EFFECT OF CHEMICAL MODIFICATION ON THE SURFACE CHARACTERISTICS AND SOME MECHANICAL PROPERTIES OF SISAL FIBRES.

BY

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MSC/SCIE/04307/2010-2011

A THESIS SUBMITTED TO THE SCHOOL OF POST GRADUATE STUDIES,
AHMADU BELLO UNIVERSITY, ZARIA.

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF A
MASTER DEGREE IN TEXTILE SCIENCE AND TECHNOLOGY.

DEPARTMENT OF TEXTILE SCIENCE AND TECHNOLOGY,
FACULTY OF SCIENCE.
AHMADU BELLO UNIVERSITY, ZARIA.

NIGERIA.

MAY, 2013.

DECLARATION

I declare that the work in this Thesis entitled EFFECT OF CHEMICAL MODIFICATION ON THE SURFACE CHARACTERISTICS AND SOME MECHANICAL PROPERTIES OF SISAL FIBRES has been carried out by me in the Department of Textile Science and Technology. The information derived from the literature, has been duly acknowledged in the text and a list of references provided. No part of this thesis report was previously presented for another degree or diploma at this or any other Institution.

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CERTIFICATION

This is to certify that the thesis "EFFECT OF CHEMICAL MODIFICATION ON THE SURFACE CHARACTERISTICS AND SOME MECHANICAL PROPERTIES OF SISAL FIBRES", by Nwadiokwu Ehiedu Sunny, meets the regulations governing the award of Master Degree in Textile Science and Technology of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

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ACKNOWLEDGEMENTS

I would like to express my profound appreciation to my major Supervisor, Professor O.K. Sunmonu, whose fatherly advice, all-round contribution and encouragement led to the success of this work. Words are not enough to thank you sir. Special thanks also go to my minor supervisor Mr. K.O. Fashola for his immense assistance throughout the period of this research.

I also wish to extend my gratitude to the staff and management of Sheda Science and Technology Complex (SHESTCO), Abuja, especially Mrs. Iwok and Mrs. Onodu, for their assistance in carrying out the SEM analysis. I acknowledge the assistance of the H.OD, Dr A Danladi and staff of the Department of Textile Science and Technology, Ahmadu Bello University, Zaria, for their various contributions to the success of this work. My heartfelt thanks go to Mr. Joshua of the Standards Organization of Nigeria (Textile and Leather laboratory), Kaduna, for his assistance in carrying out the tensile properties tests using the Instron Tensile Tester. I also want to thank Mr. Dickson for his prayers, typesetting, editing, and other assistance during this research.

A big thank you to my parents for their financial and moral support through out the period of this research work. I am also indebted to my elder brother Dr. Ifeanyi John Nwadiokwu, whose financial and moral support assisted in the completion of my work. I am grateful to my colleagues Rita, Kunle, Zaks, Tony and Auta, whose friendliness and brotherly companionship boosted my morale during my course of study. I pray for you all. Greetings to my special friend Karen for being there throughout the period of this research.

This acknowledgement will not be complete if I don't humbly give my unreserved thanks to God almighty for his guidance, sustenance, health, protection and everything. May your name be praised forever.

ABSTRACT

The effect of chemical modification on the surface characteristics and some mechanical properties of sisal fibres have been investigated. The sisal fibres were chemically modified using different concentrations of caustic soda while varying the treatment time. The surface characteristics of the untreated and treated samples were studied using the Scanning Electron Microscope (SEM). The moisture content and regain of the fibres were also investigated. The samples were also assessed for their tensile properties at different gauge lengths using the Instron tensile tester. The results obtained showed that alkali modification had appreciable effect on the surface characteristics, moisture regain and tensile properties of the sisal fibres (Tenacity, Breaking extension, Young's initial modulus and Work of rupture). The SEM micrographs revealed the rough surface topography of the sisal fibres caused by alkali modification which increased with increased concentration. Moisture content and regain also increased with increasing concentration. This increases the accessible surface of the sisal fibres, due to the increase in the amorphous region owing to the swelling of the fibre. Tenacity, work of rupture and breaking extension increased with increasing concentration of caustic soda while Young's modulus decreased. However, these tensile properties decreased with increase in gauge length due to the "weak-link effect". The time of treatment has no significant effect on all the properties investigated.

TABLE OF CONTENTS

Title Page

Declaration ii

Certification iii

Acknowledgement iv

Abstract v

List of Plates ix

List of tables x

List of figures xi

List of Appendices xiii

Chapter one

1.0 Introduction 1

1.1 Statement of research problems 2

1.2 Research objectives 3

1.3 Justification 3

1.4 Scope of research Study 4

Chapter two

2.0 Literature review 5

2.1 Sisal Plants 5

2.2 Sisal Leaves 6

2.3 Sisal Fibre 7

2.3.1 Structure of Sisal Fibres 9

2.3.2 Chemical Composition of Sisal Fibres 11

2.3.3 Sisal Fibre Processing/Extraction 11

2.3.4 Properties of Sisal Fibres 12

2.2.5 Uses of Sisal Fibres 12

2.4 Modification of Sisal Fibres 16

2.4.1 Interface Modification 16

2.4.2 Chemical Modification 17

2.5 Alkali Treatment of Sisal Fibres 18

2.6 Environmental Impact 19

Chapter Three

3.0 Materials and Method 21

3.1 Sisal Fibres 21

3.2 Chemicals 21

3.3 Equipment and Instrumentation 21

3.4 Method 21

3.4.1 Extraction of Sisal Fibres 21

3.4.2 Chemical modification of the Fibres 22

3.5 Determination of Moisture content and Regain 22

3.6 Determination of Surface Characteristics 23

3.7 Determination of Mechanical (Tensile) Properties 23

3.8 Effect of Concentration of Caustic Soda and Treatment Time . .
24

Chapter Four

4.0 Results and Discussion . . . . . .
25

4.1 Results from Extraction of Sisal Fibre . . . . . .
25

4.2 Results from Alkali Modification of Sisal Fibre . . . . . .
25

4.3 Moisture content and regain for Sisal Fibres . . . . . .
26

4.4 Assessment of Surface characteristics (SEM Analysis) . . . . .
27

4.5 Assessment of the Mechanical (Tensile) Properties . . . . .
36

4.5.1 Fibre Tenacity (diff. conc., diff. gauge length, const. time) . . .
36

4.5.2 Fibre Tenacity (const.conc.,diff gauge length, diff. time) . . . .
39

4.5.3 Breaking Extension (diff. conc., diff. gauge length, const. time) . . .
42

4.5.4 Breaking Extension (const. conc., diff. gauge length, diff. time) . . .
44

4.5.6 Young's Initial Modulus (diff. conc., diff. gauge length, const. time) . .
47

4.5.7 Young's Initial Modulus (const. conc., diff. gauge length, diff. time) . .
49

4.5.8 Work of Rupture (diff. conc., diff. gauge length, const. time) . . .
51

4.5.9 Work of Rupture (const. conc., diff. gauge length, diff. time) . . . 54

Chapter Five

5.0 Conclusion and Recommendations 57

5.1 Conclusion 57

5.2 Recommendations 58

References 59

Appendix 68

List of plates

Plate 1 SEM micrographs at constant 5 mins time but at different concentrations . . 29

Plate 2 SEM micrographs at constant 10mins time but at different concentrations . . 30

Plate 3 SEM micrographs at constant 15 mins time but at different concentrations . 31

Plate 4 SEM micrographs at constant 5% concentration but at different time. . . 32

Plate 5 SEM micrographs at constant 10% concentration but at different time. . . 33

Plate 6 SEM micrographs at constant 15% concentration but at different time. . . 34

Plate 7 SEM micrographs at constant 20% concentration but at different time. . . 35

Table

Table 4.1 Moisture content and regain for sisal fibres at various conc. and at diff time. .

26

List of figures

Figure 2.1 The Sisal Fibre 7

Figure 2.2 Schematic sketch of a Sisal Fibre cell 8

Figure 4.3.1 Tenacities at different conc., gauge length but at Constant 5 mins time . 39

Figure 4.3.2 Tenacities at different conc., gauge length but at Constant 10 mins time . 39

Figure 4.3.3 Tenacities at different conc., gauge length but at Constant 15 mins time . 40

Figure4.3.4 Tenacities at different time, gauge length but at Constant 5% conc. . . 43

Figure 4.3.5 Tenacities at different time, gauge length but at Constant 10% conc. . 43

Figure 4.3.6 Tenacities at different time, gauge length but at Constant 15% conc. . 44

Figure 4.3.7 Tenacities at different time, gauge length but at Constant 20% conc. . 44

Figure 4.4.1 Breaking Ext. at different conc., gauge length but at Constant 5 mins time . 47

Figure 4.4.2 Breaking Ext. at different conc., gauge length but at Constant 10mins time . 47

Figure 4.4.3 Breaking Ext. at different conc., gauge length but at Constant 15 mins . 48

Figure 4.4.4 Breaking Ext. at different time, gauge length but at Constant 5% conc. . 51

Figure 4.4.5 Breaking Ext. at different time, gauge length but at Constant 10% conc. . 51

Figure 4.4.6 Breaking Ext. at different time, gauge length but at Constant 15% conc. . 52

Figure 4.4.7 Breaking Ext. at different time, gauge length but at Constant 20% conc. . 52

Figure 4.5.1 Young's Mod.at different conc., gauge length but at Constant 5 mins time . 55

Figure 4.5.2 Young's Mod.at different conc., gauge length but at Constant 10minstime . 55

Figure 4.5.3 Young's Mod at different conc., gauge length but at Constant 15 mins time. 56

Figure 4.5.4 Young's Mod.at different time, gauge length but at Constant 5% conc. . 59

Figure 4.5.5 Young's Mod. at different time, gauge length but at Constant 10% conc. . 59

Figure 4.5.6 Young's Mod.at different time, gauge length but at Constant 15% conc. . 60

Figure 4.5.7 Young's Mod.at different time, gauge length but at Constant 20% conc. . 60

Figure 4.6.1 Work of Rupt.at different conc., gauge length but at Constant 5 mins time. 63

Figure 4.6.2 Work of Rupt.at different conc., gauge length but at Constant 10mins time . 63

Figure 4.6.3 Work of Rupt.at different conc., gauge length but at Constant 15 mins time. 64

Figure 4.6.4 Work of Rupt.at different time, gauge length but at Constant 5% conc. . 67

Figure 4.6.5 Work of Rupt.at different time, gauge length but at Constant 10% conc. . 67

Figure 4.6.6 Work of Rupt.at different time, gauge length but at Constant 15% conc. . 68

Figure 4.6.7 Work of Rupt.at different time, gauge length but at Constant 20% conc. . 6

List of Appendices

Appendix A: Data Tables

Table A1 Tenacities at different conc., gauge length but Constant 5 minutes time . 68
Table A2 Tenacities at different conc., gauge length but Constant 10 minutes time . 68
Table A3 Tenacities at different conc., gauge length but Constant 15 minutes time . 69
Table A4 Tenacities at different time, gauge length but Constant 5% concentration . 69
Table A5 Tenacities at different time, gauge length but Constant 10% conc entration. 70
Table A6 Tenacities at different time, gauge length but Constant 15% concentration . 70
Table A7 Tenacities at different time, gauge length but Constant 20% concentration . 71
Table A8 Breaking Ext. at different conc., gauge length but Constant 5 minutes time. 71
Table A9 Breaking Ext. at different conc., gauge length but Constant 10minutes time. 72
Table A10 Breaking Ext. at different conc., gauge length but Constant 15 minutes time. 72
Table A11 Breaking Ext. at different time, gauge length but Constant 5% concentration. 73
Table A12 Breaking Ext. at different time, gauge length but Constant 10% concentration. 73
Table A13 Breaking Ext. at different time, gauge length but Constant 15% concentration 74
Table A14 Breaking Ext. at different time, gauge length but Constant 20% concentration. 74

Table A15 Young's Mod.at different conc., gauge length but Constant 5 minutes time . . 75

Table A16 Young's Mod.at different conc., gauge length but Constant 10minutes time 75

Table A17 Young's Mod at different conc., gauge length but Constant 15 minutes time. 76

Table A18 Young's Mod.at different time, gauge length but Constant 5% concentration. 76

Table A19 Young's Mod. at different time, gauge length but Constant 10% concentration. 77

Table A20 Young's Mod.at different time, gauge length but Constant 15% concentration 77

Table A21 Young's Mod.at different time, gauge length but Constant 20% concentration. 78

Table A22 Work of Rupt.at different conc., gauge length but Constant 5 minutes time . . . 78

Table A23 Work of Rupt.at different conc., gauge length but Constant 10minutes time. . 79

Table A24 Work of Rupt.at different conc., gauge length but Constant 15 minutes time. 79

Table A25 Work of Rupt.at different time, gauge length but Constant 5% concentration. 80

Table A26 Work of Rupt.at different time, gauge length but Constant 10% concentration. 80

Table A27 Work of Rupt.at different time, gauge length but Constant 15% concentration. 81

Table A28 Work of Rupt.at different time, gauge length but Constant 20% concentration. 81

CHAPTER ONE

1.0 INTRODUCTION

Plant fibres are rich in cellulose and they are a cheap, easily renewable source of fibres with the potential for polymer reinforcement. The presence of surface impurities and the large amount of hydroxyl groups makes plant fibres less attractive for reinforcement of polymeric materials. The attractive features of these fibres are light weight, non-toxicity, friendly processing and absorption of CO_2 during their growth (Abdelmouleh *et al,* 2007; Tserki *et al,* 2005). These benchmark properties open the wide area for natural fibres to be used in composite sector with consequences for the partial replacement of traditional synthetic fibres. With the increase of environmental protection consciousness, natural fibres as a group of environmental friendly reinforcements are in considerable demand in composites (Bledzki *et al,* 2001; Ashori, 2008). Natural fibres such as flax, hemp, sisal, nettle and jute are the most common reinforcing elements (Ashori, 2008; Ganster and fink, 2006).

Alkalization has been known to modify plant fibres promoting the development of fibre-resin adhesion, which then will results in increased interfacial energy and hence, improvement in the mechanical and thermal stability of the composites (Mwaikambo and Ansell, 2002).

The uses of natural fibres to make low cost and eco-friendly composite materials are a subject of great importance. Nature provides us with an abundance of raw materials including sisal fibre which comes from the widely grown sisal plant. Sisal fibre is a hard fibre extracted from the leaves of sisal plant (*Agave sisalana*). Though native to tropical and subtropical North and South America, sisal plants are widely grown in tropical

countries of Africa, the West Indies and the Far East (Bisanda and Ansell 1994). Tanzania and Brazil are the two main producing countries of sisal fibres. (Chand *et al*, 1998). Though Sisal fibre is one of the most widely used natural fibres (Y.Li *et al*, 2000) and accounts for almost half the total production of natural fibres, a large quantity of this economic and renewable resource is still under-utilized.

Using sisal fibre as reinforcement in composites has raised great interest and expectations among material scientists and engineers. However, sisal fibre-reinforced composites generally have poor interface (Bledzki *et al*, 1996). Sisal fibre is lignocellulosic in nature; the presence of a large amount of hydroxyl groups is responsible for the hydrophilic nature of sisal fibre. Several fibre surface treatment methods have been studied to improve the adhesion properties between sisal fibres and a surrounding matrix. The chemical modification of natural fibres improves the adhesion of fibre-matrix interfaces in a composite, which in turn improves the mechanical properties of the composites. In certain cases it was found to bring about changes in the morphology, chemical groups and hydrophilicity of the fibres (Tragoon *et al,* 2007). An effective method includes alkali treatment to increase the roughness of fibre surface hence increasing the surface area available for contact with matrix.

In this research, alkali treatment was employed to chemically modify sisal fibres using different concentrations of (NaOH) 5%, 10%, 15% and 20% respectively. The influence of this modification on the surface characteristics, moisture regain and mechanical properties were assessed and analysed.

1.1 STATEMENT OF RESEARCH PROBLEMS

One of the major problems associated with natural fibres is their inherent constituents such as hemicelluloses, pectins, lignin and waxy substances which impede their uses in textile and composite applications. These inherent constituents act as cementing substances on the sisal fibre, giving it a smooth surface which leads to incompatibility with the polymer matrix (Li *et al*, 2008; Corrales *et al*, 2007) and reduction in mechanical properties. Therefore, chemical modification is required to remove these cementing substances and create a rough surface to make the sisal fibre amenable for use in composites.

1.2 RESEARCH OBJECTIVES

The main objective of the present study is to chemically modify the sisal fibres and assess the effect of the chemical modification on the surface characteristics and mechanical properties of the fibres. Specific objectives of the study are given below:

i. Extraction of the sisal fibres.

ii. Chemical modification of the sisal fibres using different concentrations of caustic soda.

iii. Assessment of the effect of the chemical modification on the surface characteristics of the fibres.

iv. Assessment of moisture content and regain.

v. Assessment of the effect of chemical modification on mechanical properties of the fibres.

1.3 JUSTIFICATION

Due to the drawbacks associated with using natural fibres in composites as enumerated in the statement of research problems above, this study intends to explore the chemical

modification of sisal fibres as a way of improving its properties to make it amenable for use in composites and in other areas of applications. The success of the chemical modification will reduce cost of composites as sisal fibres are readily and abundantly available at little or no costs in the country. Thus, the study can be likened to a "waste to wealth" venture.

1.4 SCOPE OF RESEARCH STUDY

The scope of this research study covers the chemical modification of sisal fibres using varying concentrations of caustic soda at varied time. Surface characteristics were assessed using Scanning electron microscope (SEM) to study the surface of both the unmodified and modified sisal fibres. The moisture regain and moisture content were also assessed. Mechanical properties studied were restricted to tensile properties, such as, tenacity, breaking extension, work of rupture and Young's modulus using the Instron Tensile Tester.

CHAPTER TWO

2.0 LITERATURE REVIEW

New environmental legislations as well as consumer pressure has forced the manufacturing industries to search for new materials that can substitute for conventional non-renewable reinforcing materials, such as carbon or glass fibres. On account of this, in recent years, the use of natural fibres such as banana, sisal, hemp, flax, jute, coconut and oil palm have attracted scientists and technologists for applications in consumer goods, low-cost housing and other civil structure (Justiz-Smith *et al*, 2008; Spinace *et al*, 2009; El-Taybe 2009). Natural fibres are very attractive for composite materials because of their advantages compared to synthetic fibres, These include lower levels of skin irritations and respiratory system during handling, good recyclability, abundant supply, low cost, low density, high specific strength to weight ratio, non-toxicity, and biodegradability (Spinace *et al,* 2009; Gu 2009; John 2009).

2.1 The sisal plant

The sisal plant produces approximately 200 - 250 leaves throughout its productive period. The life span of sisal plant is 7-10 years. The shape of sisal leaves is like sword and is about 1.5 to 2 meters tall. Young leaves may have a few minute teeth along their margins, but lose them as they mature. A good sisal plant yields about 200 leaves with each leaf having a mass composition of about 4% fibre, 0.75% cuticle, 8% other dry matter and 87.25% moisture. Thus a normal sisal leaf weighing about 600g yields about 3% by weight of fibre with each leaf containing about 1000 fibres. The fibre is extracted from the leaf

either by retting, by scraping or by retting followed by scraping or by mechanical means using decorticators. Diameter of the fibre varies from 100mm to 300mm (Mukherjee and Satyanarayana, 1984).

Propagation of sisal is generally by using bulbils produced from buds in the flower stalk or by suckers growing around the base of the plant, which are grown in nursery fields until large enough to be transplanted to their final position. These methods offer no potential for genetic improvement. Multiplication of selected genetic material using meristematic tissue culture (MST) offers considerable potential for the development of improved genetic material.

2.2 Sisal leaves

The sisal leaves contains three types of fibres: (1) mechanical, (2) ribbon and (3) xylem.

1. The mechanical fibres are mostly extracted from the periphery of the leaf. They have a roughly thickened-horse shoe shape and seldom divide during the extraction processes. They are the most commercially useful of the sisal fibre.
2. Ribbon fibres occur in association with the conducting tissues in the median line of the leaf. They are the longest fibres and compared with mechanical fibres they can be easily split longitudinally during processing.
3. Xylem fibres have an irregular shape and occur opposite the ribbon fibres through the connection of vascular bundles. They are composed of thin-walled cells and are therefore easily broken up and lost during the extraction process. (Bisanda and Ansell, 1992).

2.3 Sisal Fibres

Sisal fibres are plant based which are lignocellulosic in nature and composed of cellulose, hemicelluloses, lignin, pectin and waxy substances (Mohanty *et al*, 2000). Cellulose gives the strength, stiffness and structural stability of the fibre, and is the major frame work components of the fibre (Leonard and Martin, 2002). According to the type of fibre, cellulose has its own cell geometry which is responsible for the determination of mechanical properties of plant fibres. Hemicelluloses occur mainly in the primary cell wall and have branched polymers carbon sugars with varied chemical structure. It comprises a group of polysaccharides compiled of a combination of five and six carbon ring sugars. It differs from cellulose in three aspects firstly it contains several sugar units. Secondly, they exhibit a considerable degree of chain branching containing pendent side groups which give rise to its ion crystalline nature. Thirdly, is the degree of polymerization which is about 50-30 in the case of hemicelluloses. However, in cellulose it is 10-100 times more than that of hemicelluloses. Hemicellulose is very hydrophilic, soluble in alkali and easily hydrolyzed in acids. Lignin is amorphous and has an aromatic structure (Fakirov and Bhattacharya, 2007). Lignin is a complex hydrocarbon polymer with both aliphatic and aromatic constituents and it is totally insoluble in most of the solvents and cannot be broken down into monomeric units. Lignin is totally amorphous and hydrophobic in nature. It is not hydrolyzed by acids, but soluble in hot alkali, readily oxidized and easily condensable with phenol (Bismarck *et al*, 2005). It is important to remove lignin because, despite the fact that it increases the stiffness of the fibre, it is inflexible and prevents the re-orientation of the fibres required for the proper transfer of load (Santos *et al*, 2006; Esmeraldo 2006; Saha *et al*, 2010; Venson, 2008). In addition lignin acts as cement between fibrilles, and when removed, allows an increase in surface area, thereby improving the fibre-matrix

adhesion provided by alkaline treatment. Pectin structure is complex, their side chains are often cross-linked with the calcium ions and arabinose sugars. Pectin is a collective name for heteropolysachrides. They provide flexibility to plants. Waxes make up the last part of fibres and they consist of different types of alcohols. The lignin, hemicelluloses and pectin provides the adhesive tohold the cellulose frame-work structure of the fibre together. Natural fibres also composed of a small amount of organic (extractives) and inorganic (ash) components. Organic extractives are responsible for the colour, odour and decay resistance, and inorganic matters enhance abrasive nature of the fibre (Saheb and Jog, 1999).

Their structural compositions (such as cellulose, hemicelluloses, lignin, pectin and waxy substances) allow moisture absorption from the environment which leads to poor bonding with the matrix materials (Doan *et al,* 2006). Certain chemical treatment on natural fibres is needed to enhance the performance as reinforcement in polymer composite materials. The chemical modification directly influences the fibres structures and changes their compositions. As a result, it facilitates better bonding with the matrix materials. This provides better mechanical and thermal properties of fibre and composites (Abdelmouleh *et al,* 2007).

Sisal fibres are made from sisal plant leaves. The word sisal means cold water. Sisal fibre occupies 6th place among fibre plants, which represent 2% of the world's production of plant fibres (plant fibres provide 65% of the world's fibres).



Fig 2.1 The sisal fibre (Rong *et al,* 2001).

2.3.1 Structure of Sisal Fibres

Sisal fibre is fairly coarse and inflexible. It is valued for cordage use because of its strength, durability, ability to stretch, affinity for certain dyestuffs, and resistance to deterioration in salt water. Sisal is a strong, stable and versatile material and it has been recognized as an important source of fibre for composites. (Joseph *et al*, 1997; Singh *et al,* 2003; Luyt and Malunka, 2005).

A sisal fibre in cross-section is built up of about 100 fibre cells. Figure 2.2 below shows a schematic sketch of a fibre cell. The cell walls consist of several layers of fibrillar. In the primary wall, the fibrillae have a reticulated structure. In the outer secondary wall (S_1), which is located inside the primary wall, the fibrillae are arranged in spirals with a spiral angle of 40° (for sisal fibre) in relation to the longitudinal axis of the cell. The fibrillae in the inner secondary wall (S_2) of sisal fibres have a sharper slope, 18° to 25°. The thin, innermost, tertiary wall has a parallel fibrillar structure and encloses the lumen. The fibrillae are, in turn, built up of micro-fibrillae with a thickness of about 20µm. The microfibrillae are composed of cellulose molecular chains with a thickness of 0.7µm and a length of a few µm.

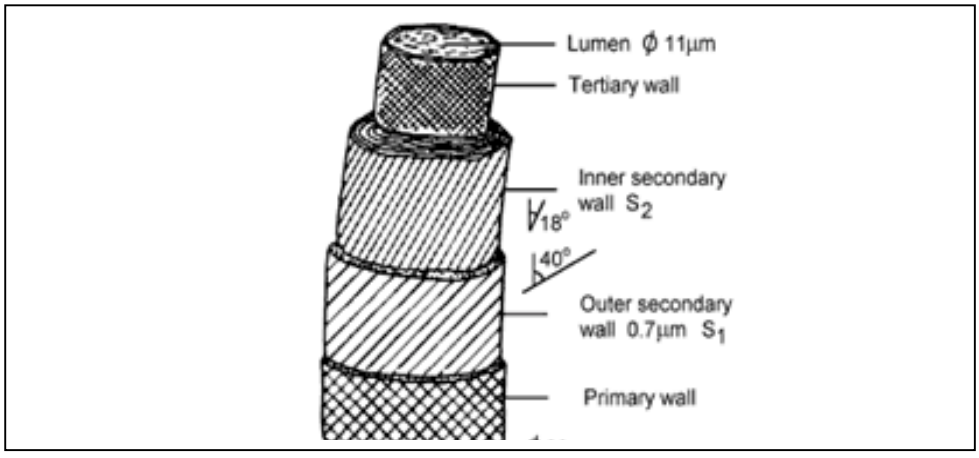


Fig 2.2 Schematic sketch of a fibre cell (Rong *et al,* 2001)

The sisal fibre is mainly cellulosic in nature. Cellulose structure is composed of crystalline and amorphous regions. Strong intra-molecular hydrogen bonds with large molecules are formed by the crystallite cellulose. Compactness of crystalline region creates cellulose blocks that makes it difficult for chemical penetration to occur. However, dyes and resins are absorbed by amorphous region easily. In addition to this, plant fibres e.g. sisal are highly polar and hydrophilic in character as hydroxyl groups are present in their structures. Other non fibre sisal plant materials have their own importance inthe form of medicine such as camellia sinesis and their leaves are used for making cigarettes and its powder is used for relief of asthma (Kumar *et al,* 2010). Much of natural product obtained from plants have its own medicinal values such as biologically active phytochemicals which are normally present in leaves, roots, barks and flowers (Thillai and Venkataraman, 2010) and there are number of medicinal plants which possess anti fertility property. (Dubey *et al,* 2011).

The fibre is actually a bundle of hollow sub-fibres. Their cell walls are reinforced with spirally oriented cellulose in a hemi-cellulose and lignin matrix. So, the cell wall is a composite structure of ligno-cellulosic material reinforced by helical microfibrillar bands of cellulose. The composition of the external surface of the cell wall is a layer of lignaceous material and waxy substances which bond the cell to its adjacent neighbours. Hence, this surface will not form a strong bond with a polymer matrix. Also, cellulose is a hydrophilic glucan polymer consisting of a linear chain of 1, 4-β-bonded anhydroglucose units (Li *et al,* 1987). And this large amount of hydroxyl groups will give sisal fibre hydrophilic properties. This will lead to a very poor interface between sisal fibre and the hydrophobic matrix and very poor moisture absorption resistance.

2.3.2 Chemical Composition of Sisal Fibres

The chemical composition of sisal fibres have been reported by several groups of researchers. For example, Wilson indicated that sisal fibre contains 78% cellulose, 8% lignin, 10% hemi-celluloses, 2% waxes and about 1% ash by weight. Rowell found that sisal contains 43 – 56% cellulose, 7 – 9% lignin, 21-24% pentosan and 0.6 -1.1% ash. More recently, (joseph *et al* 2001) reported that sisal contains 85-88% cellulose. These large variations in chemical compositions of sisal fibre are a result of its different source, age, measurement methods, etc. Indeed, Chand and Hashmi (1993) showed that the cellulose and lignin contents of sisal vary from 49.62-60-60.95 and 3.7-4.40%, respectively depending on the age of the plant.

2.3.3 Sisal Fibre Processing/Extraction

The processing methods for extracting sisal fibres include (1) retting followed by scraping and (2) mechanical means using decorticators (i.e. beating the sisal).Mechanical process yields about 2-4% fibre (15 kg per 8 h) with good quality having a lustrous colour while the retting process yields a large quantity of poor quality fibres. The leaves were crushed and beaten manually by a smooth edged stick so that only fibres remain. After extraction, the fibres were washed thoroughly in plenty of clean water to remove the surplus wastes such as chlorophyll, leaf juices and adhesive solids. The fibres are then dried in open hot air. The fibre quality depends upon its moisture content so thus proper drying is important. The dried sisal fibre strands are usually creamy white in colour (Murherjee and Stayanarayana, 1984).

2.3.4 Properties of sisal fibre

The physical properties of sisal fibres vary from source to source (Chand *et al,* 1998). Fibres from natural sources have been considered as the most promising dressing material. Sisal fibre is exceptionally durable with a low maintenance with minimal wear and tear and it is recyclable. The length of sisal fibre is between 1.0 and 1.5 m and the diameter is about 100-300 mm. (Bisanda and Ansell, 1991). Sisal fibres are obtained from the outer leaf skin, removing the inner pulp. Sisal fibres are anti static, do not attract or trap dust particles and do not absorb moisture or water easily. The fine texture takes dyes easily and offers the largest range of dyed colours of all natural fibres. It exhibits good sound and impact absorbing properties. Its leaves can be treated with natural borax for fire resistance properties. Among the various natural fibres, sisal fibre is fairly coarse and inflexible. It possesses moderately high specific strength and stiffness, durability, ability to stretch, and resistance to deterioration in salt water (Sapuan and Harimi, 2003; Joseph *et al,* 1999). Therefore, it can be used as a reinforcing material in polymeric resin matrices to make useful structural composite materials (Silva *et al,* 2010; Singh *et al,* 2011).

2.3.5 Uses of sisal

From ancient times, sisal has been the leading material for agricultural twine because of its strength, durability, ability to stretch, affinity for certain dyestuffs, and resistance to deterioration in salt water. It is mainly used as ropes in marine industry and agriculture. (Mukherjee and Satyanarayan,1984), Other applications of sisal fibres include twines, cords, upholstery, padding and mat making, fishing nets, fancy articles such as purses, wall hangings *etc.*(Chand,*et al,*1984). It has also been used to manufacture corrugated roofing panels that are strong and cheap with good fire resistance. (Bisanda, 1992), Apart from

ropes, twines and general cordage sisal is used in low-cost and specialty paper, dartboards, buffing cloth, filters, geotextiles, mattresses, carpets, handicrafts, wire rope cores and Macrame. Sisal fibres are great alternative to plastic. Woven floor covering, floor tiles, rugs, wall coverings, wainscoting and fabric panels, Handbags, shopping bags etc are now made from Sisal fibre. Now sisal has been utilized as an environmentally friendly strengthening agent to replace asbestos and fibre glass in composite materials in various uses including the automobile industry. The lower grade fibre is processed by the paper industry because of its high content of cellulose and hemicelluloses. The medium grade fibre is used in the cordage industry for making: ropes, baler and binders twine. Ropes and twines are widely employed for marine, agricultural, and general industrial use. The higher-grade fibre after treatment is converted into yarns and used by the carpet industry.

Other products developed from sisal fibre include spa products, cat scratching posts, lumbar support belts, rugs, slippers, cloths and disc buffers. Sisal wall covering meets the abrasion and tearing resistance standards of the American Society for Testing and Materials and of the National Fire Protection Association. Sisal carpet does not build up static nor does it trap dust. Spot and high spill areas can be removed by dry cleaning powder. Sisal is used by itself in carpets or in blends with wool and acrylic for a softer hand. Sisal is also valuable forage for honey bees because of its long flowering period. It is particularly attractive to them during pollen shortage. The honey produced is however dark and has a strong and unpleasant flavor. Natural fibres found to have extensive applications in building and civil engineering fields. In case of synthetic fiber based composites, despite the usefulness in service, these are difficult to be recycled after designed service life. However, natural fibre based composites such as sisalis environment friendly to a large

extent. Though hydrophilic character of sisal fibres would leads to composites with weak interface but pretreatments are aimed at improving the adhesion between fibres and matrix. In pretreatments,the hydroxyl groups get activated and can effectively interlock with the matrix (Xli *et al,*2007; Eichhorn *et al,*2003; Sain and Panthapulakkal, 2005; Weyenberg *et al,* 2003).

Traditionally Sisal fibres can be classified into a variety of grades which include; the lower, medium and higher fibres, which are used for paper, rope and carpet making, respectively. Sisal has however found new applications in a variety of industries which include; the medical field (Thatoi *et al*, 2008), the automotive and Building industry (Salazar *et al*, 2011; Filho *et al*, 2009). Apart from the sisal fibre which constitutes 4% of the sisal leaf, the sisal industry generates a lot of biomass commonly referred to as sisal waste. The biomass obtained during sisal decortications has for a long time been a nuisance to the sisal factories. While the factories were struggling to cope with the sisal waste another event seemingly unrelated came to the rescue of the sisal factories. The high rate of depilation of natural resources has compelled man to optimize the use of resources. Terms like "eco-friendly" or "green" processes are now common words in many government and industrial circles. While Kotchoni and Gachomo, (2008), paints a picture of bio-fuel as a promising concept Ramachandra, (2008) assumed that green fuel is here with us and he goes ahead to design a model which can be used to assess the need for biogas for a given region. Green energy is here to stay, since the fossil fuels are getting depilated at a high rate. In Tanzania, sisal biomass, which was a nuisance 20 years ago, is being used to generate electricity hence, improving the profitability of the sisal industry. Sisal biomass can also be used to manufacture bio-fertilizers, since crop residue have proven potential for the manufacture of

fertilizers (Taiwo, 2011). Scientists are currently carrying out research to use sisal waste for bio-energy generation, livestock feed as well as organic fertilizer. This enhanced interest and investment in research and development will improve production and hopefully the profitability of the sisal industry.

The increasing use of sisal in a variety of industries demands a thorough understanding of the properties of the fibre. According to De Andrade and Filho, (2007) the sisal plant leaf is a composite structure that is made up of three types of fibres: Mechanical, Ribbon and xylem fibres. Every sisal fibre contains numerous elongated individual fibres (fibre-cells). Each individual fibre-cell is made up of four main parts, namely the primary wall, the thick secondary wall, the tertiary wall and the lumen. Sisal leaves can be harvested from the age of 2 years. Harvesting can continue for 9 to 12 more years, producing over 200 leaves in its lifespan. This research concentrated on the study of the characteristics of the sisal fibre. Previous works indicated that the mechanical and chemical characteristics of sisal may vary depending on growing and processing conditions. The mechanical propertiesof the fibre have for example been shown to be dependent on the age of the fibre (Chand and Hashmi,1993). A regression model designed by Mukherjee and Satyanarayana, (1996) to study sisal fibre revealed that the structure and properties of sisal are affected by cellulose content, microfibrillar angle and other structural parameters which include cell dimensions and defects. Therefore, characterization of the sisal will be important, since it will shed light on the quality of the sisal and hence encourage its appropriate application.

The use of sisal in a wide range of application has lead to an increase in sisal demand. This has been accompanied by an improvement of the prices of sisal, hence making sisal production a very profitable venture.

2.4 Modification of Sisal Fibres

2.4.1 Interface Modification

Interface plays an important role in the physical and mechanical properties of composites (Zhou *et al*, 1995; Kim *et al,* 1993).The fibre-matrix interface is the diffusion or reaction zone, in which fibre and matrix phases are chemically and/or mechanically combined. Interfacial adhesion between fibre and matrix plays a predominant part in characterizing the mechanical properties of the composites. If there is a poor adhesion across the phase boundary, then relatively weak dispersion of force occurs and resulting poor mechanical properties of the composite (Wang *et al*, 2007). For reinforcement of natural fibre in composites, several problems occur along the interface due to the presence of hydrophilic hydroxyl groups on the fibre surface. This hydrophilic nature hinders effective reaction with the matrix. In addition to this, pectin and waxy substance covers the reactive functional groups of the fibre and act as a barrier to interlocking with the matrix. To optimize effective interfacial bonding between fibre and matrix, the fibre surface needs tobe modified with different chemical treatment. Chemical treatments expose more reactive groups on the fibre surface and thus facilitate efficient coupling with the matrix. As a result, better mechanical and thermal properties of the composites can be achieved (Dash *et al,* 2000).

2.4.2 Chemical Modification

Chemical treatments can be applied to the fibres to change surface tension and polarity through modification of fibre surface (Scandola *et al,* 2000). Chemical modification has been known to enhance interface between the hydrophilic sisal fibre and the hydrophobic polymer matrices. Both mechanical and moisture absorption resistance properties can be improved. An indept account of surface modification of cellulosic fibres has been reported by Belgacem and Gandini, (2005). The authors are of the opinion that the most promising approach of chemical modification seemed to be the one that gave rise to continuous covalent bond between the fibre surface and the matrix.These treatments are mainly chemical methods with the goal of modifying the surface of the fibres. (Bassadok *et al*, 2009; Pietak *et al,* 2007) e.g alkali treatment, acetylation treatment, silane treatment and so forth. According to Alvarez *et al*, (2006), although the surface of the sisal fibres can be modified by these treatments, but they found out that alkalization was more effective.The reaction of sodium hydroxide with cellulose is thought to be as follows.

Cell - OH + NaOH → Cell - ONa + H_2O

It is worth pointing out that alkalization polymerizes the native cellulose molecular structure producing short length crystallites. However, there seems to be varying interpretations of the term 'alkalization'. The standard definition for Alkalization proposed by ASTM D (1983) is "the process of subjecting a vegetable fibre to the action of a fairly concentrated aqueous solution of a strong base so as to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties". Zeronian, (1985), proposes another definition of alkalization, which is suitable for basic

research and states that: Alkalized cellulose is "a sample of cellulose which has been treated with a solution of an alkali hydroxide of sufficient strength to cause essentially complete conversion of the crystal structure from cellulose I to II". (Atkins, 1979) reports that alkalization allows total Conversion of cellulose I to cellulose II to take place.

2.5 Alkali treatment of Sisal Fibre

Alkali treatment of natural fibres is the common method to produce high-quality fibres Alkali treatment can removenatural and artificial constituents such as hemicelluloses, lignin, pectin, wax and oil covering materials (Xue, *et al,* 2007; Leonard *et al,* 2002; Dipa *et al,* 2001). In addition, alkali treatment reduces the level of fibre aggregation which leads to fibre fibrillation, i.e. breaking down the fibre bundle into smaller fibrils.This increases the effective surface area available for wetting by the matrix resin and thereby making the surface rougher. (Troedec *et al,* 2008; Esmeraldo, 2006; Gomes *et al,* 2007).This increases the fibre aspect ratio caused by reduced fibre diameter and produces a rough surface topography,which offer better fibre/matrix interface adhesion and increase in mechanical properties (Joseph *et al,* 2000). Several workers have performed work on alkali treatment (Joseph*et al,* 2000; Streekala *et al,* 2001; Morrison *et al,* 2000; Mishra *et al,* 2002) and reported that alkalization leads to an increase in the amount of amorphous cellulose at the cost of crystalline cellulose and the removal of hydrogen bonding in the network structure. The important modification occurring here is the removal of hydrogen bonding in the network structure. Moreover, alkalization increases the number of possible reactive sites and allows better fiber wetting. Alkali solution not only affects the

cellulosic components inside the plant fiber but also affect the non cellulosic components such as hemicellulose, lignin and pectin (Weyenberg *et al,* 2006).

Mwaikambo and Ansell, (2006) carried out the alkali treatment of sisal fibres and analyzed the changes with respect to the diameter and internal structure, such as cellulose content, crystallinity index, and micro-fibril angle. Alkalization was found to change the internal structure of sisal fibres which exhibited approximately same specific stiffness as that of steel. The crystalline nature of the treated fibres was also found to increase due to alkali treatment. Their results indicated that the structure of sisal fibre can be chemically modified to attain properties that will make the fibre useful as a replacement for synthetic fibres

2.6 Environmental impact

Increasing environmental awareness throughout the world has prompted design of materials that are compatible with the environment. Currently, synthetic fibres such as glass, carbon andaramid are being widely used in polymer composites because oftheir high stiffness and strength properties (Rout *et al,* 2001). However, these fibres have serious drawbacks in terms of biodegradability, initial processing cost, recyclability, energy consumption, machine abrasion, health hazards etc. (Bledzki and Gassan, 1998). Despite this, most significantly, adverse environmental impact alters attention from synthetic fibre to natural fibres. Introduction of biofibres such as natural fibres from annually renewable resources are gaining interest to be used as reinforcement in polymer composites to provide benefits to the environment with respect to the degradability and utilization of renewable materials (Rout *et al,* 2001).

Sisal farming initially caused environmental degradation, because sisal plantations replaced native forests, but is still considered less damaging than many types of farming. No chemical fertilizers are used in sisal production, and although herbicides are occasionally used, even this impact may be eliminated, since most weeding is done by hand. Although the effluent from the decortications process causes serious pollution when it is allowed to flow into water courses, plans are been laid down to use the waste as bio-fuel.

CHAPTER THREE

3.0 MATERIALS AND METHOD

3.1 Sisal Fibre

The sisal fibres used for this study were obtained from the Botanical garden of the Ahmadu Bello University, Zaria.

3.2 Chemicals

Chemicals used are: Aqueous sodium hydroxide solution (5, 10, 15 and 20% by weight) and acetic acid (2% by weight).

3.3 EQUIPMENT/INSTRUMENTATION

The following equipment/instruments were used for the study:

i. Scanning Electron Microscope(EVO MA10 Carl Zeiss, High resolution SEM)

ii. Instron Tensile Tester (model 1025).

iii. Oven

iv. Digital Weighing Balance.

v. Basic laboratory glasswares, such as, conical flasks, beakers, measuring cylinders, thermometer, stirring rod, and volumetric flask

3.4 METHOD

3.4.1 EXTRACTION OF THE SISAL FIBRES

The leaves were crushed and beaten manually by a smooth edged stick so that only fibres remain. After extraction, the fibres were washed thoroughly in plenty of water to remove

surplus wastes such as chlorophyll, leaf juices and adhesive solids (hemicelluloses). The fibres were then dried in open air. Dried sisal fibre strands are usually creamy white in colour.

3.4.2 Chemical Modification of the Fibres

2 grams of the sisal fibres were soaked in 5, 10, 15 and 20% NaOH solution for 5, 10 and 15 Minutes each at a temperature of 65^0C. These fibres were further rinsed with water followed by neutralization in 2% acetic acid solution. A final rinse in water and then dried at room temperature.

$Cell–OH + NaOH \rightarrow Cell–O^-Na^+ + H_2O$

It has been known that during alkali treatment, the OH group present in cellulose fibres reacts with sodium hydroxide and this interaction between the fibre and NaOH is an acid-base reaction that should not occur in quantitative manner due to the weak acid character of the hydroxyl groups.

3.5 Determination of moisture content and moisture regain

The sisal fibres were weighed and dried in an oven at a temperature of 103°C for 30 minutes, followed by cooling for 30 minutes and then weighed again. This step was repeated until the weight was constant. The moisture content of the test specimen was expressed as a percentage loss in weight of the initial weight using the following equation;

$$\text{Moisture Content} = \frac{W_0 - W_1}{W_0} \text{ x } 100\ (\%)$$

Where Wo is the weight of fibre before drying and W_1 is the weight of fibre after drying. Also the moisture content is related to moisture regain using the following formula (Booth, 1969).

$$R = \frac{M}{1 - \left[\frac{M}{100}\right]}$$

Where R=moisture regain

M =moisture content

The effect of concentration of alkali and time on the moisture regain and moisture content were determined for both the modified and unmodified sisal fibres. Ten tests were carried out and the mean values are reported in table 4.1

3.6 DETERMINATION OF SURFACE CHARACTERISTICS

The nature of the surface of the sisal fibre is very important as this determines to a large extent the properties of the fibre. In order to evaluate changes in the surface morphology, the surface characteristics of the untreated and chemically modified sisal fibres were studied using the Scanning Electron Microscope (SEM). The SEM micrographs were used to study the surface of the unmodified sisal fibre as compared to the surface of the chemically modified fibres. The results obtained are presented on plates I to VII

3.7 DETERMINATION OF MECHANICAL (TENSILE) PROPERTIES

The mechanical (tensile) properties of the treated and untreated sisal fibres were comparatively determined according to ASTM D3822. The following tensile properties were determined using the Instron tensile tester; tenacity, percentage extension, initial young's modulus and work of rupture. For all samples, the stress-strain curves were obtained at four different gauge lengths (10cm, 15cm, 20cm and 25cm). The rate of extension was 25% per minute and a maximum load of 50kg was used for all tests. Cross head speed was 50mm per minute and chart speed was 50mm per minute. The tenacity and percentage extension were calculated using the following relationship;

Percentage extension $= \frac{\boldsymbol{elongation}}{\boldsymbol{initiallength}} x100(\%)$

Tenacity $= \frac{Load}{Mass/Unit\ length}$ N/tex

Linear density in Tex $= \frac{w\,x\,1000}{100L}$

Where w= weight of the fibre, L=length of the fibre used

The work of rupture was obtained by calculating the area under the stress-strain curve.

The counting of squares method was used; thus:

Specific Work of Rupture $= \frac{(average\ strength\ gm)(utimate\ elongation\ (\%)}{Denier\ (100)}$

The young's initial modulus which is the slope of the stress-strain curve at the origin (after the removal of any crimp) was measured for each sample. The results are as given in Figures 4.1 to 4.28.

3.8 EFFECT OF CONCENTRATION OF CAUSTIC SODA AND TREATMENT TIME

The effect of the concentration of the NaOH and the duration of treatment on the surface characteristics and the tensile properties of the sisal fibres were investigated and appropriate graphs plotted from the results.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Results from the extraction of the sisal fibre: Extraction of the sisal fibre from its leaves by manually beating the sisal leaves with a smooth edge stick was quite tedious due to the presence of surplus wastes such as; leaf juices, adhesive solids and chlorophylls which cause serious itching on contact with the skin.

However after thorough washing in plenty of water, these impurities were largrely removed. It was then dried in open air. The dried sisal fibre strand is creamy white in colour.

4.2 Results from alkali modification of the sisal fibre: After alkali modification, it was observed that there was a colour change from the original creamy white colour of the untreated fibre to a pure cream colour which becomes more obvious according to the extent of modification. This can be attributed to the fact that the NaOH has acted on the sisal fibre altering its internal structure and morphology which reflected in the change of colour.

Another observation was on the handle of the fibre, the alkali treated sisal fibres were softer and more extensible depending on the concentration of caustic soda while the untreated fibres were hard and less extensible. This may be attributable to the fact that alkali treatment removed the residual constituents (lignin, pectin, hemicelluloses and waxy substances), causing the fibre to be soft and more extensible.

4.3 Moisture content and regain for sisal fibres

Table 4.1 Moisture content and regain for sisal fibres at various concentrations and at different times.

	Moisture Content(%)	Moisture Regain (%)	Standard deviation	C V (%)
Untreated	7.90	8.58	0.51	5.94
5%for5mins	10.15	11.29	0.56	4.96
10%for5mins	10.95	12.29	0.59	4.80
15%for5mins	11.89	13.49	0.62	4.59
20%for5mins	12.90	14.81	0.62	4.19
5%for10mins	10.60	11.86	0.57	4.80
10%for10mins	11.76	13.33	0.59	4.43
15%for10mins	12.78	14.65	0.60	4.09
20%for10mins	13.68	15.84	0.61	3.85
5%for15mins	11.58	13.09	0.57	4.35
10%for15mins	12.64	14.46	0.59	4.08
15%for15mins	13.73	15.92	0.60	3.77
20%for15mins	14.53	17.00	0.62	3.65

Usually, chemical modification causes appreciable changes in the moisture absorption for all sisal fibres. Modified sisal has higher regain compared to the unmodified ones. It can be seen from table 4.1 above, that increases in the concentration of NaOH, increase the moisture content and regain as compared to the unmodified fibre.

This increase may be due to the fact that increase in concentration of NaOH, increases the accessible surface of the sisal fibre due to increase in the amorphous region owing to the swelling of the fibres.

The effect of alkali on sisal fibres is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. Native cellulose shows a monocyclic crystalline lattice of cellulose I which can be changed into different polymeric forms through chemical treatments. Increase in the concentration of alkali will influence the degree of swelling and hence the degree of lattice formation into cellulose II (Fengel and Wengener, 1983).

Studies have shown that Na^+ has got a favourable diameter, able to widen the smallest pores in between the lattice planes and penetrates into them. Consequently, (NaOH) treatment results to higher degree of swelling (Wenyenberg *et al,* 2006).

4.4 ASSESSMENT OF SURFACE CHARACTERISTICS (SEM ANALYSIS)

The scanning electron microscope was used for these tests at different concentrations and time. The time was kept constant at 5 minutes, 10 minutes and 15 minutes. For each constant time, the concentration of NaOH was varied at 5%, 10%, 15% and 20%.

The concentration of NaOH was also kept constant at 5%, 10% 15% and 20%. For each constant concentration of NaOH, the time was varied at 5 minutes, 10 minutes and 15 minutes respectively. The SEM micrographs obtained are displayed on plates I to VII.

SEM MICROGRAPHS(PLATE I)

5 minutes treatment

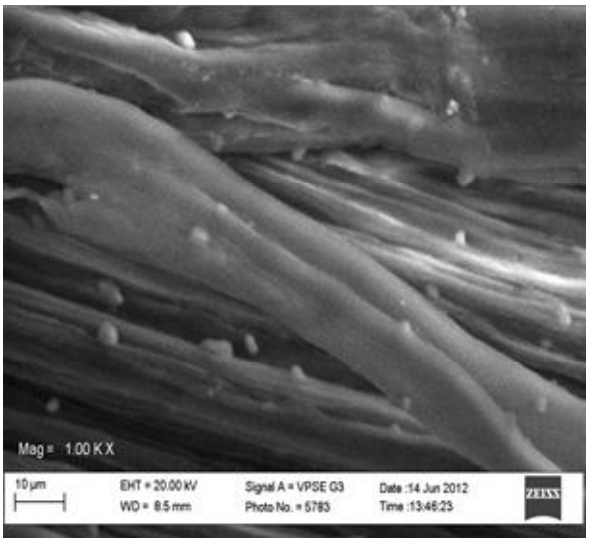


PLATE Ia UNTREATED FIBRE

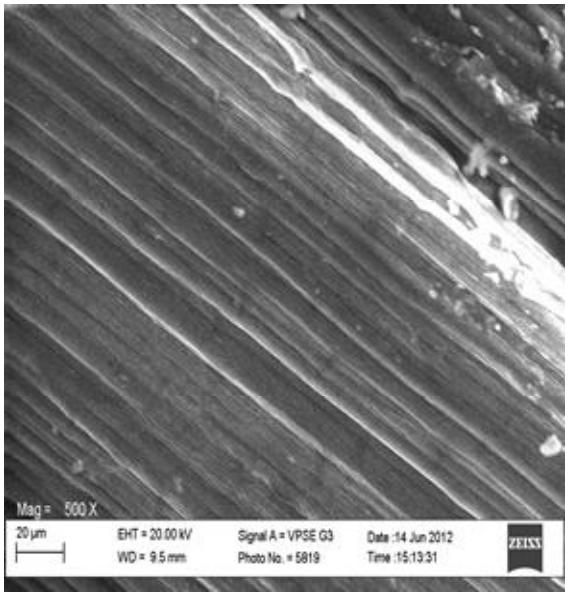


PLATE Ib 5% NaOH

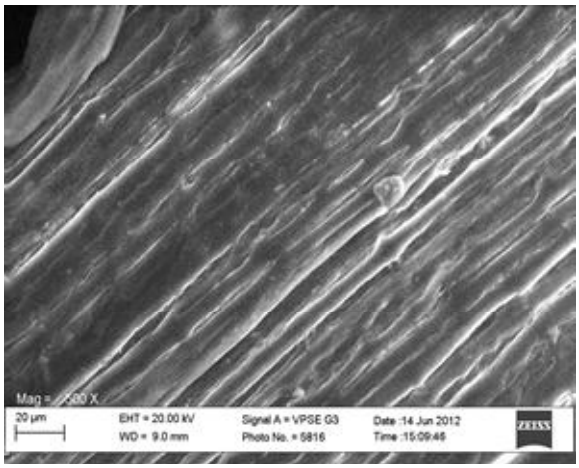


PLATE Ic 10% NaOH



PLATE Id 15% NaOH

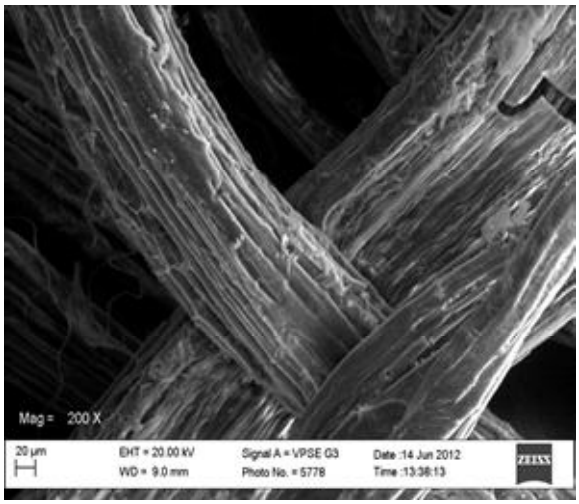


PLATE Ie 20% NaOH

SEM MICROGRAPHS(PLATEII)

10 minutes treatment

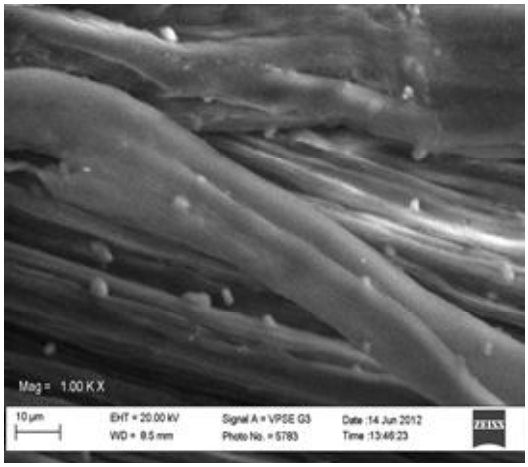


PLATE Ia UNTREATED FIBRE

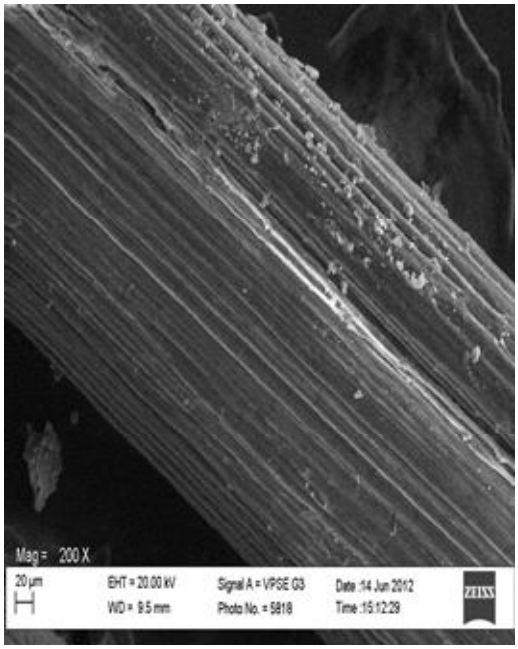


PLATE IIb 5% NaOH

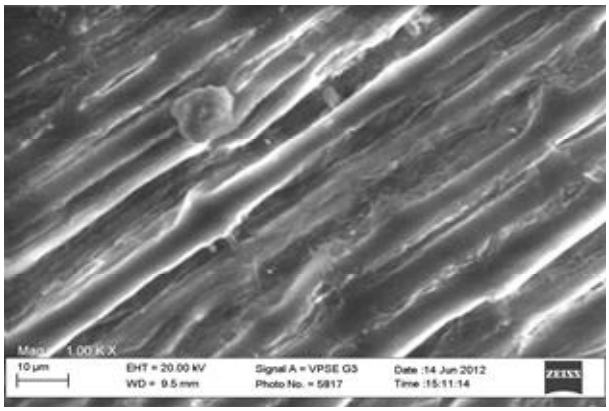


PLATE IIc 10% NaOH

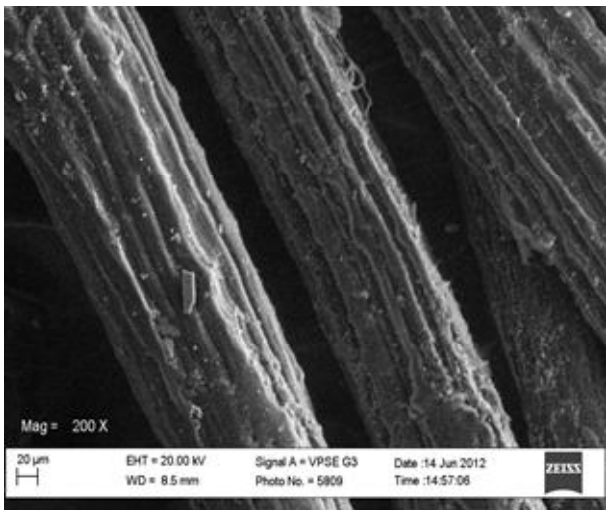


PLATE IId 15% NaOH

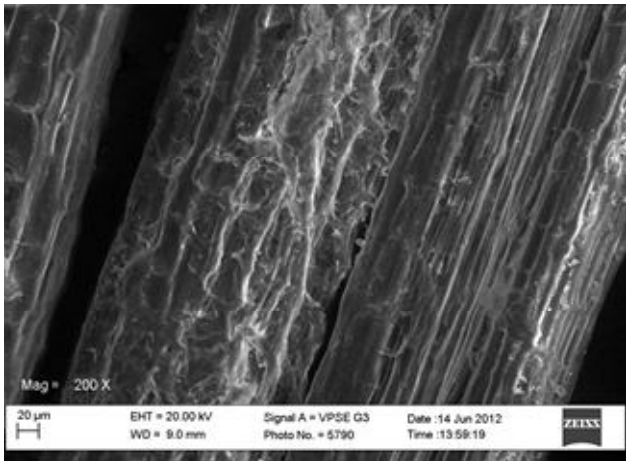


PLATE IIe 20% NaOH

SEM ANALYSIS(PLATEIII)

15 minutes treatment

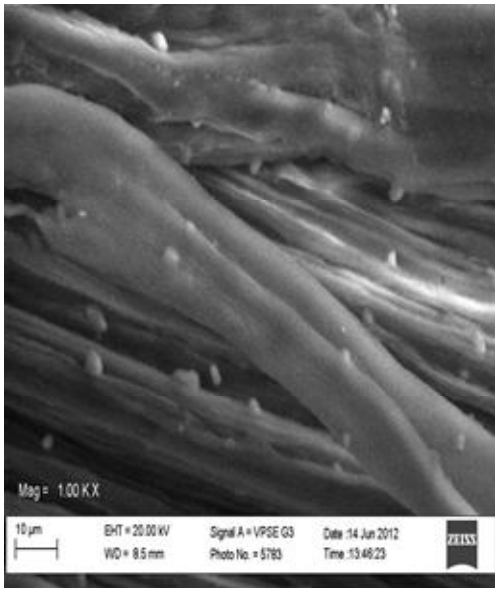


PLATE Ia UNTREATED FIBRE

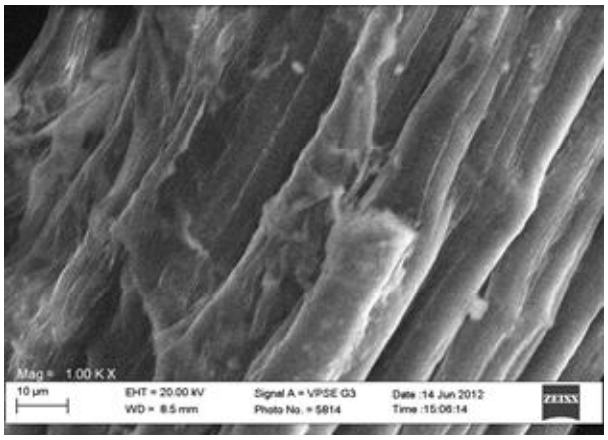


PLATE IIIc 10% NaoH

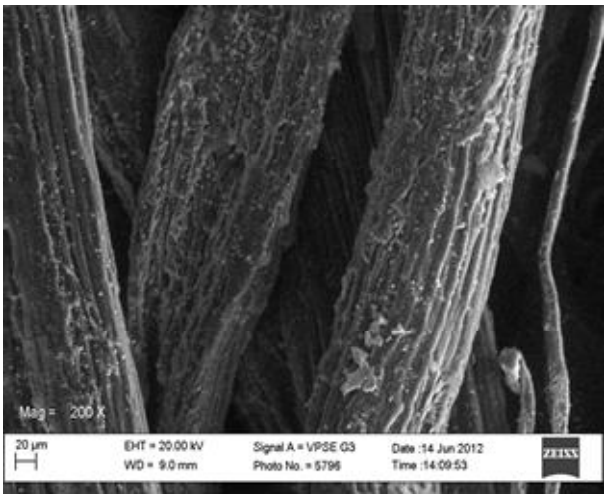


PLATE IIId 15% NaOH

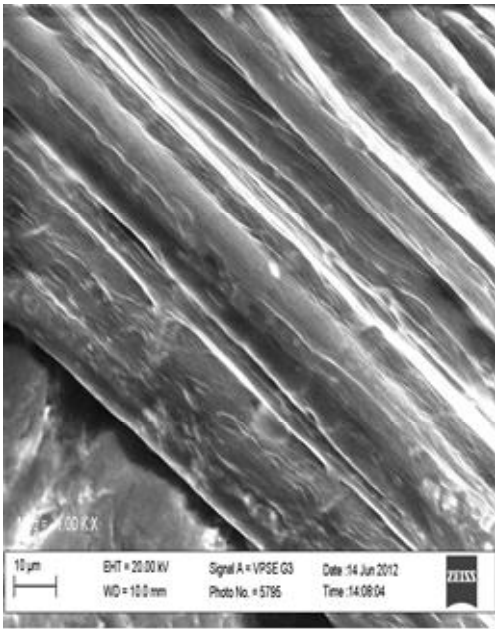


PLATE IIIb 5% NaOH

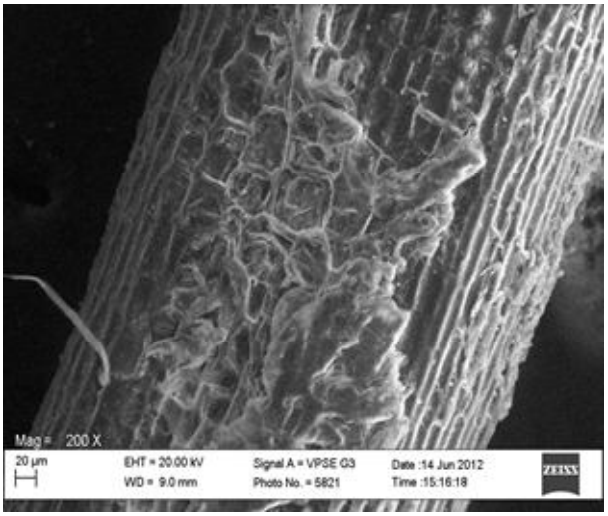


PLATE IIIe 20% NaOH

SEM ANALYSIS(PLATE IV)

5% NaOH concentration

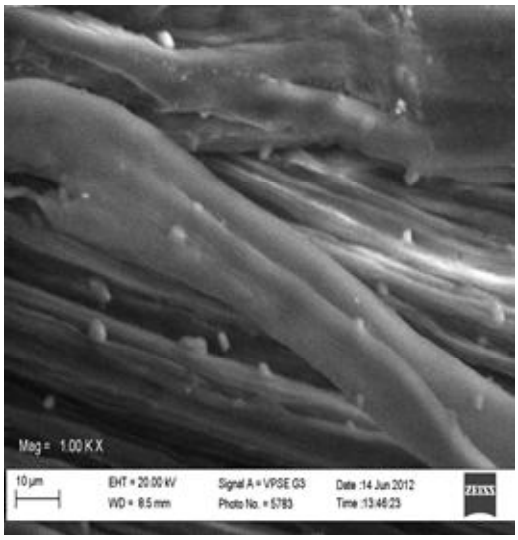


PLATE Ia UNTREATED FIBRE

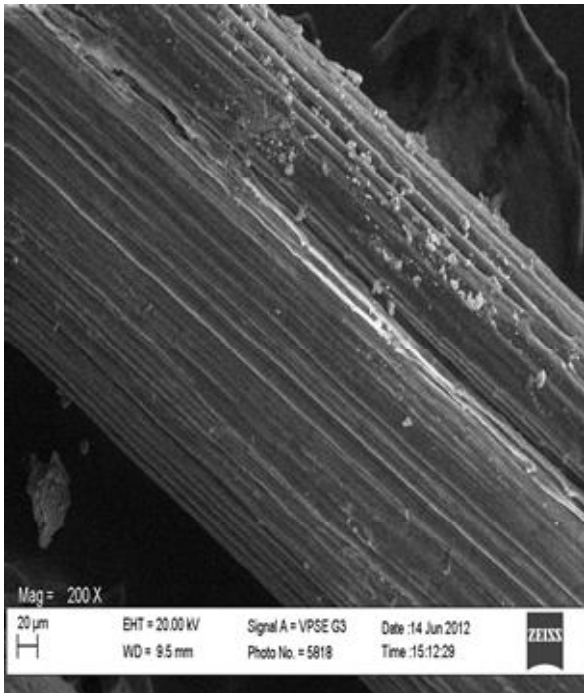


PLATE IVC 10 MINS TREATMENT

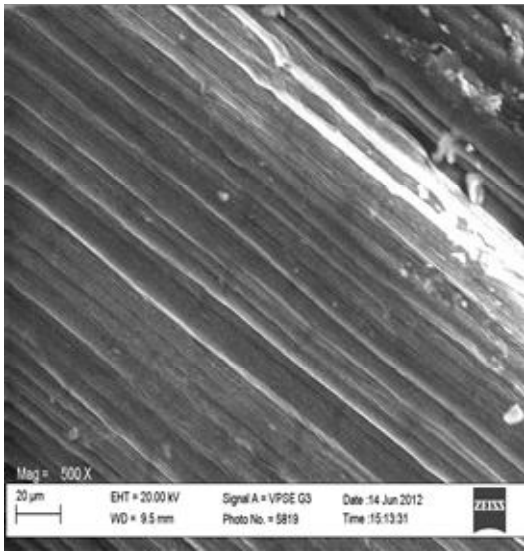


PLATE IVb 5 MINS TREATMENT

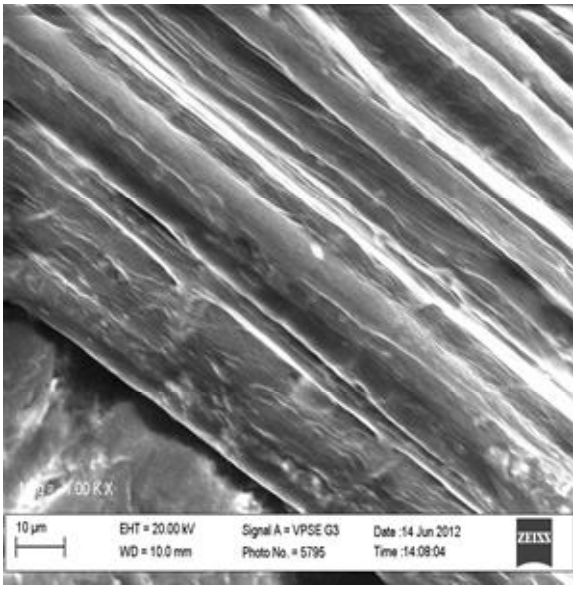


PLATE IVd 15 MINS TREATMENT

SEM ANALYSIS(PLATE V)
(10% NaOH concentration

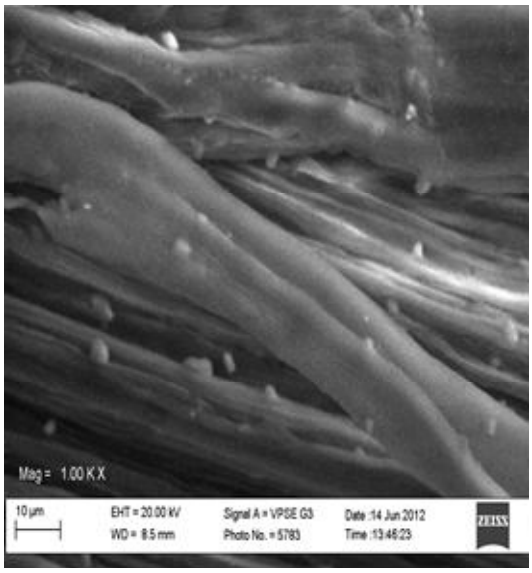


PLATE Ia UNTREATED FIBRE

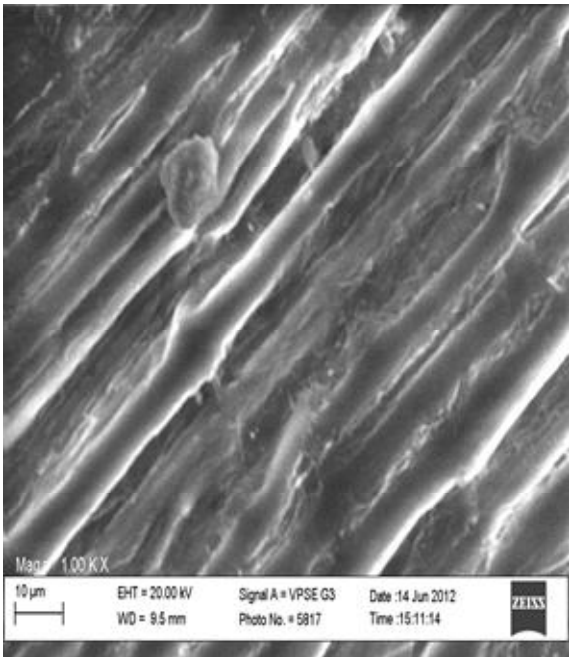


PLATE Vc 10 mins treatment

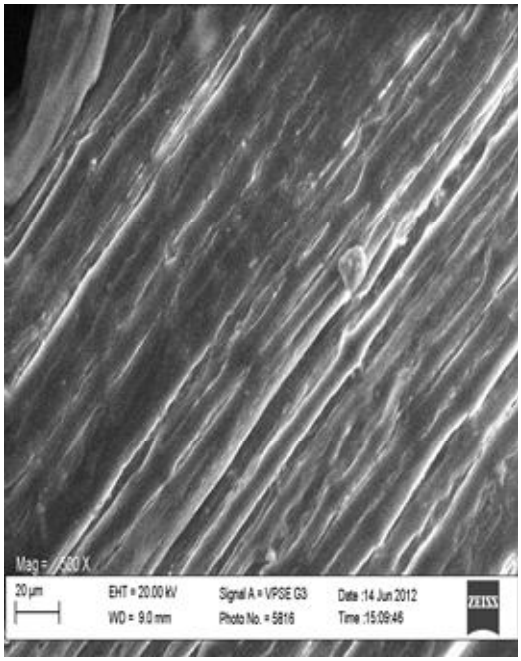


PLATE Vb 5mins treatment

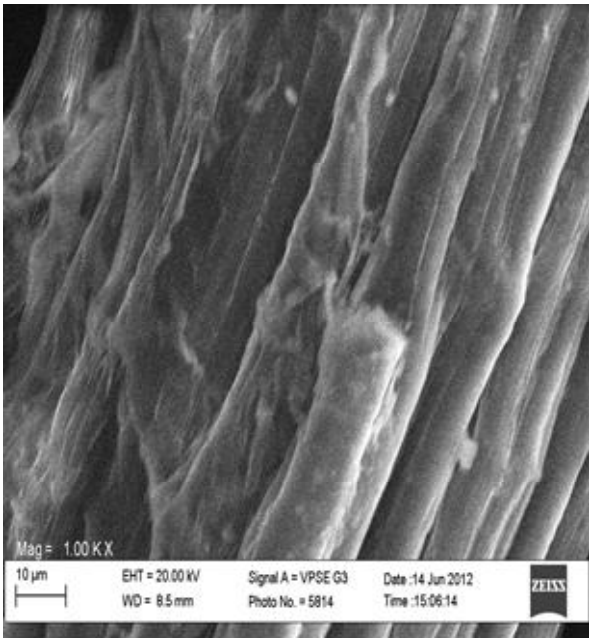


PLATE Vd15mins treatment

SEM ANALYSIS(PLATEVI)
15% NaOH concentration

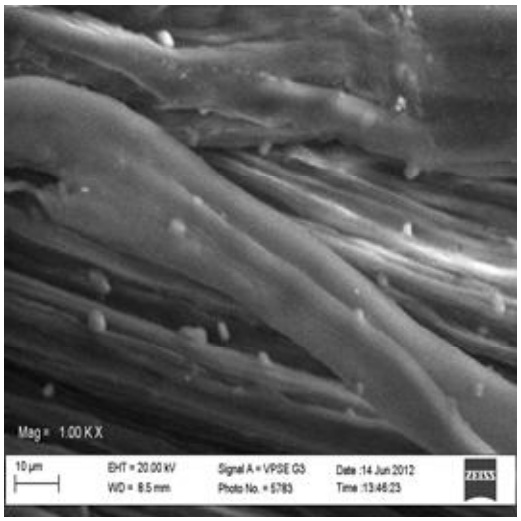


PLATE Ia UNTREATED FIBRE

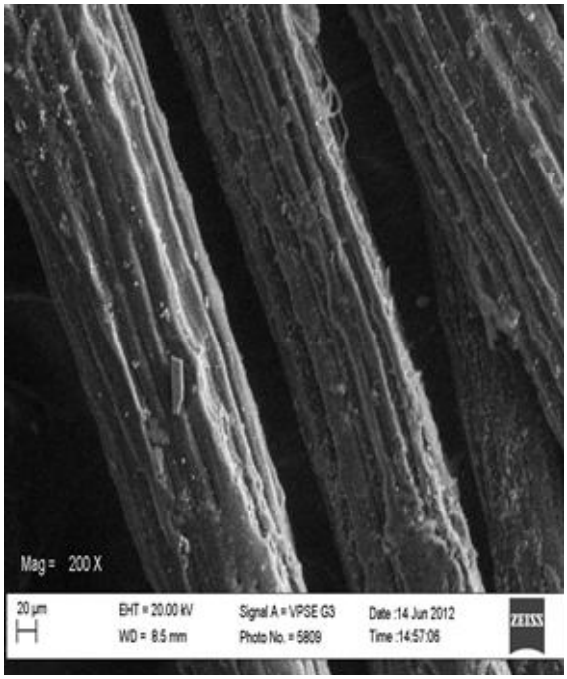


PLATE VIc 10 mins treatment

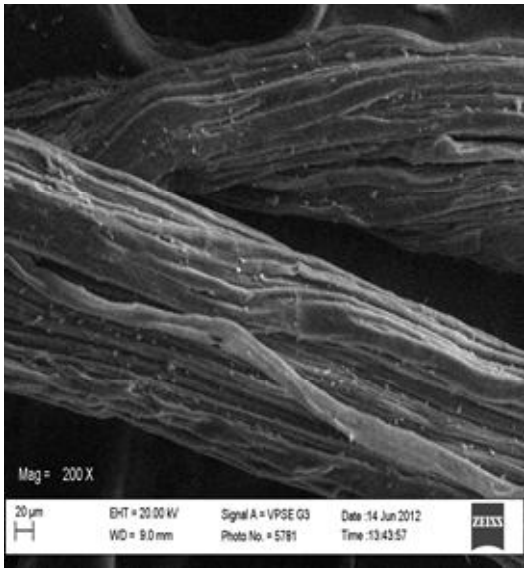


PLATE VIb 5mins treatment

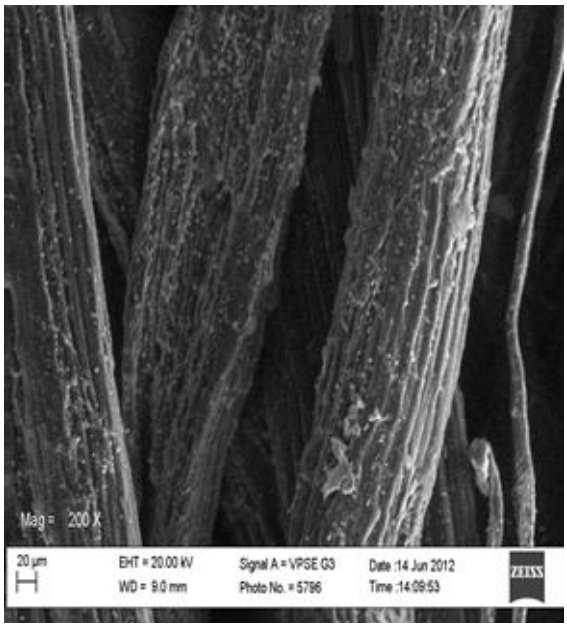


PLATE VId 15mins treatment

SEM ANALYSIS (PLATE VII)
20% NaOH concentration

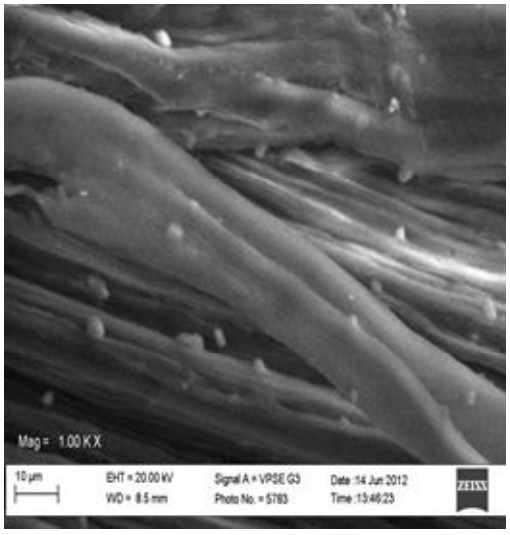


PLATE Ia UNTREATED FIBRE

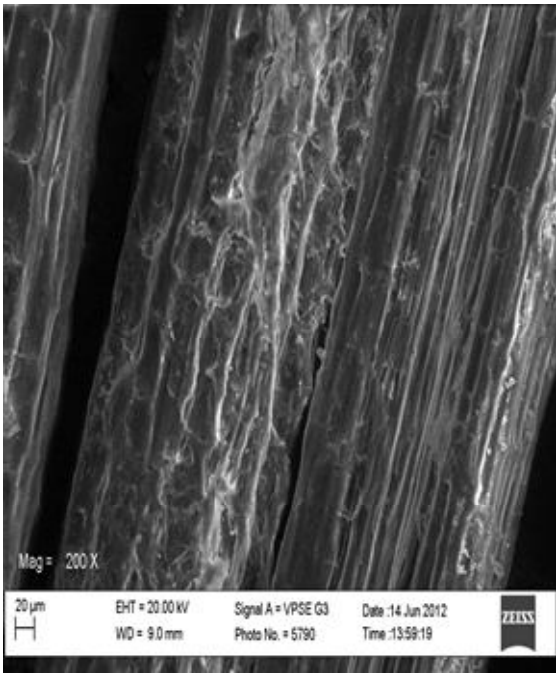


PLATE VIIc 10 mins treatment



PLATE VIIb 5mins treatment

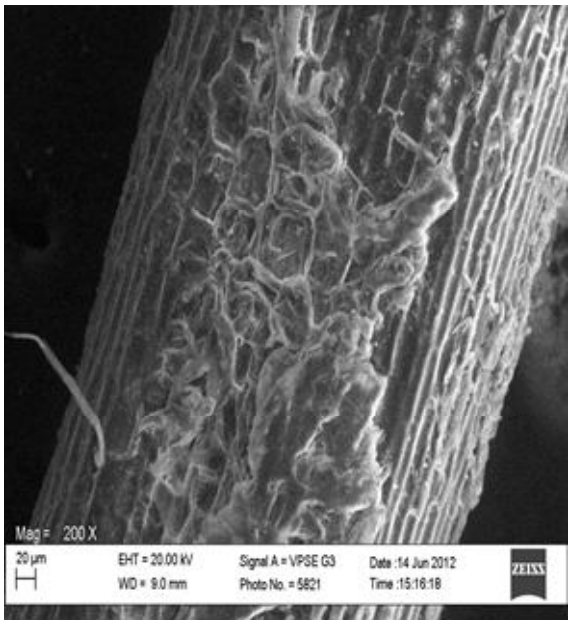


PLATE VIId 15mins treatment

From the above micrographs, Plates Ia show that the surface of the unmodified sisal fibre is wrapped with some "cementing" substances. These substances covered the residual lignin, pectins, hemicelluloses, waxes and oils. However, from Plates Ib to e, Plates IIb to e and Plates IIIb to e, it can be seen that the alkali treatment removed the "cementing substances" This increases the effective surface area available for wetting by the polymer matrix. It is achieved by increasing the fibre aspect ratio (ratio of length to diameter) by reducing the fibre diameter. This is a consequence of the alkali modification and can improve interface adhesion with the polymer matrix. According to Troedec *et al,* (2008), this chemical treatment removes extractives, waxes, and oil from fibre surface. This increases the overall roughness of the surface. After the modification, most of the fatty deposits were removed which revealed on the fibre surface empty cavities.This brings out a rough surface with consequently higher surface energy. The rough surface is made up of pits which are circular holes of about 1μm of diameter. According to Luz *et al,* (2008) and Martin *et al,* (2009), these pits are responsible for transporting water and nutrients throughout various cells to the roots and leaves. In general, pits are hidden on the surface of the untreated fibre due to the superficial layer of waxes and extractives. However, with layer removal by alkali treatments, the pits are revealed. The presence of pits and globular marks after chemical treatments is important for increase in the effective surface area. This also increases the roughness of the fibre surface and consequently improving mechanical bonding with the polymer matrix (Pietak *et al* 2007). However, from plates IVb to e to VIIb to e, increase in time of modification does not have much effect on the surface characteristics of the sisal fibres.

4.5 ASSESSMENT OF THE MECHANICAL (TENSILE) PROPERTIES

Figures 4.1 to 4.28 show the results of the tensile tests (Tenacity, Breaking extension,Young's Initial Modulus, and Work of ruture) obtained at 10cm,15cm, 20cm, and 25cm gauge length respectively for the sisal fibre samples at different concentration (5%, 10%, 15%, 20%) and varied duration (5mins, 10mins and 15mins).

4.5.1 FIBRE TENACITY

(Tenacities at different concentrations, different gauge lengths, but constant time)

Figures 4.1 to 4.3 below give the results of the tenacity values at different concentrations, different gauge lengths but constant time;

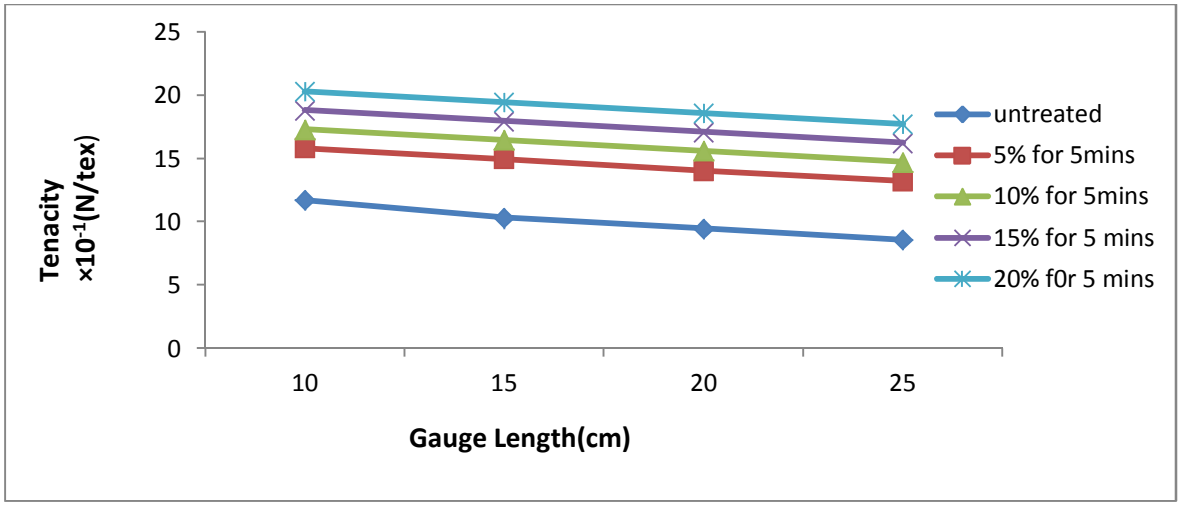


Fig 4.1 Tenacity of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (5 minutes) and at different gauge lengths.

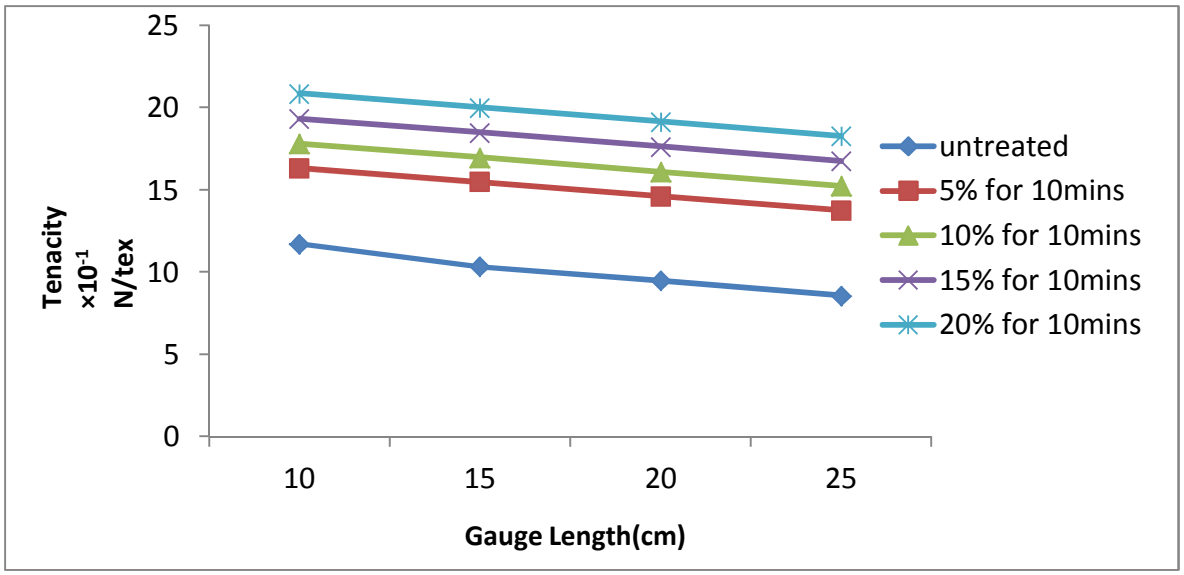


Fig 4.2 Tenacity of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (10 minutes) and at different gauge lengths.

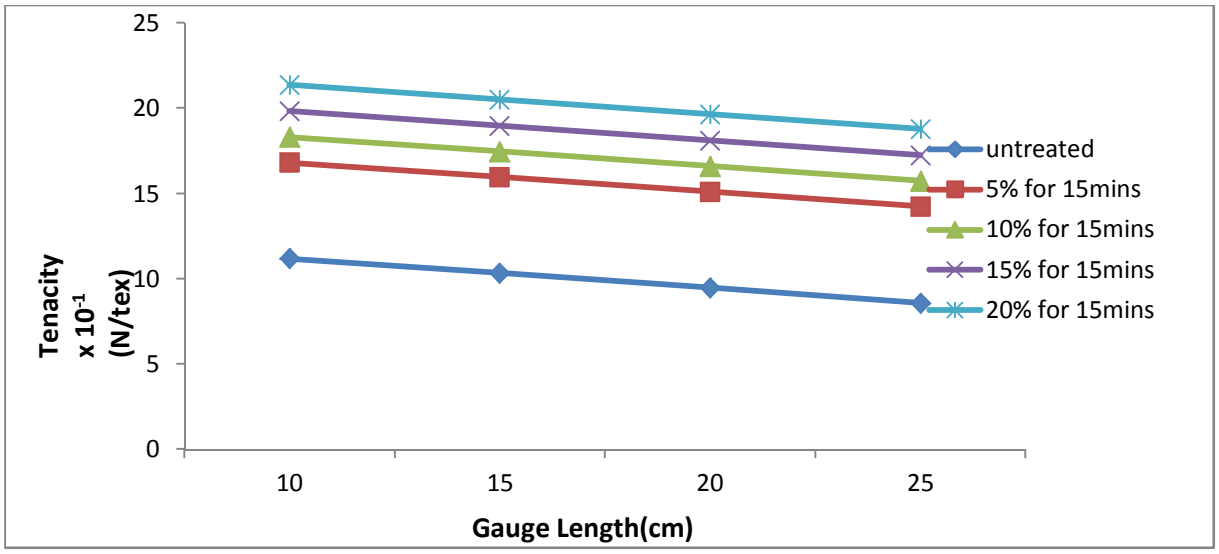


Fig 4.3 Tenacity of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (15minutes) and at different gauge lengths.

Figures 4.1 to 4.3 show that there are differences between the untreated and treated sisal fibres as the modified ones showed increase in their tenacities compared to the unmodified ones.The higher the alkali concentration, the higher their tenacities. However, both the tenacity of the unmodified and modified fibres decreased with increase in gauge lengths. This is attributable to the "weak-link effect". It can be said that the higher tenacities of the modified fibres as compared to the untreated ones, may be due to the increase in strength caused by the increase in concentrations of alkali which improves the adhesive characteristics of fibre surface by removing the residual constituents thereby producing a rough surface topography. In addition, alkali modification leads to fibre fibrillation, i.e. breaking down of the fibre bundles into smaller fibrils and consequently increases in the fibre tenacity. This has increased the proportion of the effective surface area available for contact with matrix polymer.

4.5.2 FIBRE TENACITY

(Tenacities at different time, different gauge length, but constant concentration)

Figures 4.4 to 4.7 below give the result of the tenacity values at different timing, different gauge length but constant concentration;

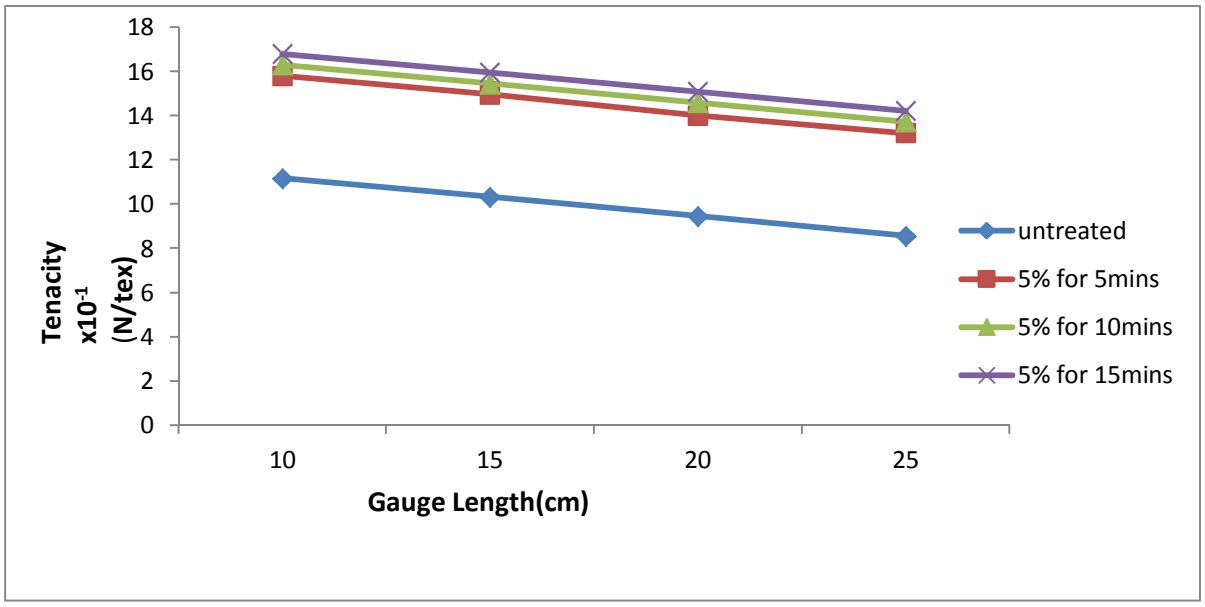


Fig 4.4 Tenacity of untreated and treated sisal fibres at constant concentration (5%) but different time (5minutes, 10minutes and 15minutes) and different gauge lengths.

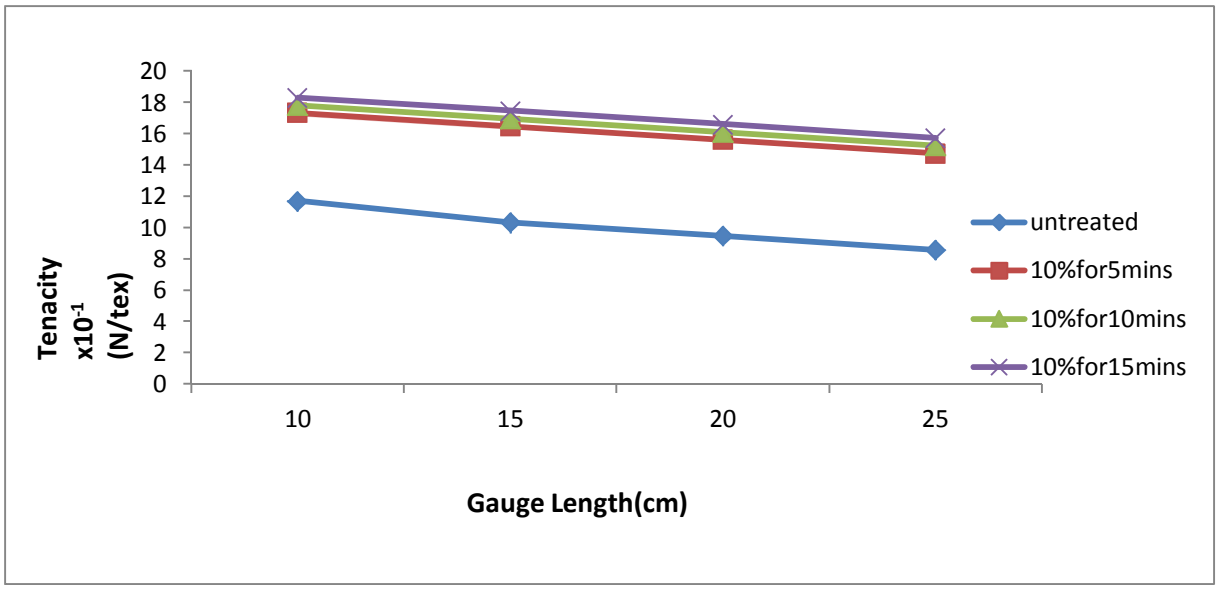


Fig 4.5 Tenacity of untreated and treated sisal fibres at constant concentration (10%) but different time (5minutes, 10minutes and 15minutes) and different gauge lengths.

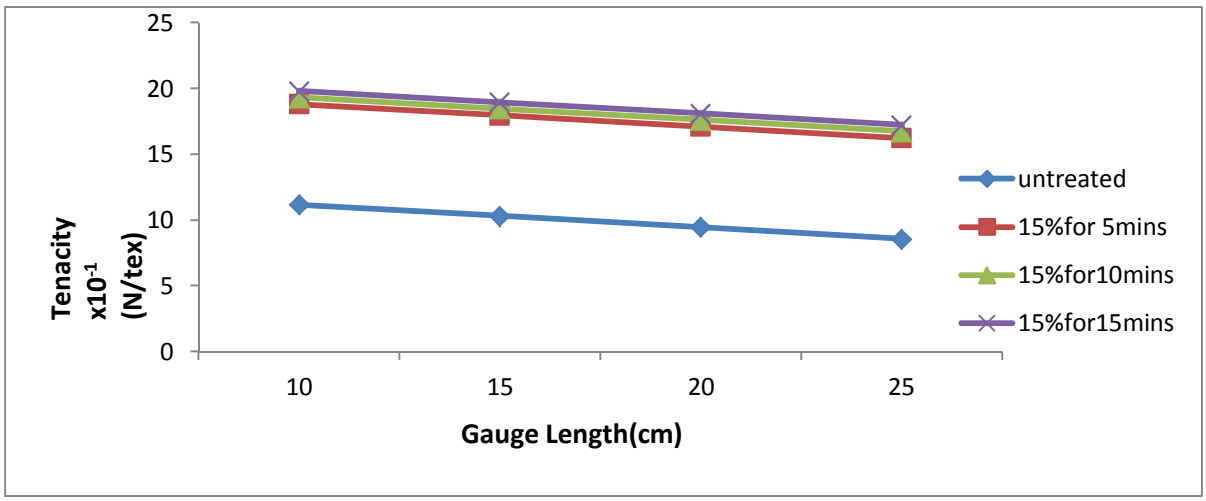


Fig 4.6 Tenacity of untreated and treated sisal fibres at constant concentration (15%) but different time (5minutes, 10minutes and 15minutes) and different gauge lengths

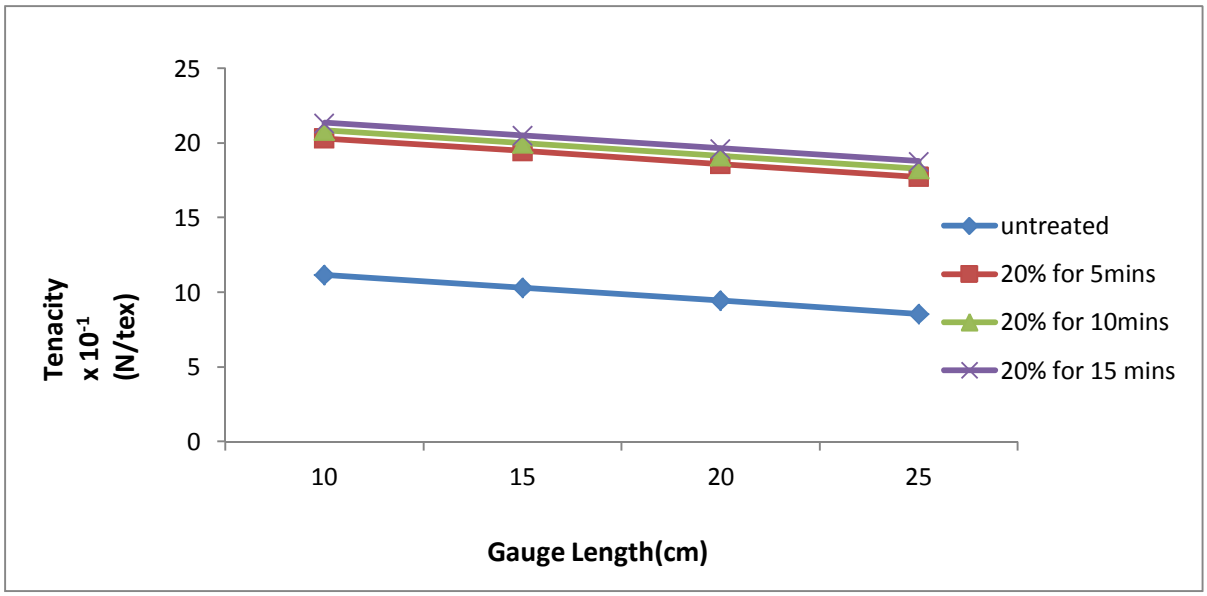


Fig 4.7 Tenacity of untreated and treated sisal fibres at constant concentration (20%) but different time (5minutes, 10minutes and 15minutes) and different gauge lengths.

Figures 4.3 to 4.7 above show the various graphs of tenacity versus gauge length at constant concentration with different time range. It can be noticed that there is a wide gap in tenacity between the untreated and treated fibres, however the differences in the time of modifications has little effect on the modified fibres at constant concentration. This is attributable to the fact that the modification was almost instantaneous at the temperature of 65°C. This is to show that increase in the time of modification does not really have a marked effect on fibre tenacity. It is also noticed that the increase in gauge length caused the tenacities to decrease. This is also due to the "weak-link effect" as mentioned earlier.

4.5.3 BREAKING EXTENSION

(Breaking extension at different concentrations, different gauge lengths, but constant time)

Figures 4.8 to 4.10 below, shows the results of the breaking extension values at different concentrations, different gauge lengths but constant time.

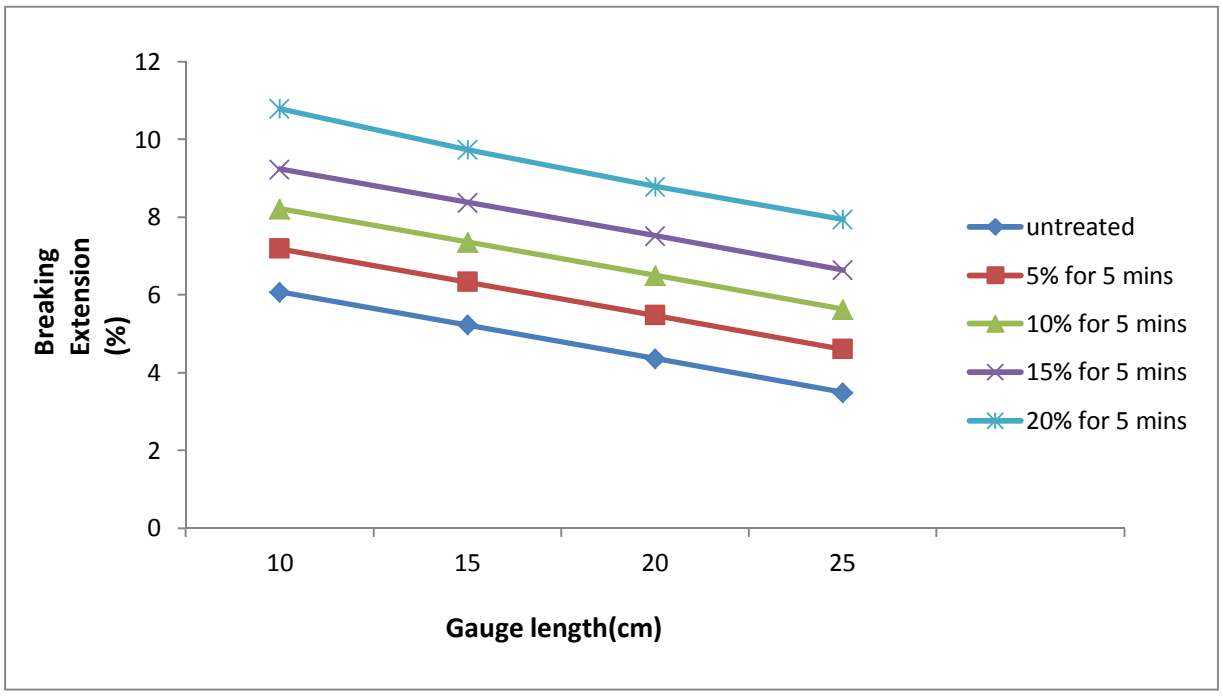


Fig 4.8 Breaking Extension of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but at constant time (5minutes) and at different gauge lengths

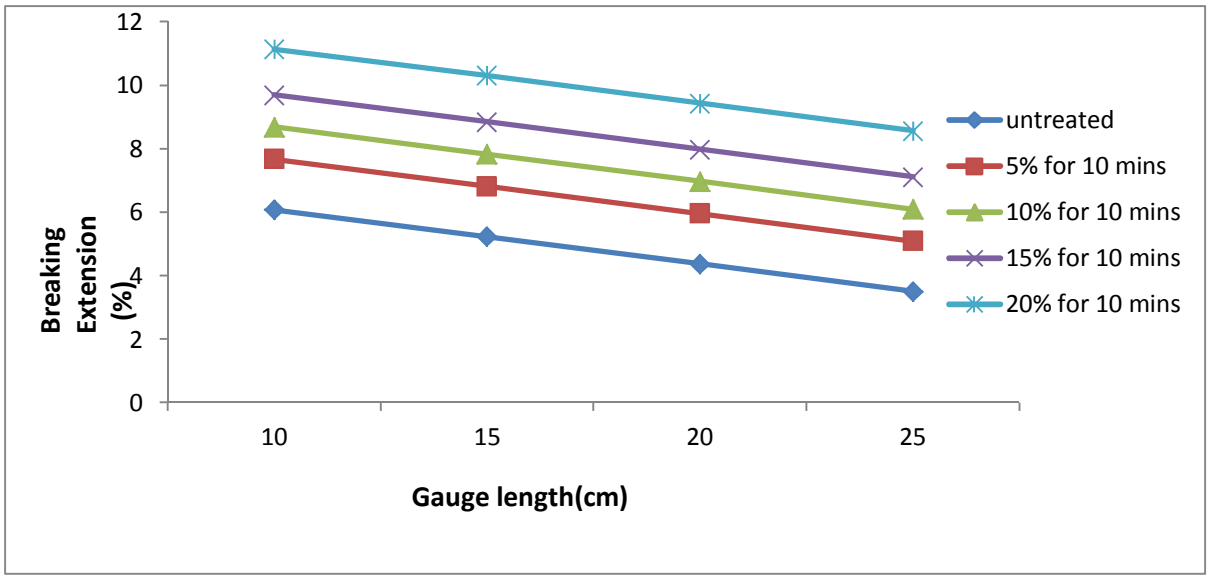


Fig 4.9 Breaking Extension of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but at constant time (10 minutes) and at different gauge lengths.

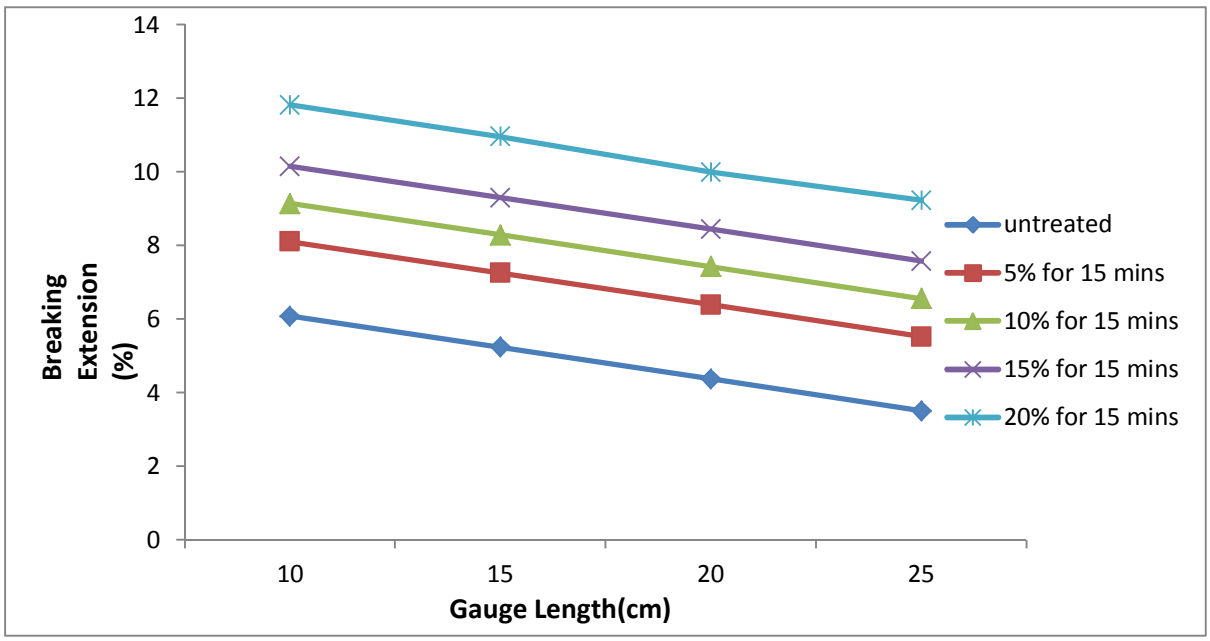


Fig 4.10 Breaking Extension of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but at constant time (15 minutes) and at different gauge lengths.

Figures 4.8 to 4.10 show that the modified samples have higher breaking extension than the untreated ones. This trend is generally expected of untreated sisal fibre because of its natural hardness and high weight. This is due to the presence of constituents such as (lignin, pectins, hemicelluloses e.t.c.) and as a result they are less extensible.

However, after modification, the sisal fibre becomes lighter due to the reduction in weight and diameter making it more extensible. This is attributable to the removal of constituents and depends on the extent of modification.Therefore increase in concentrations of NaOH, leads to increase in breaking extension of the fibres.The breaking extension decreased with increasing gauge length due to the weak-link effect.

4.5.4 BREAKING EXTENSION

(Breaking extension at constant concentration, different gauge length, and at different time)

Figures 4.11 to 4.14 below gives the result of the tenacity values at constant concentration, different gauge length but different time;

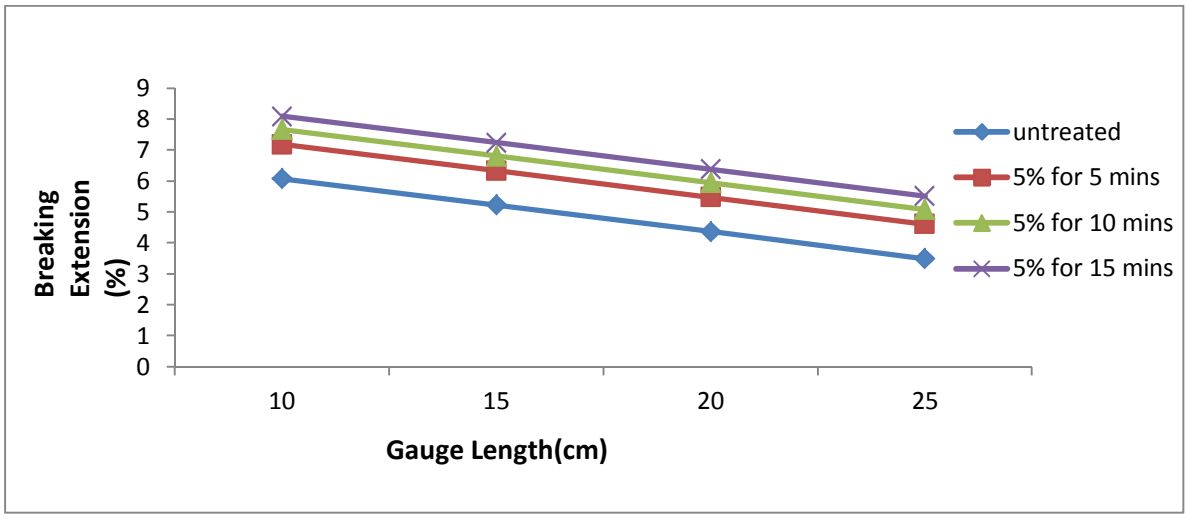


Fig 4.11 Breaking Extension of untreated and treated sisal fibres at constant concentrations (5%) but different time (5minutes, 10 minutes and 15 minutes) and at different gauge lengths.

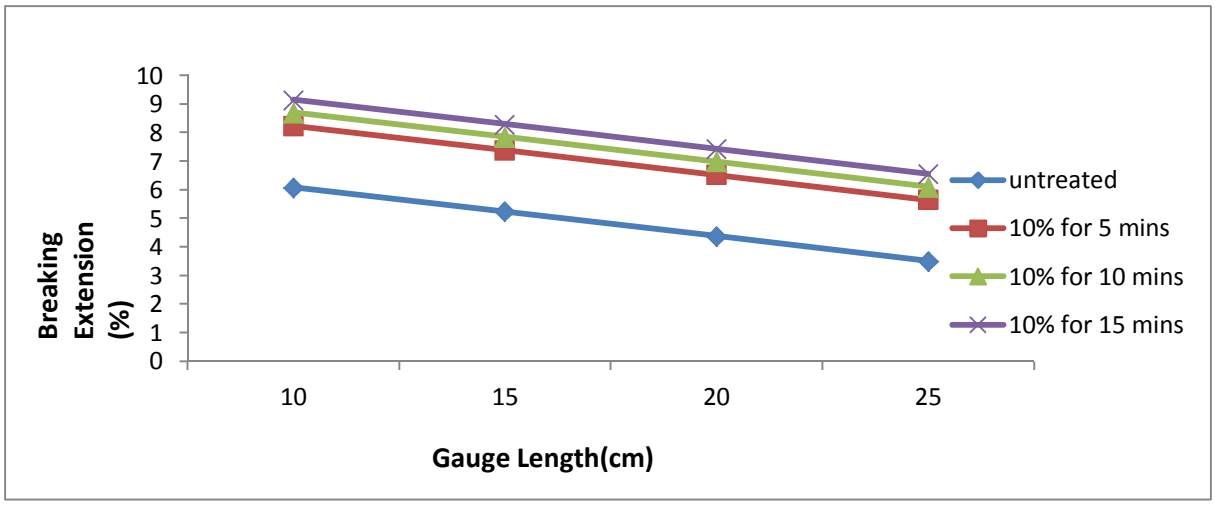


Fig 4.12 Breaking Extension of untreated and treated sisal fibres at constant concentrations (10%) but different time (5minutes, 10 minutes and 15 minutes) and at different gauge lengths.

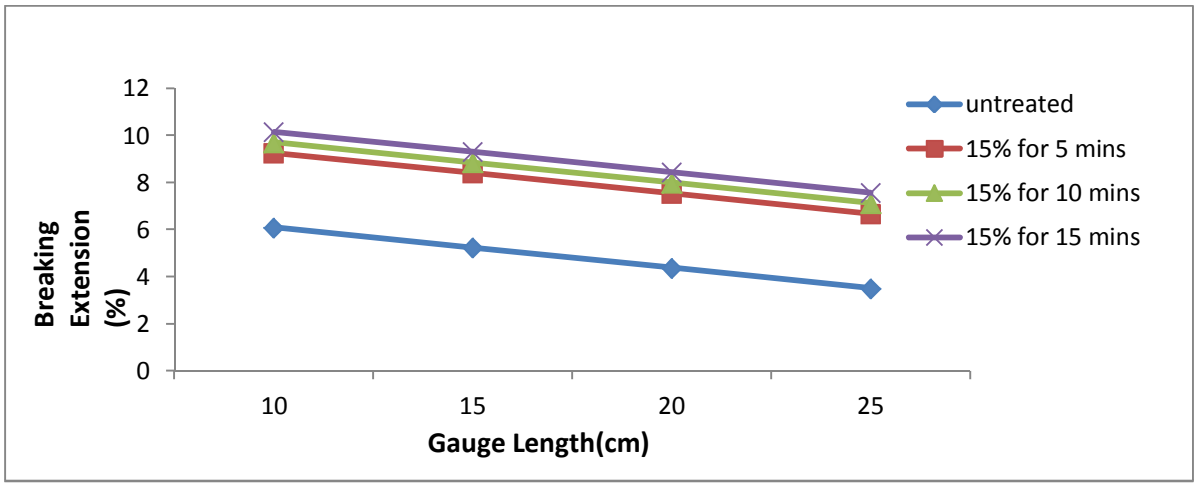


Fig 4.13 Breaking Extension of untreated and treated sisal fibres at constant concentrations (15%) but different time (5minutes, 10 minutes and 15 minutes) and at different gauge lengths.

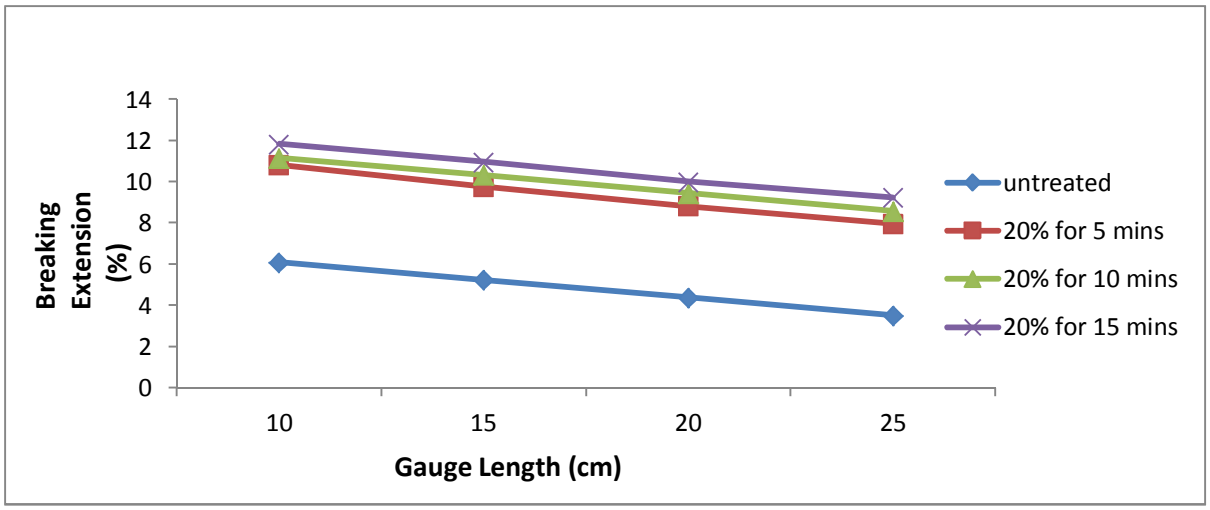


Fig 4.14 Breaking Extension of untreated and treated sisal fibres at constant concentrations (20%) but different time (5minutes, 10 minutes and 15 minutes) and at different gauge lengths.

Figures 11 to 14 above show that increase in the time of modification has little effect on the breaking extension of the sisal fibres as compared to the increase in concentration. This is also attributable to the almost instant modification at the optimum temperature of 65°C. The reduction in tenacities with increasing gauge length is also due to the weak link effect.

4.5.6 INITIAL YOUNG'S MODULUS

(Young's modulus at different concentration, different gauge length, but constant time)

Figures 4.15 to 4.17 below gives the result of the initial modulus values at different concentration, different gauge length but constant time;

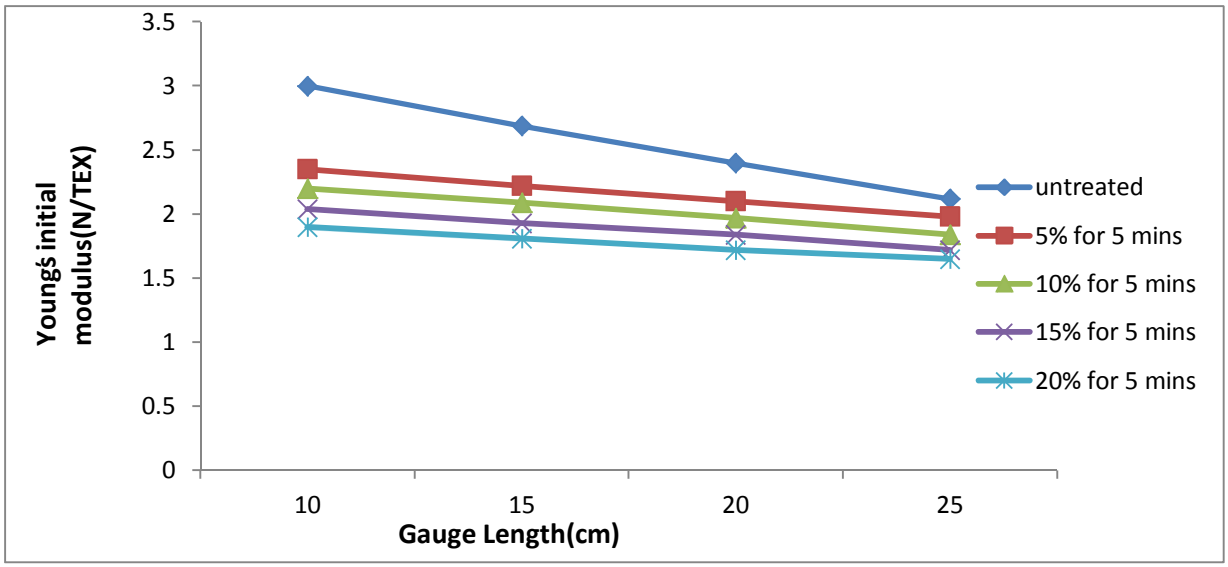


Fig 4.15 initial modulus of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (5 minutes) and at different gauge lengths.

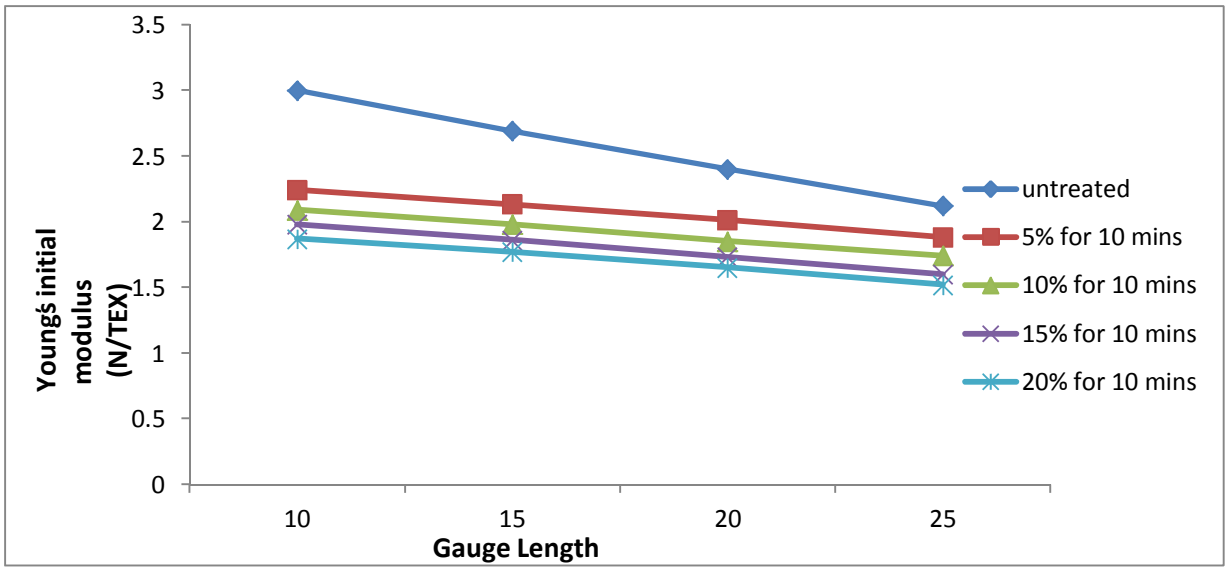


Fig 4.16 initial modulus of untreated and treated sisal fibres at different concentrations (5%, 10%, 15%and 20%) but constant time (10 minutes) and at different gauge lengths

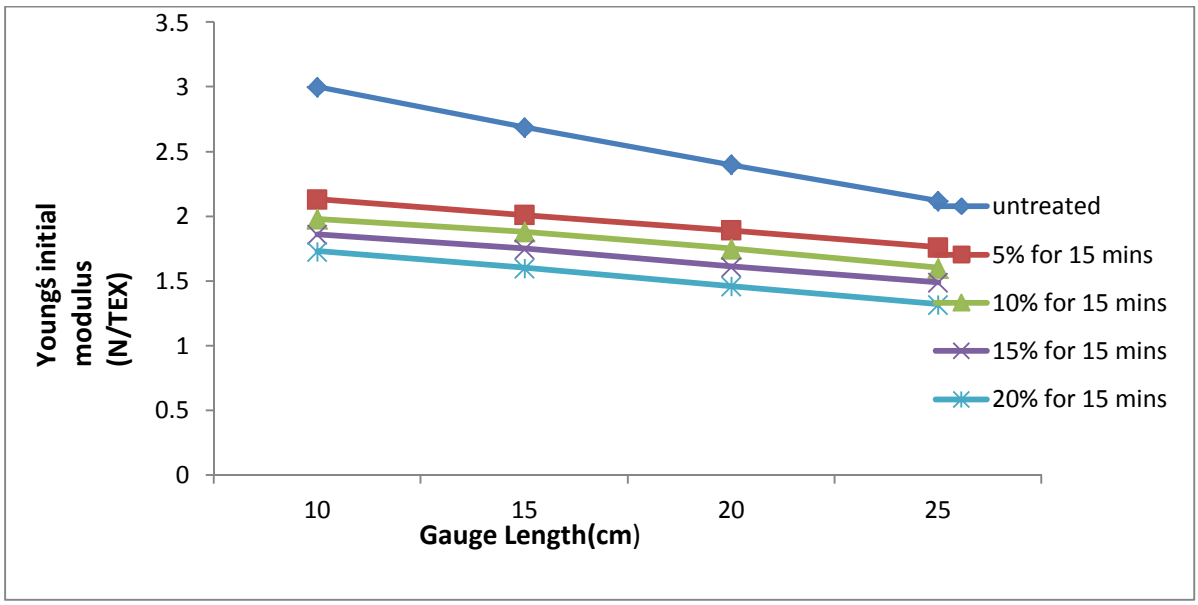


Fig 4.17 initial modulus of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (15 minutes) and at different gauge lengths

The graphs of figures 15 to 17 indicate the fact that the untreated sisal fibre has a high initial modulus compared to the treated ones.Therefore, the modified samples have low modulus as increase in concentration of NaOH reduces the modulus. A high modulus indicates inextensibility and a low modulus great extensibility. Therefore, modification increases the extensibility of the fibres, while the untreated ones are less extensible. The initial modulus for both the modified and unmodified fibres decreases with increasing gauge length also due to weak link effects.

4.5.7 INITIAL YOUNG'S MODULUS

(Initial modulus at constant concentration, different gauge length, and at different time)

Figures 4.18 to 4.21 below give the result of the initial modulus values at constant concentration, different gauge length and at different time;

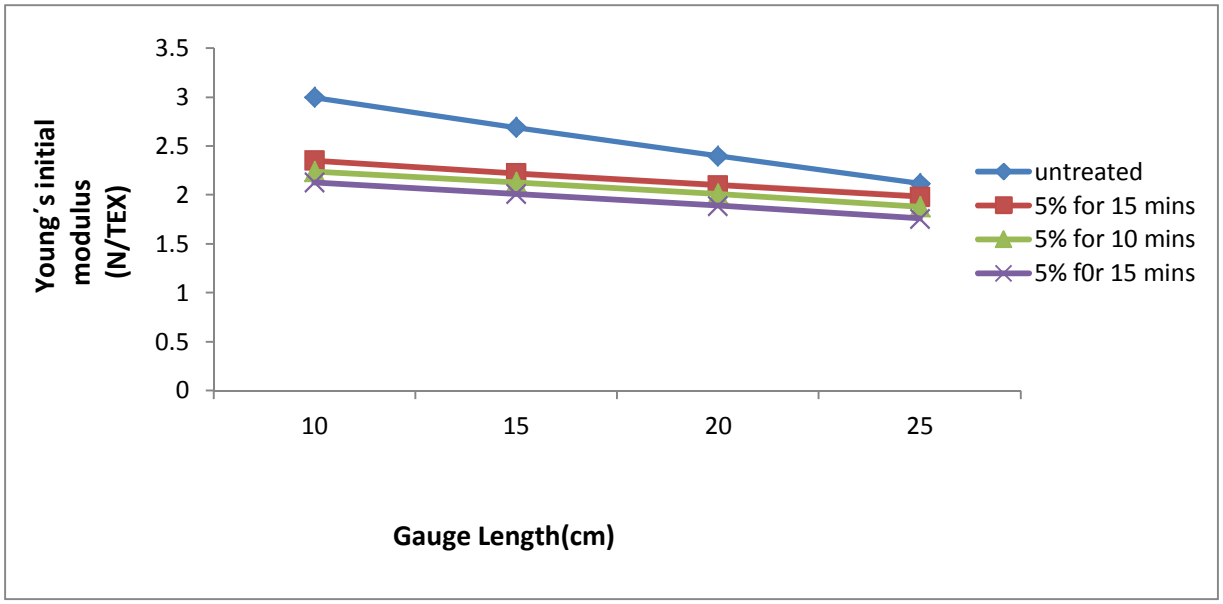


Fig 4.18 initial modulus of untreated and treated sisal fibres at constant concentrations (5%) but different time (15 minutes, 10 minutes and 15 minutes) and at different gauge lengths.

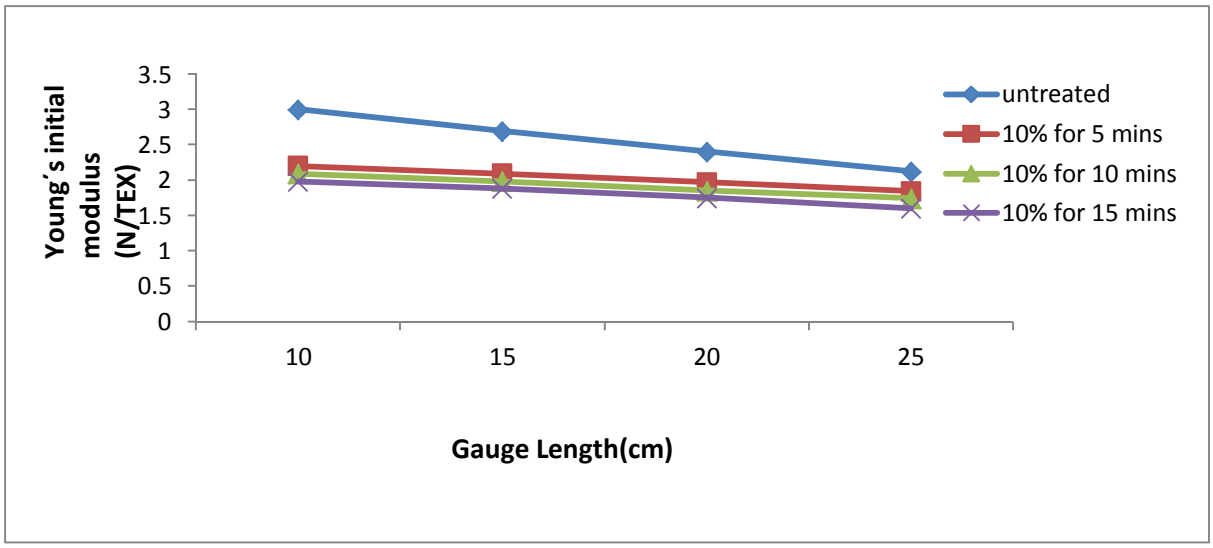


Fig 4.19 initial modulus of untreated and treated sisal fibres at constant concentrations (10%) but different time (15 minutes, 10 minutes and 15 minutes) and at different gauge lengths

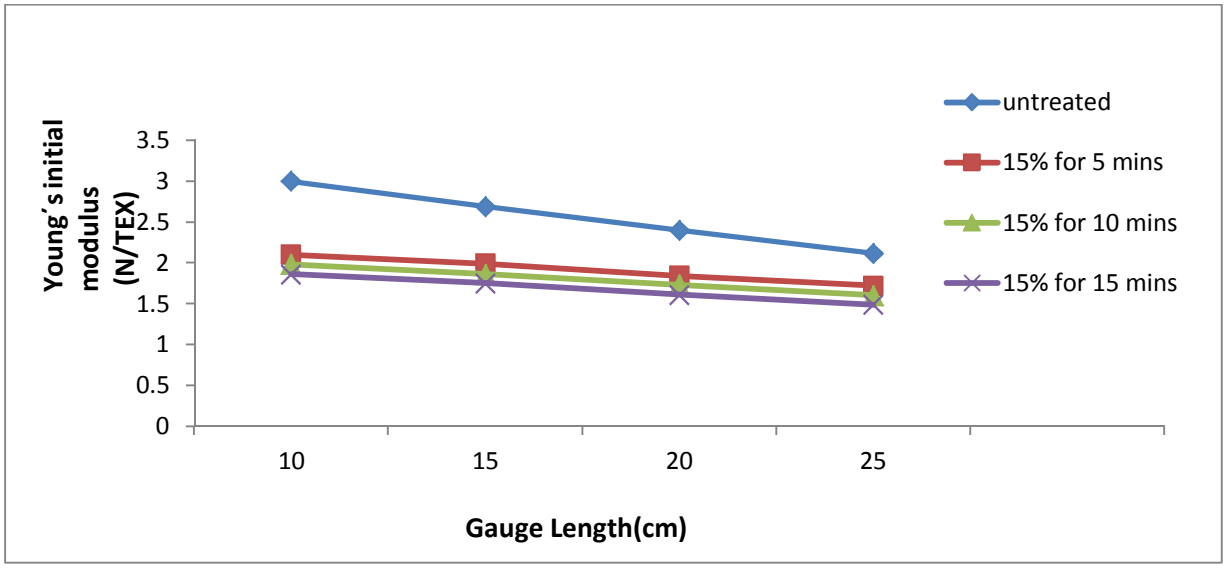


Fig 4.20 initial modulus of untreated and treated sisal fibres at constant concentrations (15%) but different time (15 minutes, 10 minutes and 15 minutes) and at different gauge lengths.

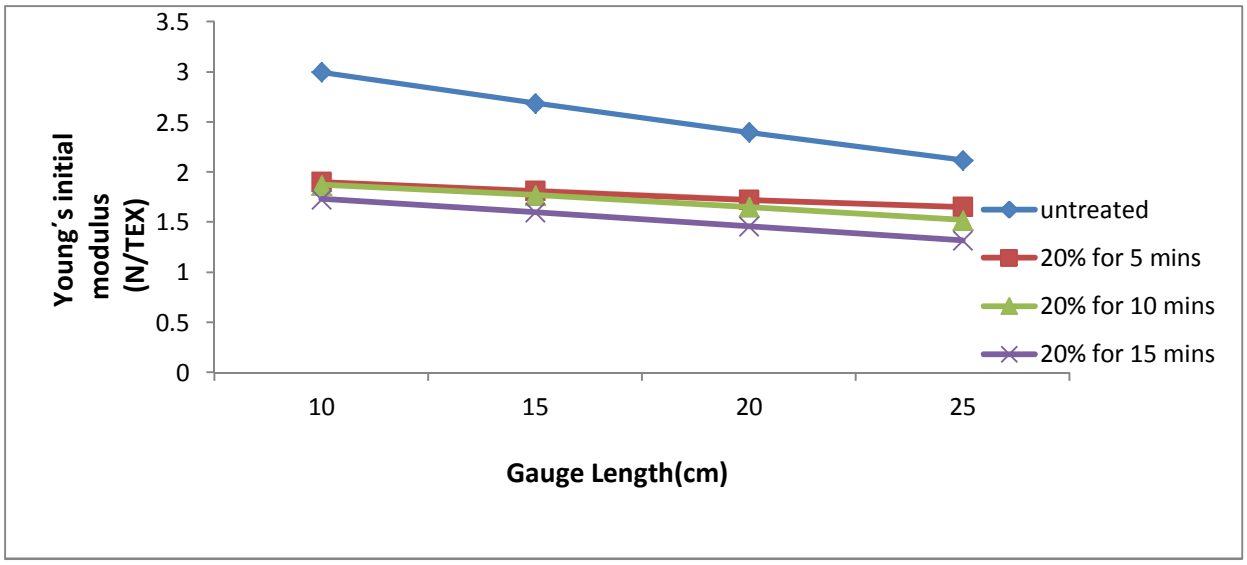


Fig 4.21 initial modulus of untreated and treated sisal fibres at constant concentrations (20%) but different time (15 minutes, 10 minutes and 15 minutes) and at different gauge lengths.

The graphs above are similar to the graphs of different concentration; however increase in time has less effect compared to that of the increase in concentration.

4.5.8 WORK OF RUPTURE

(The work of rupture at diferent concentration, different gauge length, and at constant time)

Figures 4.22 to 4.24 below gives the result of the work of rupture values at different concentration, different gauge length and at constant time.

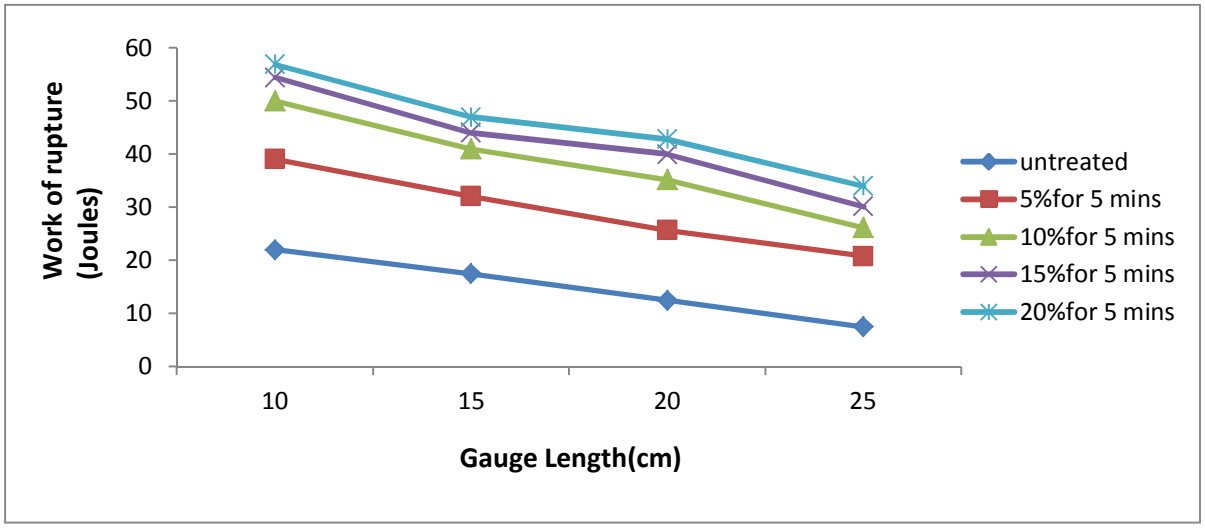


Fig 4.22 the work of rupture of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (5 minutes) and at different gauge length

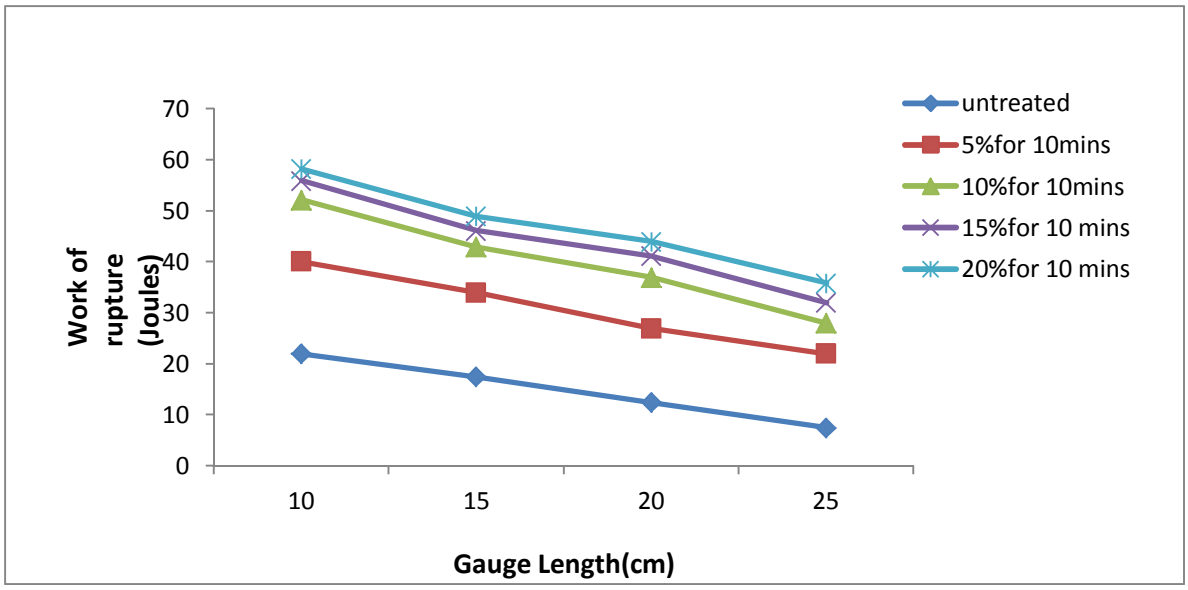


Fig 4.23 the work of rupture of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (10 minutes) and at different gauge lengths

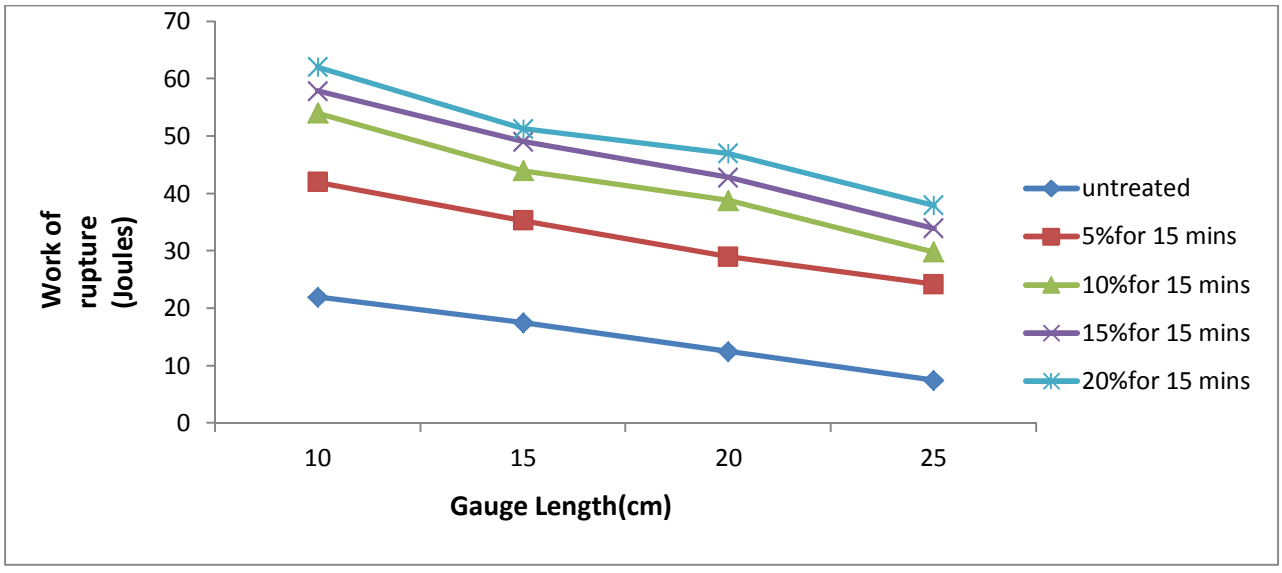


Fig 4.24 The work of rupture of untreated and treated sisal fibres at different concentrations (5%, 10%, 15% and 20%) but constant time (15 minutes) and at different gauge lengths.

Figures 4.22 to 4.24 show that the modified samples have higher work of rupture compared with the unmodified ones. The higher concentration of caustic soda, the higher the work of rupture.

Work of rupture, a measure indicating the ability to absorb work, is an important property of textile fibres. Good strength combined with elongation gives a high work of rupture, which enables the fibres to withstand sudden impacts. Thus the modified samples with the highest work of rupture are expected to absorb sudden impact more conveniently than the other fibre samples.

4.5.9 WORK OF RUPTURE

(The work of rupture at constant concentration, different gauge length, and at different time)

Figures 4.25 to 4.28 below give the result of the work of rupture values at constant concentration, different gauge length and at different time;

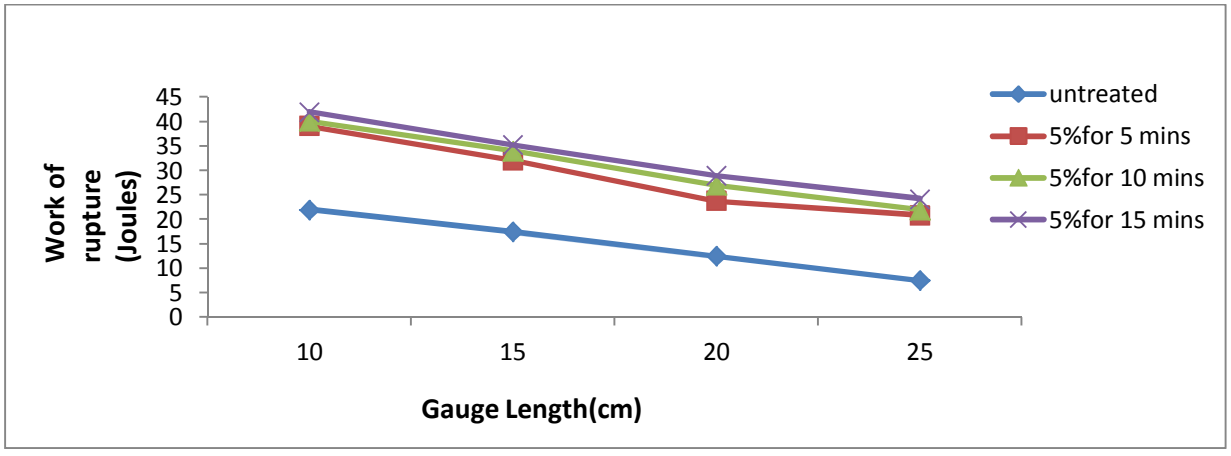


Fig 4.25 The work of rupture of untreated and treated sisal fibres at constant concentrations (5%) but different time (5minutes,10 minutes and15 minutes) and at different gauge lengths.

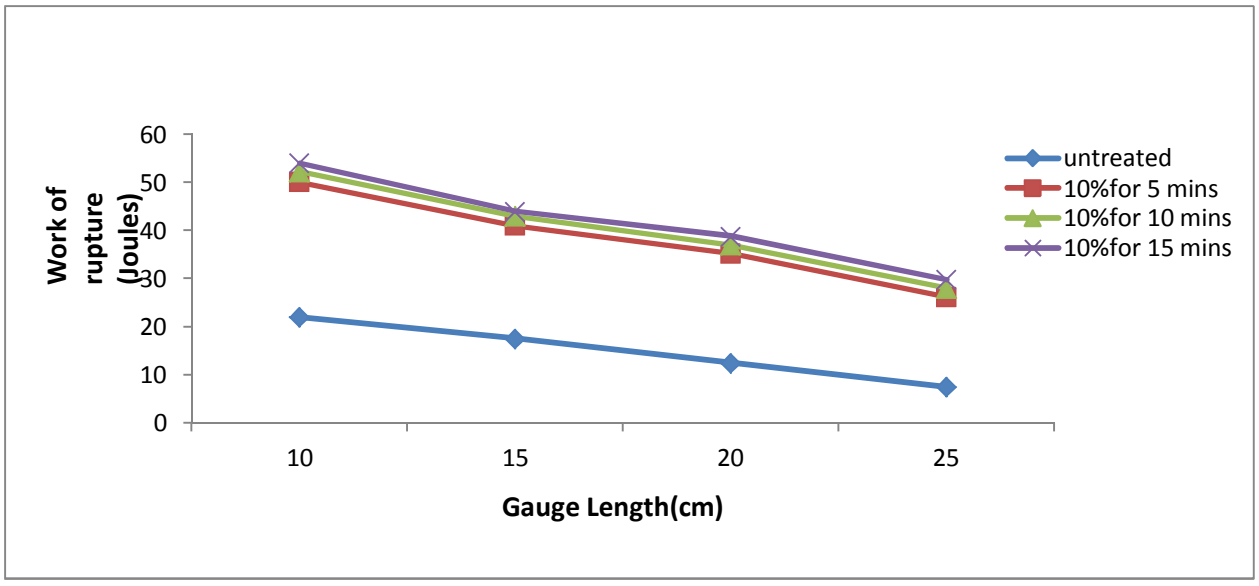


Fig 4.26 The work of rupture of untreated and treated sisal fibres at constant concentrations (10%) but different time (5minutes,10 minutes and15 minutes) and at different gauge lengths.

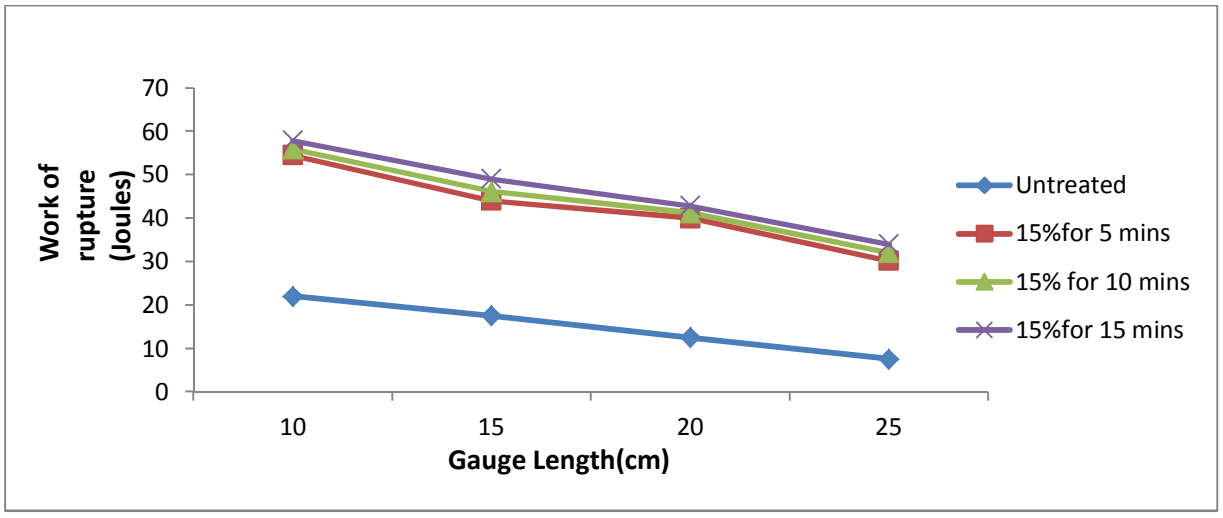


Fig 4.27 The work of rupture of untreated and treated sisal fibres at constant concentrations (15%) but different time (5minutes, 10 minutes and15 minutes) and at different gauge lengths.

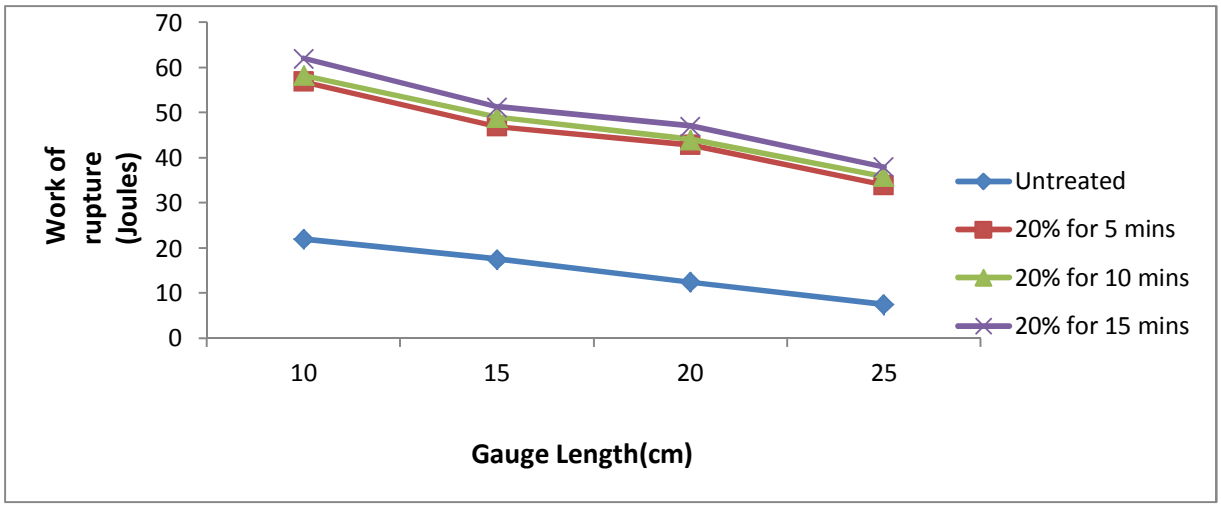


Fig 4.28 the work of rupture of untreated and treated sisal fibres at constant concentrations (20%) but different time (5minutes, 10 minutes and 15 minutes) and at different gauge lengths

The graphs are similar to the ones of increase in concentration, but increase in time has very little effects on the work of rupture during modification.

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

It was observed that the surface of the sisal fibres before modification was filled with hemicelluloses, waxes, lignin, pectin e.t.c. covered up by cementing materials. However caustic soda treatment at various percentage concentrations (5%, 10%, 15%, 20%), and at different timing (5minutes, 10minutes and 15minutes) carried out on the sisal fibres, removed the admixtures or constituents inside the fibres according to the degree of alkali modification and thereby creating a rough surface morphology as shown by the SEM Micrographs. It was also noted that increase in concentrations increases the roughness of the fibre surface and also influences the degree of swelling of the fibres and thus increasing the moisture content and regain.

The results obtained from the mechanical properties can be summarized below:

1. Increase in concentration of caustic soda, increases the tenacity, breaking extension and work of rupture of the sisal fibres.
2. Higher concentrations of caustic soda lead to low modulus and consequently higher extensibility.

However, increase in the time of modification has no marked effects on the mechanical (tensile) properties of the sisal fibres as compared to the increase in concentration of NaOH. Increase in gauge length, causes the work of rupture, tenacities, breaking extension and the young's initial modulus to decrease. This is due to "weak link effect".

It can therefore, be concluded that alkali modification has great influence on the properties of the surface characteristics, moisture regain and tensile properties of sisal fibres.

5.2 RECOMMENDATIONS

1. More useful results on the surface characteristics of sisal fibres can be obtained using the Transmission Electron Microscope (TEM) and Differential Scanning Calorimeter (DSC).

2. Another suggestion is that other chemicals, such as silanes, titanes, alkalis other than NaOH, can also be used for the modifications of sisal fibres, and the properties of treated and untreated fibres analysed accordingly.

3. Physical modification of the sisal fibres using methods such as plasma treatment can also be investigated.

4. Also, the use of the alkali modified sisal fibres in the manufacture of composites may be explored in future work.

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APPENDIX A

Data Tables

Table A1 ***(Tenacities at different concentrations, different gauge lengths, but constant time)***

5minutes treatment time

GAUGE LENGTH(CM)	**TENACITY(N/TEX) × 10^{-1}**				
	Untreated	5% Conc.	10% Conc.	15%Conc.	20%Conc.
10	11.70	15.80	17.30	18.81	20.30
15	10.32	14.95	16.45	17.96	19.45
20	9.46	14.00	15.59	17.10	18.59
25	8.56	13.20	14.72	16.23	17.72

Table A2 (*Tenacities at different concentrations, different gauge lengths, but constant time)*

10 minutes treatment time

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**				
	Untreated	5% Conc.	10%Conc.	15%Conc.	20%Conc.
10	11.70	16.30	17.80	19.32	20.85
15	10.32	15.45	16.95	18.47	20.00
20	9.46	14.59	16.09	17.61	19.14
25	8.56	13.72	15.22	16.74	18.27

Table A3 ***(Tenacities at different concentration, different gauge length, but constant time)***

15 minutes treatment time

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**				
	Untreated	5%	10%	15%	20%
10	11.70	16.80	18.30	19.81	21.34
15	10.32	15.95	17.45	18.96	20.50
20	9.46	15.09	16.59	18.10	19.64
25	8.56	14.22	15.72	17.23	18.77

Table A4 ***(Tenacities at different time, different gauge length, but constant concentration)***

5% concentration treatment

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**			
	Untreated	5 minutes	10 minutes	15 minutes
10	11.17	15.80	16.30	16.80
15	10.32	14.95	15.45	15.95
20	9.46	14.00	14.59	15.09
25	8.56	13.20	13.72	14.22

Table A5 ***(Tenacities at different time, different gauge length, but constant concentration)***
10% concentration treatment

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**			
	Untreated	5minutes	10 minutes	15 minutes
10	11.17	17.30	17.80	18.30
15	10.32	16.45	16.95	17.45
20	9.46	15.59	16.09	16.59
25	8.56	14.72	15.22	15.72

Table A6 ***(Tenacities at different time, different gauge length, but constant concentration)***

15% concentration treatment

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**			
	Untreated	5 minutes	10minutes	15 minutes
10	11.17	18.81	19.32	19.81
15	10.32	17.96	18.47	18.96
20	9.46	17.10	17.61	18.10
25	8.56	16.23	16.74	17.23

Table A7 ***(Tenacities at different time, different gauge length, but constant concentration)***

20% concentration treatment

GAUGE LENGTH(cm)	**TENACITY(N/TEX) × 10^{-1}**			
	Untreated	5 minutes	10 minutes	15 minutes
10	11.17	20.30	20.85	21.35
15	10.32	19.45	20.00	20.50
20	9.46	18.59	19.14	19.64
25	8.56	17.72	18.27	18.77

Table A8 ***(Breaking extension at different concentrations, different gauge lengths, but constant time)***

5 minutes treatment time

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	6.08	7.19	8.22	9.24	10.80
15	5.23	6.34	7.37	8.39	9.75
20	4.37	5.48	6.51	7.53	8.80
25	3.50	4.61	5.64	6.65	7.95

Table A9 ***(Breaking extension at different concentrations, different gauge lengths, but constant time)***

10 minutes treatment

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	6.08	7.67	8.69	9.70	11.15
15	7.25	6.82	7.84	8.85	10.32
20	6.39	5.96	6.98	7.99	9.44
25	5.52	5.09	6.10	7.12	8.57

Table A10 ***(Breaking extension at different concentrations, different gauge lengths, but constant time)***

15 minutes treatment

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	6.08	8.10	9.14	10.15	11.82
15	5.23	7.25	8.29	9.30	10.96
20	4.37	6.39	7.43	8.44	9.99
25	3.50	5.52	6.56	7.57	9.23

Table A11 ***(Breaking extension at constant concentration, different gauge length, and different time)***

5% treatment

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**			
	Untreated	5 minutes	10 minutes	15minutes
10	6.08	7.19	7.67	8.10
15	5.23	6.34	6.82	7.25
20	4.37	5.48	5.96	6.39
25	3.5	4.61	5.09	5.52

Table A12 ***(Breaking extension at constant concentration, different gauge length, and different time)***
10% treatment

GUAGE LENGTH(cm)	**BREAKING EXTENSION (%)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	6.08	8.22	8.69	9.14
15	5.23	7.37	7.84	8.29
20	4.37	6.51	6.98	7.43
25	3.5	5.64	6.10	6.56

Table A13 ***(Breaking extension at constant concentration, different gauge length, and different time)***
15% treatment

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	6.08	9.24	9.70	10.15
15	5.23	8.39	8.85	9.30
20	4.37	7.53	7.99	8.44
25	3.5	6.65	7.12	7.57

Table A14 ***(Breaking extension at constant concentration, different gauge length, and different time)***
20% treatment

GAUGE LENGTH(cm)	**BREAKING EXTENSION (%)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	6.08	10.80	11.15	11.82
15	5.23	9.75	10.32	10.96
20	4.37	8.80	9.44	9.99
25	3.50	7.95	8.57	9.23

Table A15 ***(Young's modulus at different concentration, different gauge length, but constant time)***

5 minutes treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20%Con
10	3.00	2.35	2.20	2.04	1.90
15	2.69	2.22	2.09	1.93	1.81
20	2.40	2.10	1.97	1.84	1.72
25	2.12	1.98	1.84	1.72	1.65

Table A16 ***(Initial modulus at different concentration, different gauge length, but constant time)***

10 minutes treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	3.00	2.24	2.09	1.98	1.87
15	2.69	2.13	1.98	1.86	1.77
20	2.40	2.01	1.85	1.73	1.65
25	2.12	1.88	1.74	1.60	1.52

Table A17 ***(Initial modulus at different concentration, different gauge length, but constant time)***

15 minutes treatment

GAUGE LENGTH	**INITIAL MODULUS(N/TEX)**				
	Untreated	5% Conc.	10% C0nc.	15% Conc.	20% Conc.
10	3.00	2.13	1.98	1.86	1.73
15	2.69	2.01	1.88	1.75	1.60
20	2.40	1.89	1.75	1.61	1.46
25	2.12	1.76	1.60	1.49	1.32

Table A18 ***(Initial modulus at constant concentration, different gauge length, and different time)***
5% treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	3.00	2.35	2.24	2.13
15	2.69	2.22	2.13	2.01
20	2.40	2.10	2.01	1.89
25	2.12	1.98	1.88	1.76

Table A19 ***(Initial modulus at constant concentration, different gauge length, and different time)***

10% treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	3.00	2.20	2.09	1.98
15	2.69	2.09	1.98	1.88
20	2.40	1.97	1.85	1.75
25	2.12	1.84	1.74	1.60

Table A20 (*Initial modulus at constant concentration, different gauge length, and different time)*

15% treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	3.00	2.04	1.98	1.86
15	2.69	1.93	1.86	1.75
20	2.40	1.84	1.73	1.61
25	2.12	1.72	1.60	1.49

Table A21 ***(Initial modulus at constant concentration, different gauge length, and different time)***
20% treatment

GAUGE LENGTH(cm)	**INITIAL MODULUS (N/TEX)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	3.00	1.90	1.87	1.73
15	2.69	1.81	1.77	1.60
20	2.40	1.72	1.65	1.46
25	2.12	1.65	1.52	1.32

Table A22 ***(The work of rupture at different concentration, different gauge length, and constant time)***
5 minutes treatment

GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	22.00	39.07	50.01	54.46	56.90
15	17.50	32.08	41.01	44.00	46.94
20	12.50	25.72	35.20	39.98	42.82
25	7.50	20.80	26.20	30.15	34.00

Table A23 ***(The work of rupture at different concentration, different gauge length, and constant time)***
10 minutes treatment

GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	22.00	40.09	52.20	55.98	58.29
15	17.50	34.01	42.98	46.14	49.01
20	12.50	26.99	37.00	41.20	44.04
25	7.50	22.01	28.04	32.06	35.90

Table A24 ***(The work of rupture at different concentration, different gauge length, and constant time)***
15 minutes treatment

Gauge length(cm)	**WORK OF RUPTURE (JOULES)**				
	Untreated	5% Conc.	10% Conc.	15% Conc.	20% Conc.
10	22.00	42.00	54.04	57.86	62.09
15	17.50	35.29	44.01	49.06	51.29
20	12.50	29.00	38.80	42.85	47.01
25	7.50	24.22	29.84	33.99	38.00

Table A25 ***(The work of rupture at constant concentration, different gauge length, and different time)***
5% treatment

GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	22.00	39.07	40.09	42.00
15	17.50	32.08	34.01	35.29
20	12.50	25.72	26.90	29.00
25	7.50	20.80	22.01	24.22

Table A26 ***(The work of rupture at constant concentration, different gauge length, and different time)***
10% treatment

GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	22.00	50.01	52.20	54.04
15	17.50	41.01	42.98	44.01
20	12.50	35.20	37.00	38.80
25	7.50	26.20	28.04	29.84

Table A27 ***(The work of rupture at constant concentration, different gauge length, and different time)***

15% treatment

01GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	22.00	54.46	55.98	57.86
15	17.50	44.00	46.14	49.06
20	12.50	39.98	41.20	42.85
25	7.50	30.15	32.06	33.99

Table A28 ***(The work of rupture at constant concentration, different gauge length, and different time)***
20% treatment

GAUGE LENGTH(cm)	**WORK OF RUPTURE (JOULES)**			
	Untreated	5 minutes	10 minutes	15 minutes
10	22.00	56.90	58.29	62.09
15	17.50	46.94	49.01	51.29
20	12.50	42.84	44.04	47.01
25	7.50	34.00	35.90	38.00