EXPLORATION AND EXTRACTION OF COLOURING SUBSTANCES FROM HERBS FOR USE AS DYES ON COTTON FABRICS IN LOKOJA, NIGERIA

By

EBENEZER IGBEKELE ALESAGBA

DEPARTMENT OF INDUSTRIAL DESIGN
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA

OCTOBER, 2014

EXPLORATION AND EXTRACTION OF COLOURING SUBSTANCES FROM HERBS FOR USE AS DYES ON COTTON FABRICS IN LOKOJA, NIGERIA

By

Ebenezer Igbekele ALESAGBA, B. A*INDUSTRIAL DESIGN*(ZARIA) 2005
NO: P13EVID8015

A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES, AHMADU BELLO UNIVERSITY ZARIA

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OFA
MASTER OF ARTS DEGREE IN INDUSTRIAL DESIGN

DEPARTMENT OF INDUSTRIAL DESIGN
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA

OCTOBER, 2014

DECLARATION

I declare that the work in this thesis entitled **"Exploration and Extraction of Colouring Substances from Herbs for Use as Dyes on Cotton Fabrics in Lokoa, Nigeria"** has been performed by me in the Department of Industrial Design (Textile Section), Ahmadu Bello University, Zaria, under the supervision of Dr. S. Maiwada, Dr. P. O. Ada and Prof. M. K. Yakubu. The information derived from the literature has been duly acknowledged in the texts and lists of references provided. No part of this thesis was previously presented for the award of another degree or diploma at any institution of learning.

Alesagba Ebenezer Igbekele		October 10, 2014
.....	.....	.....
Name of Student	Signature	Date

CERTIFICATION

This thesis entitled **"Exploration and Extraction of Colouring Substances from Herbs for Use as Dyes on Cotton Fabrics in Lokoja. Nigeria"** by Alesagba Ebenezer Igbekele meets the regulations governing the award of the degree of M. A. (Industrial Design) of Ahmadu Bello University and is approved for its contribution to knowledge and literary presentation.

---------- ----------

Dr. S. Maiwada.
Chairman, Supervisory Committee Date

---------- ----------

Dr. P. O. Ada.
Member, Supervisory Committee Date

---------- ----------

Professor. M. K. Yakubu.
Member, Supervisory Committee Date

---------- ----------

Dr. S. Maiwada.
Head of Department Date

---------- ----------

Professor A.Z Hassan
Dean, School Of Postgraduate Studies Date

DEDICATION

This thesis is specially dedicated to the Almighty God “JEHOVAH” and the family of Alesagba for their kindness, love and support.

ACKNOWLEDGEMENT

The researcher wishes to express his gratitude for the dynamic educational leadership of Dr. S. Maiwada, the Chairman, Supervisory Committee and the Head of Department, Industrial Design, A.B.U., Zaria with who in the researcher discussed most of his problems and who read the draft of this thesis, made suggestions and advised the researcher at every stage of this research.

The researcher wishes to also record special thanks to Dr. P.O. Ada, member of the supervisory Committee, who not only read the draft, but also gave useful advice to the success of this work. My special appreciation also goes to Prof. M. K. Yakubu, one of my supervisors and the "HOD", of the department of Textile Science and Technology, A.B.U., Zaria for his contributions to this work. I also thank Mall. Buhari for his advice on the practical work as well as the support of Mall.Nuhu, and Mall. Abddul, of the above department for allowing me to use the facilities in their research laboratory, my profound thanks to Professor Bello and Professor Kolawole of the same department as well. My special thanks to Dr. O.A. Ajayi of Department of Chemical Engineering for his untiring encouragement throughout the period of this research.

I am thankful to Dr. M. S. Abubakar, HOD, of Pharmacognosy and Drug Development. Alhaji Adamu Mohammed (herbarium) Chief Technologists and Kabiru Ibrahim of Department of Pharmacognosy and Drug Development, A.B.U., Zaria for giving me the opportunity to use the equipment in their laboratory. I also appreciate the effort of Mrs. M. Omopariola during the sample analysis of dye samples at National Research Institute for Chemical Technology (NARICT) Zaria. My unreserved thanks to Mrs Chukwu Chinonyerem Gladys and Buhari of Department of Chemical Laboratory, African Textile Manufacturers Ltd, Challawa, Kano, during the period of carrying out light fastness test.

I gave my heartfelt thanks to the following lecturers of Department of Industrial Design, ABU Zaria. Dr. Gwari W. B., Dr. S.A. Dutsenwai the post graduate coordinator, Professor Kparevzua, B. A., Prof. Ahuwan, Dr. Garkida, Dr. Azi, and Mrs. Ali and the supports of the

Chief Technologist, Mr. Y. Bobzon and his family, Mr. Peter, Mall. Yusuf and Mall.Balarabe.

My special appreciation to Mr. G. A. Bolorunduro, Segun Bolorunduro, Mrs. Balagbogbo P., Rukayatu Toyin, Madam Olori Orebiyi, Mr. Owolabi J. F., Olugbeja T. O., Mr. Elijah Ayigun, and the family of brother Chidi in Kano for their support throughout the period of the field work.

My boundless gratitude also goes to Prof. Mathew Idowu Ajibaro, the Rector, Kogi State Polytechnic, who gave me the chance to do this study, by allowing me to go in service training throughout the study. I acknowledged the numerous assistances received from Dr. Haruna A. A., Director, Academic Planning, Mr. B. Y. Ebute former Dean D.I.D., Mr. Alonge Dele, the present Dean, School of Arts, Design and Printing, Mr. Jones Kolade, Dele Baba and all the staff of Department of Industrial Design, Kogi State Polytechnic, Lokoja for their support and cooperation during the research work and study. My special appreciation goes to Dr. Kashim Isah Bolaji, of the Department of Industrial Design, F.U.T.Akure., for his advice and contributions to the write up of this project. My thanks also goes to all the authors, i used their books and pictures as references in this project.

I am extremely grateful to my parents, brothers and sisters most especially Mr. Rotimi Alesagba, Mrs Ruth Adebayo, as well as my sweet heart, Mrs Mercy Alesagba, Damilola Alesagba and Hannah Daniel for their love, patience, sacrifice, cooperation, support and understanding. Special thanks and appreciation to Mr. Orenibi Taiye and Bello Bamidele Oke for taking time, in spite of their tight schedule to type this work and successful completion of it to the end.

Lastly, I would like to put on record my appreciation for the contribution of many other people who are not specifically mentioned due to lack of time and space. May Almighty God Jehovah reward them all.

ABSTRACT

The research entitled “Exploration and extraction of colouring substances from herbs for use as dyes on cotton fabrics in Lokoja, Nigeria" was designed to explore improved methods, for extraction of dyes in powdery form for the purpose of preserving the dyes from selected local herbs in Lokoja, Nigeria, using organic solvents. The aim of this study was to explore experimentally quantitative and qualitative extracts from local herbs with a view to initiate indigenous solution at producing cost effective dyes that will be affordable to textile designers. One of the objectives of the study was to identify and collect dye-yielding herbs that are found within Lokoja environment. It also focuses on answering such pertinent questions as: what is the extent of research verification of herbs within Lokoja environment that prove to be adequate as textile dye yielding substance? The findings from the study revealed that dyes of good quality were extracted from the local herbs.By using Soxhlet extraction (hot extraction), maceration and extraction through boiling with water methods. The result showed that maceration (That is, cool method of extraction) was the best method for extraction of herbs, based on the fact that the process did not involve heating before dyeing processes, along with the process of carrying out washing and colour fastness tests. The type of colours extracted from the local herbs included various shades of yellow and khaki brown discovered from the root of terminalia glaucescens (white Afara). The type of mordant used to aid colour fastness of the dye extracts, were mordants (chemicals) such as sodium chloride, copper II sulphate, alum, Iron, wood ash, potassium di-chromite on cotton fabric to ascertain their usability. Conclusively the results of the experiments performed and findings indicated that alum, potash, wood ash are suitable local additives for herbs dyeing, while the best mordanting techniques for them is 'pre-mordant', or “mordant – with”. It

is therefore recommended that with the various shades of dyes produced from the herbs, it makes free lancing by resist dyer easy to accomplish and setting up small scale industries.

TABLE OF CONTENTS

Title- - - - - - - - - - - Pages

Title page - - - - - - - - - - i

Declaration - - - - - - - - - - ii

Certification - - - - - - - - - - iii

Dedication - - - - - - - - - iv

Acknowledgement - - - - - - - - - v-vi

Abstract - - - - - - - - - - vii

Table of contents - - - - - - - - - viii

List of tables - - - - - - - - - - xii

Tables: Samples of dyed fabric rating - - - - - xii

List of plates - - - - - - - - - - xiii

List of figures - - - - - - - - - - xiv

List of appendices (letters)- - - - - - - - - xiv-xv

Definition of operational terms/ Abbreviations as used in the study - - xvi-xvii

CHAPTER ONE

1.0 Introduction - - - - - - - - - - 1

1.1 Background of the study - - - - - - - - 1

1.2 Statement of the problem - - - - - - - - 2

1.3 Aim of the study - - - - - - - - - 3

1.4 Objectives of the study - - - - - - - - 3

1.5 Research questions - - - - - - - - 4

1.6 Justification of the study - - - - - - - - 4

1.7 Basic assumptions of the study - - - - - - - 5

1.8 Significant of the study - - - - - - - - 5-8

1.9 Scope of the study - - - - - - - - - 8

1.10 Delimitation - - - - - - - - - - 9

1.11 Limitation - - - - - - - - - - 9

CHAPTER TWO

2.0 Review of related literature - - - - - - - - 10

2.1 Introduction - - - - - - - - - - 10

2.2 History of natural dyes - - - - - - - - 10-12

2.2.1 Natural dyes extraction - - - - - - - - 12-13

2.2.2 Type of natural dyes - - - - - - - - - 14

2.2.3 Advantages of natural dyes - - - - - - - - 14-15

2.2.4. Disadvantages of natural dyes - - - - - - - 15-16

2.3 Effect of geographical environment on colours by dye-yielding herbs - 16-17

2.4 Equipment, material and facilities for natural dye extraction - - 17-18

2.5 Methodologies/procedures for natural dye extraction - - - 18-20

2.6 Mordant requirement for herbs - - - - - - - 20-21

2.7 Precautionary measures of synthetic dyes - - - - - 21-25

CHAPTER THREE

3.0 METHODOLOGY - - - - - - - - 26

3.1 Introduction - - - - - - - - - 26-27

3.2 Materials and methods - - - - - - - 27

3.2.1 Specimens of the study - - - - - - - 28

3.2.2 Conditioning of the specimens - - - - - - 28-29

3.2.3 Description of the herbs - - - - - - - 29-31

3.3 The field works and laboratory experiments. - - - - -31-32

3.3.1 Collection of raw materials - - - - - - - 32-33

3.3.2 Procedures, time of collection and treatments of raw materials. - - 33-35

3.4 Experimental procedures - - - - - - 35-38

3.4.1 Identification of raw materials - - - - - - 38-39

3.4.2 Colour test and dye characteristics - - - - - - 39-40

3.4.3 Dye test - - - - - - - - - 40

3.5 Solubility test - - - - - - - - - 40-41

3.6 Dyes extraction and quantity available for extraction - -42-46

3.6.1 The UV spectral scan analysis test - - - - - - 47

3.6.2 Powder technology of dyes - - - - - - - 47-49

3.7 Mordanting procedures - - - - - - - 49-51

3.7.1 Dyeing procedures. - - - - - - - - 52

3.7.2 Standard preparation of dye solution - - - - - 52-54

3.8 Colour fastness tests - - - - - - - - 54-56

i. Wash fastness test - - - - - - - 57-60

ii. Light fastness test. - - - - - - - 60-65

iii. Perspiration and water fastness test. - - - - - 65-68

CHAPTER FOUR

4.0 Result and discussion - - - - - - - 69

4.1 Introduction - - - - - - - - - 69-70

4.2 Dye extraction and quantity extractable - - - - - 70-72

4.3 The Ultra Violet Spectrophotometer (UV) scan results. - - - 73-74

4.4 Assessment of wash fastness test - - - - - - 74-75

4.5 Assessment of perspiration fastness tests - - - - - 75-76

4.6 Assessment of light fastness tests. - - - - - - 76-78

CHAPTER FIVE

5.0 Summary, Conclusions and Recommendations - - - - 79

5.1 Introduction - - - - - - - - - 79-81

5.2 Summary of findings - - - - - - - - 81-82

5.3 Correlation of research findings in relation with previous research - - 83-86

5.4 Conclusion - - - - - - - - - 87

5.5 General recommendation - - - - - - - 87-89

6.0 References - - - - - - - - - 90-96

List of tables

Table 3.1 Description of the herbs - - - - - - - 97

Table 3.2 Washing fastness ISO standard guideline - - - 98

Table 3.3 Humiditycontrol solution standard - - - - 98

Table 4.1 Solubility test result for different solvent used in the study - 99-100

Table 4. 2 The quantity of dye extracted from the each parts of the three herbs - - - - - - - 100

Table 4.2.1 The Ultra Violet Spectrophotometer scans (UV) - - 101

Table 4.3 Wash fastness rating of sodium chloride with various timing on cotton fabric - - - - - - - 102

Table 4.4 Wash fastnesses rating of on cotton fabric for 2 hours each (120 mins.) - - - - - - - - 103

Table 4.5 Acids and alkaline perspiration rating of sodium chloride on cotton fabric with various timing - - - - - 104

Table 4.6 Acid and alkaline perspiration rating of the dyes on cotton for 2 hours each (120 mins)- - - - - - 105

Table 4.7 Light fastness rating on degree of fading compared with the wool standards sodium chloride with various timing - - 106

Table 4.8: Light fastness ratings on degree of fading compared with wool standards for 2hrs - - - - - - 107

Table 4.9 Samples quality control sheet: African textile Kano - - 108

Table 4.10A Samples of dye fabrics rating: wash fastness and perspiration test serial No 1- 89. - - - - - 109-119

Table 4.10B Samples of dye fabrics rating: light fastness test serial No 1-8 - - - - - 120-125

LIST OF PLATES

Plate 2.1 when mixed with the anthocynanins found in cabbage, acids and bases can produce a variety of colours - - - - 12

Plate 2.2 Jeans - - - - - - - 126

Plate 2.3 The Blue dyestuffs used for dyeing Jeans. - - 126

Plate 2.4 Colouring yarn. - - - - - - 126

Plate 2.5 Flowering herb. - - - - - - 127

Plate 2.6 Herb. - - - - - - - 127

Plate 2.7 Shades of Yellow/Wheat. - - - - 127

Plate 3.1 Harvesting. - - - - - - 128-129

Plate 3.2 Cutting. - - - - - - - 129

Plate 3.3 Drying. - - - - - - 130

Plate 3.4 Pounding and Grinding. - - - - 130

Plate 3.5 Extraction of dye techniques.- - - - - 133

Plate 3.6 Removal of Liquid dye extractions. - - - - 133

Plate 3.7 Dyeing processes. - - - - - - 138

Plate 3.8 Equipment - - - - - - - 138

Plate 3.9 Wash fastness tests. - - - - - - 139

Plate 3.10 Light fastness test: Standard MBTL fading (Mercury blended tungsten lamp) - - - - -140

Plate 3.11 Perspiration tests. - - - - - - 141

Plate 3.12 Dyed fabric samples. - - - - - - 142

List of figures

Figure 3.1	Map of the research study areas, town in Lokoja, Kogi State-	128
Figure 3.2.1	Soxhlet (Extraction apparatus)- - - - -	131
Figure 3.2.2	Heating bath (Used for evapouration). - - -	132
Figure 3.3.1	Determination of the maximum absorption of spectra (λmax) of the dye Leave of Locust Beans. - - -	134
Figure 3.3.2	Back of Locust Beans - - - - -	134
Figure 3.3.3	Locust Beans seeds. - - - - - -	135
Figure 3.3.4.	Leaves of Teak. - - - - - -	135
Figure 3.3.5.	Back of Teak. - - - - - - -	136
Figure 3.3.6.	Leaves of White Afara. -- - - - -	136
Figure 3.3.7.	Back of White Afara. - - - - -	137
Figure 3.3.8.	Root of White Afara. - - - - -	137
Figure 3.4	Launder O-meter (digiWASH SS^{TM}) - - - -	138
Figure 3.5	Standard MBTL fading (Mercury blended tungsten lamp) -	139
Figure 3.6.	Perspiration (SS). - - - - - - -	140

List of appendices: (letters)

Appendix. A1. Letter: To whom it may concern. - - - - 143

Appendix. A2. Request for solvents. - - - - - - 144

Appendix. A3. Letter of introduction: Dr. Salihu Maiwada - - - 145

Appendix. A4. Letter of introduction: Dr. M.K. Yakubu. - - - 146

Appendix. A5. Letter of acknowledgments: the researcher - - - 147

Appendix. A6. Acceptance letter: personnel manager African textile Kano - 148

Appendix. A7. Letter of introduction: Bayero University Kano - - 149

Appendix. A8. Reference letter: Kaduna polytechnic - - - 150

Appendix. A9. Letter to the supervisors of the researcher: senior lab Tech of African Textile Kano - - - - - - - 151

Appendix. A10. Application for proposal defence: the researcher - - 152

Appendix. A11. Application for seminar presentation - - - 153

Appendix. A12. Application for extension of registration period - - 154

Appendix. A13. Application for external defence - - 155

Definition of operational terms/abbreviations as used in this study

(a) Operational terms as used in this study.

Abete: Wood ash solution used as mordant for dyeing fabrics in Yoruba land.

Mordanting: When dyeing with most natural dyes, the fibre or fabric to be dyed will undergo some special preparation or treatment. This is to enable the colour to be fixed onthe fibre or fabric. This process is known as mordanting. Most common mordantsis be used in this study are: alum, chrome, tin, copper and iron.

Mordant Dyes: These classes of natural dyes are sometimes referred to adjective dyes. This has little or no colouring power in itself. So the fabric to be dyed with these dyes has to undergo the mordanting process in this study.

Non-Mordant Dyes: This is sometimes called substantive dyes. Unlike the mordant dyes, non-mordant dyes give their colour directly to the fabric. The process of mordanting is not as important as with the mordant dyes. Example of non-mordant dyes are the one derived from lichen.

Solvent: This is a substance (usually in a liquid form) whichhas the ability to dissolve the dye substance such as water and ethanol. This will be used in this study.

(b) Operational abbreviations as used in this study:

CC: Colour change.

CS: Colour stain.

DBH: Diameter at Breast Height,

D.M: Dry Matter.

FRA: Forest Reserved Areas in Kogi State.

HC: Humidity control solution.

ISO: International Standard Organization.

LSF: Light control solution.

P^H Is the degree of acidity or alkalinity of any solution.

SS: Stainless steel.

TBL: The back of locus beans herb.

TBT: The back of teak herb.

TBW: The back of white afara herb.

TLL: The leaves of Locust beans herb.

TLT: The leaves of teak herb.

TLW: The leaves of white afara herbs.

TRW: The root of white afara herbs.

TSL: The seeds of locust beans.

UV: (Utra violet spectrophoto meter). This spectrophoto meter is used to measure absorbance and transmittance of coloured solution at a specified wavelength.

WA: Wood ash.

CHAPTER ONE

1.0 Introduction.

The study on the exploration and extraction of dyes from various herbs as conducted by various researchers (Islam 2000 and Siva 2007) showed that there are needs to conduct further researches on the determination of quantity extractable from various parts of herbs that produce such dyes. It was indicated by Mike (2010) that any dye applied on fabric should not be easily removed by primary laundering operation, and must not fade rapidly when exposed to light. This explained that dyes as colour forming substance intentionally applied to the fabric by special treatment must produce a possible permanent concentration of colour.

The selection of the herbs as source of dyestuff was due to previous observation from local herb sellers. The juice of the herbs when it comes in contact with certain part of fabric, a stain or colour appears after a short time. It was further observed that these stains were not always easily removed. This gave an indication that textile dyes may be obtained from such herbs.

1.1 Background of the study.

Traditionally, the production of local dyes from herbs has been a century old practice. Nigerians and people from other countries have been engaged in production of dyes and application of these dyes on fabric before the arrival of the

colonials (Sarmah, *et al.,* 2009). Dye extracts can be derived from trees or herbs (leaves, barks, nuts, seeds and roots).

The application of local dye from the herbs' is still been practiced among dyers who used them to dye their fabrics. Alau (1986) emphasized the need for the continuity of art of embellishing textile materials with natural colorants in order to maintain the practice of the indigenous dyers. Adeleye *et al.,* (2003) observed that local herbs sellers referred to these herbs as "Elewe Omo" in Yoruba land. "It means the seller of children herbs". He further noticed that these herbs have different colours such as green, yellow, and brown just to mention, but a few. As these colours from herbs drop on fabrics, they stain them.

However, this indigenous technology is rapidly being eroded as its transmission to succeeding generations has drastically reduced. This is due to availability of modern industrially processed dyes. Few examples are synthetic dyes, such as azo, direct, azoic, etcetera. These synthetic dyes are produced from aromatic amines which is believed to cause cancer of the bladder.

This study explored the extraction of colour substances from herbs within Lokoja for possible use as "dye" on "cotton" fabric. This is because some Nigerians, hitherto, have despised the home-made dyes and are unrepentantly depending on imported goods such as synthetic dyes.

1.2 Statement of the problem.

It is observed that almost all the dyes used in Nigeria are imported. As a result of which Government huge taxation and sometimes ban of textile materials as synthetic dyes makes it more difficult for fabric dyeing in textile institution. The source of acquiring dyes, therefore, has become difficult for Kogi State Polytechnic

Lokoja and Federal College of Education, Okene. It is also observed that studio practical experiments in textile design on fabric and research related to extraction of natural dyes have not been exhausted. It is equally observed that Kogi State possesses the desired herbs and is richly endowed with natural resources such as dye yielding herbs, particularly in Lokoja and its environs. The herbs available in Lokoja have not been adequately studied and documented. This study therefore, is concerned with extraction of dyes from local herbs which will go a long way at giving alternative to importation, thereby reducing cost of production.

1.3 Aim of the study.

- The aim of this study is to explore experimentally quantitative and qualitative extracts from local herbs from Lokoja with a view to provide indigenous solution to production of cost effective dye that is affordable to textile designers.

1.4 Objectives of the study.

The objectives of the study are to:

(i) identify and collect dye-yielding herbs that are found in Lokoja environment.

(ii) determine the appropriate method of extracting the dye substance from the herbs.

(iii) assess the type of colour dye hues obtained from the extracted dyes of herbs in the study area.

(iv) identify the type of solvent that can be used to dissolve the type of dye extract from the specimens.

(v) determine the type of mordant that can aid fastness of the extracted dye substances in washing, light and perspiration.

1.5 Research questions.

i. Has the local herbs in Lokoja environment of Kogi State been adequately identified and assessed as textile dye yielding substances?

ii. What are the appropriate methods for extracting dye from the local herb found in Lokoja?

iii. What type of colour dyes are extracted from to the local herbs in the study area?

iv. What type of solvent can be used to dissolve the dye substances to be extracted from the specimen?

v. What type of mordant can be used to aid colour fastness of the dye extracts dye?

1.6 Justification of the study:

- This study which focuses in dye extraction from herbs will minimize the problems associated with synthetic dyes. These problems are muthfanrious, which include increase hazards, memory and psychological problems, increase heart beat, etc which are harmful to workers in textile industries and institutions in Nigeria

- This study may encourage Government to revert to the use of herbs which are environmentally friendly for dyeing fabrics.
- Also, it would create job opportunity and economic development.

1.7 Basic assumptions of the study.

It is assumed that the findings in this study will aid in the supply of dyes for local textile design need in Lokoja, Nigeria. It also assumed that:

a. The true position of dye yielding materials of Lokoja, Nigeria can be determined through laboratory procedures.

b. Extracts can be preserved in powdered form for further references.

c. Herbs selected can generate colour or dye for textile use.

d. Dye producing herbs exist in Lokoja, Nigeria and;

e. Extracting agents other than distilled water will be applied scientifically to extract dyes.

1.8 Significance of the study.

The significance of this research is based on its educational, economic, socio-cultural value and technological importance. The followings are the major points for the significance of the study that will be discussed below:

(a) Educationally

Large numbers of available literatures on exploration, extraction and preservation of dye from herbs have been written by foreign authors and few authors only concentrated on the extraction of colouring substances from herbs as dyes for possible use on cotton fabrics in Zaria, Kano, Abeokuta, in Nigeria. There seems

not to be information of dye extraction in Lokoja in open literature. For instance, Siva (2007) recorded that in Britain, a shrub known as black berry, yielded a brownish grey colour with alum mordanted wool. In another related report, Labil (2010) documented that in United State of America, the black berry, yielding grey with alum mordant wool, indicating that the conditions and environment of growth of the same herbs species may affect the colour extract from it. The types of fabrics mentioned by the above authors (wool) are not easily available in Lokoja, Nigeria. The research findings on exploration and extraction of colouring substances from herbs for possible use as dyes on cotton fabrics would contribute to the existing literature, in the dye extraction from herbs. It would guide the students of textiles and their institutions about the wash and light fastness tests.

(b) Economically

Nigeria populace should move toward self- reliance and economic independence. In Nigeria cotton is produced locally, the dyes produce from herbs will be in large quantity and cheap enough to create design on fabrics from textile institutions and industries. It is noted by Mosaku (1987) that dye extraction will reduce the degree of the dependence on imported dye staffs and conserve our foreign exchange as more researchers delve into investigating local sources and materials such as dyes extracted from herbs and refine them for export and local use. However their significant importance also includes:

- Provision of job opportunities or employments to young textile graduates in Lokoja, Nigeria.
- provide immediate availability of dyes for use in small and medium scale industries, instead of depending on imported synthetic dyes.

- give room for division of labour to be established within textile industries, dyers and farmers as:
  - help local craftsmen commissioning people on personal basis for the collection of local herbs for colouring substances in order to use them as dye-stuff on cotton fabrics.
  - create more horticultural job for people who are not employed as they planted more herbs for textile institutions, that require application of natural herbs on fabrics. Therefore, this initiative will reduce the scarcity and cost of dyestuffs in Nigeria.

(c) Culturally

The findings will identify Kogi State as one of the dye producing area in Nigeria. Man cannot use fabric without beautifying it with colour that will enhance the beauty of the textile fabric. For this reason Kparevzua (2009) states that man's striving for beauty through the ages has been expressed in the use of clothing for personal adornment, as well as utility. The fabric used for these purposes essentially required dyes in them. Therefore, colour sometimes speaks louder than words. It appeals and serves, as a universal mode of expression as related to cultural and social values. For example the flag of Nigeria symbolizes green, white and green. Also, it is a tradition in Kabba town (Kogi State, Nigeria) that a man that wears red cap is regarded as a chief. Kparevzua (2009) maintains that fabrics to most consumers have no meaning without a colour.

Therefore, the importance of natural dyes on fabrics cannot be over emphasized. This explains that most of the consumers of textiles select fabrics based on their

choice of colours, as they appeal to them more than the texture and the characteristics of the fabrics. Alau (1986) describes colour as a very private and personal response to what individual look at. This ascertains that dyes play vital roles on fabrics as they serve as means of communication and beauty to various groups of people.

(d) Technologically

This research will encourage the technological extraction of dye yielding herbs in Nigeria. The technology for production of natural dyes could vary from simple aqueous to complicated solvent systems, to sophisticated supercritical fluid extraction techniques, depending on the product and purity required. Purification will entail filtration and drying of the herbs by spraying or under vacuum or using a freeze drying technique. Use of bio-technological methods to increase the yield of colourants in herbs is also being attempted in several laboratories in Nigeria (Adamu, 2010). In this study, Maceration and soxhlet methods will improve extraction of herbs, instead of traditional method of water and ash for extraction and dyeing. Technologically, it encourages the extractions of dyes in Nigeria on a large scale:

- Bigger companies can come and plant more herbs.
- Extraction of dyes.

1.9 Scope of the study.

This study is mainly an experimental type, limited to extraction of dyes and dyeing of cotton fabrics. Viz.

- **Parkia biglobosa** (*Botanical name*), **African Locust bean** (*common or trade name*) Dorawa (*Hausa*), Iru (*Yoruba*) *Local names*
- **Tectona grandis** (*Botanical name*) **Teak** (*Common name*) Labadiya *(Hausa)* Igi-oba *(Yoruba) Local name. Labadiya (Hausa).*
- **Terminalia glaucescens or Terminalia laxiflora,** (*Botanical names*) **White afara** (*common name*) Baushe (*Hausa*) Idi apata/ Idi-odan *(Yoruba) local names.*

1.10 Delimitation

The study has been delimited to those dyes that were extracted from three herbs namely, **Parkia biglobosa, Tectona grandis and Terminalia glaucescens or Terminalia laxiflora** respectively. The parts of the herbs from extraction will be carried out include the leaves, barks, seed, and root. Therefore, any other herbs that were not listed in the study were not included. The study covers Lokoja and its environs. The dyes extracted were applied on only cotton fabrics. Five selected mordant and six different solvents were employed.

1.11 Limitation

Due to paucity of time and finance, the study was limited to the extraction of dyes from herbs and these were applied on experimental cotton fabrics only.

CHAPTER TWO

2.0 Review of related literature.

2.1 Introduction

This chapter required review materials that are related to studies in the areas of dyes extraction and technology. The following mentioned are highlight of areas reviewed: history of dye, types of dye, dye extraction, equipment, materials, facilities, precaution and procedures among others.

2.2 History of natural dyes:

Natural dyestuff and dyeing are as old as textiles themselves. (Chenedu 1998). Man has always been interested in colours. The art of dyeing has a long history and many of the dyes dates back to prehistoric era. Although it was not easy in the ancient times to dye a cloth of certain colour, for no synthetic dyes were known, some of the materials used by the Israelites for the Tabernacle were dye "coccuss scarlet". This phrase is used rather than just the word "scarlet" as it is believed to be the most ancient dyestuff record. (Anonymous 2008, Sanees *et al.*, (2001)

Furthermore, Sarmah, *et al.,* (2000) and Islam (2000) agreed that the use of natural dyes dated back to more than 5,000 years, with Madder (*Rubiacordifolio*) dye found in some parts of Nigeria. Emu (1984) stated that nobody knows its exact-origin, but dyeing was practiced in many places all over the world and cannot be traced to one particular area. He goes further to say that it was practiced in China between the year 618 – 906 AD; it was practiced also in South-East, Asia, Indonesia, Peru and parts of Africa. Ajarosi (2009), Rukayatu (2009) and Owolabi (2010) confirmed

that Kogi State is endowed with a wealth of natural flora and fauna, which provide the basic resources for a rainbow of natural dyes. Natural dyes are environmentally friendly, for example, turmeric, the brightest of naturally occurring yellow dyes is a powerful antiseptic and revitalizes the skin while indigo yields a cooling sensation.

Gulrajani (1992), *The Columbia Encyclopedia* (2004) mentioned that by the 4th Century AD, dyes such as woad, madder, weld, Brazil wood, Indigo and a dark reddish-purple were known. Brazil was named after the woad found there. Gulrajani (2001) opines that "Henna was used even before 2500BC, while saffron is mentioned in the Bible." The first use of their blue dye, was by the ancient Britons, and may have originated in Palestine, where it was found growing wild. Indigo is one of the most well known of the dyes herb and also one of the oldest, and still being used in tropical Africa to dye cloths, particularly in West Africa where the tie-dyeing has developed to an impressive level.

Ogunbowale, *et al.*, (2009) noticed that Nigerians enjoy home dyed fabrics made with cotton materials that are conducive to our weather and convenient for occasions. They appreciate the gesture of former president *General Olusegun Obasanjo* who patronizes indigenous textile industry regularly as he is uniquely clad in dyed Nigerian cloth. The diverse and entire cultural group in Nigeria actively engaged in dyeing trade or business either on individual basis, small groups or as cottage industry as well as small and medium enterprises respectively.

The colourants extracted from herbs are being used as dyes, this has some significant achievement for man. Colour substances can be applied in a solution or dispersion, to a substrate, thus giving it a colour appearance. Therefore, Gwarzo and Alabi (2009) defined dye as a substance used to dye material permanently in such a

way and manner that the imparted dye seem to be part of the material and not merely applied which can be natural or synthetic dyes. Therefore, to extract dyes that can be applied on fabrics permanently becomes greatest thrills and challenges manufacturers of dyes.

2.2.1 Natural dyes extraction.

James (2000) states that almost all parts of the herbs like root, bark, leaf, fruit, wood, seed, flower, etc produce dyes. He noticed that it is interesting to know that over 2000 pigments are synthesized by various parts of herbs, of which only about 150 have been commercially exploited. According to Chandramonli (1995) it was believed that nearly 450 herbs are known to yield dyes in Nigeria and other parts of the world. Herbs are considered to be the most important as ten of these are extracted from roots, four from barks, five from leaves, seven from flowers, seven from fruits, three from seed (Gulrajani 2001).

Many natural dyestuff and stains were obtained mainly from herbs and served as sources of natural dyes, producing different colours like red, yellow, blue, black, brown and a combination of these dyes is shown in plate 2.1:



(Plate.2.1) *when mixed with the anthocynanins found in cabbage, acids and bases can produce a variety of colours.*
Source: James Elkin (2000) Amazon.com

As stated by Siva (*2007),*the increasing market demand for dyes and the dwindling number of dye-yielding herbs led to the emergence of synthetic dyes like aniline and coal-tar, which threatened total replacement of natural dyes. Even today, some dyes continue to be derived from natural sources, for example, dyes for lipstick, fabrics, leathers are still obtained from Bixa Orellana L. Lithospermum erythrorhizon Sieb of Zucci, are from indigo. The Wealth of India Dictionary (2003) states that the content or amount of dye present in the herbs varies greatly depending on the season as well as age of the herb. There are also several factors which influence the content of the dye in each dye-yielding herb. In some cases, the dye content has not been thoroughly studied so far. This therefore, provides manufacturers greatest thrills and challenges.

The study explained that Nigerian dyed cloths with natural herb, most especially indigo has become popular in Europe and America in recent years. *James (2000)* asserts that Indigotin itself is not present in the indigo herb, but is obtained from the glycoside indican hydrolysis, followed by air oxidation in alkaline solution. He further stated that, in practice, the hydrolysis of indican is carried out by crushing the herbs in water, when enzymes known as *B* –glycosidase catalyze the fermented herb, then, the hydrolysis of the glucose which is Indigo became a deep blue water- insoluble dye with a bronze tinge. This reduced deep blue to indigo white known as a bright yellow compound. Debajit (2008) also supports that Indigo dyes are mostly made into jeans in Plate.2.2 appendix.

2.2.2 Types of natural dye.

According to Gwari (1989), dye generally can be divided into two groups; Natural and Synthetic. Siva (2007), Mbahi, (2001)explained that Natural dyes can be sorted into three categories: the one obtained from plants, animals and minerals. WildMint (2009) says until the late 19th century, natural dyes were used to colour yarns used for weaving in plate 2.4 appendix.

Information about dyeing practices before the 16th century is limited, but it is known that all dye matters come from mineral pigments, and herbs. Interestingly, James, (*2000*) stated that betalains are only found in one sub-group of flowering herbs (Caryophylalles or Centrospermae). Bougainvillea, certain cacti, and amaranth are all examples of this family.

The examples of herbs were seen in plate 2.5 to 2.7 appendix respectively.

2.2.3 *Advantages of natural dyes.*

Isam (2000) opined that dyes extracted from herbs are environment friendly as stated bellow:

* Natural dyes are less toxic, less polluting, less health hazardous, non-carcinogenic and non-poisonous.

* They are harmonizing colours, gentle, soft and subtle, and create a restful effect. Above all, they are environment friendly and can be recycled after use.

* High diversity of rich and complex natural dye colour.

* Different colours go well together and rarely clash.

* Beauty of the results when using natural dyes.

* Excitement of unexpected results.

* Satisfaction of growing your own dye herbs.

* Self-sufficiency if you grow your own herbs for herbs dyes.

* Not dependent on non-renewable materials.

* Allow for endless experimentation.

* Allow for replication of ancient techniques.

* Mature with age when exposed to sunlight and normal use, developing a patina as colours mellow.

2.2.4 *Disadvantages of natural dyes.*

Siva (2007) explains that there are some limitations in extracting natural dyes as well. He mentions tedious extraction of colouring component from the materials, low colour yield and long time of extraction; make the cost of dyeing with natural dyes considerably higher than with synthetic dyes. Some of the natural dyes are fugitive and need a mordant for enhancement of their fastness properties. Also,

there are problems like difficulty in the collection of herbs, lack of standardization, lack of availability of precise technical knowledge of extracting and dyeing technique and species availability.

Tyrian purple is obtained from the rare Mediterranean molluse murex brandavis. In order to obtain 14g of the dye about 1200 molluses are needed.

Furthermore, it requires the following:

* Large quantities in comparison to chemical dyes. However, many dyes are now sold as natural dye extracts which does not require large quantities, but, small amount of conhineal, brazo wood, and logwood that dye large amount of fabric.

* Longer time is required for natural dyeing. However, if the dyers are using the sun as an energy source, it reduces the time for dyeing with natural dye extracts which are quick to use.

* Natural dyeing may seems costly – but the main cost is the dyer's time; if researcher grows his own dye-yielding herbs, it becomes cost-effective.

The "imperfection" in the colour produced with natural dyes create a depth that is marvelous. The colour creats illusion of singing, to reflect the light so much more deeply, and using natural materials is one more way to become close to mother earth.

2.3 *Effect of geographical environment on colours by dye-yielding herbs:*

There wereeffects of geographical environment on colours by dye-yielding herbs. Wild Mint (2009) gave evidence that every kind of herb represent a product of a geographical environment, the quality of which depends on

climate, soil, water and other conditions. Therefore, herbs in Western part of Nigeria might produce better dyes than in the Northern parts of Nigeria or vice versa, because of the soil contents, rainfall, and weather, etcetera.

2.4 Equipment, materials and facilities for natural dye extraction:

Wangboje (1985) reiterates the need for experimental research into local resources using as much local materials as possible, in order not only to meet the challenges posed on dwindling resources, but, also, to ensure its relevance to the needs of the society where they live. Gwarzo and Alabi (2009) saw the importance of dyed fabric as it is used to beautify dressing and as they used dye fabric to decorate homes and environments. Emu (1984) states that tie-dyeing is a method of tying or stitching fabric together, preventing the absorption of dyes in certain areas of the fabric when dipping it into dye pot.

George (1979) mentioned the sources of colours as herbs, mineral, and earth. He says that indigo, green, red, purple and yellow are from herb sources.

(i) Indigo – this colour is gotten from the West-African herb, Lonchocarpus Syanescens' otherwise known as *Elu* in Yoruba. It is used for dyeing with textile material such as the Yoruba tie and dye known as *Adire*.

(ii) Green – this is from the leaves of Byrsocarpus Coccineus (*"Omoje in Yoruba)*. Also, it is got from the leaf of the Caladium Lily (*Eje Jesu "in Yoruba)* and Cocoyam.

Joanne (1987) said Nupe women, however, use leaves from an indigo tree and prepare their dye in pots, similar to the practice of Yoruba women in the south. While in the South, Yoruba women placed indigo cakes in a pot which is underneath another perforated pot which contains ash. Water is poured into the top filter through the perforated holes into the bottom pot and caused fermentation to occur slowly. Bliss (1980) found out that to extract dye from herbs, the dye materials ought to be covered with water and brought to simmer state (*180^0F – 190^0F) or (64^0 C – 70^0C).* This is done for about one hour. According to Joanne, (*1987)*, water can be added later if it is discovered that too much evaporation occurs. She believes that one pound *(0.5 kilogram)* of wool yarn is appropriate to obtain reasonable shades of colour. After the dye has simmered for one hour she reports, the dye is allowed to cool with the herbs in it before straining out the herb materials.

2.5 *Methods/procedures for natural dye extraction:*

Krishnamurthy *et al*., (2002) Gwari (2008) explains that dyes might have been discovered accidentally, but their use has become so much part of man's customs that it is difficult to imagine a modern world without dyes. The art of dyeing spread widely as civilization advanced. Siva (*2007)* stated that primitive dyeing techniques included sticking herbs to fabric or rubbing crushed pigment into cloth. The methods became more sophisticated with time and techniques using natural dyes from crushed fruits, berries and other herbs, which were boiled into fabric and which gave light and water fastness, were developed. Some of the well-known ancient dyes include

madder, a red dye made from the roots of the *Rabia tinctorum L.*, yellow from the stigma of the Saffron plant *(crocus Sativus L.,*)

Gwari (2006) in Nkeonye and Bello (1987) agreed that for fabric to be *dyed* it should be hygroscopic, that is having the quality and capacity to absorb moisture and dye such a cellulose materials (*cotton*)

As a rule, natural dyes are extracted from herbs by pounding, shredding or cutting them up. Herbs parts are then placed in water and heated to a temperature just at the boiling point. Until the colour is added to mordant-saturated materials, the dye would then adhere to the fibre of the material Mordants help set colour permanently into fibre.

McRae et al. (1991) observed that, some mordants are very toxic; it is recommended that dyers use aluminum ammonium sulphate. This form of alum is commonly used as a picking agent. For example Native Americans use a number of naturally – occurring mordants which includes natural alum precipitated from some dry soils tannic acid from sumac (*berries, branches, or leaves*), dye made from wood ashes, urine, sheep manure were mixed with water and later smoked to be baked.

The similarities in production of dyes are listed by Gulrajani (2009) as follows:

(i) In the colour extraction process, the herbs must be sourced, crushed and pounded.

(ii) In the extraction of indigo dye, the indigo balls will be dissolved in water and filtered through indigo wood ash to obtain the products.

(iii) Most of the herb sources of other colours excluding indigo are boiled in water before colour can extracted. Some of these colour extracts need mordants.

2.6 Mordant requirements for herbs:

Dyes do not interact directly with the materials they are intended to colour. Natural dyes are not substantive and require a mordant to fix to the fabric and prevent the colour from either fading on exposure to light or bleeding during washing. These compounds bind the natural dyes to the fabrics. A mordant is an element which aids the chemical reaction that takes place between the dye and the fibre, so that the dye is absorbed.

All natural dyes need mordants to help them adhere to the fabric. Those that do not require mordants, include lichens and walnut hulls, as they have provided bond between the fabric and the dye. Gwari (1986) says that all dyes belong to one of the following two classes:

(i) Substantive dyes: This class gives colour directly to the fabric.

(ii) Adjective dyes: This class includes a greater number of dyes that require the use of mordant to bring out colour. Common mordants are alum (usually used with cream of tartar, which helps evenness and brightens slightly); iron (or copper) (which darken colours bringing out green shades); tin (usually

used with cream of tartar, which blooms or brightens colours, especially reds, oranges and yellows), and blue vitriol (which darken colours and brings out green shades).

There are three types of mordant. According to James (2000) these include:

Metallic mordants: Metal salts of aluminum, chromium, and iron, copper and tin are used.

Tannins: myrobalan and sumach commonly used in textile industry;

Oil mordants: these are mainly used in dyeing Turkey red colour from madder. The main function of the oil mordant is to form a complex or solid bond with alum used as the main mordant.

2.7 Precautionary measures of synthetic dyes:

Research has shown that synthetic dyes are suspected to release harmful chemicals which are allergic, carcinogenic and detrimental to human health. Anonymous (2007a) says "the first dye that was not derived from natural sources is attributed to William Henry Per'kin in 1956. An exhibit explains Perkin's discovery Mauve, or mauveine, an intense purple dye. Today, 8,000 types of synthetic dyes are manufactured.

Anonymous (2007a) noticed that people are sensitive to pesticides, cosmetics, paints and perfumes, which result in heart palpitation, extreme muscle weakness, vomiting and outburst of crying. And this can lead to a mental disorder – Schizophrenia.

Furthermore, Anonymous (2004) Pushpa and Sengupha (1992) stated that modern technological developments have brought with them, a vast multifold increase in the kind of hazards to which workers are being exposed. The dangers are frequently so specialized that no single type of clothing will be adequate for work outside the normal routine. Anonymous (2002) stated that studies in the 1970s suggested that certain synthetic dyes cause cancer. As these potential health hazards became known, natural dyes began making a comeback in patronage. The nation of Peru, for instance, now produces some 85 percent of the world's supply of cochineal. Anonymous (2000) says that most of the products release low level of potentially harmful chemicals, such as formaldehyde, into the recycled air. Rugs add to the problem by absorbing various cleaners and solvents and releasing them over a long period of time.

These symptoms generally disappear when the affected people leave that environment. In addition, Anonymous (2007b) further informs that in some cases

"patients may develop multiple chemical sensitivities" as examined by the British medical journal the Lancet. Dr. Claudia Miller in Awake Journal in which it was revealed that more than a dozen countries reported psychological problems after well-identified chemical exposures, which may have resulted as either toxic exposure or sick building (syndrome).

Chemical hazard exposure can lead to psychological problems and many doctors believe that most of the workers in textile industries can contact this problem as result synthetic dye exposure as reported by Dr. Claudia Miller and cited in

Anonymous (2004). He further reports that the reverse is also true that psychological problems, can contribute to the development of chemical sensitivities. To prove this right, Anonymous (2007b) states that in United State, surveys among various groups of population suggested that between 15 and 37 percent of the population consider their people especially sensitive or allergic to common chemicals and chemical odors, such as, fresh dye, new rug, and perfumes. Above products are common in Nigerian environment, especially synthetic dyes.

In another development, Anonymous (1998) noticed that they have to consider the risk of injury or death. The situation is much the same with synthetic dyes – their disadvantages. The MTBE (Methyl teriary butyl ether) clears the air in many cities in United States. Some synthetic dyes have been banned and taken off the market, altogether because of the damage they cause to the environment and human health. The *World Book* Encyclopedia in Anonymous (2004) states that even the most careful testing "cannot always reveal the possibility that a product might produce an unexpected harmful effect."

Some areas in India experienced severe water pollution after thousand of idols were dropped into stream and lakes. In order to limit water pollution, the residents states "Magazine Down to Earth" quoted in Anonymous (2004) mentions that India and everywhere, maker of idols or manufacture materials revert to using traditional dyes instead of synthetic paints, otherwise" says the magazine, the rivers that Hindus Worship or manufacturing materials such as fabric may be poisoned by the idols they worship or the manufactured materials.

In another related literature review Anonymous (2007b) explained that a survey of more than 4,300 youths between the ages 12 and 24 indicate that today's youth's are interested in traditional values, such as using traditional resources. The Globe and Mail Augus Reid Group, agreed that the most important value is keeping the natural resources made from herb dyes. They also agreed that there was not an environmental problem when manufacturing materials were painted with colours made from flowers or vegetable dyes. However, when synthetic dyes made with heavy metals and carcinogens were introduced in some areas of India and other part of the world experienced severe pollution and it resulted in severe health hazards to mankind in general.

After the accidental synthesis of Maurine by Perkin in Germany in 1956 and its subsequent commercialization, coal tar dyes began to compete with natural dyes. Singh (2001) stated that the advent of synthetic dyes caused rapid decline in the use of natural dyes, which were completely replaced by the former within a century.

However, research has shown that synthetic dyes are suspected to release harmful chemicals that are allergic, carcinogenic and detrimental to human health. Ironically, Germany that discovered 920 synthetic dyes became the first country to ban certain 920 synthetic dyes in 1996, (Singh and Singh 2002)

Conclusively, Siva (2007) Anonymous (2008) explained that the disadvanges of synthetic dyes are more than the advantages, and therefore requested that dyers should be more comfortable in making use of natural dyes. He listed the problems of synthetic dyes as:

* lack of availability of different dye that are not resisted to fabric but, fade more than natural dyes.

* chemical dyes fade more than nature dyes

* affect dyer's health.

Dyers require the following:

* quick results that can be repeated accurately.

* more calculation and precise measurement.

* skill to find dyes that combine together effectively.

* dyes often look bright and garish.

CHAPTER THREE

3.0 Methodology

3. 1 Introduction

This chapter deals with all methods and procedures that were adopted in accomplishing the set objectives. It includes the materials, samples for the study, the field works and laboratory experiments. This process includes harvesting of herbs, and identification of the raw materials. It also involves the fabric preparation, extraction procedures and the amount of dyes that would possibly be extracted, mordanting, dyeing procedures and colour fastness tests which involved wash fastness test, light fastness test, and perspiration test that were analyzed. The procedures for each herbs extraction and dyeing experiment in the laboratory with all apparatus/equipment used are shown in Plate 3.1 -3.12 appendix

These dyes that were extracted and tested on the cellulosic cotton fabrics were analyzed in the following laboratories:

a) The Department of Textile Science and Technology Laboratory, A.B.U. Zaria for the processes of experimenting and applying the dye extracts on cotton fabric.
b) Department of Pharmacognosy and Drug Development, Pharmacognosy Laboratory, A.B.U., Zaria, for extraction of dyes from herbs.
c) National Research Institute for Chemical Technology, Zaria, for the UV spectral scan analysis to determine the amount of dye absorbance.
d) African Textiles manufacturers Ltd., at Kano, for the colour fastness tests.

Cotton fabric was chosen and examined because of its strong affinity for vegetable dyes as well as its availability in local markets in Lokoja, Nigeria

The following instruments and procedures were employed: Discussion and Interview, Experiment and Analysis. The study was designed in two phases consisting of field works and laboratory experiments.

3.2 Materials and Methods:

The materials comprise herbs that need to be documented to prevent them from being lost forever as the herbs properly employed and available as dye- yielding herbs in Kogi State. (See fig.3.1) appendix presents the map of Kogi State showing area of study Lokoja. Materials of the study consist also of various parts of leaves, seeds, roots, and bark, of indigenous dyeing herbs from Lokoja. The three herbs consist of eight branches listed below:

- **Parkia biglobosa** (*Botanical name),* **African Locust bean**
- **Tectona grandis** (Botanical name) **Teak** (English)
- **Terminalia glaucescens** (Botanical names) **White afara** (English)

The following eight parts of the various herbs will be examined namely:

i Parkia biglobosa *(Locust bean)*: **leave, seeds, and bark.**

ii Tectona grandis (*Teak*): **Leave, bark.**

iii Terminalia glaucescens: (*White afara)***leave, roots, bark.**

3.2.1 *Specimens of the study:*

The test sample for this study consists Parkia biglobosa *(Jaq.) R,* Tectona grandis *lin. F,* and terminalia glaucescens *Plaanch.,* in Lokoja Local Government Area of Kogi State. The choice of study was guided by homogeneity and similarities in geographical location, their climate condition and the availability of the dye raw materials in kogi state. The following institutions contributed to the development and growth of those herbs, namely; Kogi State Ministry of agriculture, Department of Forestry, in Lokoja. The Department of Horticulture, Landscape, and Technology, College of Agriculture, Ahmadu Bello University, Kabba,

3.2.2 Conditioning of specimens:

The herbs were conditioned to a room temperature (25^0C) relative humidity (RH) 65$\pm$ 2^0C and temperature (T) 21$\pm$ 2^0C exposed to standard laboratory temperature of not more than 27^0C-30^0C for 24hrs. Thereafter, it was crushed and spread indoors to dry. Distilled water was gotten from the Chemistry labouratory A.B.U. Zaria. White cotton fabric was purchased easily at International Market Lokoja, Kogi State. Six selected mordants or dye assistants that were used in dyeing processes included the following: Local alum, wood ash, Sodium chloride (Common salt), Iron II sulphate, Copper II sulphate, Potassium di-chromate. The following solvents: Distilled

water Ethanol (C_2H_5OH), Toluene, Acetone and Dimethyl formamide (DMF) used for solubility study to determine the best solvent for extraction.

After the extraction the shimadzu UV-Vis Spectrophotometer model Uv-2550 was used to scan the results for the absorbance tests of the extract from the herbs. Then the dyed fabric was subjected to wash fastness test with the machine known as machine wheel or (Washing Fastness Tester – Launder meter) for washing dyed fabrics. Thereafter light and perspiration tests were carried out with alkaline, acidic solution and humidity control according to ISO standard. All the tests were graded with grey scales instrument called digi WASH SS^{TM}.

3.2.3 *Description of the Herbs.*

Each specie collected, was arranged alphabetically and was provided with correct botanical name, available local name brief description, flowering and fruiting season; uses and method of propagation.

i **Family:** LEGUMINUSAE –MIMOSACEAE.

Botanical name: Parkia biglobosa *(Jaq.) R*

Common names: African locust bean.

Local names: Dorawa *(Hausa)*, Iru (*Yoruba)*

Description:Tree up to 20m high, not buttressed with low branches that are low are bearing a wide spreading crown,

deciduous, flowering white leafless. Leaflet 35- 65cm on each side of the rachis of each pinna, corolla lobes very short, leaves rachis grayish and leave alternate.

Parts used: leaf, bark and seed.

Constituents: Tannins, Saponnins and Alkaloids, Cyanogenetic glycoside, Saponin.

ii **Family:** VERBENACEAE.

Botanical name: Tectona grandis*linn. F.*

Common name: Teak.

Local name: Labadiya (*Hausa),* Igi Oba *(Yoruba,* Shinasi *(Kabba)*

Description: A medium size tree 25m to 30m tall and approximately1m diameter at breast height (dbh), under favorable site conditions. It may grow up to height of 50m with a dbh of 2m. Leaves are simple ovate to round. Flowers are small bulb numerous and white. Fruits are round, hard and about 1cm in diameter, which turns brown when it is mature. A fruit contains 1 to 4 Seeds.

Parts to be used: Leaf, root

Constituents:

iii. **Family:** COMBRETACEAE.

Botanical name: Terminalia glaucescens *Planch.*

Common name: white Afara

Local names: Baushe *(Hausa)*

Idi- apata / Idi- odan *(Yoruba)*

Description: Tree 20m high with dark grey deeply fissured bark.

Parts used: Bark, root and fruit.

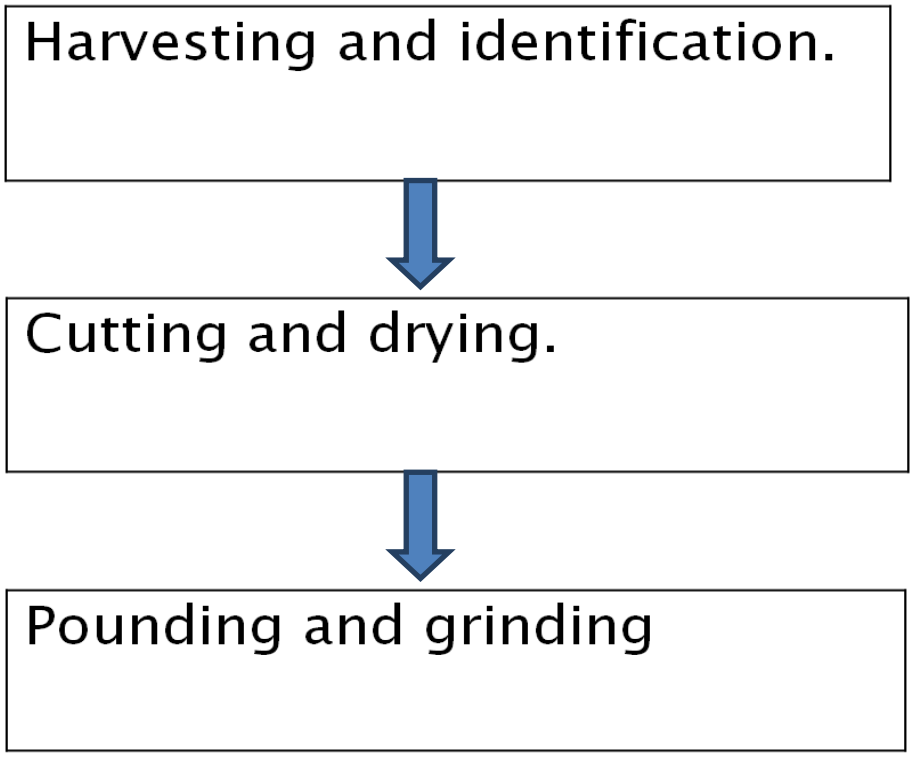
Constituent: Alkaloid. (See table 3.1)

3.3 The Field works and laboratory experiments:

This study was divided into phases one and two respectively.

(a) Phase one- Field work:

Field works procedures:



3.3.1 *Collection of raw materials:*

The herbs served as main raw materials which are derived from the branches of leaves, roots, and barks of the above herbs as mentioned and described. These were gotten in Forrest reserves at Lokoja, and the cotton fabric was purchased at the International Market in Lokoja. For this reason, Mosaku (1987) suggested five stages which dye herb can be introduced in'to the fabric:

- Collection of raw materials (dye herbs.)
- Identification of the raw materials collected.

- Extraction of dyes from dye herbs.
- Experimentation with the dye extracts.
- Colour fastness test.

The above methodwere adopted for the purpose of this study.

3.3.2 *Procedures, time of collection and treatment of raw materials:*

The cotton material that was used for the laboratory tests was first identified by burning test. Extraction of dyes and the fastness tests was carried out in the laboratory. Collection of dye materials, differ according to climatic conditions and geographical locations. Nyam (1985) and Mike (2010) gave the time of the year when the dye raw material should be collected and the growing condition of the dye yielding herbs determines the quantity and quality of dye substances that most vegetable material contains. For this reason, therefore the appropriate time for collecting various dye material is as follows:

(i) ***Roots:*** Roots were collected during the dry season (*October to March),* when the sap is up.

(ii) **The bark of the plant:** The bark was collected same as above.

(iii) **Stem, Seeds and Leaves:** This group was collected late in the season of growth as the herb part is still in physical condition during the period of

(*October to March*).

The following procedures were used for collection of herbs in this work as follows:

The herbs were conditioned to a room temperature (20^0C) relative humidity (RH) 65$\pm$ 2^0C and temperature (T) 21$\pm$ 2^0C expose to standard laboratory temperature of not more than 27^0C-30^0C for 24hrs. The harvesting of the herbs was as follows:

Step l: Harvesting of herbs from the forest reserved area of mount Patti hill in Lokoja and environment of Kogi State. (Plate 3.1)

Step II: The chopping, crushing and pounding of herbs collected, was by the process of drying gradually at room temperature (of about 20^0C). It was dried in an oven condition of 20^0C. These were stored in containers or polythene bags for subsequent experiments. (Plate 3.2 – 3.4)

Step lll: The herbs were dried at room temperature (of about 20^0C) for two weeks, (14days), in order to allow the process of pounding with mortar/pestle and the use of grounding mill into crumbing or powder form. Further, the crumb forms were kept in the oven at 20^0 c for 24 hours.

Step IV: Pictures, diagrams, tables, figures, and some of the tools and equipment used for the study were shown at the appropriate section of the text for proper documentation. The detailed analysis and progressive results of the study is presented in chapter four of the

study. The practical works were carried out along with technical assistance. The dried materials were referred to as “herbs”.

(b) Phase two- Laboratory experiments:

3.4 Experimental procedures:

Labouratory experiments conducted were after the field works, the herbs were taken into the laboratories for identification, dyes extraction and other experiments such as wash fastness, light fastness and perspiration fastness tests. The equipment, materials and reagents of various tests are listed below:

- ***Materials and equipment used:***

1 Small pieces of fabric

2 Filter paper.

3 Organic solvent and solutions

4 Stirring rod.

5 Mortar.

6 Oven.

7 Evaporating dishes.

8 10 x150 m test- tube (pyrex).

9 Measuring cylinder.

10 Weighing balance.

11 Boiling water bath.

12 Percolator: For soaking the crumb (crude dye)

13 Dye herb crumbs.

14 Electric machine:

15 Funnel with stand and filter paper.

16 Water bath: For boiling the solvent with the dye herb crumbs.

17 Rubber Tubes

18 Solvents: Distilled water, Ethanol (c_2H_5OH), Toluene, Acetone and DFM

19 The UV-Vis Spectrophotometer with model Uv- 2550 shimadzu

20 Six selected mordants or dye assistants used in dyeing Processes were:

Local alum, wood ash, Sodium chloride (Common salt), Iron II sulphate, Copper II sulphate, Potassium di-chromate.

21 solvent added included: Acetone (CH_6)2co, DFM, Ethanol (C_2H_5OH), Toluene (CH_3)2CO, and Distilled water respectively

22 Wash wheel or launder O-meter (digi WASH SS^{TM})

23 Thread.

24 Needle.

25 Volumetric flask.

26 Soap solution: Sodium Oleate powder, distilled water.

27 Grey scales: The instrument used for this is called digi WASH SS^{TM} (Wash Fastness Tester – Launder meter). To determine the cc and cs grading for wash fastness, light fastness and perspiration fastness tests

28 digi – light^{TM} (light fastness tester) Standard MBTL fading (Mercury tungsten lamp)

29 Rectangular cells.

30 Double slot mask (2/3 area exposed)

31 Single slot mask (1/3, 1/4, 2/4, 3/4)

32 Cylindrical cells.

33 Control fluid filler.

34 Stainless steel clamps.

35 Balls

36 Voltage stabilizer-1K.V.A.

37 Blue wool light fastness standard.

38 Perspiration and water fastness test, the following solutions were introduced:

- **Standard alkaline solution:**

I L-Histiline Monohydrochloride Monohydrate ($C_6HgO_2N_3HCIH_2O$).

ii Sodium Chloride (NaCl)

iii Disodium Hydrogen Orthophosphate Dihydrate

($Na_2HPO_4.2H_2O$).

iv Distilled water.

v Volumetric flask.

- **Standard acidic Solution:**

i L-Histidine Monohydrocloride Monohydrate

($C_6H_6O_2N_2HCl.H_2O$)

ii Sodium Chloride (NaCl).

iii Sodium Dihydrogen Orthophosphate Dihydrate.

iv Distilled water.

v Volumetric flask.

✓ **Testing:**

The experiment involved the following tests: Identification of raw materials, colour tests and dye characteristics, dye tests, solubility tests, dye extractions, wash fastness and colour fastness tests. The following procedures were followed.

3.4.1 *Identification of raw materials:*

Identification of the dye materials was carried out before the collection of the raw materials. Siva (2007) stated that to indentify

dye herbs, the botanical, local, and common name must be established. This was obtained at the Department of Pharmacognosy and Drug Development, Ahmadu Bello University, Zaria, the Department of Horticulture, Landscape, and Technology, and Kogi State Ministry of Agriculture, Department of Forest, Lokoja, Kogi State for confirmation. Dye materials were extracted from fresh herbs and some of them dried them in a well ventilated space to preserve them. They are retained in the space until they are completely dry.

3.4.2 *Colour test and dye characteristics:*

Adeleye (2003) defined dyes as substances used to colour materials permanently in a manner that the colour imparted is regarded as a part of the materials and not merely adhered to the surface as painting. This definition clears the point that not all dye substances have the attributes of a dye. Islam (2000) mentioned that dye stuff must have the property of fixing and attaching itself to the material to be dyed. In this direction, the materials collected will be subjected to colour dye characteristics test recalling the above attributes.

Thereafter, small quantities of the crumbs (5gm) was weighed and placed inside small evaporating dishes and some drops of solvent was added which included: Acetone $(CH_6)_2CO$, DFM, Ethanol

(C_2H_5OH), Toluene $(CH_3)_2CO$, and Distilled water respectively. The colourless solvents were poured on the herb crumbs that extracted the dyes. The changes of the solvent were observe at 30mins interval. The resulting solutions were used to dye cotton fabric.

3.4.3 *Dye test:*

In order to be sure that dye product were applied on the fabric, test to characterize the dye produced was carried out. Pieces of filter paper and fabrics were immersed half way into the solvents as mentioned above for five minutes and immediately were removed. The filter papers and the fabrics were rinsed thoroughly with running tap water, spread in the room temperature and left to dry. As the drying was completed, it was observed that some stains were left on the materials used for testing. The dyes on the materials were very pale.

3.5 Solubility test:

According to Gwari (1989) solubility could be defined as the amount of the solute necessary to saturate a certain amount of solvent at a given temperature and pressure. The speed at which the operation takes place mostly depends on the extent of solid- solvent surface area interaction, the compatibility and the speed of mixing or stirring. For the solubility test in this study, 5g of dye material was dissolved in 100mls of solvent. The objective was to find out the appropriate

solvent that can be employed to dissolve the crude dye stuff, in order to aid the extraction processes. The extracting solvent was the substance required for selection without extracting other impurities. The herbs were conditioned at room temperature (20^0C) and relative humidity (RH) 65$\pm$ 2% (standard laboratory condition) for 24hrs.

Step1: Powder form of the crude dye was obtained from already indentified eight herbs species. 1gm of each was placed in clean dye test tubes.

Step2: 20mls of each of the solvent was added to each of the 8 test tubes and then shaken vigorously for five minutes.

Step3: The 8 test tubes were labeled as follows: TBL, TBT, TBW, TLL, TLT, TLW, TRW and TSL, to reflect the eight samples. The procedure was repeated. The boiled solution was observed to see how the solvent reacted with the dyes stuff. The solvents are Acetone $(CH_6)2CO$, DFM, Ethanol (C_2H_5OH), Toluene $(CH_3)2CO$, and Distilled water. The result of the observation is presented in a table in chapter four.

Step 4: In carrying out these tests, dye stuff weight, solvent volume and other environmental conditions were kept constant.

3.6 Dye extraction and quantity available for extraction.

The dye was prepared by boiling the crushed powder with water, but sometimes it was left to steep in cold water. The solution then obtained was used to dye coarse cotton fabrics. Alum was used as a mordant. Flowers of Butea Monosperma (Lam) Taubert yield an orange-coloured dye, which was not fast and was easily washed away. For the purpose of colouring, the material is steeped in a hot or cold decoction of the flowers. A more permanent colour was produced either by first preparing the cloth with alum and wood ash, or by adding these substances to the dye bath.

The extraction process was the separation method that involves the removal or transfer of a substance from one material phase into a second phase.

When the phases are solid and liquid, according to Gwari (1989) the method is solid- liquid extraction. The general method of vegetable dye extraction has been the solid- liquid extraction. This includes the following:

- **Methods of dye extraction that was employed in this study:**

I. Maceration method known as Cool extraction.
II. Soxhlet extraction technique.
III. Extraction method of boiling with water.

i***Maceration method or cool extraction:***

Rita (2009) stated that the length of time for soaking dye herbs depend on the type and condition of the herb. The observation of Mustard (1997), Wildmit (2009) were that a dry bark or roots after a day's soaking might still need about three hours boiling. Casstino (1975), Simon (2000) stressed the fact that any category of herbs should be soaked at least over- night. The method of soaking fibrous herbs for three days was adopted in this study. This indicates that each of the eight crude dyes were soaked for three days respectively, i.e. crumbs were soaked for three days for the first extraction without boiling and six days for the second and nine days for the third extraction until the crude dyes were extracted. After each period of soaking the solvent was filtered away from the crumb particles using a funnel placed on four and made in a three dimensional cone shape placed in the glass funnel on the stand. The solvent which was initially colourless but, it became coloured when soaked with the dye herb.See plate 3.5b in appendix.

- **Methods:**

Step1: This indicates that each of the eight crude dyes was soaked for three days respectively, i.e. crumbs were soaked for three days for the first extraction without boiling and six days for the second and nine days for the third extraction until the crude dyes are extracted.

Step2: After each period of soaking the solvent was filtered away from the crumb particles using a funnel placed on four and made in a three

dimensional cone shape placed in the glass funnel on the stand. The solvent which was initially colourless and became coloured when soaked with the dye herb.

Step3: The coloured solvent was poured on the filter paper through the funnel placed on the stand and the filtered extracted dripped into the beaker.

Step 4: After filtration a fine fluffy powder were left on the body of the filter paper. When the solvent was filtered from the crumbs there were still some colours that were left in the herb substance. This was determined by the intensity of the first extract. The solvent was measured for each crude herb to be extracted.

The result of the extract that was gotten after this was processed from a highly concentrated dye solution. The concentrated dye solution was measured, labeled and preserved for subsequent experiment. The result was presented in chapter four.

ii. Soxhlet extraction technique:

Soxhlet extraction was hot extraction method. This method was employed mainly to obtain pure samples for testing purposes.

The only solvent used was hot Ethanol (C_2H_5OH) for this technique based on solubility study employed.

Stage 1: 25 gms of each of the crude dye derived from TBL, TBT, TBW, TLL, TLT, TLW, TRW and TSL, were placed in the porous thimbles (A) one after the other. This was placed into the inner tube of the soxhlet apparatus. The apparatus were fitted to round bottomed flask, (c) containing 250mls of solvent, distilled water and Ethanol (C_2H_5OH) and supported in the thermostatically paraffin bath, (K). The reflux condenser, (D), was fitted on the apparatus. Seven circles ran each as in case of the TBL, TBT, TBW, TLL, TLT, TLW, TRW and TSL.This has five circles each. (see plate 3.5a and fig.3.2.1 adopted).

The time set for the extraction ranges from 8 -12 hours, depending on running circles of the soxhlet system. With these processes, it took each of the part of herbs mentioned above to run 12 hours respectively. Locus bean seeds and white afara took 8 hours to have the five circles respectively based on their quick extraction. The solvents that were used ware distilled water and Ethanol (C_2H_5OH) based on solubility test.

Stage 2: The second stage of this procedure was that, the extracted dye solutions was reserved as samples for measurement and subsequent dyeing. For the determination of the quantity of dyes available for extraction, the extracted dye solutions obtained from the herbs were poured in the dish and placed on the thermostatically control paraffin oil bath where

the solutions obtained from them were evapourated respectively and allowed to dry naturally. These solid dyes were the amount of dyes available from each extraction. Therefore, the results for the various weights that exist according to the quantities of each dye are presented in chapter four.

iii Extraction by boiling with water:

Step 1: The beaker was rinsed and the dye substance crumb measured into it. The materials for extraction were measured into beaker with the already measured crumb.

Step 2: The soaked substance was left for a day following the suggestion of Simon, (2000) confirming that after a day's soaking, it would be placed on the hot plate to boil for three hours, as Wildmit (2009), ascertained that a dry bark is to be soaked for a day and still require three hours boiling as recommended by him.

Step 3: The dye bath was left to cool and after which the residue was removed. The substances became the dyeing material. Only two of the herbs were tried with this method.

3.6.1 *The UV spectral scan analysis test:*

The maximum absorbance for each extracted dye(various herbs)was determined using UVspectraphotometer available atNational Research Institute for Chemical Technology, Zaria, (NARICT).

See fig.3.3.1 – 3.3. in appendix.

3.6.2 *Powder technology of dye:*

The powder technology of dye is a way of preservation of dye herbs and dye bath in order to avoid damp. The extracted dye was kept in a room temperature for a day and in an oven for one day, so that all moisture was removed. The oven temperature was very low (20^0C) to dehydrate the specimen further. The oven door is opened half way as the samples were been dried as recommended by Islam (2000). He further said that before putting them in the oven, proper labeling is very important. After a day, it should be wrapped with newspaper and kept in a polythene sheet to avoid moisture absorbance from the surrounding.

Step 1: The solvent in dye bath evaporated, this process separated the

solvent from the dye substances. The filtered solvent was placed in the bath for four hours during which, the solvent continued to evaporate gradually until it was completely evaporated. The evaporation became necessary as water bath kept boiling leaving the

dye stuff in the glass beaker. The residue in the beaker was in crystal form. See plate 3.6 and fig. 3.2.2, adopted for this study.

Step 2: To further dry the dye extract it was kept in the desiccators, adding some drying agent (H_2SO_4 .Conc Sulphuric acid). The crystal was kept in the desiccator; some sodium Benzoate will be added as preserving agent.

Step 3: This sodium Benzoate was mixed with the dye extract to avoid dye lose as stated by Mustard (1997) and Bin (1985). The shinning crystal obtained was stored in an air tight bottle. The same procedure was used for water extraction. The difference was that the evaporation of water from dye may take a week or more; this depends on the quality of the dye solution. A point of caution was given by New Feeds (2006) that too much boiling can destroy dye content in a dye herb. After boiling for four hours it was kept in the desiccators for another three days with concentrated hydrochloric acid as the drying agent and was mixed with sodium benzoate as the drying agent for colour loss.

Some of this preservation was adopted in this study for alkaline solution extract. This method of preservation eliminated the tedious process of pounding, soaking and boiling that would be necessary, if only the dye herbs were preserved. It was confirmed that for any singular prescribed medium of dyeing operation, dissolving the crystalline dye in the prescribed medium

was the only operation required to be neat, simple and fast (Adeleye and Opia 2006).

3.7 Mordanting procedures:

Siva (2007) explained that before dyeing fabrics with some natural dyes, such fabrics should undergo certain special preparation as mordanting in order to allow the dye fix onto the fabric. Wild (2009) posits that almost all the natural dyes with the exception of few have no dyeing energy, except when the dye stuff are used in conjunction with mordants. Gwari (1989) provided the three methods that mordants can be applied on fabric as seen below:

1. Pre-mordanting or mordanting before: This is the process where the substrate is mordanted prior to dyeing of the fabric.
2. The fabric, mordant and the dye were together in same dye bath.
3. The dyeing of the fabric came up first, then followed by mordanting;

The first suggestion above was adopted in this study, as pre- mordanting or mordanting before was found appropriate in this study. All the related literatures confirmed that pre-mordanting before were found to be the most appropriate method found in dyeing any vegetable or natural herbs because of its non-dulling effect when applied onto cotton fabric. Although, the main reason for non-adding of any mordant or non mordant treatment was to determine either the dye is substantive or adjective.

Step 1: The cotton fabric purchased at Lokoja International Market, Lokoja Kogi State, was burnt as related literatures indicated that the cotton must burn quickly without melting leaving a little ash with a resemblance like that of paper. This is to confirm that it is a cotton fabric.

Step 2: The cotton fabric tested for mordanting in this study was desized, scoured and bleached. It is known either as poplin or "Dan Funtua".

Step 3: The mordanting procedures was carried out at room and boiling temperature both cold and hot, whereas the time in which the samples stayed in the mordant solution was varied from 20mins to 2hours. The mordanting was at constant temperature and with varying times taken on the processes.

Step 4: The uses of six different types of mordants were experimented namely: Local alum (potash), wood ash, Sodium chloride (Common salt), Iron II sulphate, Copper II sulphate, Potassium di-chromate.

i. Hot mordanting:

Step 1: 5gms by weight of the selected mordant: Local alum (potash), wood ash, Sodium chloride (Common salt), Iron II sulphate, Copper II sulphate,

Potassium di-chromate, were dissolved in 100mls of distilled water with 5% solutions.

Step 2: The samples were put into hot water for 20mins first, in order to wet the fabric as recommended by Mosaku (1987) as it helps in achieving level of mordanting. Later the mordants were introduced on the wetted cotton fabric one after the other. The temperature was raised to 100^0C and maintained while the stirring were at 10mins interval.

Step 3: The samples were tested on time variations of 20mins, 40mins, 60mins, 80mins and 120mins respectively.

Step 4: After the samples stayed in the mordant solutions for the required period. The mordanting fabrics were removed, squeezed and rinsed in water. These samples were kept for subsequent dyeing.

ii The cold water mordanting:

Step 1: Cold distilled water mordanting had the same recipe, except that the dyeing was done at reduced temperature of 20^0C for four hours.

Step 2: In order to dissolve the mordant, the solution was stirred up for Four hours. After the samples have stayed in the mordant solution for the required time, they were removed, squeezed and rinsed in water for the subsequent dying.

3.7.1 *Dyeing procedures.*

The dyeing procedure adopted for study utilization different measurements of materials that were tested for dyeing in the experiments. The final measurement that was adopted included the following:

i. 0.5grams of fabric.

ii. 5 grams of dye.

iii. Organic solvent 10ml. and 90ml of distilled water ml. or 2 percent of each of the solvent solution.

iv. 800ml Stainless glass beaker**.** (See plate 3.7 in appendix).

3.7.2 *Standard preparation of dye solution:*

A stock was prepared by dissolving 1gm of dry powder in 100ml of distilled water. To calculate the quantities required for any experiment, the following were used.

No. of ml of stock solution required = $\frac{W \times P}{C}$

Where W =weight (in grams) of sample of fabric was dyed.

P = Percentage of dye or assistant used.

C = concentration of stock solution. Gile (1974)

❖ ***Method for preparation of dye bath:***

The already prepared powder dye was dissolved in a beaker with 10ml of water, at 100^0C . After the dye has been dissolved in 10ml of organic solvent solution, then 90 ml of water was added to make the 100ml specified measurement for the dye

bath respectively. This dye bath formed all the base measurement for the entire eight dye specimen used in this study.

This dyeing process employed both hot and cold dye bath in this study:

(i) Hot dyeing:

Mosaku (1987) suggested that at 10mins of dyeing half tea spoon of cream of tartar should be added to aid level dyeing. He stated further that in another 20mins of dyeing half tea spoon of glauber salt is added into the dye bath to assist in exhausting the dye, that it minimized stirring regularly. She further explained that cream of tartar in glauber salt needed not to be pre-dissolved before adding dye bath in cold water dyeing. The above mordants were not used in this study, because of the difficulty in getting them. This study utilized 100ml dye solution prepared in a glass beaker weighing 800ml. The already mordanted fabric cotton were wet and immersed in the Luke warming dye bath for 20mins. The control time the fabric stayed in dye bath with vigorous stirring was 2hours. After the intervention of 10mins water was added when evapouration was fast taking place. Then rigorous stirring at 20mins interval was employed to aid in exhausting the dye. When the 2hrs stipulated as dyeing time was over the dye bath was removed from the hot plate. The fabric was left to cool in the dye bath before removing them accordingly. After this stage, the fabric removed, squeezed,

rinsed thoroughly in running tap water and dried under laboratory condition. Thereafter, the dyed fabric was removed for light and perspiration fastness test. (Plate 3.7 in appendix)

(ii) Cold dyeing:

Step 1: This was conducted the same way as the above, except that with the cold mordant that has absence of heat application. Water was not added, fabrics took long period of twelve hours as comparing to the ranging of time above. After that period, the fabric was removed, squeezed, rinsed thoroughly in running tap water and dried under laborator condition.

3.8. Colour fastness tests:

The main aim of dyeing was not only to apply colour on fabric, but to fix it in the fabric and achieve relative permanence. Thomson (1981) asserts that the principal fastness properties which all dyes must possess include: ability to withstand such daily activities as washing, light and perspiration. To support this, Bin (1985) says that the most important test for vegetable dye is light and washing for the reasons listed below:

- ✓ ***Testing natural dyes for fastness:***

(i) Obtain as many different fibres of materials as possible and dye each of this in the dye bath.

(ii) Then, obtain pieces of several fabrics and dye, in the same dye baths. Took note of whether, there were differences in dye obtains with a single fibre as compared to a piece of cloth.

(iii) Took samples of the dyed piece of cloth and fasten to a white cloth. Make up the solution of as many detergents, soap powder and soap as possible.

(iv) Cut the square of cloth into separate sections and immerse a piece of dyed cloth attached to its white piece into each solution. Make sure that the solution is the same temperature and that the pieces are put in the same temperature and that the pieces are put at the same time and taken out at the same time.

(v) Test for sun-light fastness by leaving pieces of dyed cloth near a window for several days. If possible cover half of each piece with paper before exposing, so that the degree of fading can be noticeable.

The colour fastness properties of these natural dyes were applied on fabric. In this study the resistance of a material that changes any of the dye characteristics or to the extent of transferring colourants to the adjacent white materials in touch. Therefore, colour fastness was rated either by loss of depth of dye in original sample or it was also expressed by staining scale,

i.e. the company's white material got tinted or stained by the dye of the original fabric.

However, among the various methods of test for dye fastnesses carried out on textiles fabrics at African Textile, Kano. These tests were carried out, in Line with the ISO test no 4 standard that was adopted. This study examined, wash fastness, light fastness, and perspiration fastness tests as the procedures for the 82 dye samples intended to produced as explained below;

(i) Wash fastness test:

In this test, Biodun, (1995) says fastness can be tested by washing a small amount of the naturally dyed yarn or fabric in hot water and soap along with some white cotton, wool yarn or fabric. If the y a r n or the fabric becomes stained with the dye, the dyer knew just how much the dye bleeds. with grey scales the instrument used for this was called digi WASH SS^{TM} (Wash Fastness Tester – Launder meter). It was used to determine the colour change (cc) andcolour stain (cs) grading for wash fastness, light fastness and perspiration fastness tests

Siva (2007) affirmed that study was conducted on colour fastness to washing with the use of ISO 3 method in a composite specimen was prepared by stitching two pieces of un-dyed fabric measuring 5cm x 10cm on the dyed fabric to be tested and dropped it into the steel containers of the wash-wheel after which the amount of soap solutions where poured into the steel container

according to the weight of the fabric. This was calculated according to the liquor ratio of 50: 1 of the soap solution containing 5g of soap and 2g of anhydrous sodium carbonate and heated to 60 ± 2^0c. He also mentioned that the dyed fabric was subjected to the above temperature for minutes and afterward rinsed on twice in the running tap water for 10 minutes to remove any surface colour. The stitched material was removed and then dried in the air. The change in colour of the uncovered portion of the fabric and the staining of the undyed fabric was assessed with the grey scale for shade changed and staining.

Furthermore, Ashis (2009) studied the light fastness and wash fastness of Christive (2007) and Siva (2007) under standard condition (50^0C) and also at 20^0C with a washing formulation used in conservation work for restoration of old textiles. Some dyes undergo marked changes in hue on washing due to the presence of even small amounts of alkali in washing mixtures, highlighting the necessity to know the pH of alkaline solutions used for the cleaning of textiles dyed with natural dyes. As a general rule, natural dyes show moderate wash fastness on cotton, as assessed by the ISO 4 test. (See plate 3.9). This study reported on the 82 dye samples.

Step 1**:** The samples in between the two pieces of adjacent fabric are sown along the four sides for better result.

Step 2: The test was carried out in wash wheel or launder O – meter.

The dyed cotton fabric sand witched between an un-dyed cutting of the same cotton fabric and another as stated in the standard are placed in washing solution at $95^0 \pm 2$C as the standard for test solution for fastness to washing of International Standard Organization (ISO4) (see table 3.2) in the steel jars of eight with tight fitting lids placed inside the machine name in wash wheel or launder O – meter.. These jars were rotated in a constant temperature water bath for the length of 30 mins per 1S0standards. (Plate 3.9, fig. 3.4)

Step 3: The cotton fabrics were taken out, rinsed and dried below 60^0C or at room temperature. The change in colour of the dyed fabric and standard fabric were compared with each order to determine the stain rating of colour change and colour stain with the two grey scales of rating based on ISO 2-5 standards. The result was presented in chapter four. The 1SO describes five tests for different degree of several washing by increasing temperature and time and addition of recipe of soap as presented in a table 3.2. This study adopted 1S0 test no 4 for the fabric wash fastness test.

Step 4: The washed is fabric dried under room condition.

Step 5: After grey scaling, the washing fabric was gum on the quality

control sheet for ISO standard grading at African Textile Manufacturer Ltd, Kano.

- ***Standard Soap Solution:***

0.5% Sodium Oleate solution as standard soap solution (as per ISO 4 standards). This was adopted as well.

- **Method of preparing the standard soap solution:**

(a) 100ml of distilled water was taken into a volumetric flask.

(b) 5gms of Sodium Oleate powder was kept in the water and dissolve it thoroughly by agitating or shaking the volumetric flask. That is standard soap solution for wash fastness test.

1. After the main bath is preconditioned, the temperature and time was chosen for ISO4 Test4 95$\pm$ 2^0C temperature and 30 mins timeas was stated for ISO test 4 see table.3.2. Fill each jar with necessary amount of standard soap solution, by weighing each sample to give liquid to composite specimen ratio at 50:1.

3. Open the beakers and place one composite specimen in each jar containing test liquid for test 4 as stated in the above table. Place 10 stainless steel balls (SS) of 6mm, diameter. Fit the beaker to their respective position by pressing against the spring and rotating it.

4. As the above temperature is attaineds, the timer was set for 30 minutes for Test 4.

5. The start switch was shut on. After the set time is reached, the motor stops automatically with an alarm/buzzer. Remove the entire beaker and take out all the composite specimens and SS balls with the help of filter unit.

6. The composite samples was rinsed twice in cold water and then in cold running tap water for 10mins and squeeze along the two sides. Open the composite samples and dry in air at temperature not exceeding 60^0C with three pieces (test specimen and two adjacent fabrics) in contact only along the remaining line of stitching.

7. The change in colour of the treated test specimen was evaluated by comparing with untreated piece of the sample using the standard grey scale for evaluation of change in colour. Also treated adjacent fabric pieces were compared with untreated adjacent fabric pieces using the standard grey scale for evaluating staining. Note that treated test specimen and the two adjacent fabrics will be allowed to cool and dry to regain their normal moisture content before evaluation. Examining the colour change (CC) and colour stain (CS) with grey scales was adopted.

ii. ***Light fastness test:***

All dyed fabrics experienced change in colour when exposed to daylight for long period of time and because of this action, dyed fabric faded away. The severity of exposure in both time and intensity of light determines the extent of change in colour. The colour fastness is denoted by the number of the light fastness

standard fabric that has changed colour to the same extent as the test specimen. It was expressed as the numerical rating for light fastness. (Ashis 2009)

The digi – light^{TM} (light fastness tester) which was designed to test colour fastness of fabrics to sunlight was used. This test was carried out by exposing a set of light standard fabric 1 to 8 and a set of test specimens to sunlight till the desired fading of colour is obtained. The change in colour of exposed area of the test fabrics and the light fastness standard fabrics were examined, rated and the relative degree of colour fastness of test specimens determined. (See plate 3.10, fig. 3.5)

There are two methods recommended in the ISO standard for mounting and testing of sample fabric for light fastness test as follows:

Method 1: This is considered and used in case of dispute that requires set of light fastness standards for each test sample fabrics.

Method II: This enables a large number of test samples of differing light fastness rating to be against a single set of light fastness standards.. This study adopted method II for the 82 samples of dyed fabric used for this study.

✓ **Method II :**

(a) One strip (5cm x 10cm) each of 1 to 8 light fastness standards (LFS) on the specimen card was mounted and put the same in one Type 'A' rectangular cell.

(b) Test specimen strip on the specimen card was mounted and the same was put in another type 'A' rectangular cell. Each Type 'A' rectangular cell accepted up to 10 test specimen.

(c) ¼ of the total length of each sample tests was covered as well as LFS single slot mask (¼ cover). Therefore in both the regular cells, the single slot mask was also put (¼ area covered).

(d) Both the rectangular cells to light were exposed.

(e) The effects of light by lifting the mask periodically and inspecting the LFS were followed.

(f) When a change in colour of the exposed portion of the LFS No. 3 have just perceived equal to grey scale grade 4 – 5, the samples were inspected and assessed their preliminary light fastness by comparing any change that occurred with the changes that occurred in standard patterns 1, 2 and 3.

(g) The single slot mask (¼ cover) in exacting the same position was replaced and continued to expose until a change in colour of the exposed portion of the LFS No. 4 were perceived, with equal to grey scale grade 4 – 5.

❖ ***The samples exposed to light move to stage 2.***

(h) The single slot mask (¼ area) with slot mask (½ cover) was replaced as shown in the in both the cells together.

(i) It was exposed continuously until a change in colour of the exposed portion of LSF No. 6 as perceived equal to scale grade 4 – 5.

❖ ***The samples exposed to light moved to stage 3.***

(j) The single slot mask (½ areas,) was replaced with single slot mask (¾ cover) . This was also done in both cells (rectangular cell that have been tested samples and rectangular cell having the LFS).

(k) Both the cells were exposed until either:

(a) a contrast is produced on standard pattern of equal to the contrast illustrated by grade on the grey sale or

(b) a contrast equal to grade 3 on the grey scale has been produced on the most resistant sample which occurs FIRST.

The three covers were removed and terminated the experiment.

❖ Then the light fastness were assessed as below according ISO standard:

(a) The final assessment in numerical ratings was based on contrast equal to grey scale grade 4 and/or grade 3 between exposed and unexposed portions of the samples.

(b) The change in colour of the specimen was compared with the changes which have occurred in the standard patterns under suitable illumination.

(c) The light fastness of the samples was the number of LFS which shows similar change in colour (this was visual contrast between the exposed and unexposed portions of the specimen).

❖ **The Humidity Control as adopted by ISO standard:**

(a) The light fastness of a pattern varies according to its moisture during exposure, and this was governed by the humidity of the air in contact with the pattern, termed the effective humidity.

(b) The effective humidity was a complex factor, which took into account not only the relative humidity of the surrounding air, but also its temperature, and the surface temperature of the pattern.

(c) The average value for day light exposures has been found to be 65% which was obtained as shown in the table 3.3 by placing a 2ml, saturated solution of sodium chloride, (Humidity Control Solution) at the bottom of the cell was to help the control of fluid filler. This reduced the effect heat on the on the dye fabric inside the slot cell.

(d) A control field filler (syringe) used for placing humidity control solution (H.C solution) into the cells.

(e) In the rectangular cells humidity control solution were been placed only 2ml with the syringe into the hole provided so that it went at the bottom.

(f) In the cylindrical cells, half an hour were allowed, before inserting the samples, to ensure the H.C solution has drained from the sides of the tube to the bottom.

(g) Sample has not become contaminated with H.C solution, since some liquids can cause colour changes.

(h) Stoppers were replaced tightly, so as to ensure that the effective humidity was maintained in the test.

(i) H.C solution was replaced when they become discoloured. Before putting in new solution, clean out and dry cells.

iii ***Perspiration and water fastness test:***

Colour fastness of dyed fabrics against perspiration is determined by exposing the fabric samples to the action of both alkaline and acidic reagents, while in contact with un-dyed adjacent fabric on both sides. The test specimen along with adjacent fabric were placed between plastic (acrylic) Separator plates placed under a fixed load, inside an incubator maintained at 37^0C. The apparatus used for this test is called Perspirometer (SS), which is made of stainless steel frame which

accepts twenty one 115cm x 60mm acrylic separator plates. Twenty specimen of 100 x 40 mm was placed between the places, under a load of 5kg, Ashis, (2009). The following procedures ISO standard was adopted.

(See plate 3.11, fig.3.6.)

1. The composite fabrics sample thoroughly was wet in alkaline solution at a liquor ratio of 50.1 and allowed it to remain in the solution at room temperature for 30 minutes. It was pressed and stirred from time to time to ensure good and uniform penetration of the liquour. The solution was poured off and the excess liquor of the fabrics samples was wiped. the composite specimen was put between the two acrylic separator plates.

2. The second fabric samples were wet in Acidic solution by the method as stated above.

3. After loading all the test specimens as above, the acrylic separator plates were placed, one over the other and all these were placed in between the two loading stainless steel plates. The stacked was pushed in till it was in contact with the rods at the back and placed the calibrated weight on the top of the apparatus. The springs were allowed to get compress so that the required load is applied on stacked test specimens. Both the knobs on the sides of the unit position were locked and the weight from the top loading platform was removed.

4. The loaded apparatus inside thermostatically incubator maintained at 37^0C was placed and allowed the apparatus in the incubator for four hours.

5. At the end of this period, the apparatus was removed from the incubator.

6. The Acrylic plate along with composite fabric samples was removed from the apparatus.

7. The test fabric sample was separated from the adjacent fabric and to evaluate the

   * change in colour of the treated test fabric samples and

   * The degree of staining of the two pieces of adjacent fabrics. This was assessed with two separate grey scales meant for the purpose.

❖ **Preparation of reagent for testing perspiration:**

The human perspiration can either b alkaline or acidic hence to undertake the perspiration test; the reagents were prepared according to ISO standard as explained below:

(a) Standard alkaline solution:

Prepare the solution by dissolving the following chemicals in one litre of distilled water.

(i) 0.5gms of L-Histiline Monohydrochloride Monohydrate ($C_6HgO_2N_3HCIH_2O$).

(ii) 5gms of Sodium Chloride (NaCl) NaCl , (iii) 2.5gms of Disodium Hydrogen Orthophosphate Dihydrate ($Na_2HPO_4.2H_2O$).

(iv.) Volume of distilled water – 100ml.

Put in a volumetric flask and agitate the solution thoroughly, it was brought to pH8 with O.IN (normal) Sodium hydroxide solution.

(b) Standard acidic Solution:

Prepare the solution by dissolving the following chemicals in one litre of distilled water.

(i) 0.5gm of L-Histidine Monohydrocloride Monohydrate ($C_6H_6O_2N_2HCL.H_2O$)

(ii) 5gms of Sodium Chloride (NaCL).

(iii) 2.2gms of Sodium Dihydrogen Orthophosphate Dihydrate.

(iv) Volume of distilled water 100ml.

Put in a volumetric flask and agitates the solution thoroughly; it was brought to pH 5.5 with 0.1N (normal) Acidic Solution. The above standards soap solutions was adopted. (See plate 3.8)

CHAPTER FOUR

4.0 Result and discussion.

4.1 Introduction

This chapter deals with the presentation and analysis of the results of the practical experiments performed on the specimens studied. The general objectives of the study as mentioned earlier in chapter one was to find out the quality and the quantity of dye contents that could be extracted from the eight parts of the various three dye-yielding herbs namely:

1. Parkia biglobosa (locus beans): leaves, seed, and bark.
2. Tectonia grandis (Teak): leaves and bark.
3. Terminalia glaucescens: (White afara): leaves, roots, bark.

All that formed the basis of the analysis of the study were the dye specimens collected from the three herbs, as well as various local additives and mordants dye experiments performed by applying them as colourant substances, and examination of colour fastness tests on cotton fabrics. The plate 3.12 presents the practical processes of the documentation.

The analysis presentation in Table 4.1 shows the solubility test of 5gm of dye specimens in 100mls of solvent results for 5mins each. From the table 4.1, it was observed that five different solvents were tested to dissolve the dye specimens obtained from seeds, leaves, barks and roots of the three herbs. These solvents are distilled water, ethanol, Dimmethy Formamide(DMF), toluene and acetone. Most

of these solvents are partial soluble and others full soluble of the content dye available. In the study the best soluble solvent was Ethanol for all the contents in the specimens were fully soluble.

4.2 Dye extraction and quantity extractable.

The table 4.2 represented various quantity weights of dye contents produced according to the amount of dye extracted from 150g of herbs from the leaves, barks, seeds and roots of the three herbs used in the study.

a. ***Quantity produced:***

In table 4.2, the column entitled quantity of dyes extracted; the

Results were presented as follows:

(1) Parkia biglobosa (locus beans): Seed – 11.03 grams, Leaves – 10.68 grams, Bark – 8.65 grams.

(2) Tectonia grandis (Teak): Leaves – 8.36 grams,

Barks – 3.01 grams.

(3) Terminalia glaucescene (White afara): Roots –

31.74 grams, leaves – 29.5 grams, barks – 4.58 grams.

b. Comparing different parts of the herbs that yielded more weight of dyes.

It was observed that in different part of the herbs, the weights were not the same as shown in table 4.2 as follows:

I. Parkia biglobosa (locus beans): The weight of the seeds was more than the leaves and the back of the herbs.

II. Tectonia grandis (Teak): The leaves were more than the back of the herb.

III. Terminalia glaucescene (White afara): The roots were heavier than the leaves and bark of the herb, while both the bark and root of Terminalia glaucescene (white Afara)

were very light.

c. The method of extractions:

Maceration method known as cool extraction, soxhlet extraction technique and extraction method of boiling with water were discussed in chapter three (plate 3.5). From the results obtained, it was observed that the three methods can be equally employed to obtain both qualitative and quantitative dye of the herbs. The visual

examination and the numerical rating of colour fastness test shown in table 4.3 proved that there were no significant different between hot and cold temperature of the dyes extracted. The maceration method known as cool extraction was mainly used in the study, because the specimen will not undergo much heat before colour fastness test. These will avoid reduction of dye contents as reported by Siva (2007) Laursen (2009) and Rita (2009)

The time for the extraction in the maceration, soxhlet and boiling, however varied. From the results it was observed that extraction at soxhlet and boiling temperature was also effective and simpler, but the specimen will undergo more heat before the colour fastness test as mentioned by Siva (2007) and Rita (2009). This research was carried out in the Pharmacognosy Laboratory, Department of Pharmacognosy and Drug Development, A.B.U., Zaria. The result of the three methods used in the study, showed that there was little difference in the quantity of dye extracted from each part of the herbs.

4.3 The Ultra Violet Spectrophotometer (UV) scan results.

The entire extracted dyes would not be completed and regarded as positive and standard experiments, without the confirmation of Ultra Violet Spectrophotometer (UV) scan results. The spectrophotometer measured absorbance and the transmitted coloured solution at a specified wavelength. The specimen in each graph stated the maximum absorbance of each dye that was under-studied as confirmed by krishnamurrthy *et al.,* (2002) and Ameuru (2009). This analysis test was carried out at National Research Institute for Chemical Technology, (NARICT.) Zaria. It was scan known as UV-Vis Spectrophotometer, model Uv-2550 shimadzu. The graphs in fugures 3.3.1- 3.3.8 in appendix showed wavelength and the results of the maximum absorbance of each extracted dye from various herbs studied and verified by the NARICT.

UV visible spectra of any dye show its peaks at predominating wavelength, indicating main hue. For natural dyes the spectra specially indicated different peaks for mixed colourants available in their extract in both UV and Visible region. UV Visible Spectroscopic studies of different natural dyes were carried out by Bhattacharya (1999) and Ameuru (2009) using different solvents for extraction. But in the study only ethanol solvent was used for the extraction as other solvents have been tested for solubility study tests were partially dissolved (table 4.2.1) as stated below:

- The result of all the specimens are as follows: the leave of locus beans showed 665.40nm W/L 2.551nm Abs, the seed gave 664.60nm W/L

and 0.433nm Abs, the bark 653.80nm W/L and 0.139nm Abs. The teak herbs produced 664.80nm W/L and 0.404nm Abs, while the bark showed 659.00nm W/L and 0.167nm Abs. The white afara leaves gave 664.80nm W/L and 0.815nm Abs. (see table 4.2.1) The back showed 664.50nm W/L and 0.221nm Abs. The root reported 663.60nm W/L and 0.270nm. The study was although scientific in nature, but it was useful in understanding the UV absorbance criteria as these were indication of many application information that showed possible fading and absorbance behavior under UV – light sunlight and those reports are important. (Ashis, 2009) (Table 4.2.1)

4.4 Assessment of wash fastness test.

The resistance of dyeing materials to laundry such as washing is referred to as wash fastness test Ameuru (2009). This is of great importance to the consumer, and there are several washing tests that are applied according to the purpose for which the material is intended. The present study of wash fastness test adopted from International Standard Organization was (ISO wash test no. 4) in table 3.1.

The results were graded with grey scale according ISO standard. In tables 4.3and 4.4 the summary result were record on wash fastness test are generally good with little stain on adjacent white fabric (CS). For instance, the rating were fair to good to very good for various dyes, for examples in TLW dyed with (Iron II sulphate), TBL dyed with (Potasium dichromate),TLT and TRW dyed with copper sulphate,

TBL dyed with sodium chloride show rating of fair (i.e 2-3). The rating of good are as follows: TLT dyed with (Iron II sulpate), TRW dyed with (Potasium dichromate), TLT, TRW dyed with (Copper sulphate) and TSL (Sodium chloride) dyed with the grading 3-4.

In the study the rating of very good were graded on alum, wood ash, iron II sulphate, potassium dichromate, copper sulphate and sodium chloride with 4-5 rating in table 4.4 and table 4.9A with little stain of adjacent white fabric of various parts of the three herbs.

4.5 Assessment of perspiration fastness tests.

Perspiration measures resistance of dyed substrate to body fluid when in use especially areas where these fluids emission are prominent, Ameuru (2009). In the present study the test was adopted on the dyed cotton fabric both in acidic and alkaline medium and the results are assessed on change of shade (cc) and colour stain (cs) using grey scale as well as degree of staining of the adjacent white fabric similar to wash fastness, the result are shown in tables 4.5 and 4.6, table 4.9A and plate 3.11 respectively.

The fastness to perspiration in table 4.5 presented, the various time that the cotton fabrics stayed in dyeing processes and table 4.6 observed time limit of 2hrs period of dyeing with various mordants employed. Table 4.5 states that all the dyes were generally fair to good both in acidic and alkaline medium. All the mordants in table 4.6, shows that the dyes are good or very good with grading 4, and 4- 5. TRW, TLL,

except TWB, TRW. TLW those are fair these with poor grading of 3, 3-4 respectively in the column of colour stain (cs) as well colour change (cc).

In alkaline medium, dyes of all mordants, also show rating 5 excellent, 4-5 or 4 with very good and 3 fair respectively. The only additional observation is that the acidic ratings are higher than the alkaline ratings. Ameuru (2009) explained that as the result of repulsion between negative affinities that is produced in alkaline medium as opposed to the acidic medium condition where there is positive affinity of fibre which will produce superior fastness properties than alkaline medium condition.

4.6 Assessment of light fastness tests.

Light fastness test is the resistance of colour of dyed materials to the influence of light. The fastness assessment can be done using daylight which takes a long period or by a xenon arc known as digi – light^{TM} (light fastness tester), which is the accelerated version of the test Ashis (2009) and Ameuru (ibid). In the present studies, to achieve quick results, the xenon arc known as digi – light^{TM} (light fastness tester) was employed and the evaluation of results based on the eight wool standards 1 to 8. See plate 3.10, fig, 3.3 in appendix.

Fading on exposure to light is undoubtedly the most complex of the reactions which dyes undergo on a substrate, and much research has been devoted to discovering its causes. First, it is important to appreciate that all organic colouring matters fade when exposed to light, whereas hardly any inorganic colourants and no ‘abstract’ colour do so (Ameuru, 2009). Energy in the form of light is absorbed by the dye and causes some of its molecules to unstable. They are then in a so called excited

state and under these conditions; the dye may react with surrounding materials, such as oxygen in the air or sometimes with the fibre itself. It thus decomposes and loses its colour.

There is no absolute value for the light fastness of any dye, the value obtained depends not only on the nature of the dye but also on its method of application, the depth of dyeing, the type of fibre, the moisture content (regain) of the fibre, the relative humidity and the oxygen content of the atmosphere, the nature of illumination and other factors (Ameuru, 2009).

Table 4.7, presenting the various time that the cotton fabrics stayed in dyeing processes with light fastness rating on degree of fading compared with the used of sodium chloride with various timing, while table 4.8 graded the light fastness rating on degree of mordants fading compared with wool standard of time limit of 2hrs period.

The result in table 4.7 states that all the dyes were generally fair to good in various timing, the hours gave these results: LTT 6, TBT 5, TBL 7, TBW 7, TRW 7, TLW 6, TLW 6, TLL 6, while TSL was poor with the rating of 3.

In table 4.8 the light fastness ratings compared with wool standard of different mordants for 2hrs indicated that most of the parts of the three herbs selected in this study were excellent, good, fair and poor respectively. For example sodium chloride as mordant was good with the rating of 7 in some of the herb parts. Copper II sulphate was poor with TLT, TBT, TSL, and TBL with 3 rating. Alum rating is excellent with TRW with the rating of 8 and poor with TBT 3, and TSL 2, wood ash

with TSL rating of 3 respectively. There were general good light fastnesses rating in all the dyed cotton fabric ranging from 4 to 8. (Table 4.9 B)

The light fastness rating of the dyes on cotton fabric indicated dyes 5 -6 gave very good rating which are in line with the fact that the lower the sulphonic acid groups, the better are photo- stability, Ashis (2009) and Ameuru (2009) had investigated this in details. They explained that the number of sulphonic acid group affects photo- stability from the point of view of surface activity in relation to aggressive state. The same phenomena can be used to explain the good light fastness rating of dyes.

The better light fastness obtained on cotton fabric is based on the fact that the more the exposure of fabric darker it becomes. This implies that cotton fabric react with dye enhance the better light fastness results were achieved.

CHAPTER FIVE

5.0 Summary, Conclusions and Recommendations.

5.1 Introduction

This chapter contains the summary of the findings from study, conclusions and recommendations drawn from the study. These include the statements the problems, objectives, procedures and methodology of specimens the study. The conclusions drawn were based on the results of the experiments performed and findings in various tables in chapter four in the appendix. The recommendations were made in line with the findings from the study.

The objectives of the study were exploring and extracting of colour substances from herbs for the possible use as dyes on cotton fabrics, determining the quantity and maximum quality that were extracted from a particular weight 150gm., of herbs in Lokoja, Nigeria. It also examines the fastness properties of such dyes on cotton fabric. It is an experimental study that determined a way of preserving such extracted dye stuffs in a way that could be used dyeing on cotton fabric without the loss of colour and for the analysis that could be used for the purpose of large scale production. The concern of the study therefore, was about the sources of scarcity of natural dyes in studio based textile design in Nigeria higher institution for example Kogi State Polytechnic Lokoja. It also determines the wash Fastness, perspiration and light fastness tests. The study examines the precautions of synthetic dyes to dyers and the societies at large as it's affected their health.

The specimens of the study were three dye yielding herbs namely: Parkia biglobosa, Tectona grandis, Terminalia glaucescenes from which the eight parts of these herbs were examined, include the roots, barks and leaves respectively. The areas of coverage were Lokoja and environs of Kogi State, Nigeria. The materials used included Cotton fabric, and distilled water that was collected from physical laboratory chemistry department, A.B.U. Zaria. The six selected mordants were parts of materials in the study.

The experimental procedures of the specimens collected were designed in the following:

1. Ways by which dyes can be extracted from parkia biglobosa, Tectona grandis, Terminalia glaucescens species of dye yielding herbs.

2. Methods of examining the solvent by which dyes can be extracted in much quantity from certain parts of the above dye – yielding herbs in the study.

3. Ways of findings an appropriate solvent which can easily dissolve the dyes powder of the yielding herbs.

4. Ways of finding the quality of the dye – yielding herbs in the study.

5. Six different mordants (Copper II sulphate, Sodium chloride (common salt), Local alum (Potash), Iron II sulphate, wood ash, Potassium di- chromate.) were used in the study.

6. The colour fastness on wash, perspiration and light fastness test as ISO standard test no 4 were carried out in the study.

7. Precautionary measures of synthetic dyes were examined.

To achieve the above objectives, the study was further divided into the following areas of operations.

a The collection of the herbs, description of dye-yielding herbs, colour tests and preservation, preparation of the dye bath.

b Solubility test.

c. The Extraction process of dyes: Quantity available for extraction.

d The mordanting process.

e. The dyeing process.

f The colour fastness tests.

5.2 Summary of findings:

Based on the analysis of the specimens contained in chapter four (4) of the study, the following findings are pertinent:

1. The most appropriate way of extraction of dyes was by maceration method or cool extraction and soxhlet extraction technique. Both processes were used, in order to obtain qualitative and quantitative dyes from *Parkia biglobosa, Tectona grandis, and Terminalia glaucescens* respectively.
2. Ethanol and distilled water were the most solvents that easily dissolved dye extracts from herbs in powder form.

3. Various shades of yellow, khaki brown, to certain extent light brown and grey from the eight parts of the various herbs that were discovered when treated with mordants before dyeing, however, have good colour shades were obtained without the use of mordants, but it was observed such that no-mordant dyed samples tend to lose a considerable amount of colour when wash – fastness properties test were conducted in the study,
4. No significance difference exists between hot and cold water/ethanol dyeing with respect to the colour fastness properties of the herbs.
5. It is observed that dyestuff obtained from the herbs is directly applied on cotton fabric with the aids of mordants.
6. Six different mordants produced different colours with the same dyestuff made it possible for the dye to bind permanently on the fabric. This is to mention that mordant does not only support in varying the colour shades of the same herbs but also makes the colour fast. These mordants were local alum, wood ash sodium chloride (common salt), iron II sulphate, copper II sulphate, and potassium di-chromate.
7. Various weights existed according to the quantity of dye contents extractable from leaves, barks, seed and root of the three herbs. It is also clear that root and leaves of terminalia glaucescens contained many quantities than the remaining six parts of the herbs studied. Thus, it indicates that these two parts were the most favourable parts of the extracted dye from the herbs studied. Similarly, there exists difference too, in the fastness properties of the dye contents extractable from leaves, bark, seed and roots of the herbs.

5.3 Correlation of research findings in relation with previous research.

Using the review of related literature and the experiments conducted for the study as a basis, this research confirmed that herbs have odour during processing, future research should discover if natural herbs odour is hazardous to humans health. This was in agreement with the statements of Maiwada (2010) and Singh et al., (2005) noticed that herbs give odours when being processed into dyes. Singh et al., (2005) stated that the dye produced good quality according to the degree of the odour. Also, based on the finding of the study it was also found that mordanting had a lot of advantages in herb dyeing. Krishmamurth (2005), reports that mordanting is very important as dyeing, as he believed that the success of herbs dyeing rest chiefly on the mordanting processing. This study reinforced the reliability of their findings statements.

This study found that the most appropriate way by which dyes can be extracted from herbs that consist the leaves, root, barks should be cut into smaller units, crushed and dried, then the dyer leaves the yarn in the dye bath, the deeper or richer the colours depend on time period in dye bath. For this reason, Mike (2010), reported the process of chopping off the herb into small pieces, the process of pounding, the crumbs and dyed materials weighted and measured bottles with cover and solution measured and amount of water poured into the bottom flask, and placed on the water bath was also observed and confirmed in this current study.

It confirmed in the study that the ranges is between 20 minutes to 2 hours (120 minutes) of heating gave the best colours. This is still in line with the studies of

Saliu (1983), and Siva (2000) that explained that herb is by boiling the dye material for 30 minutes after a specific length of soaking. The dye materials should be heated to a boiling point. It does not need to stay overnight, as it wastes time. More so, cool dyeing is recommended as it reduces the destruction of dyes contents from heat. Siva (2007) asserted that tedious extraction of colouring component from the herbs materials, is low in colour and long time of processing makes the cost of dyeing with natural dyes higher than synthetic dyes, but when mordants were added, it enhanced their fastness properties, the time of processing is accelerated in the study.

Simon (2000) observed that any category of herbs should be soaked at least over-night, or should be soaked for three days given up its colour and strains out the herbs. Ashris (2009) ascertained that a dry bark is to be soaked for a day, still required three hours boiling and stayed for one hour on the dye bath. This study discovered that those procedures wasted time, that there is no need for soaking of the herbs materials (root and bark). The maximum time the fabric entered into the dye bath and temperature is raised according to Siva (2007), Gwari (1989), Joanne ( ), Blish (1980), found that to extract dye from herbs, the dye material ought to be covered with water and brought to Simmer state (180^0f – 190^0f) or (64^0C – 70^0C), Siver (2007) adds that leaves, roots, bark, fruit and flour to a boiling point of 100^0C is enough for extraction.

Gwarzo, et al (2009) mentioned that dye as substance used for colouring materials permanently are very important and are part of the materials that Mustard (1997)

Siva (2007) documented that black berry yielded a brown grey with Alum mordant with wool. This study confirmed that alum produced variety colour of shades.

The Columbia Encyclopaedia (2004), Walton (1988), Castino, R. (1975) and Christive (2007) in their reports stated that herbs produced varied results from one dye pot to the next. They further said that number of variables – availability of dye, dye stuff, the different site climate (geographical region) or sheer vagaries of archaeological discovering determined the colours produced. This statement was confirmed in the study especially as the one of materials transferred to other dyeing pot stained part of the fabric with defferent colour to produce another result. Therefore extra-ordinary careful is needed to discover the exalt colour produced from the herbs.

In another development New Feed (2006), Alau (1986) mentioned the method of using maceration technique in cold water dye extraction in order to extract dye, the weight of the dye powder (raw material) must be dissolved in a required volume of water for twelve hours before boiling for sixty minutes. Using laboratory suction pup, the boiled solution, should be allowed to cool before straining out to extract dye solution. These reports have reinforced the reliability of using the soxhlet extraction apparatus and the maceration techniques as employed in the study.

Orebiyi (2010) reported the process of extracting dye from herbs locally in Kabba with the use of wood ashes known as ‘Abete’ for five days. The study observation was that, when the dye materials with wood ashes is heated for 2 hours. It gave the same desired result. Siva (2007) explained that the village dyeing reduction is

achieved by natural fermentation which takes place in large open vats. After soaking the fabric in the indigo solution, when dried and oxidation by the air before one can see the colour deposited on the fabric. This study shows that, one can easily recognize the hue of the colour before final oxidation takes place.

Gwarzo et al (2009), Krishmamurthy et al (2002) and Wangboje (1985) reported the need for experimental study into local resources as using local materials provide natural colours, this resulted to excitement, mysterious, and perplexing. This study also confirms that various colours produced through the experimental study show that the colour extracted from herbs were amazing and exciting to the visually.

Annonymous (2002), Singh et al (2002) explained that Germany discovered 920 synthetics dyes as they became the first country to ban the synthetic dyes in 1996. These dyes caused danger to human health in general and the environment. Annonymous, (ibid) mentioned that in India synthetic dyes caused health problems, therefore, and reverted to the use traditional dyes instead of synthetic dyes. To be more sure Abass (2009) noticed that new discovery of synthetic dye hazard has sensitization effect from handling the dry powder has pose a great threat to public health safety, Based on this, the research were on natural dyes that reduces the risk that causes humans health and environment problems. The study worked on natural dyes that and suggested that Nigeria at large should revert to natural dye extracted from herbs.

5.4 Conclusion.

Based on the results of the experiments performed in the study, it is concluded that:

1. Alum, potash, wood ash are suitable local additives for herbs dyeing, while the best mordanting techniques for them is 'pre-mordant', or by mordant – with.

2. The results obtained from the laboratory tests, depicts that herbs produced wide range of colours that can be applied as dye on cotton fabric for both textile industries as well as instructional dyes for textile institutions. These colours appeal the more to social, economic, cultural, aesthetic and emotional value of the people as it is environmental friendly.

3. Soxhlet extraction apparatus and maceration techniques can both be used in the extraction processes in order to obtain qualitative and quantitative dyes from parkia biglobosa, Tectona grandis and Terminalia glaucescens.

4. Various mordants produced different final results from the same dye.

5.5 General recommendation:

a. Based on the findings from this project it is significance for government to look inward on available human and natural resources that can be used to develop textile industries in the areas of production dyes from herbs, like china and India.

b. The quantity of dye extracts obtained from herbs in the study reveals that it could be economical and easily available mordants can help local dyers,

schools and workshops in Lokoja Nigeria to have enough dyes for their works. It reduces the scarcity of dyes and current high cost of dyestuff in the local market.

c. With the various shades produced from the herbs. The requirements of free-lance resist dyers can be met. This will make it possible to set up small scale industry in textile.

d. There should be opportunity in such that a conference can be organized by Ministry of Education, Ministry of Science and Technology, Universities in order to display, educate, harness, collect discovery for consequential national development.

e. For future research the following educational recommendation are made:

- Other herbs of local origin that contain colourant suitable for production of dyes should be explored.
- More ways of extracting dyes from herbs than three ways used in the study should be explored.
- Most of agents and equipment used for light fastness test are not readily available. More researches should be done on local method of producing them.
- The use of other fabric (wool) other than cotton should be tried.
- Administratively, since the supply of synthetic dyestuffs are causing health and environment for our local textile dyers,

schools and workshops. The federal and state governments as well as textile industries should encourage the growth of dye-yielding herbs for studies.

Institutions of higher learning and private organization should be encouraged to build some research institutes, such as Textile institutes where intense work of this research are will be conducted.

Workshops and seminars on local ways of sourcing dyes materials should be from time to time organized and give publicity. It is noteworthy that, if these studies and other recommendation are employed this will save Kogi State and Nigeria at large from the huge amount spent annually for importation of synthetic dyes.

6.0 REFERENCES

Abbas, K.A. (2009) Environmental Management in the Textile Industry: APanacea for sustainable Development: Proceedings of 1st Annual conference of the Association of TextileTechnologists of Nigeria. (ATTN) (Kaduna). Pp. 11-12

Adeleye, I. and Opia, L. (2006) Antimicrobial activity of extraction of local cough.Mixture onupper respiratory tract. West Indies.53 (3): 188 – 190. http//ajol.info/index.php/ajid/ retrieved on 27 October, 2010.

Adetoro, S. A. (1981): Development in action- traditional dyeing and resist Methods: an article on N.Y.S.C Programme given to the Radio Television Kaduna for broadcast. A.B.U. Zaria.

Alau, D. D. (1986), Exploratory Extraction of Dyes from Local Plants in Southern Zaria. Unpublished

M.A. Thesis. Department of Industrial design, Ahmadu Bello University, Zaria. P1.

Anonymous (1998): Chemicals- Friend and Foe? *In Awake December 22, (ISSN 0005-237X)* Watchtower…Society, New York, INC.; Pp. 4-6.

Anonymous (2000) When Chemical makes you sick.*In Awake August 8, (ISSN 0005-237X)* Watchtower…Society, New York, INC.; Pp. 5-6.

Anonymous (2002a): The Cochineal- A very special Insect. *In Awake July 8, (ISSN 0005-237X)* Watchtower…Society, New York, INC.; P. 22.

Anonymous (2002b):Watching the World. *In Awake July 22, (ISSN 0005-237X)* Watchtower…Society. New York, INC.; P. 29.

Anonymous (2004): We appreciate the Cloth we wear? *In Awake February 8, (ISSN 0005- 237X)* Watchtower…Society., New York, P 4

Anonymous (2007): I was Allergic to Toxic Chemical. *In Awake June 8, (ISSN 0005-237X)* Watchtower…Society, New York. INC.P 3.

Anonymous (2008): Multiple Chemical sensitivity's a mysterious malady. *In Awake August 8, (ISSN 0005-237X)* Watchtower…Society, New York, INC.; Pp 3-5.

Ashis, K. S., and Priti A. (2009).Application of natural dyes on textiles. Institute of Jute Technology, Indian Journal of Fibre and Textile Research Volume 34 December, India.

Bin, B. N. (1985).Determination of the Amount of dye extraction from plants and examination of their fastness properties to cotton fabrics.Unpublished M.A. Thesis, Department.of Industrial Design, A. B. U. Zaria.

Biodun, N. (1995). Dyes from Plants their extraction and Application to Textiles.JidePublishing House. Lagos: P. 22.

Blanc, R., Espejo, T., Moutes, A. L., Tores D., Crovetto G., Navalon, A. and Vilchez, J. L. J. (2006).Chromatography p105. . http://tamm,chat-2009-2010 Retrieved on 3 July, 2010

Bliss, A. (1980) North America Dye *Plants.*Charles Scribers sons. New –York:

Castino, R. (1975) Spinning and Dyeing the Natural way. London: Exams Brother Limited.http://.ingentaconnet.com/ Retrieved on 20 June, 2010.

Chandraouli, K.V. (1995) Sources of Natural Dyes in India- A Compendium with Regional Names, PPST Foundation, Chemical."http://.ingentaconnet.com/ Retrieved on 10 June, 2010.

Chinedu, C. C. (1998): Contemporary Nigerian Art and its Classifications. Delta State: Delsu Consult Publishing House.

Christive, B. N. (2007) Modified from Cotton, Mary-Rusele, and Hopi Dye: Flags: Museum of Northern Arizona. Indian Natural Dye. . http://.ingentaconnet.com/ retrieved on 8 June, 2010.

Debajit, M. (2008): Arumachi Pradesh State Council for Science and Technology vihar, Itamager- 791,113 New projects India. -http://.ingentaconnet.com/ retrieved on 25/08/2010.

Emu, O. (1984): Certificate Art for Junior and Senior Secondary Schools. University Press Plc. Ibadan. Pp. 69-70.

George, T. (1979): Art Teaching in African Schools. Heinemann Educational Books (Nig.) Ltd. Ibadan.

Gile, C.H. (1974) Labouratory course in dyeing2.The society of dyers and colourist Bradford Pp. 33 -37.

Gulrajani, M. L. (1992): Introduction to Natural Dyes, Indian Institute ofTechnology. New Delhi. Indian Natural Dye. Pp. 26, 191-201. http://.ingentaconnet.com/. Retrieved on 11/06/10.

Gulrajani, M. L. (2001): Present State of Natural dye, Indian J fibre. Text Res. Indian Natural Dye. Pp. 26, 191-201. http://.ingentaconnet.com/Retrieved on 10/01/10.

Gwari, W. B. (1989) Extraction and Determination of Dye Contents from Cashew Plants using some selected Mordants. Unpublished M. A thesis submitted to the Department. of Industrial Design, Ahmadu Bello University Zaria. P.19

Gwari, W. B. (2006): The Concept of Man –Made Fibre on Textile Design and their uses by Consumers. A conference paper presented, at the Celebration of Zaria Art School at 50. Theme: A Golden Jubilee of Zaria. Art School in National Development Zaria.

Gwari, W. B. (2008) A study of Computer- Aided Textile Design and its Application in Traditional Textile centres in Zaria and katsina cities. Unpublished Ph.D. Thesis Department. of Industrial Design, A. B. U. Zaria. Pp 9-10.

Gwarzo, A. I. and Alabi, U.D. (2009) Application of Design Elements and Principles in Fashion for Sustainable Economic Development: Zaria: Proceedings of 1st Annual conference of the Association of Textile Technologists of Nigeria.(ATTN), (Kaduna)

Hagg, I. (1984) Die Textilfundr aux dem Hafan Von Haithabu. Neumunster:Karl Wachholz verlag.

http:// www. Kidgardening.com/ retrieved on 15 January, 2010.

Islam, M. (2000): Certain Fibre yielding plants in the Eastern region constraints.Aruncachel for News. Indian Natural Dye. Pp 85-92. http://.ingentaconnet.com/Retrievedon10/10/09.

Jain, S. K.and Rao, R.R. (1989) "Handbook of Field and Herbarium methods, today and tomorrow Printers New Delhi. Indian Natural Dye. Retrieved on 8 June, 2010

Jain, S.K. (1989) Methods and Approaches an Enthobotany, Society of Ethnobotanists,
Lucknow. Indian Natural Dye. Retrieved on 10/10/09 James, E. (2000) Introduction to natural dyes. Amazon.com retrieved on 25 June, 2009.

Joanne, B. E. ( ) *Nigerian Handcrafted Textiles;* Ile-Ife: University of Ife-Ife Press Nigeria. Pp 22-30.

Kparevzua, B.A. (2009): INDE 505: Designer and Client Relation. Lecture Note.

Krishnamurmy, K.V. Siva, R.,and Senthil, K.T. (2002) Natural Dye-yielding Plants ofShervaroy Hills of Eastern Ghats in Proceeding of National Seminar on the Conservation, the Eastern Ghats, Environment Protection Training and Research Institute Hyderabacl 24- 25 march 2002. Pp. 151-153. Retrieved on 30 June, 2009.

Krishnamurthy, T. (2005): Minor Forest Products of India, Oxford and IBH, New Delhi.Indian Natural Dye. Pp 340- 364.http://tamm,chat-2009-2010 Retrieved on 8 June, 2010.

Labil, O. (2010) Natural dyes "Pa Da Cotton Textile Museum" Thailand, hand looms indigo, Blog 2009 –2010 #6: pa http://tamm,chat-2009-2010Retrieved on 12/05/10.

McRae, D. and Bobbi, A. A. (1991) Natures Dye pot: A Resource Guide for Spinners, Weavers and Dyers. Texas: fiber works, publications – 944, 577-02-4., Microsoft student DVD,
http://tamm,chat-2009-2010 retrieved on 27 October, 2010.

Mike, R., (2010) (Wild Colour – Exciting Colour from Natural Dyes. http: // tamm.chat-2009-2010 blogspot.com/ retrieved on 25 June, 2011. Miyamalsu, F. (tamm.chat-2009-2010 biogspot.com.) retrieved 0n 16/06/10

Mosaku, O. (1987) Extraction and Preservation of Dyes and dyeing using selected Plants in Nigeria. Unpublished M.A Thesis submitted to the Department of Industrial Design. Ahmadu Bello University, Zaria.

Musa, A. (1985). Source of Local Colour in Kano and their Suitability forContemporary use. Unpublished M. A. Thesis.submitted to the Department of Industrial Design, Ahmadu Bello University, Zaria.

Mustard, F.E. (1997) Dyeing the Natural Way.Great Lakes Living Press Limited, Publishers matter son. Illinois.

National Bureau of plant Genetic Resource (2000) Indian Natural Dye. Retrieved on 8 June, 2010.

Noah, B. (1981): Colour for Textile Ancient and Modern. Seminar paper: Department of Textile Science and Technology, Ahmadu Bello University, Zaria.

Nworgu, G. (1991), Educational Research: basic methodology. Wisdom Publishers limited, University of Nigeria. Nsuka.

Ogunbowale, W.O., Maiwada, S., Bello, K. A. and Kolawole, E.G. (2009): Networking/ Cluster Strategies for Developing the Handcrafted Textiles Small and medium Enterprises (SMEs) Nigeria: Proceedings of 1st Annual conference of the Association of Textile Technologists of Nigeria. (ATTN), (Kaduna 2008). Pp. 37-38.

Oguntona, T. (1984): The Dynamic of Colour in Environment Design.A.B. U. Zaria. Seminar of African Art in Historical Perspectives.Held at the University of Lagos. December, 11- 12,

Olugbemi, P. O., Ajulo, O. and Abubarkar, F. I. E.G. (2009): The effect of Government import Prohibition Policy Vis – A –Vis smuggling on The Nigerian Textile Industry. roceedings of 1st Annual conference of the Association of Textile Technologists of Nigeria. (ATTN), (Kaduna)

Pushpa, B. and Sengupha, S. (1992): The importance of fibre The Textile Institute: Protective Clothing Textile Clothing. Volume 22, Num. 22/2/9/4. India. Pp.1, 85.

Saliu, A. R.(1983): Dye Extraction from natural Plants in Zaria. Unpublished P.G.D.E Thesis, Institute of Education.Ahmadu Bello University, Zaria.

Sarmah, A., Haridansa, K. and Bliss, N. S. (2000) Development of Medicinal Plant as Economic ventures in Arumachal Pradesh, Prospects and Constraints Arumachal for News. Indian Natural Dye. Pp. 18, 89-92. *http://www.questa.com./*Retrieved 10/10/2009.

Sanees, U. Ramatu, A. A. and Abdulkadir, S. (2009): An Empirical Study of SMEs in the Textile and Clothing Town of Khalkaranji. Zaria: Proceedings of 1st Annual conference of the Association of Textile Technologists of Nigeria. (Kaduna 2008). Pp. 38-39.

Sieber, R.S. (1978): African Textile and Decorative Arts.Museum of Modern Arts. New York.

Singh, R. Jain, A., Panwan, S., Gupta, D., and Khare, S. K. (2005): Microbial activities of natural dyes. Pigm.Natural Dye.Indian.Pp. 66, 99-102.*http://www.questa.com./* Retrieved on 10 October, 2010.

Singh, V. and Singh, R. V. (2002): Healthy hue, Down to earth. Pigm.Natura Dye. Indian *http://www.questa.com./*Retrieved on 10 October, 2010.

Siva, R. (2003)Assessment of genetic variation in some dye yielding plants using Isozyme data, PhD. Thesis, Bhara thidasan University Tiruchcrapalli.Pigm.Indian Natural Dye.*http://www.questa.com./* Retrieved on 8 June, 2010.

Siva, R. (2007) Status of Natural Dyes and Yielding Plants in India.*Current Science*. Vol 92, No 1, 10 April, Indian Natural Dye. Pp. 7, 916-925. *http://www.questa.com./*Retrieved on 8 June, 2010.

Storey, J. (1978):Manual of Dyes and Fabrics. London: Thomas and Hudson Ltd.

The Columbia Encyclopedia (2004) sixth edition. Copyright Columbia University Press licensed from Lernout & Hauspie speech product.*http://www.questa.com./* retrieved 10 March, 2009.

*The Wealth of India (*2003): A Dictionary of Indian Raw Materials and Industrial Products. Council of science, communication and information. New Delhi. http://www.questa.com./ retrieved 10 March, 2009.

Thomason, H., (1981): *Fibres and Fabrics of today.* Third Edition, Heinemann Education Books Lt London:

Wangboje, S. I., (1985): An address delivered by Professor Wangboje, Deputy V.C. University of Benin, on the occasion of the opening of Art Exhibition, mounted by the Benin School of Art A.B.U. Zaria. 3, June, 1985.

Wealth of India Dictionary (2003): Indian Natural Dye. Retrieved On 10/01/10.

www.business-nigeria.com/map, retrieved 10 March, 2009.
www.mapsofworld.com/niger, retrieved 10 March, 2009.

www.worldatlas.com. Retrieved 10 March, 2009.

www.lonelyplanet.com/./nigeria/ retrieved 10 March, 2009.

Interviews and discussion:

Adamu, M., (2010): Department of Pamacognosy and Drug Development, Ahmadu Bello University, Zaria. (Herbarium)

Ajarosi, (2009): Kogi State Ministry of Agriculture, Department of Forest, Lokoja, Kogi State.

Maiwada, S., (2010): Department of Industrial Design, A. B. U. Zaria.

Orebiyi, O., (2010): Former Commissioner of Women affairs, Kogi State.

Owolabi, J. F., (2010): Department of Horticulture and landscape, Technology, College of Agriculture, Ahmadu Bello University, Kabba.

Rukayatu, T., (2009): Herbs Seller. No K112, Taiwo Rd, Lokoja, Kogi State, Nigeria.

Appendix: **List of tables:**

Table 3.1 Description of the herbs:

Extraction points	Family	Botanical names	Common names	Local names	Descriptions
Leaf, bark, and seed	Verguninusae - Mimosaceae	*Parkia biglobosa (Jaq.)R*	African local bean.	Dorawa *(Hausa)* ,Iru *(Yoruba)*	Tree up to 20m high, leaflet 35-65 cm on each.
Leaf, root	Verbenaceae	Tectona grandis *linn F.*	Teak	Labadiya *(Hausa)* Igi Oba*(Yoruba)* Shinasi *(Kabba)*	A medium size tree 27m to 30m tall. Height of 50m with a dbh of 2m.
Bark, root, and fruit	Combretacae	Terminalia glaucesens.	White Afara	Baushe *(Hausa)* Idi- apata/ Idi odan *(Yoruba)*	Tree 20m high with dark grey deeply fissured bark

Sources: Results analysis by the researcher (2013)

Table 3.2: Washing fastness ISO standard guideline:

ISO Test No.	Recipe	Temperature	Time
1.	0.5% Soap	$40^0 \pm 2^0$C	30 mins
2.	0.5% Soap	$50^0 \pm 2^0$C	45 mins
3.	0.5% Soap $\pm$ 0.2% Na_2CO_3	$60^0 \pm 2^0$C	30 mins
4.	0.5% Soap $\pm$ 2% with 10 balls Na_2CO_3	$95^0 \pm 2^0$C	30 mins
5.	0.5% Soap $\pm$ 0.2% with 10 balls Na_2CO_3	$95^0 \pm 2^0$C	4 hours

Source: Paramount since 1964 ISO certified company. (User's manual).

Table 3.3: Humidity control solution standard:

ISO TestNo	Effectively Humidity %	Humidity Control Solution
1	10	Saturated solution of ZnC|l12 \9at least 245g/100cc
2	20	Saturated solution of KOH.
3	45	Saturated solution of K_2CO_3 (at least 105g/100cc
4	65	Saturated solution of NaNo3 (at least 75g/100cc)
5	90	Saturated solution of BaCl2 (at least 31g/100cc)
6	100	Water.

Source: Paramount since 1964 ISO certified company. (User's manual)

Solubility test results for 5 mins each.

Table 4.1: Solubility test result for different solvent used in the study:

S/N	Solvent	Herbs	Behaviour observed in Solvent	Colour Developed
1	DMF	Terminalia glaucescene Leaf	Soluble	Light – yellow
		Bark	Soluble	Red
		Root	Soluble	Yellow
		Tectona grandis (Teak) Leaf	Soluble	Red
		Bark	Soluble	Red
		Parkia biglobosa (locus bean) Leaf	Soluble	Yellow Green
		Bark	Soluble	Brown
		Seed	Soluble	Light Yellow
2	Toluene	Terminalia glaucescene Leaf	Not soluble	Light Yellow
		Bark	Slightly soluble	Red
		Root	Soluble	Yellow
		Tectona grandis Leaf	Soluble	Red
		Bark	Slightly soluble	Red
		Parkia biglobosa Leaf	Soluble	Green
		Bark	Not soluble	Whitish in nature
		Seed	Soluble	Yellow
3	Acetone	Terminalia glaucescene Leaf	Slightly soluble	Light Yellow
		Bark	Slightly soluble	Light Red
		Root	Slightly soluble	Yellow
		Tectona grandis Leaf	Soluble	Yellow
		Bark	Slightly soluble	Light brown
		Parkia biglobosa Leaf	Soluble	Green
		Bark	Slightly soluble	Light Brown
		Seed	Slightly soluble	Yellow
4	Ethanol	Terminalia glaucescene Leaf	Soluble	Yellow
		Bark	Soluble	Red
		Root	Soluble	Yellow

		Tectona grandis Leaf	Soluble	Yellow
		Bark	Slightly soluble	Red
		Root	Soluble	Yellow
		Parkia biglobosa Leaf	Soluble	Green
		Bark	Soluble	Yellow
		Seed	Soluble	Yellow
5	Distilled water	Terminalia glaucescene Leaf	Very slightly soluble	Light Yellow
		Bark	Very slightly soluble	Light Yellow
		Root	Slightly soluble	Light Yellow
		Tectona grandis Leaf	Very slightly soluble	Light Green
		Bark	Very slightly soluble	Light Red
		Parkia biglobosa Leaf	Very slightly soluble	Yellow
		Bark	Very slightly soluble	Pink
		Seed	Very slightly soluble	Grey

Sources: Results analysis by the researcher (2013)

Dye extraction and quantity extractable.

Table 4. 2: The quantity of dye extracted from the each parts of the three herbs.

S/N	Botanical Name	Local Name	Part of Herb used	Herb tag	Solvent used in volume ethanol	Total wgt of Sample	Bottle wgt	Extraction of Dyes	Sample of colour developed
1.	Terminalia glaucescens	Igi Idi Yoruba	Leaves	01	250mls.	74.05g	45.00g	29.5g	Yellow
			Bark	06	250mls	55.52g	50.94g	4.58g	Red
			Root	03	250mls	76.84g	45.09g	31.75g	Light yellow
2.	Teclona grandis	Igi Oba Yoruba	Leaves	04	250mls	57.46g	49.10g	8.36g	Red
			Bark	08	250mls	43.93g	32.28g	8.65g	Red
3.	Parkia biglobosa	Igi Iru Yoruba	Leaves	02	250mls	73.38g	62.70g	10.68g	Yellow
			Bark	07	250mls	60.22g	57.21g	3.01g	Yellow
			Seeds	05	250mls	68.98g	57.95g	11.03g	Green

WGT: Weight g: grammes.

Sources: Results analysis by the researcher (2013)

4.2.1 The Ultra Violet Spectrophotometer scans (UV):

S/N	**Parts**	**Herbs**	**Weave land**	**Max. Abs**
1	Leaves	Locus beans	665.40nm W/L	2.551nm Abs
2	Seed	Locus beans	664.60nm W/L	0.433nm Abs
3	Back	Locus beans	653.80nm W/L	0.139nm Abs.
4	Leaves	Teak	664.80nm W/L	0.404nm Abs
5	Back	Teak	659.00nm W/L	0.167nm Abs.
6	Leaves	White afara	664.80nm W/L	0.815nm Abs.
7	Back	White afara	664.50nm W/L	0.221nm Abs.
8	Root	White afara	663.60nm W/L	0.270nm Abs.

Sources: Results analysis by the researcher (2013)

Table 4.3: Wash fastness rating of sodium chloride with various timing on cotton fabric.

S/N	MORDANT	TYPE OF DYES	120 MINS		100 MINS		80 MINS		60 MINS		40 MINS		20 MINS	
			CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC
1.	Sodium Chloride	TLT	4	4	4	3	4	3 – 4	4	2 – 3	4	3	4	2 – 3
2.	Sodium Chloride	TBT	4	4	4	3 – 4	4	3 – 4	4	4	4	4	4	4
3.	Sodium Chloride	TSL	3 – 4	2	2 – 3	2	4	3 – 4	4	2 – 3	4	2	3	2
4.	Sodium Chloride	TBL	4 – 5	4	4	4 – 5	4	4	4	4 – 5	4 – 5	4	4	4
5.	Sodium Chloride	TBW	2 – 3	4	2 – 3	3 – 4	4	4	2 – 3	2 – 3	4 – 5	4 – 5	4	4
6.	Sodium Chloride	TRW	4	2 – 3	3	3 – 4	4	3	3 – 4	3	3 – 4	3	3 – 4	3
7.	Sodium Chloride	TLW	4	4	2 – 3	4	3	4	3	3 – 4	3 – 4	3 – 4	3	3
8.	Sodium Chloride	TLL	4	4	4	4	4 – 5	4	4	4	4	4	4	4

Sources: Results analysis by the researcher (2013)

Table 4.4: wash fastnesses rating of mordants on cotton fabric for 2 hours each (120 mins.)

	MORDANT	ALUM (POTASH)		WOOD ASH		IRON II SULPHATE		POTASIUM DICROMATE		COPPER SULPHATE		SODIUM CHLORIDE	
S/N		CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC
1.	TLT	4 – 5	4	4	2 – 3	3 – 4	3 – 4	4 – 5	4	2 – 3	2 – 3	4	4
2.	TBT	4 – 5	2	2 – 3	4	2 – 3	3 – 4	4 – 5	4	4	4	4	4
3.	TSL	4	2	2 – 3	4	4	3 – 4	4 – 5	4 – 5	3 – 4	3 – 4	3 – 4	2
4.	TBL	4 – 5	4	4	4	4	4	2 – 3	4	4	4	4 – 5	4
5.	TBW	4 – 5	4	4	4	4 – 5	3 – 4	4	4	4	4 – 5	2 – 3	4
6.	TRW	4 – 5	4 – 5	3 – 4	3	4	3 – 4	3 – 4	3	2	4	4	2 – 3
7.	TLW	4 – 5	4 – 5	2 – 3	4	4	3 – 4	4 – 5	4	3 – 4	3 – 4	4	4
8.	TLL	4 – 5	3 – 4	4	4	4	4	4 – 5	4 – 5	3 – 4	3 – 4	4	4

CC: Change in shade (colour change) **CS:** Standing on adjacent fabric (colour stain)

Sources: Results analysis by the researcher (2013)

Table 4.5: Acids and alkalin perspiration rating of sodium chloride on cotton fabric with various timing.

S/N	MORDANT	TYPE OF DYES	120 MINS				100 MINS				80 MINS				60 MINS				40 MINS				20 MINS			
			ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE	
			CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC
1	Sodium Chloride	TLT	4	4	4	4	4-5	4	4	4	4	4	4	4	4	4	3-4	3	4	4	3	4	3-4	4	4	4
2	Sodium Chloride	TBT	4	4 – 5	4	4-5	4	4-5	4	3-4	3-4	4	4	4	4	4-5	4	4-5	4	4	4	4-5	4	4	4	4 – 5
3	Sodium Chloride	TSL	3 – 4	4	2 – 3	4	4	4-5	3-4	4-5	4	4	3-4	4	3-4	4	3-4	3-4	4	4-5	3-4	4-5	4	4-5	4	4 – 5
4	Sodium Chloride	TBL	4 – 5	4 – 5	4 – 5	4-5	4-5	4	4-5	4	4-5	4	4-5	4	4-5	3-4	4-5	3-4	4-5	4	4-5	4	4-5	4	4-5	4
5	Sodium Chloride	TBW	3 – 4	3	3	3-4	3-4	3-4	3-4	4	4	4	4	4	4	4	4	4	4	4	4	4	4-5	4-5	4-5	4 – 5
6	Sodium Chloride	TRW	3 – 4	3	4	3-4	4	3-4	3-4	4	3	3	2-3	4	3-4	3-4	4	3-4	3	3	3-4	4	3	3-4	3	4
7	Sodium Chloride	TLW	3	2 – 3	3	3	4	3-4	3-4	3-4	3	4	4	4	4	3	4	4	3-4	3-4	4	4	3	3-4	4-5	4 – 5
8	Sodium Chloride	TLL	4 – 5	4	4 – 5	4	4-5	4	4-5	4	4	4	4	4	4-5	4	4-5	4	4-5	4	4	4	4-5	4-5	4-5	4 – 5

Sources: Results analysis by the researcher (2013)

Table 4.6: Acids and alkaline perspiration rating of dyes on cotton for 2 hours each (120 mins)

	MORDANT	ALUM (POTASH)				WOOD ASH				IRON II SULPHATE				POTASSIUM DICROMATE				COPPER II SULPHATE				SODIUM CHLORIDE			
		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE		ACID		ALKALINE	
		CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC
1	TLT	4	4	4–5	4	4	4– 5	4	4	4-5	4-5	4-5	4 – 5	4 – 5	4	4 – 5	4	4	3-4	4	3-4	4	4	4	4
2	TBT	4 – 5	2	4–5	2-3	4	3– 4	4	3-4	4	4	4	4	4 – 5	4-5	4 – 5	4-5	4	4-5	4	4-5	4	4-5	4 – 5	4
3	TSL	4 – 5	4– 5	4	4–5	4 –5	4	4– 5	4	3-4	3-4	3-4	3 – 4	4 – 5	4-5	4 – 5	4-5	4	4-5	4	4	4	2– 3	4	4
4	TBL	4 – 5	4	4–5	4–5	4 – 5	4	4-5	4	4-5	4	4-5	4	4 – 5	4	4 – 5	4	4	4	4	4	4-5	4-5	4 – 5	4 – 5
5	TBW	4 – 5	3– 4	4–5	3–4	4 – 5	4	4-5	4	4-5	3- 4	4-5	4	4 – 5	4	4 – 5	4	4	4-5	4	4-5	3-4	3	3	3
6	TRW	4 – 5	5	5	5	4	3– 4	4	3-4	4-5	4-5	4-5	4 – 5	4	4-5	4	4-5	4	3-4	4	3-4	3-4	3	4	3 – 4
7	TLW	4	4	4–5	4–5	4	4	4	4	4-5	4-5	4-5	4 – 5	4 – 5	4	4 – 5	4	4	3-4	4	4-5	3	2-3	3	3
8	TLL	5	5	5	5	4	4– 5	4-5	4	4-5	4	4-5	4	4 – 5	4-5	4 – 5	4-5	4	4	4	4	4-5	4	4 – 5	4

Sources: Results analysis by the researcher (2013)

Table 4.7: Light fastness rating on degree of fading compared with the wool standards: sodium chloride with various timing

S/N	MORDANT	TYPE OF DYES	120 MINS	100 MINS	80 MINS	60 MINS	40 MINS	20 MINS
1.	Sodium Chloride	TLT	6	5	5	5	5	3
2.	Sodium Chloride	TBT	5	5	5	4	4	4
3.	Sodium Chloride	TSL	3	3	3	3	3	3
4.	Sodium Chloride	TBL	7	7	6	6	6	6
5.	Sodium Chloride	TBW	7	7	6	6	6	6
6.	Sodium Chloride	TRW	7	7	7	7	7	6
7.	Sodium Chloride	TLW	6	6	6	6	7	7
8.	Sodium Chloride	TLL	7	7	7	7	7	7

Sources: Results analysis by the researcher (2013

Table 4.8: Light fastness ratings on degree of mordants fading compared with wool standards for 2hrs

\	**Mordants**	**Alum (Potash)**	**Wood Ash**	**Iron II Sulphate**	**Potasium Dicromate**	**Copper II Sulphate**	**Sodium Chloride**
S/N	**DYES**						
1.	TLT	3	3	5	5	3	7
2.	TBT	3	3	5	5	3	7
3.	TSL	2	3	5	5	3	7
4.	TBL	4	7	6	6	3	7
5.	TBW	6	3	5	5	5	7
6.	TRW	8	7	5	6	7	6
7.	TLW	4	3	5	5	7	7
8.	TLL	5	7	7	5	3	7

Table 4.9 Sample of quality control sheet: African Textile Kano

AFRICAN TEXTILE MANUFACTURERS LTD
QUALITY CONTROL SHEET

Design No..... Meters Printed..... Date.....

Colour Way

Standard sample	Covered sample	Uncovered sample	Washed White	Standard White	Ironing	Rubbing	Perspiration
					Dry	Dry	Acid
					Damp		Alkali
						Wet	
					Wet		Washing Fastness..... Ironing..... Rubbing..... Perspiration..... Light Fastness.....

Processing Mgr. Quality Control Officer Remark

Source: African Textile Kano (2013)

Table 4.10A: Samples of dye fabrics rating: wash fastness and perspiration tests. Serial No 1-89

.2

WASH FASTNESS TEST ON COTTON FABRIC FOR 2 HOURS EACH (120 MINUTES)

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
1	TLT with Alum							
2	TBT with Alum							
3	TSL with Alum							
4	TBL with Alum							
5	TBW with Alum							
6	TRW with Alum							
7	TLL with Alum							
8	TLW with Alum							

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

WASH FASTNESS TEST ON COTTON FABRIC FOR 2 HOURS EACH (120 MINUTES)

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
9	TRW with Wood ash							
10	TBW with Wood ash							
11	TLW with Wood ash							
12	TBT with Wood ash							
13	TLT with Wood ash							
14	TSL with Wood ash							
15	TBL with Wood ash							
16	TLL with Wood ash							

Source: Original fabric samples for fastness test by the researcher (2013)

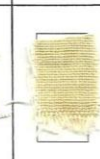
WASH FASTNESS TEST ON COTTON FABRIC FOR 2 HOURS EACH (120 MINUTES)

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
17	TBT with Iron II Sulphate							
18	TBL with Iron II Sulphate							
19	TLL with Iron II Sulphate							
20	TRW with Iron II Sulphate							
21	TLW with Iron II Sulphate							
22	TLT with Iron II Sulphate							
23	TSL with Iron II Sulphate							
24	TBW with Iron II Sulphate							

Source: Original fabric samples for fastness test by the researcher (2013)

4.2

WASH FASTNESS TEST ON COTTON FABRIC FOR 2 HOURS EACH (120 MINUTES)

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
25	TLL with Potassium dichromate							
26	TLW with Potassium dichromate							
27	TBT with Potassium dichromate							
28	TSL with Potassium dichromate							
29	TBW with Potassium dichromate							
30	TRW with Potassium dichromate							
31	TBL with Potassium dichromate							
32	TLT with Potassium dichromate							

Original fabric samples for fastness test by the researcher (2013)

WASH FASTNESS TEST ON COTTON FABRIC FOR 2 HOURS EACH (120 MINUTES)

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
33	TBW with Copper II Sulphate							
34	TRW with Copper II Sulphate							
35	TLW with Copper II Sulphate							
36	TLT with Copper II Sulphate							
37	TLL with Copper II Sulphate							
38	TBT with Copper II Sulphate							
39	TSL with Copper II Sulphate							
40	TBL with Copper II Sulphate							

Original fabric samples for fastness test by the researcher (2013)

4.1

WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
41	TBW with Sodium chloride (20mins)							
42	TBW with Sodium chloride (40mins)							
43	TBW with Sodium chloride (60mins)							
44	TBW with Sodium chloride (80mins)							
45	TBW with Sodium chloride (100mins)							
46	TBW with Sodium chloride (2hrs)							
47	TBW with Sodium chloride (20mins)							
48	TBW with Sodium chloride (40mins)							

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
49	TLW with Sodium chloride (60mins)							
50	TLW with Sodium chloride (80mins)							
51	TLW with Sodium chloride (100mins)							
52	TLW with Sodium chloride (2 hrs)							
53	TLW with Sodium chloride (20mins)							
54	TLW with Sodium chloride (40mins)							
55	TLW with Sodium chloride (60mins)							
56	TLW with Sodium chloride (80mins)							

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

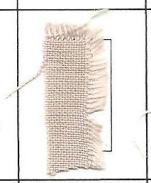
WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
57	TRW with Sodium chloride (2hrs)							
58	TSL with Sodium chloride (20mins)							
59	TSL with Sodium chloride (40mins)							
60	TSL with Sodium chloride (60mins)							
61	TSL with Sodium chloride (80mins)							
62	TLT with Sodium chloride (20mins)							
63	TLT with Sodium chloride (40mins)							
64	TLT with Sodium chloride (60mins)							

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

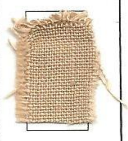
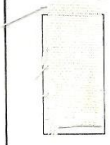
WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
65	TLT with Sodium chloride (80mins)							
66	TRW with Sodium chloride (80mins)							
67	TRW with Sodium chloride (2hrs)							
68	TSL with Sodium chloride (2hrs)							
69	TSL with Sodium chloride (80mins)							
70	TLT with Sodium chloride (80mins)							
71	TLT with Sodium chloride (40mins)							
72	TBT with Sodium chloride (20mins)							

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

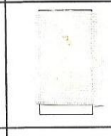
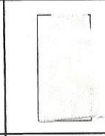
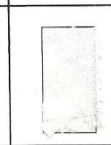
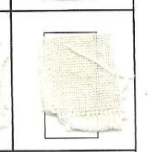
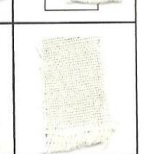
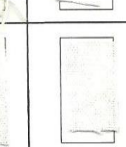
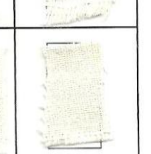
WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
73	TBT with Sodium chloride (40mins)							
74	TBT with Sodium chloride (60mins)							
75	TBT with Sodium chloride (80mins)							
76	TBT with Sodium chloride (100mins)							
77	TBT with Sodium chloride (2hrs)							
78	TLL with Sodium chloride (20mins)							
79	TLL with Sodium chloride (40mins)							
80	TLL with Sodium chloride (60mins)							

Source: Original fabric samples for fastness test by the researcher (2013)

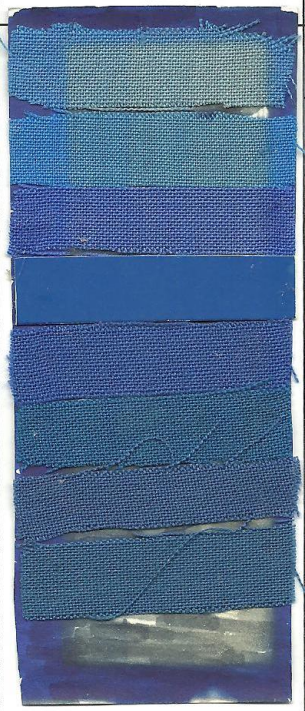
Table 4.1

WASH FASTNESS OF SODIUM CHLORIDE WITH VARIOUS TIMING ON COTTON FABRIC

S/N	Description of samples	Change in Colour (CC)			Staining Test		Perspiration	
		Standard Sample	Covered Sample	Uncovered Sample	Wash White	Standard White	Acid	Alkaline
81	TLLwith Sodium chloride (80mins)							
82	TLL with Sodium chloride (100mins)							
83	TLL with Sodium chloride (2hrs)							
84	TBL with Sodium chloride (20mins)							
85	TBL with Sodium chloride (40mins)							
86	TBL with Sodium chloride (60mins)							
87	TBL with Sodium chloride (80mins)							
88	TBL with Sodium chloride (100mins)							
89	TBL with Sodium chloride (2hrs)							

Source: Original fabric samples for fastness test by the researcher (2013)

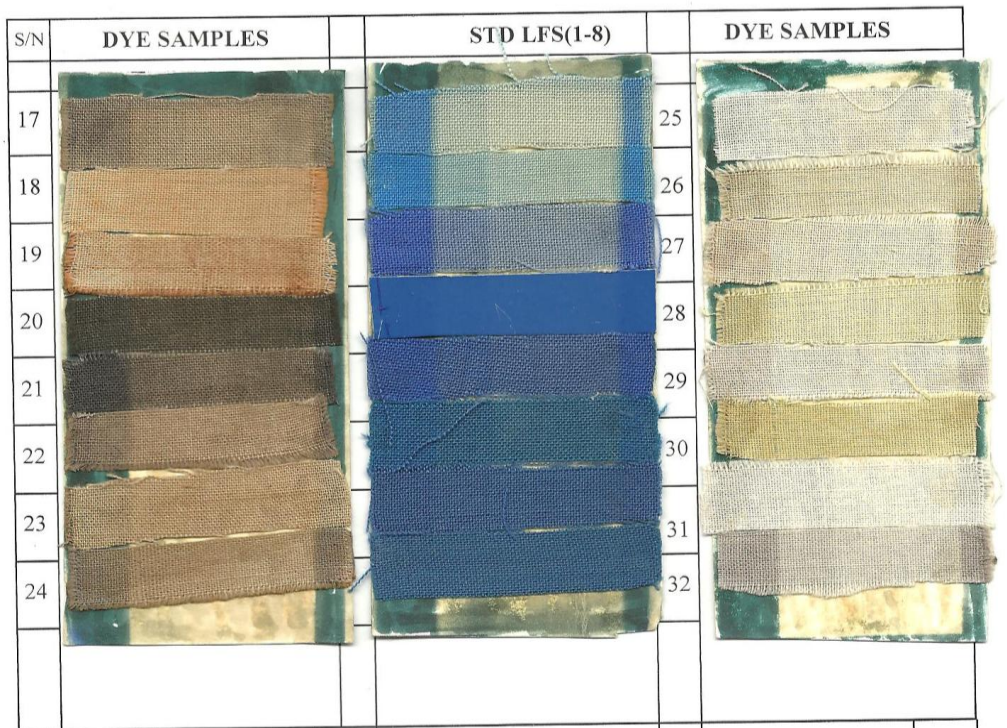
Table 4.10B: Samples of dye fabrics rating: **Light fastness test. Serial No 1-89.**

S/N	DYE SAMPLES		STD LFS(1-8)		DYE SAMPLES
					
1				9	
2				10	
3				11	
4				12	
5				13	
6				14	
7				15	
8				16	

S/N	Description of Sample	Rating	Humidity - 65^0	S/N	Description of Sample	Rating
1	TLT with Alum	6		9	TRW with wood ash	7
2	TBT with Alum	4		10	TWB with wood ash	3
3	TSL with Alum	2		11	TLW with wood ash	3
4	TBL with Alum	4		12	TBT with wood ash	3
5	TBW with Alum	6		13	TLT with wood ash	3
6	TRW with Alum	8		14	TSL with wood ash	4
7	TLL with Alum	5		15	TBL with wood ash	7
8	TLW with Alum	4		16	TLL with wood ash	7

Source: Original fabric samples for fastness test by the researcher (2013)

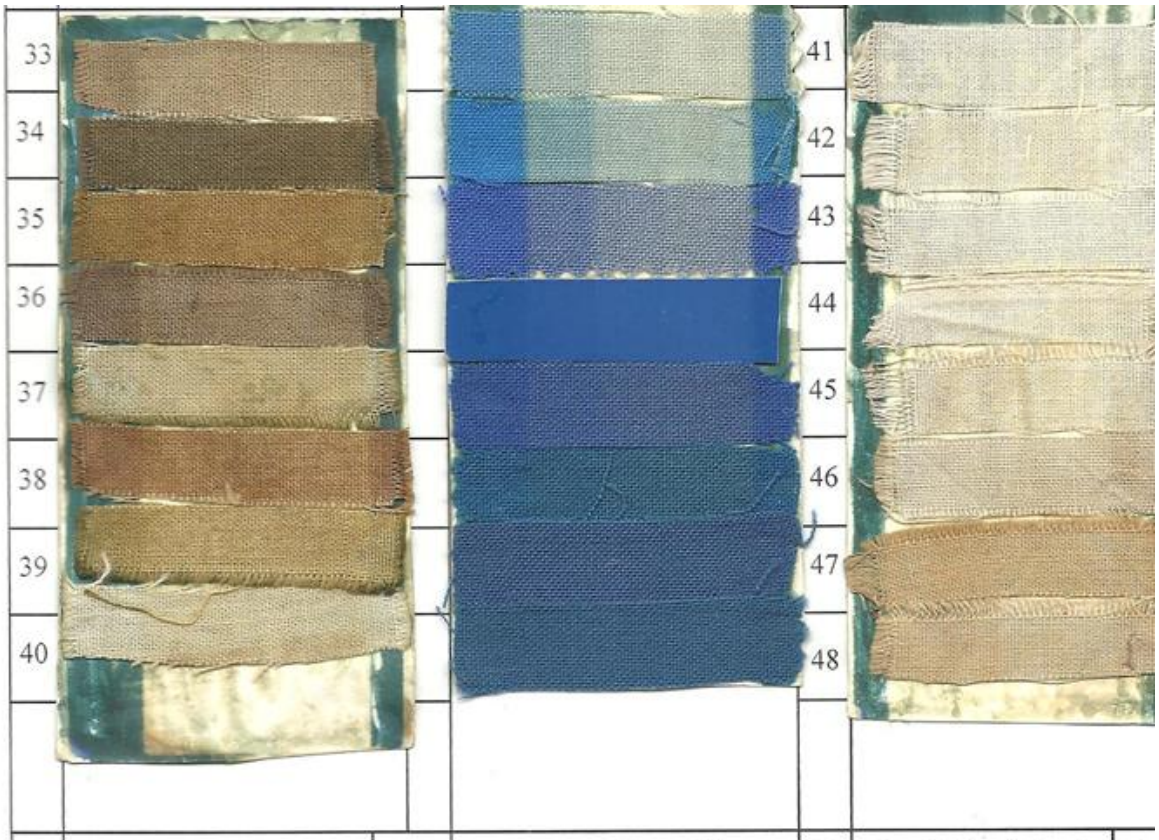
Continuation



S/N	Description of Sample	Rating	Humidity - 65^0	S/N	Description of Sample	Rating
17	TBT with Iron II Sulphate	5		25	TLL with Potassium dichromate	5
18	TBT with Iron II Sulphate	6		26	TLW with Potassium dichromate	5
19	TLL with Iron II Sulphate	7		27	TBT with Potassium dichromate	5
20	TRW with Iron II Sulphate	5		28	TSL with Potassium dichromate	5
21	TLW with Iron II Sulphate	5		29	TBW with Potassium dichromate	5
22	TLT with Iron II Sulphate	5		30	TRW with Potassium dichromate	6
23	TSL with Iron II Sulphate	5		31	TBL with Potassium dichromate	6
24	TBW with Iron II Sulphate	5		32	TLT with Potassium dichromate	5

Source: Original fabric samples for fastness test by the researcher (2013)

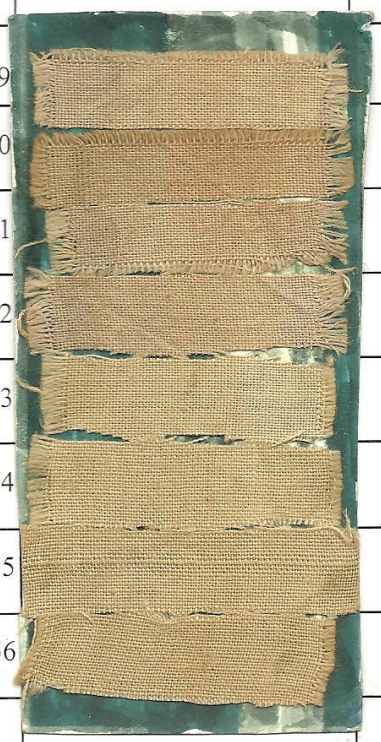
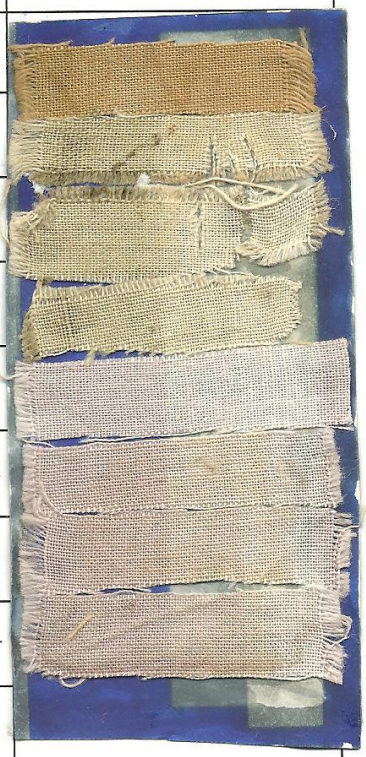
ontinuation



S/N	Description of Sample	Rating	Humidity - 65°	S/N	Description of Sample	Rat
33	TWB with copper II Sulphate	5		41	TBW with Sodium chloride (20mins)	6
34	TRW with copper II Sulphate	7		42	TBW with Sodium chloride (40mins)	6
35	TLW with copper II Sulphate	7		43	TBW with Sodium chloride (60mins)	6
36	TLT with copper II Sulphate	3		44	TBW with Sodium chloride (80mins)	6
37	TLL with copper II Sulphate	3		45	TBW with Sodium chloride (100mins)	6
38	TBT with copper II Sulphate	3		46	TBW with Sodium chloride (2hrs)	7
39	TSL with copper II Sulphate	3		47	TLW with Sodium chloride (20mins)	7
40	TBL with copper II Sulphate	3		48	TLL with Sodium chloride (40mins)	7

Source: Original fabric samples for fastness test by the researcher (2013)

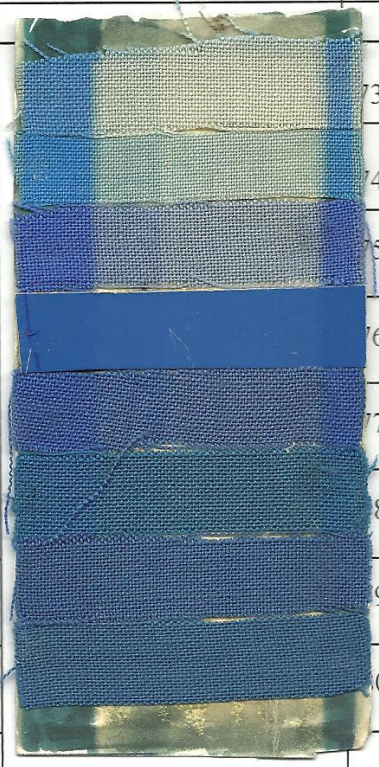
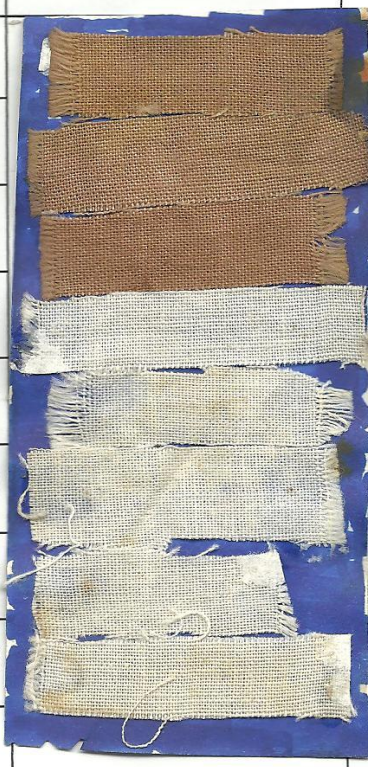
Continuation

S/N	DYE SAMPLES	STD LFS(1-8)	DYE SAMPLES
49			
50			
51			
52			
53			
54			
55			
56			

S/N	Description of Sample	Rating	Humidity - 65^0	S/N	Description of Sample	Rating
49	TLW with sodium chloride (60mins)	6		57	TRW with sodium chloride (2hrs)	6
50	TLW with sodium chloride (80mins)	6		58	TSL with sodium chloride (20mins)	3
51	TLW with sodium chloride (100mins)	6		59	TSL with sodium chloride (40mins)	3
52	TLW with sodium chloride (2hrs)	6		60	TSL with sodium chloride (60mins)	3
53	TRW with sodium chloride (20mins)	7		61	TSL with sodium chloride (80mins)	3
54	TRW with sodium chloride (40mins)	7		62	TLT with sodium chloride (20mins)	3
55	TRW with sodium chloride (60mins)	7		63	TLT with sodium chloride (40mins)	5
56	TRW with sodium chloride (80mins)	7		64	TLT with sodium chloride (60mins)	5

Source: Original fabric samples for fastness test by the researcher (2013)

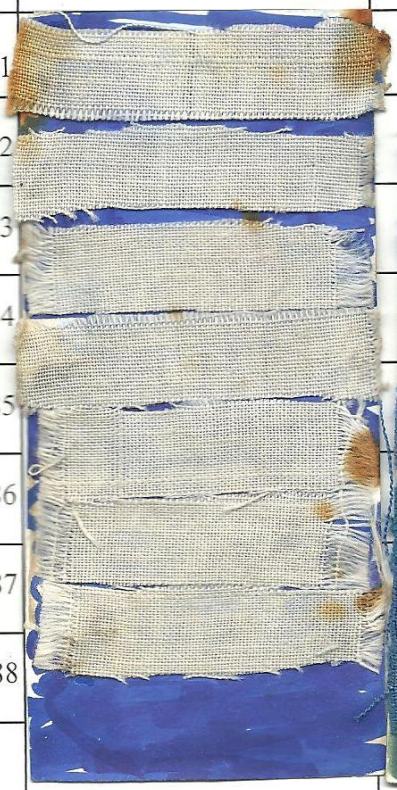
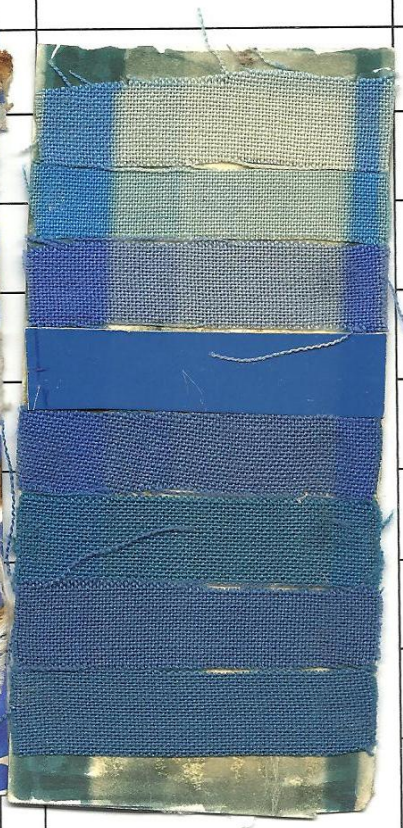
Continuation

S/N	DYE SAMPLES		STD LFS(1-8)		DYE SAMPLES
65				73	
66				74	
67				75	
68				76	
69				77	
70				78	
71				79	
72				80	

S/N	Description of Sample	Rating	Humidity - 65^0	S/N	Description of Sample	Rating
65	TLT with sodium chloride (80mins)			73	TBT with sodium chloride (40mins)	4
66	TRW with sodium chloride (80mins)	5		74	TBT with sodium chloride (60mins)	4
67	TRW with sodium chloride (2hrs)			75	TBT with sodium chloride (80mins)	5
68	TSL with sodium chloride (2hrs)	3		76	TBT with sodium chloride (100mins)	5
69	TSL with sodium chloride (80mins)	3		77	TBT with sodium chloride (2hrs)	5
70	TLT with sodium chloride (80mins)	5		78	TLL with sodium chloride (20mins)	7
71	TLT with sodium chloride (40mins)	6		79	TLL with sodium chloride (40mins)	7
72	TBT with sodium chloride (20mins)	4		80	TLL with sodium chloride (60mins)	7

Source: Original fabric samples for fastness test by the researcher (2013)

Continuation

S/N	DYE SAMPLES		STD LFS(1-8)		DYE SAMPLES
81				89	
82					
83					
84					
85					
86					
87					
88					

S/N	Description of Sample	Rating	Humidity - 65^0	S/N	Description of Sample	Rating
81	TLLwith sodium chloride (80mins)	7		89	TBL with sodium chloride (2hrs)	7
82	TLL with sodium chloride (100mins)	7				
83	TLL with sodium chloride (2hrs)	7				
84	TBL with sodium chloride (20mins)	7				
85	TBL with sodium chloride (40mins)	7				
86	TBL with sodium chloride (60mins)	7				
87	TBL with sodium chloride (80mins)	7				
88	TBL with sodium chloride (100mins)	7				

Picture plates



(Plate.2.2) *Jeans. Source: James Elkin (2000) Amazon.com*



(Plate 2.3) The Blue dyestuffs used for dyeing Jeans. The ubiquitous blue of indigo is as familiar as blue jeans. Source: James Elkin (2000) Amazon.com.



(Plate 2.4) Colouring yarn
Source: James Elkin (2000) Amazon.com.



(Plate 2.5 a) (Plate2.5 .) (Plate2.5, c)*Flowering herb.*
Flowering herb.Floweringherb.
Sources: James Elkin (2000) Amazon.com.



(Plate.2.6) *Herb*
Sources: James Elkin (2000) Amazon.com.



(Plate.2.7) *Shades of Yellow/Wheat*
Sources: James Elkin (2000) Amazon.com.



(fig 3.1) *Map of the research study areas, town Lokoja in Kogi State. Source.*www.business-nigeria.com/map. www.worldatlas.com.

Field Works









Plate 3.1: Harvesting: (a) Mount Patti Forest Reserve (b) Locust beans leaves (c) White Afara roots (d) Back of white Afara (e) The seed of Locust beans (f) Teak leaves.
Source: Researcher at field works (2013)



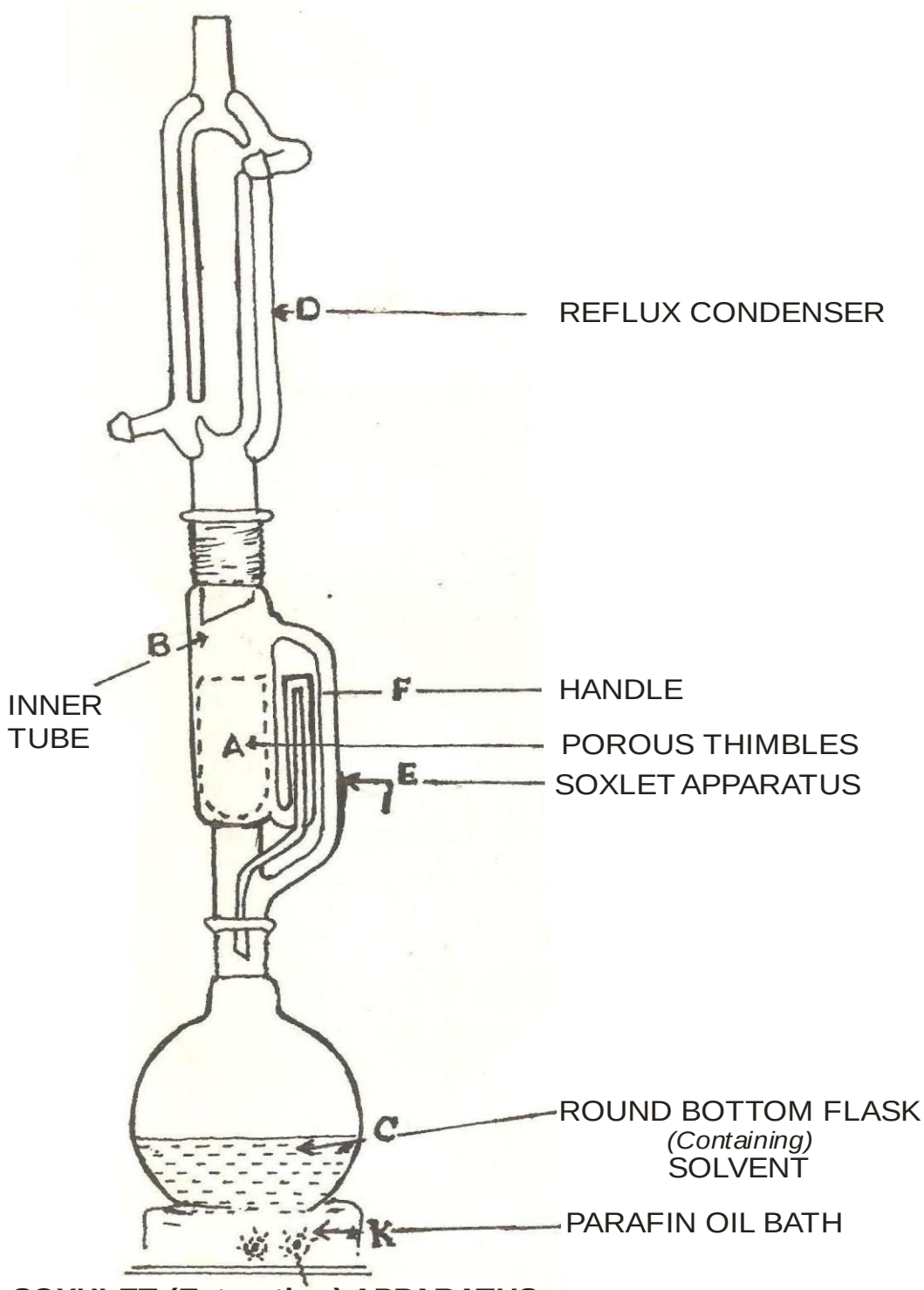
Plate 3.2: Cutting (a) The back of Teak (b) The back of Locust beans (c) The back of White Afara
Source: Researcher at field works (2013}



Plate 3.3: Drying (a) Leaves of Teak. (b) Root of White Afara (c) The Seed of locust beans
Source: Researcher at field works (2013)



Plate 3.4: Pounding and Grinding (a) Leaves of White Afara (b) Root of White Afara (c) Leaves of Locust beans (d) Leave of Teak (e) Back of Teak (f) Back of White Afara.
Source: Researcher at the labouratory (2013)



SOXHLET (Extraction) APPARATUS

Fig 3.2.1: Adopted from Gwari (1989) p 56.

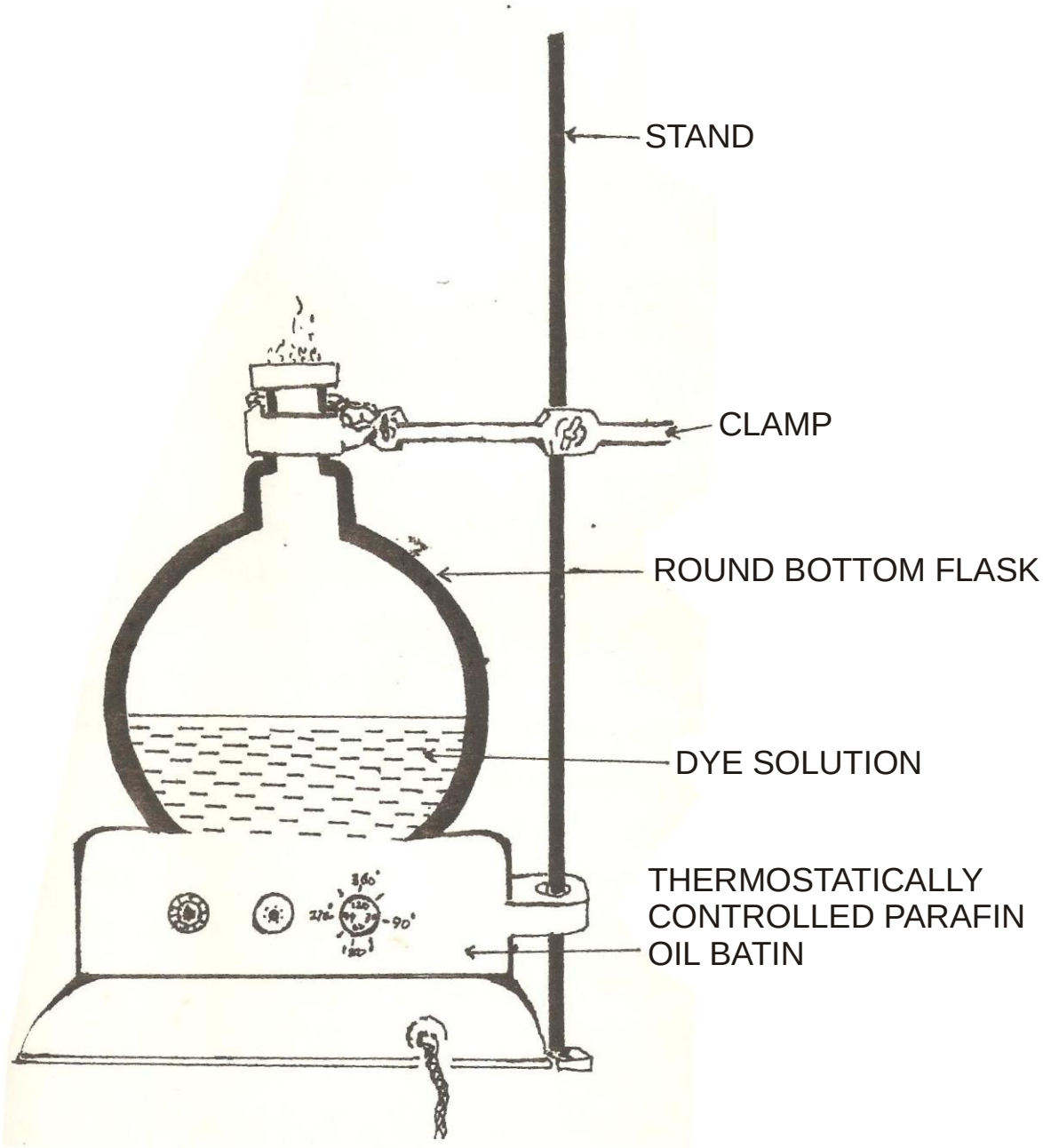


Fig 3.2.2 Adopted from Gwari,(1989) p.59.

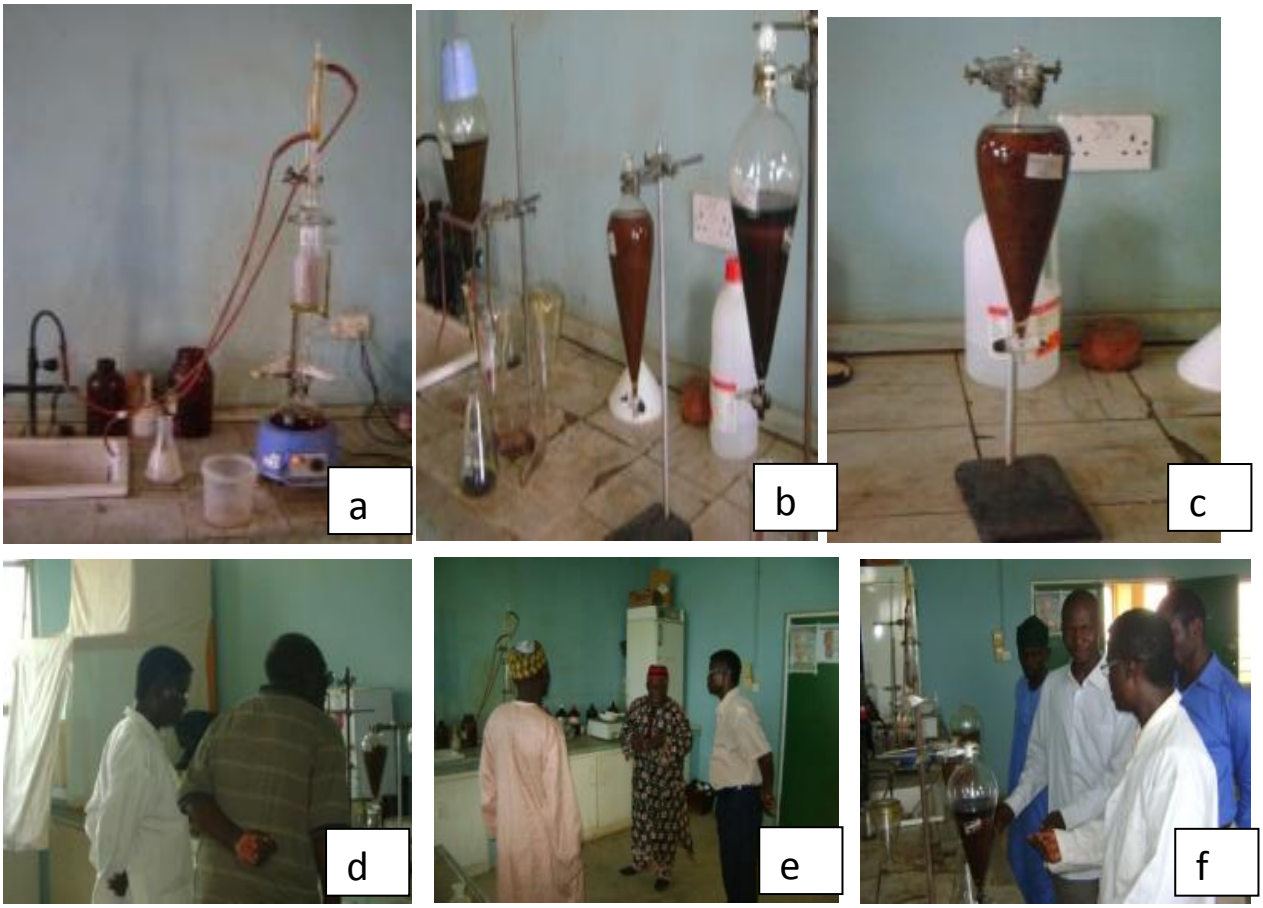


Plate 3.5: Extraction of dye techniques (a) Soxhlet technique (b) Maceration method (c) Extraction of Afara(d) Mr. Bobzon observing the extraction process (e) Dr. Ada and Mal. Adamu supervising the extractionprocesses. (f) Technical staff assisting in the extraction processess.
Source: Researcher at the labouratory (2013)



Plate 3.6: Removal of Liquid dye extractions (a) Liquid extraction (b) Electric Baths for evaporation of liquid (c) Powdered Dye.
Source: Researcher at the labouratory (2013)

UV SPECTRAL SCAN RESULTS

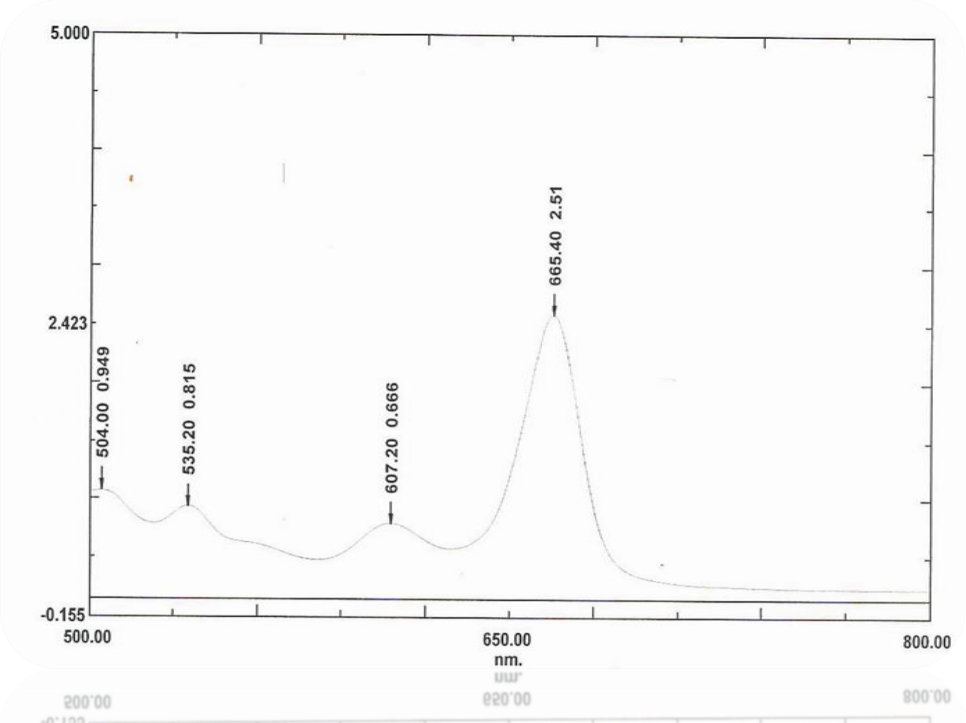


Figure3.3.1: Determination of the maximum absorption of spectra (λ max) of the dye (Leave of Locust Beans)

Source: Researcher Graph results on dye extracted (2013)

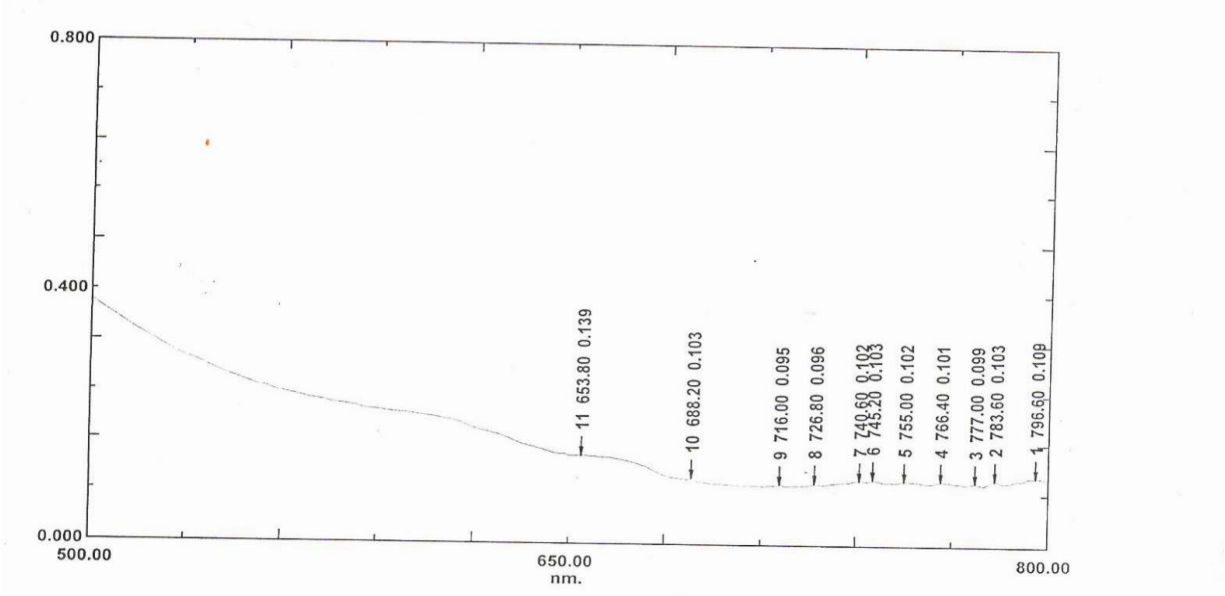


Figure3.3.2: Determination of the maximum absorption of spectral (λmax) of the dye (Back of Locust Beans)

Source: Researcher Graph results on dye extracted (2013)

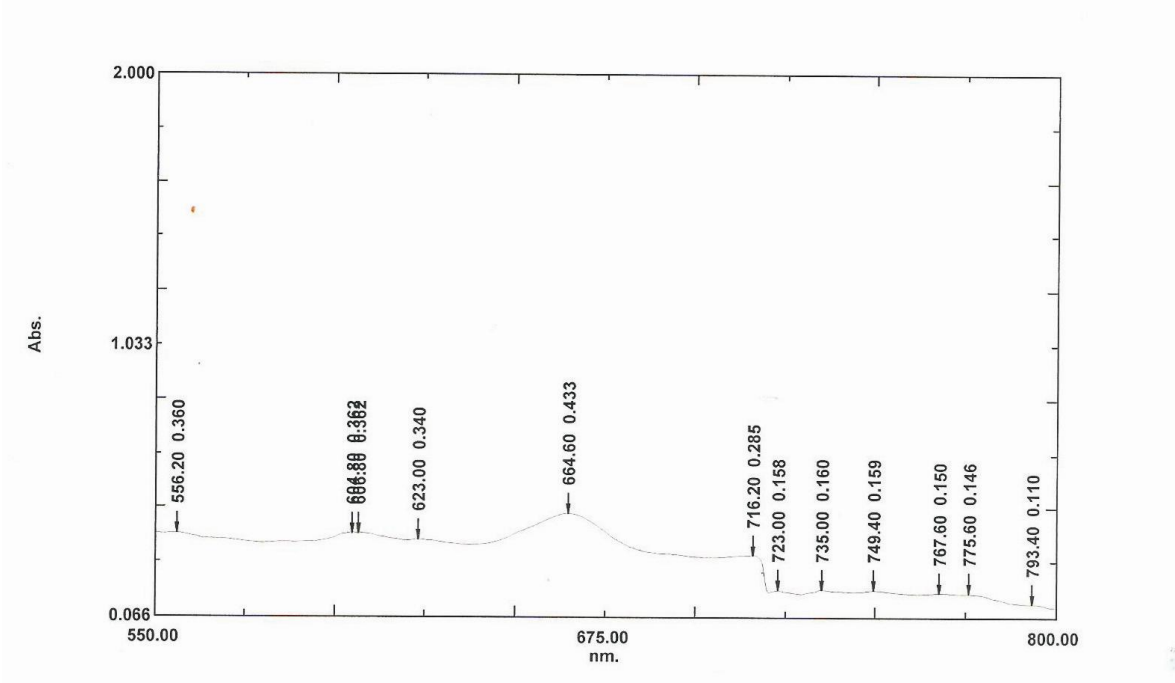


Figure3.3.3: Determination of the maximum absorption of spectral (λmax) of the dye (Locust Beans Seed)
Source: Researcher Graph results on dye extracted (2013)

,

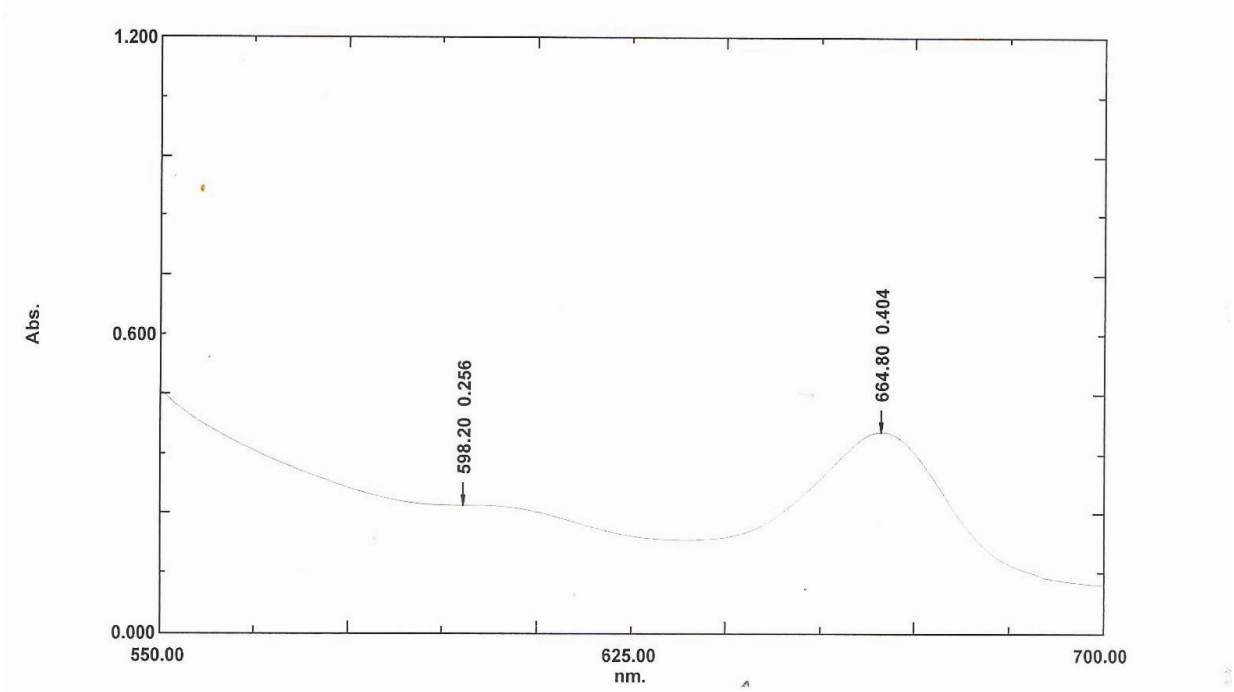


Figure3.3.4: Determination of the maximum absorption of spectral (λmax) of the dye (Leave of Teak)
Source: Researcher Graph results on dye extracted (2013)

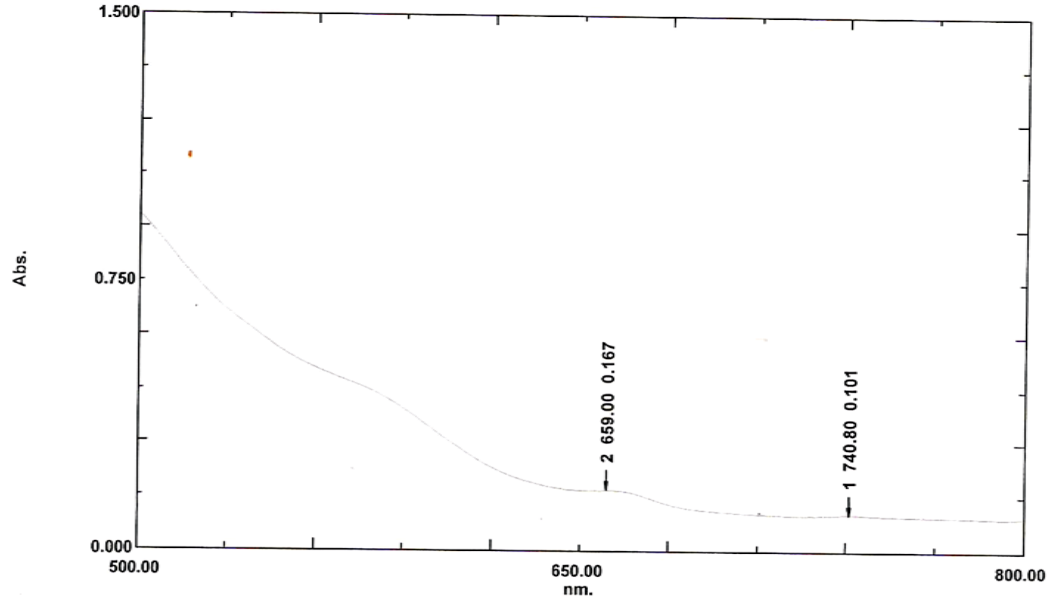


Figure3.3.5: Determination of the maximum absorption of spectral (λmax) of the dye (Back of Teak)

Source: Researcher Graph results on dye extracted (2013)

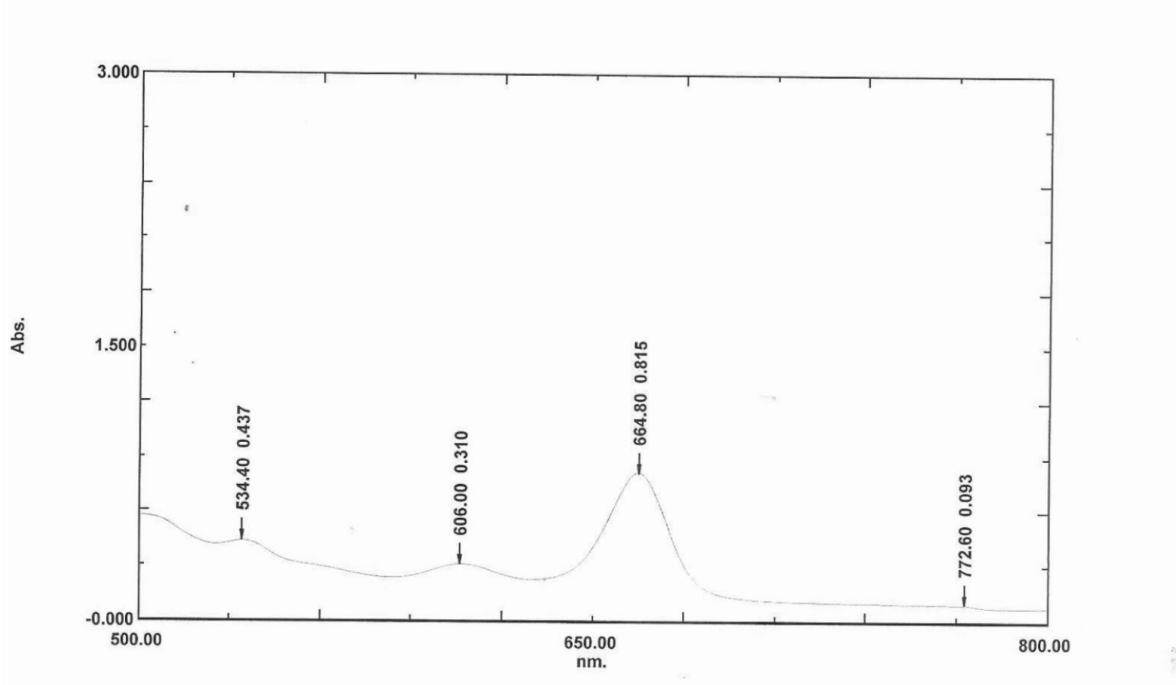


Figure3.3.6: Determination of the maximum absorption of spectral (λmax) of the dye (Leaf of White Afara)

Source: ResearcherGraph results on dye extracted (2013)

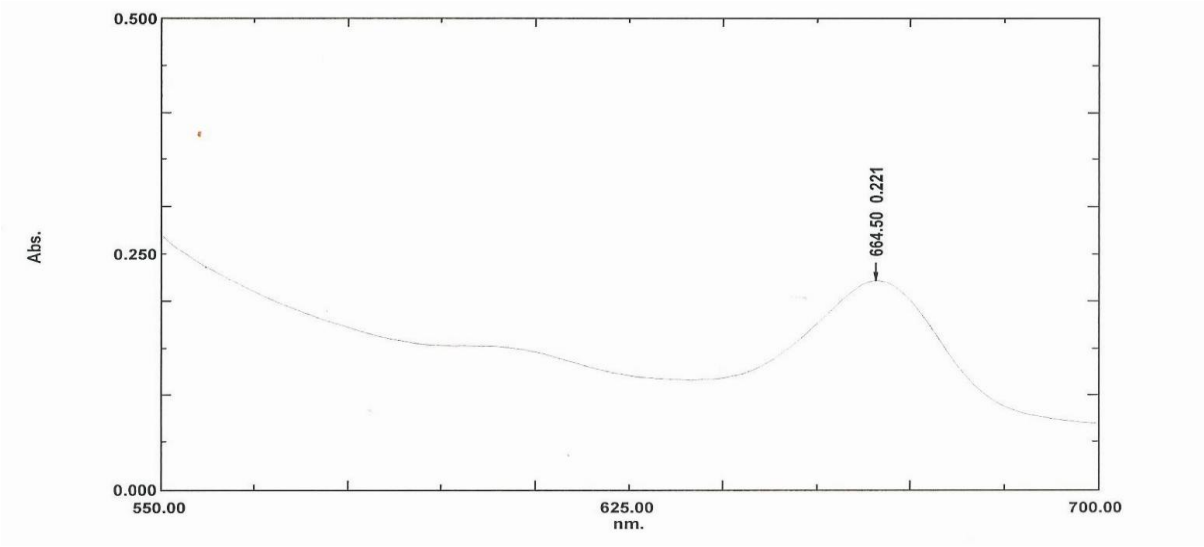


Figure3.3.7: Determination of the maximum absorption of spectral (λmax) of the dye (Back of White Afara)

Source: Researcher Graph results on dye extracted (2013)

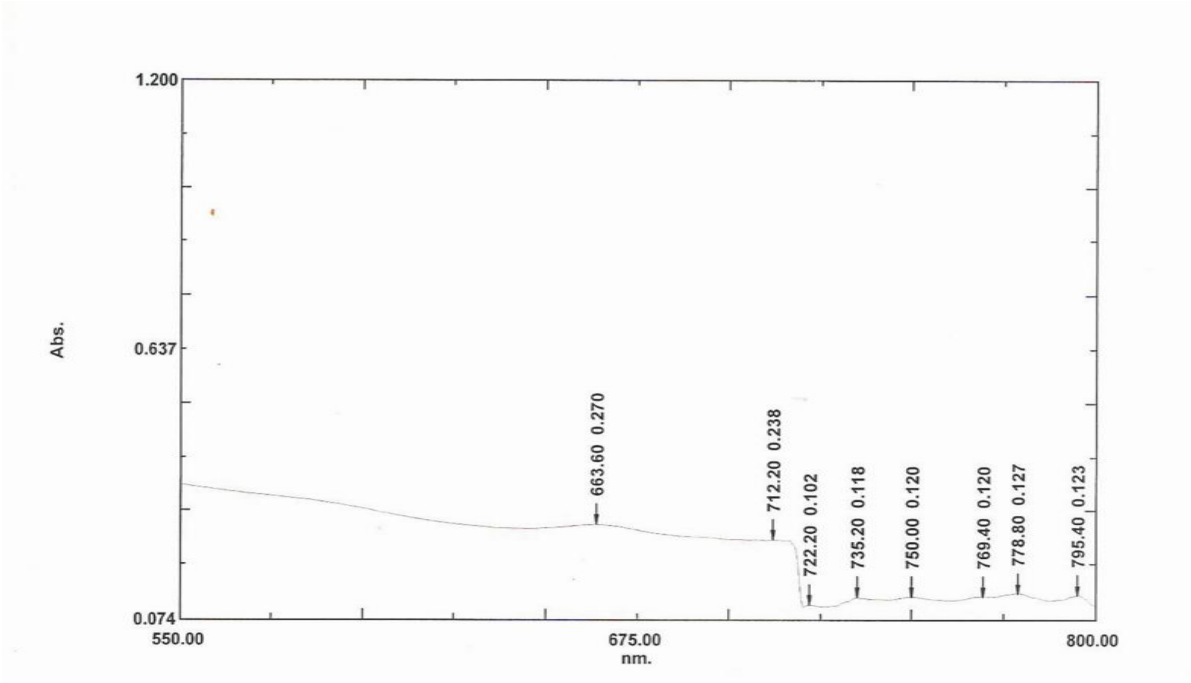


Figure3.3.8: Determination of the maximum absorption of spectral (λmax) of the dye (Root of White Afara)

Source: ResearcherGraph results on dye extracted (2013)

Dyeing processes:

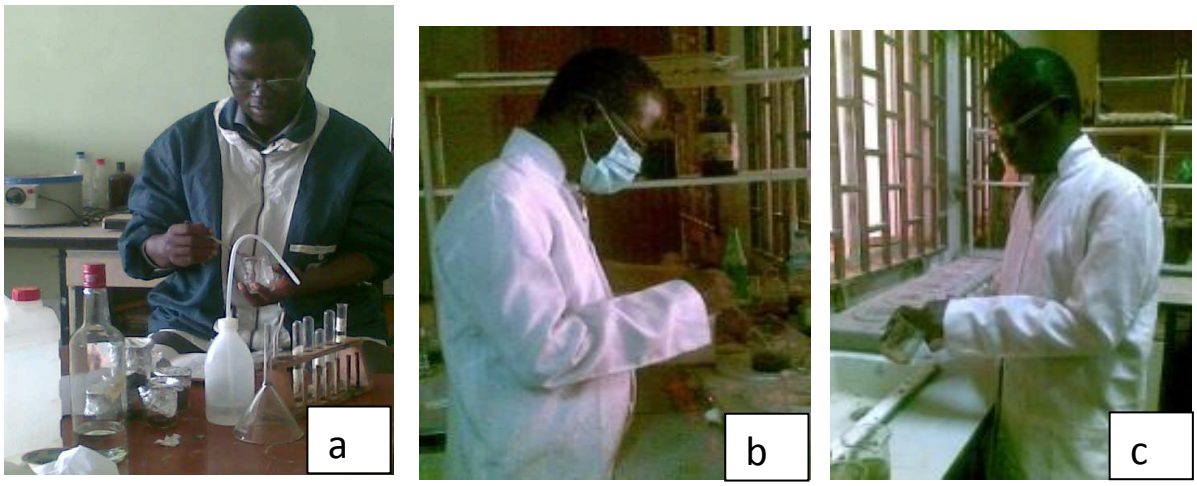


Plate 3.7: Dyeing processes: (a) Solubility text. (b) Dyeing of fabrics. (c) Rinsing of dye fabrics.
Source: Researcher at the labouratory (2013)

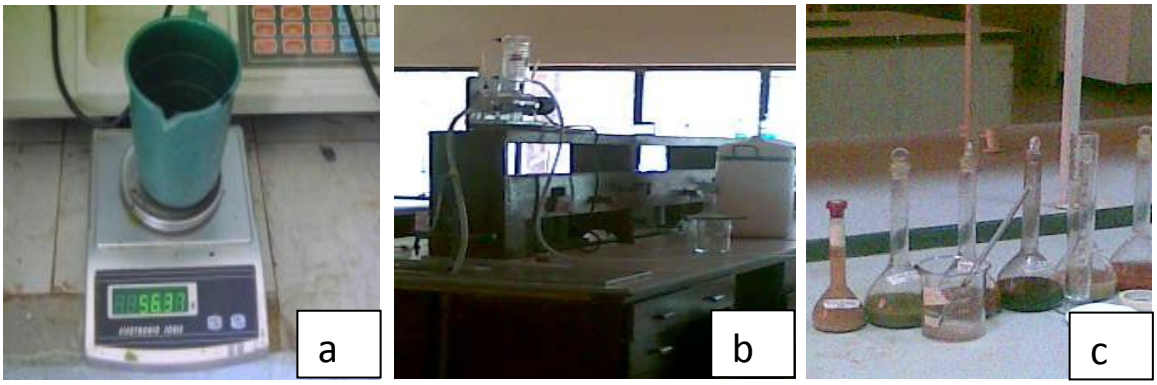
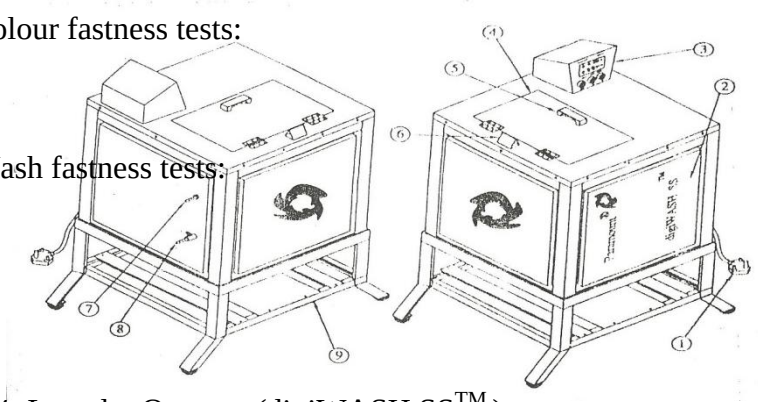


Plate 3.8: Equipment: (a) Weighing scale (b) Distilled water processes (c) Conical flasks containingprocessed dyes.
Source: Researcher at the labouratory (2013)

Colour fastness tests:

Wash fastness tests:



3.4: Launder O-meter (digiWASH SS^{TM})
Source: Paramount since 1964 ISO certified company. (User's manual)

(1.) Main lead with plug. (2.) Side cover 04 Nos. (3.) Control panel box. (4.) Door. (5.) Door handle. (6.) Door supporter. (7.) Over flow nozzle. (8.) Inlet/outlet valve (on both above valves the plastic tubing supplied is to be fitted) (9.) Stainless steel stand.

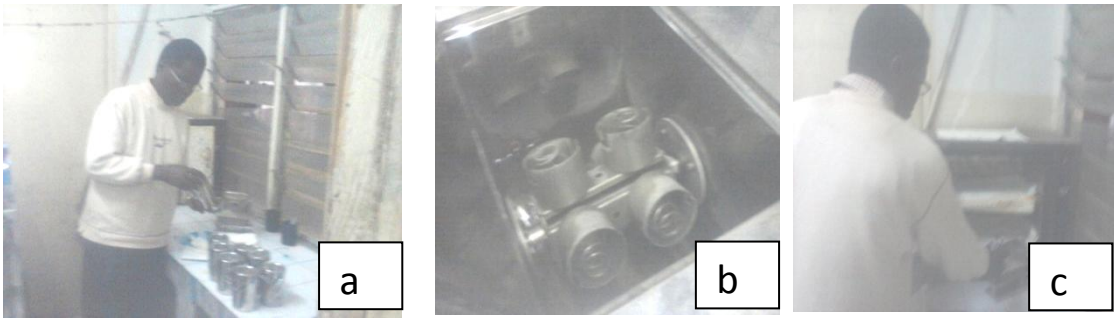


Plate 3.9 Wash fastness tests. (a) Beakers container for fabrics washing tests (b) Wash wheel machine for fabric samples: beaker's based. (c) Removal of the wash fabrics tests from the wash wheel machine.

Source: Researcher at the labouratory (2013)

Light fastness tests:

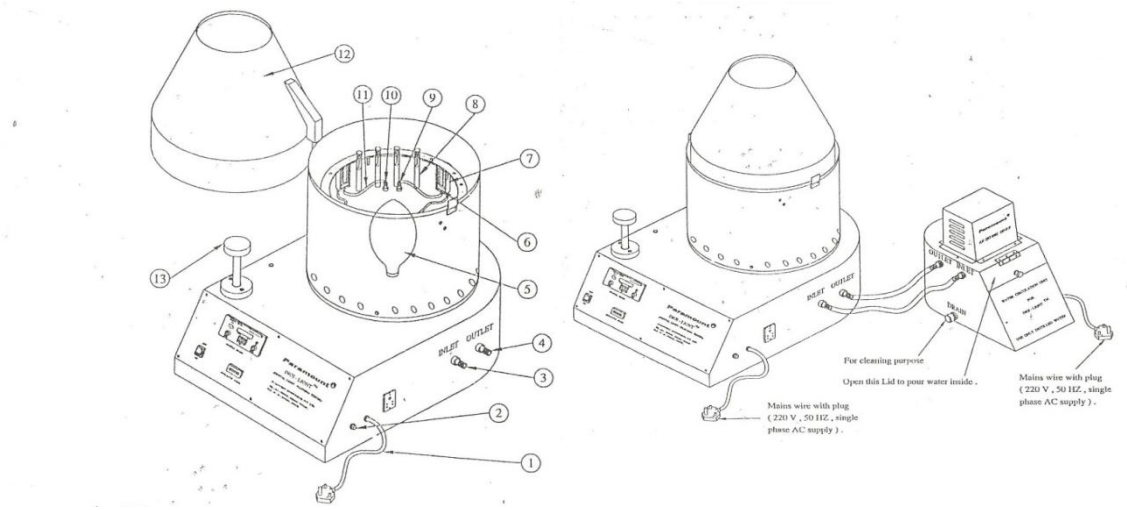
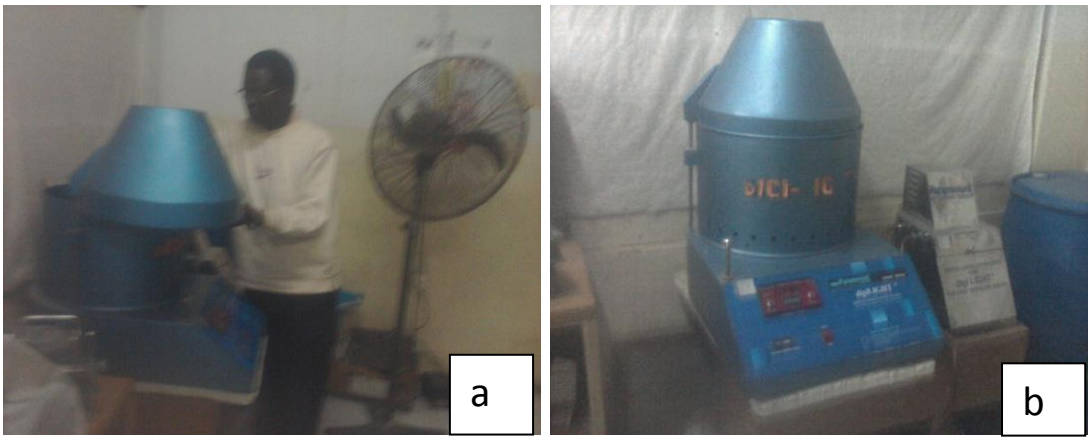


Figure 3.5 a (Opened) b (Closed)
Standard MBTL fading (Mercury blended tungsten lamp)

(1) Main wire with plug (To operate on220 volts, 50HZ Single phase AC supply) (2.) Fuse (5 amps.)
(3.) Outside water inlet nozzle (outer) (4.) Outside water outlet nozzle (outer)
(5.) Mercury blended tungsten lamp (MBTL) fading lamp. (6.) Type 'A' rectangular cells.
(7.) Spring mounted cradles (holder) Type 'A'. (8.) Type 'C' Cylindrical Specimen Holder.
(9.) Water inlet nozzle (inner) (10.) Water outlet nozzle (inner). (11.) Water circulation pipe.
(12) Top lid (top cover) of the unit. (13.) Push down lever to open lid (cover)
Source: Paramount since 1964 ISO certified company. (User's manual)



(a) Opened. (b) Closed.Plate

3.10: Light fastness test: Standard MBTL fading (Mercury blended tungsten lamp)

Source: Researcher at the labouratory (2013)

Perspiration fastness tests

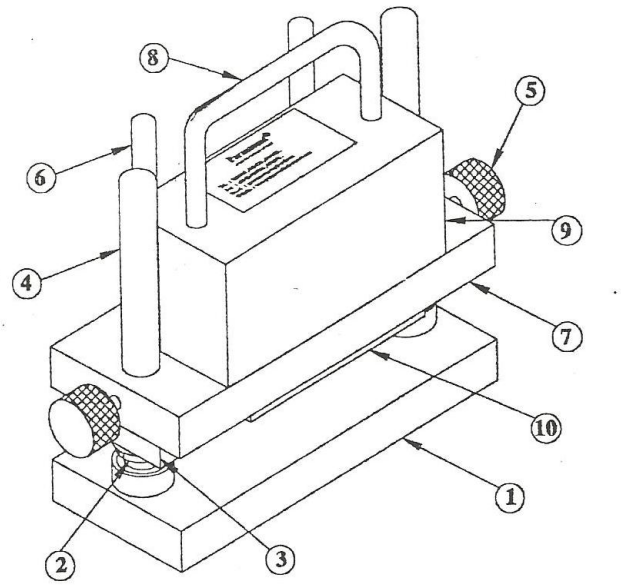


Figure 3.6: Perspiration (SS)

(1.) Base of the unit. (2.) Compression S.Steel springs -02 Nos. (3.) Center loading plate of the unit.
(4) Front rods -02 nos. (5.) Knobs for tightening the top plate -02 Nos. (6.) Back rods- 02 Nos.
(7.) Top loading plate. (8.) Handle (calibrated dead weight) (9.) Calibrated dead weight.
(10.) Acrylic separator plate.
Source: Paramount since 1964 ISO certified company. (User's manual)

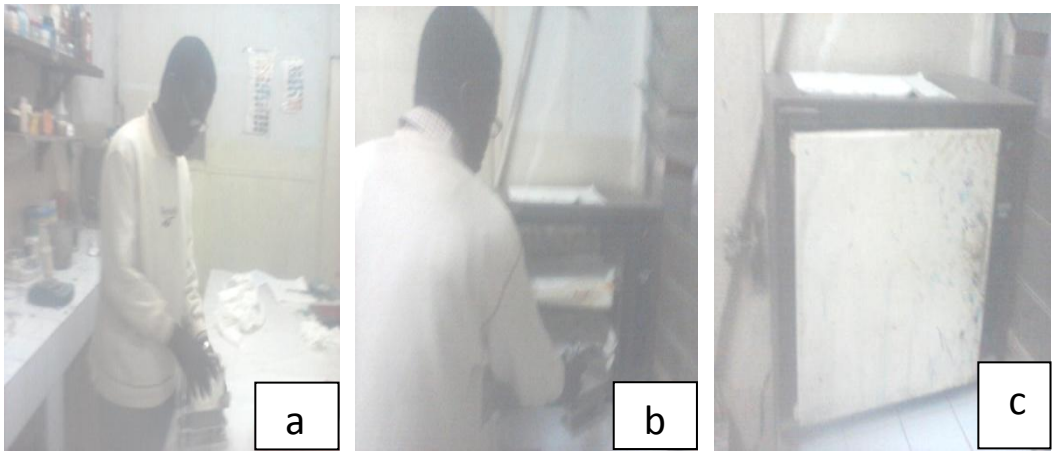


Plate 3.11 Perspiration tests: (a) Fabrics perspiration with Perspirometer (SS). (b) Opened.(c) Closed.
Source: Researcher at the labouratory (2013)

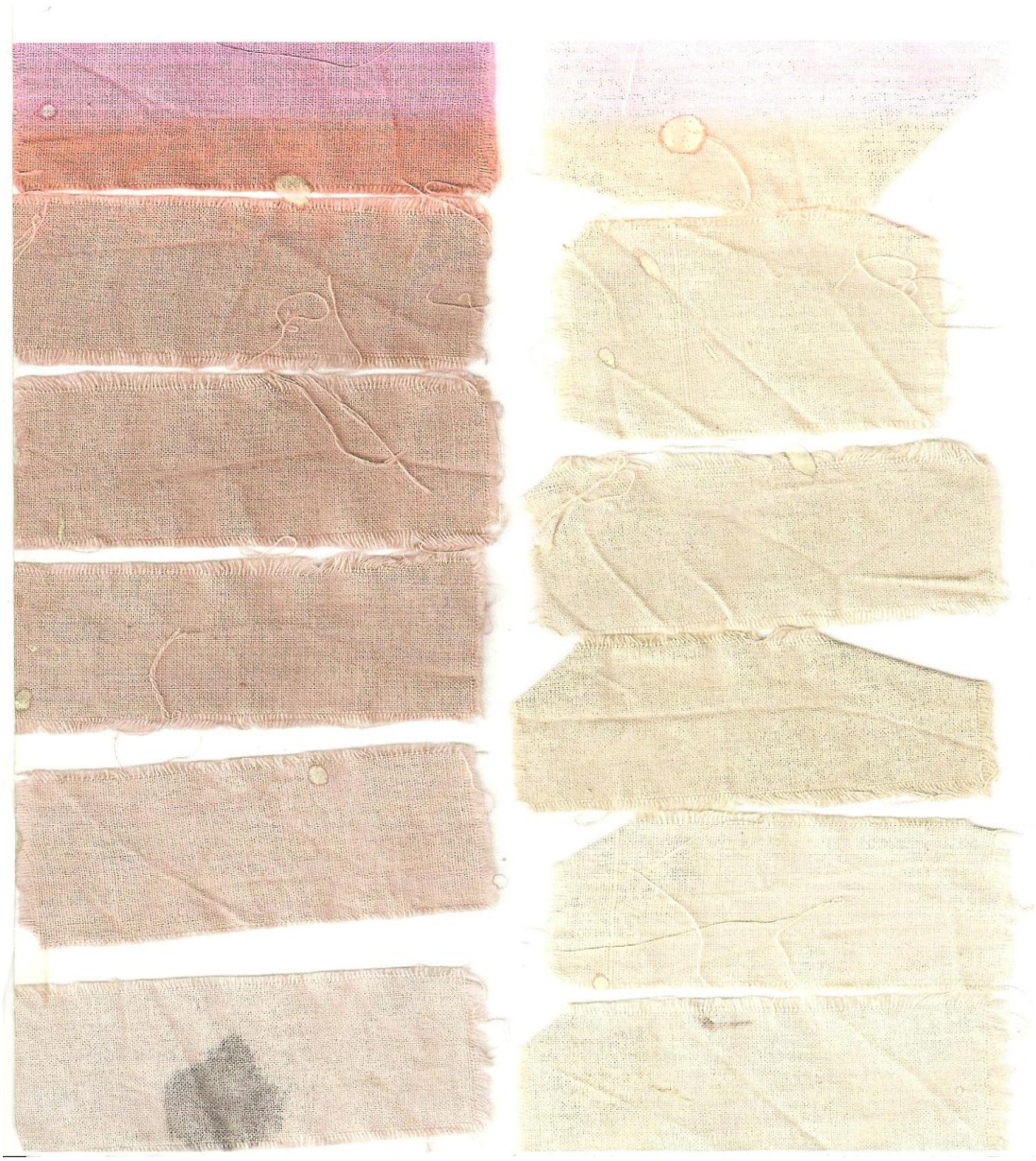
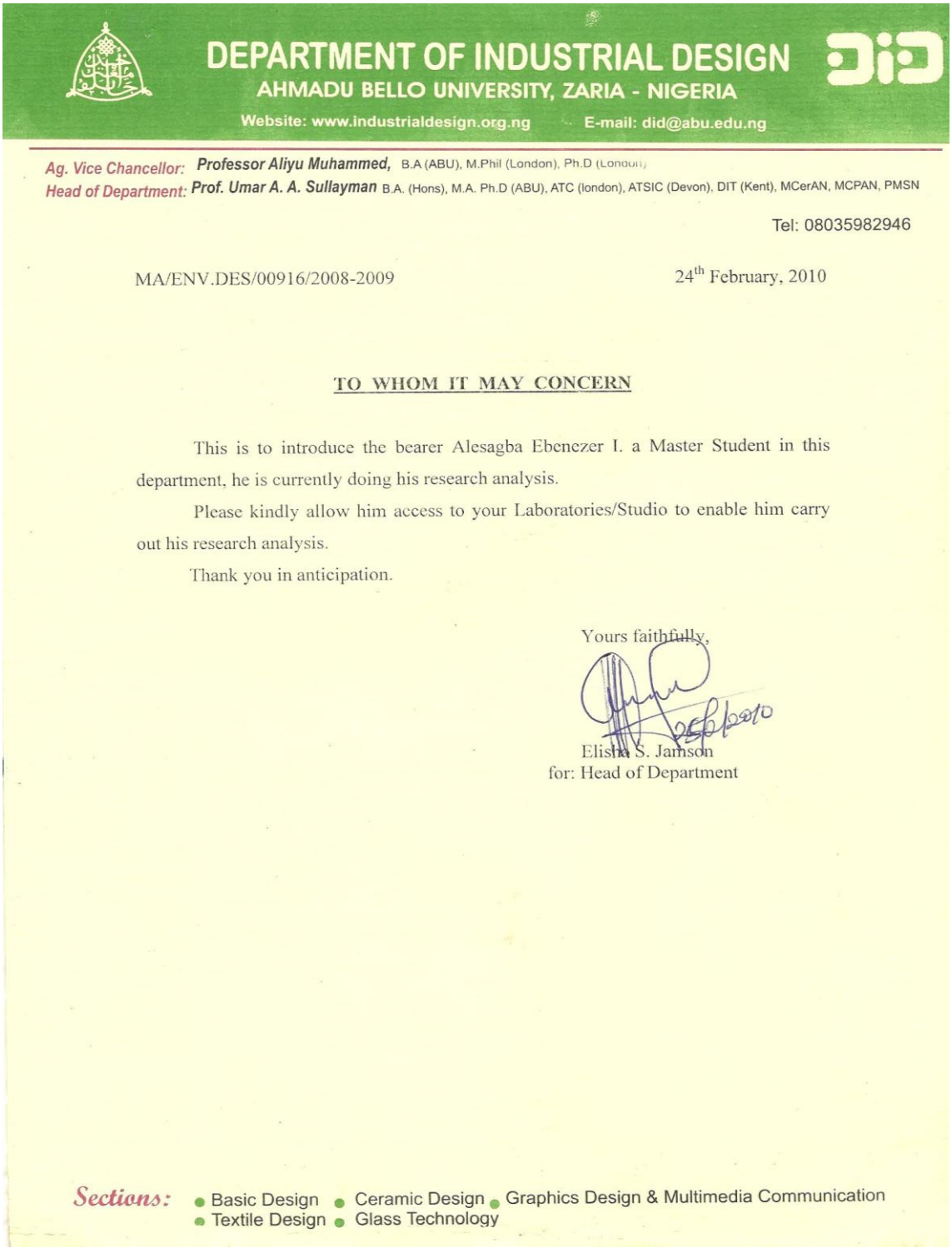


Plate 3.12 Dyed fabric samples. Source: Researcher at the laboratory (2013)

Appendices

Appendix. A1. Letter: To whom it may concern



DEPARTMENT OF INDUSTRIAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA - NIGERIA
Website: www.industrialdesign.org.ng E-mail: did@abu.edu.ng

Ag. Vice Chancellor: Professor Aliyu Muhammed, B.A (ABU), M.Phil (London), Ph.D (London)
Head of Department: Prof. Umar A. A. Sullayman B.A. (Hons), M.A. Ph.D (ABU), ATC (london), ATSIC (Devon), DIT (Kent), MCerAN, MCPAN, PMSN

Tel: 08035982946

MA/ENV.DES/00916/2008-2009 24th February, 2010

TO WHOM IT MAY CONCERN

This is to introduce the bearer Alesagba Ebenezer I. a Master Student in this department, he is currently doing his research analysis.

Please kindly allow him access to your Laboratories/Studio to enable him carry out his research analysis.

Thank you in anticipation.

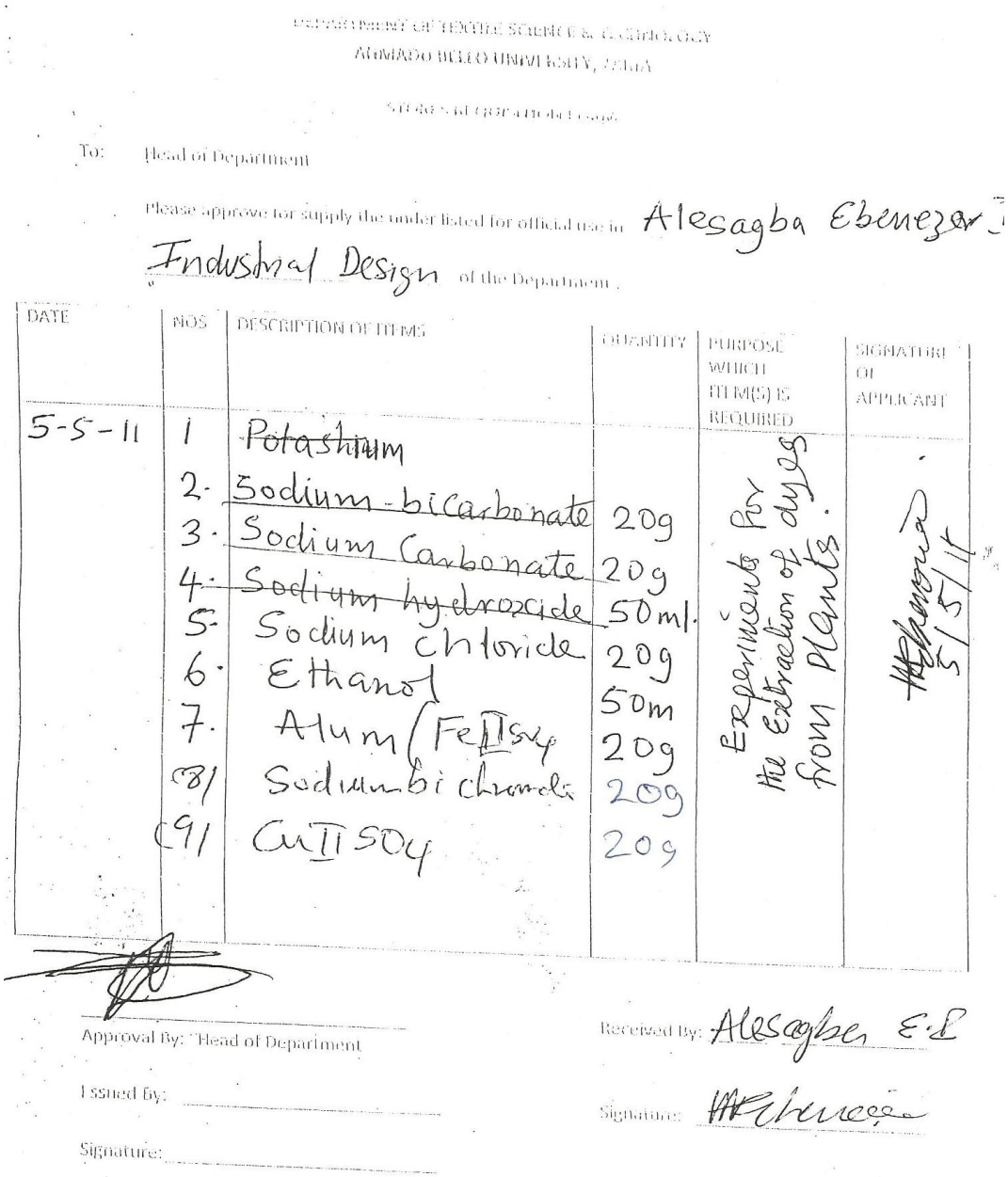
Yours faithfully,

25/2/2010

Elisha S. Jamson
for: Head of Department

Sections: Basic Design Ceramic Design Graphics Design & Multimedia Communication Textile Design Glass Technology

Appendix. A2. Request for solvents



DEPARTMENT OF TEXTILE SCIENCE & TECHNOLOGY
AHMADU BELLO UNIVERSITY, ZARIA

STORES REQUISITION FORM

To: Head of Department

Please approve for supply the under listed for official use by Alesagba Ebenezer J.
Industrial Design of the Department.

DATE	NOS	DESCRIPTION OF ITEMS	QUANTITY	PURPOSE WHICH ITEM(S) IS REQUIRED	SIGNATURE OF APPLICANT
5-5-11	1	Potashium		Experiments for the extraction of dyes from plants.	5/5/11
	2.	Sodium-bicarbonate	20g		
	3.	Sodium Carbonate	20g		
	4.	Sodium hydroxide	50ml.		
	5.	Sodium chloride	20g		
	6.	Ethanol	50m		
	7.	Alum/FeIISO4	20g		
	8/	Sodiumbichromate	20g		
	9/	CuIISO4	20g		

Approval By: Head of Department

Received By: Alesagba E.E

Issued By:

Signature:

Signature:

Copper Sulphate
Sodium chromate

Appendix. A3. Letter of introduction: Dr. Salihu Maiwada

DEPARTMENT OF INDUSTRIAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA - NIGERIA
Website: www.industrialdesign.org.ng E-mail: did@abu.edu.ng

Vice - Chancellor: Professor Abdullahi Mustapha, B.Sc (Hons) Pharm (ABU) Ph.D (London), FPSN
Head of Department: Dr. Salihu Maiwada, B.A., M.A., Ph.D(ABU) MSNA, MATTN

Tel: 08028185081
069879415

06/05/2011

The Head,
Department of Chemistry,
Ahmadu Bello University,
Zaria.

Dear Sir,

LETTER OF INTRODUCTION

The bearer Alesagba Ebenezor is a master student who is undergoing his research work. He would want to use some materials/solvents in your Department. The items are as follows:

1. Copper II sulphate – 20g,
2. Sodium bi chromate – 20g.
3. Alum (Fe II su_4)
4. Ethanol – 100ml
5. Sodium Chloride – 20g.
6. Distilled water.

Please kindly accord him assistance to enable him complete his research work.

Thank you.

Yours faithfully,

Dr. Salihu Maiwada
Head of Department

Sections: Basic Design Ceramic Design Graphics Design & Multimedia Communication
Textile Design Glass Technology

Appendix. A4. Letter of introduction: Dr. M.K. Yakubu

DEPARTMENT OF TEXTILE SCIENCE AND TECHNOLOGY

AHMADU BELLO UNIVERSITY, ZARIA, NIGERIA

VICE-CHANCELLOR: PROFESSOR ABDULLAHI MUSTAPHA
B.Sc. (Hons) Pharm. (A.B.U), Ph.D. (London), FPSN

HEAD OF DEPARTMENT: DR. M. K. YAKUBU
B.Sc., M.Sc., Ph.D (ABU), MMSN, MPIN, MAT
GSM: 08029533411, 08094607196
E-mail: koko80001@yahoo.com

Date: 11- 07 – 2011

The Personnel Manager,
African Textile Manufacturer
Kano.

Dear Sir,

LETTER OF INTRODUCTION

The bearer Mr. Alesagba Ebenezer Igbekele with Registration Number M.A/ENV-DES/00916/2008-09 is a student of this Department currently on his research work.

Kindly assist him to carryout the Wash, Light and Perspiration Fastness on his samples.

Thank you.

Yours faithfully,

Dept. of Textile Science & Technology ZARIA NIGERIA Ahmadu Bello University

Dr. M. K. Yakubu
Head of Department

Appendix. A5. Letter of acknowledgments: the researcher

Department of Industrial Design,
Ahmadu Bello University,
Zaria.
13-07-2011

The Personnel Manager,
African Textile Manufacturer,
Kano.

Through.

The Head of Department,
DPF 1
African Textile Manufacturer,
Kano.

Dear Sir,

Ref:- Letter of Introduction Dated 11-07-2011 From Ahmadu Bello University Zaria.

I Alesagba Ebenezer Igbekele with Registration Number M.A/ENV-DES/00916/2008-2009 of the above department wish to apply to this organisation to please acknowledge the receipt of the letter to allow me Carryout the necessary research of Wash, light and Perspiration of my fabric samples at chemical laboratory.

I am hoping that the program of this research will take me from 11th - 29th July 2011.

My head of department will like an acceptance letter for the period mentioned above.

Thanks for your understanding and Cooperation.

Yours faithfully,
Alesagba Ebenezer Igbekele

Appendix. A6. Acceptance letter: personnel manager African textile Kano

AFRICAN TEXTILE MANUFACTURERS LTD.

RC No. 32300

Manufacturers of Refined African Prints - Abada, Wazobia Duniya...

SUHAIL AKAR'S LANE,
BEYOND COCA COLA,
CHALLAWA INDUSTRIAL ESTATE,
P.O. BOX 284,
KANO,
NIGERIA.

TEL: 234 (64) 311074-6
234 (64) 891570
234 (64) 977356
FAX: 00 871 762 553 041#
E-MAIL: Afriwax@atmltd.net
WEBSITE: www.atmltdonline.com

BRANCHES: KANO, LAGOS, IBADAN ONITSHA & LOME

July 15, 2011

The Head of Department
Department of Industrial Design
Ahmadu Bello University
Zaria, Nigeria

ACCEPTANCE LETTER

We write to inform you that **Mr. Alesagba Ebenezer Igbekele Reg. No. M.A/ENV-DES/00916/2008-2009** of the above mentioned institution has been accepted to carryout his research program in our company from **11th – 29th July 2011**.

Thanks,

Yours faithfully,
For: African Textile Manufacturers Ltd.

Ebiteh N. Godwin
Personnel Manager

Appendix. A7. Letter of introduction: Bayero University Kano

AFRICAN TEXTILE MANUFACTURERS LTD.

RC No. 32300

Manufacturers of Refined African Prints - Abada, Wazobia Duniya...

SUHAIL AKAR'S LANE,
BEYOND COCA COLA,
CHALLAWA INDUSTRIAL ESTATE,
P.O. BOX 284,
KANO,
NIGERIA.

TEL: 234 (64) 311074-6
234 (64) 891570
234 (64) 977356
FAX: 00 871 762 553 041#
E-MAIL: Afriwax@atmltd.net
WEBSITE: www.atmltdonline.com

BRANCHES: KANO, LAGOS, IBADAN ONITSHA & LOME

December 15, 2011

The Head Department
Science Laboratory
Bayero University
Kano

LETTER OF INTRODUCTION

The bearer **Mr. Ebenezer Igbekele** of A.B.U Zaria has been with us since July 2011 carrying out the fastness test on his dyes production but along the line our light fastness machine bulb got burnt thereby making it impossible to conclude this test.

I hereby ask that you kindly assist him to carry out this light fastness test in your laboratory if the equipment is available.

Thanks,

Yours faithfully
For: African Textile Manufacturers Ltd

Ebiteh N. Godwin
Personnel Manager

Gladys C. Chukwu
Snr. Lab Technologist

Appendix. A8. Reference letter: Kaduna polytechnic

AFRICAN TEXTILE MANUFACTURERS LTD.

RC No. 32300

Manufacturers of Refined African Prints - Abada, Wazobia Duniya...

SUHAIL AKAR'S LANE,
BEYOND COCA COLA,
CHALLAWA INDUSTRIAL ESTATE,
P.O. BOX 284,
KANO,
NIGERIA.

TEL: 234 (64) 311074-6
234 (64) 891570
234 (64) 977356
FAX: 00 871 762 553 041#
E-MAIL: Afriwax@atmltd.net
WEBSITE: www.atmltdonline.com

BRANCHES: KANO, LAGOS, IBADAN ONITSHA & LOME

20th June 2012.

The Head of Department,
Science Laboratory,
Kaduna State Polytechnic,
Kaduna State.

Sir,

REFERENCE FOR EBENEZER I. ALESAGBA

I wish to let you know that the above mentioned has been conducting the light fastness test of his products in our chemical laboratory until the pump of the cooling tank got broken leading to the burning of the pump meter.

On this note we are refering him to your institution to enable him conclude the said experiment.

Thanks for your acceptance.

Snr. Lab Tech. 26/06.

Personnel Manager

Appendix. A9. Letter to the supervisors of the researcher: senior lab Tech of African TextileKano

AFRICAN TEXTILE MANUFACTURERS LTD. RC No. 32300

Manufacturers of Refined African Prints - Abada, Wazobia Duniya...

SUHAIL AKAR'S LANE,
BEYOND COCA COLA,
CHALLAWA INDUSTRIAL ESTATE,
P.O. BOX 284,
KANO,
NIGERIA.

TEL: 234 (64) 311074-6
234 (64) 891570
234 (64) 977356
FAX: 00 871 762 553 041#
E-MAIL: Afriwax@atmltd.net
WEBSITE: www.atmltdonline.com

BRANCHES: KANO, LAGOS, IBADAN ONITSHA & LOME

20th June 2012

The Supervisor of Mr. Ebenezer I. Alesagba,
Department of industrial design,
Ahmadu Bello University,
Zaria,
Kaduna State.

Sir,

I wish to inform you that Mr. Ebenezer I. Alesagba of the above department has been carrying out the fastness properties of his products in our laboratory since July 2011, having concluded the washing and perspiration fastness, commenced the light fastness property of which many samples has been tested but along the line the machine started having series of problems, first it was power and latter the cooling tank pump got burnt of which the management is yet to fix.

On this note I advised him to discuss with you in any way forward as it may take some time for the machine to be put in order. However, we have assisted him with many sample tests that he is much conversant with.

Thanks for your consideration.

Snr Lab Tech. 25/6/12

Personnel Manager

Appendix. A10. Application for proposal defence: the researcher

Department of Industrial Design,

Faculty of Environmental Design,

Ahmadu Bello University, Zaria.

9th August, 2012.

The P.G. Coordinator,

Department of Industrial Design,

Faculty of Environmental Design,

Ahmadu Bello University, Zaria.

Through

The Major Supervisor, Approved for Major Supervisor. 9/8/2012

Department of Industrial Design,

Faculty of Environmental Design,

Ahmadu Bello University, Zaria.

Dear sir,

Application for proposal defence.

I wish to apply for proposal defence at the above department. My name is Alesagba Ebenezer Igbekele with reg. no: MA/ENV.DESIGN/00916/2008-09.

The topic of my proposal defence is "Exploration and extraction of colouring substances from herbs for possible use as herbs on cotton fabrics" and I have met the departmental requirements for proposal defence.

I will be very grateful, if this application can be considered. Thanks.

Yours faithfully,

Alesagba Ebenezer Igbekele.

MA/ENV.DESIGN/00916/2008-09.

Appendix. A11. Application for seminar presentation

Department of Industrial Design
Faculty of Environmental Design
Ahmadu Bello University, Zaria
18th June, 2013.

The P.G. Coordinator,
Department of Industrial Design
Faculty of Environmental Design
Ahmadu Bello University, Zaria

Through,

The Chairman, Approved. Opt for chairmans 19/6/2013
Supervisory Committee,
Department of Industrial Design
Faculty of Environmental Design
Ahmadu Bello University, Zaria

Dear Sir,

Application for seminar presentation

I wish to apply for seminar presentation on the topic: Exploration and extraction of colouring substances from herbs for possible use as dyes on cotton fabrics in Kogi State. I have finished my course works as well as defended my proposal in the research carried out at the Department. My name is Alesagba Ebenezer Igbekele with reg. no: M.A/ENV.DESIGN/00916/2008-09.

I shall be very grateful, if this application can be considered. Thanks in anticipation.

Yours faithfully,

Alesagba Ebenezer Igbekele.

Appendix. A12. Application for extension of registration period

Department of Industrial Design

Faculty of Environmental Design

Ahmadu Bello University, Zaria

18th June, 2013.

The Dean,

School of Postgraduate Studies,

Ahmadu Bello University, Zaria

Through,

The P.G. Coordinator,

Department of Industrial Design

Faculty of Environmental Design

Ahmadu Bello University, Zaria

Recommended for Approval

18/06/13

Dear Sir,

Application for Extention of Registration Period

Due to non- completion of my study during the initial scheduled period by the University, I hereby wish to apply for an extension of my study period. This will enable me to complete the programme successfully.

My name is Alesagba Ebenezer Igbekele a post graduate student of M.A Industrial Design Department, with reg. no: M.A/ENV.DESIGN/00916/2008-09. Presently, the study has reached the level of seminar presentation a stage to the final defence.

I shall be grateful, if my application is being considered favourably. Enclosed herewith is the photocopy of my admission letter for your perusal. Thanks in anticipation.

Yours faithfully,

Alesagba Ebenezer Igekele.

Appendix. A13. Application for external defence

Textile Section,
Department of Industrial Design,
Ahmadu Bello University,
Zaria.
10th June, 2014.

The Dean,
School of Postgraduate Studies,
Ahmadu Bello University,
Zaria.
Through:

The Dean,
Faculty of Environmental Design,
Ahmadu Bello University,
Zaria.

Recommended

DEANS OFFICE
Faculty of
DEPT. OF INDUSTRIAL DESIGN
DATE
SIGN
A.B.U. ZARIA

The Head,
Department of Industrial Design,
Ahmadu Bello University,
Zaria.

Recommended

Through:

Recommended

The Chairman, Supervisory Committee,
Dr. S Maiwada,
Department of Industrial Design,
Ahmadu Bello University,
Zaria.

Sir,

APPPLICATION FOR EXTERNAL DEFENCE

I hereby humbly apply for consideration to sit for my external defence, I have fulfilled the requirement of semester course work/studio work and seminar presentation.

The title of my thesis is: "Exploration and extraction of colouring substances from herbs for possible use as dyes on cotton fabric in Lokoja, Nigeria".

I shall be grateful if you would consider and fix a suitable date for the examination.

Thanks in anticipation.

Yours Faithfully,

Alesagba Ebenezer Igbekele.

P13EVID8015