EFFECT OF DIFFERENT FILLER TREATMENTS ON THE PROPERTIES OF FLEXIBLE POLYURETHANE FOAM COMPOSITES

BY

ABUBAKAR MUSTAPHA

(2006/24157EH)

A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING,
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA
NIGER STATE, NIGERIA

IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF
BACHELOR OF ENGINEERING (B.ENG) DEGREE IN CHEMICAL ENGINEERING

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CERTIFICATION

This is to certify that this project report entitled 'EFFECT OF DIFFERENT FILLER TREATMENTS ON THE PROPERTIES OF FLEXIBLE POLYURETHANE FOAM COMPOSITES' by Abubakar Mustapha meets the requirements for the partial fulfillment of the award of Bachelor of Engineering (B.Eng) degree in Chemical Engineering, Federal University of Technology, Minna.

Aboje A. Audu
Project Supervisor
Name and Signature

24/10/11
Date

Head of Department
Name and Signature

Date

External Examiner
Name and Signature

23/02/2012
Date

DECLARATION

I declare that the work in the project report (thesis) entitled **EFFECT OF FILLERS ON THE PROPERTIES OF FLEXIBLE POLYURETHANE FOAM** has been performed by me under the supervision of A. A. Aboje. No part of this project report was presented for another degree or diploma elsewhere at any institution to the best of my knowledge.

Abubakar Mustapha		24/10/11
Student Name	Signature	Date

DEDICATION

This project is dedicated to almighty Allah (S.W.T) the most compassionate and the most merciful, the one who in his infinite mercy as seen me through the course of carrying out this research work and throughout my years in my undergraduate programme. I also dedicate this document to my beloved, caring and wonderful parents Alh Abubakar Mohd and Haj Fatima Abubakar, to my brothers and sisters, my good friends and my dearest Habiba Abubakar; they all stood by me throughout the programme. I love you all and May almighty Allah reward all your efforts abundantly.

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Special recognition goes to National Research Institute for Chemical Technology (NRICT) Zaria and its workers, both for their assistance over this research. In particular, I'd like to acknowledge Mr. Obina for his many helpful discussions on the complexities of foam. Finally, I need to thank them and everyone else in the laboratory.

Vita foam Nigeria Plc. and its workers also deserve a word of thanks for their support. I enjoyed the opportunity to be able to see how foam production works, and I greatly appreciate your courtesy and many helpful discussions. In particular, I'd like to recognize the production manager, Alh Bello for working so closely with me in the characterization effort.

Mr. Darlington E. Onyeama, who helps in looking for some of the chemicals, he has really done me a big favour and I thank him for that.

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Finally, and I put them last only to emphasize them most, I want to express my love and thanks to my whole family. To my perennially patient parents, I thank you for your moral support, spiritually, financially and care through all the years. My beloved father, Alh Abubakar Mohd he is my mentor; how I wish I can ever be like him. My one and only mum, haj Fatima Abubakar, she is the best of all mothers; I know that I owe them much of what I am and what I will become. You will always be in my memory, heart and soul. Also I thank my brothers and sisters, for their friendship and support for the past several years. I love you all and may Allah (S.W.T) reward you abundantly.

ABSTRACT

It has become apparent that what is of great interest to researchers is the improvement in the mechanical properties of polymers, and also to produce at optimum cost. It has therefore become imperative to consider inorganic substance suitable as reinforcement agent in polyurethane foam. In this study, materials such as calcite ($CaCO_3$) and carbon black (Charcoal) at size particle 500 μm and 350 μm were used to improve the mechanical properties of flexible polyurethane foams. Loading was varied from the range of 5 % - 30 % in to the foam formulation. Properties of these composites were studied as functions of filler contents in the matrix polyurethane foam. These properties include the density, compression set, Hardness index, tensile strength, elongation, water absorption and cost analysis. Based on the obtained result, the study indicates that 20 % of the foam sample filled with carbon black has no loss in its thickness after been compressed. The addition of filler increases the density of the foam but reduces the tensile strength and elongation at break with an increasing amount of filler in the polyurethane matrix for the entire foam sample. Cost analysis of the production revealed that cost of producing flexible polyurethane has been optimized with addition of filler composition in the foam formulation.

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Chapter One

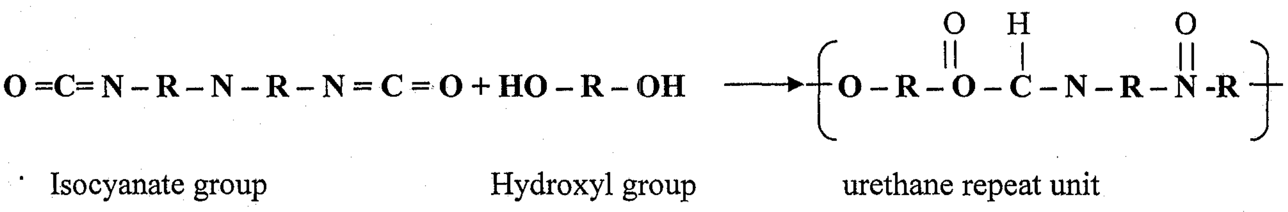
1.0 INTRODUCTION

1.1 Foam

Polyurethane flexible foams exist in many forms and have been increasingly used over the last thirty years. Flexible foam is applied in durable products with long lifetimes, usually more than ten years. The products are as diverse as comfortable foam cushioning for furniture or light weight seats to crash padding in cars. Polyurethane flexible foam products have become a valuable part of our modern life through improving comfort, increasing safety and reducing fuel consumption. In addition, they are recyclable, (http//www.isopa.org).

Polyurethane foam usually consists of the chemicals; 50 % polyol, 40 % polyisocyanate and 10 % water and other chemical. Polyisocyanates and polyols are liquid polymer that when combined with water produces an exothermic (heat generating) reaction forming the polyurethane. The molecular structure, amount and reaction temperature of each additives determine the characteristic and subsequent use of the foam, for instant, a switch in the blowing agent may requires an increase in water and a switch in surfactants to maintain optimum bubbles sizes and the formation rate during foaming. The density of the foam is determined by the amount of blowing, the stiffness and the hardness of the polyurethane which can also be toiled by changing the level of polyol in the chemical formulation, (http//www.crosslink.com).

Flexible polyurethane foam can be produce in different varieties depending on the type of monomer been used. Other additives can be used to improve their fire performances, stability in difficult chemical environment, colour and properties of the polyurethane products, (http//www.wiki.com).



$$O{=}C{=}N-R-N-R-N=C=O+HO-R-OH \longrightarrow \left[O-R-\overset{\overset{O}{\|}}{O}-\overset{\overset{H}{|}}{C}-N-R-\overset{\overset{O}{\|}}{N}-R\right]$$

Figure 1.1 Structure of PU unit

The first step of this reaction result in the chemical linking of two molecules leaving a reactive alcohol on one side and reactive isocyanate (NCO) on the other side. These groups react further with other monomer to form a longer molecule. These are a rapid process which yields high molecules weight material even at room temperature, (Http//www.crosslink.com).

Fillers are introduced into foam formulations to increase foam density, load bearing ability and sound attenuation. When introduced and processed accurately, overall cost of production can also be significantly reduced.

In this research study, filler materials such as calcite ($CaCO_3$) and carbon black (charcoal) is used to influence the properties of flexible polyurethane foams. Properties of these composites were studied as function of filler contents in the matrix polyurethane foam.

1.2 Aims and Objectives of the Research Study

1. To produce polyurethane foam.
2. To study the effect of Fillers on the products.
3. To test for the properties of the product obtained seeing that is in conformity with standard.

1.3 Scope of Study

The scope of this research will be limited to the followings

i. Production of flexible polyurethane foam using different filler treatment.
ii. To carry out the quality control tests of the products obtained.
iii. Cost optimization of production.

1.4 Statement of the Problem

The non-availability of some of these raw materials and high cost of materials due to importation, contributing to high production cost.

1.5 Justification of the Research

Fillers are known to have positive and desirable effects on the polyurethane foam produced, this work attempts to investigate and compare the positive and negative effects of the fillers (Calcium carbonate and Charcoal) used on foam properties and thus look for a way to optimize the positive effects.

Cost of production of foam will be reduced by incorporating fillers in the formulation, since the polyurethane foams produced will be compared by subjecting them to various physical analysis and tests, the work will also help determine which of the filler used produces better foam based on the physical properties and cost of foam. These results will provide foam companies a comprehensive view of which of the filler is more applicable for foam production in Nigeria.

Chapter Two

2.0 LITERATURE REVIEW

Polyurethane are single most versatile family of linear polymer that have a molecular back bone containing carbamate group [-$NHCO_2$]. Polyurethane can also be called polycarbamates, which belongs to a large class of compound called polymer. Polymers are macromolecule made up of smaller repeating unit known as monomers. The IUPAC abbreviation of polyurethane is PUR but commonly abbreviated as PU. They are called polyurethane because in their backbone they have urethane linkages, (Medinat R. 2010).

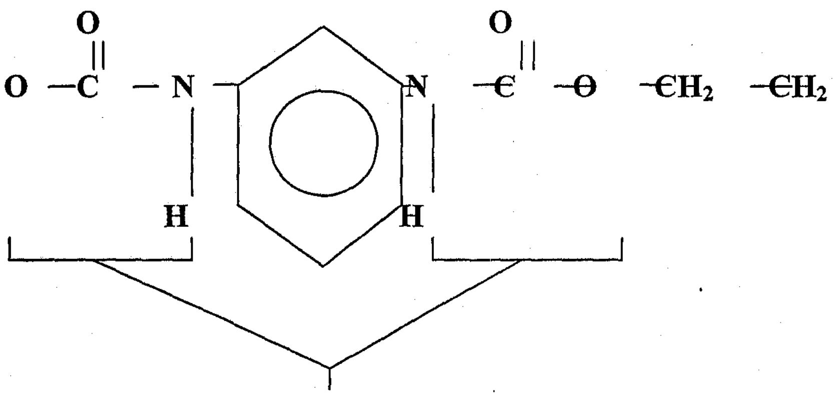


Figure 2.1 Urethane linkages in polyurethane

Polyurethane polymer are formed through step growth polymerization by reacting monomers containing at least two isocyanate functional groups with another monomers containing at least two hydroxyl alcohol group in the presence of a catalyst, (http//www.wikipedia.polyurethane).

Furthermore, Polyurethane is categorized as flexible, rigid form, fibers, elastomeric (elastic polymer), surface coating and adhesives. Elastomeric polyurethane has elastic properties while maintaining some rigidity, such as in the wheels of a dolly that absorb shocks but do not compress too much. Polyurethane can also be extremely flexible when used as a form insulator in construction or a foam cushion in upholstery. It can deformed over and over and still maintain its original shape in other words, flexible polyurethane has a structural memory. Other polyurethane include thermoplastic, which resemble other kind of plastic, metal or fiber glass. Thermoplastic polyurethane is rigid and smooth with sealed surface impermeable to water, these are used when strength, durability is important such as in seat at an airport terminal or packaging create on a truck. Some thermoplastics polymers are difficult to recycle, but they can be reused, (Medinat R. 2010).

Polyurethane are mostly used in high resiliency flexible form seating, rigid form insulation panels, gasket, durable elastomeric wheel and tires, high performance adhesives, surface coating and sealant, carpet underlay, (http//www.wikipedia.org).

2.1 Historical Development

In 500BC, rubber was made by Mayans and Aztecs, they were made originally from natural latex, a white sap produce from rubber tree known as "Hevea Brasiliensis", (Medinat R. 2010).

Simple urethanes were prepared by Wartz in the middle of nineteenth century. These compounds were obtained by the reaction of alkanol such as ethanol and isocyanate such as phenyl isocyanate. It was found that this reaction was cartelized by tertiary amines and salt of heavy metal, (Medinat R. 2010).

Ottobayer and his coworker in 1937 at the laboratory of I.G. Farben in germany review this reaction. They discovered that using poly addition principle to produce polyurethane from liquid disocyanate and liquid polyether polyester diol seemed to point to special opportunities when compared to the already existing plastic that were made by polymerizing. The polyurethane obtained from these reactions is used as adhesives, coating, fibers, elastomers, cellular product and molded article, (http://en.wikipedia.org).

At early 20th century, various types of specially manufactured foam are used. Foam in this case meaning "bubby liquid" also produced as an often unwanted by-product in the manufacture of various substances. Since then, foam has been discovered and continuously modified by the nature of the chemicals used, (http://en.wikipedia.org).

At late 1950's, moldable polyurethane were produced such as mattresses and pillows. It was not until 1952 that polyisocyanate became commercially available. Foam production began in 1954 based on toluene disocyanate (TDI) and polyester polyols. The invention of these foams initially was called imitation Swiss cheese by the inventor. These materials were also used to produce rigid foams, gum, rubber and elastomer, (Medinat R. 2010).

In 1956, the first commercially available polyether Polyol, Poly [Tetramethylene ether] glycol was introduced by Du Pont, these is a process of polymerizing Tetrahydrofuran, a less expensive Polalkylene glycol were introduced with a base and Dow chemical. In the following year, 1957, polyether Polyols offered technical and commercial advantages such as low cost, ease of handling and a better hydrolytic stability over polyester polyols and quickly replaced them in the manufacture of polyurethane goods, (http//www.wikipedia.org).

In 1960 more than 45,000 metric tons of flexible polyurethane foams were produced. As the decade progressed, the availability of chlorofluoroalkane blowing agents, inexpensive polyether polyols, and methylene diphenyl diisocyanate (MDI) heralded the development and use of polyurethane rigid foams as high performance insulation materials. Rigid foams based on polymeric MDI (PMDI) offered better thermal stability and combustion characteristics than those based on TDI. In 1967, urethane modified polyisocyanurate rigid foams were introduced, offering even better thermal stability and flammability resistance compared to low-density insulation products. During the 1960s, automotive interior safety components such as instrument and door panels were produced by back-filling thermoplastic skins with semi-rigid foam, (http//wikipedia.org).

In early 1980's, polyurethane foam is often made by adding small amount of volatile materials known as blowing agent such as trichlorofluoromathane and other halo alkanes such as hydrochlorofluorocarbon1, 1-dichloro-1-fluoro ethane to the reaction mixture. Also, water-blown microcellular flexible foam was used to mold gasket for panel and radial seal air filters in the automotive industry. Since then, increasing energy price and the desire elimination PVC plastic from automotive application have greatly increased market share. Highly filled polyurethane elastomers and more recently unfilled polyurethane foams are now used in high-temperature oil filter application, (http//www.wikipedia.org).

Building on existing polyurethane spray coating technology and polyetheramine chemistry, extensive development of two-component polyurea spray elastomers took place in the 1990s. Their fast reactivity and relative insensitivity to moisture make them useful coatings for large surface area projects, such as secondary containment, manhole and tunnel coatings, and tank liners. Excellent adhesion to concrete and steel is obtained with the proper surface treatment and primer. During the same period, new two-component polyurethane and hybrid polyurethane-polyurea elastomer technology was used to enter the marketplace of spray-in-place load bed liners. This technique for coating creates a durable, abrasion resistant composite with the metal substrate, and eliminates corrosion and brittleness associated with drop-in thermoplastic bed liners, (http//www.wikipedia.org).

In 2004, the potential for polyol derived from vegetable oil to replace petrochemical-based polyols was developed, due to the rising cost of petrochemical feed stocks and also due to an enhanced public desire for environmentally friendly green product made by ford motor company, (http//www.wikpedia.org).

2.2 Definition of Foam

Foam is a colloidal suspension of a gas in a liquid created by the agitation. Foams are cushioning material formed by the creation of gas bubble in a base material such as natural rubber, synthetic rubber or other elastomeric material. They can be flexible or rigid depending on the molecules. Low density foams are dispersion of gas in relatively small volumes of solid having for example a density less than 0.1g/cm^3. Medium foams, they have a density higher than 0.4g/cm^3 i.e contains small volumes of gas in the matrix. Foams are basically of two types which include; flexible foams having an open cell structure and can be produced in both high and low densities. Application include cushioning for furniture, automobile, mattresses and pillows e.t.c. Rigid foams are highly-crossed linked polymers with a closed cell structure that prevents gas movement. Their main application is as insulation for buildings, refrigerator, freezers and refrigerated transport vehicles, (Medinat R. 2010).

Polyurethane foam is a plastic material in which a proportion of solid phase is replaced by gas in the foam of numerous bubbles (cell). The gas may be in a continues phase to give an open cell material that will allow free movement of air between the foam cavities or it may be discontinuous i.e close cell material that will not allow free movement of air between the foam cavities, (Medinat R. 2010).

2.3 The Basic Chemistry of Polyurethanes

The first essential component of a polyurethane polymer is the Isocyanate. Molecules that contain two Isocyanate groups are called di-Isocyanate. These molecules are also referred to as monomers or monomer units. A pre-polymer is formed when the stoichiometric ratio is equal to 2:1. Important characteristics of Isocyanate are their molecular backbone, % NCO content, functionality, and viscosity, (http//en.Wikipedia.org).

The second essential component of a polyurethane polymer is the Polyol. Molecules that contain two hydroxyl groups are called diols, those with three hydroxyl groups are called triols, et cetera. Polyols are polymers in their own right. They are formed by based-catalyzed addition of propylene oxide (PO), ethylene oxide (EO) onto a hydroxyl or amine containing initiator, or by poly-esterification of a di-acid, e.g polyester polyols. The choice of initiator, extender, and molecular weight of the polyol greatly affect its physical state, and the physical properties of the polyurethane polymer. Important characteristics of polyols are their molecular backbone, initiator, molecular weight, % primary hydroxyl groups, functionality, and viscosity, (http//en.Wikipedia.org).

2.3.1 Reactions involve in production of polyurethane

2.3.1.1 *Polymerization reaction*

The polymerization reaction is catalyzed by tertiary amines, such as Dimethylethanolamine, and one of the most desirable attributes of polyurethanes is their ability to be turned into foam.

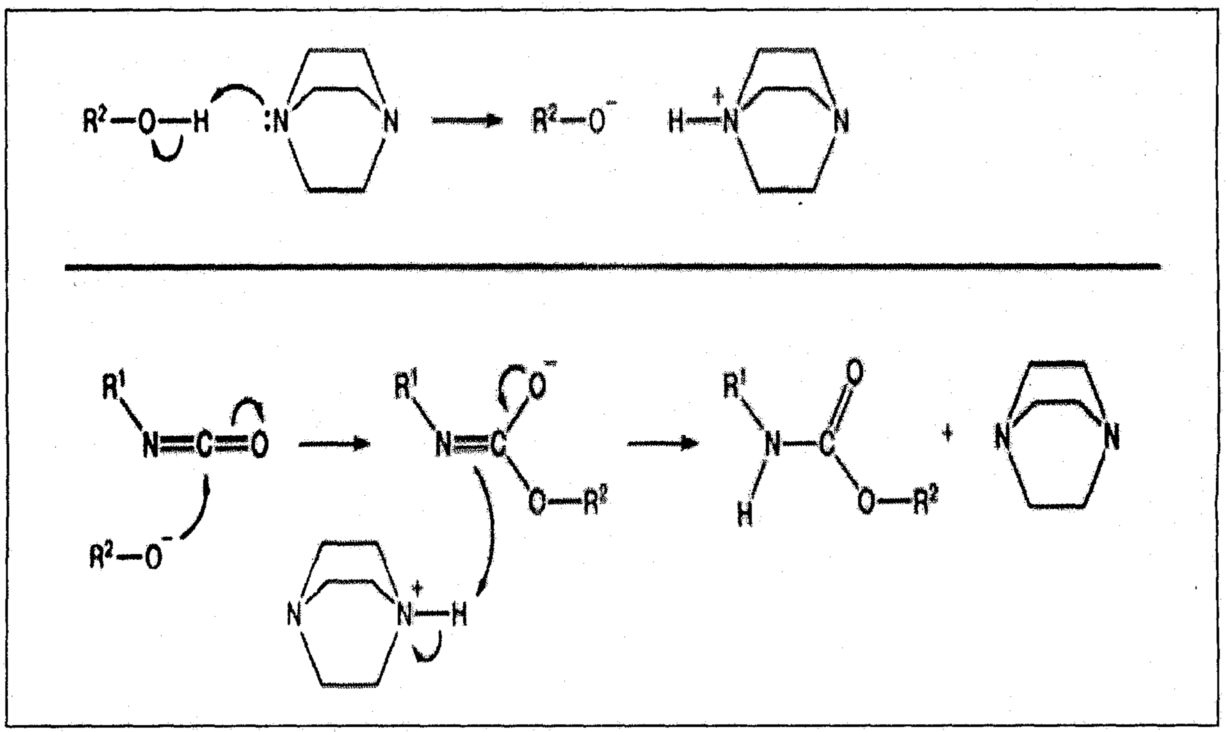
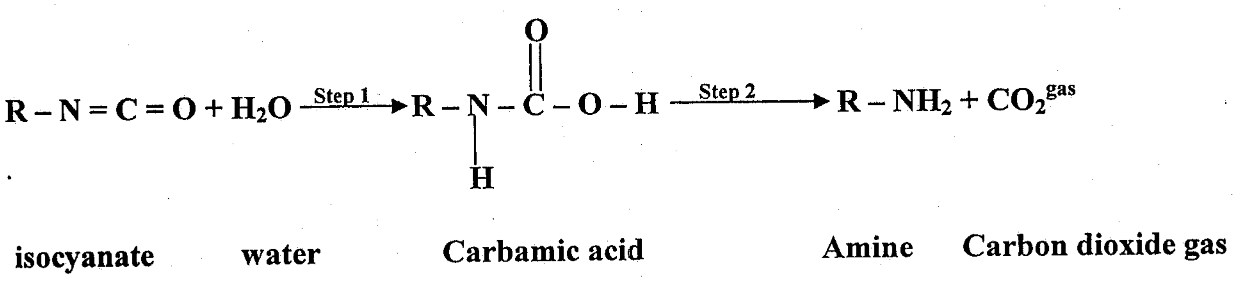


Figure 2.2: Polyurethane Reaction Mechanism Catalyzed by a Tertiary Amine

R^1 and R^2 groups may also contain isocyanate or isocyanate-reactive group when extended to poly functional reactants, this reaction provide a direct route to cross linked polymers. The hydrogen on the nitrogen atom of the urethane group is capable of reacting with additional isocyanate.

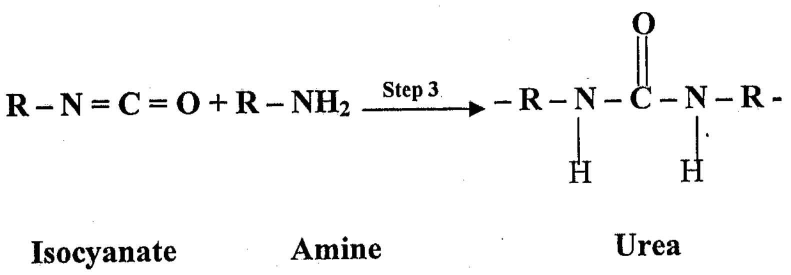
2.3.1.2 *Gas producing reaction*

Blowing agents such as water can be incorporated into the poly side or added as an auxiliary stream. Water reacts with the isocyanate to create carbon dioxide gas, which fills and expands cells created during the mixing process. The reaction is a three step process. A water molecule reacts with an isocyanate group to form a carbamic acid. Carbamic acids are unstable, and decompose forming carbon dioxide and an amine. The amine reacts with more isocyanate to give a substituted urea. Water has a very low molecular weight, so even though the weight percent of water may be small, the molar proportion of water may be high and considerable amounts of urea produced. The urea is not very soluble in the reaction mixture and tends to form separate "hard segment" phases consisting mostly of polyurea. The concentration and organization of these polyurea phases can have a significant impact on the properties of the polyurethane foam.



$$R-N=C=O+H_2O \xrightarrow{\text{Step 1}} R-\underset{H}{\underset{|}{N}}-\overset{O}{\overset{\|}{C}}-O-H \xrightarrow{\text{Step 2}} R-NH_2+CO_2^{gas}$$

Figure 2.3: Carbon dioxide gas formed by reacting water and isocyanate



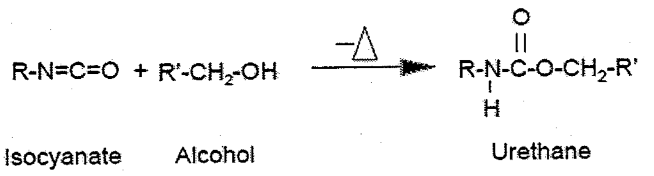
$$R-N=C=O+R-NH_2 \xrightarrow{\text{Step 3}} -R-\underset{H}{\underset{|}{N}}-\overset{O}{\overset{\|}{C}}-\underset{H}{\underset{|}{N}}-R-$$

Figure 2.4: Blow reaction

Since the polymerization reaction is exothermic, these blowing agents volatilize into a gas during the reaction process. They fill and expand the cellular polymer matrix, creating foam. It is important to know that the blowing gas does not create the cells of foam. Rather, foam cells are a result of blowing gas diffusing into bubbles that are nucleated or stirred into the system at the time of mixing, (http//www.wikipedia.org).

2.3.1.3 *Gelation reaction*

The polyurethane linkage is produced by the reaction of alcohol functionality with an Isocyanate group as shown in Figure 2.5. This addition reaction is exothermic with a heat of reaction of approximately 24 kcal/mol of urethane. The nature of the R and R' groups shown in Figure 2.5 can vary depending on the selection of components in the formulation. Generally, one of these components is multifunctional so that these reactions lead to a covalent network.



$$R\text{-}N\text{=}C\text{=}O + R'\text{-}CH_2\text{-}OH \xrightarrow{-\Delta} R\text{-}\underset{H}{\underset{|}{N}}\text{-}\overset{O}{\overset{\|}{C}}\text{-}O\text{-}CH_2\text{-}R'$$

Figure 2.5: The Gelation or Cross-linking Reaction

2.4 Components of Polyurethane Foam

For the manufacture of polyurethane polymers, two groups of at least Bi-functional substances are needed as reactants; compounds with Isocyanate groups, and compounds with active hydrogen atoms. The physical and chemical character, structure, and molecular size of these compounds influence the polymerization reaction as well as ease of processing and properties of the finished polyurethane. In addition, additive such as catalysts, surfactants, blowing agents, cross linkers, flame retardants, light stabilizers, and fillers are used to control and modify the reaction process and performance characteristics of the polymer.

Table 2.1: Typical Components and their Normal Usage in Flexible Polyurethane Foam Formulations.

Component	Weight Added [Parts Per Hundred Polyol]
Polyol	100
Water	1.5-7.5
Inorganic Fillers	0-150
Silicone Surfactant	0.5-2.5
Amine Catalyst	0.1-1.0
Tin Catalyst	0.0-0.5
Chain Extender	0-10
Cross Linker	0-5
Additive	Variable
Auxiliary Blowing Agent	0-35
Isocyanate	25-85

2.4.1 Polyol

A Polyol is generally an alcohol functionalized low molecular weight polymer but it can also contain additional Isocyanate-reactive groups. This very broad definition is required, because many types of Polyol structures are employed to achieve desired combinations of properties and processing requirements. It should be kept in mind that their specific composition is frequently tailored to meet very specific requirements.

The properties of the final foam are highly dependent on the composition of the Polyol, this compositions are:

- Functionality
- Hydroxyl number
- Equipment weight
- Primary hydroxyl content
- Cloud point
- Reactivity

The most important composition that should be noted which will be used in calculating the amount of your Isocyanate needed in the formulation is the hydroxyl number.

2.4.2 Isocyanate

Is an acid been represented by the general formula R – N = C – O where R is predominantly alkyl and aryl, stable Isocyanate in which N = C = O group is link to element such as Sulphur, Silicon, Phosphorus, Nitrogen or Hydrogen. Most member of this class of compound is liquid that are sensitive to hydrolysis and strong lachrymator.

The most reactive material required to produce polyurethane are Isocyanate, which are highly reactive alcohols. The most widely used Isocyanate employed in polyurethane production is Toluene-Di-Isocyanate (TDI) and polymeric Isocyanate (PMDI). The most popular used TDI is that which contain a mixture of two isomers, 2,4 and 2,6-Di-Isocyanate Toluene isomers consisting of 80 % of the 2,4-isomer and 20 % of the 2,6-isomer presented below

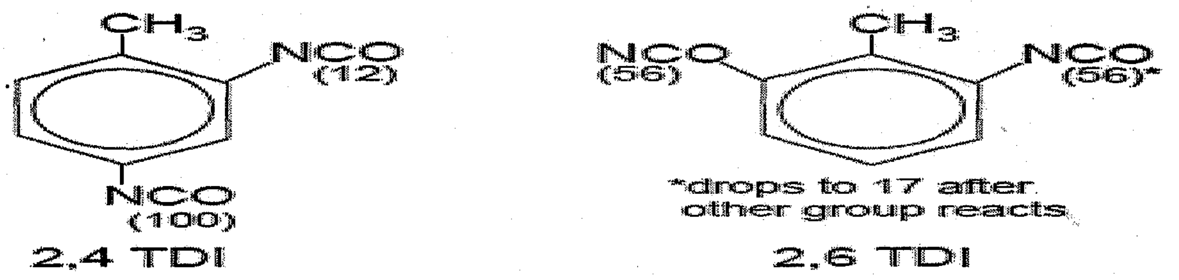


Figure 2.6: Isomers of Toluene-Di-Isocyanate (TDI)

Toluene-Di-Isocyanate is clearly colourless, low viscous liquid with a characteristic pungent smell. It is a toxic chemical and must be handled with care but is prone to freezing at temperature below 20 °c.

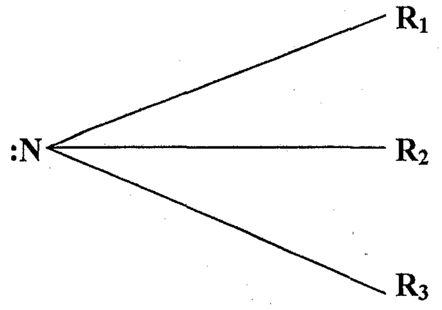
2.4.3 Activators

2.4.3.1 *Catalyst*

They are introduced to start up and maintain the reaction between Di-Isocyanate and water. Example of catalyst is Amine, this Amine primarily aids the blowing reaction and to lesser extent the gelling reaction. Amines are low viscous liquids which usually have an unpleasant fishy smell and are corrosive. The catalytic activity of Amine is due to the presence of a free election pair on the Nitrogen atom.

$$HO-CH_2-CH_2-\overset{H}{\overset{|}{N}}-CH_2-CH_2-OH$$

Figure 2.7: Structure of Dimethylethanolamine (DMEA)



Where :N = Free Electron

R = Polymeric end

Figure 2.8: Tertiary Amine Structure

2.4.3.2 *Stannous Octoate (Tin II Octoate)*

This promotes the polymer foaming or gelation reaction. It is a viscous liquid, light yellow to brown color with an unpleasant smell. The chemical needs special handling because it is easily hydrolyzed and oxidized in the presence of water and amines. When exposed to air, the water vapour in the surrounding air is enough to react with and render it important. Generally, stannous octoate is refers to as a cross-linking agent because the chemical strongly influence the cohesion and hardening of foam.

2.4.4 Surfactant or Stabilizer

This plays the following roles in the production of polyurethane foam:-

i. Acts as an emulsifier i.e brings together other incompatible ingredient.
ii. Assists the mixing of the components to form a homogeneous liquid.
iii. Lowers bulk surface tension.

iv. Prevent cell collapse during the rising stage and enhances formation of cells during the initial mixing stages.

An example of surfactant is Silicon oil which is a light colored and moderately viscous liquid. A minimum level of silicone oil is required in a given foam formulation to have well-structured foam.

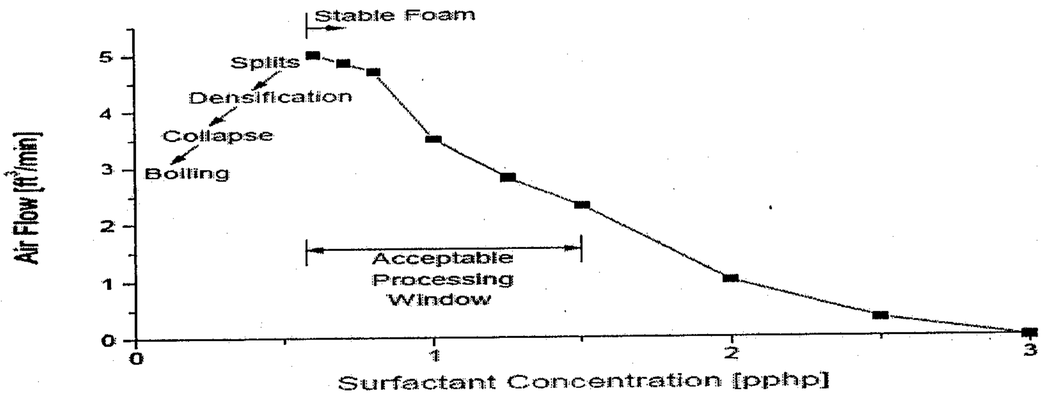


Figure 2.9: Effect of Concentration for a Typical Silicone Surfactant on Airflow

2.4.5 Additives

2.4.5.1 *Inorganic Fillers*

Fillers are introduced into foam formulations to increase foam density, load bearing ability and second attenuation. When expertly introduced and processed, overall cost of processing can also be significantly reduced. However fillers used must be free of moisture and impurities. Experience has shown that up to 30 parts per hundred parts of polyol can be used with little or no difficulty.

Certainly there are difficulties encountered when fillers are used. Some are:

- Due to their weight, the powder tends to settle out of the polyol mixture, and preventing this will result to constant agitation.
- At relative high filler concentration, the gelling reaction will be affected and this may call for re-balancing of the whole formulation.

2.4.5.2 *Colourants*

In order to achieve specific properties, it is sometimes necessary to include an additive. Some additives are used to make the product more appealing to consumers. For example colorants (e.g. metal oxides, carbon black, or azo/diazo dyes) may be added to conceal product yellowing or achieve a design specification. The most convenient colourants to use are those that are either soluble in water or polyol.

2.4.6 Auxiliary Blowing Agent and Water

These chemicals include Methylene chloride, Freon II. They are introduced into foam formulation to give additional blowing effects to the foaming reaction. They also make it possible to produce soft foams at all densities, which can be a chemical without increasing the water level.

The water use for polyurethane foam reacts with isocyanate to generate heat, carbon dioxide and polyurethane molecules and the volume of carbon dioxide is large enough to be the main blowing during the foam expansion. It also promotes flow ability in rigid foams; products are incorporated into polymers structure. The liquid quality of water introduced into the formulation must be carefully monitored as to prevent excessive heat generation.

2.5 List of polyurethane applications

Polyurethane products have many uses. Over three quarters of the global consumption of polyurethane products is in the form of foams, with flexible and rigid types being roughly equal in market size. In both cases, the foam is usually behind other materials: flexible foams are behind upholstery fabrics in commercial and domestic furniture; rigid foams are inside the metal and plastic walls of most refrigerators and freezers, or behind paper, metals and other surface materials in the case of thermal insulation panels in the construction sector. Its use in garments is growing: for example, in lining the cups of brassieres. Polyurethane is also used for moldings which include door frames, columns, balusters, window headers, pediments, medallions and rosettes. The new iPad Smart Cover is made of polyurethane, (http//wikipedia.org).

2.6 Foaming reaction

The foaming reaction stages are the period between chemical mixing and the full attainment of solid block height. It is during the foaming stage that all characteristics of the foam are formed and for this reason, much understanding of events taking place is necessary if foam qualities are to be manipulated to give good effects.

The reaction stage can be divided into three stages:

- Cream time
- Rising time
- Curing time

2.6.1 Creaming time: is the first sign that chemical reactions have started. Noticeably, the chemical mixture has a slightly white colour and it has been determined that this period lasts for about 12 seconds. As soon as generation of CO_2 gas starts, the cream time ends, but the duration of this depends on the amount of amine used.

2.6.2 Rising time: is the period when all the foam expansion takes place, which last for 60-80seconds. The expansion of foam takes place as follows:

- Cell structure are formed
- Gas reaction accompanied by generation of CO_2 takes place
- Evaporation of auxiliary blowing agent if present in formation.

2.6.3 Curing time: when the foam full rise is achieved, the foam is initially a soft gelatinous mass and the period between attainment of full rise and complete solidification of foam is the curing time. This is essentially the time most cross-linking occurs.

2.7 Foam Characterization

2.7.1 Compressibility Test

The value obtained from this test is a measurement of the ability of foam to recover after subjection to constant deflection compression. The compression set can simply be defined as the difference between the initial thickness and final thickness of foam as a percentage of its original thickness. Mathematically, it is expressed as

$$\text{Compression set} = \frac{T0-Tf}{T0} \times 100 \qquad 2.1$$

Where T_0 is the original thickness of the test-piece and T_f is the thickness of the test-piece after recovery. Whenever the value of compression set is obtained is reported, test conditions must also be stated and these are: level of compression, time and temperature.

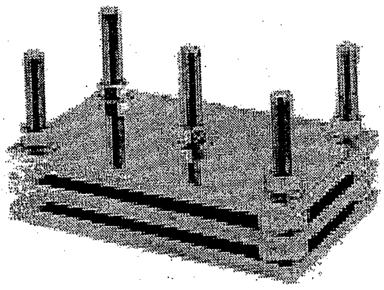


Figure 2.10 a typical compression setting device

2.7.2 Indentation Hardness Test

The indentation hardness index of foam is measurement of its load-bearing properties. In real terms, the index is the force required to depress by 25 %, 40 % and 65 % a small circular plate into the foam. The testing machine is capable of indenting the test-piece between a supporting surface and an indenter which has a uniform relative motion in a vertical direction.

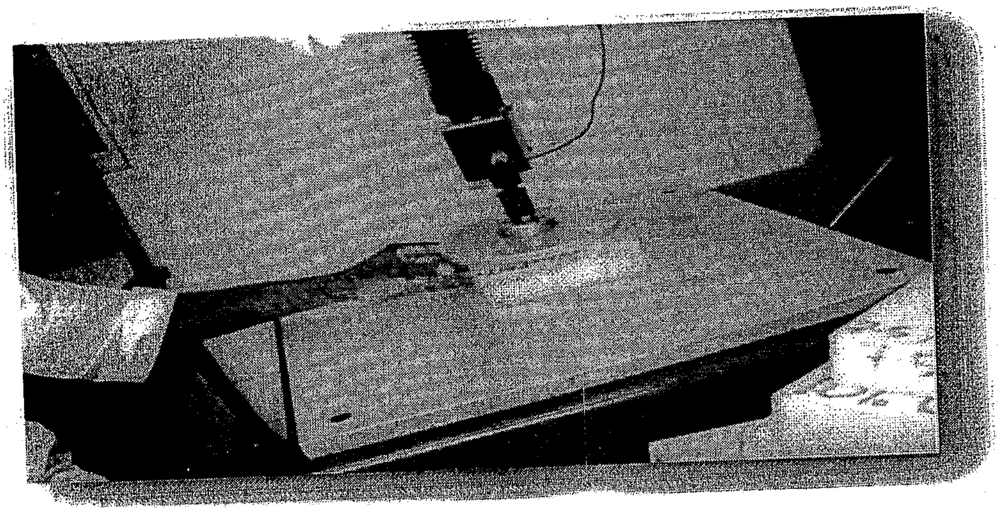


Figure2.11: Indentormetor (Hampden EC30, V2.48/024)

2.7.3 Density test

The measured density of any foam is an apparent or bulk density and not polymer density. Density of foam has a parallel relationship with both its cost and load-bearing ability. High densities will generally result in higher costs and improved load-bearing properties.

Mathematically, Apparent density $= \frac{Mass\ (Kg)}{Volume\ (m3)}$ 2.2

2.7.4 Tensile Test

A tensile test gives information about the elasticity of foam and also indicates the strength of the foam under tension. By definition therefore, tensile strength is the maximum force required to break a test- piece, divided by the original cross-sectional area.

The elongation at break is the change in gauge length of the test piece, determined at the time of break, and expressed as percentage of its original gauge length.

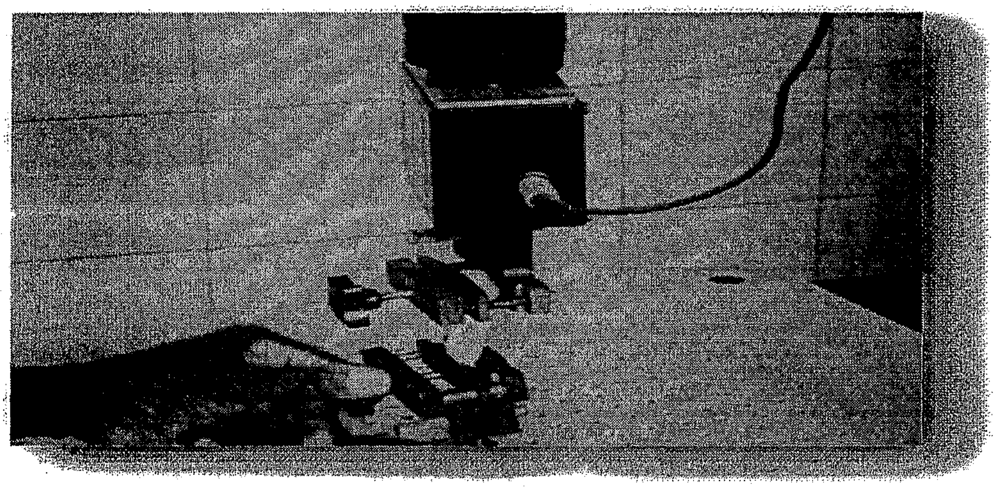


Figure 2.12: Foam samples undergoing a tensile test

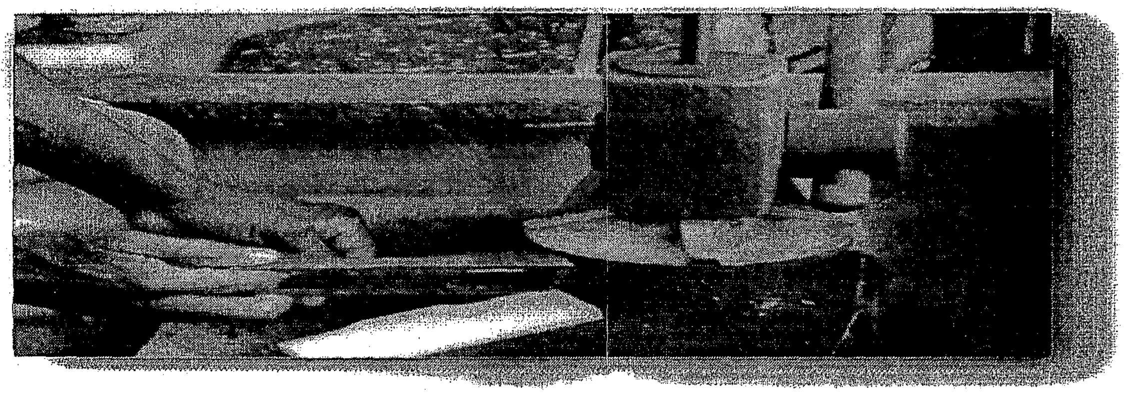


Figure 2.13 Dumb Bell Shape Machine

2.7.5 Water Absorption test

Water absorption was determine by immersing the test sample restrained by a metal net, beneath the tested sample and place on the top surface of the water for 24 hours after removal from the water, the sample were blown free of surface water and weighed. The percentage absorption was calculated as follows.

$$\% \text{ water absorption} = \frac{\textit{Final weight} - \textit{initial weight}}{\textit{Initial weight}} \times 100 \qquad 2.3$$

2.8 Health and Safety

Fully reacted polyurethane polymer is chemically inert. No exposure limits have been established by OSHA (occupational safety and health administration). It is not regulated by them for carcinogenicity.

Polyurethane polymer is combustion solid and will ignite if exposed to an open flame. It begins to break down at about 240 °C (464 °F). Decomposition can produce isocyanates, carbon monoxide, nitrogen oxide and hydrogen cyanide; firefighter should wear self-contained breathing apparatus in enclosed areas. Polyurethane polymer dust can cause irritation to the eyes and lungs. Proper hydrogen control and personal protective equipment, such as gloves, nose masks, respirators, mechanical ventilation and protective clothing and eye wear should also be used.

Liquid resin blends with Isocyanate may contain hazardous or regulated components. They should be handled in accordance with manufacturer recommendations found on product labels, and in material safety data sheet and product technical literature. Isocyanates are known skin and respiratory sensitizers and proper control should be put in place to prevent exposure to Isocyanate liquid and vapour.

3.4 Sample Preparation

3.4.1 Formulation Tables for Experimental Procedure

Table 3.3: Control sample for PU foam formulation

S/N	Component	Weight (g)
1	Polyol	100.00
2	TDI	53.00
3	Water	5
4	Amine	0.12
5	Silicone	1.02
6	Stannous Octoate	0.28
7	Methyl Chloride	4.96

Table 3.4: Percentage composition of fillers to polyol

S/N	POLYOL (g)	$CaCO_3$ (g)	CHARCOAL (g)
1	95	5	5
2	90	10	10
3	85	15	15
4	80	20	20
5	75	25	25
6	70	30	30

The formulation at Table 3.2 is used to produce a control test for the polyether polyol based polyurethane foam and Table 3.3 was used to produce samples at different percent load of the fillers.

Let the sample with 5 %, 10 %, 15 %, 20 %, 25 %, and 30 % of $CaCO_3$ be A_1, A_2, A_3, A_4, A_5, A_6, and samples with the same percentage composition for charcoal be B_1, B_2, B_3, B_4, B_5, and B_6 respectively.

Also the control sample test should be represented by C.

Chapter Three

3.0 RESEARCH METHODOLOGY

3.1 Production Methods/Method Used

There are various methods of foam production. According to the known chemistry of polyurethane, various required components are brought together in certain ways to produce desired foam.

There are three methods of foam production and these are:

i. Batch process
ii. Semi-continues process
iii. Continues process

3.1.1 Batch Process

This is suitable for small scale production which involves the charge of the feed into the system and product is withdrawn all at the end of the desired conversion. No mass crosses the system boundaries between the time the feed is charged and product is withdrawn.

3.1.2 Semi-Continues Process

This is a process whose characteristics are between that of batch and continuous process. In this process, recycle operation can be carry out.

3.1.3 Continues Process

This is a process in which the feed and product flows continuously throughout the duration or process. It often involves many complex operations.

3.2 Method Used

In the course of this research work, the batch method was used, because it is usually used in carrying out a lab-mix.

3.3 Instruments, Equipment and Materials

The instrument, equipment and materials used in this research are:

Table 3.1: instrument and equipment

S/N	EQUIPMENT	FUNCTION
1	Stainless steel spatula	
2	Digital weighing balance	used for weighing
3	Pyrex glass beakers	storage containers
4	5ml syringes	
5	Stopwatch	used for timing
6	Dropper bottle	used for addition of water
7	Cardboard paper	used as mold
8	Indentometer (Figure 2.11, Hampden EC30, V2.48/024)	indenting machine
9	Compression device (Figure 2.10)	compressing setting device
10	Dumb bell shape machine (Figure 2.13)	cutting out the shape sample for tensile test
11	knife edge cutter machine (Figure 2.14)	cutting of the block samples
12	Tensile Strength testing machine (Figure 2.12)	tensile test device

Table 3.2: list of chemicals and their manufacturer

S/N	Chemicals	Manufacturer
1	Polyether Polyol	Korea polyol co. Ltd, Korea
2	2,4-Toluene di-Isocyanate (TDI)	Meryer Chemical Technology, Shanghai company
3	Dimethylethanolamine (DMEA)	Dow Chemicals, Europe
4	Silicone oil	Innovative Chemical
5	Stannous Octoate	Dow Chemicals, Europe
6	Methylene Chloride	Dow Chemicals, Europe
7	Calcium Carbonate ($CaCO_3$)	Freedom Nigerian company
8	Charcoal	Local Supply

3.5 Experimental Procedure

The steps followed in the course of this research using batch method are outline below

3.5.1 Foam Preparation

Using a batch reactor all the reagents are mixed into a 1000 ml beaker for each formulation, the required amount of all the functional groups, activators, Stabilizer, additives and others such as ABA and water were measured using syringes and dropper bottle.

Water, silicone oil and Amine were mixed thoroughly for about 10 seconds, after which the mixture was transfer into the polyol and was mixed thoroughly for 30 seconds. MC and Stannous Octoate was added and mixed for about 20 seconds. Finally, TDI was added into the mixture and rapidly mixed for 3-5 seconds respectively. The mixture was then smartly poured into the prepared mold.

3.5.2 Preparation of PU Foam with addition of Fillers.

The process is almost the same with that of foam preparation expect that the inorganic fillers were synthesized with the amount of each chemical component chosen to obtain a target of 21 kg/m^3. The percentage weight of the polyol and corresponding percentage weight of fillers were initially mixed thoroughly creating a complete dispersion of the fillers into the oil. $CaCO_3$ and Charcoal of particle sizes 500 µm and 350 µm. all other raw materials were added to the mixture and were mixed thoroughly. The diisocyanate was directly charged into the mixture and complete formulation was stirred thoroughly for 4 seconds. The foam formulation was the immediately poured into an open box. Six foam batches for. each filler concentration were produced with identical chemical formulation. After 10 min, the foams were removed from the mold and were allow curing.

3.6 Determination of Foaming Reaction

During the experimental procedure, cream, rising and curing time were noted and was recorded as shown in the table below.

Table 3.5 Foaming Reaction

Time	C	A_1	A_2	A_3	A_4	A_5	A_6	B_1	B_2	B_3	B_4	B_5	B_6
Creaming(sec)													
Rising(min)													
Curing(hr)													
Weight(g)													

Chapter Four

4.0 RESULTS AND DISCUSSION

4.1 RESULTS

4.1.1 Observation Records during Foaming Reaction

Table 4.1 Foaming Reaction

Time	**C**	**A_1**	**A_2**	**A_3**	**A_4**	**A_5**	**A_6**	**B_1**	**B_2**	**B_3**	**B_4**	**B_5**	**B_6**
Creaming (sec)	8	10	10	10	10	10	10	12	12	12	12	12	12
Rising	1.31	1.25	1.23	1.29	1.22	1.20	1.23	1.20	1.12	1.26	1.16	1.18	1.35
Curing(hr)	24	24	24	24	24	24	24	24	24	24	24	24	24
Weight(g)	129.5	137.6	129.7	131.1	128.6	129.5	127.9	136.8	139.3	132.7	136.6	134.4	123.

4.2 Results on the Quality Control Test

Table 4.2 Result of the physical/mechanical properties on calcite samples

S/N	**Experiment**	C	A_1	A_2	A_3	A_4	A_5	A_6
1	Density(Kg/m^3)	20.89	21.5	21.26	21.85	22.17	22.72	24.59
2	Hardness Index(KN)	2.3	2.18	2.3	3.2	3.6	3.98	4.06
3	Compression test 75% (%)	3.8	8.0	7.4	7.6	8.0	12.0	11.11
4	Tensile Strength(KN/M^2min)	99.00	100.69	81.94	74.78	69.28	53.6	47.35
5	Elongation ()	148.38	121.52	100	96.53	93.00	87.60	80.15
6	Water absorption test	10.35	13.52	6.63	19.45	18.04	13.21	5.75

Table 4.3 Result of the physical/mechanical properties on carbon black samples

S/N	**Experiment**	C	B_1	B_2	B_3	B_4	B_5	B_6
1	Density(Kg/m^3)	20.89	22.07	22.84	21.06	22.03	23.17	25.10
2	Hardness Index(KN)	2.3	2.3	2.5	2.7	3.9	4.07	4.38
3	Compression test 75% (%)	3.8	29.11	52.38	3.7	0.0	6.89	7.69
4	Tensile Strength(KN/M^2min)	99.00	94.00	70.00	59.33	47.96	37.50	32.47
5	Elongation ()	148.38	104.86	80.00	69.63	61.50	57.00	48.90
6	Water absorption test	10.35	9.14	11.61	8.44	18.74	13.02	6.42

Table 4.4 Cost Analysis for the foam samples

% Filler	Polyol	TDI	Water	Silicone	St. Octoate	Amine	MC	Filler	Total Cost	% Reduction
0%	100	53	5	1.02	0.28	0.12	4.96	0		
Cost	19.5	14.045	0	0.893	0.42	0.064	0.595	0	35.52	0.00
5%	95	53	5	1.02	0.28	0.12	4.96	5		
Cost	18.53	14.045	0	0.893	0.42	0.064	0.595	0.006	34.55	2.72
10%	90	53	5	1.02	0.28	0.12	4.96	10		
Cost	17.55	14.045	0	0.893	0.42	0.064	0.595	0.013	33.58	5.46
15%	85	53	5	1.02	0.28	0.12	4.96	15		
Cost	16.58	14.045	0	0.893	0.42	0.064	0.595	0.019	32.62	8.89
20%	80	53	5	1.02	0.28	0.12	4.96	20		
Cost	15.6	14.045	0	0.893	0.42	0.064	0.595	0.025	31.64	10.92
25%	75	53	5	1.02	0.28	0.12	4.96	25		
Cost	14.63	14.045	0	0.893	0.42	0.064	0.595	0.031	30.68	13.63
30%	70	53	5	1.02	0.28	0.12	4.96	30		
Cost	13.65	14.045	0	0.893	0.42	0.064	0.595	0.038	29.71	16.37

4.2 Discussion of Result

4.2.1 Effect of Fillers on Foam Rising Time

Table 4.1 shows the observation made on the samples during experimentation. It could be seen that the rising time of all the samples with filler composition except that of 30 % carbon black that is higher than the control sample. All the samples were subjected to the same curing time of 24 hrs.

It was observed that at 15 %, 25 %, and 30 % composition of carbon black filler, the sample have pinholes as a result of excess air injection in the foaming mixture or over-mixing.

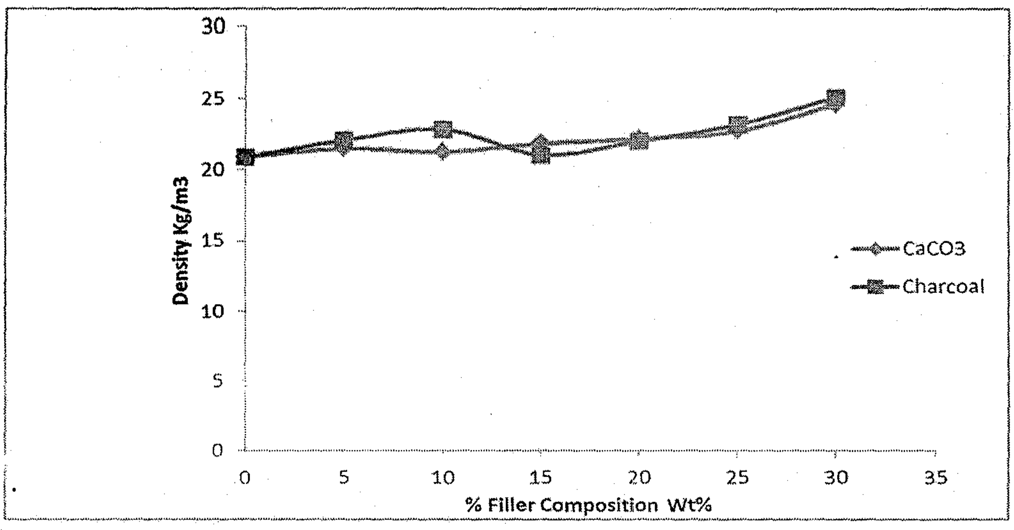


Figure 4.1 Effect of filler on Foam density

4.2.2 Effect of Filler on the Foam Density

Foam density is a specific measurement of how much weight in kilograms polyurethane foam can handle per cubic meter. This density rating tells you the strength of the foam. The result obtained indicated that the 100 % Polyol composition foam has density of 20.89 kg/m^3 approximately equal to 21 kg/m^3 which was the intended density.

Figure 4.1. shows increase in density with increase in percentage filler composition. At 15 % composition of filler, foam density was 21.06 kg/m^3 approximately the same with that of the desired density and at the same time cost of production is optimized.

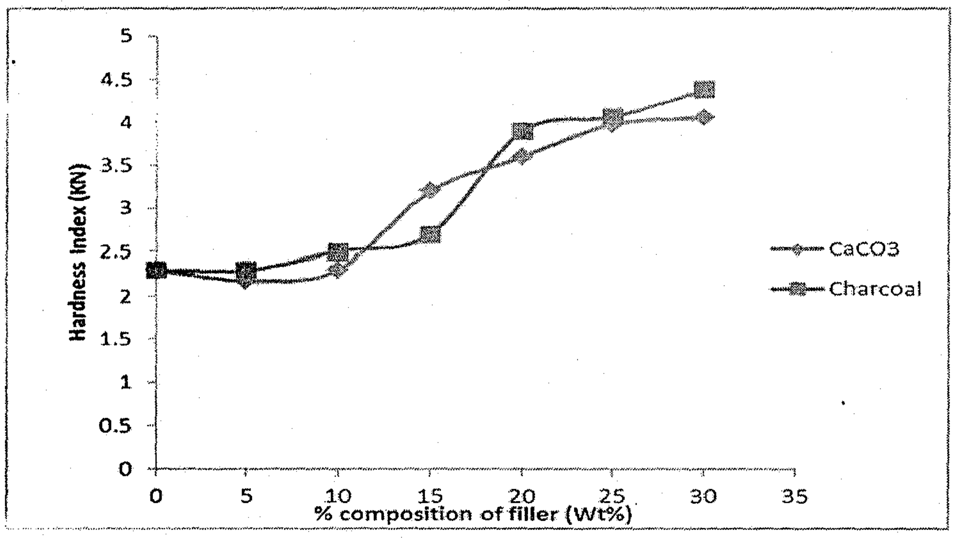


Figure 4.2 Effect of filler on the support factor of flexible Polyurethane foam

4.2.3 The Effect of Filler Composition on the Hardness Index of the Foam Sample

The support factor is the ratio of 65 % IFD (indentation force deflection) divided by the 25 % IFD (indentation force deflection). The result obtained indicated that the 100 % Polyol composition foam have the hardness index of 2.3, the higher the number, the greater the difference between the surface firmness and the deep support. Higher support factors allow desirable softness and a firm inner support.

From figure 4.2, it indicates that at 5 % and 10 % composition of $CaCO_3$ filler have a reduced hardness index, after which there is an increase on the support factor for both the $CaCO_3$ and Carbon black. The support factor according to ASTM D-3489-82 is expected to have a range of 2.3 - 4.38. However, this implies that the cushioning quality of the foam samples varies with filler concentration.

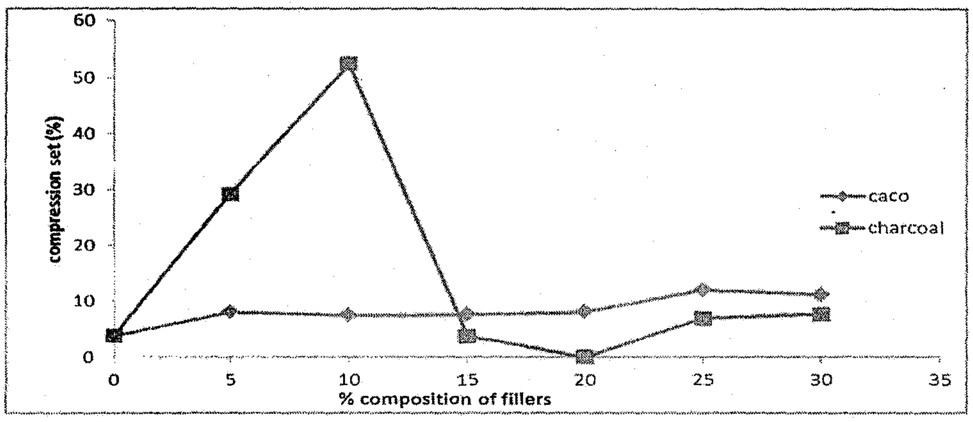


Figure 4.3 Effect of filler on compression setting of flexible polyurethane foam

4.2.4 Effects of Filler Composition on Recoverability and Resilience of Flexible Polyurethane Foam.

The measurement of foam's ability to recover after compression was observed. The percentage compression shows the loss in thickness after the test was performed. The control sample with 0 % composition of filler has 3.8 % loss in thickness after the test while this increases rapidly at 5 % and 10 % carbon black filler composition. As shown in fig. 4.3, at 15 % filler composition of carbon-black has 3.7 % loss in thickness lower than that of the control sample and at 20 % carbon black filler concentration, its shows no loss in thickness indicating a better ability to recover after compression.

For the calcium carbonate, fig. 4.3 shows that the minimum compression coefficient occurred at 0 % filler composition.

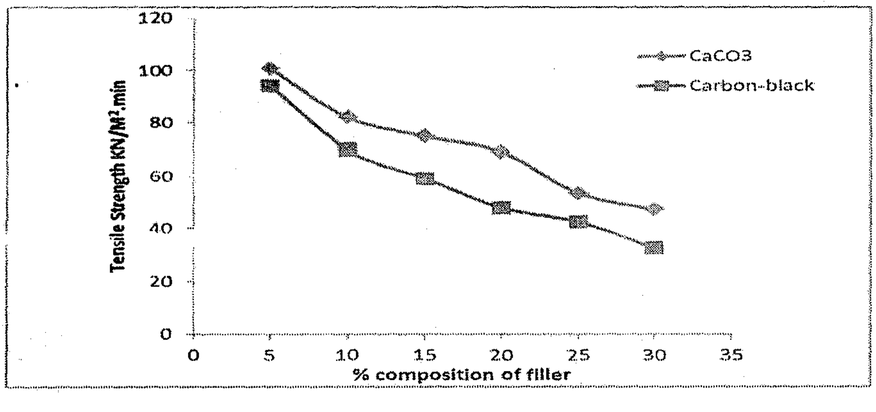


Figure 4.4 Effect of filler on the tensile strength of the foam sample

4.2.5 The Effect of Fillers on the Tensile Strength of Flexible Polyurethane Foam

Tensile strength is resistance of a material to a force tending to tear it apart, measured as the maximum tension the material can withstand without tearing.

As shown in figure 4.4, the tensile strength was found to decrease generally, as the filler composition increases from 5 % to 30 %. The result obtained indicated that the 100 % polyol composition foam fell within this range 99 kN/m^2min. However, the effects of fillers are observed to affect this property with the closet value to the standard being 5wt % $CaCO_3$ and Carbon black composition having a value of 100.69/94 kN/m^2min. However, the results also indicated that the tensile strength of the Carbon-black filled foams were lower than those of $CaCO_3$ filled foams. This indicates that $CaCO_3$ filled foams are better in terms of this property.

Elongation is defined as the extent to which a foam sample can be stretched before it breaks and it is expressed as a percentage of its original length. The results indicate that for 100 % polyol foam has an elongation of 148.38 %.The elongation points at breakage was found to decrease generally, as the filler composition increases from 5% to 30 %. Higher elongation indicates higher ductility. However, the effects of fillers are observed to affect this property. The results also indicated that the elongation points at breakage of the $CaCO_3$ filled foams were higher than those of Carbon-black filled foams. This indicates that $CaCO_3$ filled foams are better in terms of this property.

4.2.6 The Effect of Water Absorption on the Polyurethane Foam

Absorption of water can result through diffusion and condensation. It is observed from table 4.2 and 4.3 that the percentage of water absorption at 20 % concentration for both calcium carbonate and charcoal filler shows a good texture of the foams. Also, it could be deduced from table 4.2 and table 4.3 that at 30 % concentration of both filler samples have the lowest percentage of water absorption due to their closed cell structure. This indicates that fillers at 20 % composition are better in terms of this property.

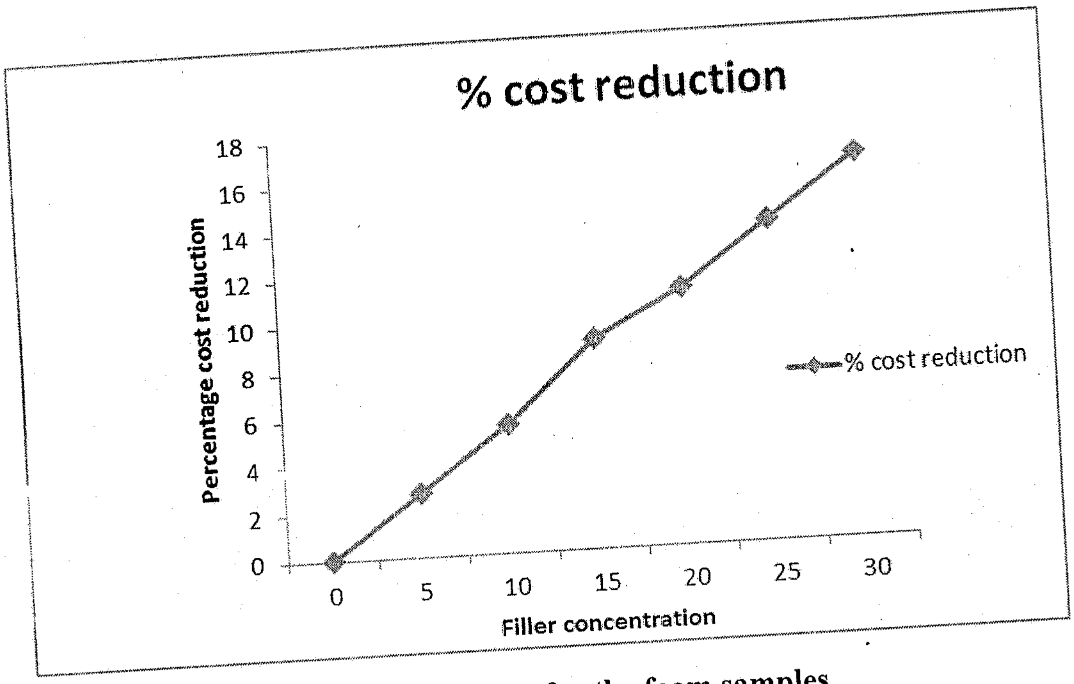


Figure 4.5 Percentage cost reduction for the foam samples

4.2.7 Effect of Fillers on Cost Analysis of the Foam Samples

Figure 4.2 shows how cost in production of foam reduces with filler concentration. This explains the optimization of production cost at each filler concentration.

Chapter Five

5.0 Conclusion and Recommendation

5.1 Conclusion

The aim of this research work is to study the effect of calcium carbonate and charcoal as filler in polyurethane foam production and their effects on the physical properties of foam.

The following conclusions can be drawn from the investigation carried out in this work.

1. Charcoal was successfully use as filler for production of flexible polyurethane foam.
2. Desired foam density could be achieved irrespective of the filler composition even though filler tends to increase foam density. This could be achieved by adjusting the concentration of other chemicals in the formulation.
3. The results of physico-mechanical properties showed variations in properties of the foams produced with increasing filler load. The study shows that at 15 % charcoal filled foams, has 3.7 % loss in thickness lower than that of the control sample and at 20 % carbon black filler concentration, its shows no loss in thickness indicating a better ability to recover after compression.
4. The indentation force deflection test shows that the introduction of filler in the foam formulation also gave foams with improved load bearing ability, with the maximum load bearing ability occurring at 30 % filler composition for both the calcium carbonate and charcoal filled foams.
5. The most important fundamental requirement in production is optimization of cost. From the result obtained, it has been observed that the greater the quantity of filler to replace the polyol, the higher the percentage reduction in the cost of producing flexible polyurethane foam.
6. From all the result obtained, it was determined that best filler composition to be used is 20 % charcoal and calcium carbonate filler concentration. This is because the foam sample had the best resilience and recovery at these filler concentration based on

compression coefficient obtained from the calculation. Also the cushioning quality of the foam which depends on the support factor also gave relatively good values for the 10 % calcium carbonate and 5 % charcoal filler composition.

7. From the look of charcoal filled samples, charcoal can also be used as colourant in producing foam.

It is therefore justifiable and reasonable to conclude from the analysis and tests carried out that the introduction of filler into foam formulations will not only optimized the cost of production of the flexible polyurethane foam, but also help to maintain or improve upon the foam qualities and physical properties thereby establishing the significance of this research.

5.2 Recommendation

In light of the experimentation performed, the result obtained, the test carried out, and the discussion, the following recommendations may be made to further this research.

1. Further research on the effect of charcoal filler on the properties of polyurethane foam need to be investigated by increasing the composition up to 50 % concentration.
2. Other inorganic fillers such as ground limestone, barium sulphate (barytes), clays (china, kaolins e.t.c) can also be used to investigate their effects on the properties of polyurethane foam.
3. The effects of Charcoal filler should also be investigated on individual chemical ingredients of polyurethane foam, in order to establish a standard.
4. Polymer testing laboratories is needed to facilitate full characterization of a polyurethane foam sample.
5. Government should increase its funding to polymer research, because with materials, new materials can be produced.

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DECLARATION

I declare that the work in the project report (thesis) entitled **EFFECT OF FILLERS ON THE PROPERTIES OF FLEXIBLE POLYURETHANE FOAM** has been performed by me under the supervision of A. A. Aboje. No part of this project report was presented for another degree or diploma elsewhere at any institution to the best of my knowledge.

Abubakar Mustapha		
Student Name	Signature	Date

APPENDIX

CALCULATIONS FOR QUALITY CONTROL TEST OF ALL THE SAMPLES

FOAM DENSITY

Density $= \frac{Mass\ (Kg)}{Volume\ (m3)}$

Where volume = length × breadth × height

Unfilled Sample

Mass = 0.1295

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16.4}/_{100}) = 0.0062$

Density = 20.89Kg/m^3

Samples with $CaCO_3$ Filler

At 5% filler composition

Mass = 0.1376

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16.9}/_{100}) = 0.0064$

Density = 21.5Kg/m^3

At 10% filler composition

Mass = 0.1297

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16}/_{100}) = 0.0061$

Density = 21.26Kg/m^3

At 15% filler composition

Mass = 0.1311

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{15.9}/_{100}) = 0.006$

Density = 21.85Kg/m^3

At 20% filler composition

Mass = 0.1286

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{15.5}/_{100}) = 0.0058$

Density = 22.17Kg/m^3

At 25% filler composition

Mass = 0.1295

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{15}/_{100}) = 0.0057$

Density = 22.72Kg/m^3

At 30% filler composition

Mass = 0.1279

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{13.8}/_{100})$ = 0.0052

Density = 24.59Kg/m^3

Samples with Carbon-black filler

At 5% filler composition

Mass = 0.1368

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{21}/_{100})$ = 0.0062

Density = 22.07Kg/m^3

At 10% filler composition

Mass = 0.1393

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16}/_{100})$ = 0.0061

Density = 22.84Kg/m^3

At 15% filler composition

Mass = 0.1327

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16.5}/_{100})$ = 0.0063

Density = 21.06Kg/m^3

At 20% filler composition

Mass = 0.1366

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{16.4}/_{100})$ = 0.0062

Density = 22.03Kg/m^3

At 25% filler composition

Mass = 0.1344

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{15.3}/_{100})$ = 0.0058

Density = 23.17Kg/m^3

At 30% filler composition

Mass = 0.1230

Volume = $(^{19}/_{100}) \times (^{20}/_{100}) \times (^{13}/_{100})$ = 0.0049

Density = 25.10Kg/m^3

COMPRESSION SET

% Compression Set = $\frac{T0-Tf}{T0} \times 100$

Where

T_0 = Original thickness of test sample

T_f= Thickness of test sample after recovery

% = Percentage

Unfilled Sample

$T_0 = 2.6$

$T_f = 2.5$

$= \frac{2.6-2.5}{2.6} \times 100 = 3.8\%$

Samples with Carbon Black Filler

At 5% Filler Composition

$T_0 = 2.4$

$T_f = 1.7$

$= \frac{2.4-1.7}{2.4} \times 100 = 29.11\%$

At 10% Filler Composition

$T_0 = 2.1$

$T_f = 1.0$

$= \frac{2.1-1.0}{2.1} \times 100 = 52.38\%$

At 15% Filler Composition

$T_0 = 2.7$

$T_f = 2.6$

$= \frac{2.7-2.6}{2.7} \times 100 = 3.7\%$

At 20% Filler Composition

$T_0 = 2.6$

$T_f = 2.6$

$= \frac{2.6-2.6}{2.6} \times 100 = 0\%$

At 25% Filler Composition

$T_0 = 2.9$

$T_f = 2.7$

$$= \frac{2.9-2.7}{2.9} \times 100 = 6.89\%$$

At 30% Filler Composition

$T_0 = 2.6$

$T_f = 2.4$

$$= \frac{2.6-2.4}{2.6} \times 100 = 7.69\%$$

Samples with Calcium Carbonate Filler

At 5% Filler Composition

$T_0 = 2.5$

$T_f = 2.3$

$$= \frac{2.5-2.3}{2.5} \times 100 = 8.0\%$$

At 10% Filler Composition

$T_0 = 2.7$

$T_f = 2.5$

$$= \frac{2.7-2.6}{2.7} \times 100 = 7.4\%$$

At 15% Filler Composition

$T_0 = 2.6$

$T_f = 2.4$

$$= \frac{2.6-2.4}{2.6} \times 100 = 7.6\%$$

At 20% Filler Composition

$T_0 = 2.5$

$T_f = 2.3$

$$= \frac{2.5-2.3}{2.5} \times 100 = 8.0\%$$

At 25% Filler Composition

$T_0 = 2.5$

$T_f = 2.3$ $= \frac{2.5-2.3}{2.5} \times 100 = 12.0\%$

At 30% Filler Composition

$T_0 = 2.7$

$T_f = 2.4$

$$= \frac{2.7-2.4}{2.7} \times 100 = 11.11\%$$

HARDNESS INDEX

$$\text{Support Factor} = \frac{65\%\ IFD}{25\%\ IFD}$$

IFD = (indentation force deflection)

Table showing data for support factor calculation

	Indentation force deflection test		
Samples	**65%IFD**	**25%IFD**	**Hardness Index**
Unfilled	105	46	2.3
A_1	131	60	2.18
A_2	158	69	2.3
A_3	149	47	3.2
A_4	186	52	3.6
A_5	179	45	3.98
A_6	187	46	4.06
B_1	143	63	2.3
B_2	154	61	2.5
B_3	169	63	2.7
B_4	179	46	3.9
B_5	159	39	4.07
B_6	193	44	4.38

WATER ABSORPTION

$$\%\text{ water absorption} = \frac{\text{Final weight} - \text{Initial weight}}{\text{Initial weight}} \times 100$$

Table showing data for calculating water absorption

Sample	**Final weight**	**Initial weight**	**% absorption of water**
Unfilled	142.9	129.5	10.35
A_1	156.2	137.6	13.52
A_2	138.3	129.7	6.63
A_3	156.6	131.1	19.45
A_4	151.8	128.6	18.04
A_5	144.8	129.5	13.21
A_6	135.7	127.9	5.75
B_1	149.3	136.8	9.14
B_2	157.6	139.3	11.61
B_3	143.9	132.7	8.44
B_4	162.2	136.6	18.74
B_5	151.9	134.4	13.02
B_6	130.9	123.0	6.42