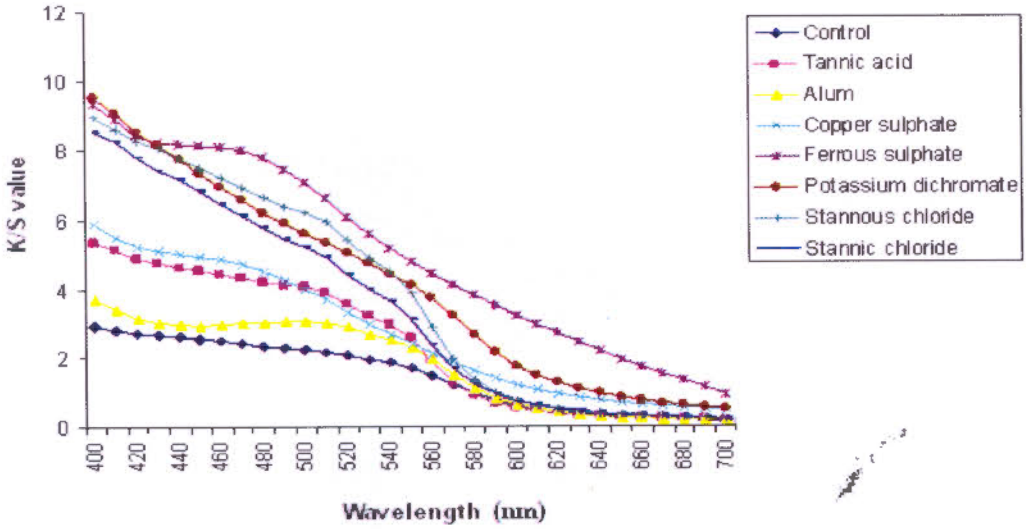


Figure 4.57 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *R. cordifolia*

Table 4.48 Characteristics for cotton fabric dyed with methanolic extract of *R.cordifolia*

Method	Mordant	L*	a*	b*	c	H	K/S
Pre-mordanting	Control	72.64	0.004	21.97	21.97	90.04	10.19
	Tannic acid + Dye	74.29	-0.49	26.08	26.08	91.13	16.33
	Alum	74.28	-0.46	26.06	26.06	91.06	16.40
	Copper sulphate	74.01	0.55	25.94	25.94	88.73	81.86
	Stannous chloride	76.29	0.06	30.50	30.50	89.84	17.18
	Ferrous sulphate	72.14	2.71	20.86	21.04	82.56	39.92
	Pot. dichromate	73.39	0.90	23.91	23.92	87.80	29.23
	Stannic chloride	74.94	0.97	27.71	27.73	87.95	21.38

The best values are obtained for methanolic extract of *R. cordifolia* with ferrous sulphate (K/S value of 39.92) and the order of reactivity is Fe> Cr > Cu > Al > Sn (IV) > Sn (II) for cotton samples as shown in Figure 4.58. The cotton samples were pre treated with tannic acid before mordanting.

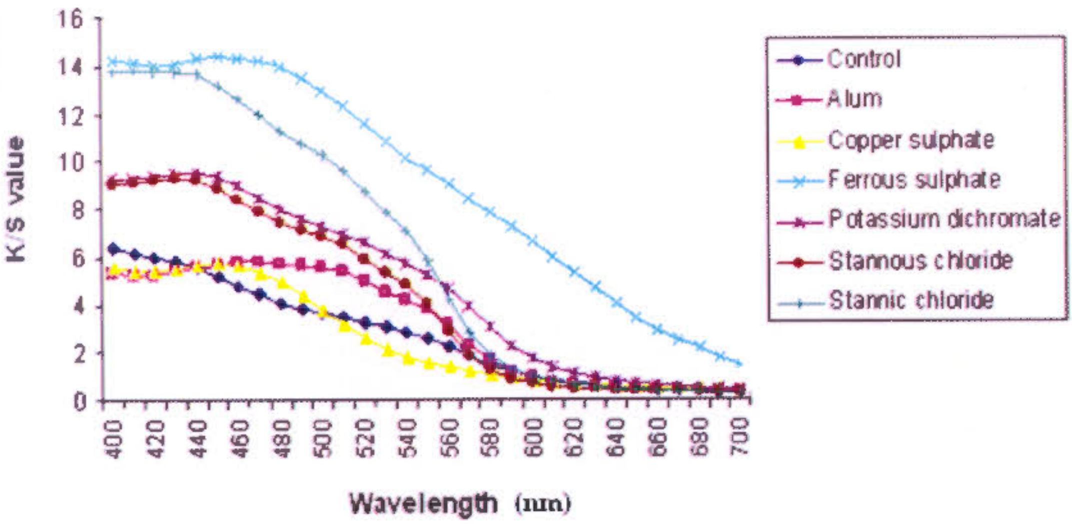


Figure 4.58 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *R. cordifolia*

Table 4.49 Characteristics for silk fabric dyed with methanolic extract of *R. cordifolia*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	62.99	-0.69	48.52	48.53	90.85	51.74
	Alum	65.19	-1.35	52.12	52.14	91.52	56.11
	Copper sulphate	62.89	-2.99	48.31	48.31	93.58	58.74
	Stannous chloride	62.51	-2.90	47.52	47.61	93.52	61.72
	Ferrous sulphate	57.41	2.97	38.09	39.09	85.60	83.68
	Pot. dichromate	61.34	0.25	45.73	45.73	89.64	57.40
	Stannic chloride	57.26	-1.95	38.24	38.29	92.96	61.75

The best values are obtained with ferrous sulphate (K/S value of 83.68) and the order of reactivity is Fe> Cu > Cr > Al > Sn (IV) > Sn (II) for silk samples as shown in Figure 4.59.

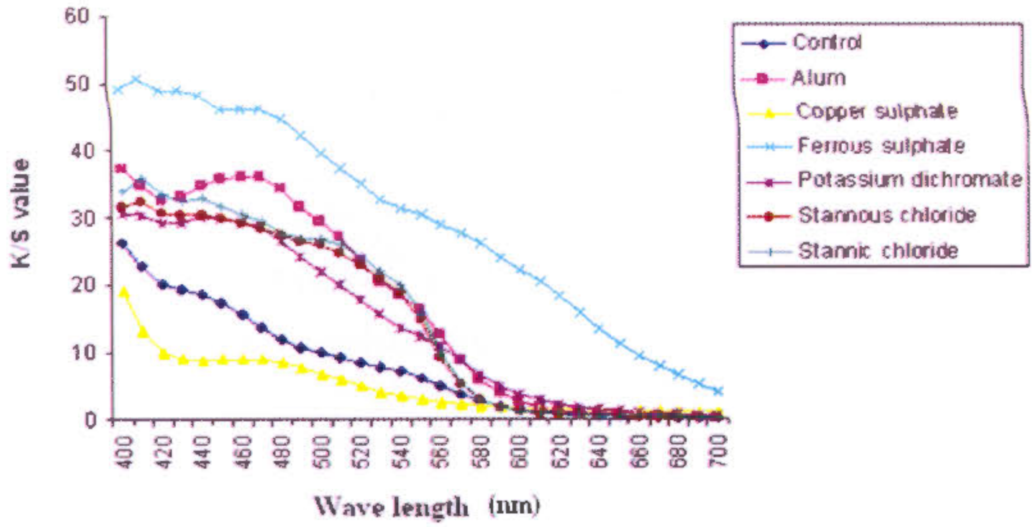


Figure 4.59 Change in K/S values with different mordants for wool yarns after dyeing with methanolic extract of *R. cordifolia*

Table 4.50 Characteristics for wool yarn dyed with methanolic extract of *R. cordifolia*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	56.45	3.87	46.52	46.68	85.21	76.98
	Alum	48.77	7.32	33.21	34.01	77.54	37.16
	Copper sulphate	47.95	-2.29	30.17	30.26	94.38	81.36
	Stannous chloride	55.79	4.83	45.34	45.60	83.88	84.94
	Ferrous sulphate	57.39	5.46	48.09	48.40	83.47	183.67
	Pot. dichromate	54.07	4.87	42.61	42.88	83.43	104.88
	Stannic chloride	48.92	8.21	32.90	33.91	75.95	80.56

The best values are obtained with ferrous sulphate (K/S value of 183.67) and the order of reactivity is Fe> Cu > Cr > Al > Sn (IV) > Sn (II) for wool yarns as shown in Figure 4.59.

Effect of mordanting conditions

The colorimetric data obtained from dyed fabrics and yarn which had been pre treated with tannic acid/metal mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pre treatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to control samples. It also increased the colour strength and flattened the shade of the dyeings. It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison. Therefore, in pre mordanting technique, the dyed fabrics were mordanted with stannic chloride, stannous chloride, ferrous sulphate, copper sulphate, potassium dichromate and alum. The conventional dyeing of cotton, silk and wool fabric and yarn with and without mordant by the methanolic extract of *R. cordifolia*, show that metal mordant showed very good results. The dye uptake values are 14.8 %, and 33.5 % for without and with metal mordants respectively. In the case of conventional dyeing, the dye uptake values are 38 % and 47 % for dye–alone and dye–metal simultaneous mordanting methods respectively. The effectiveness of metal mordant–*R. cordifolia* in better dye uptake may appear to be more as compared to without mordanting, and the reduction in effluent pollution as well as improved fastness properties outweighs its benefit.

(c) Fastness properties of dyed fabrics

Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional dyeing with different metal modants with methanolic extract of *R. cordifolia* are shown in Table 4.51.

Table 4.51 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional heating with different metal modanting with methanolic extract of *R. cordifolia*

Fabric (Mordant)	Fastness values					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Control)	2-3	2	2	2-3	2-3	2
Cotton (Alum)	4	4	3–4	3-4	3-4	4
Cotton ($FeSO_4$)	4-5	4	4	4	4	4-5
Cotton ($CuSO_4$)	4	4	4	4	4	4
Silk (Control)	2	2	2-3	2-3	2	2
Silk (Alum)	4	4	4	4	4	4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	4-5
Silk ($CuSO_4$)	4-5	4	4	4	4	4-5
Wool (Control)	3-4	3-4	3	3	3	3-4
Wool (Alum)	4	4	4	4	4	4
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	5
Wool ($CuSO_4$)	4-5	4	4	4	4	4-5

WF = wash fastness, LF = light fastness , Per – Perspiration fastness, Rub – Rubbing fastness.

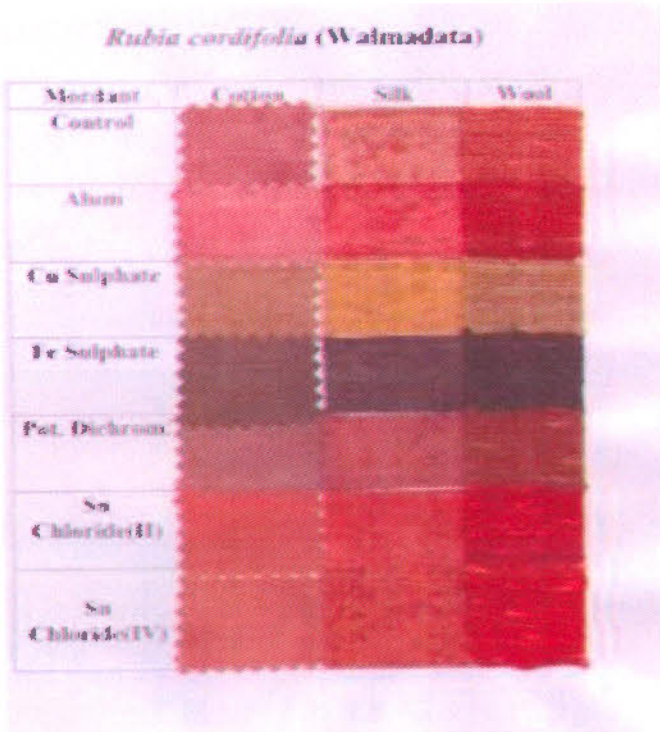


Figure 4.60 Fabric samples dyed with methanolic extract of *R. cordifolio* with different mordants

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of *R. cordifolia* and fabric dyed with *R. cordifolia* extract in mg/l are tabulated in Table 4.52.

Table 4.52 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.12	ND	0.03	ND	ND	ND	0.43	0.14
Dyed fabric (mg/l)	0.02	0.12	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	134 -250								

ND – Not detected up to 10 mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *R.cordifolia*.

R. cordifolia was found to have good agronomic potential as a dye crop in western province of Sri Lanka. Metal mordants when used in conjunction with *R. cordifolia* were found to enhance the dye-ability due to the Al contents present in the leaves. Enhancement of dye uptake was 23.5 % with Copper sulphate, 33.5 % with alum and 14.8 % without any mordant. Use of mordant not only enhances the fastness properties but also gives good colorimetric data on dyeing. Even the fastness properties in this case show good results, with mordanted dye, developed for the ease of industrial application offers an eco friendly process which should be popularized as an alternate method to the existing dyeing methods.

4.12.10 Turmeric (*Curcuma domestica)*

a

b

Figure 4.61 a,b *C.domestica* plant and root

Family	Zingiberaceae
Species	*C. domestica*
Source	Available across the country

(a) Characterization

Visible spectrum of *C. domestica* extract:

The methanolic extract from *C. domestica* shows peak at 205 nm and 220 nm.

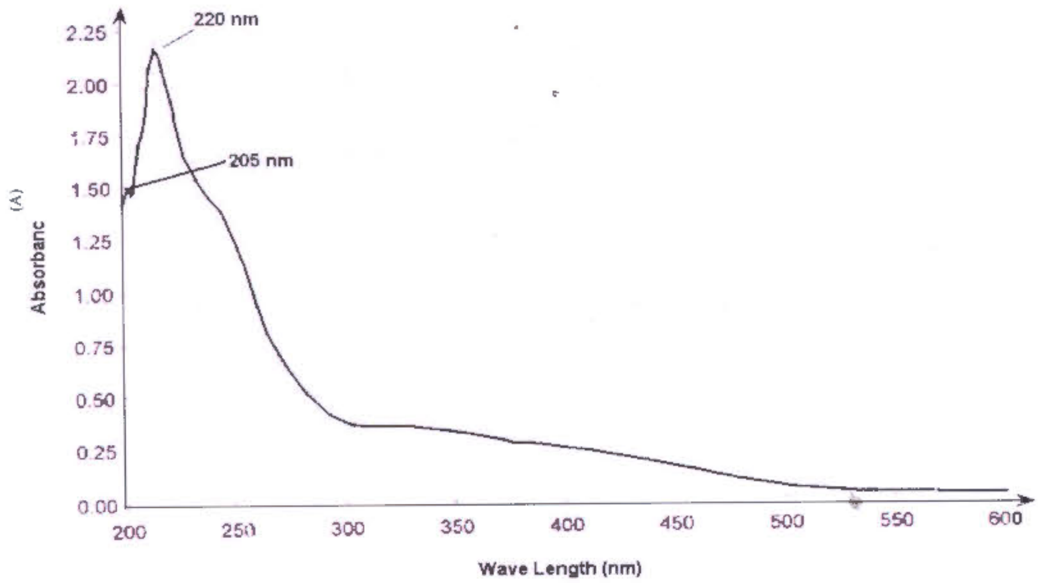


Figure 4.62 UV-Vis spectrum of methanolic extract of *C. domestica*

(b) Colour measurements: K/S were measured for cotton, silk fabrics and wool yarn as shown in Figures 4.63 – 4.65 and CIE Lab values are shown in Tables 4.53 – 4.55.

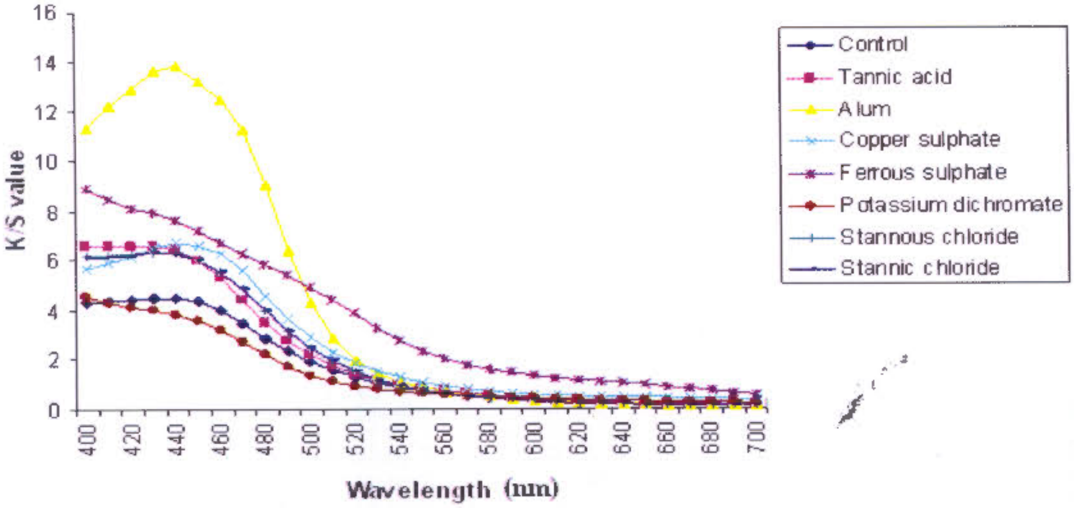


Figure 4.63 Change in K/S values with different mordants for cotton fabrics after dyeing with methanolic extract of *C. domestica*

The sequence of K/S values according to the mordant activity is Al > Fe > Cu > Sn (II) > Sn (IV) > K in cotton for methanolic extract of *C. domestica*. The best values are obtained with Alum (K/S value of 92.42) as metal mordant.

Table 4.53 Characteristics for cotton fabric dyed with methanolic extract of *C.domestica*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	50.265	8.79	26.61	28.02	71.68	55.66
	Control + Tan. acid	50.314	9.76	27.01	28.72	70.09	68.21
	Alum	51.800	8.44	29.56	30.74	74.03	92.42
	Copper sulphate	50.306	7.29	26.87	27.84	74.78	89.98
	Ferrous sulphate	43.126	1.44	7.30	7.44	78.82	70.11
	Pot. dichromate	50.887	7.28	27.82	28.76	75.30	83.44
	Stannous chloride	52.850	10.64	31.68	33.42	71.41	57.01
	Stannic chloride	53.694	11.80	33.33	35.36	70.46	58.81

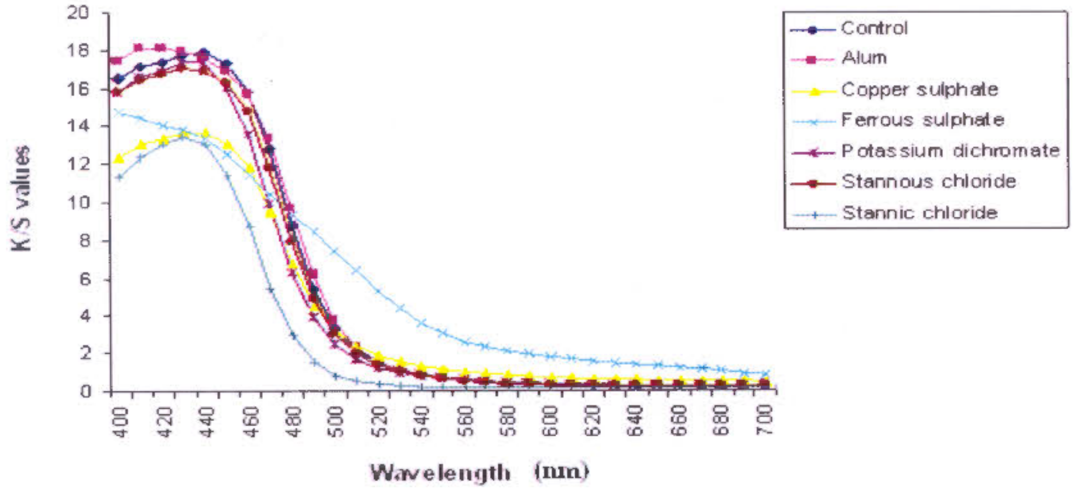


Figure 4.64 Change in K/S values with different mordants for silk fabrics after dyeing with methanolic extract of *C. domestica*

The sequence of K/S values is as Fe > Cu > K > Sn (IV) > Al > Sn (II) in silk for methanolic extract of *C. domestica,* the absorption of colour by silk fabric was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 197.44 with corresponding CIE Lab values.

Table 4.54 Characteristics for silk fabric dyed with methanolic extract of *C.domestica*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	60.097	6.24	30.61	31.24	78.44	32.96
	Alum	59.769	4.38	29.52	29.85	81.51	45.26
	Copper sulphate	59.629	5.68	30.25	30.78	79.32	59.51
	Ferrous sulphate	50.203	1.28	4.03	4.23	72.32	197.44
	Pot. dichromate	59.250	5.83	29.17	29.74	78.65	59.35
	Stannous	60.046	6.47	30.40	31.08	77.93	39.88
	Stannic chloride	60.978	8.94	32.90	34.10	74.77	48.19

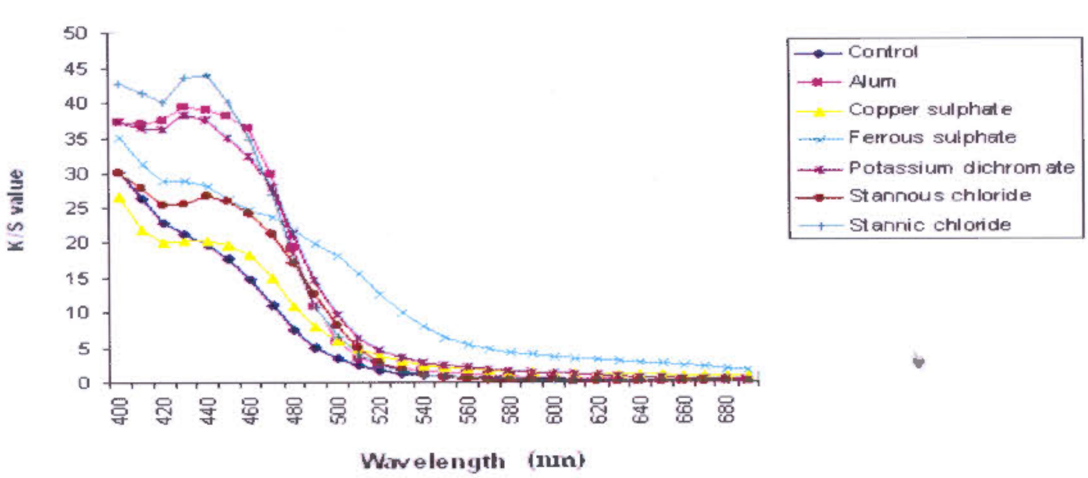


Figure 4.65 Fabric samples dyed with methanolic extract of *C.domesica* with different mordants

The order of K/S values is Fe > Cu > K > Sn (IV) > Sn (II) > Al in wool for methanolic extract of *C. domestica,* the absorption of colour by wool yarn was enhanced when using Ferrous sulphate as metal mordant with optimum K/S value of 335.45 with corresponding CIE Lab values.

Table 4.55 Characteristics for wool yarn dyed with methanolic extract of *C.domestica*

Method	Mordant	L*	a*	b*	C	H	K/S
Pre-mordanting	Control	57.567	8.23	32.88	33.89	75.91	43.26
	Alum	57.633	8.70	33.03	34.16	75.21	79.39
	Copper sulphate	55.814	4.88	29.60	30.00	80.60	133.54
	Ferrous sulphate	46.34	2.19	5.06	5.52	6658	335.45
	Pot. dichromate	54.605	5.21	27.83	28.32	79.34	130.17
	Stannous chloride	56.154	9.91	30.16	31.74	71.78	80.30
	Stannic chloride	58.300	9.51	34.18	35.48	74.43	80.94

Effect of mordanting conditions

It was observed that the pre mordanting technique with metal mordants imparted good fastness properties to the cotton, wool and silk fabric. Control samples without mordant were also prepared for comparison.

The colourimetric data obtained from dyed fabrics and yarn which had been pretreated with tannic acid/metal mordants in the case of cotton and only metal mordants in the cases of silk and wool reveal that pretreatment markedly improved the wash fastness, in terms of change of shade of the dyed fabrics with respect to control samples. It also increased the colour strength and flattened the shade of the dyeings.

(c) Fastness properties of dyed fabrics

Results are shown in Table 4.56 of metal mordanted samples for the dyeing conditions.

Table 4.56 Fastness properties of dyed cotton, silk fabrics and wool yarn under conventional conditions of metal modanting with methanolic extract of *C.domestica*

Fabric (Mordant)	**Fastness values**					
	WF	Per_{acidic}	Per_{basic}	Rub_{dry}	Rub_{wet}	LF
Cotton (Control)	3-4	3	3	3	3	3-4
Cotton (Alum)	4	4	3–4	3-4	3-4	3
Cotton ($FeSO_4$)	4-5	4	4	4	4	3
Cotton ($CuSO_4$)	4	4	4	4	4	3-4
Wool (Control)	3	3	3	3	3	3
Wool (Alum)	4	4	4	4	4	3
Wool ($FeSO_4$)	5	4-5	4-5	4-5	4-5	3
Wool ($CuSO_4$)	4-5	4	4	4	4	3
Silk (control)	3	3	3	3	3	3
Silk (Alum)	4	4	4	4	4	3-4
Silk ($FeSO_4$)	5	4-5	4-5	4-5	4-5	3-4
Silk ($CuSO_4$)	4-5	4	4	4	4	3-4

WF = wash fastness, LF = light fastness , Per – Perspiration fastness, Rub – Rubbing fastness.

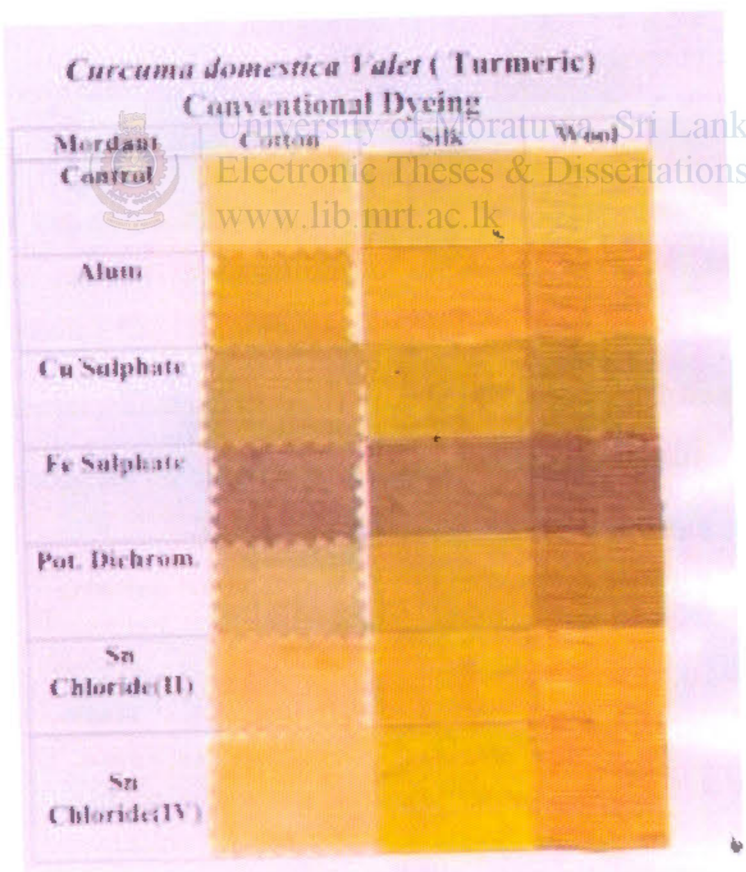


Figure 4.66 Fabric samples dyed with methanolic extract of *C.domestica* with different mordants

The dye uptake in case of conventional cotton dyeing ranges from 51-60 %, for silk 56-72 % and wool 55-65 % with different mordants. The effectiveness of metal mordant-*curcuma* in better dye uptake appears to be an improved process resulting in good dye adherence which results in good fastness properties its is a good source of natural dye for conventional dyeing as observed during the course of this study. In the case of curcuma, light fastness is slightly lower. With the use of proper mordanting conditions this poor quality also can be over come.

(d) Characterization of environmental impact

Trace elements in the dye and Chemical Oxygen Demand (COD) of methanolic extract of C. *domestica* and fabric dyed with *C. domestica* extract in mg/l are tabulated in Table 4.57.

Table 4.57 Characterization of environmental impact

Trace elements (mg/l)	**Cu**	**Zn**	**Cd**	**Co**	**Pb**	**As**	**Hg**	**Ni**	**Cr**
In the dye (mg/l)	ND	0.02	ND	0.0	ND	ND	ND	0.13	0.11
Dyed fabric (mg/l)	ND	0.05	ND	ND	ND	ND	ND	ND	ND
COD of the extract (mg/l)	120 -200								

ND – Not detected up to 10 mg/l

Results indicate that elements of major concern to the environment are absent in the dye solution and one can expect an effluent of much less impurities from dye operation involving *C.domestica.*

4.13 Environmental emission characteristics of effluents (COD Analysis)

Full COD data summary for the 10 dye samples are presented in Table 4.58.

Table 4.58 COD data summary

Extracted Natural Dye	**COD Range (mg/l)**
Rambutan	237-300
Marigold	200-210
Kothala	175-260
Weniwalgata	145-260
Big onion	154-300
Mangustine	180-250
Jak saw dust	149-200
Waste tea leaves	270-300
Walmadata	134-250
Turmeric	120-200

From the 10 analysed samples of extracted natural dyes, COD values of 05 samples are in the range of Central Environmental Authority's permissible range (250 ppm) for discharge into surface waters with a 1:8 dilution. Therefore some of the natural dyes in their chemical oxidative strength is environmentally compatible and can be considered as acceptable. Even though COD of other samples are in higher side of the permissible level (260-300 ppm), by using simple biological techniques these levels can be reduced to acceptable levels than in the case of synthetic dyes. Synthetic dyes need higher amount of chemical and auxiliaries for the treatment of effluents as they display high values for COD. Typical effluent strengths from local dyeing plants today are in the range of 1500-2000 mg/l. This observation is quite interesting and is a strong plus factor for the use of natural dyes. No prior literature exists in this area for any comparative analysis.

Table 4.59 Tolerance limits for effluents from textile industry discharged into inland surface waters (CEA, 2007)

Determinant	Tolerance Limit
pH value at ambient temperature	6.5 – 8.5
Temperature, ^{o}C, max.	40 measured at site of sampling
Total suspended solids, mg/l, max.	<50
Biochemical Oxygen Demand (BOD_5)	<60
Chemical Oxygen Demand (COD), mg/l,	250
Oils and grease, mg/l, max.	10.0
Phenolic compounds (as phenolic OH),	1.0
Sulphides, mg/l, max.	2.0
Chromium, total, mg/l, max.	2.0
Hexavalent Chromium, mg/l, max.	0.5
Copper, total, mg/l, max.	3.0
Zinc, total, mg/l, max.	5.0

4.14 Basic economic analysis

5.0 g of mercerized cotton fabrics were dyed to get standard depth shades equivalent to synthetic dyes (direct dyes) used in Sri Lankan textile dye industry. The required amounts of natural dye extracts were calculated and tabulated as follows.

Table 4.60 Cost of natural dyeing to get standard depth

Name of the sample	Botanical Name	Volume of Extra. Dye needed to get std. depth (ml)	Cost of Extraction (Rs.)	Cost of Raw Mat. (Rs.)	Cost of Dyeing (Rs.)	Total Cost to Dye 1 kg of fabric(Rs.)
Kothala	*S. reticulate*	970	428	700	660	2758.00
Turmeric	*C. domestica*	440	600	400	485	1925.00
Venival	*C.fenestratum*	600	540	550	470	2160.00
Walmadata	*R. cordifolia*	700	660	540	865	2765.00
Tea	*C. senensis*	850	550	200	775	2375.00
Rambutan	*N.Lappaceum*	450	470	125	480	1525.00
Mangus	*G.mangostana*	900	480	100	465	1945.00
Marygold	*T.erecta*	950	570	50	440	2010.00
Jak	*A.heterophyllus*	650	669	50	460	1829.00
Big Onion	*A. cepa*	760	680	60	475	1975.00

The cost of dyeing of 1kg cotton fabric with synthetic dyes is about Rs. 150/= in the industry (With Direct Dyes). Replacement cost is somewhat higher than synthetic dyes. Even though the cost is high still it is possible to replace synthetic dyes by accepted % because there is a trend to accept green dyes in the world. From the above results it can be concluded that a considerable amount of synthetic dyes can be replaced by using natural dyes.

4.15 Storage of Dyes (Preparation of Ready to dye concentrate of natural dyes)

If the raw material cost, transport cost etc., can be reduced to a certain extent these standards can be achieved with minimum cost involved. Low cost techniques should be adopted in order to prepare RTDC (Ready to Dye Concentrates) of natural dyes. Ready to Dye Form of natural dye extracts prepared in the laboratory are shown in the Figure 4.62.

1. *S. Reticulata*	6. *C. Domestica*
2. *R. Cordifolia*	7. *C. Senensis*
3. *C. Fenestratum*	8. *G. Mangostana*
4. *N. lLappaceum*	9. *A. Heterophyllus*
5. *A. Cepa*	10. *T. Erecta*

Figure 4.67 Ready to use Dye Concentrate of natural dyes.

4.16 Opportunity to use bio materials for similar colours with different mordants and substrates

It is important to consider similarities of colours produced by these natural materials. By considering the colours developed by each and every bio-material, colours obtained can be categorized as follows. The data presented in the CD are the supporting material for this classification.

Table 4.61 Categorisation of colour obtained from bio-materials

Brown colour		Yellow colour		Red colour	
N. lappaceum	wet & dry	*C. fenestratum*	Wet	*A. cepa*	Dry
G. mangostana	wet	*C. domestica*	wet & dry	*R. cordifolia*	Arid
C. senensis	wet	*T. erecta*	wet & dry		
S. reticulata	Wet & dry	*A. heterophyllus*	wet & dry		

Some of these bio materials are restricted to certain areas of the country. Therefore investigations were made on several materials which could produce the same colour. The availability of materials in different zones is also shown in the Table 4.61. Since some of the raw materials selected out of ten were seasonal, there can be problems in non seasonal period due to shortage in raw materials, production of dye materials. To avoid shortage in raw material supply during this period, possibility of replacement of colours were investigated. From the shades obtained for cotton, silk and wool fabrics following replacement possibilities emerge. Even though these raw materials could be stored in dry form safely during the season, this option is important for sustainability of natural dye industry in Sri Lanka.

Table 4.62 Bio materials with similar colours

colour	Cotton	silk	Wool
Brown			
N. Lappaceum	*C. senensis* , *A.cepa*, *S. reticulata* , *G. mangostana*	*C. senensis* , *S. reticulata* , *G. mangostana*	*C. senensis* , *S. reticulata* , *G. mangostana*
G. mangostana	*C. senensis* , *A.cepa*, *S. reticulata*, *N. lappaceum*	*C. senensis* *S. reticulata* *N. lappaceum*	*C. senensis* *S. reticulata* *N. lappaceum*
C. senensis	*A.cepa*, *S. reticulata* *G. Mangostana* *N. Lappaceum*	*S. reticulata* *N. Lappaceum* *G. Mangostana*	*S. reticulata* *N. Lappaceum G. Mangostana*
S. reticulata	*C. senensis* *A.cepa* *G. Mangostana* *N. lappaceum*	*C. senensis* *G. Mangostana* *N. lappaceum*	*C. senensis* *G. Mangostana N. Lappaceum*
Yellow			
Weniwelgata	*C. domestica*, *T. eecta* *A. heterophyllus*	*C. domestica* *T. eecta* *A. heterophyllus*	*C. domestica* *T. erecta* *A. heterophyllus*
Turmeric	*C. fenestratum* *T. erecta* *A. heterophyllus*	*C. fenestratum* *T. erecta* *A. heterophyllus*	*C. fenestratum* *T. erecta* *A. heterophyllus*
Marigold flower	*C. domestica* *A. heterophyllus* *C. fenestratum*	*C. domestica* *A. heterophyllus* *C. fenestratum*	*C. domestica* *A. heterophyllus C. fenestratum*
Jak saw dust	*C. domestica* *T. erecta* *C. fenestratum*	*C. domestica* *T. erecta* *C. fenestratum*	*C. domestica* *T. eecta C. Fenestratum*
Red			
Big Onion skin	*R. cordifolia*	*R. cordifolia*	*R. cordifolia*
Walmadata	*A. cepa*	*A. cepa*	*A. cepa*

After careful investigation of fabric samples dyed with extracts of plant materials in the precence of six different types of metal mordants, the following observation can be made.

Table 4.63 Bio materials with different mordants

Plant extract	Type of mordant	Corresponding K/S value		
		cotton	silk	Wool
N. lappaceum	$FeSO_4$	39.92	83.68	183.67
T. erecta	$FeSO_4$	187.77	271.63	256.54
S. reticulata	$K_2Cr_2O_7$ (cotton) , $FeSO_4$ (silk and wool)	39.77	81.93	125.28
C. fenestratum	$CuSO_4$ (cotton),Alum(wool),$FeSO_4$ (silk)	81.86	73.68	137.16
A. cepa	$SnCl_4$ (cotton) , $FeSO_4$ (silk and wool)	158.97	227.11	539.51
G. mangostana	$FeSO_4$	77.70	93.32	129.57
A. heterophyllus	$FeSO_4$	39.52	45.62	64.15
C. sinensis	$FeSO_4$	34.92	73.68	58.67
R. cordifolia	$FeSO_4$	39.92	83.68	183.67
C. domestica	Alum (cotton) , $FeSO_4$ (silk and wool)	92.42	187.44	335.45

4.17 Analysis of questionnaire

Following results were obtained from the analysis of sample results of questionnaire.

Total number of participants - **30**

People those who have already started natural dyeing in the industry: **03**

(Selling products in exhibitions)

Out of theses three there is only one person still carrying out natural dyeing

Those who are supposed to do natural dyeing in future -**19**

From the above analysis those who have targeted specific market is illustrated in the following pie chart.

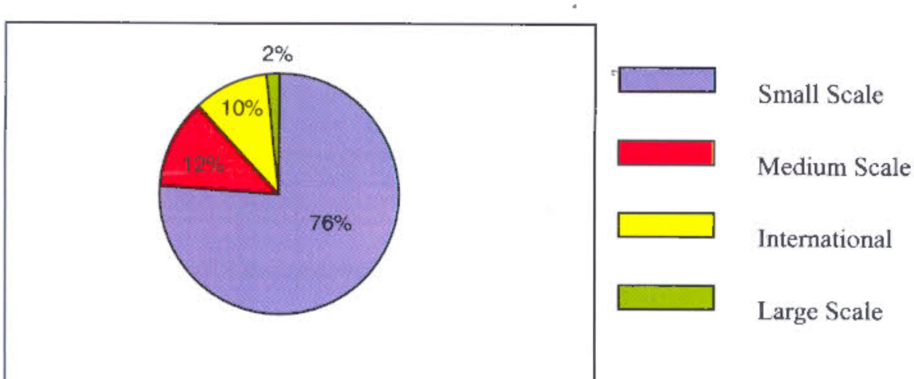


Figure 4.68 Analysis of questionnaire

4.18 The ways of finding resources:

From the above analysis ways of finding resources for natural dyeing was also investigated. Following chart illustrates the percentages.

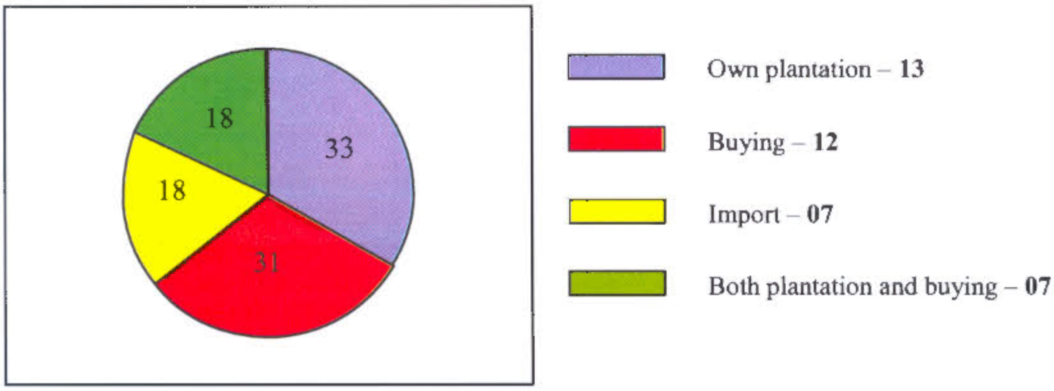


Figure 4.69 Sourcing resources

4.19 The views and comments from the exhibition – over the years are

- Art gallery Exhibition in Sri Lanka (year 2004)

  There was a good demand for natural dyed cotton sarees than other natural dyed products. Some customers were not satisfied with the prices of natural dyed products and there was not a sale just only research to recognize the natural dye market by displaying natural dyed products.

- Art Gallery exhibition (year 2005)

  Significant number of people who were interviewed, suggested expanding natural dyeing to a larger scale.

- Exhibition at BMICH (Year 2005)

  No formal stall for naturally dyed products. Some few items were sold prepared in
  this maner.

- Exhibition at BMICH (Year 2006)

  There was a separate stall for natural dyed products.
  Manufacturing price of a saree – Rs. 1500 -1600/=
  Selling price at the exhibition – Rs. 3000 - 3500/=
  Good demand for all the natural dyed products. All good quality products found ready acceptance and were sold out quickly.

- Exhibition at BMICH (Year 2007)

  It has been the most successful exhibition to-date in this area with a significant participation. The most interesting stall was "Natural Dyed Textiles" stall presented by the Department of Textile Industries, Sri Lanka. It was appreciated by many people and all the products were sold within the first two days. It is possible to market small amount of high price products and if one is selling large amount of products one would have to lower the price by about 50 %. Some photographs taken at these exhibitions are shown below.

Figure 4.70 Some views of the exhibitions

Chapter Five

CONCLUSIONS AND RECOMMENDATIONS

This chapter summarises the general conclusions from this research. This work is the first systematic study on the possibility of natural dyeing in Sri Lanka. Natural dyes cannot be used as simple alternatives to synthetic dyes and pigments. They do, however, have the potential for application, in specified areas, to reduce the consumption of some of the more highly polluting synthetic dyes. The characterisation of waste materials removes a major limiting factor associated with plant materials.

5.1 Findings of the study

Globally the textile industry is undergoing changes. The rapid changes in trends and fashion and the demand for good fastness properties on different substrates require a basic database describing possible applications of natural dyes.

The following conclusions were drawn from the investigations on screening, extraction and characterization of dye sources which proved that the objectives envisaged have been achieved to an appreciable extent. To achieve at least a partial replacement of synthetic dyes by natural dyes, the technical aspects of dyeing, defined by the demands of a modern dye house, have to be considered at the same time as the demands of the producer/manufacturer of the dye, e.g., the farmer / growers and waste collectors.

More than 60 % of tested dyeings achieved acceptable all round colour fastness properties while the ten selected bio materials indicated over 90 % acceptable fastness properties for cotton, silk and wool.

Strong positive environmental performance had been shared with natural dyes. The comparison between wastes after natural dyeing with the conventional process, revealed that a significant lowering of the chemical load, released with dye house effluents can be expected.

5.2 Analysis of individual dye yielding plant materials

Detailed analysis of ten best dye yielding bio materials were carried out as the final part of the study following the initial screening of 47 plants. This revealed the suitability of selected materials for textile dyeing and finishing industry.

The conclusions from the study are stated in association with each study objective.

(a) To investigate the natural dye producing plants in the world and those which are indigenous to Sri Lanka.

After carrying out a comprehensive literature survey the natural dye producing plants in the world were investigated. A considerable amount of natural dye producing plants are available all over the country. Globally about 55% of dyeing is contributed to the textile industry by natural dyes. Especially in India, manufacturing and dyeing factories are available in large scale to meet the local and international demand of dyed fabrics. Although there are about thousands of bio-materials are available worldwide and about 90 bio-materials as potential dye giving sources have been identified in Sri Lanka in this study (Annexure B).

(b) To study different techniques of natural dyeing available in the world and to investigate the traditional dyeing techniques practiced in Sri Lanka and their current status.

Modern colourant extraction techniques like superficial liquid extraction, sonicator extraction, solvent extraction etc., are also available world wide. In India new techniques like sonicator dyeing and high temperature pressure dyeing are being carried out with natural dyes. These state of the art techniques are used worldwide to meet the current demand in the textile industry. Traditional dyeing techniques are also investigated as a part of literature survey to acquire knowledge on how these were being carried out in the past to meet their day to day requirements. In rural temples these traditional methods are still being used, especially in dyeing of robe of buddhist priests. Currently most of the robes are dyed using synthetic dyes and the traditional techniques are diminishing. Newer techniques of natural dyeing should be introduced to obtain same or similar depth of shades which are equivalent to synthetic dye shade depths.

(c) To select plant materials which go as waste but still contain dye materials in relatively large quantity.

Out of the ninety plant materials gathered 47 bio materials were selected for investigation. Priority was given those which go as waste, but still contains dye materials in relatively large quantities. These selected wasteful biomaterials are abundantly available all over the country though some bio-materials are seasonal. Therefore proper storage facilities should be introduced to be consumed during off seasons. Another aspect is the different varieties of wasteful bio-materials. For example Rambutan contains two varieties namely yellow and red. On the basis of varieties, there should be consideration of the extractability, dye ability, stability of the shades produced and depth of shades of these verities. Matrix for analysis of possible options was introduced which could be followed in future.

(d) To select a method which is ecologically friendly and with less health hazards from a selected plant material.

Utilization of ultrasound energy for dyeing cotton with natural dyes is a definit improvement in the dyeing process. The method selected was sonicator dyeing which demonstrated the dyeing at lower operation temperature (Low energy dyeing was carried out mostly at room temperature, with not much need of heating the dye bath). The mechanical agitation causes slight rise in temperature, which helps in dyeing with less energy. In this method ultrasound energy of 20 kHz frequency was utilized. This would be a very low cost dyeing method that could be used in Sri Lanka and is recommended for further analysis.

Even heat sensitive dyes can be used in sonicator dyeing very comfortably without undergoing decomposition. The dye uptake is very good in sonicator dyeing. The same bath can be recharged and reused. Due to higher dye uptake the effluent is fairly clear thus least amount of dye discharge to the environment.

(e) To investigate its suitability as a textile dye and to indicate pathways for large scale exploitation to support the local textile industry.

The samples were dyed with natural dyes to get same standard depth shade as can be obtained from synthetic dyes. Then the replacement cost was calculated and it shows some what higher than the synthetic dye cost. Even though the cost is high still it is possible to replace synthetic dyes by acceptable percentage because there is a trend to accept green dyes in the world. It is possible to improve on this analysis with a better scale up study.

(f) To investigate the role of ultrasonic and conventional dyeing processes. These high-energy releasing devices used for dyeing are advancements over the conventional heating method.

Comparative study of Conventional vs Sonicator dyeing

- The conventional method of dyeing has been to boil the fabric or yarn in dye bath, till the desired colour is obtained.
- Enormous amount of heat is consumed in terms of heating the dye bath.
- Some dyes, which are heat sensitive, cannot be used in conventional dyeing because prolonged heating decomposes the dye molecules.
- The dye uptake by the fabric is also far from exhaustion and as a result, fair amount of dye is wasted.

(g) To emphasize on making ready- to- use newer natural dyes for commercial use. These plant extracts are otherwise sticky masses, not very easy to handle and store.

Extracted dye samples can be used as ready to dye stage by adding stabiliser to the extract. The stabilizer added was methyl paraban and it last for about one month. But shelf life of these samples also should be determined for manufacturing processes. This was not done in this research due to time constraints.

Finally the results can be summarised as follows :-

Table 5.1 Bio materials with different mordants

Plant extract	Type of mordant	Corresponding K/S value		
		cotton	silk	wool
N. lappaceum	$FeSO_4$	39.92	83.68	183.67
T. erecta	$FeSO_4$	187.77	271.63	256.54
S. reticulata	$K_2Cr_2O_7$(cotton), $FeSO_4$ (silk and wool)	39.77	81.93	125.28
C. fenestratum	$CuSO_4$(cotton),Alum(wool),$FeSO_4$ (silk)	81.86	73.68	137.16
A. cepa	$SnCl_4$ (cotton) , $FeSO_4$ (silk and wool)	158.97	227.11	539.51
G. mangostana	$FeSO_4$	77.70	93.32	129.57
A. heterophyllus	$FeSO_4$	39.52	45.62	64.15
C. sinensis	$FeSO_4$	34.92	73.68	58.67
R. *cordifolia*	$FeSO_4$	39.92	83.68	183.67
C. domestica	Alum (cotton) , $FeSO_4$ (silk and wool)	92.42	187.44	335.45

Table 5.2 Categorisation of colour obtained from bio-materials

Brown colour		Yellow Colour		Red Colour	
N. lappaceum	wet & dry	*C. fenestratum*	Wet	*A. cepa*	dry
G. mangostana	wet	*C. domestica*	wet & dry	*R. cordifolia*	arid
C. senensis	wet	*T. erecta*	wet & dry		
S. reticulata	Wet & dry	*A. heterophyllus*	wet & dry		

Dye exhaustion percentages of 10 bio-materials can be summerised according to the textile substrates as follows. (i.e. for cotton, silk and wool).

Table 5.3 Dye exhaustion percentages of 10 bio-materials

Plant extract	% dye uptake		
	Cotton	Silk	Wool
Rambutan (*N. lappaceum)*	50-55 %	52-62 %	60-65 %
Marigold (*Tegetus erecta)*	45-52 %	38-46 %	37-51 %
Kothala Himbutu (S. *reticulata)*	31-50%	37-52%	38-50 %
Weniwalgata (*C.fenestratum)*	31-50%	37-52%	38-50 %
Big onion (*A. cepa)*	65-68 %	70-74 %	78-82 %
Mangus *(G.mangostana)*	64-70%	75-82%	80-84%
Jak (A. *heterophyllus)*	55-62 %	68-70 %	59-73 %
Walmadata (*R. cordifolia)*	23- 42%	33 - 48%	14-65%

5.3 Positive environmental performance

Inductively Couple Plasma Spectrophotometer study on effluent from dye baths showed that the concentration of toxic heavy metals in dyes and the dyed fabrics was much below the stipulated limits and in most cases below detectable levels if present at all. Comparative charts of concentration of toxic heavy metals in different dyes are given. The emphasis was on effluent strength and to completely classify these extracts as eco-friendly dyeing process. As dyeing with synthetic colours represents an enormous ecological problem, there are environmental benefits to be had from greater use of renewable primary products and resources to be found in many fields: the use of non-renewable resources is minimized, ecological damage is reduced over the complete production chain and the value of agricultural areas is increased. Additionally, jobs are created and safeguarded with a regional creation of value and a simultaneous use of renewable raw materials. In the current economic context these jobs could be termed as green jobs.

The criteria for sustainable use of natural dyes in modern textile dye houses are:

Low resource consumption (life cycle approach)

Low emissions (life cycle approach)

5.4 Recommendations

The concept of Ready to use dye concentrate has been introduced in the study. The stabilization chemicals identified with some preliminary shelf life stability studies. A further study of this area is important because the international market also should be targeted with natural dyes. Shelf life of the individual bio-materials are also should be investigated to consider them as marketable products. These natural dye concentrates should be in powder form as in the case of synthetic dyes. If it is in liquid form it is difficult to handle such as transport problems may arise. Therefore these dyes should be in powder form in order to handle easily.

Toxicology of the extracted dyes should be investigated because prolonged contact of the skin with these dyes may cause health issues, viz skin rashes, allergies and skin cancer etc., which can be of chronic type or immediate response.

Colour shades also can be varied according to the geographical conditions like soil, climate. This also should be investigated.

5.5 Conclusion

It was proven that plant dyeing on an industrial scale is possible as the experiments on plant dyeing were positive. The production of dye-prototypes needs further research in order to evaluate the consequences of various preservation and standardization techniques. The study had concluded the production of dye concentrates and stabilization aspects for shelf life of dyes.

Use of ultra sound appears to be quite a premising approach. sonicator dyeing (at 20 kHz) used in case of natural dyeing has also been proved to give excellent dyeing performance. Therefore it was concluded that if some how these unconventional devices can be used at pilot scale, it would be a significant development in the field of dyeing industry.

From all the results obtained and the conclusions drawn, it can be said that the dyes extracted from waste bio-materials could contribute considerably to a cleaner dyeing industry. Some of the natural dyes in their chemical oxidative strength is environmentally compatible and can be considered as acceptable.

The findings and the conclusions of the present study may lead the textile chemists to work on making the present textile industry an eco-friendly industry. Thus it can be concluded that the use of natural dye in dyeing various textiles on a commercial basis will be a safe practice that could save our environment and improve the health of mankind for Sri Lanka it can be a significant green industry.

To conclude, there is an urgent need for proper collection, documentation, assessment and characterization of dye yielding plants and their dyes, as well as research to overcome the limitation of natural dyes. After due characterisation process development will involve a modeling and simulation step. This would involve developing relevant extraction and dyeing methods to predict scale-up operations.

List of Publications originated from the thesis

The following publications were originated from the studies on natural dyes.

1. Samudrika U.G., "Investigation of jak leaves and saw dust for potential colouring agent for textile substrates", Proceedings of the 12^{th} ERU Symposium, Oct., 2006 , pp 103-105
2. Samudrika U.G., "Investigation of jak leaves and saw dust for potential colouring agent for textile substrates", Proceedings of the 62^{nd} Annual Sessions, Institute of Engineers, Sri Lanka, Dec.2006, pp 65
3. Samudrika U.G., "Investigation on Cashew nut Shell Liquid as potential colouring and finishing agents for Textile substrate ", Proceedings of the 62^{nd} Annual Sessions, 2006, Institute of Engineers, Sri Lanka, Dec.2006, pp 63
4. Samudrika U.G., "Ultrasound techniques in Natural Dye application to Textiles" Proceedings of the 13^{th} ERU Symposium, Nov.2007, pp 46-48
5. Samudrika U.G., "An Environmental performance analysis of natural dyes" ,Proceedings of the 14^{th} ERU Symposium, Nov.2008, pp 26-27
6. Samudrika U.G., "Extraction of Textile colourants from kitchen wastes", Proceedings of the 14^{th} ERU Symposium, Oct. 2008 , pp 43-44
7. Samudrika U.G., "Extraction, Isolation & Identification of colouring substances in local natural dye yielding plants – jak saw dust", Proceedings of the 14^{th} ERU Symposium, Oct. 2008 , pp 138-139
8. Samudrika U.G., "Investigation of Domestic kitchen wastes as potential colouring agent for textile substrates", Proceedings of the 64^{th} Annual Sessions, Institute of Engineers, Sri Lanka., Dec.2008, pp 267
9. Samudrika U.G., "An Environmental burden analysis of synthetic dye vs. natural dyes" , Proceedings of the 64^{th} Annual Sessions, Institute of Engineers, Sri Lanka , Dec. 2008, pp 63

International Publications

10. Vankar P. S., Shanker R. , Wijayapala S. , de Alwis A.P.P., de Silva N.G.H., "Dyeing of cotton, wool and silk with extract of *Nephelium Lappaceum* (Rambutan) pericarp ", Asian Textile Journal – July 2007 , pp 66 – 70
11. Vankar P. S., Shanker R. , Wijayapala S. , de Alwis A.P.P., de Silva N.G.H. ,"Dyeing of cotton, wool and silk with extract of *Salacia Prenoids* (Kothala Himbutu)", Asian Textile Journal , June 2007, pp 69 – 74
12. Vankar P. S, Shanker R. , Wijayapala S. , de Alwis APP, de Silva NGH, "Dyeing of cotton, wool and silk with extract *Coscinium fenestratum* (Weniwal) ", Asian Textile Journal ,October 2007, pp 59 – 64
13. Vankar P. S., Shanker R. , Wijayapala S. , "Improved wash and light fastnesses by sonicator dyeing of cotton, silk and wool with *Curcuma domestica valet* extract", International Dyer193 (7), June 2008 , pp 38-42
14. Vankar P. S., Shanker R. , Wijayapala S. , "Dyeing of Cotton, Wool and Silk with extract of *Allium Cepa*" , Journal of Pigment and Resin Technology , Jan. 2009

15. Vankar P. S, Shanker R. , Wijayapala S. , "Dyeing Cotton, Silk and Wool yarn with extract of *Garcinia mangostana pericarp*," , the Journal of Textile and Apparel, Technology and Management (JTATM), Volume 6 , Issue 1 , 2009
16. Vankar P. S., Shanker R., Wijayapala S. , "Utilisation of temple waste flower *T. erecta (*Marigold) for dyeing of Cotton, Wool and Silk on industrial scale", the Journal of Textile and Apparel, Technology and Management (JTATM), Volume 6, Issue1, 2009

ANNEXURE – A

Questionnaire

University of Moratuwa.
Department of Textile and Clothing Technology.

- Name :
- Designation :
- Factory Name :

1. Have you already started Natural dyeing in Textile and Apparel industry?
   Yes
   No

2. Or do you hope to start in future?
   Yes
   No

3. If you already started when? (The year)

4. At that time what is your targeted market? (pl. select)
   International
   Local – Small scale
   Medium scale
   Large scale

5. If you deal with international market, what are the brands that you deal with?

6. Do you use your own brand name?
   Yes
   No

7. If you target local market, how do you sell your products?
   Through your own shop (name, location)
   Through distribution to other shops
   Through exhibitions

8. Is there a sufficient demand for your product?
   Yes
   No

9. Do you produce the quantity of market demand?
   Yes
   No

10. At present what is your targeted market?

International

Local- small scale

Medium scale

Large scale

12. What are the yarns that you use for Natural Dyeing?

Cotton

Silk

Wool

Nylon

Synthetic

13. What do you think of the demand of Natural Dyeing Market?

Increasing

Decreasing

Average

14. What do you think the cost of the Natural dyed products?

Same as the synthetic dyed product

Higher than the synthetic dyed products

Less than synthetic dyed products

15. Do you have any competition with synthetic dyeing product?

Yes

No

16. Mostly plants have been used to extract Natural Dye. Do you think it will effect to the environment?

Yes

No

17. How do you find resources?

Own plantation

Buying from local market

Import.

ANNEXURE B

A list of bio-materials used for natural dye sources

No.	Name	Botanical name	Family	Parts used
1	Thekka	*Tectona grandis Linn. f.*	Verbenaceae	Leaves, Bark
2	Dan pothu	*syzygium cumini L. Jamun*	Myrtaceae	Stem, Bark
3	Kohomba	*Azadirachtin indica*	Meliaceae	Bark,
4	Rambutan	*Nephelium lappaceum L.*	Sapindaceae	Skin
5	Bulath	*Piper Betle L.*	Piperaceae	Leaves
6	Jak	*Artocarpus hetorophyllus*	Moraceae	Saw dust
7	Weniwel	*Coscinium fenestratum Ga.col.*	Menispermaceae	Stem
8	Kurundu	*Cinnamomum verum Synonym c.*	Lauracea	Bark
9	Kothala Himbutu	*Salicia reticulata*	Hippocrateacaea	Bark
10	Delum	*Punica granatum*	Lithracaea	fruit skin
11	Rath Handun	*Pterocarpus Santalinus L.f.*	Fabaceae	Stem
12	Ranawara	*Gassia auriculata*	Cesalpinaceae	Flowers
13	Aralu	*Terminalia Chebula*	Combretaceae	Fruit
14	Bulu	*terminalia belerica*	Combretaceae	Fruit
15	Munamal Pothu	*Mimusops elengi L.*	Sapotaceae	Stem
17	Mangus	*Garienia mangostana L.*	Clusiaceae	Skin
16	Welmadata,	*Rubia Cordifolia L.*	Rubiaceae	Root and Stem
18	Daspethiya	*Tegetus erecta*	Asteraceae	Petals
19	Big onion	*Allium cepa L.*	Eliaceae	Skin
20	Wada	*Hibiscus rosa-sinensis*	Malvaceae	Flowers
21	Tea	*Camellia Sinensis L.*	Theaceae	Used leaves
22	Katarou	*Clitoria ternatea L.*	Fabaceae	Flowers
23	Kuppamenia	*Acalypa indica L.*	Euphorbiaceae	Leaves
24	Kopi	*Coffea Arabica L.*	Rubiaceae	Leaves, seeds
25	Kottamba	*Terminalia catappa L.*	Rubiaceae	Riped leaves
26	Devadara	*Erithroxylum monogynum Linn.*	Euphorbiaceae	Stem
27	Beet root	*Beta vulgaris Linn.*	Amaranthaceae	Rysome
28	Kaippu	*Acacia catechu Lnn.*	Fabaceae	wood
29	Pethangi	*Caesalpinia sappan*	Fabaceae	wood
30	Marathondi	*Lawsonia intermis L.*	Lythraceae	wood, leaves
31	Seyum wel	*Oldeulandia umbellate L.*	Rubiaceae	Roots and tubers
32	Rasandun	*Berberis aristata*	Berberidaceae	Roots and tubers
33	Ahu, Dumbu	*Morinda citrifolia L.*	Rubiaceae	Roots and tubers
34	Kudu miris	*Toddalia asiatica Lamk*	Rutacea	Roots and tubers
35	Kaha	*Curcuma domestica Valet L.*	Zingiberaceae	Roots and tubers

36	Kela gas	*Butea monosperma Lam.*	Fabaceae	Flowers
37	Sepalika	*Nyctanthus arbo-tristis L.*	Oleaceae	Flowers
38	Maliththa	*Woodfordia fruitticosa*	Lythraceae	Flowers
39	Anaththa	*Bixa Ovellana L.*	Bixaceae	Seeds
40	Nil awariya	*Indigofera tinctora L.*	Fabaceae	Leaves and stem
41	Hamparilla	*Mallotus phillipiueses Lam.*	Euphobiaceae	Stem
42	Welikaha	*Memecyloa capitellatuae L.*	Melastomataceae	Rhysome
43	Sen kottan	*Semecarpus anacardium L.*	Anacardiaceae	seed
44	Kumbuk	*Terminalia arjuna L.*	Combretaceae	Bark
45	Hik	*Lannea coromandelica L.*	Anacardiaceae	Bark
46	Ipil Ipil	*Leucaena leucocephala L.*	Fabaceae	Bark
47	Gas Penela	*Sapindus trifoliatus L.*	Sapindaceae	Seed
48	Wal inguru	*Zingiber cylindricum Moon*	Zingiberaceae	Rhysome
49	Puwak	*Areca catechu L.*	Erecaceae	Seed
50	Kothala Himbutu	*Salicia reticulata L.*	Hippocrateacaea	wood and root
51	Bowitia	*Osbekia aspera L.*	Melastomataceae	Bark
52	Mal ehela	*Cassia fistula L.*	Fabaceae	Bark
53	Mahogani	*Swietenia mahagoni L.*	Meliaceae	Bark, Saw dust
54	Bulu	*Terminalia berelia L.*	Combretaceae	Bark
55	Madan	*Syzygium cumini L.*	Mirtaceae	Bark
56	Rath mal	*Ixora coccinea L.*	Rubiaceae	Flowers
57	Kaju	*Anacardium occidentale L.*	Anacardiaceae	Bark And fruit
58	Sera	*Cymbopogon cutratus L.*	Poaceae	Rhysome
59	Inguru	*Zingiber cylindricum Moon*	Zingiberaceae	Rhysome
60	Rata kaha	*Bixa orellana L.*	Bixaceae	Seed
61	Alisarin	*Hydrorcy anatharaquinones*		Stem
62	Masakka	*Quercus Infectoria L.*	Fagaceae	Rhysome
63	Pipigngna	*Cucumis sativusl L.*	Cucurbitaceae	Fruit
64	Gammalu	*Pterocarpus marsupium Roxb.*	Fabaceae	Stem
65	Rata-embilla	*Morus Tinctoria l.*	Moraceae	Fruit
66	Annasi	*Ananas Comosus L.*	Bromeliaceae	Leaves
67	Kesel	*Musa Sapientum L.*	Musaceae	Muwa
68	Goraka	*Ggarcinia Cambogia L.*	Clusiaceae	Fruit
69	Nelum	*Nelumbo Nucifera gaertn*	Nelumbonaceae	Flowers
71	Carrot	*Daucus carrota L.*	Apiaceae	Fruit
72	Daisiya	*Chrysanthemum Leucanthemum L.*	Asteraceae	Flowers
73	Grass	*Zingiber cylindricum Moon*	Zingiberaceae	Leaves
74	Rosa	*Rosa Indica L.*	Rosaceae	Flower
75	Suriyakantha	*Helianthus Annuus L.*	Asteraceae	Flowers

76	Thakkali	*CSolanum Lycopersium L.*	Asteraceae	Fruit
77	Eucalyptus	*Eucalyptus globules L.*	Myrtaceae	Bark
78	Lemon grass	*Zingiber cylindricum Moon*	Zingiberaceae	Leaves
79	Nivithi	*Basella alba L.*	Basellaceae	Seed
80	Kudalu	*Impatiens flaccida*	Balsaminaceae	Flower
81	Canas	*Canna ediulis L.*	Cannaceae	Flower
82	Boganvila	*Bougainvillea spectabilis L.*	Nyctaginaceae	Flower
85	Beligeta	*Aegle marmelos L.*	Rutaceae	Fruit
86	Kaduru	*Cerbera manghas*	Apocynaceae	Fruit
87	Katakaluwa	*Myrica nagai L.*	Myricaceae	Fruit
88	Thembili	*Eugenia bracteata L.*	Myricaceae	Husk
89	Jak	*Artocarpus heterophylus L.*	Moraceae	Stem/ root
90	Turmeric	*Curcuma Domestica Valet L.*	Zingiberaceae	Rysome

ANNEXURE –C
Sieve Analysis Data

	Raw Material	Mesh size (µm)			
		0	**150**	**250**	**355**
1	Walmadata	1120	375	290	215
2	Madan Pothu	1350	500	100	50
3	Venival	1200	420	300	80
4	Turmeric	1015	550	150	285
5	Kothala Himbutu	1215	300	415	70
6	Delum (Skin)	1150	500	200	150
7	Rath Handun	1075	600	250	75
8	Ranawara	1150	700	250	200
9	Aralu	1050	700	200	50
10	Bulu	1025	450	400	125
11	Munamal Pothu	1065	600	200	135
12	Jak	1100	700	100	100
13	Mangustene	1050	450	300	200
14	Rambutan (Yellow)	1095	600	175	130
15	Rambutan (Red)	1035	500	300	165
16	Merygold (Orange)-1	1100	700	100	100
17	Merygold (Orange)-2	1030	500	270	200
18	Big Onion	1020	430	395	155
19	Red onion	1025	550	225	200
20	Eucalyptus	1025	650	200	125
21	Nuga	1045	550	225	180
22	Beli	1200	460	215	125
23	Iramusu	1115	600	200	85
24	King coconut	1065	565	275	95
25	Areconut	1075	655	185	85

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