ADDIS ABABA UNIVERISITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

SCHOOL OF GRAGUATE STUDIES

INVESTIGATION THE PHYSICAL PROPERTIES AND OPTIMIZATION THE PROCESS PARAMETERS OF THE BIO COMPOSITE SYNTHESIZED FROM RECYCLED POLYETHYLENE TERE PHATALATE (PET) AND AGAVE SISALINA (SISAL) PLANT FIBER FOR HOUSE CELLING APPLICATIONS.

BY

DANIEL TASEW

January 2020

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Investigation the physical properties and optimization the process parameters of the bio composite synthesized from recycled polyethylene tere phatalate (pet) and agave sisalina (sisal) plant fiber for house celling applications

A Thesis Submitted to Graduate School of Addis Ababa University in Partial Fulfillment of the Requirement of the Degree of Master of Science in Chemical Engineering (Process Stream)

By

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Advisor: Dr. Beteley Tekola

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School, chairperson name

DECLARATION

I here declare that the research entitled by investigation the physical properties and optimization the process parameters of the bio composite synthesized from recycled Polyethylene Tere Phatalate (pet) and Agave Sisalina (sisal) plant fiber for house celling applications is my own independent work and has no previously been submitted to any other universities in order to obtain degree.

Signature ----------

Date ----------

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ACRONYMS

PMC	Polymers-matrix composites
PET	Polyethylene Tere Phatalate
CCIID	Chemical and construction industry development institute
MMC	Metal matrix composites
CMC	Ceramics matrix composites
PMC	Polymer matrix composites
FRP	Fiber reinforced polymer
PRP	Particle reinforced polymer
CM	Centimeter
mm	Millimeter
m	Meter
MPa	Mega Pascal
Gpa	Gigaa Pascal;
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
DMT	Dimethyl terephthalate
Tg	Glass transition temperature
dℓ/g	Deciliters per gram
IV	Intrinsic viscosity
CHDM	Cyclohexane di methanol
PET-G	Polyethylene terephthalate glycol-modified

Abstract

Sisal natural fiber - recycled pet bio composite is a composite material produced from sisal fiber and polyethylene Tere Phthalate (PET) injection pressing and by using hot pressing. The recycled pet has been utilized as the matrix and the natural sisal fiber acts as reinforcement in the course of the production of the composite material. The composite material has been made for the house ceiling application.

In the investigation the physical properties and optimization the process parameters of the bio composite synthesized from recycled Polyethylene Tere Phatalate (pet) and Agave Sisalina (sisal) plant fiber for house celling applications, the ratio of the fiber and the pet were15/85, 25/75and 35/65 and Press times for all sample taken 10 minutes. The sisal fiber treated with 10, 15, 20 % alkali solution with treatment time 24, 48 and 72 hours. The effects of treatment time, sisal to pet ratio and concentration of alkali on internal bonding and flexural bending were investigated by using a full factorial design using Design Expert 7.0.0 and determined the optimal value.

Characterization of the physiochemical properties of the bio composite was analyzed and the results obtained from experiments that internal bonding 1.36N/mm^{2,} flexural strength 520 N/mm^2 and the fiber has been characterized as 0.75% cuticle, 8% dry matter, 13.1 % moisture content, density of the fiber 1.33g/m^3, tensile strength 600-700 Mpa, yongus modulus 10.40 GPa, elongation of 2-3 % and 0.9 % ash content.

The obtained products were tested for their physical and mechanical properties such as density, internal bonding (IB) according to German DIN 52352 Standard and flexural bending test. The results indicated that all properties of test were coinciding with wood related particleboards.

The optimal value of the internal bonding strength and flexural bending strength of the composite at sisal to pet composition ratio of 25/75 in %, treatment time at 72hr , alkali concentration 20% and plate temperature of 246 ^{o} C are 1.36 N/mm^2 and 520 N/mm^2 respectively.

Key words: Sisal fiber, Polyethylene TerePhatalate, Internal Bonding Strength, Flexural strength,

CHAPTER ONE

1. INTRODUCTION

1.1. BACK GROUND

The global market pressure requires faster product development and reduces time to market (Patel, B.C, 2012). Customer driven product customization and continuous demands for cost savings are forcing companies to look for new technologies and processes that can cope with high volume production in a quicker and cheaper manner (Ramakrishna Malkapuram, 2009).

Materials containing fibers or particle reinforcement belong to the class of materials known as composites. Many of our modern technologies require materials with unusual combination of properties (Omar Faruk; Andrzej, 2012). This is especially true for materials that are needed for aerospace, underwater, transportation applications. Air carafe engineers are increasingly searching for structural materials that have low density, high strength, stiffness, high abrasion and corrosion resistant. Material property combinations and ranges have been and are being extended by the development of composite materials (Salma Siddika; 2013).

Generally, composites are produced when two or more materials are joined together to give combination of properties that can not be attained otherwise (Han-Seung Yang, 2004**)**. According to the principles of combined action, better property combinations are fashioned by the combination of two or more distinct materials.

Polymers-matrix composites (PMC) consist of polymer resins (high molecular weight reinforcing plastics) as the matrix with particulate reinforcement medium (Netra**,** 2012**).** The functions of the matrix are to transfer stress between the reinforcing fiber and particulate, to act as a glue to hold the fiber together, and to protect it from mechanical and environmental damages (Omar Faruk; Andrzej, 2012).

Particulate composites have distinct properties like improved strength and contain large amount of coarse particles that do not block slip effectively (Nadir, Ayrilmis, 2011).

Mechanical properties of polymers can be controlled by the incorporation of particles in the polymer matrix. In view of this back ground, this study aimed to evaluating the mechanical

properties of sisal fiber particulate reinforcement in polymer polyethylene TheraPhatalate(pet) matrix composites in order to develop material for house celling application.

1.2. STATEMENT OF PROBLEM

Ethiopia is country with an increase of plastic packaging industries. One of the packaging materials that can mostly be used is Polyethylene Thera Phatalate (pet) which is mostly used for packing portable water, soft drinks, and juices and so on.

According to chemical and construction industry development institute (CCIIDI), 8,457,340.48 killo grams of pet resins have been used and transformed into pet bottle annually and after usage this amount of pet bottle waste was disposed to our environment. Even if there are certain companies that collect theses wastes, crashed in to chip form and export it to some other countries, the amount of uncollected pet bottle waste is still paramount.

Moreover, some countries' government including the China government is banning any recycled materials from entering to their countries so that the companies that have been exporting so far are facing new challenges. Thus, in order to overcome these challenges, technologies that transform these pet recycled to some other valuable product must be considered. One of the possible technologies that can give a relief is that manufacturing of composite materials from the recycled materials and natural fibers.

1.3. OBJECTIVES

1.3.1. GENERAL OBJECTIVES

The main objective of this study to investigate the physical properties and optimize process parameters of the bio composite synthesized from recycled Polyethylene Tere phatalate (pet) and Agave Sisalina (Sisal) plant fiber for house celling applications.

1.3.2. SPECIFIC OBJECTIVES

- To prepare bio composite materials from pet recycled and sisal fiber.
- To investigate the optimal condition for the composite with desirable quality.

- To determine the mechanical properties of the bio composites made from recycled pet and sisal fiber.
- To characterize the bio composite material made.

1.4. SIGNIFICANCE OF THE STUDY

The significance of investigation the physical properties and optimizing process parameters of the bio composite synthesized from recycled pet and sisal fiber can be seen in different perspectives:-

- Provide the identification of best production method for the production of bio composite from recycled pet and sisal fiber.
- Provide economic feasible option to produce the composite material locally.
- To minimize environmental pollution due to the damping of pet
- From the output of the research, investors, farmers, researchers and high learning institutions will be beneficiaries.

CHAPTER TWO

2. Literature review

2.1. Introduction

2.1.1. Composite materials

A composite is a combination of two or more materials in which one of the materials, called the reinforcing phase, is in the form of fiber, sheets, or particles, and is embodied in the other materials called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Composites typically have a fiber or particle phase that is stiffer and stronger than the continuous matrix phase and serve as the principal load carrying members. The matrix acts as a load transfer medium between fibers, and in less ideal cases where the loads are complex, the matrix may even have to bear loads transverse to the fiber axis. The matrix also serves to protect the fibers from environmental damage before, during and after composite processing. When designed properly, the new combined material exhibits better strength than would each individual material. Composites are used not only for their structural properties, but also for electrical, thermal and environmental application (Patel, 2012).

Composites are multi-functional material systems that provide characteristics not obtainable from any discrete materials. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics (Jartiz, 1965).

The composite should not be regarded simple as combination of two materials. In the broader significance, the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them, (Kelly, 1967).

The composites are compounded materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short coming in order to obtain improved materials (Beghezan, 1966).

Composite materials as heterogeneous materials consisting of two or more solid phases having intimate contact with each other on a microscopic scale (Han-Seung Yang, 2004).

The following are some of the reason why composites are selected for certain applications.

- High strength to weigh ratio (low density, high tensile strength)
- High creep resistance
- High tensile strength at elevated temperatures
- High toughness

2.2. CLASSIFICATION OF COMPOSITES

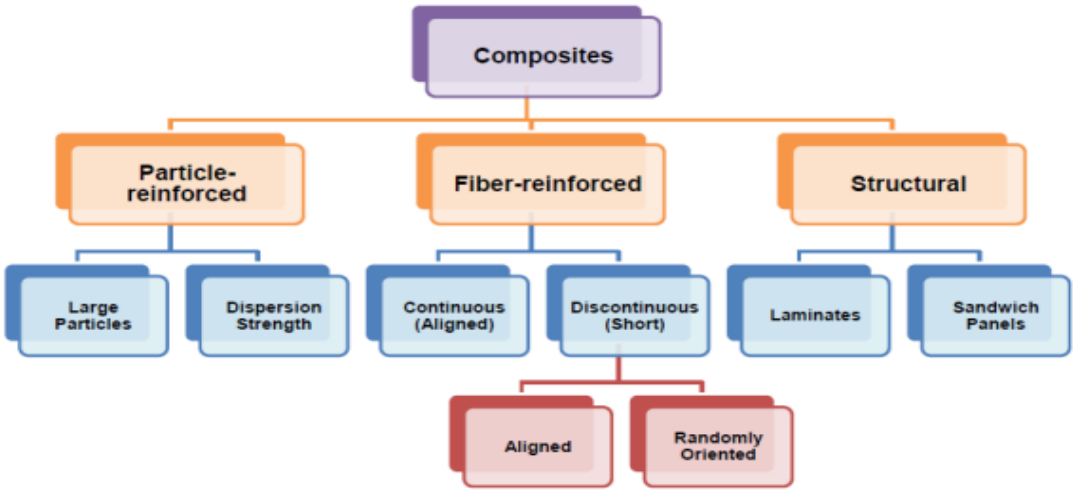


Fig 2.1. Composite materials classifications

2.2.1. Fibrous composites

A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimension of the reinforcement determines its capability of contributing its properties to the composites. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might otherwise lead to failure, particularly with brittle matrices (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3).

Man-made filaments or fibers of non – polymeric materials exhibits much higher strength along their length since large flaws, which may be present in the bulk material, are minimized because of the small cross sectional dimensions of the fiber. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3).

2.2.2. Particulate composites

In particulate composites the reinforcement is of particle nature. It may be spherical, cubic, tetragonal, a plate or of other regular or irregular shape. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composites to limited extent. Particles fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities , improve performance at elevated temperatures, reduce friction ,increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage. (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3)

According to the type of matrix material, composites are classified as:-

- Metal matrix composites(MMC)
- Ceramics matrix composites(CMC)
- Polymer matrix composites(PMC)

2.2.3. Metal matrix composites

Higher strength**,** fracture toughness and stiffness are offered by metal matrix composites. Metal matrix compositescan with stand elevated temperature in corrosive environment than polymer composites. Titanium, aluminum and magnesium are the popular metalused in composite that are particularly usefully for aircraft applications. Because of these attributes metal matrix composites are under consideration for wide range of application (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3).

2.2.4. Ceramic matrix composites

One of the main objectives in producing ceramic matrix composites is to increase the toughness. Naturally it is hoped and indeed often found that there is a concomitant improvement in strength and stiffness of ceramics matrix composites (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3).

2.2.5. Polymer matrix composites

Most commonly used matrix materials are polymeric. In general the mechanical properties of polymers are inadequate for many structural purposes.In particular their strength and stiffness are low compared to metal and ceramics. These difficulties are overcome by reinforcing other materials with polymers. Secondly, the processing of polymer matrix composites need not involve high pressure and does not require high temperature. Also equipment required for manufacturing polymers matrix composites are simpler (Omar Faruk, 2012)

For this reason, polymer matrix composites developed rapidly and soon became popular for structural applications. Two types of polymers composites are:-

A/fiber reinforced polymer (FRP)

B/particle reinforced polymer (PRP)

2.2.5.1. Fiber reinforced polymer (FRP)

These are composites composed of fibers and a matrix. Fibers are the reinforcement and the main source of strength while the matrix glues all the fibers together in shape and transfers stresses between the reinforcing fibers. Sometimes, fillers might be added to smooth the manufacturing process, impact special properties to the composites, and/or reduce the product cost (Polymer matrix composites Materials usage, design, and analysis, 1997 Volume 3).

2.2.5.2. Particle reinforced polymer (PRP)

These are composites in which particles are used for reinforcing include ceramics and glasses such as small mineral particles, metal particles such as aluminum and amorphous materials,

including polymers and carbon black. Particles are used to increase the modules of the matrix and to decrease the ductility of the matrix (Brandrup, 1999).

2.2.6. Hybrid composites

Hybrid composites are more advanced composites as compared to conventional FRP composites. Hybrids can have more than reinforcing phase and a single matrix phase or single reinforcing phase with multiple matrix phases or multiple reinforcing and multiple matrix phases. They have better flexibility as compared to other fiber reinforced composites. Normally, it contains high modules fiber with low modulus fiber. The high modulus fiber provides the stiffness and load bearing qualities, whereas the low-modulus fiber makes the composites more damage tolerant and keeps the material cost low. The mechanical properties of hybrid composites can be varied by changing volume ratio and stacking sequence of different plies (Jevanthi, S and Janci Rani, J 2011).

2.3. Natural fiber reinforced composites

The interest in natural fiber-reinforced polymer composites materials is rapidly growing both in terms of their industrial and fundamental research. They are renewable, cheap, completely or partially recyclable, and bio degradable. Plants such as flax, cotton, hemp, jute, sisal, kenaf, pipaple, bamboo as well as wood ,used from time to immemorial as a source of liginocellulose fibers, are more and more often applied as the reinforcement of composites. Their availability, renewability, low density, and price as well as satisfactory mechanical properties make them attractive ecological alternatives to glass, carbon and man-made fibers used for the manufacturing of composites. The natural fiber containing composites are more environmentally friendly, and are used in transportation (automobiles, railway coaches, aerospace), military application, building and construction industries (ceiling partition board), packaging ete (Chandramohan, D and Marimuthu, K, 2011).

2.4. Natural fibers

There are two kinds of fibers available i.e. natural fiber and synthetic fiber. Natural fiber, being nontoxic and harmless .is composed of vegetable ,minerals and animals where vegetables

utilized for this purpose are bamboo, kemp, sisal ,flax ,banana while the animal components includes wool, skin, hair (Andrea, D.J and Brown, W.R 1993)

The mineral like ceramics and asbestos are required for manufacturing natural fibers. The second form, synthetic fiber is manufactured by men. It includesglass fiber, carbon fiber and aramid, etc (E. W. Godwin and Matthews, F.L 1980)

There is a huge market and demand for the fiber reinforced polymeric composite specifically for the synthetic fiber reinforced composite as it is utilized in marketing pipes, in tanks, sports goods and construction of bridges, boat hull, automotive industry and aircraft secondary structure (Jevanthi, S and JanciRani, J 2011).

Fig.2.2. Extracted Natural fiber

Source: - Milkias Dandessa, extraction of natural fiber, 2017

2.5. Application of natural fiber composites

The exploitation of natural fiber composites in various applications has opened new avenue for both academicians as well as industries to manufacture a sustainable module for future applications of composites (Cheung, 2009).

2.5.1. Automobile components industry

In United States, composites building materials are being made from straw .many automobile components are already produced with natural composites, mainly based on polyester or polypropylene and fibers like flax, hemp or sisal. The adoption of natural fiber composites in this industry is led by motives of price, weight reduction, and marketing rather than technical demands (Mesfin Kebede, 2015).

Germany is the leader in use of natural fiber composites. The German auto manufacturers like Mercedes, BMW, AUDI and Volkswagen have taken the initiative to introduce the natural fiber composite for interior and exterior car body applications (Mesfin Kebede, 2015).

The first commercial example is the inner door panel in 1999 s-class Mercedes Benz, made in Germany, 35% Baypreg F semi-rigid (PUR) elastomer and 65% of blend of sisal, hemp and flax. Another paradigm of natural fiber composite application appeared commercially in 2000, when Audi launched A2 mid-range car in which the door trim panel were made from polyurethane reinforced with mixed sisal and flax fibers. Natural fiber composites can be very cost effective materials for the following applications (Mesfin Kebede, 2015).

2.6. Sisal plant

Sisal (agave sisalna) is a plant native to tropical and subtropical north and South America, the plant is now widely grown in tropical countries of Africa, the West Indies and Far East. The plant has a life span of 7to 10 years and typically produces 200 ±250 commercially usable leaves.

A sisal leave weighing about 600gm will yield 20% weight of fiber with each leave containing about 1000 fibers and each leave has 20% fiber,0.75% cuticle,8% dry matter,87.25% water (www.daff.gov.za).

Fig.2.3.Sisal plantation

Source: - www.daff.gov.za

2.6.1. Classification of the plant

The botanical name of the sisal plant is genus agave L agave sisalana and the agavaceae family, which contains about 300 species. There are many varieties of agave agave plant throughout the world (www.daff.gov.za).

2.6.2. Distribution of the plant

Sisal plant was originally grown in southern Mexico but widely naturalized and cultivated in many other countries .It has been widely introduced in tropics and subtropics, in India between 885-1892, in Tanzania in 1893, in Kenya in 1903-1908.Until 1960s, Tanzania was the leading producer of sisal, but since then Brazil has become the major world producer of sisal followed by Tanzania, Kenya, Madagascar and China. Other countries in Africa that commercially produce the sisal plant include Guinea, Ethiopia, Malawi, Mozambique, Angola, South Africa and Morocco.(www.daff.gov.za).

2.6.3. Morphology of the plant.

2.6.3.1. Mature plant

Sisal is a tall perennial monocotyledon, hard plant that can grow well fast all year round and attains a height of 15.2cm in 9 months after planting and 0.6m at the end of two years. The plant grows for 7-12 years producing 120-180 leaves depending on the location, altitude, level of rain fall and verity of the plant (www.daff.gov.za)

2.6.3.2. The stems of the plant

Two or three years after transplanting, a 20cm tall stem is formed, which will reach a height of about 1.2m. Fleshy stems develops from underground buds at the base of the plant, first growing sideways and then upwards to form new plants. These new plants are called suckers (www.daff.gov.za).

2.6.3.3. The roots of the plant

The sisal plant has a shallow, fibrous root system that is a maximum of 60cm deep. The 2-4 mm thick root arises from leaf scars at the base of the bole beneath the soil surface and extends horizontally up to 5m away from the mother plant and form suckers, which can be used for propagation.

2.6.3.4. The leaves of the plant

The plant has a stiff, heavy, persistent leaves that are 0.6 to 1.2 m long, 10.2 to 20.3cm wide and 2.5 to 10.2 cm thick when matured. The leaves are spirally arranged around the trunk, green in color and covered by a layer of wax. The fibers are found in the leaves (www.daff.gov.za).

2.9. Diversity of sisal plant in Ethiopia

In Ethiopia, the production of Agave sisalana is quite minimal. Nevertheless, most part of the country semi-arid and it accounts for about 71% of the entire 1.115km^2 land area about 46% with that of Agave Americana .Most of the areas being dry land implies that there is a fertile ground and tremendous potential to produce Agave sisalana on small scale basis(by farmers) and on large scale basis. Since the crop is quite labor intensive, it could offer a great deal of job opportunities for the large rural population (sisal initiative Ethiopia brochure).

Year in E.C	Production in tones/year
2007	600
2008	602
2009	461
2010	541
2011	623

Table 2.1. Sisal fiber production in Ethiopia

Source FAO fact sheet 2011 about sisal production in Ethiopia

2.10. Sisal fiber

Sisal fiber is a kind of natural fiber which possessed a high specific strength and modulus and recyclability. The fiber is a bundle of hollow sub-fibers which have a cell wall reinforced with spirally oriented cellulose, in hemi cellulose and lignin matrix. So, the cell wall is a composite structure of lignocellulose material reinforced by helical micro bands of cellulose (Murherjee PS, Satyanarayana KG, 1984).

Fiber	**Tensile Strength (Mpa)**	**Young's Modulus (GPa)**	**Specific Strength (MPa/kg m $^{-3}$)**	**Density (g/cm^3)**	**Elongation at break (%)**	**moisture absorption in percent**
Sisal	600-700	10.40	0.3910	1.33	2-3	11

Table 2.2.Physico mechanical properties of sisal fibers

Source: - Sarah E. Royse University of Nebraska-Lincoln, seroyse@huskers.unl.edu

Fiber	Cellulose (wt %)	Hemicellulose (wt %)	Ligning (wt %)	Waxes (wt %)
Sisal	65	12	9.9	2

Table 2.3. Chemical composition of sisal fiber

Source: - Hindi Publishing Corporation International Journal of Polymer Science Volume 2015, Article ID 243947).

2.10.1. Extraction Methods of sisal fibers

Natural cellulose fibers are extracted from lignocellulosic by products using bacteria and fungi, mechanical and chemical methods. Retting is also a traditional method to extract fibers which uses bacteria and fungi in the environment to remove pectin,lignin and other substances (Han, 1998 and Suhaib A. Sheik).

Bacteria such as Bacillus and Clostridium, used in water retting,and fungi such as Rhizomucorpusillus and fusarium lateritium ,used in dew retting are found to be most effective in their ability to attack the non-cellulosic substance and separate the fibers from core.

Although atmospheric retting provides better quality fibers, it relatively requires relatively longer duration and it is difficult to control. The relatively more common chemical retting methods use alkalis, mild acids and enzymes for fiber extraction. Sodium hydroxide is the most commonly used chemical for fiber extraction.

Acids suchas sulfuric acid and oxalic acid in combination with a detergent are also used for fiber extraction. Chemical concentration, temperature and the duration of treatment are the main factor for the quality of extracted fiber. Sisal fiber can be extracted in different way (Han, 1998 and Suhaib A. Sheik).

2.10.2. Chemical extraction method of sisal fiber

For chemical extraction, alkali treatment is used. The alkali NaOH, sulfuric acid, hydrogen peroxide, protease, pectinase, and sodium citrate can be used. The main disadvantage of chemical extraction is to produce agood quality fiber it can take 35-40 days. There is also a lot of wastage in the process (Han, 1998 and Suhaib A. Sheik).

2.10.3. Mechanical extraction method of sisal fiber

It is extraction of fiber using simple machine consisting of single roller which rolls on a fixed support. The roller is provided with horizontal stainless steel blades with blunt edges. Mechanical extraction using simple machine can reduce labor work and it can increase fiber production by 20-25 times as compared to manual process.

The fully grown plant isused to extract the fibers. The green skin is thrown away retaining the white portionwhich will be processed into knotted fibers. Once dried, the fibers are ready for knotting. Each fiber is separated according to sizes and grouped accordingly (Han, 1998 and Suhaib A. Sheik).

2.10.4. Manual extraction method of sisal fiber

Sisal fiber can be extracted from waste stalk and leaf of sisal plant.But most abundant of sisal fiber is obtained from the leaves of the sisal plant. It can be peeled of easily using ribbons or hand. It is the simple stripping process inwhich trunks are pulled apart and sheath is undressed. Finally the fiber is obtained by removing pulpy part using ribbons.

2.10.5. Alkali treatment of sisal fiber

Alkali treatment the most common and well known type of treatment process in sisal and other natural fibers. During alkali treatment, the most important factors are concentration of alkali, temperature at which fiber is treated with alkali and time length for which the fiber treated with alkali. The possible reaction during alkali treatment is the following (Han,1998 and Suhaib A. Sheik).

$$\text{Fiber–OH} + \text{NaOH} \longrightarrow \text{Fiber-O-Na}^{+} + \text{H}_2\text{O}$$

2.11. Trends of using sisal fiber in Ethiopia

In Ethiopia, there is a trend of using sisal fiber for the production of ropes, bags for packing coffee. But there is also a useof sisal fiber in some part of Ethiopia for the same purpose stated above even if it is not inthe scale that sisal fiber is used. Unfortunately even if there a considerable amount Sisal cultivation in Ethiopia, there is no observable trend of using the fiber.

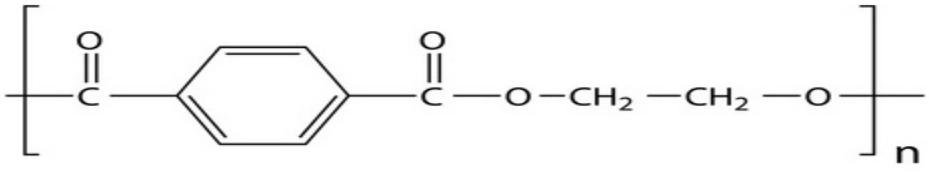
In Ethiopian rural area, farmers who are cultivating sisal in small scale and Ethiopian local farmers have no idea of using the sisal fiber for commercial proposes. It is hard to find literature studied utilizing sisal fiber in context of Ethiopia (Mesfin Kebede, 2015).

2.11.1. Sisal fiber based studies in Ethiopia

In Ethiopia there is no considerable research done on sisal fiber based studies. However, in Ethiopia there is a huge production of sisal plant especially in SNNP region. The only research that can be stated as a research on sisal fiber may be a research done by Mesfin Kebede, Addis Ababa University, on composite material production from sisal fiber and epoxy resin for car body application in the year 2015.

2.12. Pet (Polyethylene Tere Phatalates)

Polyethylene terephthalate (sometimes written poly(ethylene terephthalate),commonly abbreviated PET is the most common thermoplastic polymer resin of the polyester family and is used in fibers for clothing, containers for liquids and foods, thermo forming for manufacturing, and in combination with glass fiber for engineering resins (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).



Molecular Structure of Polyethylene Terephthalate
PET Chemical Formula: $(C_{10}H_8O_4)_n$

The majority of the world's PET production is for synthetic fibers (in excess of 60%), with bottle production accounting for about 30% of global demand. In the context of textile applications, PET is referred to by its common name, polyester, whereas the acronym PET is generally used in relation to packaging.

Polyester makes up about 18% of world polymer production and is the fourth-most-produced polymer; polyethylene (PE), polypropylene (PP) and polyvinyl chloride (PVC) are first, second and third, respectively (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.12.1. Pet manufacturing technology

Polyethylene terephthalate may exist both as an amorphous (transparent) and as a semi-crystalline polymer. The semi crystalline material might appear transparent (particle size less than 500 nm) or opaque and white (particle size up to a few micrometers) depending on its crystal structure and particle size (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

The monomer bis (2-hydroxyethyl) terephthalate can be synthesized by the esterification reaction between tere phthalic acid and ethylene glycol with water as a byproduct, or by trans esterification reaction between ethylene glycol and dimethyl terephthalate (DMT) with methanol as a byproduct. Polymerization is through a poly condensation reaction of the monomers (done immediately after esterification/trans esterification) with water as the by product.

2.12.2. Discovery and History of Polyethylene terephthalate

Pet was patented in 1941 by John Rex Whin field, James Tennant Dickson and their employer the Calico Printers' Association of Manchester, England. E. I. DuPont de Nemours in Delaware, United States, first used the trademark Mylar in June 1951 and received registration of it in 1952. It is still the best-known name used for polyester film. The current owner of the trademark is DuPont Teijin Films US, a partnership with a Japanese company (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

In the Soviet Union, PET was first manufactured in the laboratories of the Institute of High-Molecular Compounds of the USSR Academy of Sciences in 1949, and its name "Lavsan" is an acronym thereof (The PET bottle was patented in 1973 by Nathaniel wyeth (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.12.3. Physical properties

PET in its natural state is a colorless, semi-crystalline resin. Based on how it is processed, PET can be semi-rigid to rigid, and it is very lightweight. It makes a good gas and fair moisture barrier, as well as a good barrier to alcohol (requires additional "barrier" treatment) and solvents. It is strong and impact-resistant. PET becomes white when exposed to chloroform and also certain other chemicals such as toluene (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

About 60% crystallization is the upper limit for commercial products, with the exception of polyester fibers. Clear products can be produced by rapidly cooling molten polymer below Tg glass transition temperature to form an amorphous solid. Like glass, amorphous PET forms when its molecules are not given enough time to arrange themselves in an orderly, crystalline fashion as the melt is cooled. At room temperature the molecules are frozen in place, but, if enough heat energy is put back into them by heating above Tg, they begin to move again, allowing crystals to nucleate and grow. This procedure is known as solid-state crystallization.

When allowed to cool slowly, the molten polymer forms a more crystalline material. This material has spherulites containing many small crystallites when crystallized from an amorphous solid, rather than forming one large single crystal. Light tends to scatter as it crosses the boundaries between crystallites and the amorphous regions between them. This scattering means that crystalline PET is opaque and white in most cases. Fiber drawing is among the few industrial processes that produce a nearly single-crystal product (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.12.4. Chemical properties of pet

Excellent resistance to alcohols, aliphatic hydrocarbons, oils, greases and diluted acids. Moderate resistance to diluted alkalis, aromatic & halogenated hydrocarbons (Sheffield academic press, 2002)

2.12.5. INTRINSIC VISCOSITY

One of the most important characteristics of PET is referred to as intrinsic viscosity (IV). The intrinsic viscosity of the material, found by extrapolating to zero concentration of relative viscosity to concentration which is measured in deciliters per gram (dℓ/g). Intrinsic viscosity is dependent upon the length of its polymer chains but has no units due to being extrapolated to zero concentration (www.wikipedia.org).

properties of pet	
Density	1.38 g/cm3 (20 °C)
Melting point	> 250 °C,
Boiling point	> 350 °C (decomposes)
Solubility in water	Insoluble
Thermal conductivity	0.15W/m.k
Refractive index	1.57–1.58
Heat capacity (C)	1.0 kJ/(kg·K)[1]
Coefficient of friction	0.2 - 0.4
impact strength ($J.m^{-1}$)	13-35
tensile modulus (Gpa)	2-4
Tensile strength (MPa)	190 - 260
Water absorption over 24 hours (%)	0.1

Table 2.5. Physical properties of pet

Source: Stephan Favilla and Pet Good fellow *A ZoM, 2010*

2.12.5. Co polymers of pet

In addition to pure (homo polymer) PET, PET modified by copolymerization is also available. In some cases, the modified properties of copolymer are more desirable for a particular application. For example, cyclohexane di methanol (CHDM) can be added to the polymer backbone in place of ethylene glycol.

Since this building block is much larger (6 additional carbon atoms) than the ethylene glycol unit it replaces, it does not fit in with the neighboring chains the way an ethylene glycol unit would. This interferes with crystallization and lowers the polymer's melting temperature. In general, such PET is known as PETG or PET-G (Polyethylene terephthalate glycol-modified; Eastman Chemical, SK Chemicals Selenis are some PETG manufacturers). PETG is a clear amorphous thermoplastic that can be injection molded, sheet extruded or extruded as filament for 3D printing. It can be colored during processing. Replacing terephthalic acid (right) with isophthalic acid (center) creates a kink in the PET chain, interfering with crystallization and lowering the polymer's melting point. Another common modifier is isophthalic acid, replacing some of the 1, 4-(Para-) linked terephthalate units. The 1, 2-(ortho-) or 1, 3-(meta-) linkage produces an angle in the chain, which also disturbs crystallinity (David w. brooks, pet materials and technology, Sheffield press,2002).

Such copolymers are advantageous for certain molding applications, such as thermoforming, which is used for example to make tray or blister packaging from co-PET film, or amorphous PET sheet (A-PET/PETA) or PETG sheet. On the other hand, crystallization is important in other applications where mechanical and dimensional stability are important, such as seat belts. For PET bottles, the use of small amounts of isophthalic acid, CHDM, diethylene glycol (DEG) or other co monomers can be useful: if only small amounts of co monomers are used, crystallization is slowed but not prevented entirely. As a result, bottles are obtainable via stretch blow molding ("SBM"), which are both clear and crystalline enough to be an adequate barrier to aromas and even gases, such as carbon dioxide in carbonated beverages (David w. brooks, pet materials and technology, Sheffield press,2002).

2.13. Degradation of pet

PET is subjected to various types of degradations during processing. The main degradations that can occur are hydrolytic, and probably most important, thermal oxidation. When PET degrades, several things happen: discoloration, chain scissions resulting in reduced molecular weight, formation of acetaldehyde, and cross-links ("gel" or "fish-eye" formation). Discoloration is due to the formation of various chromophoric systems following prolonged thermal treatment at elevated temperatures. This becomes a problem when the optical requirements of the polymer are very high, such as in packaging applications. The thermal and thermo oxidative degradation results in poor processibility characteristics and performance of the material (www.wikipedia.org).

One way to alleviate this is to use a copolymer. Co monomers such as CHDM or isophthalic acid lower the melting temperature and reduce the degree of crystallinity of PET (especially important when the material is used for bottle manufacturing). Thus, the resin can be plastically formed at lower temperatures and/or with lower force. This helps to prevent degradation, reducing the acetaldehyde content of the finished product to an acceptable (that is, unnoticeable) level. Another way to improve the stability of the polymer is to use stabilizers, mainly antioxidants such as phosphites. Recently, molecular level stabilization of the material using nanostructured chemicals has also been considered.

At least one species of bacterium in the genus Nocardia can degrade PET with an esterase enzyme. Japanese scientists have isolated a bacterium Ideonellasakaiensis that possesses two enzymes which can break down the PET into smaller pieces that the bacterium can digest. A colony of I. sakaiensis can disintegrate a plastic film in about six weeks (www.wikipedia.org).

2.14. Acetaldehyde of pet

Acetaldehyde is a colorless, volatile substance with a fruity smell. Although it forms naturally in some fruit, it can cause an off-taste in bottled water. Acetaldehyde forms by degradation of PET through the mishandling of the material. High temperatures (PET decomposes above 300 °C or

570 °F), high pressures, extruder speeds (excessive shear flow raises temperature), and long barrel residence times all contribute to the production of acetaldehyde. When acetaldehyde is produced, some of it remains dissolved in the walls of a container and then diffuses into the product stored inside, altering the taste and aroma. This is not such a problem for non-consumables (such as shampoo), for fruit juices (which already contain acetaldehyde), or for strong-tasting drinks like soft drinks. For bottled water, however, low acetaldehyde content is quite important, because, if nothing masks the aroma, even extremely low concentrations (10–20 parts per billion in the water) of acetaldehyde can produce an off-taste (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.15. Bottle processing equipment

There are two basic molding methods for PET bottles, one-step and two-step. In two-step molding, two separate machines are used. The first machine injection molds the preform, which resembles a test tube, with the bottle-cap threads already molded into place. The body of the tube is significantly thicker, as it will be inflated into its final shape in the second step using stretch blow molding (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

In the second step, the preforms are heated rapidly and then inflated against a two-part mold to form them into the final shape of the bottle. Preforms (un inflated bottles) are now also used as robust and unique containers themselves; besides novelty candy, some Red Cross chapters distribute them as part of the Vial of Life program to homeowners to store medical history for emergency responders (wikipedia.org).

In one-step machines, the entire process from raw material to finished container is conducted within one machine, making it especially suitable for molding non-standard shapes (custom molding), including jars, flat oval, flask shapes, etc. Its greatest merit is the reduction in space, product handling and energy, and far higher visual quality than can be achieved by the two-step system (wikipedia.org).

2.16. Pet bottle recycling

PET bottle recycling is more practical than many other plastic applications because of the high value of the resin and the almost exclusive use of PET for widely used water and carbonated soft drink bottling. When recycling polyethylene terephthalate or PET or polyester, in general two ways have to be differentiated.The chemical recycling back to the initial raw materials purified tere phthalic acid (PTA) or dimethyl terephthalate (DMT) and ethylene glycol (EG) where the polymer structure is destroyed completely, or in process intermediates like bis (2-hydroxyethyl) terephthalate. The mechanical recycling where the original polymer properties are being maintained or reconstituted (recycling of plastic, Cambridge university press, 2013).

Pet recycling in Ethiopian case is emerging technology in which the wasted pet collected, crushed and exported by private companies.

No.	Name of companies	Location	Annual capacity ton/year
1.	Coba impact private limited company	Addis ababa	3500
2.	Ekt ceramics and plastics plc.	Addis ababa	4000
3.	Assay chemical fiber plc.	Burayu	3600
4.	Huyang recycling plc.	Sebeta	2500
5.	Orex international plc.	Addis ababa	3000
	Total		**16,600**

Table 2.6 Pet Recycling companies in Ethiopia

Source: chemical and construction industry development institute.

2.16. Applications of recycled pet

The PET-based compounds are also suitable for construction (e.g. as structural members), equipment housings (e.g. printer and copier parts), agricultural applications (e.g. mower and tractor engine covers), materials handling (e.g. pallets and trays), furniture (e.g. office chair bases), as well as electrical and electronic applications and automotive components.

PET may be considered a low-cost raw material for the production of engineering compounds due to its widespread availability from recycled beverage bottles. Its abundant availability and good molecular weight make it an excellent precursor for the production of toughened

compounds that can in many cases compete directly with toughened and glass-filled nylons at a considerable cost advantage (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.16.1 Advantages & Key Properties of PET Resin

It has higher strength and stiffness

It is very strong and lightweight & hence easy and efficient to transport

It is known for its good gas (oxygen, carbon dioxide) and moisture barrier properties

It exhibits excellent electrical insulating properties

It has broad range of use temperature, from -60 to 130°C

It has low gas permeability, in particularly with carbon dioxide

It is suitable for transparent applications, when quenching during processing

It doesn't not break or fracture. It is practically shatter-resistant and hence, a suitable glass-replacement in some applications

It is recyclable and transparent to microwave radiation

It is approved as safe for contact with foods and beverages by the FDA, Health Canada, EFSA & other health agencies (PET Packaging Technology, David w and Geoff, Sheffield academic press, 2002).

2.16.4. Limitations of Polyethylene Terephthalate

Lower impact strength

Lower moldability , due its slow crystallization rate

Affected by boiling water

Attacked by alkalis and strong bases

Attacked at high temperatures (>60°C) by ketones, aromatic and chlorinated hydrocarbons and diluted acids and bases

Poor burning behavior

2.17. Compression Moulding

Compression molding is mainly used for the manufacturing of thermosetting plastic materials such as electric switches, ceiling apparatus, and other consumer oriented apparatus (surrender allu, mold design, India, 2008).

2.17.1. Compression Mould Classification

They are classified in to three main categories. These are:-

- Hand moulds
- Semi- automatic moulds
- Automatic moulds

2.17.1.1. Hand Moulds

Hand moulds are usually small in size and do not weight more than 10kg for ease of handling. The material is put in to cavities and the two halves of the mould are assembled and placed between the platens of the press.

After the press has been closed and the parts moulded, the mould is removed from the press and the parts ejected from the mould (Surrenderallu, mould design, India, 2008).

2.17.1.2. Semi- Automatic Moulds

These are varying greatly in size .These moulds are mounted on the platens of the press by clamps. The mould is loaded with material and the two halves closed. After the species have been moulded, some sort of injection mechanisms pushes the parts out of the cavity (Surrenderallu, mold design, India, 2008).

2.17.1.3. Automatic Moulds

They are similar to semi- automatic moulds but do not need and operator once the mould has been set up for production run. The automatic moulds, through the use of the combination of loading devices, timing devices positive ejective systems, sweeps, micro switches, safty devices and cleaning apparatus ,lend themselves well for moulding many articles. There is a purpose for

each type of mould, and many factors such as the size of the piece part and the production requirements, determine which of the three issues (surrender allu, mold design, India, 2008).

2.18. Factors that affect compression moulding

There are three important factors that must be control in compression moulding. These are:-

- Temperature
- Pressure
- Curing time

2.18.1. Temperature

Thermo setting materials which are used in compression moulding are cured by heat and pressure. Heating is an important phase in moulding operation. Heat softens the materials sufficiently to allow it to flow under the influence of the press pressure in to any opening in the mold to the desire shape. The heat can also cause a chemical change or polymerize the material in to its hard infusible finished state (Surrender allu, mold design, India, 2008).

Temperature for thermo setting materials vary from 270 to 350^{o}F and the temperature for moulding various materials can be determined by experimentation or by getting information from manufacturers some particular material. Mould temperature must be maintained with in plus or minus 5^{o} F. Generally, the mould temperature depends on the geometry of the moulded article, the type of mould (Surrender allu, mold design, India, 2008).

2.18.2. Pressure

Pressure needed to mould a particular article depends on the flow characteristics of material, the cavity depth, and the projected area of piece part. Generally, it is recommended that minimum moulding pressure of 225kg/cm^2 of projected area to be used. However, in practice, about 375kg/cm^2 of projected area is used to compensate for any variables that may be encountered (Surrender allu, mold design, India, 2008).

2.18.3. Curing time

In compression moulding, curing time is the time elapsed when the movement of the press stops until the pressure on the mould is released. It ranges from 1 minute to 15 minutes (Surrender allu, mold design, India, 2008).

2.19. Injection moulding

Thermo plastic materials are used in injection moulding. The thermo plastics soften and become plastic in state by the application of heat and harden when cooled. There is no chemical reaction taking place during the process of heating and cooling. These materials have linear molecular chains that flow over each other when heated and solidify into a new shape when cooled without significant chain breakage (R.J. Crawford, plasticengineering, 1987).

2.19.1. Principle and process of injection moulding

The process of injection moulding essentially consists of plasticizing the raw material in a cylinder by the application of heat and then injecting it under pressure through a nozzle by means of a ram into a closed mould, where it is allowed to cool, and then discharged by opening the mould and removing the moulded component. There are two methods employed to inject the plastic material into the mould: one by means of plunger and another by means of a screw which transports the materials during which time it is plasticized and then it is injected (R.J. Crawford, plastic engineering, 1987).

2.19.2. Injection moulding

Injection moulding is one of the most versatile processing methods by means for manufacturing small clips to large industrial scales. Technological advancement was taken place over the last two decades. Sophisticated micro processed control of injection moulding; structural from moulding, two color injection moulding, gas assisted injection moulding etc., are gaining more and more popularity. Precision moulds are essential to meet the quality requirements of the end product (R.J. Crawford, plastic engineering, 1987).

From a practical point of view, a classification of injection moulds should be based on the main design features and manner of operation. These includes

- The type of gating and means of de-gating
- The type of ejection
- The presents or absence of external & internal under-cuts on the products
- The manner in which the product is released form the moulds

The different types of injection moulds are;

- Stranded moulds (two or three plate moulds)
- Split cavity moulds
- Stripper plate moulds
- Stack moulds
- Hot runner moulds

Thermo plastic materials are used in injection moulding. The thermo plasticssoften and become plastic in state by the application of heat and harden whencooled. There is no chemical reaction taking place during the process ofheating and cooling. These materials have linear molecular chains that flowover each other when heated and solidify into a new shape when cooled without significant chain breakage (R.J. Crawford, plastic engineering, 1987).

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2.19.4. Injection moulding machines:

The injection moulding machine essentially consists of two units namely the mould clamping unit and the injection unit. The mould clamping unit performs the function of closing the mould, locking the mould and opening it, the injection unit. The mould clamping unit performs the functions of closing the mould, locking the mould and opening it. The injection unit plasticizes the material and injects it in to the moulds (R.J. Crawford, plastic engineering, 1987).

The left side is the clamping unit and the right side the injection unit. The mould clamping unit comprises of the fixed, movable and end platens. The cover half of the mould is mounted on the fixed platen and the ejector half of the mould is mounted on the fixed platen and the ejector half of the mould is mounted on the movable platen.

The fixed and end platens are held by four tie bars and the movable platen moves over the four tie bars. There are, in general, two methods employed to move the movable platen and to apply the locking force – these are direct hydraulic locking system and the mechanical toggle system (R.J. Crawford, plastic engineering, 1987).

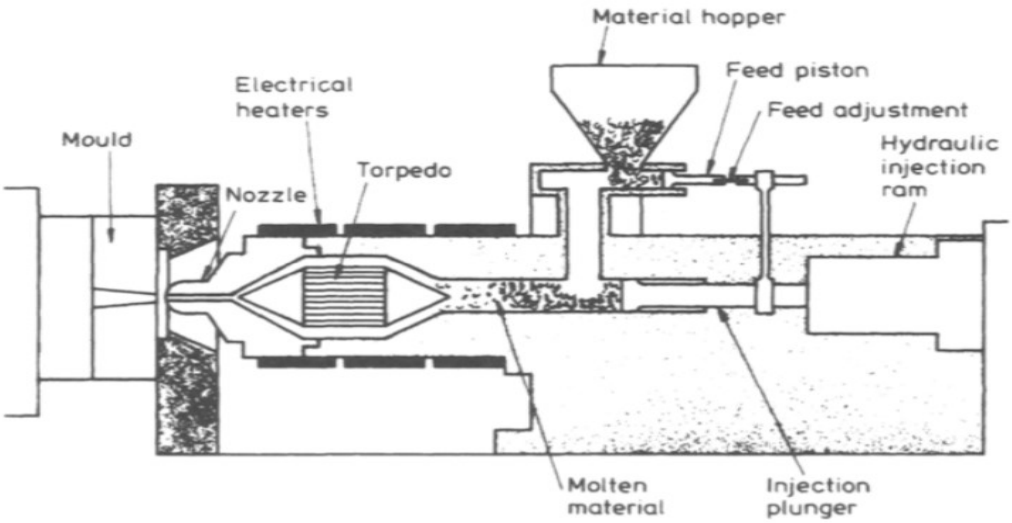


Fig 2.4. Injection molding machine

Source :-(R.J. Crawford, plastic engineering, 1987)

The injection unit is required to plasticize the material and inject it into the mould cavity and exert a holding pressure on the molten material in the mould cavity. Almost all of the injection moulds are of the semi – automatic or automatic type. Depending on the size of piece part and the size of press available, moulds are made in single or multiple cavity.

A variety of ejector systems, which are generally an integral part of the mould, are used to eject the piece parts, runners etc., for the mould. All injection moulds are cooled by having water circulate through channels drilled into the various part of the mould. Injection moulding offers a very efficient and economical method of moulding pieces from a wide range of thermoplastic materials. Parts moulded by this method possess very little or no flash, thus eliminating secondary operation of sanding, filing, and tumbling.

Very high production rates are possible because of shorter moulding cycles than in moulding thermo – setting materials of comparable size. There is no wastage of material in the injection moulding of the thermo – plastic materials, as the scrap, runners, sprues, and incompletely – moulded pieces are reground and re moulded (R.J. Crawford, plastic engineering, 1987).

2.19.5. Composite processing process flow

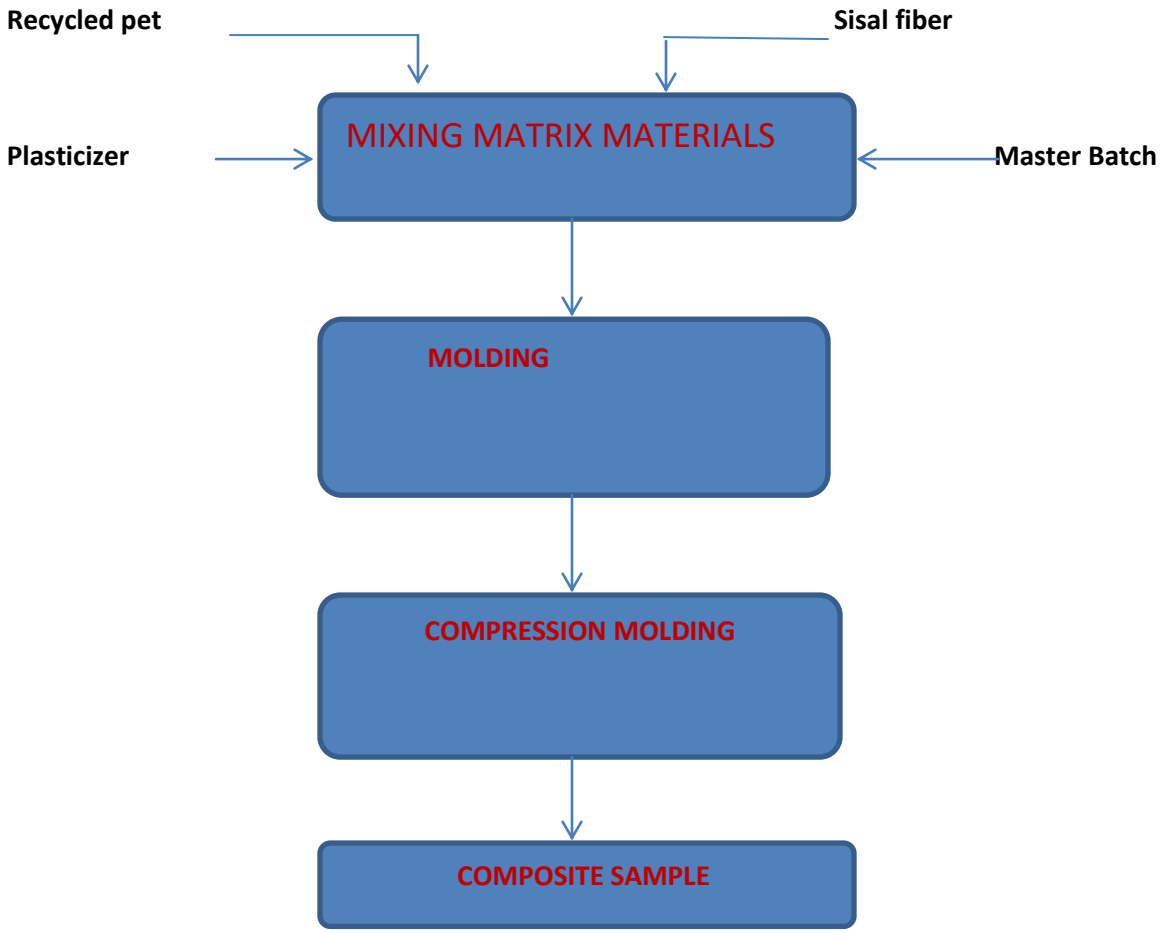


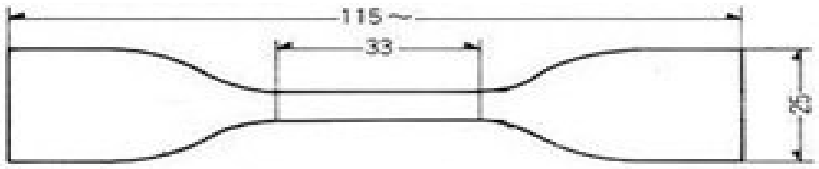
Figure 2.5. Schematics flow diagram for the fabrication of sisal fiber reinforced recycled pet resin composites

2.20. Universal testing of composite

2.20.1. Tensile testing

Tensile testing is one of the most fundamental tests for engineering, and provides valuable information about a material and its associated properties. These properties can be used for design and analysis of engineering structures, and for developing new materials that better suit a specified use (Stephan Favilla, 2010).

This test method uses standard "dumbell" or "dogbone" shaped specimens under 14mm of thickness. A universal testing machine (tensile testing machine) is needed to perform this test.



Analysis obtained:

- Tensile Strength
- Elongation at Yield
- Elongation at Break
- Nominal Strain at Break (Grip Separation)
- Modulus of Elasticity
- Secant Modulus
- Poisson's Ratio (Requires Transverse Extensometer)

Equipment required:

1. Universal testing machine (tensile testing machine)
- Needs to be servo controlled to keep a constant rate of speed.

- Capacity needs to be enough for your materials. A 1,000 lbf single column system is usually sufficient for most non-reinforced plastics. A 2,000 lbf dual column system is also very common. A high capacity 10,000 lbf model is sometimes needed for larger samples and/or stronger materials such as reinforced plastics or composites.

2. Extenso meter

   Required when measuring modulus, yield, and modulus for two reasons: 1) the linear region of plastics is very small and happens suddenly so grip separation is just not accurate enough. 2) Dumbbell specimens do not have uniform widths so there will be errors when both the wide and narrow sections of the dumbbell shaped specimen elongate at different rates.

3. Data Acquisition

   Software or suitable electronics are required to operate the machine and to take the measurements. Basic systems will provide the raw data, and stress-strain charts. Using these sources of data, one can determine and calculate all of the analysis listed above. However, fully PC based systems have the capability to calculate all of these automatically. For example, our MTEST Quattro testing software has built in support for ASTM D638 and all of these calculations are provided immediately after performing the test.

4. Tensile Grips

   Any grip with serrated faces is usually adequate for this test. It is possible to use wedge, pneumatic, vise, or other self-tightening grips such as eccentric roller or scissor grips.

Test Specimen The test specimen shall consist of a strip 12.70 mm wide by 76.20 mm in length and at least 10 μm in thickness. The width of the specimen should not deviate by more than 2% over the length of the specimen between the grips. The thickness of the films shall not vary by more than 10% over the entire film. A minimum of ten specimens are required (Stephan Favilla,2010)

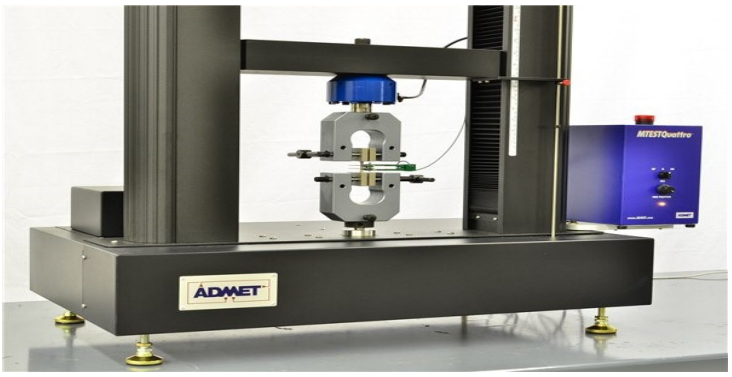


Fig ASTM D 882 Standard Test Methods for Tensile Properties of Thin Plastic Sheeting

Tensile test of pet specimens followes ASTM Standard test method D638 (ASTM 1997a). The test specimens conditioned at temperature of 23 ± 2°C and relative humidity of 50 ± 5% for more than 40 h prior to the test. The Instron Universal testing machine or Instron Model 1122 testing machine can used to perform the tensile strength test at a crosshead speed of 5 mm/min. Each test shall be performed until tensile failure occurred. The test shall be conducted on 3 specimens per pet sample. The maximum (peak) load (Fmax, N) will be read from the instrument. The tensile strength (σt, Pa) then calculated according to the following equation (Stephan Favilla, 2010).

$$\sigma_t = \frac{Fmax}{A}$$

Where, where A = cross-sectional area in m^2

2.20.2. Percent elongation

The percent elongation of PET is the percentage increase in length of the specimen at its breaking point and is calculated from the following equation:

$$\% \text{ elongation} = \frac{L-LO}{LO} X100$$

Where L_0 = original measured length (m),

And L = length of the specimen at its breaking point (m).

$$\text{percent elongation} = \frac{(\text{elongation at rupture}) \times 100}{(\text{initial gage length})}$$

2.20.3. YOUNG'S MODULUS

Young's modulus is calculated by drawing a tangent to the initial linear portion of the stress strain curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. For purposes of this calculation, the tensile stress shall be calculated by dividing the load by the average original cross section of the test specimen. The result is expressed in giga pascals (GPa) and reported (Stephan Favilla,2010).

$$\text{Young's modululus} = \frac{\dfrac{(\text{load at point on tangent})}{(\text{original width})\ (\text{original thickness})}}{\dfrac{(\text{elongation at point on tangent})}{(\text{initial gage length}}}$$

The tensile properties determined using this test method will vary with method of specimen preparation, specimen thickness, specimen width, rate of grip separation, initial gauge length, type of grips used, and method of measuring extension. The tensile strength and elongation are sensitive to the specimen dimensions and any flaws in the specimen. Young's modulus is an index of the stiffness of the specimen and is sensitive to the rate of grip separation. Note that materials that fail by tearing give anomalous data that cannot be compared with those from normal failure (Stephan Favilla, 2010).

A tear failure is a tensile failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous stress-strain curve. Results obtained using different specimen dimensions or at different rates of grip separation are not comparable; consequently, when trying to make quantitative comparisons between specimens or between laboratories, these factors must be carefully controlled (Stephan Favilla, 2010).

CHAPTER 3

3. Materials and methods

3.1. Pet (Poly Ethylene Tere Phatalate)

The matrix used for this study is recycled pet chips which were collected from the recyclers in Addis Ababa, Ethiopia.

3.2. Sisal fiber

Suitable quantity of sisal plant leaves were collected from south regional state, at Hawassa Tikur Wuha. The fibers were extracted through hand extraction with knife. Initially, the leaves were trimmed in longitudinal direction into strips for ease of fiber extraction. The peel was clamped between the wood plank and knife and hand-pulled through in longitudinal direction gently, removing the resinous material,then the extracted fiber washed with pure water in order to loosen, and separated the fiber until individual fibers are obtained then the extracted fibers was sun-dried. Once dried, the sisal fibers were ready for fabrication of test pieces of extracted sisal fiber.

3.3. Chemicals

- Sodium hydroxide (NaOH) RANKEM,S0290
- Plasticizer (De Oxi Phthalate Esters, DOP)
- Master Bach.

3.4. Equipments

- NABERTHER MGmbH Furness
- Molder or metal plate,
- Flexural Strength Tester (UC SM), 200N/M^2- 600N/M^2 testing range
- Beakers,
- Reagent bottles,

- Stirrer,
- Beam balance
- Burette,
- Internal bond testers, ZWICK/ROELL testing machine 1.1N/mm^2 - 1.4N/mm^2 testing range
- Thermometer digital
- Pressing machine,
- Metal mold,
- Oven,
- Crucible
- Tong
- Desiccator
- Scissor
- thickness gauge (analog)meter
- Instron Universal tensile testing machine Model 1122

3.5. Sample preparations

3.5.1. Preparation of sisal fiber

The fibers were cut into ~ 9.0 mm using a pair of scissors chopped sisal fiber is shown in fig 3.2

(b) **(b)** **C**

Fig 3.2**.** Preparation of sisal fiber

Suitable quantity of sisal plant leaves was collected from south regional states at Hawassa, tikur wuha, Ethiopia. After cutting at their base from the harvest. The fibers are extracted through

hand extraction with knife. Initially the leaves trimmed in longitudinal direction into strips for ease of fiber extraction. The peel is clamped between the wood plank and knife and hand-pulled through in longitudinal direction gently, removing the resinous material then the extracted fiber washed with pure water in order to loosen, and separate the fiber until individual fibers are obtained. Then the extracted fibers are sun-dried which whitens the fiber. Once dried, the sisal fibers are ready for fabrication of test pieces extracted sisal fiber is shown in figure 3.2C.

3.5.2. Alkali treatment of sisal fiber

Alkali treatment is the simplest method of chemical treatment of fibers; In order to observe the effect of concentration of the alkali, it has been taken 20 %, 15 % and 10 % of alkali solution during the course of fiber treatment.

During this process dried and chopped sisal fibers were taken in to 9 different beakers. Then in the first 3 beaker of fiber holding 20 %, 15 % and 10 % of NaOH solution , treatment time was taken as 24 hours, in the second 3 beaker of fiber holding 20 %, 15 % and 10 % of NaOH solution , the treatment time taken as 48 hours and with the last 3 beakers of sisal fiber holding 20 %, 15 % and 10 % of NaOH solution, treatment time taken as 72 hours .

The fibers were then washed thoroughly with water to remove the excess of NaOH sticking to the fibers. Lastly, the fibers were allowed to dry in sun light for at least 4 day as shown in figure.

Fig 3.2. Alkali treatment of sisal fiber

3.5.5. Determination of ash content of the fiber

Ash content was determined using the ASTM D 2017 (1998). Two grams of three different samples in three different crucibles were prepared, weighed and recorded and then allowed to Furness at 600 ^{o} C until a complete ashing is achieved. The crucible together with the ash was then transferred into a desiccator for cooling for 15 minutes. The cooled samples were then weighed. The ash content is calculated by using the equation:

$$\textbf{Ash content (fiber)} = \frac{w2-w0}{w1-w0} X100$$

W_0 = the weight of the crucible,

W_1 = the weight of the crucible + sample before incineration,

W_2 = the weight of the crucible + sample after incineration

3.5.6. Determination of water absorption of the fiber

The effect of water absorption on sisal fiber were investigated in accordance with ASTM D 1037(1991) .the specimen were dried in an oven at 50 ^{0}c and then they were allowed to cool till they reached room temperature. The specimen was weighted .water absorption tests were conducted by immersing the fiber in distilled water in a plastic tube at room temperature for different time durations. Once in 24 hours, the samples were taken out from the water and all surface water was removed and weighted. The moisture absorption was calculated by the weight difference. Finally the water absorption was calculated as follows.

$$\textbf{MA of the fiber (\%)} = \frac{wi-wf}{wi} \text{x100}$$

Where,

wi = initial weight of fiber

wf = final weight of fiber

3.6. Determination of tensile strength of pet

Tensile test of pet specimens followed ASTM Standard test method D638 (ASTM 1997). The test specimens were conditioned at temperature of 23 ± 2°C and relative humidity of 50 ± 5% for more than 40 h prior to the test. The Instron Universal testing machine or Instron Model 1122 testing machine was used to perform the tensile strength test at a crosshead speed of 5 mm/min. Each test was performed until tensile failure occurred. The test was conducted on 3 specimens per pet sample. The maximum (peak) load (Fmax, N) was read from the instrument. The tensile strength (σt, Pa) was then calculated according to the following equation

$$\sigma_t = \frac{Fmax}{A}$$

Where, where

Fmax = maximum (peak) load

A = cross-sectional area (m^2)

Test Procedure:

1. Cut or injections mold your material into one of the five “dumbbell” shapes. The exact shape you use is dependent upon your material’s rigidity and thickness.
2. Load the specimen into tensile grips.
3. Attach the extensometer to the sample
4. Begin the test by separating the tensile grips at a constant rate of speed. Speed depends on specimen shape and can range from 0.05 – 20 inches per minute. The target time from start of test to break should be from 30 seconds to 5 minutes.
5. End the test after sample break (rupture)

3.7. Determination of percent elongation

Percent elongation is calculated by dividing the elongation at the moment of rupture by the initial gauge length and multiplying by 100. When gauge marks only this length is used in the

calculation, otherwise the distance between the grips is used as the initial gauge length. The result is expressed in percent and reported to two significant figures. Or extensometers are used to define a specific test section.

$$\text{Percent elongation} = \frac{elongation\ at\ rapture}{initial\ gage\ length} \text{x} 100$$

$$\textbf{\% elongation} = \frac{\boldsymbol{L-LO}}{\boldsymbol{LO}} \boldsymbol{X100}$$

Where L_0 = original measured length (m),

And L = length of the specimen at its breaking point (m).

3.8. Water absorption of pet

The water absorption test followed ASTM standard test method D570 (ASTM 1998). Before the measurement, the sample was dried in an air oven at 50°C for 24 h, cooled in a desiccator, and immediately weighed to the nearest 2 g which is then taken as the dry initial weight of the sample, M0. Then the specimen was placed in a container of distilled water maintained at a temperature of 23 ± 1°C for 24h. After 24 h, the specimen was removed from water; water was wiped off the surface of the sample with a dry cloth; and the sample was immediately weighed , which is designated as M1. For each pet sample, 3 specimens were measured. The water absorption of the sample was calculated as percent weight change (M %) determined as follows:

$$\textbf{MA of the fiber (\%)} = \frac{\boldsymbol{w0-w1}}{\boldsymbol{w0}} \textbf{x100}$$

Where,

W0 = initial weight of pet

W1 = final weight of pet

3.9. Composite sample preparation

The composite was made by taking the fiber and the recycled pet in ratio that is 15/85 % fiber /pet, 25/75 % fiber/pet and 35/65 % fiber/pet and melted in 246 ^{o}C and transferred to handmade

iron mould and allowed to compression. 10 Mpa of pressure was maintained and it requires 20 minutes for curing each 100 gram composite made at room temperature. After curing period, the produced composite material was removed. A typical hydraulic press was used for curing the composite.

3.10. Determination of density bio composite

The density of bio composite is determined as follows. First, measured the weight of bio composite in weighing balance, second measured the dimension in length and width of bio composite by using flexible meter, and third measured the thickness using the thickness gauge (analog) meter at the 4 points and taken the mean values. Then, calculate the gross density of the bio composite using the following formula:

$$\textbf{Gross density (g/cm}^3\textbf{)} = \frac{Wbc}{T\,xW\,x\,L}$$

Where

W $_{bc}$- weight of bio composite

T- Thickness

L- Length

W- Width

3.11. Determination of Internal Bonding Strength

Fig 3.4. internal bonding testing

The internal bonding was tested and evaluated at wood and forest industry development institute with internal bonding tester. First, the samples were polished with glue at both sides and attached

with woods at both sides as shown in the fig 3.4. Each sample of the composite allowed to the testing machine and record the force at which the samples were disintegrated.

3.12. Determination of Flexural Strength

The flexural strength was determined by taking the sample form composite material produced and measured the mass of each sample and calculated the average thickness of sample. The width of specimen, b=9.5mm, length of specimen, L=9.5mm, and the thickness of the sample, t= 1mm, with support span-to-depth ratio of 16:1 and support span length51.2 mm rate of cross head motion 0.5mm/min and measured the weigh each test piece in the precision balance and calculate the gross density of each test piece. Fix the bending Yoke, support bar in the ZWICK/ROELL machine and adjust the distance of the support at Ls=Bending length test piece 9.5mm as shown in figure 3.6. Then force was applied manually until the sample broken down then recorded the force by which the samples were raptured. The flexural strength was expressed as modulus of rupture (MR) in psi (MPa) (John, J, Gangadhar, 2001).

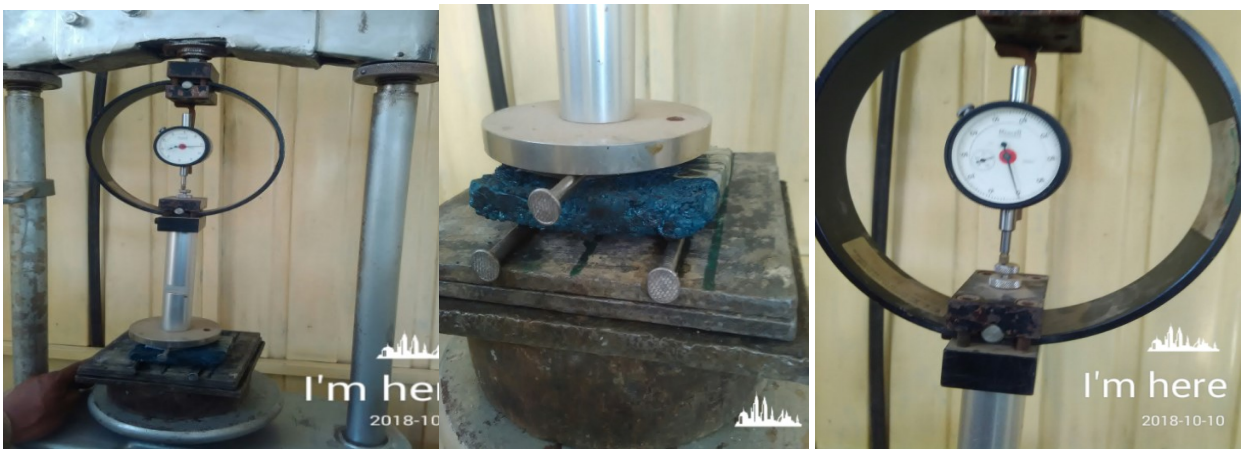


Fig 3.6. Fluxiral Strengh Sesting Machine

Source: - Photographed Sample at Mafcoon Engineering Laboratory.

The load was applied in the middle span of the specimen at a speed of 0.5mm/min. The span length was 51.2mm as shown in figure 3.7. Then the flexural testing was conducted in the testing range of 200 $\frac{N}{M2}$ – 600 $\frac{N}{M2}$which is integrated with the standard UTM test machine (John, J, Gangadhar, 2001).

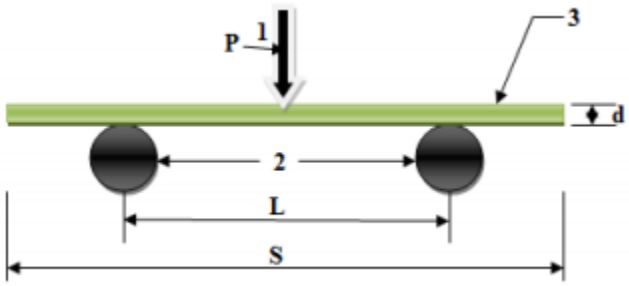


Fig. 3.7.Three bending test

Under three point bending in figure 4.1when the load P is applied at mid span ofa rectangular specimen ofspan L between two rollers, the highest flexural strength is determined by:

$$\sigma_{bf} = \text{Smax} = \frac{3 \times L \times P}{2(b \times d^2)}$$

The deflection of the specimen by considering specimens as a beam (Dc) from the center as illustrated in figure 4.1can be expressed as:

$$Ec = \frac{r \times L^2}{6d}$$

The maximum flexural strain εf also calculated from:

$$\varepsilon_f = \frac{6 \times Ec \times d}{L^2}$$

The bending elastic modulus (E) is determined from the slope of the Load-deflection curve in a linear region and mathematically expressed as:

$$E = \frac{mXL3}{4X(bxd3)}$$

Where

σ_{bf}=E= calculated fracture stress (flexural strength), MPa

P = load at a given point on the load- deflection curve, N

E_c= is the maximum deflection at the center of the specimen (camber distance), mm

m= is the slope of the tangent to the straight-line portion of the load-deflection beam.

L= support span of specimen, mm

b = width of the specimen tested, mm

d = depth of the specimen, mm

r = strain, mm/mm

Smax = is the maximum strength of the material and it is also given in Mega Pascal (MPa)

3.13. Design of Experiment

The design experiment was used according to the standard method for scientific research. Three factors with three levels full factorial design were used in experiment to make the bio composite at different process parameters. The three factors are concentrations of alkali, fiber to recycled pet composition and treatment time.

The fiber was treated with 20, 15, 10 percent of alkali, therefore the 3k design; three factors with three levels with one response variable which is mechanical property (internal bonding strength) of composite was measured.

In this study, statistical experimental design techniques were used to determine the effect of the alkali concentration, pet to fiber composition, treatment time in the production of the bio composite.

A total of 27 experiments were conducted for optimization of the production of the composites and the effect of each factor was analyzed by taking minimum and maximum values from operating condition which has significance influence on the properties of the composites.

The maximum and minimum values of variables of process parameters of synthesized composite were presented as follow.

	Variables	Units	Lower Level(-)	Medium Level	Higher Level(+)
1	Alkali concentration	%	10	15	20
2	Treatment time	hr	24	48	72
3	Composition ratio of sisal to recycled pet	%	15/85	25/75	35/65

Table 3.1. The maximum and minimum values of variables of process parameters

The codified (x_1, x_2 and x_3) and respective non codified levels(alkali concentration in percent, treatment time of fiber in hours and composition ratio of sisal to recycled pet in percent) for optimization of propose were described below.

Exper. run	Codified levels of variables			Non Codified levels of variables		
	X1	X2	X3	Alkali concentration (%)	treatment time (hr)	Composition ratio of sisal to recycled pet (%)
1	0	-1	+1	15	24	35/65
2	-1	0	+1	10	48	35/65
3	-1	+1	+1	10	72	35/65
4	-1	-1	+1	10	24	35/65
5	+1	-1	-1	20	24	15/85
6	+1	-1	0	20	24	25/75
7	-1	+1	-1	20	72	15/85
8	0	0	+1	15	48	35/65
9	+1	-1	+1	20	24	35/65
10	0	+1	0	15	72	25/75
11	0	+1	+1	15	72	35/65
12	+1	0	-1	20	48	15/85
13	0	0	-1	15	48	15/85
14	0	-1	-1	15	24	15/85

15	-1	-1	-1	20	24	15/85
16	+1	+1	+1	20	72	35/65
17	0	+1	-1	15	72	15/85
18	+1	0	+1	20	48	35/65
19	+1	+1	0	20	72	25/75
20	+1	0	0	20	48	25/75
21	+1	+1	-1	20	72	15/85
22	0	0	0	15	48	25/75
23	-1	-1	0	10	24	25/75
24	-1	+1	0	10	72	25/75
25	0	-1	0	15	24	25/75
26	-1	0	-1	10	48	15/85
27	-1	0	0	10	48	25/75

Table .3.2. Codified levels of variables and non-codified levels of variables.

CHAPTER FOUR

4. Result and Discussion

4.1. Characterization of Sisal fiber and the Polyethylene Tere Phatalate

4.1.1. Determination of moisture content and ash content of sisal fiber

<table>
<tr><td colspan="4">water absorption of Sisal Fiber</td><td colspan="7">Sisal Fiber Ash Content Determination</td></tr>
<tr><td colspan="2">Sample weight(in gram)</td><td>Moisture content (%)</td><td>Average MC (%)</td><td colspan="7">Before</td></tr>
<tr><td colspan="2" rowspan="2"></td><td rowspan="2"></td><td rowspan="2"></td><td>weight</td><td>C_1</td><td>C_2</td><td>C_3</td><td>C_1+ fiber</td><td>C_2 + fiber</td><td>C_3 + fiber</td></tr>
<tr><td>Initial weight</td><td>Final weight</td><td>13.5%</td><td>20.75</td><td>24.87</td><td>23.43</td><td>21.750</td><td>26.87</td><td>25.43</td></tr>
<tr><td>60.00</td><td>67</td><td>11.6</td><td colspan="7">After</td></tr>
<tr><td>76</td><td>91</td><td>19.7</td><td>weight</td><td>C1</td><td>C 2</td><td>C 3</td><td>C1+ fiber</td><td>C2 + fiber</td><td>C3 + fiber</td></tr>
<tr><td>91</td><td>103</td><td>13.1</td><td></td><td></td><td></td><td></td><td>21.741</td><td>26.878</td><td>25.439</td></tr>
<tr><td colspan="4" rowspan="4"></td><td colspan="4">Change in Weight</td><td>0.009</td><td>0.008</td><td>0.009</td></tr>
<tr><td colspan="4">Average the Ash Content</td><td colspan="3">0.009</td></tr>
<tr><td colspan="4">% Ash content</td><td colspan="3">0.9</td></tr>
<tr><td colspan="7">C_1,C_2 and C_3 are Crucibles</td></tr>
</table>

Table 4.1. Water absorption and Ash content of sisal fiber

The standard Water absorption of sisal fiber is expected to be in a range from 5% up to 15% and an average value of Water absorption obtained from the experiment as shown in the above Table 4.1 was 13.1%.

The Ash Content of sisal fiber obtained from the experimental result was 0.9 percent. It was calculated as follows ;

$$\textbf{Ash content (fiber)} = \frac{w2-w0}{w1-w0} X100$$

W_0 = the weight of the crucible,

W_1 = the weight of the crucible + sample before incineration,

W_2 = the weight of the crucible + sample after incineration

The result obtained for ash content compared adequately with what was observed for sisal fiber by Chow *et al.* (2008). The standard ash content of natural fiber is 1 %. Therefore, the result obtained is acceptable.

4.1.2. Characterization of recycled pet

4.1.2.1. Physico mechanical properties of pet

Properties	Unit	Test 1	Test 2	Test 3	Average
Tensile Strength	(Mpa)	210	204	216	210
percent elongation	(%)	85	87	87	86.3
Moisture absorption	(%)	0.09	0.081	0.082	0.08

Table 4.2. Physico mechanical properties of pet

The standard tensile strength of the polyethylene tere phthalate is in a range from 190 Mpa up to 260Mpa (Stephan Favilla,2010) and an average value of tensile strength of the polyethylene tere phthalate obtained from the experiment as shown in the above Table 4.2 was 210 Mpa. The tensile strength (σt, Pa) was then calculated according to the following equation.

$$\sigma_t = \frac{Fmax}{A}$$

Where:

σ_t = tensile strength

F max =the maximum peak load

A= Area of the sample

The standard percent elongation of the polyethylene tere phthalate is in a range from 65% up to 165 % (Stephan Favilla, 2010) and an average value of percent elongation of the polyethylene

tere phthalate obtained from the experiment as shown in the above Table 4.2 was 86.3%. The percent elongation was then calculated according to the following equation.

$$\text{Percent elongation} = \frac{elongation\ at\ rapture}{intial\ gage\ length} \text{x} 100$$

The standard moisture absorption of the polyethylene tere phthalate is 0.1 (Stephan Favilla, 2010) and an average value of moisture absorption of the polyethylene tere phthalate obtained from the experiment as shown in the above Table 4.2 was 0.08.

The moisture absorption was then calculated according to the following equation.

$$\textbf{MA of the pet (\%)} = \frac{wi - wf}{wi} \textbf{ x100}$$

Where,

wi = initial weight of pet

wf = final weight of pet

4.2. Effect of internal bonding strength process variables

Based on analysis of variance (ANOVA), the internal bonding strength was significantly affected by various interactions between the process variables. Instead, significant individual process variables that affect internal bonding strength are concentration of alkali, A , treatment time, B, and sisal to recycled pet ratio, C. This result demonstrated the advantage of using design of experiments in capturing the interaction between variables that affects the internal bonding strength of the composite.

4.2.1. Effect of concentration of alkali on the internal bonding strength of composite

The concentration of alkali affects the internal bonding strength of composite positively. As it is easily can be observed from Fig.4.1, the concentration of alkali increases, the internal bonding strength also increases. After alkali treatment, it is seen that the treatment has removed the surface debris from the fiber; most of the lignin and pectin are removed resulting in a rough surface. This rough surface facilitates both mechanical interlocking and bonding reaction due to

the exposure the hydroxyl groups to the matrix, thereby increasing the fiber-matrix adhesion (Gonzalez-murilo C,Ansell M P).

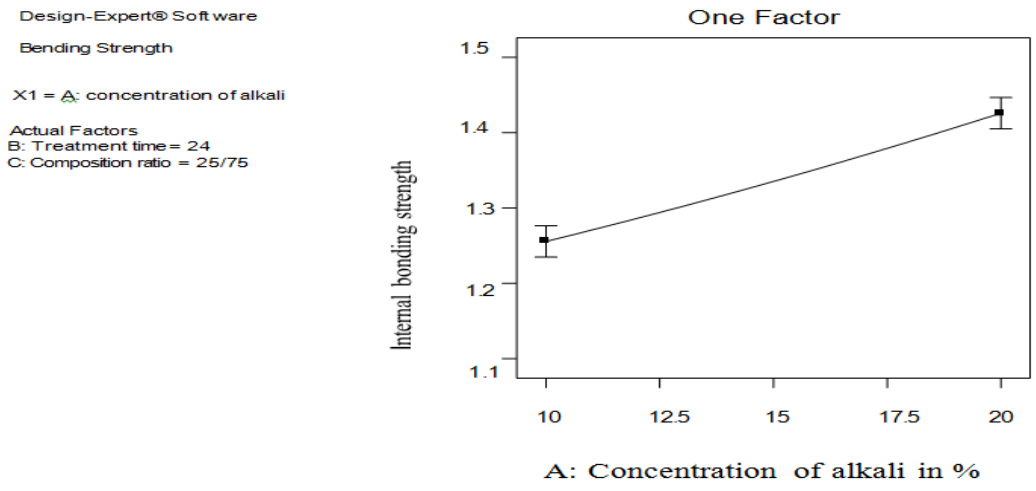


Figure 4.1. The effect of alkali concentration in the internal bonding strength of composite

4.2.2. Effect of treatment time on the internal bonding strength of composite

The treatment time affects the internal bonding strength positively. As it is easily can be observed from Fig.4.2, the treatment time increases, the internal bonding strength of composite also increases.

The sisal fiber was treated with different treatment time of 24, 48 and 72 hours. It indicates that more lignin and polluters removed from the fiber surface without degrading the fiber. The increased properties are also caused by the increasing of interfacial bonding between the fiber and the polymer matrix due to the effect of cellulose fiber purification (Dikuncoro diharo 2017).

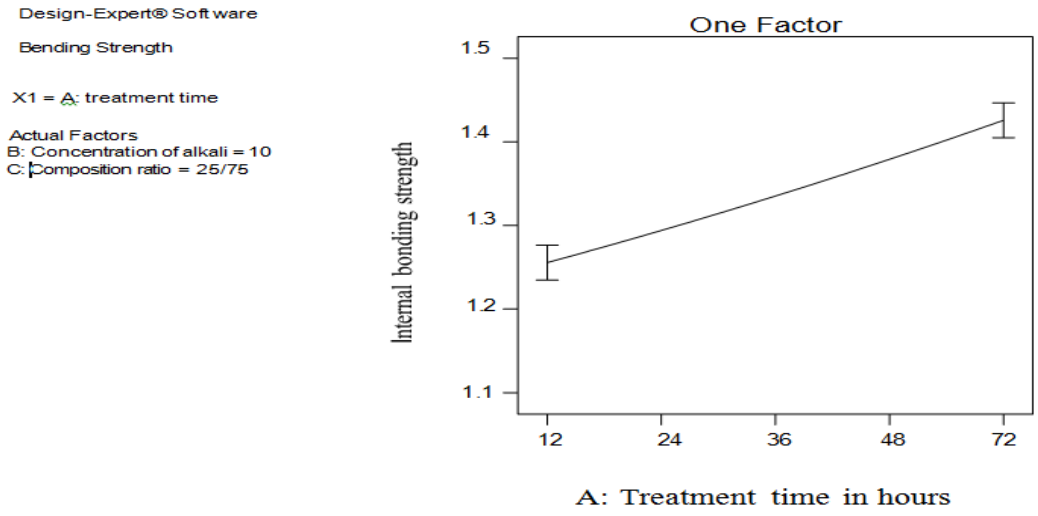


Figure 4. 2. The effect of treatment time in the internal bonding strength of composite

4.2.3. Effect of composition of sisal fiber to recycled pet ratio on the internal bonding strength of composite

The composition of sisal fiber to recycled pet ratio affects the internal bonding strength positively. As it is easily can be observed from Fig.4.3, the composition of sisal fiber to recycled pet ratio increases, the internal bonding strength of composite also increases. As the ratio of composition in the composite increases nearest to certain optimal level, the physical properties will increases (Hile gebreal gemechu, 2017).

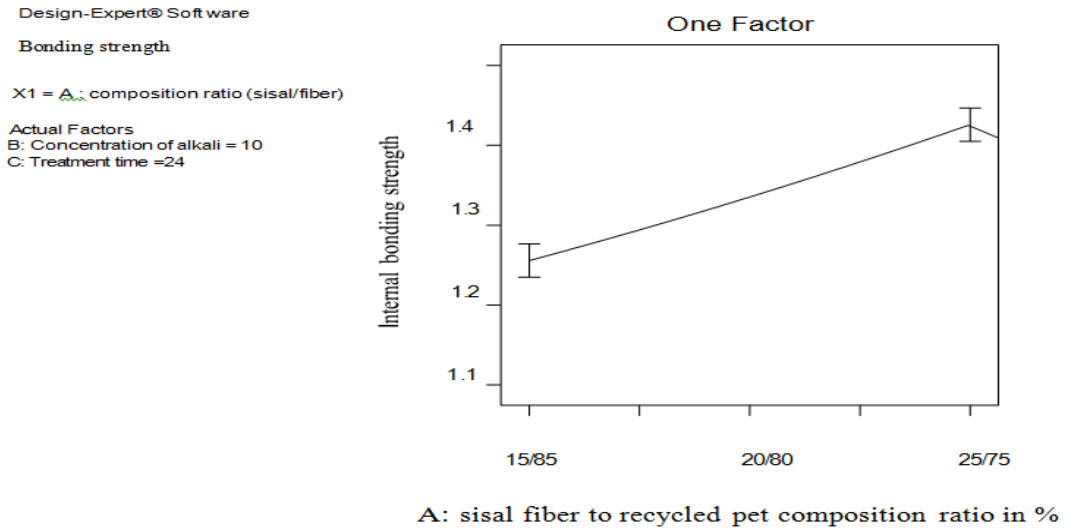


Figure 4.3. The effect of sisal fiber to recycled pet ratio in the internal bonding strength of composite.

As shown from Fig. 4.1, 4.2 and 4.3 above the effect of one factor is not sufficient in describing the effects of the parameters have on internal bonding strength.

This suggests that the interactions between the internal bonding parameters were also found significant. This result agrees with the ANOVA output for the regression model equation and coefficients.

4.3. Effect of Interaction between Process Variables

The process variables were found to have significant interaction effects. Figure 4.4, 4.5 and 4.6 show the interaction between composition ratio, treatment time, and alkali concentration, respectively, on bonding strength.

Generally, an increase in composition ratio is found to some decrease the internal bonding strength. This is due to the higher fiber content leads to weak the internal bonding in between the fiber and the pet. It also another notable observation is that at higher range of alkali concentration, higher treatment time always resulted in the internal bonding strength of composite.

This phenomenon is further supported by a fact that composition ratio is the most significant process variable that affect the internal bonding strength of composite as indicated by highest F value in the ANOVA .

The RSM was used to optimize the conditions of process parameters for production and to understand the interaction of the factors affecting the internal bonding strength of composite. Figure 4.4, 4.5, 4.6 and 4.7 show surface plots between the independent and dependent variables for different fixed parameters.

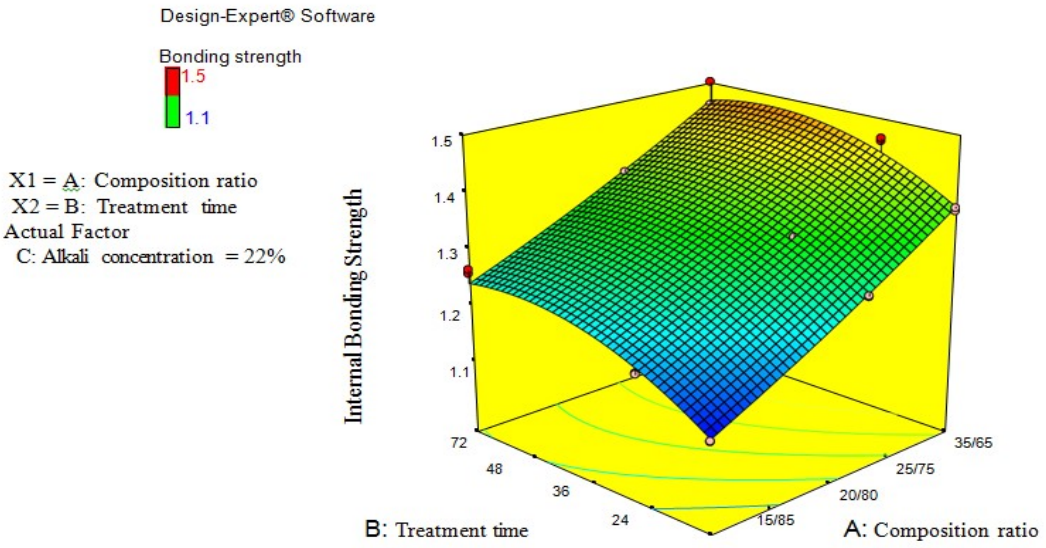


Figure 4.4. Surface plot of the interaction effect of B & A versus internal bonding strength

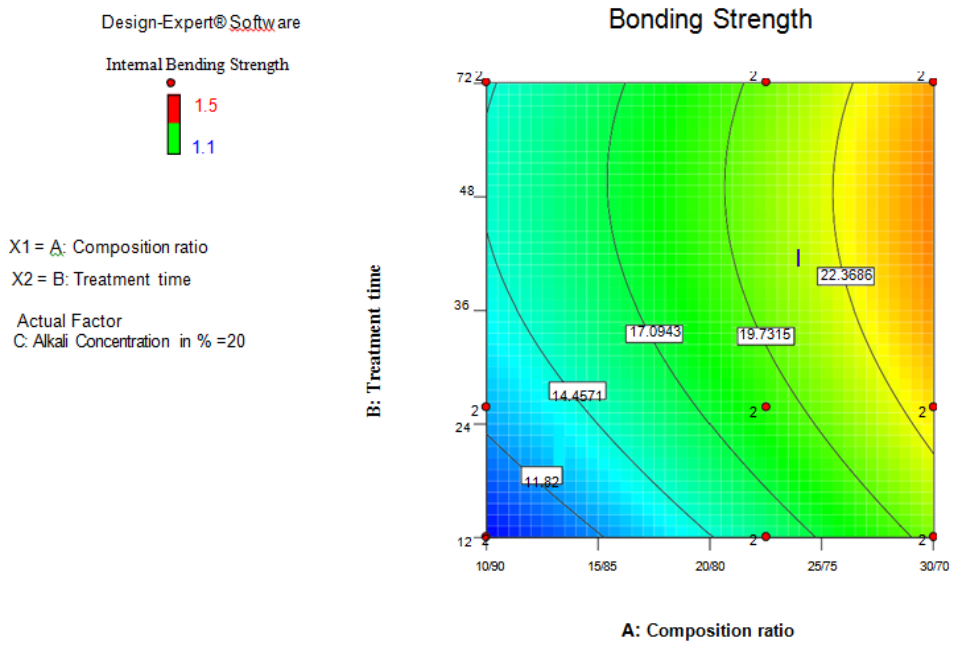


Figure 4.5. Contour plot of the interaction effect of B & A versus the internal bonding strength

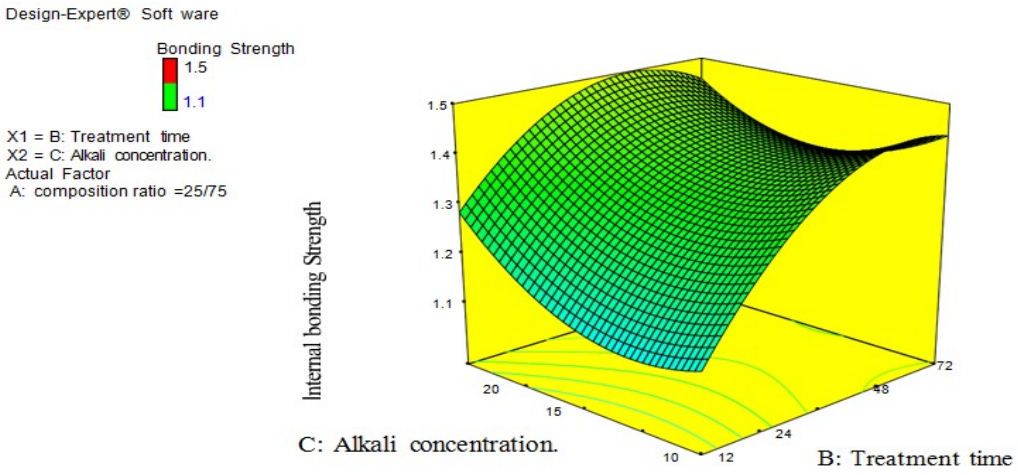


Figure 4.6 Surface plot of the interaction effect of C and B versus the internal bonding strength

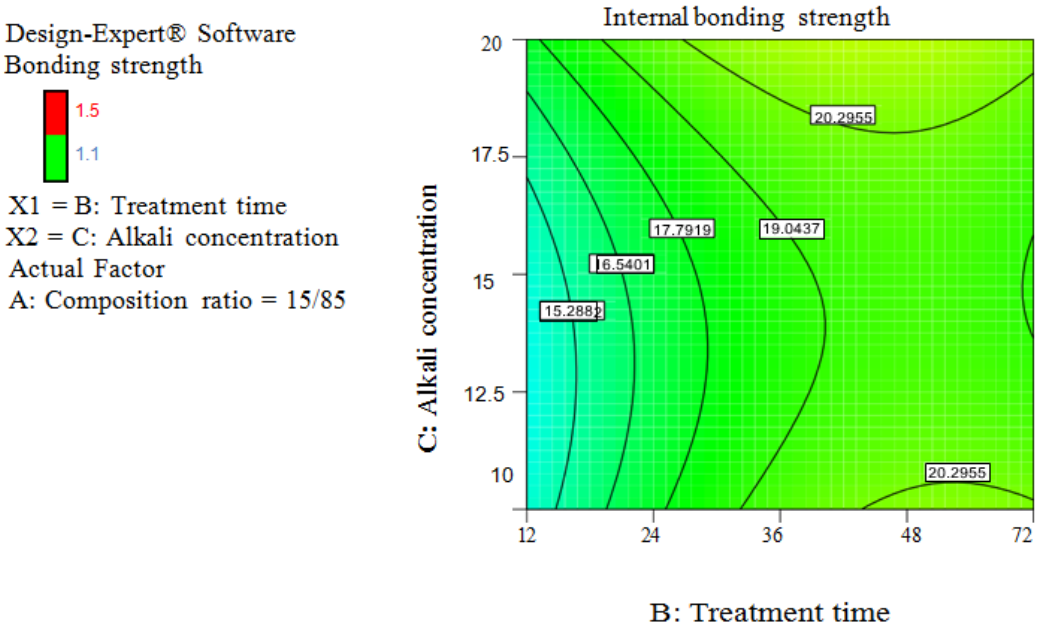


Figure 4.7 Contour plot of the interaction effect of C and B versus the internal bonding strength

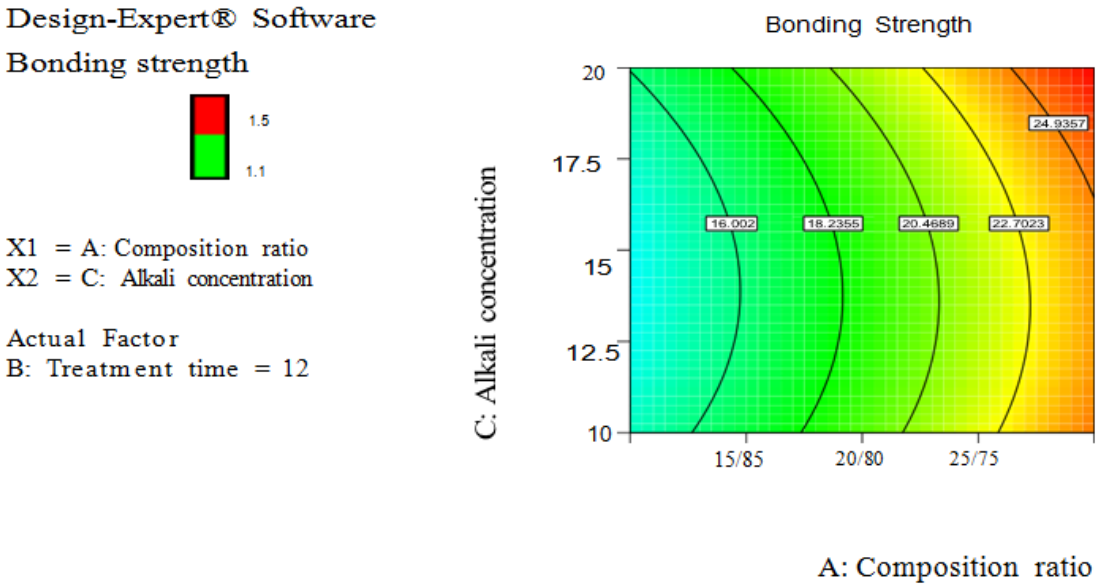


Figure 4.8. Contour plot of the interaction effect of C and A versus the internal bonding strength

The surface and contour plots in figure 4.7 and 4.8 respectively, shows that the interaction between treatment time and composition ratio on the internal bonding strength. As it can easily see from the two plots in the above, the interaction effect of two process variables affects the internal bonding strength positively at higher values. The reason behind this is there are more lignin and polluters removed from the fiber surface without degrading the fiber. The increased properties are also caused by the increasing of interfacial bonding between the fiber and the polymer matrix due to the effect of cellulose fiber purification (Dikuncoro diharo 2017).

4.4. Statistical Analysis of the Experimental Result

In this study Statistical experimental design techniques were used to determine the effects of concentration of alkali, treatment time of fiber and recycled pet to sisal fiber composition ratio in the mechanical properties of the composite. A total of 27 experiments were conducted for optimization of process parameters of sisal recycled pet composite and the effect of each factor was analyzed by taking minimum and maximum values from operating conditions which has significance influence on the physical properties of the composite. The internal bonding strength values obtained from the experiments were used as response parameters for optimization.

Run No.	Factor A.	Factor B.	Factor C.	Response
				Internal Bonding N/M^2
1	0	-1	+1	1.16
2	-1	0	+1	1.31
3	-1	+1	+1	1.30
4	-1	-1	+1	1.25
5	+1	-1	-1	1.26
6	+1	-1	0	1.20
7	-1	+1	-1	1.33
8	0	0	+1	1.27
9	+1	-1	+1	1.34
10	0	+1	0	1.23
11	0	+1	+1	1.29
12	+1	0	-1	1.22
13	0	0	-1	1.21
14	0	-1	-1	1.19
15	-1	-1	-1	1.17
16	+1	+1	+1	1.31
17	0	+1	-1	1.25
18	+1	0	+1	1.35
19	+1	+1	0	1.36
20	+1	0	0	1.28
21	+1	+1	-1	1.32
22	0	0	0	1.25
23	-1	-1	0	1.18
24	-1	+1	0	1.34
25	0	-1	0	1.24
26	-1	0	-1	1.33
27	-1	0	0	1.24
" +1'' represents high level of variable and " - 1'' represents low level of variable and and "0" represents the medium level of variance				
A - Concentration of alkali in % (10%,15% and 20%), B- Treatment time in Hr(24Hr,48Hr and 72 Hr), C – sisal –recycled pet composition ratio in % (15/85,24/75 and 35/65)				

Table 4.3. Experimental result of internal bonding of each run

The resulting data, table 4.1, were analyzed using design expert7 software to determine the effect of Concentration of alkali, Treatment time, and sisal –recycled pet composition ratio on the physical properties of the composite. The dependent variable used as a response parameter was the internal bonding strength of the composite. All experiments were carried out in a randomized

order to minimize the effect of unexpected variability in the observed response due to extraneous factors. Table 4.2 summarizes the design expert of the experiment.

Study Type	**Factorial**
Initial design	3level factorial
Center point	0
Design model	2F
Runs	27
Blocks	No blocks

Table 4.4. Summary of the design expert

Source	Sum of square	Df	Mean square	F-value	P-value pro>f	Remark
Model	0.024	18	2.678	3.84	<0.0083	significant
A- Concentration of Alkali	8.011	1	8.011	1.15	<0.0471	
B- Treatment time	0.012	1	0.012	17.76	<0.0462	
C- Composition ratio	1.974	1	1.974	2.83	<0.0109	
A^2	5.796	1	5.796	8.30	<0.0104	
B^2	3.717	1	3.717	0.53	0.4756	
C^2	3.388	1	3.388	0.49	0.4955	
AB	1.156	1	1.156	1.65	0.2155	
AC	7.467	1	7.467	1.07	0.3156	
BC	5.159	1	5.159	0.74	0.4020	
Residual	0.012	8	6.983			
Lack of fit	8	8	0.33	2.50	<0.0162	significant
Pure error	8.5	26	0.33			

Table 4.5. Analysis of variance (ANOVA)

The Values of "Prob> F" less than 0.0500 indicate model terms are highly significant. In this case A, B, and A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

This shows that the concentration of alkali, treatment time, Composition ratio and the square of the concentration of alkali affect the internal bonding strength much significantly. Thus, from these statistical tests, it was found that the model is adequate for predicting the internal bonding strength of composite material within the range of variables studied.

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 2.50 implies the Lack of Fit is not significant relative to the pure error. There is only 1.62% chance that a “Lack of fit F-value” this large could occur due to noise. Significant lack of fit is bad because we want the model to fit.

The multiple regression coefficients are obtained by employing a least square technique to predict a quadratic polynomial model for the composite (Table 4.6). The actual internal bonding strength of composite at different process parameters was calculated. The model was tested for adequacy by analysis of variance.

Standard order	Actual value	Predicted value	Residual	Leverage	Studentized residual	Run order
1	0.81	0.85	0.040	0.259	1.748	25
2	0.74	0.75	7.717	0.343	0.360	18
3	0.76	0.78	0.014	0.343	0.664	2
4	0.83	0.82	0.011	0.259	0.501	13
5	0.78	0.77	6.916	0.259	0.304	20
6	0.77	0.76	0.013	0.509	0.718	3
7	0.75	0.75	3.879	0.509	0.210	7
8	0.85	0.83	0.021	0.509	1.160	15
9	0.83	0.80	0.035	0.343	1.619	6

10	0.80	0.81	0.012	0.259	0.531	22
11	0.76	0.77	0.012	0.509	0.674	21
12	0.78	0.78	7.083	0.343	0.331	11
13	0.75	0.76	0.013	0.343	0.617	24
14	0.79	0.79	6.696	0.259	0.294	8
15	0.81	0.79	0.019	0.259	0.849	10
16	0.82	0.79	0.034	0.343	1.610	12
17	0.85	0.83	0.016	0.343	0.740	23
18	0.75	0.78	0.031	0.343	1.441	26
19	0.80	0.81	0.015	0.509	0.808	4
20	0.74	0.77	0.030	0.343	1.422	19
21	0.79	0.82	0.022	0.509	1.210	5
22	0.80	0.79	9.889	0.343	0.462	17
23	0.84	0.86	0.015	0.343	0.718	14
24	0.81	0.79	0.019	0.259	0.826	27
25	0.86	0.82	0.040	0.343	1.886	1
26	0.75	0.77	0.020	0.509	1.077	9
27	0.76	0.85	0.017	0.509	0.913	16

Table 4.6. Experimental and predicted values of internal bonding strength of composite

4.5. Development of regression model equation

The model equation that correlates the response (internal bonding strength) of the composite materials in terms of coded value after excluding the insignificant terms was given in below. The predicted model for percentage of internal bonding strength (IB) in terms of coded and actual factors is given below.

4.6. Final Equation in Terms of Coded Factors:

1.0/ (Internal bonding)(N/mm^2)

=+0.81- 6.671E-003 * A-0.026 * B-0.010 * C-0.031 * A2+7.871E-003 * B2-7.515E-003 * C2+9.813E-003 * A * B-7.888E-003 * A * C+6.557E-003 * B * C

4.7. Final Equation in Terms of Actual Factors:

1.0/ (internal bonding) (N/mm^2)

= +0.81209- 6.67130E-003 * A - 0.026248 *B 0.010473 * C - 0.031080 * A2 +7.87089E-003 * B2 -7.51482E-003 * C2+9.81294E-003 * A * B -7.88832E-003 * A * C+6.55650E-003 * B * C

Where,

A= Concentration of Alkali

B=Treatment time

C= Composition ratio

4.8. Model adequacy check

A line of unit slope, i.e. line of perfect fit with points corresponding to zero error between predicted values and actual values are also shown in figure 4.5. This plot therefore, shows the performance of the correlation in an obvious way. The results in figure 4.5 demonstrated that the regression model equation provided a very accurate description of the experimental data, in which all points are very close to the line of perfect fit. This result indicates that it was successful in capturing the correlation between the three composite production process variables to the internal bonding strength.

Based on a 95% confidence level. F-value is a test for comparing model variance with residual (error) variance. If the variances are close to the same, the ratio will be close to one and it is likely that any of the factors have a significant effect on the response with the P-value less than 0.05.

It is calculated by model mean square divided by residual mean square. Here the model F-value of 3.84 implies the model is significant.

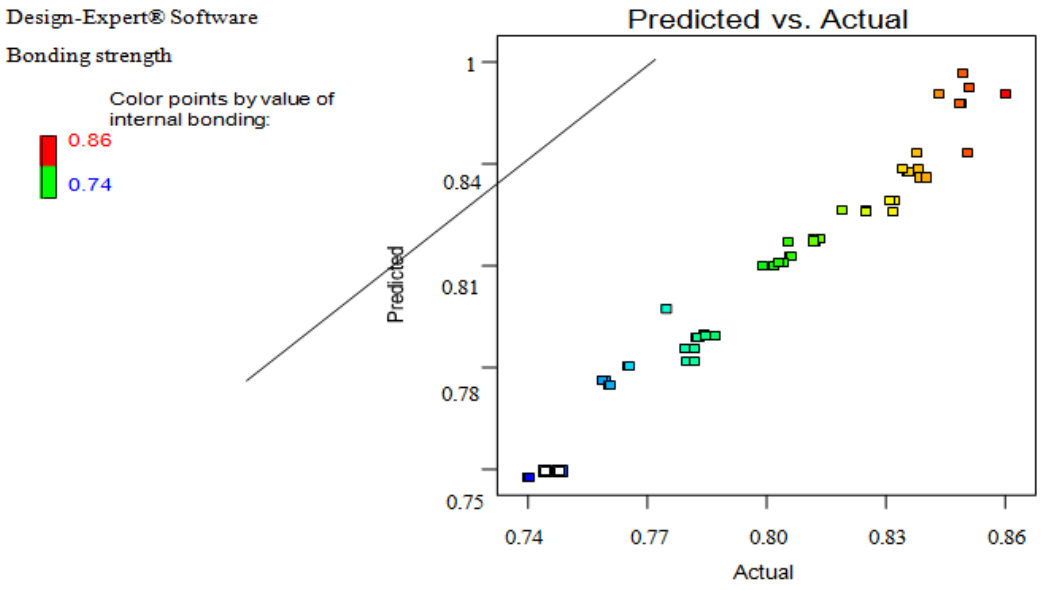


Figure 4.5 Predicted versus actual internal bonding strength of composite

4.9. Determination of density of bio composite

Run No.	Mass(gram)	Volume(cm^3)	Density(g/cm^3)
Sample	55.5	90.25	0.615

Table 4.7. Density of composite material

The values of bulk density measured for sisal fiber pet recycled bio composite made by0.95cm X0.95cm X1cm iron mold was calculated as 0.615g/cm^3. The composite materials and some partion borads were classified as medium density boards, which refers to board with bulk density between 0.6 and 0.8 g/cm^3 (Técnicas, 2006).

The average value of bulk density for industrial panels found by Mendes RF M. L, 2012 were 0.683g/cm^3 for Pinus, 0.655g/cm^3 for Eucalyptus and 0.699g/cm^3 for sugarcane bagasse. Despite in the lack of significant difference, the sisal fiber-pet recycled composite had a lower density compared with Pinusand Eucalyptus. So, according to a results obtained from the experiment,

there is no significant difference in density among natural fiber reinforced composite (Técnicas, 2006).

4.10. Optimization of the parameters in the mechanical properties of bio composite

The individual and interaction effects of process parameters have shown that these parameters have a positive effect at their higher values on the internal bonding strength. But the effects have to be optimized in order to have a better composite product at optimum condition. The DOE software suggests the optimum values for the three process variables.

These are composition ratio at 25/75 in %, treatment time at 72hr. and alkali concentration 20%, an optimum value of 1.36 N/mm^2 internal bonding strength was obtained. The experiment was conducted the suggested values to verify optimum values and a good approximation of the predicted optimum value in internal bonding strength was obtained, which is 1.4 N/mm^2 For wooden made partition boards and ceilings made from natural fiber reinforced composite (Técnicas,2006).

The ANOVA output shows that the composite production process is highly and significantly affected by the composition ratio, treatment time, alkali concentration and their interactions.

CHAPTER FIVE

6. Conclusion and recommendation

The sisal fiber was extracted manually from Ethiopian Highland sisal plant, and then the alkaline treatment was carried out. Next to that, sisal fiber reinforced pet composite was manufactured and its mechanical performance such as the internal bonding is determined using laboratory experiment. All the numerous experimental test results gathered an important information about sisal fiber reinforced pet composite. Moreover such tests constitute fundamental confirmation of the reliability of the material and of its usage in ceiling application. During the course of the study about the internal bonding of the composite made from sisal fiber and recycled pet, few points can be concluded as follows:

- A polymer matrix composite contains the sisal fiber as reinforcement was successfully fabricated.
- Different mechanical properties were determined from different Sisal to recycled pet percentage
- 20% NaOH alkali treatment is an effective chemical treatment in removing the lignocellulose materials and improving interfacial adhesion between sisal fibers and the pet. After treatment, sisal fiber are bonded better with matrix pet and the pet is able to penetrate into sisal fiber core, i.e. the interfacial adhesion has been greatly improved after 20% NaOH solution treatment in this research .
- From the internal Bonding Experimental test results, it is found that 25/75 %treated sisal fiber has better internal bonding properties.
- From the internal Bonding Experimental test results, it is found that better internal bonding properties obtained from the fiber that treated with 72hr.
- The treated sisal fiber/pet composite system has a high mechanical performance especially the internal bonding strength. So this research muscularly gives confidence to utilize the advantages offered by renewable resources and its application in some aspects of celling application.

6.2. Future work and recommendation

The following study can throw more light into the application of SFRPC for house celling application. The possibility of finding the appropriate SFRPC for house celling application depends on the accurate knowledge of the fabrication and proportion of sisal fiber and the matrix material. The following studies could be performed to analyses more details in this topic.

- Characterization can be done by using different types Natural fibers to improve the strength.
- Characterization of the fibers can be done by the using different fabrication techniques on SFREC's
- Characterization of the fibers can be done by the using Mat type and also with Fine powder type.
- Further we can make use of Advanced or Bio-Matrix materials (high density polyethylene or propylene etc.).
- Testing different sisal plant species to obtain the fiber with remarkable characteristics.
- SEM and Finite Element Analysis can be carried out.
- Testing like Fatigue test, shear test, Impact test, Moisture content test and thermal test must be done intensively.
- Design of natural fibers extraction processing machine must be done.

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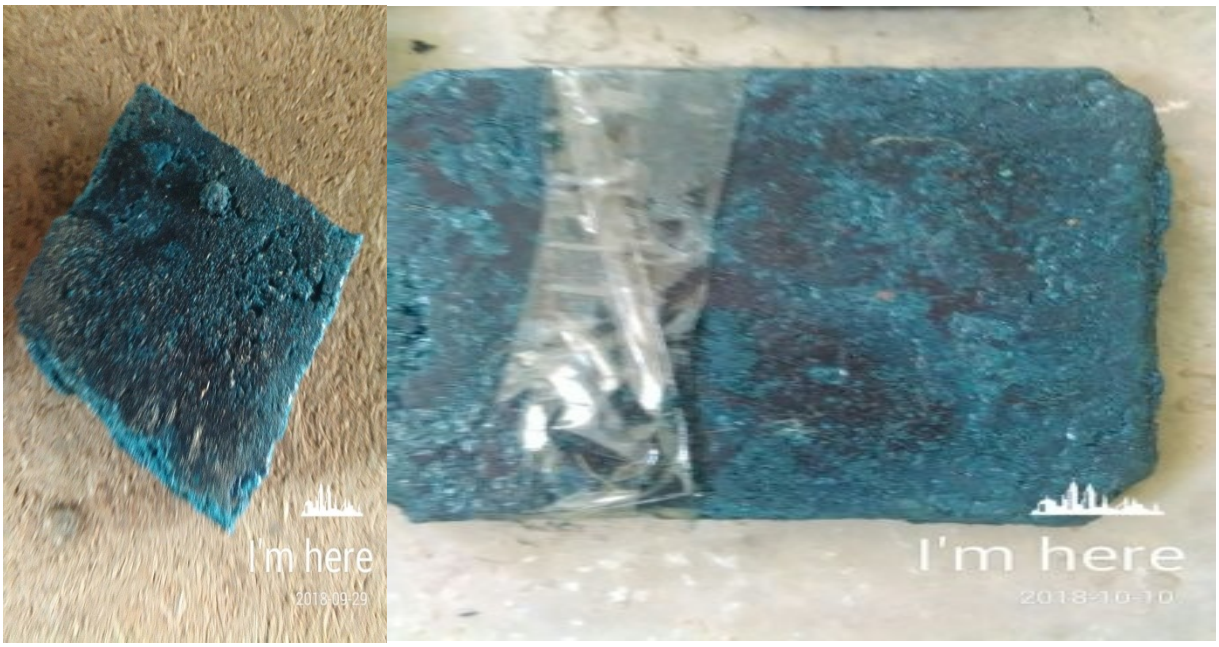
Appendix

SAMPLE PREPARATIONS

FIBER EXTRACTION AND CHEMICAL TREATMENT OF FIBER

MIXING THE RAW MATERIALS AND MOLDING



MOLDED SAMPLES OF BIOCOMPOSITES

FLXURAL TESTING METHODES

INTERNAL BONDING TESTING