

VIBRATION DAMPING CHARACTERISTIC AND SOUND ABSORPTION OF  
BIO-POLYMER FROM WASTE COOKING OIL

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## ABSTRACT

Waste cooking oils are problematic disposal especially in the developed countries. Options for disposing of waste cooking oil are limited. Pouring waste cooking oil down to the drain or sewers leads to clog and odour as well as damage wastewater leading to problems for humanlife. Thus, in this research, vegetable waste cooking oil is used as raw material to produce foam. This foam was crosslink with flexible isocyanate type Polymethane Polyphenyl (Modify polymeric-MDI) or rigid isocyanate type Polymethane Polyphenyl Isocyanate (Diisocyanate-diphenylmethane-MDI) and further processed into granulate (powder) to be able to hot compress by hot compression moulding technique. These fabricated samples were namely as hot bio-polymer flexible (HBF) and hot bio-polymer rigid (HBR). HBF or HBR samples with three different thickness/laminated structure namely as 5.4 mm (F1), 10.8 mm (F2), 16.2 mm (F3) as well as 5.4 mm (R1), 10.8 mm (R2), 16.2 mm (R3) respectively of each type cross linking agent (flexible and rigid) was examined by using vibration transmissibility test and sound absorption test. Vibration transmissibility test was generated at 1 mm, 1.5 mm, 0.1 g and 0.15 g base excitation while the mechanical properties were examined through compression test, thermogravimetric analysis (TGA) and density test. The morphological structure of HBF and HBR were observed by scanning electron microscope (SEM). The results showed that HBF is denser than HBR that is  $1241.15 \text{ kg/m}^3$  and  $1183.4 \text{ kg/m}^3$  respectively. Therefore, HBF gave higher damping ratio to transmit vibration with F1 revealed the most excellent vibration damping of 0.671 at 1.5 mm base excitation meanwhile, the vibration absorption of highest thickness gives highest percentage of increment of absorption for F3 and R3 with 95.9 % and 91.8 % respectively. The decreasing of HBF thickness evidently increased the damping ratio up to 45 %. R1 shows an excellent sound absorption coefficient ( $\alpha$ ) of 0.72 at highest frequency level (5000 Hz) but shifted to lower frequency of 2500 Hz down to 1900 Hz for higher thickness samples up to 16.8 mm with maximum  $\alpha$ . In conclusion, this hot

compression technique is useful as processing method as an alternative recycling for bio-polymer generated from recyclable of waste vegetable cooking oil.

## ABSTRAK

Sisa minyak masak adalah pelupusan bermasalah terutamanya di negara-negara maju. Pilihan untuk melupuskan sisa minyak masak adalah terhad. Selain itu, membuang sisa minyak masak ke dalam longkang dan pemetung membawa kepada penyumbatan dan bau yang akan membawa masalah kepada kehidupan. Oleh itu dalam kajian ini, sisa minyak masak sayur telah digunakan sebagai bahan mentah untuk menghasilkan span. Span ini telah disilang dengan menggunakan ejen penyilang fleksibel jenis Polymethane Polyphenyl Isocyanate (Modified polymeric-MDI) dan penyilang tegar jenis Polymethane Polyphenyl Isocyanate (Diisocyanate-diphenylmethane-MDI) dan seterusnya diproses menjadi butiran (serbuk) dan selepas itu dimampat dengan menggunakan pemanasan mampat. Sampel fabrikasi ini dinamakan sebagai bio-polimer fleksibel panas (HBF) dan bio-polimer tegar panas (HBR). HBF dan HBR sampel diuji dengan tiga ketebalan/lapisan berbeza iaitu 5.4 mm (F1), 10.8 mm (F2), 16.2 mm (F3) dan juga 5.4 mm (R1), 10.8 mm (R2), 16.2 mm (R3) bagi setiap ejen penyilang fleksibel dan tegar dan telah diuji dengan menggunakan ujian getaran kebolehtantaran dan ujian penyerapan bunyi. Ujian getaran kebolehtantaran telah dijana pada 1 mm, 1.5 mm, 0.1 g dan 0.15 g pada pengujaan asasnya manakala sifat mekanikal telah diperiksa melalui ujian mampatan, analisis termogravimetri (TGA) dan juga ujian kepadatan. Struktur morfologi HBF dan HBR telah diperhati dengan menggunakan mikroskop imbasan elektron (SEM). Hasil kajian menunjukkan bahawa HBF lebih tumpat daripada HBR yang masing-masing bernilai  $1241.15 \text{ kg/m}^3$  dan  $1183.4 \text{ kg/m}^3$ . Oleh itu, HBF memberikan nisbah redaman yang lebih tinggi untuk menghantar getaran yang mana F1 memberi redaman getaran yang paling baik iaitu 0.671 pada pengujaan asas 1.5 mm. Sementara itu, penyerapan getaran pada ketebalan tertinggi memberikan peratusan kenaikan penyerapan tertinggi bagi F3 dan R3 iaitu masing-masing sebanyak 95.9 % dan 91.8 %. Apabila ketebalan HBF berkurangan nisbah redaman meningkat sehingga 45 %. R1 menunjukkan keputusan pekali penyerapan bunyi ( $\alpha$ ) yang paling

baik iaitu 0.720 pada fekuensi tertinggi (5000 Hz) tetapi beralih kepada frekuensi yang lebih rendah sebanyak 2500 Hz ke 1900 Hz untuk sampel ketebalan yang lebih tinggi sehingga 16.2 mm dengan  $\alpha$  maksimum. Kesimpulannya, teknik mampatan panas ini berguna sebagai kaedah pemprosesan sebagai alternatif kitar semula untuk bio-polimer yang dihasilkan daripada sumber kitar semula sisa minyak masak.

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## LIST OF SYMBOLS

Tan $\delta$	-	damping factor
$\sigma$	-	stress
$\varepsilon$	-	strain
$\rho$	-	Density
$k, K_c$	-	stiffness
$x, z$	-	Displacement in uniaxial compression
$t$	-	Time
$C_c$	-	Damping coefficient or damping constant
$A$	-	Contact area
$D$	-	Original cushion thickness
$W, M, m$	-	Total mass or total riding mass
$\omega_n$	-	Natural frequency
$\omega$	-	Forced frequency
$\omega_b$	-	Base frequency
$x, X$	-	Passenger or mass motion (output)
$y, Y$	-	Base motion (Input)
$m\ddot{x}$	-	Inertia force
$r$	-	Frequency ratio
$K_x, K_y, K_z$	-	Stiffness in x, y, or z axis
$S$	-	Structural coefficient
$V$	-	Volume
$\alpha$	-	Sound absorption coefficient or viscoelastic parameter
NCO	-	Reactive isocyanate group (-N=C=O)
$R$	-	Reactive group from isocyanate
$R'$	-	Reactive group from polyol



$n$	- 1, 2, 3 ...
$\beta$	- logarithmic decrement
$i$	- 1, 2, 3 ...

**LIST OF ABBREVIATIONS**

PU	- Polyurethane
PET	- Polyethylene Terephthalate
wt.	- weight
HB	- Hot Bio-polymer
HBF	- Hot Bio-polymer Flexible
HBR	- Hot Bio-polymer Rigid
F1	- One laminated structure of HBF
F2	- Two laminated structure of HBF
F3	- Three laminated structure of HBF
R1	- One laminated structure of HBR
R2	- Two laminated structure of HBR
R3	- Three laminated structure of HBR
SEM	- Scanning Electron Microscopy
ASTM	- American Society for Testing and Materials
MDI	- Methylene Diphenyl Diisocyanate
TDI	- Toluene Diisocyanate
IIR	- Butyl Rubber
PP	- Polypropylene
SDOF	- Single Degree Of Freedom
UTHM	- Universiti Tun Hussein Onn Malaysia
VCS	- Vibration Control System
RSTD	- Resonance Search, Track and Dwell
IIR	- Infinite Impulse Response
UTM	- Universal Testing Machine
IFD	- Indentation Force Deflection

NaOH	- Sodium Hydroxide
NRC	- Noise Reduction Coefficient
H	- Hydrogen
C	- Carbon
O	- Oxygen
CH <sub>2</sub>	- Hydrocarbon
OH	- Hydroxide
N	- Nitrogen
NH <sub>2</sub>	- Amine
H <sub>2</sub> O	- Water
CO <sub>2</sub>	- Carbon Dioxide
rpm	- revolutions per minute
CaCO <sub>3</sub>	- Calcium Carbonate or Calcite
NA-MMT	- Sodium Montmorillonite
Ca	- Calcium
Cl	- Chlorine
Fe	- Iron
K	- Potassium
Mg	- Magnesium
Si	- Silicon
Zn	- Zinc
Al	- Aluminum
Cu	- Copper
Ti	- Titanium
Comp.	- Compression
Stand. Dev.	- Standard Deviation

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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 General Introduction**

Polymer composite is one of the examples of the material deals with the revolution. Even it is more widespread acceptance and use in various fields, and this makes it one of the important engineering materials because these materials have a distinct advantage of the properties compared with other materials. Composite materials are generally used for buildings, bridges and structures such as boat hulls, swimming pool panels, race car bodies, shower stalls, bathtubs, storage tanks, imitation granite and cultured marble sinks and counter tops. The most advanced examples perform routinely on spacecraft in demanding environments. While the recyclable materials such as waste cooking oil, paper powder, and iron powder can be reused in the production of a polymer composite (Averous and Pollet, 2012). This is the good step in addressing pollution problems involving discard items.

#### **1.2 Cooking oil**

Cooking oil of plant, animal or synthetics fat used in frying, baking and other types of renewable resources for food preparation and flavouring that do not involve heat such as salad dressings and bread dips. Cooking oil typically in liquid form although some oils that contains saturated fat such as coconut oil, palm oil and palm kernel oil.

Proper disposal of used cooking oil is an important waste management concern. As the oil crisis and global warming deepen, bio based materials have

received particular attention. Vegetable oils are becoming extremely important as renewable resources for the preparation of polyols required for the polyurethane industry (Sharma and Kundu, 2008). Polyols from natural oils such as soybean and palm oils as refer to Figure 1.1 are increasingly being viewed by industry as a viable alternative to hydrocarbon based feedstock (Sharma and Kundu, 2008). Actually, these oils are annually renewable, and are cost competitive as well as environment friendly. Referring to the market summary published by the United Soybean Board in February 2000, vegetable oil-based polyurethane is used as binders and agricultural films (Sharma and Kundu, 2008). In this research, the monomer from the waste vegetable oil is used as bio-polymer foam to produce high dense solid for other applications.



Figure 1.1: Natural oils

### 1.3 Bio-polymer

The interest for biodegradable polymers and natural fibre-reinforced polymers has recently grown due to the increasing environmental concerns (Benjamin and Jorg, 2008). Bio-polymers are polymers that are biodegradable. The input materials for the production of these polymers may be either renewable which is based on agricultural plant or animal products or synthetic. There are four main types of bio-polymer based on starch, sugar, cellulose and synthetic materials. Since they are polymers, bio-polymers contain monomeric units that are covalently bonded to form larger structures (Averous and Pollet, 2012). There are three main classes of bio-polymers based on different monomeric units used and the structure of the bio-polymer formed

(i) polynucleotides, which are long polymers, composed of 13 or more nucleotide monomers, (ii) polypeptides, which are short polymers of amino acids and (iii) polysaccharides, which are often linear bonded polymeric carbohydrate structures (Chandra and Rustgi, 1998).

Current and future developments in biodegradable polymers and renewable input materials focus relative mainly to the scale up production and improvement of product properties (Proterra International Center, 2012). Larger scale production will increase availability and reduce prices. Currently either renewable or synthetic starting materials may be distressed to produce bio-degradable polymers. Proterra International Center (2012) stated that two main strategies may be followed in synthesizing a polymer. One is to build up the polymer structure from a monomer by a process of chemical polymerization. The alternative is to take a naturally occurring polymer and chemically modify it to give it the desired properties. The disadvantage of chemical modification is however that the biodegradability of the polymer may be adversely affected. Therefore it is a failure for incidental seeks a compromise between the desired material properties and bio-degradability (Proterra International Center, 2012).

#### **1.4 Green technology**

Yijun and Gengxin (2011) mentioned that the applied research of green production technologies is the mainstream of the current human civilization consciousness that advocates energy conservation and environmental protection, the development of the new energy for environmental protection for saving the energy consumption of the existing products and benefits the society. The main problem should be aimed and made improvement on the efficiency of the energy conservation and environmental protection in order to reduce unnecessary energy waste.

Viet *et al.*, (2011) described that in an operation context, internal today would focus on cost minimization and risk management techniques. For example firms would focus to decrease environmental burden and associated costs of raw materials or disposal and incorporate their employees concerns in decisions and ensure proper worker safety and health standard. Efficient utilization of land, water, energy and other natural resources makes business more productive.

## 1.5 Environmental impact

Due to the importance of environmental issues, governments begin to make laws and regulations to protect the environment. For example, the Restriction of Hazardous Substances (RoHS) of the European Union restricts the use of six toxic materials in the manufacturing of all electronic and electrical equipment (Jack *et al.*, 2010). So, consumers are increasingly willing to purchase eco-friendly or so called green products even though these product are often more expensive. Since the protection on the environment has emerged as one of the hottest global trends, it is important for firms to understand how to design and manage green product (Jack *et al.*, 2010).

Bio-polymers are sustainable, carbon neutral and renewable, because they are made from plant materials which can be grown many years indefinitely. These plant materials come from agricultural non food crops. Therefore, the use of biopolymers would create a sustainable industry (Wikipedia Foundation, 2012) such as corn, soybean and etc. The feedstocks for polymers derived from petrochemicals will eventually run out. In addition, biopolymers have the potential to cut carbon emission and reduce carbon dioxide quantities in the atmosphere. This is because the carbon dioxide released when they degrade can be reabsorbed by crops grown to replace them which is makes them close to carbon neutral (Wikipedia Foundation, 2012).

Abundant researches have been performed to help corporation to become more environmentally friendly through process re-engineering such as reuse, remanufacturing, recycling and collaboration. Vachon and Klassen (2008) study the impact of environmental collaboration in the supply chain on manufacturing and environmental performance. They conclude that environmental collaboration with suppliers is mainly linked to superior delivery and flexibility while collaboration with customers is mostly linked to better quality performance.

## 1.6 Background of research

Flexible polyurethane (PU) foam is one of the major productions from urethane material (Zhang, 2008). It is widely used as cushioning material in applications of transportation and packaging. By proper choice of raw materials, additive and manufacturing technology, properties of flexible PU foam can be changed to satisfy



desired application. In recent years, users demand a more comfortable compartment and now designed for better riding comfort and sound absorption (Broos *et al.*, 2000). It is desirable to improve the vibration characteristic of flexible PU foam whereas with respect to sound absorption, noises such as from engine, road as well as wind should be absorbed and reduced (Koshute *et al.*, 2001).

As the cost of polymer, plastics and timber get higher, there is a need to develop an alternative source or recycling of waste as a substitute to the function they perform in engineering manufacture (Atuanya *et al.*, 2011). Waste recycling is an interesting approach to achieve an efficient integrated manner of managing municipal solid waste (Atuanya *et al.*, 2011).

Therefore, the purpose of the research is to develop flexible bio-polymer and rigid bio-polymer from recycling polymer material based on waste bio-polymer granulate foam to measure damping characteristics with different of thickness based on flexible and rigid crosslinking agents. Bio-polymer foam was produced by using waste cooking oil monomer. The flexible bio-polymer and rigid bio-polymer were fabricated by using hot compression technique from granular particles of bio-polymer foam namely as hot bio-polymer flexible (HBF) and hot bio-polymer rigid (HBR) respectively. The characteristics of the HBF and HBR will investigated and analyzed in detail in providing superior damping to absorb vibration and sound in particular as well as to offer reduction as a recyclable renewable material.

## **1.7 Problem statement**

In recent year, waste cooking oil has proven to be problematic material (Food Standards Agency, 2004). Many people cannot solve the problem and did not know how to manage that problem instead of poured into the drain or toilet. In reality, it is believed that waste cooking oil can produce bio-polymer foam based product. It has the potential by improving its physical and mechanical properties and can generate to reduce the environmental pollution and also as an alternative to petroleum based products.

In previous research, the problem of noise and vibration absorbing material in an automotive field or packaging needed some new material that can be used to replace or as alternative the existing products. Recently, the use of renewable sources in the preparation of various industrial materials has been revitalized because of the

environmental concerns. Thus, the problem is to generate new idea and formulae either in automotive or packaging or building production application. According to the existing product, the applications in automotive such as car dashboard, door panel and engine casing are produced by using natural fiber and petroleum based. Meanwhile, in this research, waste cooking oil monomer is used by adding crosslinker as bio-polymer foam to replace the existing commercialized substances. The cost effective recycling route for thermosetting polymers has not established, it may restrict manufacturer's choice of materials to optimise the functional performance of components (Hulme and Goodhead, 2003).

Polyurethane Foam Association (1994) noted that flexible PU foam scrap is an easy product to recycle and now generating revenue for many end-users. It has made great strides in technology and end-use applications to address waste problem. It is suitable to increase the utilization of the secondary raw materials. Referring to Polyurethane Foam Association (1994), by recovering and reusing scrap foam, the economic and environmental value will cover as well as it can be used to generate revenue, offset raw material cost and alleviate solid waste disposal problems.

Therefore, the crumbling technology opens new ways for the recycling of PU foams and composites. It can be performed by using conventional crushing (Valclavik *et al.*, 2012). A knife grinder can be used both for flexible and rigid PU foam to provide a particle size shape. Thus, hot compression method was used to produce hot bio-polymer (HB) to focus on mechanical properties of the materials such as vibration test and acoustical test due to the less research pointed on the mechanical properties on polymer composites (Gibson, 2000).

## **1.8 Hypothesis of research**

The hypotheses of the research are:

- i. To prove that with a proper weight percentage (wt%) of treated bio-polymer foam, a new fabricated HBF and HBR may offer good vibration or sound damping material
- ii. To prove that the thickness of HB influences the vibration and sound damping characteristic

- iii. The HBF and HBR have the potential to increase the dissipation of energy through absorption of vibration and sounds for comfortableness of any suitable application.

## 1.9 Objectives of research

The objectives of this research are:

- i. To determine the crosslinking agent of flexible and rigid isocyanate to crosslink the bio-monomer (synthesis from waste vegetable oils) into bio-polymer foam and fabricated by hot compression equipment as flexible (HBF) and rigid (HBR) bio-polymer.
- ii. To measure the physical property, and mechanical property and its vibration characteristic of HBF and HBR based on vibration transmissibility test to calculate the damping ratio ( $\zeta$ ) of two different crosslinking agents.
- iii. To measure the sound absorption of HB through acoustical testing at low to high frequency and to compare the sound absorption coefficient ( $\alpha$ ) between two crosslinking agents.

## 1.10 Scope of Research

- i. Literature review on the fundamental process of bio-polymer foam fabrication via use of different cross linking agents to impart flexible and rigid for hot compression technique.
- ii. Developing bio-polymer foam from vegetable waste cooking oil monomer by hot compression technique as HBF and HBR.
- iii. The physical and mechanical properties of HBF and HBR were examined through density and compression test meanwhile, the morphological characteristics of the hot bio-polymer were analyzed by SEM.
- iv. The damping characteristics of HB by using vibration transmissibility test and sound absorption test according to ASTM standard were

conducted with different composition ratio or thickness of the samples.

### **1.11 Significant of Research**

The significances of the research study included:

- i. This research intended in improving or adds composite for the applications in absorbing vibration and sound especially on the inside of motor vehicle and packaging industry. This research will determine whether this is able to increase the polymer mechanical properties of HB that serves as a vibration and sound absorber.
- ii. The HB was developed for the application on the various applications especially in automotive in increasing the comfortableness for driver and passengers. The emotional disturbance to driver and passengers due to environmental effect are decreased since the noise and vibration are dissipated by HB before being transmitted to the driver and passengers.
- iii. This hot compression is an alternative recycling method for bio-polymer generated from recyclable of renewable resources.
- iv. HBF or HBR can be replace as the components in application which offer a great production of cost reduction through the use of waste cooking oil for bio-polymer foam and contributed to sustainability and green technology.

### **1.12 Thesis organization**

**CHAPTER 1** has highlighted the general introduction on this research, background of study, problem statement, hypothesis, objective, scope and its significance of research. It discussed the reason of research aimed in developing hot bio-polymer foam which can provide better vibration and sound damping.

In **CHAPTER 2**, a review of literatures focusing on polymer, composite material, foam production, damping vibration measurement and its parameter and sound absorption coefficient.

**CHAPTER 3** shows the methodology that used to conduct the whole study. The technique of HB preparation, the physical and mechanical test is described in detail.

**CHAPTER 4** until **CHAPTER 6** cover the results and discussion of the experimental carried out from this research. The particles of bio-polymer, the morphological structure, density as well as compression of HB are discussed in **CHAPTER 4**. **CHAPTER 5** shows the vibration characteristic analysis when the HB inserted in vibration transmissibility system and also in vibration absorption system. In **CHAPTER 6**, the results of sound absorption of fabricated HB were discussed in detail.

Table 1.1: Thesis organization description

<b>CHAPTER</b>	<b>Description</b>
CHAPTER 1	Introduction
CHAPTER 2	Literature Review
CHAPTER 3	Research Methodology
CHAPTER 4	HBF and HBR Production and its Physical and Mechanical Properties
CHAPTER 5	Vibration Characteristic
CHAPTER 6	Acoustic Characteristic
CHAPTER 7	Conclusion and Recommendation

Lastly, **CHAPTER 7** summarized the results and discussion of all the experimental testing. At the end of this chapter, the recommendations are list out for future study. The entire chapter is illustrated as tabulated in Table 1.1.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

In Malaysia, options for disposing of used cooking oil and grease are limited. Disposal is difficult because used cooking oil is usually in a liquid or semi-solid form and the solid waste regulations restrict the disposal of liquids in landfills (Solid Waste Program, 2011).

This chapter discusses and reviews previous studies from other researchers on development and characteristics of bio-polymer foam, bio-polymer foam as sound and noise absorption application and the testing methods used for evaluating the foam. It helps to provide more information and explanation especially in bio-polymer of its mechanical and physical properties as well as damping characteristics of hot bio-polymer for the new application especially in automotive field.

#### 2.2 Polymer

Polymer is defined as a large molecule composed of one or more repeating units which are connected by a covalent bond (Dr Esah *et al.*, 1998). It is hydrocarbon based material elements comprise of carbon, hydrogen and oxygen. It is also a material that contains a lot of parts or smaller units; chemically bound to each other.

Benjamin and Jorg (2008) described that the use of traditional composites made of glass, aramid or carbon fiber reinforced plastics have recently been discussed critically because of increasing environmental consciousness. Thus the

recent research and development efforts have led to new products based natural resources. Some of these are biodegradable polymers like PLA (polylactic acid), cellulose esters, polyhydroxyalkanoates and starch polymers (Benjamin and Jorg, 2008). Furthermore, natural fibre-reinforced polymers made with natural fibres like flax, hemp, kenaf, jute or cotton fibres are important research and development (R&D) achievements. Composites made of natural fibres and biopolymers are completely biodegradable and are called “green composites” because of their environmentally beneficial properties as shown as in Figure 2.1.

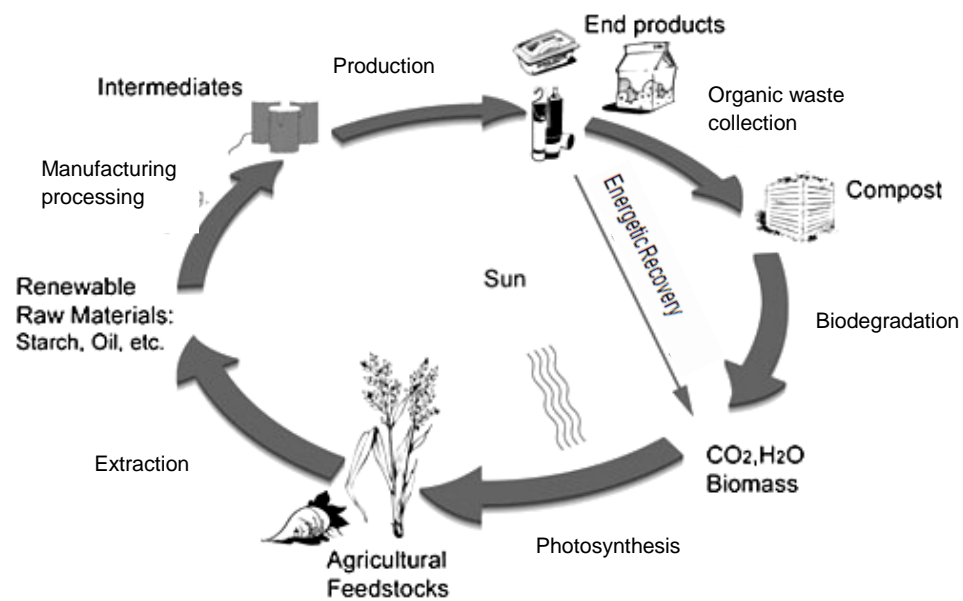


Figure 2.1: Life Cycle (Valentina *et al.*, 2008)

The polymer industry continues to evolve from its rapidly expanding petroleum-based infancy in the middle to-late twentieth century into a complicated economic, regulatory, and ecological industry (Swain *et al.*, 2004). With the increase in environmental consciousness among consumers and governments, the industry is now facing not only with manufacturing and marketing concerns but with ecological and legislative issues as well. The trend is environmental protection and regulation is toward a “cradle-to-grave” approach, in which the manufacturer is increasingly responsible for the handling of raw materials used in production and the finish products, eventual disposal (Swain *et al.*, 2004).

Mohanty *et al.*, (1999) described that when a bio-degradable material (neat polymer, blended product or composite) is obtained completely from renewable resources may call it a green polymeric material. Bio-polymers from renewable resources have attracted much attention in recent years. Renewable sources of polymeric materials offer an answer to maintaining sustainable development of economically and ecologically attractive technology. The innovations in the development of materials from bio-polymers, the preservation of fossil-based raw materials, complete biological degradability, the reduction of the volume of garbage and compost ability in the natural cycle, protection of the climate through the reduction of the carbon dioxides released, as well as the application possibilities of agricultural resources for the production of bio/green materials are some of the reasons why such materials have attracted the public interest (Mohanty *et al.*, 1999).

### **2.2.1 Polymer from renewable resources**

Oil-based bio-polymers have many advantages compared to polymers prepared from petroleum-based monomers. They are bio-degradable and in many cases, cheaper than petroleum polymers (Guner *et al.*, 2006). According to the previous researches Long *et al.*, (2006), generally polymers from renewable resources (PFRR) can be classified into three groups which are natural polymers such as starch, protein and cellulose, synthetic polymers from natural monomers, such as polylactic acid (PLA), and polymers from microbial fermentation, such as polyhydroxybutyrate (PHB). Three ways bio-polymer plastics can be produced are through converting plant sugars into plastic, producing plastics inside microorganisms and growing plastics in corn and other crops.

Long *et al.*, (2006) mentioned that one of the most promising polymers in this regard is PLA, because it is made from agricultural products and is readily biodegradable. Lactide is a cyclic dimer prepared by the controlled depolymerisation of lactic acid, which in turn can be obtained by the fermentation of corn, sugar cane, sugar beet. PLA has the tensile strength, modulus, flavour and odour barrier of polyethylene and PET or flexible PVC; the temperature stability and processability of polystyrene; and the printability and grease-resistance of polyethylene. The glass transition temperature of PLA ( $T_g$ ) ranges from 50 °C to 80 °C while the melting temperature ( $T_m$ ) ranges from 130 °C to 180 °C. PLA can be



processed by injection moulding, sheet extrusion, blow moulding, thermoforming and film forming.

Long *et al.*, (2006) mentioned bacterially synthesized PHAs attract much attention because they can be produced from a variety of renewable resources, and are truly bio-degradable and highly bio-compatible thermoplastics materials. Therefore, PHAs are expected to contribute to the construction of an environmentally sustainable society. Over 90 different types of PHAs existing of various monomers have been reported and the number is increasing such as *Ralstonia eutropha*, *Alcaligenes latus* (PHB) and etc.

Besides, in polymer industry, vegetable oils which represent a major potential source of chemicals have been utilized as an alternative feedstock for monomers (Suresh *et al.*, 2007). This is found that from Sulong and Rus (2010), PU foams made from palm oil were synthesized and metal oxide powder was doped as filler to become renewable polymer composite. The structure of the renewable polymer composite was investigated by using SEM with different percentage of filler.

Others oils such as soybean oil and rapeseed oil-based polyols have been used to make PU foams as well. Based on Suresh *et al.*, (2007), Canola-PU foam gave better compressive properties than Soybean-PU foam but less than Castor-PU foam. The differences in performance were found to be related to the differences in the number and position of OH-groups and dangling chains in the starting materials and to the differences in cellular structure.

### **2.2.2 Composites from oil-based polymers**

Guner *et al.*, (2006) said that Polymers composites are used in a wide range of application areas, such as aerospace, military, construction, electrical and electronics, medicine, marine, transportation and so on. It is known that composites consist of two or more materials forming separate phase. Some of the fillers used glass, boron and steel aromatic polyamide (Guner *et al.*, 2006).

Recently, composites prepared from oil-based polymers have become a special interest in many areas. PU resin prepared from castor oil, for example, was used to obtain graphite composite as an electrode material. Guner *et al.*, (2006) mentioned the 60 % (graphite, w/w) composite exhibited good mechanical and appropriated electric resistance, easy preparation and surface renovation.

In other research, epoxidised soybean oil-based composite was prepared and their viscoelastic properties were investigated. Authors suggested that new material prepared exhibited strong viscoelastic solid properties similar to synthetic rubbers such as compressive strength, damping, creep behaviour and etc. Other investigator manufactured materials from soybean oil-based resin and natural fibers for using as the roof, floors or walls of a house or low-rise commercial buildings. For preparing soybean oil-based resin, first, soybean oil was epoxidized and vinylated by styrene or acrylic acid (Guner *et al.*, 2006).

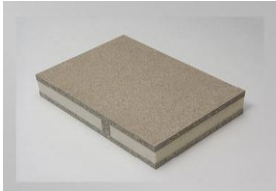


Another researcher synthesized polyurethanes with soybean oil-based polyol or petrochemical based polyol, and then prepared glass-reinforced composites from them. From the experiment, the mechanical properties revealed that properties of the soybean oil based composites were comparable with those based on petrochemical polyol. Additionally, oxidation, thermal and hydrolytic stability of soybean oil-based composite were superior to those of the latter. All results indicated that polyurethane matrix based on soybean oil is a preferable alternative to the petrochemical polyurethanes in glass-reinforced composites (Suhreta *et al.*, 2005).

### **2.3 Foam generation**

Foam is an important engineering material. It offers unique advantages in terms of low cost and weight, easy to manufacture and has good energy absorption properties. It is used in many applications; acoustic absorption, impact retardation and mechanical damping (Singh *et al.*, 2003 and Lee *et al.*, 2007). In most modern automotive seats, static comfort and vibration isolation are now achieved through the use of foam alone.

Polymer foams represent one of many types of polymeric materials such as solid (nonporous), reinforced, laminated, etc. Schematically in Table 2.1, all types of polymer foam can be classified in three generations and each type of foam has different technical characteristics and also different cellular structure (Klempner and Sendjarevic, 2004).

Table 2.1: Three generation of polymer foams (Klempner and Sendjarevic, 2004)

Generation	Type of Foam
First Generation: 1940-1970 	Isotropic foams Sandwich structures
Second Generation: 1970-1985 	Structural (Integral) foams Syntactic foams Reinforced and filled foams Foamed laminates RIM and RRIM foams Microcellular foams
Third Generation: 1985 until now 	One-sided structural foams Inverse structural foams Syntactic - structural foams Foamed syntactic foams Foamed fibres and films Macro/Micro (bi – modal) fomas Foam-in Foam structure Nano foams

Normally, PU foam is made from a formula that contains a host of ingredients selected to achieve the desired grade of foam. These components included polyol, isocyanate, water, catalyst, surfactants, cross-linkage agent, auxiliary blowing agent, and additives such as colorant, flame retardant, antistatic agent, bacteriostat or UV stabilizer. Among all, polyol and isocyanate are the main components used to forms PU linkage (Figure 2.2).

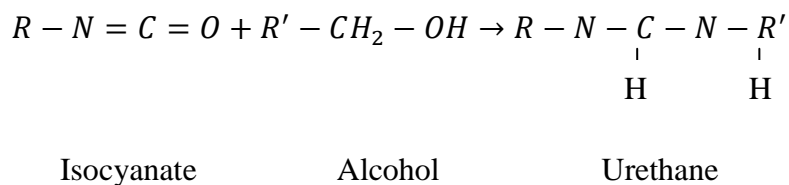


Figure 2.2: Gelation Reaction or PU Cross Linking Reaction (Kaushiva, 1999)

The chemical reaction between polyol and isocyanate is called gelation reaction (Kaushiva, 1999). It is an exothermic process for which heat of reaction had been reported approximately 24 kcal/mol of urethane (Klempner and Sendjarevic, 2004). Kaushiva (1999) noticed another chemical reaction occurred during foam generation. It is called blowing reaction (Kaushiva, 1999). The reaction illustrates in Figure 2.3. In this reaction, water reacts with isocyanate to produce carbon dioxide (CO<sub>2</sub>) which diffuses to the existing gas bubbles in the polyol and so expands the foam. Heat would generate in the mixture and it plays a large role on expanding the gas into the liquid to foam a desired cellular-structure. It was reported that the internal temperature of foam build up would build upon the order of 140 °C during the foam generation (Klempner and Sendjarevic, 2004).

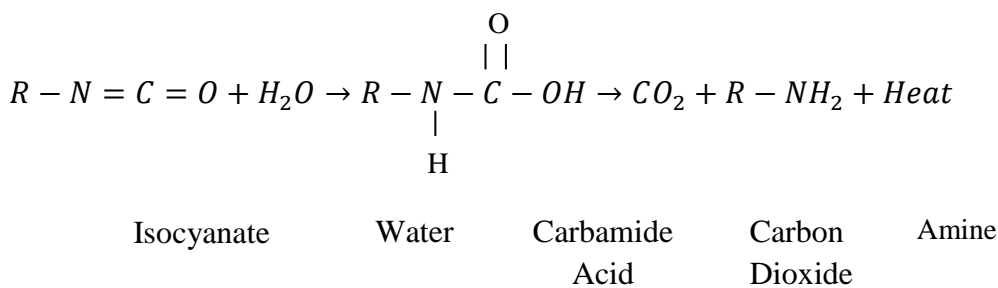


Figure 2.3: Blow Reaction between Isocyanate and Water (Kaushiva, 1999)

Other components in formula are working as surfactants such as fumed silica, dust wood, calcium carbonate are necessary components in foam formation in order to produce a well open-celled morphology. They perform to reduce surface tension in PU, emulsifying in compatible ingredients, promoting bubble nucleation, stabilizing the rising foam, thus reducing the deformation effects and the most important is to stabilize the cell wall. Klempner and Sendjarevic (2004) found surfactant prevents the coalescence of rapidly growing cells until those cells have attained sufficient strength through polymerization to become self supporting. Without this, cell coalescence would lead to a total foam collapse. In book Klempner and Sendjarevic (2004), catalyst is generally used in foam formulation and blow reaction. Besides, it is also for assuring completeness of reaction or “cure” in finished foam.

In addition, cellular structures in which adjacent cells are separated by cell walls or faces are referred to as closed cell foams, those with permeable cells as open-cell foams. There is an essential difference between open and closed cell structures concerning their mechanical behaviour. The open-cell structures are composed of a network of connected struts and therefore the main deformation mechanism is bending with additional extension and compression of the cell edges (Konstantinidis, I. C *et al.*, 2009). The closed cell structures exhibit cell walls between the cell edges that stiffen the structure. Membrane stresses in the cell walls also play a major role in the deformation mechanical. Owing to higher constraints arising from the existence of cell walls in closed cell structures, their Young's modulus is higher by several magnitudes compared with open-cell structures of the same relative density (Konstantinidis, I. C *et al.*, 2009).

### **2.3.1 Formation of foam**

Normally, most foamed polymers are produced by dispersing a gas throughout a fluid polymer phase and stabilizing the resultant foam (Klempner and Sendjarevic, 2004). Before stabilize the system, the foam is expanded by increasing the bubble size. There are many sources of gases may be involved in foaming process such as air from environment, gas such as from carbon dioxide, a low boiling liquid or gas may be generated by a chemical reaction (Klempner and Sendjarevic, 2004).

The preparation of foams by the dispersion process generally involves three steps as followed (Klempner and Sendjarevic, 2004):

- Step 1: Bubble formation. The first step in producing foam is formation of gas bubbles in a liquid system. The bubbles will usually form more easily at the liquid-solid interface
- Step 2: Bubble growth. Once a bubble was formed, it may grow by diffusion of gas from solution in the liquid phase into the bubble
- Step 3: Stabilization. There are many factors that affect the stability of bubble which are pure liquids without surface tension. Besides, a surfactant may be most effective for stabilization if it is design. Lastly, the temperature can also affect stability. An increase the temperature also increases the reaction

rates as well as reduces both viscosity and surface tension

### 2.3.2 Production of foam

According to the Sung *et al.*, (2007), flexible PU foam is produced by one-shot and free rise method. It means that the isocyanate, polyol, water and other ingredients are rapidly and intensively mixed and immediately poured to carry out the foaming. The method indicates that once the process is started, the formation of foam is exothermic until they are complete.

Basically, flexible PU foams are made through two types of fabrication process which are slabstock foam and moulded process (Polyurethane Foam Association, 1991). Slabstock foams are used for cushioning (Polyurethane Association, 1991) and in furnishing industry such as mattress and carpet backing whereas moulded foam are largely used in transportation application such as automotive seating (Zhang, 2008). For slabstock foam, two basic procedures are used. The chemical mix is poured onto the moving conveyor, where it is allowed to react and expand. The conveyor allow the foam to rise into a “bun” or slab anywhere from two to four feet high. The continuous slab is then cut, stored, and allowed to cure for up to 24 hours. The slabstock foam is performed in an open environment (Klempner and Sendjarevic, 2004). However, for moulded foams, is a process where individual items are produced by pouring foam chemicals into specially shaped moulds and allowing the foam reaction to take place. It is also call closed mould process (Klempner and Sendjarevic, 2004).

Normally, for the small or laboratory scale production, the flexible PU foams could be made as a simple hand or cup-foam mixes to techniques of box-foaming mixes (Klempner and Sendjarevic, 2004). This is also called free rise-bun. Generally, the methods to make foams in laboratory scale are follows (i) the components lists in recipe are weighed manually according to the proportion need, (ii) they are mixed using the mechanical device according to the sequence of mixture, (iii) after a mixing, the mixtures were poured into a cup or mould prepared and (iv) the flexible foam is moving out from the mould or cup and leaving for cured after a short period (Klempner and Sendjarevic, 2004). Table 2.2 is listed parameters used for flexible PU foam composites production from previous works.

Table 2.2: Parameters Use for Flexible Foam Composites Production

No	Components Mixing	Foam cured	Author	Year
1	<ul style="list-style-type: none"> <li>i. Polyol and fillers were stirred until completed homogenous</li> <li>ii. 1 minutes for mixtures and mechanical stirred when added amine, surfactants and water</li> <li>iii. Isocyanate was introducing it mixtures and stirred for 6 seconds (s)</li> <li>iv. Catalyst were added and stirred for 30 seconds (s)</li> </ul>	Leaving the foams to cure for three days	Sant'Anna., <i>et al</i>	2008
2	<ul style="list-style-type: none"> <li>i. Mechanical stirring of the polyol with water, silicone, catalyst and stannous octoate at 850 rpm for 1 minute.</li> <li>ii. Addition of toluene diisocyanate and methylene chloride to the polyol mixture and stirring for 5 s</li> <li>iii. For PU foams reinforced with PET (polyurethane terephthalate), the PET particles were added to the polyol and mixture was mechanically stirred for 15 s</li> </ul>	After foam development, the foam was demoulded and left to rest for 24 hour.	Mello., <i>et al</i>	2009
3	<ul style="list-style-type: none"> <li>i. Prior to synthesis of PU nanocomposite foams, the polyol was dehydrated under vacuum at room temperature.</li> <li>ii. For PU nanocomposites was first mixed with the polyol at 2000 rpm for 10 min using an over head stirrer equipped with a dispersion disc.</li> <li>iii. The surfactant, catalyst and distilled water were added to the CNT/polyol mixture and mixed at 2000 rpm for 1 min.</li> <li>iv. The isocyanate was added and stirred for 15 s before foaming occurred in an open cylindrical mould</li> </ul>	The foams were then transferred into an oven at 50 °C overnight and demoulded.	Verdejo., <i>et al</i>	2009

Table 2.2 (continued)

No	Components Mixing	Foam cured	Author	Year
4	<ul style="list-style-type: none"> <li data-bbox="395 353 951 472">i. The mixture of polyol with filler (calcium carbonate) was added to the content of the mixing chamber and stirred for 6 s.</li> <li data-bbox="395 488 951 651">ii. The surfactant, catalyst and water as the blowing agent were added to the mixture the polyol and filler in the mixing chamber and thoroughly premixed.</li> <li data-bbox="395 667 951 831">iii. The isocyanate was weighed directly into this mixture and the completed formulation was stirred with an overhead mechanical mixer for 4 s.</li> <li data-bbox="395 846 951 965">iv. The foam was poured in open mould which was treated with mould release agent to produce with free-rise foam</li> </ul>	After 10 min the foams were removed from the mould and left to cure for at least 7 days.	Lantiwo., <i>et al</i>	2010
5	<ul style="list-style-type: none"> <li data-bbox="395 1133 951 1200">i. Fumed silica was mixed with isocyanate using magnetic stirrer for 30 min.</li> <li data-bbox="395 1216 951 1379">ii. Polyester polyol, catalyst, blowing agent and surfactant were added and stirred using a mechanical stirrer at 500 rpm for 15 s.</li> </ul>	The mixture was poured in an open cylindrical mould and the foams were transferred into an oven at 50 °C for 3 h and then demoulded.	Ting., <i>et al</i>	2011
6	<ul style="list-style-type: none"> <li data-bbox="395 1458 951 1525">i. Different nanofillers were added in the polyol.</li> <li data-bbox="395 1541 951 1749">ii. The mixtures were initially sonicated for 20 min using an ultrasonication probe in water/ice bath and then were stirred using an overhead stirrer equipped with a dispersion disk for 6 h at 2400 rpm.</li> <li data-bbox="395 1765 951 1883">iii. The surfactant, catalyst and distilled water were added to this mixture and stirred at 2400 rpm for 3 min.</li> <li data-bbox="395 1899 951 1973">iv. Finally, the required amount of isocyanate was added and mixed again for 20 min.</li> </ul>	The foaming process occurred in an open mould	Bernal., <i>et al</i>	2012



## 2.4 Modification of polymer using fillers

Normally, fillers are solid additives and different from plastic matrices in composition and structure which are added to the polymers to raise bulk or improve the behaviour. Fillers usually applied as solid additives into the polymer to examine its physical and mechanical properties of material development (George, 2000). There are a lot of factors e.g. properties of fillers. Their fibres aspect ratio and particulate shape size, as well as filler-matrix interface would affect and govern the properties of composites material. In certain condition, air and other gases could be considered as fillers in cellular polymers. Generally, filler are defined as materials that are added to the formulation to reduce the cost.

Hence, it is important to have some basic knowledge to fillers use in any research. According to Lutz and Grossman (2000), each class of the fillers appears to exhibit specific characteristics to make them especially suited for the given application. Fillers being much stiffer and stronger than the polymer itself to increase its modulus and strength.

### 2.4.1 Composite foam and its properties

Flexible PU foams are one of the important classes plastic used in the manufacture of such materials as foam mattresses, pillow, furniture, cushioning materials for automobiles, packing, recreation, shoes and so on (Deng *et al.*, 2003; Mello *et al.*, 2009; Latinwo *et al.*, 2010 and Usman *et al.*, 2012).

In addition, PU rigid foams also have broad range of applications including thermal insulation, cushioning, buoyancy and energy absorption. PU rigid foams have low density permits the design of light and stiff compounds such as aircraft interior panels and structural shapes (Nikje and Tehrani, 2010), such as also epoxy foam. It exhibits excellent properties including low moisture absorption, low shrinkage, excellent adhesion onto all types of materials and high mechanical performance. Since early 50 s, epoxy foams have been applied to the fabrication of naval vessels, military vehicles, aircraft, building and offshore structure.

However, the discussion about rubber foam is not widely covered and few in numbers, but foamed rubber are globally manufactured and are in service all over the world (Lee and Choi, 2007).

Mello *et al.*, (2009) carried out post consumer plastic bottle waste PET (Polyethylene terephthalate), PET as reinforcement filler in flexible PU foams. The aim was to find alternatives for recycling of polymer packaging; reduce the cost material and getting a better mechanical properties from the new composite fabricated. The researchers were prepared a particulate shape in size of  $< 297 \mu\text{m}$  and it is added to PU at a concentration of 1.5 parts by hundred parts of polyol. They conducted some mechanical test such as tensile resistance, tear resistance and break strain (%) for testing in mechanical strength. The results shown by such testing showed that the mechanical performance of PET foams surpassed than standard foam for all layers (top, mid top, mid bottom and bottom).

The PET filled is also used to conduct the wear, compression strength and compression set tests. From the results obtained, a filled foam yield a better wear which has less mass losses during the wear test, a better compressive strength which indicated that PET particles can effectively absorb the compression energy and lower compression set test value.

According to the results obtained in Latinwo *et al.*, (2010) study, it was observed that filler material increased the hardness characteristics of the flexible polyurethane foam to compositions up to 35%. The researchers also found that the properties such as tensile strength and elongation at break, these properties goes down negatively when particle size of fillers were added as shown in Figure 2.4 and Figure 2.5. Among all, coarse filler filled composite obtained better tensile strength than fined filler filled composite. It is may due to fact of less surface area of coarse particles in disturbing the foam reaction (Latinwo *et al.*, 2010).

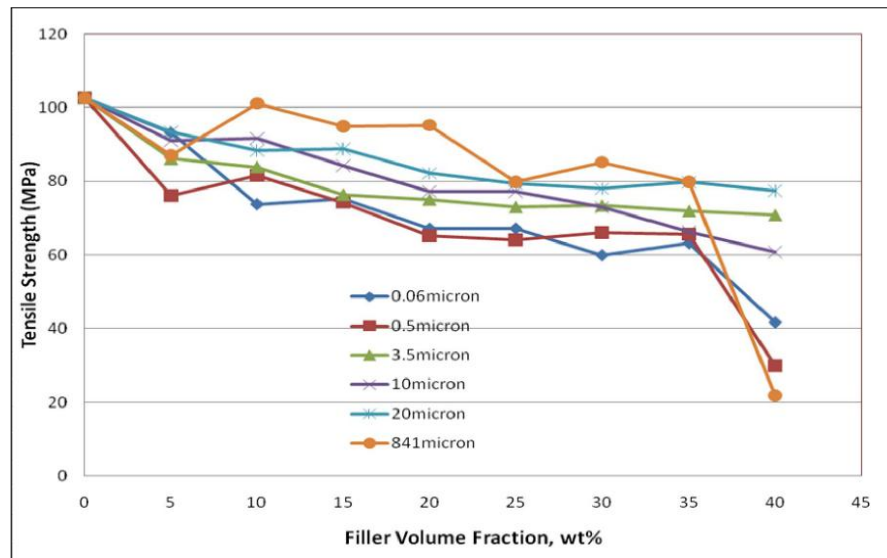


Figure 2.4: Effects of calcium carbonate of different particle sizes and compositions on the Tensile Strength of Flexible PU Foam (Latinwo *et al.*, 2010)

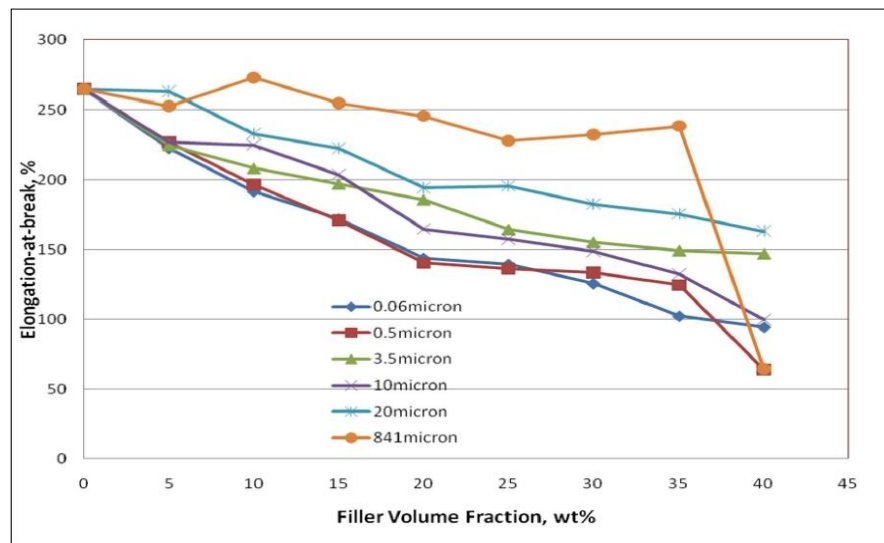


Figure 2.5: Effects of calcium carbonate of different particle sizes and composition on the Elongation-at-break of Flexible PU Foam (Latinwo *et al.*, 2010)

Recently, Usman *et al.*, (2012) has published a study related to the preparation, properties and cost in production of flexible PU foams filled with Calcium Carbonate ( $\text{CaCO}_3$ ). The purpose of their study was to achieve sustained mechanical properties and reduction in production cost. For this work, varying concentration of  $\text{CaCO}_3$  ranging from 0 to 30% introduced into the formulation. The

results indicated that, the density of the foam is improved as  $\text{CaCO}_3$  content increase to 30 %. Figure 2.6 (a) shown that it is gradually increase in density value until 20 % filler concentration before sharp rise in density afterwards. Figure 2.6 (b) shows that the elastic property of the foam was maintained between the normal ranges of 90-110  $\text{kN/m}^2$ . However, a sharp drop in the elastic property was experienced. Furthermore, in Figure 2.6 (c) the tensile strength of the foam was maintained until 20 % filler content (145-120 kPa) after there was a sharp and sudden decreased in tensile strength.

In addition, cost analysis of the cost production for formulation without filler (Sample A) and formulation with 20 % filler concentration (Sample E) is presented in Figure 2.7. Hence, the cost production was reduced by about 18 % while the foam qualities and properties were either maintained or improved upon as the  $\text{CaCO}_3$  filler was introduced into the foam formulation to an optimum 20 % content (Usman *et al.*, 2012).

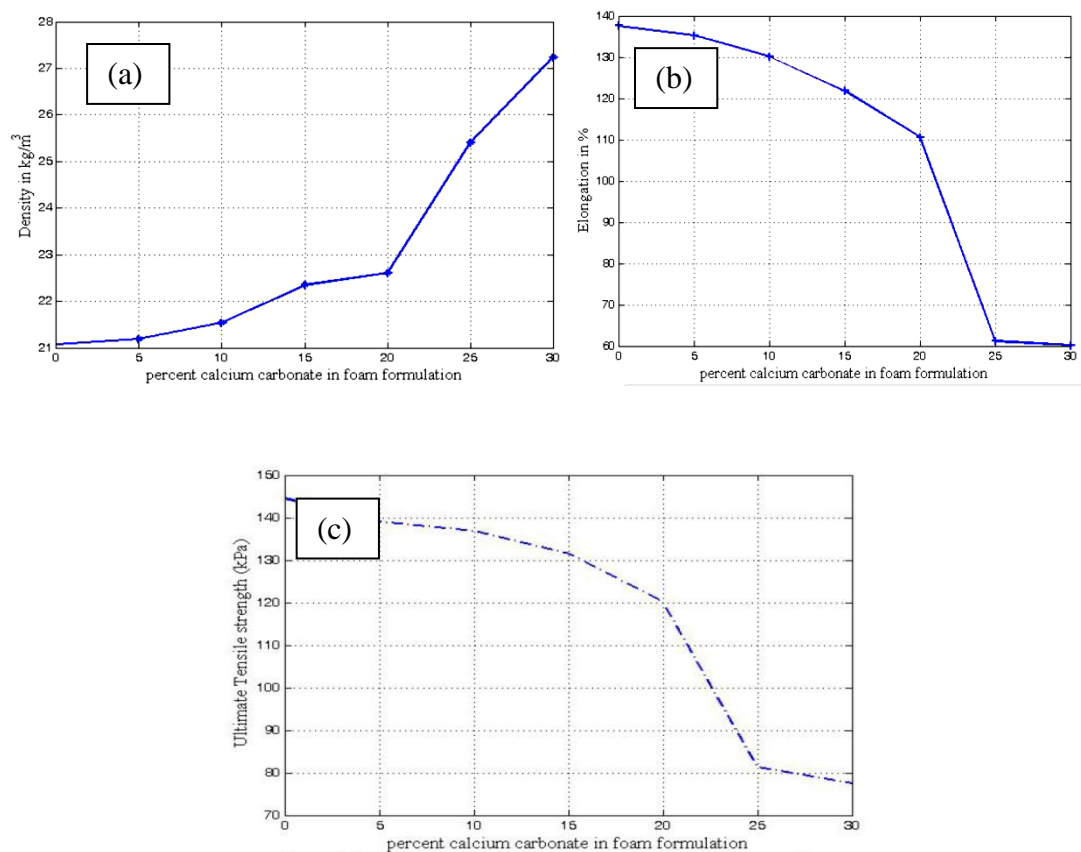


Figure 2.6: (a) density, (b) elongation and (c) tensile strength of foam with concentration of  $\text{CaCO}_3$  (Usman *et al.*, 2012)

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