

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PREPARATION AND CHARACTERIZATION OF GREEN RUBBER FOAM FROM RECLAIMED RUBBER GLOVE

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering

(Engineering Materials) (Hons.)

by

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APPROVAL

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ABSTRAK

Kajian ini mengkaji kesan natrium bikarbonat terhadap saiz busa getah yang diperbuat daripada sarung tangan getah yang dikitar semula. Kelebihan menggunakan sarung tangan getah yang dikitar semula boleh mengurangkan kos bahan mentah disamping sifat plastik yang baik dan mudah untuk dicampurkan dengan getah mentah serta keperluan pemprosesan pada suhu yang lebih rendah. Dalam kajian ini, natrium bikarbonat digunakan sebagai agen peniup dan kesannya terhadap busa getah diuji untuk sifat fizikal seperti ketumpatan, ketumpatan relatif, nisbah pengembangan buih, ketumpatan sambung silang, kadar penyerapan air, sifat mekanikal seperti ujian mampatan dan morfologi melalui kemikroskopan electron imbasan (SEM). Getah boleh kembang disediakan menggunakan pencampur dalaman Haake dibusakan melalui proses pindahan haba menggunakan pengacuan mampatan dan oven aliran udara panas. Dalam kajian ini, sifat-sifat fizikal busa getah dikaitkan secara sistematik dengan sifat mekanikal busa. Kekuatan mampatan busa didapati menurun dengan penurunan ketumpatan sambung silang dan ketumpatan relatif busa, yang dikaitkan dengan pembentukan saiz sel yang besar dan peningkatan dalam bilangan sel per unit isipadu. Kajian ini adalah kajian awal untuk potensi penggunaan busa getah dikitar semula sebagai bahan tapak kasut.

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ABSTRACT

This research studied the effect of sodium bicarbonate to foam size of rubber foam from reclaimed rubber glove. The advantages of using reclaimed rubber glove are reducing cost of raw materials, a part of good plasticity, easy to be mixed with raw rubber and requirement of low processing temperature. In this research, sodium bicarbonate was used as blowing agent to evaluate the effect of rubber foam on the physical properties such as density, relative density, expansion ratio, crosslink density, water absorption rate and mechanical properties such as compression strength and morphology through scanning electron microscopy (SEM). The expandable rubber samples were prepared using a Haake internal mixer and then expanded via heat transfer foaming process using a compression molding and an air circulating oven. In this study the physical properties of rubber foams were systematically correlated with its mechanical properties. The compression strength of the foam decreases with decreasing crosslink density and relative foam density which was associated with the formation of bigger foam cells and an increase in the number of cells per unit volume. This study is a preliminary study for the potential use of recycled rubber foam material as shoe insoles.

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LIST OF ABBREVIATIONS

ASTM	-	American Standard Test Method
TPNR	-	thermoplastic natural rubber
NR	-	natural rubber
SEM	-	scanning electron microscopy
TPE	-	thermoplastic elastomer
BR	-	polybutadiene rubber
SBR	-	styrene butadiene rubber
RR	-	reclaimed rubber
CIIR	-	chloro butyl rubber
BIIR	-	bromo butyl rubber
DOE	_	design of experimental
Rpm	-	rotation per minute
OM	-	Optical microscope
TMTD	-	Tetramethylthiuram-disulfenamide
CBS	-	Benzothiazyl-2-cyclohexyl-sulphenamide

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LIST OF SYMBOLS

oC	-	Celsius
M/S	-	meter per second
%	-	percentage
kW	-	kilo watt
min	-	minute
kg	-	kilogram
mm	~-	millimeter
μ m	-	micrometer
S	-	Second
nm	-	nanometer
g	-	Gram
Hz	-	hertz

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CHAPTER 1 INTRODUCTION

1.1 Background

In industrialized countries, rubber products are everywhere to be found, though few people recognize rubber in all of its applications. Since 1920, demand for rubber manufacturing has been largely dependent on the automobile industry, the biggest consumer of rubber products. Rubber forms a part of many mechanical devices in the kitchen, helps to exclude draughts and to insulate against noise. Sofas and chairs may be upholstered with foam rubber cushions, and beds may have natural rubber pillows and mattresses. Clothing and footwear may contain rubber for example elasticized threads in undergarments or shoe soles. Still other applications have been developed due to special properties of certain types of synthetic rubber, and there are now more than 100,000 types of articles in which rubber are used as a raw material (Pothen 2011).

The use of rubber in so many applications results in a growing volume of rubber waste. With the increase in demands, the manufacturing and use of rubber and the rubber products has increased tremendously both in the developed and less developed countries. By the middle of 1980s less than 1% of the worldwide polymer consumption was in the form of reclaim. At the beginning of 20th century half of the rubber consumed was in the form of reclaim. It is expected that in 21st century most of the scrap rubber will be recycled in the form of reclaim because of day to day increase in environmental awareness. One of the various problems which mankind faces as it enters into the 21st century is the problem of waste disposal management.

Since polymeric materials do not decompose easily, disposal of waste polymers is a serious environmental problem. Large amounts of rubbers are used as glove and tires for airplanes, trucks and car. Reclaimed rubber is the product resulting when waste vulcanized scrap rubber is treated to produce a plastic material which can be easily processed, compounded and vulcanized with or without the addition of either natural or synthetic rubbers. Reclaiming of the waste rubber can cost half that of natural or synthetic rubber, some properties that are better than those of virgin rubber, requires less energy in the total production process than does virgin material and it is an excellent way to dispose of unwanted rubber products, which is often difficult.

Although reclaim rubber is a product of discarded rubber articles it has gained much importance as additive in various rubber article formulations. It is true that mechanical properties like tensile strength, modulus, resilience and tear resistances are all reduced with the increasing amounts of reclaim rubber in fresh rubber formulation. But at the same time the reclaim rubber provides many advantages if incorporated in fresh rubber. The increase in the awareness of waste management and environment related issues has led to substantial progress in the utilization of rubber waste. Recycling materials back into its initial use often are more sustainable rather than finding new applications.

1.2 Problem statement

Various shoe sole in the market nowadays made from simple, single materials in a single layer or they can be complex with multiple structures or layers and materials. Each of its show their unique properties on their application of shoe sole which have their

advantages and disadvantages for the customer. According to the domestic waste, recycling of rubber waste poses a challenging environmental, economic and social problem. The latex industry has expanded over the years to meet the world demands for gloves, tire, latex thread and others. This industry has always been at the mercy of rapid and drastic changes, both in the cost of raw rubber and the prices of finished goods. The waste rubber formed in latex-based industries is around 10-15% of the rubber consumed. Domestic and contaminated industrial gloves that do not require specific handling are disposed along with other household wastes as non-hazardous municipal solid wastes into domestic landfills. In a landfill, residual chemicals example accelerators will leach out as the rubber biodegrades. Under standard landfill conditions, vinyl is not biodegradable but the plasticizers will leach out from the material when in contact with non-aqueous solvents. Nitrile itself is not biodegradable and the chemical by-products leaching out will be similar to those produced by NR gloves. NR latex gloves and other rubber products containing sulphur liberates sulphur dioxide and water when incinerated and the remaining carbon backbone oxidised to carbon dioxide in an aerobic environment.

The formation of a higher percentage of waste latex rubber (WLR) in latex factories is due to the unstable nature of the latex compound and the strict specifications in the quality of latex products. As waste latex rubber (WLR) represents a source of highquality rubber hydrocarbon, it is a potential candidate for generating reclaimed rubber of superior quality (Abraham, Thomas 2011). Rubber sole shows good properties in terms of lightweight, durable, flexible and can reduce shock force to the feet. Hypothesis that reclaimed rubber it will able to show similar performance in term of shoe sole as replacement for pure synthetic or natural rubber. The usage of reclaimed rubber is aimed to reduce the effect on environmental problem and at the same time produce shoe sole with significant properties at cheaper cost.

1.3 Objective

The main objectives on this research are:

- i. To prepare rubber foam from reclaimed rubber glove through melt compounding using internal mixer.
- ii. To study the effect of sodium bicarbonate loading as blowing agent in physical properties and mechanical properties.
- iii. To characterize the physical properties and mechanical properties of rubber foam made from reclaimed rubber glove.

1.4 Scope

This research is to study the effect of sodium bicarbonate loading as blowing agent to prepare the rubber foam from reclaimed rubber glove through melt compounding using internal mixer followed by various physical and mechanical testing. Then characterize the properties of rubber foam made from reclaimed rubber glove. Some analysis such as morphology was performed to support the data.

1.5 Chapter Overview

There are five chapters in this report where for the Chapter 1 that is the introduction of the research. This consists of research background, a problem statement, and objectives of the project, scope and chapter overview. Next its continue for the Chapter 2 that is the literature review and covers the fundamental of shoe sole, elastomer foam, reclaimed rubber glove and also a general overview of the current development of shoe sole in the market. Then for the Chapter 3 there is the methodology of this research, response surface methodology and it discuss the raw material specification, equipment and experimental procedures used in this study. After that, for the Chapter 4 there is results and discussion of the data. Finally, Chapter 5 is formulate procurement review and list of potential research and also proposed future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Shoe Sole

A shoe is an item of footwear intended to protect and comfort the human foot while doing various activities. Shoes are also used as an item of decoration. The design of shoes has varied enormously through time and from culture to culture, with appearance originally being tied to function. Shoes have traditionally been made from leather, wood or canvas, but are increasingly made from rubber, plastics, and other petrochemical-derived materials. The parts of a shoe are pretty common, regardless of the specific style of footwear. All shoes have a sole, which is the bottom of a shoe, which is in contact with the ground. Soles can be made from a variety of materials, although most modern shoes have soles made from natural rubber, polyurethane, or polyvinyl chloride (PVC) compounds. Soles can be simple, a single material in a single layer or they can be complex, with multiple structures or layers and materials. When various layers are used, soles may consist of an insole, midsole, and an outsole.

The insole is the interior bottom of a shoe, which sits directly beneath the foot under the foot bed. The purpose of insole is to attach to the lasting margin of the upper, which is wrapped around the last during the closing of the shoe during the lasting operation. The outsole is the layer in direct contact with the ground. Dress shoes often have leather or resin rubber outsoles; casual or work-oriented shoes have outsoles made of natural rubber or a synthetic material like polyurethane. The layer between the outsole and the insole in shoe is typically the function for shock absorption. Some types of shoes, like running shoes, have another material for shock absorption, usually beneath the heel of the foot, where one puts the most pressure down. Different companies use different materials for the midsoles of their shoes. Some shoes may not have a midsole at all. Most types of shoes are designed for specific activities. For example, boots are typically designed for work or heavy outdoor use. Athletic shoes are designed to be worn at more formal occasions, and others are designed for casual wear (James 2011). Figure 2.1 shows the example of shoe sole in shoes.

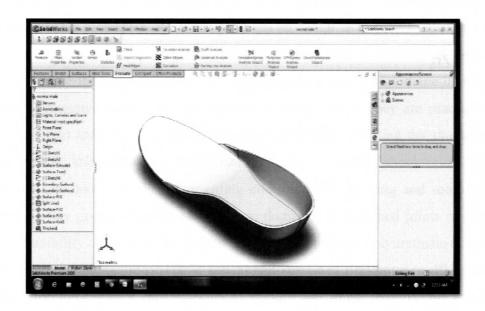


Figure 2.1: Example of shoe sole in shoes view from solid work

2.1.1 Type of shoe sole in the market

There are several types of soft shoe sole available in the market. They all facilitate foot development and walking, but differ in terms of sole construction and materials and specific benefits. These soles also are available in different sizes and thickness. The sole of the shoe should be thick enough for protection while allowing the foot to move as naturally as possible. For a shoe manufacturer it can be a challenge to meet the varied demands that consumers place on their shoes, but there is one special polymer that meets these many demands and is so extraordinarily versatile that it is now the heart and soul of good, modern shoe technology (James 2011). There are different types of soles in the market that are PU (polyurethane) Sole, TPR sole and Leather sole.

Polyurethane (PU) is lightweight and extremely durable. With slight changes in its chemical properties, it makes products as tough as the outsole on a safety shoe, as springy as the cushioning in a cross-trainer, or as striking and versatile as synthetic leather. PU double density safety shoes use polyurethane in both the hard, protective outsole and the soft, cushioning insole. They are practical and lightweight and offer a range of advantages including reducing the shock forces to the feet, knees, hips and lower back, reducing the risk of shock-related injury to the feet and lower back, providing comfort by cushioning and soothing the feet all day giving ongoing protection to damaged or inflamed joints improving the flexibility of the sole. In fashion and casual footwear, polyurethane offers the dynamite combination of visual appeal and sustained performance. PU outsoles have the light weight and high-quality texture and finish required for a fashion shoe, but they also outperform other materials in durability and resistance to oil and abrasion. Polyurethane is highly valued in the sports shoe industry because of the ease with which it can be molded, and it provides great cushioning, durability and flexibility.

Thermo Plastic Rubber, TPR is another type of shoe sole in the market. TPR is the combination of PU and rubber. Thermo Plastic Rubber soles are produced by compounding and molding rubber granules of solid raw material. Rivers use only the highest quality materials in their TPR Soles. They do not use shredded old car tires which is common in this type of sole. TPR is renowned for its slip resistance and shock absorption. In footwear industry, TPR is used in the production of shoes soles, safety shoes sole and industrial shoes sole, sports shoes sole, ski-boot soles, kiddy shoes sole and related decorative accessories, modification modifier for SMC (sheet molding compound) and other thermoset and thermoplastic composites.

Leather shoe sole is actually a car to carpet concept sole, we cannot use it daily and is not for rough use and it's using for Brogue models shoes. The properties of leather shoe sole are made from best quality leather, offers premium safety against several hazards, unique design and durable. Shoe sole leather has a good capacity for withstanding the deformations of abrasion, compression, and bending, and it must also maintain its linear dimensions upon wetting and subsequent drying. Leather shoe sole must be sufficiently dense to retain the thread tacks after part of the stitches have been abraded away on the walking surface of the sole. Leather with greater plasticity is selected for sewn methods of fastening, since excessive stiffness makes the manufacturing process difficult and the threads wear out quickly during use.

2.1.2 Design of shoe sole in the market

A shoe consists of two primary components that are the sole and the upper. The upper covers the dorsal, medial, and lateral aspects of the foot, and the sole provides stability and cushioning at the interface between the ground and the plantar surface of the foot. The shoe upper consists of toe box, heel counter, entry opening, tongue, and quarter. The sole is divided into components that are designated as anterior sole, mid sole or brand sole, and outer sole. A foot model made of wood or plastic, which is used for production of the shoe in the desired size and form, is referred to as a last. Stress on cartilage, ligaments, bones, and tendons of the lower extremity are independent of, or only minimally affected by the cushioning property of the shoe sole. The term comfort is associated with both the fit of a shoe and the extent to which the shoe supports the natural motion of the foot.

Based on recent findings, the two biggest trends in shoe sole design are individualization of fit and force generation that is similar to barefoot walking on a natural ground (Hilgers 2003). Various companies make application of these concepts in different ways. Their four top-selling models are available in four different widths for each shoe sole size where width individualization is limited to the forefoot, the midfoot and hindfoot are identical in all four models. Other companies offer as many as three widths for certain models due to costs associated with longer shelf time, this fit individualization concept has not been readily accepted by retail stores that sell the shoes sole. Figure 2.2 shows the shoe sole design in market nowadays.

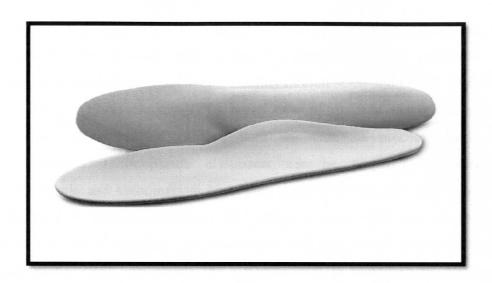


Figure 2.2: Example of shoe sole in the market

2.2 Current materials for shoe sole

Nowadays, current materials for shoe sole are made from thermoplastic elastomer, TPE(s), carbon fiber reinforcement plastic, CFRP and elastomer. Most of the shoe sole in the market are preferred from rubber materials because the properties of rubber as a shock absorption is very good and also the price is available to all.

2.2.1 Thermoplastic elastomer (TPEs)

Holden (2000) and Walker (1986) have both shown that thermoplastic elastomers (TPEs) have become a technologically important class of material in recent past years. TPEs have many of the elastomeric physical properties of rubbers such as softness, flexibility, resilience but they are processable as thermoplastics. TPEs can be able to process using conventional thermoplastic

processing techniques such as injection molding, extrusion. TPEs can also be completely reprocessable without any considerable loss in properties or processing characteristics due to recyclability of their scrap and rejects. Abdullah and Dahlan (1998) classify TPEs are a new of material that combine the properties of vulcanized rubbers with the ease of process ability of thermoplastics Thermoplastic elastomers can be prepared by blending thermoplastic and elastomers at high shear rate. Thermoplastics, for example, polypropylene (PP), polyethylene (PE) and polystyrene (PS), and elastomers, such as ethylene propylene diene monomer (EPDM), natural rubber (NR) and butyl rubber (BR), are among the materials used in thermoplastic elastomer blends.

A hard thermoplastic phase is combined with a soft elastomer phase, and the properties of the resulting TPE will be derived from the properties of each of the two phases and from the interaction between these phases. The two phases may result from simple mixing of two different polymers, as in a blend of a hard thermoplastic with a soft elastomer (Arnold & Rader, 1992). The hard phase gives these TPEs their strength and the elastomeric phase provides elasticity and flexibility to the system. Arnold and Rader (1992) state that the performance characteristics of a TPE depend on the melting point (Tm) of the hard thermoplastic phase and the glass-transition temperature (Tg) of the soft elastomeric phase. The useful temperature range of a TPE is between Tm and Tg and the TPE displays its desirable elastomeric phase melts and the TPE becomes fluid and can be processed by usual thermoplastic techniques. Below Tg the TPE becomes brittle and loses all of its useful elastomeric properties. A list of common thermoplastic elastomers used in modern world is given in Table 2.1

		Amorphous		Crystalline
	PVC	Poly Vinyl Chloride	DEPE	Poly Ethylene High density
Low Cost	SAN	Styrene Acrylo Nitrile	LDPE	Poly Ethylene High density
	PS	Poly Styrene	PP	Poly Propylene
	PMMA	Poly Methyl Metha		
	ABS	Acylonitrile		
		Butadiene Styrene		
	SMA	Styrene Maleic		
		Anhydride		
	PPO	Poly Phenlene Oxide	UHMWPE	Ultra High Molecular
Medium Cost	PC	Poly Carbonate		Poly Ethylene
	PPC	Poly Phthalate	POM	Poly Oxy Methylene
		carbonate	PA	Poly Amide
	PTFE	Poly Tentra Fluoro	PBT	Poly Butylene
		Ethylene(Teflon)		Terephthalate
			PET	Poly Ethylene
				Terephthalate
	PAR	Poly Arylate	PA-4,6	Poly Amide-4,6
High Cost	PES	Poly Ether Sulfone	PPA	Poly phthal Amide
	PEI	Poly Ether Amide	PPS	Poly phenylene sulfide
	PPSU	Poly Phenyl sulfone	LCP	Liquid Crystal Polymers
	TPI	Thermoplastic	PVDF	Poly Vinyl Diene Fluoride
		polyimide	FP	Fluoro Polymers
	PAI	Poly Amide Imide	PEEK	Poly Ether Ether Ketona

Table 2.1: Common thermoplastic elastomeric materials

The properties and characteristics of TPEs are strongly dependent on constituents of the TPE, molecular weight, relative amount of hard and soft segment and processing history. The changing of hard segment will influence the crystalline of the materials, similarly the soft segment type influences on the phase separation and hence physical properties of the final. TPEs are most used as shoe sole because it lightweight, good of energy absorption and cheaper.

2.2.2 Carbon Fibre Reinforcement Plastic (CFRP)

Carbon-fiber-reinforced polymer or carbon-fiber-reinforced plastic (CFRP) is an extremely strong and light fiber-reinforced polymer which contains carbon fibers. The polymer is most often epoxy, but other polymers, such as polyester, vinyl ester or nylon, are sometimes used. The composite may contain other fibers, such as Kevlar, aluminum, or glass fibers, as well as carbon fiber. The strongest and most expensive of these additives, carbon nanotubes, are contained in some primarily polymer baseball bats, car parts, insole in shoes and even golf clubs where economically viable.

Carbon fibre reinforced plastic (CFRP) insoles may also be manufactured to provide a lightweight, space-saving device which is of increasing importance in many sports. For example, football boots are evolving rapidly, with new designs offering slim fitting and lightweight products which often contribute to the development of bony growths such as osteophytes, possibly as a consequence of poor boot design and protection. Many devices simply cannot fit into current boot designs and even if they could, they would have a large impact on the overall weight of the boot and so a CFRP insole provides a slimmer profile due to its stiffness and rigidity.

2.2.3 Elastomer

According to Fisher (1939) state that terminology of elastomer is defined by as a natural or synthetic product that can be vulcanized, where it shows elastic behavior after crosslinked and could be stretch at least double the original length at room temperature and will return to original length after release it (Heinisch,

1966). Elastomers are rubbery polymers that can be stretched easily to several times their unscratched length and which rapidly return to their original dimensions when the applied stress is released. Elastomers are crosslinked, but have a low cross-link density. The polymer chains still have some freedom to move, but are prevented from permanently moving relative to each other by the crosslink. According to Callister (2007), elastomer is a polymeric material that may experience large and reversible elastic deformations.

2.2.3.1 Type of elastomer

Elastomer can be divided into natural rubber and synthetic rubber. Natural rubber are found in many forms such as horns of animals, tortoise shell, shellac, rosin (from pine trees), asphalt, and tar from distillation of organic materials. One of the most useful of the natural polymers was rubber, obtained from the sap of thehevea tree. Natural rubber had only limited use as it became brittle in the cold and melted when warmed. In 1839, Charles Goodyear discovered, through a lucky accident, that by heating the latex with sulfur, the properties were changed making the rubber more flexible and temperature stable. That process became known as vulcanization (David 1998).

Although many polymers were made in the following years, the technology to mass produce them was not developed until World War II, when there was a need to develop synthetic rubber for tires and other wartime applications and nylon for parachutes. Since that time, the polymer industry has grown and diversified into one of the fastest growing industries in the world. Today, polymers are commonly used in thousands of products as plastics, elastomers, coatings, and adhesives (Galimberti, 2011).

Synthetic rubber is any type of artificial elastomer, invariably a polymer. An elastomer is a material with the mechanical (or material) property that it can undergo much more elastic deformation under stress than most materials and still return to its previous size without permanent deformation. Synthetic rubber is made by the polymerization of a variety of petroleum-based precursors called monomers. The most prevalent synthetic rubbers are styrene-butadiene rubbers (SBR) derived from the copolymerization of styrene and 1,3-butadiene. Other synthetic rubbers are prepared from isoprene (2-methyl-1,3-butadiene), chloroprene (2-chloro-1,3-butadiene), and isobutylene (methylpropene) with a small percentage of isoprene for cross-linking. These and other monomers can be mixed in various proportions to be copolymerized to produce products with a range of physical, mechanical, and chemical properties. The monomers can be produced pure and the addition of impurities or additives can be controlled by design to give optimal properties (Mayers, 1996).

2.2.3.2 Reclaimed rubber

Reclaimed rubber produced from the process of de-vulcanization (recycling) of rubber scrap and waste rubber. In the reclaim rubber process de-vulcanization begins with the delinking of the sulfur elements from the rubber elements, therefore reclaiming agent, and processing oil are added in the crumb rubber at the high temperature and pressure, thereby enabling the adjectives of either natural or synthetic rubber to be reproduced (Jean 2002). In the rubber recycling process by the conventional pan method, finely ground rubber powder mixed with oils and reagents is heated with steam in a pressure vessel at temperature of ~ 200 °C for more than 5 hours .Moreover, usually this process has to be followed by several procedures (refining and straining) before obtaining the final reclaimed rubber. The reclaimed rubber obtained by this method is inferior in

quality to virgin rubber. This is due to the occurrence of unselective breakage of both the crosslinking points and main chain (C-C) bonds in the rubber.

For motor vehicles, many crosslinked rubber products are used as weather strips, hoses, vibration insulators and miscellaneous parts, except for tires, as shown in Figure 2.2 (Otsuka, 2000). Table 2.2 show the technical specification of reclaimed rubber that from different type of reclaimed rubber Superfine- HR reclaim rubber, Fine-111 reclaim rubber, Medium202 reclaim rubber , Coarse-303 reclaim ruber. In the newly developed continuous recycling process various chemical reactions corresponding to selective breakage of crosslinking points (socalled, devulcanization) can be efficiently controlled by optimizing the parameters in the reactor such as shear stress, temperature, and internal pressure (Fukumori, 2001).

The continuous recycling process for crosslinked rubber waste is performed in a modular screw type reactor as schematically shown in Figure 2.3. Change in the network structure during the recycling process under suitable process conditions (i.e., screw configuration, reaction temperature, screw rotation speed, etc.), reclaimed EPDM with good surface appearance can be continuously obtained from the head of the reactor, as shown in Figure 2.4. This was closely connected with the values of the Mooney viscosity for the rubber polymer. Properties of reclaimed rubber and its practical uses for automotive rubber products the reclaimed EPDM was compounded a crosslinked with an accelerated sulfur vulcanization system according to the conventional recipe. Figure 2.5 shows stress-strain curves for a reclaimed rubber from the developed method and for a virgin rubber. The reclaimed rubber exhibits an excellent tensile tress-strain property, almost comparable to the virgin rubber.

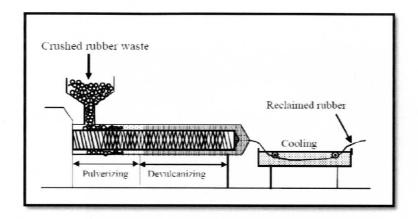


Figure23: Schematic illustration of the reactor for the product of reclaimed rubber

Type of Reclaim Rubber	Superfine- HR Reclaim Rubber	Fine-111 Reclaim Rubber	Medium-202 Reclaim Rubber	Coarse-303 Reclaim Rubber
Chemical	Juan - noutra			
Properties				
Ash (%)	7±2	7±2	7±2	7±2
Carbon Black (%)	27±3	27±3	27±3	27±3
Acetone Extract (%)	15±3	15±3	15±3	15±3
RCH Different (%)	47 min	47 min	47 min	47 min
Physical				
Properties				
Specific Gravity (%)	1140±20	1140±20	1140±20	1140 ± 20
Tensile Strength (Kg/cm ²)	40 min	40 min	35 min	25 min
Elongation Break (%)	240 min	220 min	200 min	180 min
Hardness (Shore A)	59±3	59±3	59±3	59±3
Mooney Viscosity at 100 ° C	25-45	25-45	25-45	25-55

Table 2.2: Technical specification of reclaimed rubber

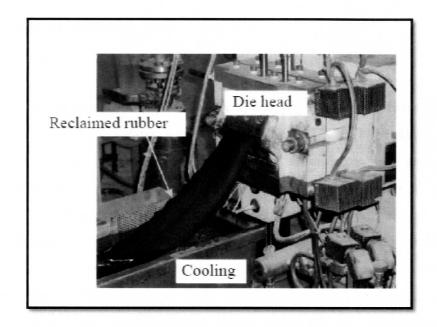


Figure 2.4: Continuous recycling process for the product of reclaimed rubber.

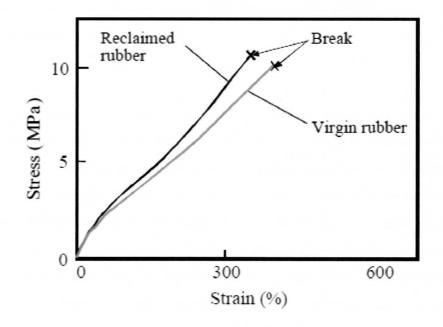


Figure 2.5: Stress-strain curves of crosslinked rubbers.

2.2.3.3 Elastomer/Rubber foam

Foams rubber refers to rubber that has been manufactured with a foaming agent to create an air-filled matrix structure (Galimberti, 2011). Nowadays, rubber foam have long been considered as an economical material for many applications due to their wide variety of advantageous properties such as light weight, buoyancy, cushioning performance, acoustic insulation and good aging. Generally rubber foam consist minimum of two phases, a solid polymer matrix and a gaseous phase derived from blowing agents. Rubber foam also known as sponge rubber which has open cell structure and usually contain sodium bicarbonate as their blowing agent. Rubber foam can be manufactured via expansion during single stage or in two phase heat transfer process. Basically the first stage of the heat transfer is identically to single stage process but opening the expandable compound is immediately transferred to a circulating hot air oven at a higher temperature.

The processing temperature is another important parameter that effects on the final foam structure and subsequently affects the mechanical properties. Increase in the temperature caused reduction in the overall crosslinking density thus larger cells produced at this temperature. The compressive behavior of rubber foam can be determined using universal testing machine. In the case of compression set, it was found that the recovery of the foam was clearly a function of strain and independent of foam density (Zunaida *et al.*, 2007). Most of rubber foam has been derived from the desire to combine its low density with some other physical properties. Although foam rubber is widely manufactured and is in service all over the world, its electrical, mechanical and physicomechanical properties are few in number.

2.3 Elastomer based sole

Elastomers are long polymer chains above their glass transition temperature. Elastomers are usually lightly cross-linked and are easily deformed. Common elastomers include polybutadiene (used in shoe soles), polyisobutylene (used in automobile tires), and polyisoprene (natural rubber). Elastomeric polymer chains can be cross-linked, or connected by covalent bonds. This process is sometimes called vulcanization. Crosslinking is initiated by heat, light, or the addition of chemicals. The demand of the sole is increasing rapidly as shoe users are growing in the country every day. The manufacturers are very much interested to attach the sole with upper portion of the shoes. As the name indicates the soles are made with rubber compound and rubber sole is increasingly popular due to its cheapness and comfort in uses (Kear, 2003).

2.3.1 Type of elastomer foam materials

Foam is a substance that is formed by trapping pockets of gas in a liquid or solid. A bath sponge and the head on a glass of beer are examples of foams. In most foam, the volume of gas is large, with thin films of liquid or solid separating the regions of gas. There are many different types of foam rubber, and they have endless uses. A large amount is used in consumer products such as furniture, cars, packaging and sole in shoe. Foam rubber refers to rubber that has been manufactured with a foaming agent to create an air-filled matrix structure. Commercial foam rubbers are generally either polyurethane foam or natural foam rubber latex (Kear, 2003).There are several types of specialized foam rubber that is usually denser than typical polyurethane and can be more elastic, resistant to damage and useful in specific situations. Charles (2002) state that a shoe sole composed of a laminate of an intermediate sole formed by cushioned

foam and an outside sole formed by a semi-hard rubber having a hardness of 50^{0} C to 70^{0} C in order to improve impact absorption. A shoe sole material obtained by foaming and curing a composition prepared by blending a polymer with syndiotactic 1,2-polybutadiene having a hardness of 98 or more and a melting point of 110^{0} C or higher and cis 1,4-polybutadiene.

2.3.2 Elastomer foam properties

A foam rubber shoe has flexible and cushioned foam rubber for the sole or upper part of the shoe. Over time, the foam rubber can crack from frequent use and over-flexing of the shoe. The present invention relates to a rubber composition for shoe soles which is lightweight and has an appropriate hardness and which is excellent in a tensile strength, tear strength, an abrasion resistance and a gripping property and a rubber foam composition for shoe soles which is excellent as well in a dimensional stability of a shoe sole after foaming. Elastomer foam properties can be focused on temperature sensitivity of foam.

2.3.2.1 Temperature sensitivity of foam

The physical properties of elastomeric foams can be greatly influenced by temperature variation and footwear foam temperature can have a direct effect on the forces transmitted to the foot. It has been shown that soft footwear foams result in a longer contact time between the foot and the ground resulting in a reduction in the peak value of the forces transmitted to the foot and skeletal system. For many activities, such as running, the wearer must perform in hot and cold temperatures which will affect the attenuating properties of the footwear. Three main factors which play key roles in modifying the temperature of footwear are foot temperature, friction and compression between foot and footwear; and the temperature of the environment. Repetitive friction and compression between foot and footwear induces a non-homogeneous elevation of temperature in the foot. This elevation varies according to activity and to the ability of the footwear materials to evacuate heat. In general the heat is transferred to the shoe components (midsole and insole) resulting in a temperature rise (James *et al.*, 2011).

Other studies have found by (Utracki,1998) state that midsole temperatures increase during the initial 15–20 min of a run followed by a relatively constant temperature period as the heat exchanges becomes balanced. They found that the average increase in midsole temperature was about 60° C with a maximum value of 80° C. This increase was believed to be due to the effect of internally generated heat resulting from repetitive compression of the air cells inside the footwear and from heat transfer from the foot. This heat transfer is significant as whilst running the maximum midsole temperature has been shown to exceed the higher of the exterior air temperature and the road temperature by around 100° C (Parsons, 2011).

Runners generally seem to use the same or similar shoes throughout the year regardless of environmental temperature. Footwear foams have a melting temperature of about 70^oC. In colder temperature conditions, foams tend to become firmer depending on their formulation and cold ambient temperatures significantly reduce the shock attenuation of commonly used running shoes. These findings suggest that although the footwear foam temperature is not identical to the environmental temperature during an outdoor activity, it is still heavily influenced by environmental temperature. Considering the wide range of extreme weather conditions in which today's athletes train, the performance of their running shoes could vary significantly (Legge, 2003).

2.4 Processing of elastomer foam

Foam rubber is found in a wide range of applications, from cushioning in automobile seats and furniture to insulation in walls and appliances to soles and heels in footwear. Foams are made by forming gas bubbles in a plastic mixture, with the use of a blowing agent. Foam manufacture is either a continuous process for making laminate or slab stock or a batch process for making various shapes by cutting or molding. There are two basic types of foam. Flexible foams have an open cell structure and can be produced in both high and low densities. Applications include cushioning for furniture and automobiles, mattresses and pillows, automotive trim, and shoe soling. Rigid foams are highly cross-linked polymers with a closed cell structure that prevents gas movement. Their main application is as insulation for buildings, refrigerators and freezers and refrigerated transport vehicles (Galimberti, 2011).

2.4.1 Blowing agent

Blowing agent is a chemical substance that is widely used in generating the gas to expand rubber, plastics and ceramics to create foam. In other words, it is called baking powder for rubber, plastics and ceramics. In present, blowing agent is used to give the following advantages and features such as lightweight, heat insulation, elasticity, permeability and shock absorber. When rubber compound is added with blowing agent, fine cell structure is formed on the vulcanisate after curing, this rubber vulcanisate is called microcellular. The cell structure of microcellular is normally closed with gas trap within the cell. Since the blowing can be controlled the final products density varies, the bigger the cell size the lower the density and the softer the material. If the cell size is small the density is higher and the harder the product. The properties of microcellular rubber is normally inferior compared to solid vulcanisate, this is because the cell initiates failure. Some of the properties deteriorated are tensile properties, hardness, tear strength, abrasion and compression set. The most preferable properties of microcellular are low density, lightweight, high impact absorption and soft that assured comfort (Mayers, 1996).

Rubber foam also known as sponge rubber which has open cell structure and usually contain sodium bicarbonate as their blowing agent. Sometimes this blowing agent is used together with nitrogen gas blowing agents to give more effective in cell structure (Coswald 2007).

2.4.2 Compounding process to produce elastomer foam

Compounding is a process of adding one or more additives to polymers to achieve one or more specific properties. In plastic processing, there is having different meanings of the terms mixing, blending and compounding. Mixing indicates the physical act of homogenization, blending usually indicates preparation of polymer blend and alloys, while compounding is the incorporation of additives into polymeric matrix, antioxidants, lubricants, pigments, fillers, or reinforcements (Utracki and Shi, 2002). Generally the polymer blend has been prepared commercially by melt mixing, solution blending and latex blending.

2.4.2.1 Melt mixing

Melt mixing process is the heterogeneous impurities can migrate across the interface between both blend phases, Several factors determine the 'possibility' for the impurities to migrate from one phase to the other phase during the melt mixing process. The longer the mixing or the more intense mixing, the higher that probability those nuclei find themselves. Then the effect of migration on the crystallization behavior will be pronounced. Furthermore the possibility of impurities to located close enough to an interface stand in direct relation to the phase morphology during the melt mixing (Bartczak et al., 1986). Melt mixing is economical way of blending different polymers to avoid problems of contamination, solvent or water removal. Mixing can be accomplished on heated roll mills, mixing extruders, and twin screw mixer. Twin screw mixer is suitable for melt mixing elastomer with plastic. Mixing on an open mill in air at elevated temperatures induces oxidative degradation. Internal mixers, mixing extruder, and twin-screw extruder are more efficient and their use gives less oxidative degradation. An internal mixer is a batch mixer, whereas a twin-screw extruder is a continuous mixer (Coran, 2001). Je and Chang (1998) The factors affecting of blend morphology during compounding are temperature, duration of mixing, the residence time, the intensity of mixing (rotor speed in an internal mixer or screw speed in a twin-screw extruder), blend composition, viscosity ratio, elasticity ratio, and interfacial tension.

2.4.2.1 a) Internal mixer

The first step of rubber processing is combining the ingredients of a compound in a mixing device. Internal mixers were first developed by Fernley H.Banbury in 1916. Today it is commonly used because they are much more productive than two roll mills. Internal mixers consist of two rotors blades turning toward each other in an enclosed metal cavity. These rotors can be either tangential or intermeshing. Mixing time is determines by the shape and size of the rotors, the rotor speed and the horsepower of the motor turning them (John S. Dick, 2001).The internal mixer is mixing instruments widely used in most laboratories working on polymer or powder material process. It requires only small amount of materials and is usually used as a preliminary testing step for verifying the melting and mixing behaviors of polymers, chemical reactions in polymers, polymer cross-linking, and polymer degradation before the actual processing in an extruder (Cha and White, 2001).

The modern internal mixer consists of a mixing chamber containing two counter rotating rotors. At the center of the top of the mixing chamber is a shaft through which compounding ingredients gain access. During most of the mixing cycle, the shaft contains a ram which presses the compounding ingredients into the chamber. At the bottom of the mixing chamber is a door which opens at the end of the cycle and dumps the mixed compound (James, 2005). In these internal mixers, both rotors must move at the same angular velocity. The Francis Shaw mixer, the "Intermix," was marketed first and received considerable attention. In time, the intermeshing mixers dominated the mechanical rubber goods industries in Europe and Japan (Dick, 2001).

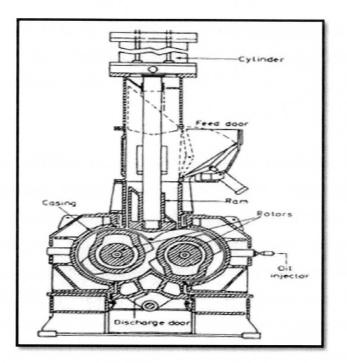


Figure 2.6: Internal mixer machine.

2.4.2.1b) Twin-Screw Extruder

A screw extruder has one screw or more than one screw. An extruder with one screw is called a single screw extruder; and it is the most common machine in the plastics processing industry. An extruder with more than one screw is called a multiscrew extruder. The most common multiscrew extruder is the twin screw extruder; it has two screws (John Wiley and Sons, 2005). There are Co-Rotating Twin Screw Extruders and Counter-Rotating Twin Screw Extruders.

There are several types of twin screw extruders. In most twin screw extruders, the screws are located side by side. If both screws rotate in the same direction, the extruder is called a co-rotating twin screw extruder (Figure 2.7). Co-rotating twin screw extruders used for compounding run at high screw speed, typically

between 200 and 500 rpm. Some of the newer very high speed twin screw extruders are capable of running at speeds over 1000 rpm, as high as 1600 rpm (John Wiley and Sons, 2005).

According to John Wiley and Sons (2005) defined the twin screw extruder rotate in opposite direction, it is called a counter-rotating twin screw extruder. It can run at high or low speed, depending on the application. High speed extruders run at around 200–500 rpm and even higher; they are primarily used in compounding. Low speed extruders run at about 10–40 rpm and are used mostly in profile extrusion applications. Most twin screw extruders for profile extrusion are counter-rotating extruders. This is because counter-rotating extruders tend to have better conveying characteristics than co-rotating extruders. Another distinguishing feature of twin screw extruders is the extent that the screws intermesh. The screws can fully intermeshing shows in (Figure 2.7a), partially intermeshing shown in (Figure 2.7b), and no intermeshing shown in (Figure. 2.7c).

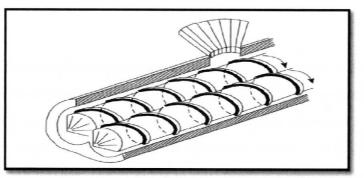


Figure 2.7: A co-rotating twin screw extruder.

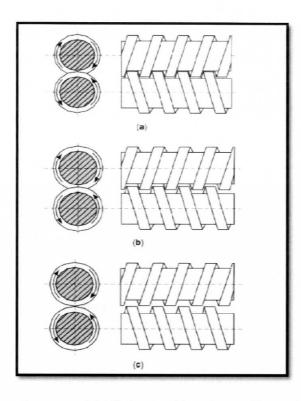


Figure 2.8: Counter-rotating screws: (a) fully intermeshing; (b) partially intermeshing; and (c) no intermeshing (tangential)

2.4.2.1c) Two Roll Mill

Mixing of rubber and other ingredients can be achieved on a two-roll mill which consists of two horizontal, parallel, heavy metal roll which can be jacketed with steam and water to control temperature. The rolls rotating in opposite directions with a pre-set, adjustable gap or nip to allow the rubber to pass through to achieve high shear mixing. The different in roll speeds is called the friction ratio. Two roll mills relatively slow method and it is batch size limited. Internal mixers overcome this problem (Dick, 2001).

The two roll mill in Figure 2.9 consist of two horizontal, parallel, heavy metal roll which can be jacketed with steam and water to control temperature. These rolls turn towards each other with a pre-set, adjustable gap or nip to allow the rubber to pass through to achieve high-shearing mixing (Dick, 2001).

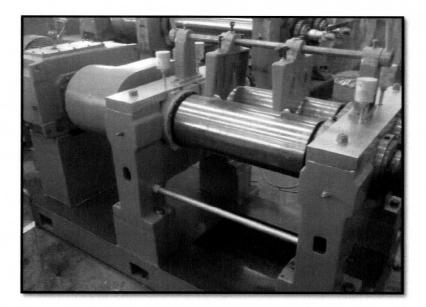


Figure 2.9: Two roll mill machin

2.4.2.2 Solution Blending

Due to the experimental difficulties in mixing and measuring highly viscous polymer blends, it is a frequently laboratory practice to attain intimate mixing by dissolving the two polymers in a common solvent then drying them. However, the morphology of polymer alloy and blend prepared by solvent casting depends on the solvent, temperature, time, and concentration (Utracki, 2000).

2.4.2.3 Latex Blending

The latex blends were used either directly, for example, as paint, adhesives or sealants, or they were spray dried or palletized. Spray drying has been the most frequently used method. Since emulsion polymerization was able to produce either a fine dispersion of homo or copolymer, or alternatively large drop aggregates, comparable to those generated in suspension polymerization. The latex blending offered not only a wide range of composition but also diverse morphologies. The disadvantage of the latex blending was high content of contaminants such as emulsifiers, residuals of the initiators, chain transfers, stabilizers and etc (Utracki and Shi, 2002).

2.4.2.4 Compression Molding

According to the Osswald and Menges (2003), compression molding is widely used in the automative industry to produce parts that are large, thin, lightweight, strong and stiff. It is also used in the household goods and electrical industries. The appropriate amounts of thoroughly mixed polymer and necessary additives are placed between male and female mold members, as illustrated in Figure 2.10.The compression molding process is fairly simple. First, the both mold pieces are heated; however only one is movable. The mold is closed, and heat and pressure are applied, causing the plastic to become vicous and flow to confirm the mold shape. Before molding, raw materials may be mixed and cold pressed into a disc, which is called preform. Preaheating of the preform reduces molding time and pressure, extends the die lifetime, and produces more uniform finished pieces (Callister, 2007).

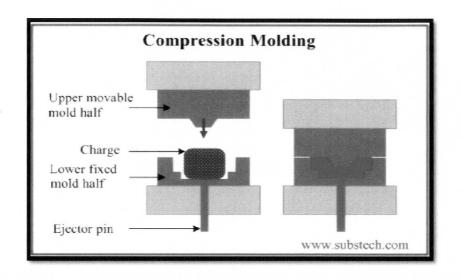


Figure 2.10: Schematic compression molding process

CHAPTER 3

METHODOLOGY

In this chapter covers the methodology of this project which divided into several sub-section as following;

- a. Raw material
- b. Characterization of raw materials
- c. Preparation of sample
- d. Testing and analysis

Figure 3.1 shows the flowchart of overall processing method, testing and analysis that will carry out in this study.



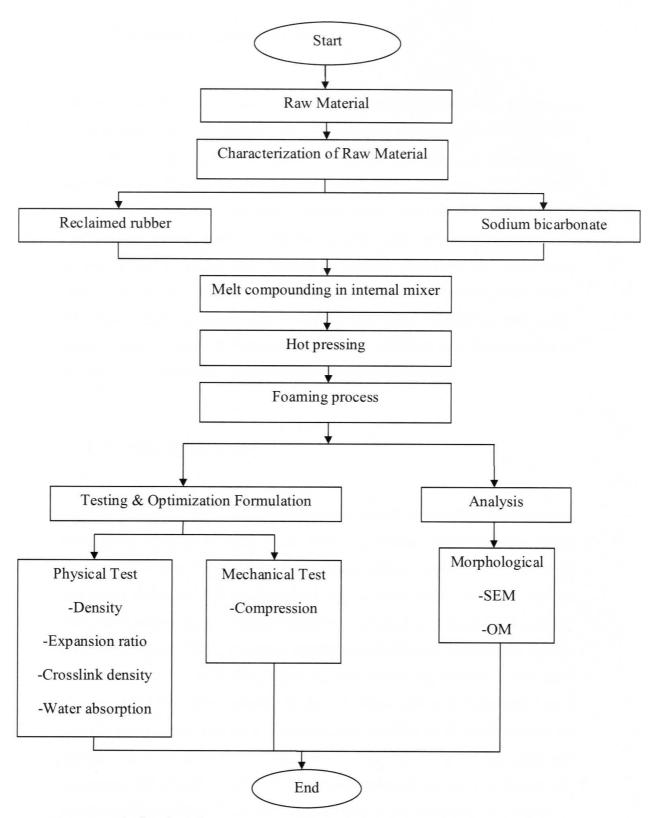


Figure 3.0: The flowchart of overall processing method, testing and analysis of rubber foam

3.1 Characteristic of raw material

The main raw materials in this study are reclaimed rubber, sodium bicarbonate and vulcanization agent (sulfur). The concentration of sodium bicarbonate as a chemical blowing agent was varied to evaluate its effect on the physical properties, mechanical properties and morphology. The expandable rubber samples were prepared using Haake internal mixer and were then expanded via heat transfer foaming process using hot compress and air circulating oven. The physical properties of the rubber foams were characterized and the results were observed to systemically correlate with the mechanical properties of the foam. All other rubber ingredients that have been used, such as sulphur, zinc oxide, stearic acid, Tetramethylthiuram-disulfenamide (TMTD) and Benzothiazyl-2cyclohexyl-sulphenamide (CBS), were from of industrial grade.

3.1.1 Reclaimed rubber glove

Reclaim rubber is vulcanized rubber products already or a rubber manufactured from waste or scrap of tires, shoes, tubes and rubber gloves and then reuse through the reclaim process by the molecules of rubber are linked. It decomposed into small molecules that can be mixed with reclaim rubber chemicals will be vulcanized rubber again. Rubplast reclaimed rubber is produced from selected discarded or worn rubber products, mainly tyres and tubes. Under strict quality control systems, a reclaimed rubber of consistency and quality is obtained. Rubplast therefore offers reliable reclaimed rubber which is both useful as a process aid material, and as a distinctive raw material in it. Rubplast can be divided into 9 groups where it has been divided according to their type and properties. Rubplast G is a derived from examination gloves and has a lower ash content and higher rubber content.Its colour ranges from lovibond 3- 7. Table 3.1 shows the Rubplas Reclaim Grades G and their properties.

Rubplas Reclaim Grades		
Item	G	
Ash %	12	
Acetone extract %	5	
Cabon black %	0	
Rubber %	80	
Specific gravity	1.05	

Table 3.1: Rubplas Reclaim Grades G

3.1.2 Sodium bicarbonate

Sodium bicarbonate has been used as the blowing agent and is purchased from Science Lab.com. Sodium bicarbonate is an organic chemical blowing agent that releases carbon dioxide gas during decomposition. It decomposes at relatively low temperature between 145° C to 150° C and often results in an open cell structure. In this study, rubber foams were prepared by varying the concentration of sodium bicarbonate which is 4,6,8,10 and 12 phr which was used as a blowing agent at fixed processing time and temperature. The influence of the sodium bicarbonate concentration on the physical properties and mechanical properties of the foam has been analyzed. Table 3.2 shows the physical properties and chemical properties of the sodium bicarbonate.

Item	Properties	
Physical state and appearance	Solid	
Odor	Odorless	
Taste	Alkaline	
Molecular Weight	84.01g/mole	
Color	White	

Table 3.2: Physical properties and chemical properties of sodium bicarbonate

3.2 Preparation of the sample and process

3.2.1 Blending of RR blends in Internal Mixer

The compounding process is performed according to ASTM D-3182 and using a Haake internal mixer (Figure3.1) working at 60^oC and a rotor speed of 60 rpm. Firstly, the component will be weighted to desire compound composition ratio of reclaimed rubber, zinc oxide, stearic acid, TMTD, CBS, sulphur and sodium bicarbonate for specific formulation as stated in Table 3.3 and in Table 3.4 shows the table of the preparations of raw materials of compound.

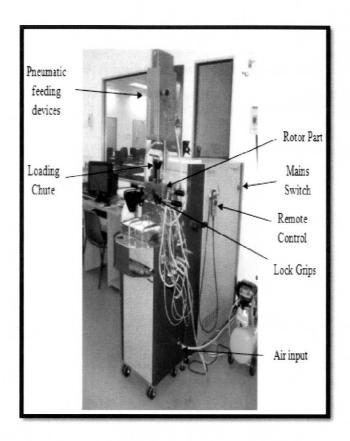


Figure 3.1: HAAKE RHEOMIX OS Internal Mixer.

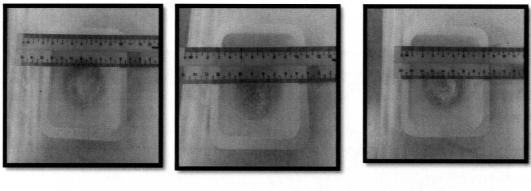


Ingredients	Composition (phr)					
	Control-	Sample-	Sample-	Sample-	Sample-	Sample-
	Sample	1	2	3	4	5
Reclaimed Rubber	100	100	100	100	100	100
Zinc Oxide	4.0	4.0	4.0	4.0	4.0	4.0
Stearic acid	2.0	2.0	2.0	2.0	2.0	2.0
Tetramethyl Thiuram Disulphate (TMTD)	2.5	2.5	2.5	2.5	2.5	2.5
Benzothiazyl- 2-cyclohexyl- sulphenamide (CBS)	1.0	1.0	1.0	1.0	1.0	1.0
Sulphur	1.0	1.0	1.0	1.0	1.0	1.0
Sodium bicarbonate	0	4	6	8	10	12

 Table 3.3: Formulation of natural rubber compounds

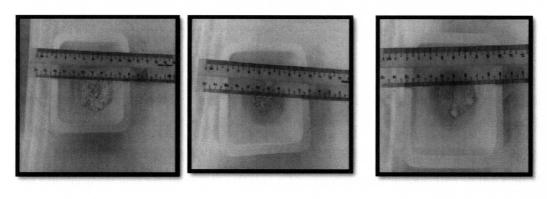


Figure 3.2: Reclaimed rubber



- a) Zinc Oxide
- b) Stearic acid





- d) Sodium
- e) CBS

f) Sulphur

bicarbonate



(c) TMTD (d) CBS (f) Sulphur

3.3 Hot pressing

3.3.1 Compression Molding Process

The rubber compound was compounded with other ingredients based on the formulations of mixture from Table 3.3 in Haake internal mixer. The curing agent which is sulphur and the blowing agent were added at the end of the process. Figure 3.2 shows that the rubber compound after mixing in the Haake internal mixer

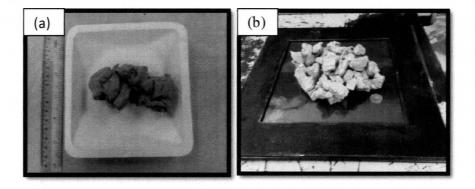


Figure 3.4: (a) Rubber compound (b) Rubber compound cut with scissor

Next step are compression moulding process. The rubber compound was cut with scissor (Figure 3.3) and weighed. The machine used in this step is Gotech (GT 7014-A) hot press machine. The hydraulic hot moulding machines (Figure 3.4) was switched on and the temperature was set to $100 \, {}^{0}$ C. Then, rubber compound is moulded in the disc shaped with the diameters of 160mm and 6mm depth using hot press for 1 minute at the temperature of $100 \, {}^{0}$ C.

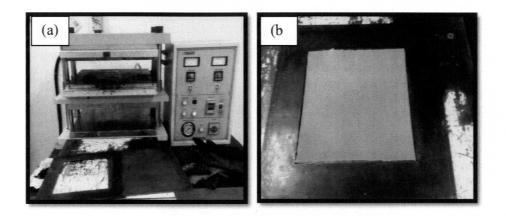


Figure 3.5: (a) Hot moulding machines (b) Mold of rubber foam

Then the mold was inserted into the first slot to compress the sample for 1 minute at the pressure meter of 110psi. After 1 minute, the mold (Figure 3.5) was taken out from the first slot and put into second slot for cooling. When the cooling step has finished, the mold was taken out and its flash was removed by using scissor.

3.4 Foaming process

The compounds were vulcanize and foamed via heat transfer process. This process was involve pre vulcanization using compression moulding at a temperature of 100^{0} C for 1 minute and followed by simultaneous curing and foaming in an air circulating oven (Figure 3.6) for 30 minutes at a temperature of 150^{0} C.



Figure 3.6: Air circulating oven

Foams are characterized by their density, cell size, shape and the modulus of the base polymer. The compounds with full vulcanized by simultaneous curing and foaming in an air circulating oven for 30 minutes at a temperature of 150 0 C. Figure 3.7 shows that the rubber foam after taking out from the oven after 30 minutes.



Figure 3.7: Rubber foam

3.5 Testing and Analysis

In the next step of this project, the samples was tested and analyzed. The testing and analysis are as following:

1) Physical test

- i) Density
- ii) Relative foam density
- iii) Expansion ratio
- iv) Crosslink density
- v) Water absorption
- vi) Retention test
- 2) Mechanical test
 - i) Compression test

3) Morphological

- i) Digital camera
- ii) Optical microscope (OM)
- iii) Scanning electron microscope (SEM)

3.5.1 Physical Test

3.5.1.1 Density Test (ASTM D3575)

The density of a sample is a property that is conveniently measured to identify a material, to follow physical changes in a sample, or to indicate the average density of a large item. Electronic densimeter (Figure 3.8) was used in this study to measure the density of samples. The sample which size of 20mmx20mmx10mm was put into the beaker and the readings was recorded. The weights of the sample were recorded in water and on air. The procedure was repeated for the other 5 specimens.



Figure 3.8: Electronic densimeter.



3.5.1.2 Relative Foam Density (ASTM D3575)

Relative foam density was the results of the density before foaming and after foaming. Before foaming the density of the sample with size of 20mmx20mmx3mm was determined with before put the sample into the oven for foaming process. Then, density of the sample after the foaming process with size of 20mmx20mmx10mm was determined again. The electronic densimeter was used with the sample of different five samples have been used for the relative foam density testing. Relative density was calculated based on the Equation 3.1 below;

Relative density =	Foam Density Solid Density	Equation 3.1
Relative defisity -	Solid Density	

3.5.1.3 Expansion Ratio (ASTM D3375-93)

Expansion ratio was measured using equation in accordance with ASTM D3575-93.The different sample with different formulation of the concentration of sodium bicarbonate as blowing agent was used to determine the expansion ratio of each sample. Height before foaming is determine where the height after the sample was compress by using hot press machine at 100° C with 1 minute and the height after foaming is height after the sample was put into the oven at 150° C with 30 minutes for foaming process. The foam expansion ratio was calculated based on the following Equation 3.2: Expansion ratio = $\frac{Hf - Ho}{Ho}$ Equation 3.2

Where,

 $H_f =$ Height after foaming

 $H_o =$ Initial height before foaming

3.5.1.4 Crosslink Density (ASTM D471)

Crosslink density was to determine the cross linking occurs between cell walls of the foam samples. Five different samples with different formulations were put into the beaker containing toluene and were left for six hours. Before the sample was put into the beaker, the original weight was measured first. Then after six hours the sample were then removed from the solvent, wiped thoroughly to remove excess solvent and weighed again as a results this value was taken as the swollen weight. The crosslink density of the sample was calculated using the Flory-Rehner Equation 3.3 as follows:

$$- \{ \ln (1-Vr) + Vr + xVr^2 \} = \Box V_0 M_c^{-1} Vr^{\frac{1}{3}}$$
 Equation 3.3

Where,

X= Interaction constant characteristic between rubber and toluene, 0.42

P= Rubber density, 0.973

V_o= Molar volume of toluene, 92.14g/mol

 V_r = Volume fraction of rubber in swollen sample

M_c= Average molecular weight between crosslink

$$Vr = \frac{(Xr/Pr)}{(Xr/pr) + (Xs/Ps)}$$

Where,

Ps= density of toluene, Pr= density of the raw rubber, Xs= mass fraction of toluene, X_r = weight of the rubber

$$Xs = \frac{(Weight of Swollen Sample - Original Weight)}{Weight of Swollen Sample}$$

 $X_r = 1 - X_s$

3.5.1.5 Water Absorption (ASTM C1083)

Water absorption was to determine how much the sample can absorb the water with the time was setting before. Five beakers containing the water then was put the different sample and left for 1 hour, 2 hours, 3 hours, 12 hours and 24 hours. The samples sizes are same for each sample that is 20mmx20mmx10mm. The original weight of the sample was measured using analytical balance. Then the samples were immersed in a beaker containing water with 450ml for each of beaker. The sample were then removed from the water with the time that was setting early, wiped thoroughly to remove excess water and weight again and the value was taken as the water absorption rate of sample. Water absorption rate was calculated based on following Equation 3.4:

Water absorption rate = Final weight sample – Initial weight sample ... Equation 3.4

3.5.1.6 Retention test (ASTM D6147-97)

Retention test was to determine the retention samples of load imposed on it by the time was specified. Five different samples was placed load with 45 kilogram on it and was left for 30 minutes, 60 minutes, 90 minutes and 120 minutes to know the extent to which the samples can withstand the load on it. The initial height of the sample was measured before placed the load. The samples sizes are same for each sample that is 50mmx50mmx10mm. After 30 minutes, the load was removed and the height of the sample was measured. The sample was left for 1 minute, 2 minutes, 3 minutes, 4 minutes and 5 minutes for the recovery. After 5 minutes, the load was placed again to the sample and repeated for 60 minutes, 90 minutes and 120 minutes. Retention time was calculated based on following Equation 3.5:

Retention time = Final height sample – Initial height sampleEquation 3.5



3.5.2 Mechanical Test

3.5.2.1 Compression test (ASTM-D575)

Sample with the dimension of 50x50x15mm was cut without any skin layer exists on the top and bottom of the rubber foam. Then, it was tested using Instron machine (Figure 3.9) with a cross head speed of 25mm/min downwards to measure the compression stress. For compression set, the sample with the dimension of 50x50x15mm was cut and put through the compression plate and compresses until 50% of the foam original thickness before exposing it in an oven with the temperature of 70^{0} C for 24 hours.



Figure 3.9: Instron machine

3.5.3 Morphological Study

3.5.3.1 Digital camera

Digital camera (Figure 3.10) is a camera that takes photographs and video by recording images on an electronic image sensor. Digital and film cameras share an optical system, typically using a lens with a variable diaphragm to focus light onto an image pickup device. The different five samples were captured image by the digital camera to view the image of the foam of rubber. The size of each sample was 50mmx10mm. The image of foam was been analyzed on result and discussion.



Figure 3.10: Digital camera

3.5.3.2 Optical microscope

Optical microscope (Figure 3.11) is an optical instrument that uses the lens and combination of lens to produce the magnified images that are too small to seen by unaided eye. Optical microscope can be use for imaging surface features at 5 to 1000x magnification features. The sample with dimension of 20mmx20mmx10mm was placed under microscope slide to see the size of foam of the sample. The light sources was switched on, the brightness of the light was adjusted by using rheostat. The diaphragm was adjusted to get the best lighting. Optical microscope was used to see the size and morphology of the foams.



Figure 3.11: Optical microscope

3.5.3.3 Scanning Electron Microscope (SEM)

Scanning electron microscopy can be use for imaging of surface features at 10 to 100 000 x Resolution of features down to 3 to 100 nm, depending on the sample. The primary use of SEM is to produce high-resolution and depth-of-field images of sample surfaces. Besides that, SEM is need provide chemical analyses of micron-sized areas of the structure revealed on these surfaces, and evaluates the crystallographic orientation of grains with diameter down to 2 to 10 μ m. SEM is use for imaging of a second phase on unetched surfaces when the second phase has a different average atomic number. In this study, Zeiss EVO-50 ESEM machine has been used (Figure 3.12).

Samples with 5mmx5mmx1mm were placed in the microscope. The samples were coated with thin layer of gold. Samples must be free from high vapor pressure liquids, such as water, organic cleaning solutions and remnant oil-base film. Test samples were placed in a vacuum chamber for viewing on the computer monitor at to 35x magnification. Polaroid photos were taken for a permanent record.



Figure 3.12: Zeiss EVO-50 ESEM machine

CHAPTER 4

RESULT AND DISCUSSION

4.0 Introduction

This chapter lists the results, analysis, data evaluation and data discussion of the study measures in chapter 3. Each of the results and discussions are in the sequence of methodology. The results and discussions are as following:

- a) Physical properties of rubber foam
- b) Mechanical properties of rubber foam
- c) Analysis

4.1 Physical properties of rubber foam

4.1.1 Density of solid rubber and rubber foam

Table 4.1 show the density of solid rubber and rubber foam. As the percentage of sodium bicarbonate increased, the percentage of foam also increased. The density of solid rubber constant but the density of rubber foam difference due to the difference percentage concentration of sodium bicarbonate used.

Sample	Percentage concentration of sodium bicarbonate (phr)	Density of solid rubber (g/cm ³)	Density of rubber foam (g/cm ³)	Increase of percentage foam (%)
CS	0	0.436	-	-
1	4	0.436	0.405	7.11
2	6	0.436	0.376	13.76
3	8	0.436	0.354	18.80
4	10	0.436	0.317	27.29
5	12	0.436	0.293	32.79

 Table 4.1: Density of solid rubber and rubber foam

Figure 4.1 illustrates that the increase percentage of foam. The increasing percentage concentration of sodium bicarbonate was increase the percentage of foam. This is because, as greater concentrations of sodium bicarbonates as blowing agent were used, it releases more carbon dioxide gas as well as allowing the sample produce more foam. This foam was expanding more, the solid state of sample reduce and consequently producing foam with lower density.

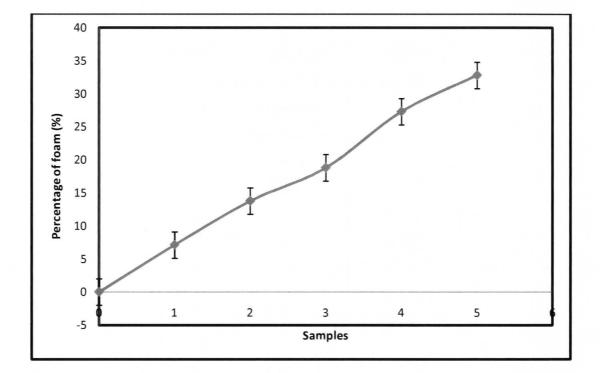


Figure 4.1: Increase percentage of foam (%)

4.1.2 Relative density of rubber foam

Table 4.2 show the relative foam density of rubber foam. As the percentage concentration of sodium bicarbonate increased, the relative density of rubber foam decreased.

Sample	Percentage concentration of sodium bicarbonate (phr)	Relative density
CS	0	-
1	4	0.928
2	6	0.862
3	8	0.811
4	10	0.727
5	12	0.672

Table 4.2: Relative foam density

Figure 4.2 illustrates the effect of the blowing agent concentration on the relative foam density of rubber foam. Increasing the blowing agent concentrations, the number of cells per unit volume also increased. From the Figure 4.2, it is found that the relative density slightly decreases. An increase in the blowing agent concentration resulted in smaller, finer and more uniform cells. The decomposition of high concentrations of carbon dioxide gas occurs simultaneously for a given time, and then more cells formed at the same time. Consequently, the number of cells per unit volume increased, resulting in a smaller average cell size in the foam. Increase in blowing agent concentration of the sample decrease the relative foam density of sample.

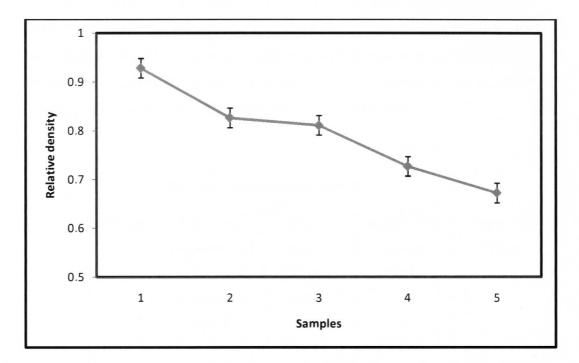


Figure 4.2: Relative foam density

4.1.3 Expansion ratio

Table 4.3 show the expansion ratio of rubber foam. Height before foaming of the sample was same because during the compression moulding process, the height of the mold that used to compress the sample by using hot press machine was same. Height after foaming of the sample shows the increased as the increased percentage concentration of sodium bicarbonate. The expansion ratio increased directly proportional to the height after foaming of the sample.

Sample	Percentage concentration of sodium bicarbonate (phr)	Height before foaming (mm)	Height after foaming (mm)	Expansion ratio
CS	0	14.48	14.48	0
1	4	14.48	16.53	0.141
2	6	14.48	17.78	0.227
3	8	14.48	18.86	0.302
4	10	14.48	19.98	0.379
5	12	14.48	21.40	0.477

Table 4.3: Expansion ratio of rubber foam

Figure 4.3 illustrates the effect of blowing agent concentration on the expansion ratio of the rubber foam. As greater concentrations of blowing agent were used, increasing the expansion ratio of rubber foam due to the more gas was produced during foaming process in the oven at 150^oC with 30 minutes. When the gas produced is increased, more foam was formed and allowing the foam to expand more and consequently produce the rubber foam with high expansion ratio. Increase the formed of foam space, decrease the solid space of the sample then increase the expansion ratio of samples.

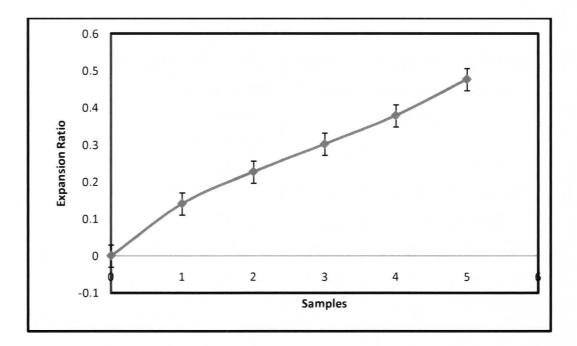


Figure 4.3: Expansion ratio of sample

Table 4.4 show the crosslink density of rubber foam. Original weight of the sample decreased with increased the percentage concentration of sodium bicarbonate but for the weight of swollen sample and crosslink density of rubber foam, it was increased with increased percentage concentration of sodium bicarbonate.

Sample	Percentage concentration of sodium bicarbonate (phr)	Original weight(g)	Weight of swollen sample(g)	Crosslink density
1	4	2.235	5.128	1.89x10 ⁻³
2	6	2.099	6.179	1.12×10^{-3}
3	8	1.915	7.129	7.51×10^{-4}
4	10	1.840	8.342	5.82×10^{-4}
5	12	1.734	9.306	4.95×10^{-4}

Table 4.4: Crosslink density of rubber foam

Figure 4.4 illustrates the effect of the blowing agent concentration on the crosslink density of the rubber foam. The crosslink density shows slightly decreased with increasing blowing agent concentration. This is because cross linking and decomposition occur simultaneously where at high blowing agent concentrations, the more carbon dioxide gas is present thus the gas phase will be more prominent than solid phase. Hence, it would be expected that similar

crosslink densities would be obtained for all the samples because the same amount of sulphur (cross linking agent) was used. However, the sodium bicarbonate used in this study decomposed endothermically this may result in cross linking deficiency as the blowing agent concentration increases. For the sample 1 with formulation of 4phr, 3.54% of the sodium bicarbonate used from the whole sample whereby for the sample 5 with the formulation of 12phr, 9.78% of the sodium bicarbonate used from the whole sample. The changes in the crosslink density of the foam may be caused by the destruction of crosslinks by the expansion of the gas during the decomposition of the blowing agent (Sombatsompop *et al.*, 2000). At high concentrations of sodium bicarbonate, more heat was absorbed from the system, hence interrupting the cross linking process.

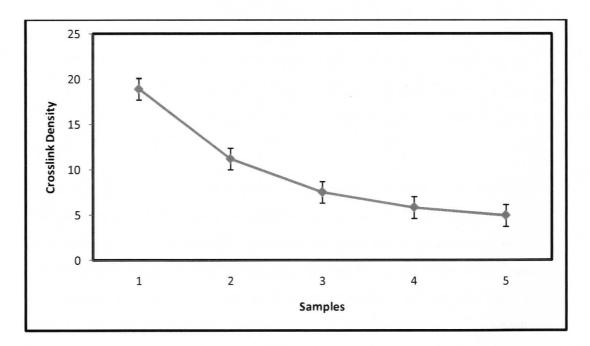


Figure 4.4: Crosslink density of sample

Table 4.5 show the water absorption rate of rubber foam. Increased the percentage concentration of sodium bicarbonate was increased the rate of water absorption. Sample 5 with highest percentage concentration of sodium bicarbonate show the highest water absorption rate.

Reading(h)	0	1	2	3	12	24
Sample(g)						
1	1.868	1.953	2.040	2.127	2.413	2.475
2	1.882	1.960	2.056	2.152	2.748	2.773
3	1.888	2.187	2.494	2.801	2.908	3.012
4	1.963	2.293	2.577	2.895	3.213	3.286
5	1.980	2.295	2.655	3.048	3.441	3.445

Table 4.5: Water absorption rate of rubber foam

Figure 4.5 illustrates the effect of blowing agent concentration on the water absorption rate of rubber foam. As greater concentrations of blowing agent were used, more gas was subsequently generated, reducing the relative foam density. The higher concentrations of blowing agent shorten the growth time of the foam, thus restricting the gas escaping through the foam surface, allowing the foam to expand more and consequently producing the foam with lower relative density (lighter). The water absorption rate slightly increased with increasing blowing agent concentrations. This is due to the fact that, at high concentrations of blowing agent, more carbon dioxide gas is released, thus is produce more foam space. It will expected that, the increase the concentrations of blowing agent more water is absorb into the sample by increase the time until after 24 hour of

water absorption. During 1 hour, 2 hour and 3 hour, the rate of water absorption increase dramatically to the sample but after 12 hour the rate of water absorb only slightly increase because of the saturated water in the sample. These results show that the increase in concentration of blowing agent will increase the water absorption rate of the sample until it comes to saturated parts.

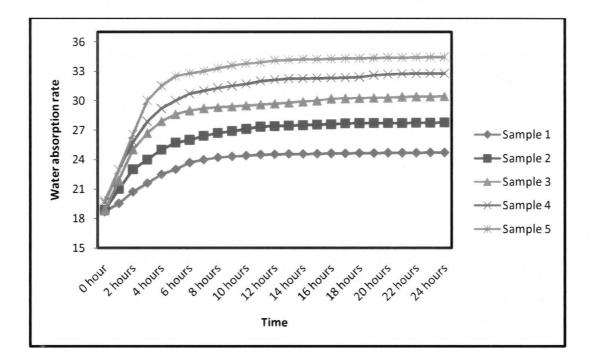


Figure 4.5: Water absorption rate of sample

4.1.6 Retention test

Table 4.6 until Table 4.10 shows the retention test for the rubber foam. Table 4.6 show retention tests for sample 1 with percentage concentration of sodium bicarbonate was 4phr, Table 4.7 show retention tests for sample 2 with percentage concentration of sodium bicarbonate was 6phr, Table 4.8 show retention tests for sample 3 with percentage concentration of sodium bicarbonate was 8phr, Table 4.9 show retention tests for sample 4 with percentage concentration of sodium bicarbonate was 610phr, whereas Table 4.10 show retention tests for sample 4 with percentage concentration of sodium bicarbonate was 612phr.

Retention(min)	0	30	60	90	120
Recovery(min)	14.83	14.51	14.50	14.49	14.49
1	14.83	14.53	14.52	14.50	14.50
2	14.83	14.54	14.53	14.51	14.51
3	14.83	14.54	14.53	14.52	14.52
4	14.83	14.54	14.53	14.52	14.52
5	14.83	14.54	14.53	14.52	14.52

Table 4.6: Retention test for sample 1 with 4 phr

Retention(min) Recovery(min)	0	30	60	90	120
0	15.28	14.93	14.92	14.91	14.91
1	15.28	14.95	14.94	14.92	14.92
2	15.28	14.96	14.95	14.93	14.93
3	15.28	14.96	14.95	14.93	14.93
4	15.28	14.96	14.95	14.93	14.93
5	15.28	14.96	14.95	14.93	14.93

Table 4.7: Retention test for sample 2 with 6phr

Table 4.8: Retention test for sample 3 with 8phr

Retention(min) Recovery(min)	0	30	60	90	120
0	15.73	15.37	15.36	15.35	15.35
1	15.73	15.39	15.38	15.37	15.36
2	15.73	15.40	15.39	15.38	15.37
3	15.73	15.40	15.39	15.38	15.37
4	15.73	15.40	15.39	15.38	15.37
5	15.73	15.40	15.39	15.38	15.37

Retention(min)					
	0	30	60	90	120
Recovery(min)					
0	16.18	15.82	15.81	15.80	15.79
1	16.18	15.84	15.83	15.82	15.80
2	16.18	15.85	15.84	15.83	15.81
3	16.18	15.85	15.85	15.84	15.82
4	16.18	15.85	15.85	15.84	15.82
5	16.18	15.85	15.85	15.84	15.82

Table 4.9: Retention test for sample 4 with 10phr

Table 4.10: Retention test for sample 5 with 12phr

Retention(min)					
	0	30	60	90	120
Recovery(min)					
0	16.65	16.26	16.25	16.24	16.23
1	16.65	16.28	16.27	16.26	16.25
2	16.65	16.29	16.28	16.27	16.26
3	16.65	16.30	16.29	16.27	16.26
4	16.65	16.30	16.29	16.27	16.26
5	16.65	16.30	16.29	16.27	16.26

Figure 4.6 illustrates that the retention test of rubber foam. The retention test for rubber foam was same with increased percentage of sodium bicarbonate as blowing agent because the size and shape of foam does not involved. Size and shape of foam does not involve during retention test because the foam properties was flexible to recovery it height either with larger size of foam or lower size of foam. When the foam was left for a long time, it was capable to return its original shape.

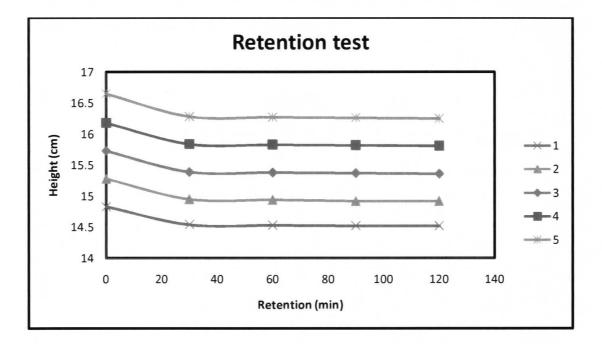


Figure 4.6: Retention test of sample

4.2 Mechanical properties of rubber foam

4.2.1 Compression testing

Table 4.11 show the stress vs. strain of rubber foam. The increase percentage of concentration of sodium bicarbonate, the stress of the sample decrease but strain of the sample increase. Sample 5 has the highest percentage of sodium bicarbonate but has lowest stress and highest strain.

Sample	Percentage concentration of sodium bicarbonate (phr)	Stress (kPa)	Stroke Strain (%)
1	4	105	46.0
2	6	95.5	50.0
3	8	83.0	52.5
4	10	73.0	54.0
5	12	55.0	56.0

Table 4.11: Stress vs. strain

Figure 4.7 illustrates that the effect of blowing agent concentration on the foam mechanical properties of stress strain of foam sample. As compression set, it is determine that by adding more blowing agent concentration, less value of compression set are obtained. The cell sizes were played an important role in compression set test. Increase the blowing agent concentration, the stress and strain required to compress the sample is decrease. This is due to the fact that increase the formations of foam of the sample give the more empty space exist that from increases the producing of gas carbon dioxide during foaming process.

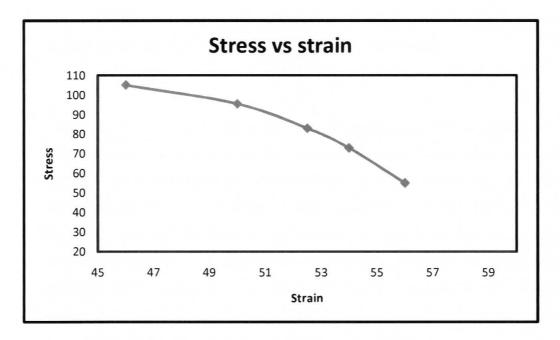


Figure 4.7: Stress vs. stroke strain of the foam sample

4.3 Analysis

Morphological analysis was performed on every sample which had different formulations to support the data of physical testing and mechanical testing of rubber foam.

4.3.1 Digital camera

Figure 4.6 shows the morphology of average cell sizes of rubber foam at different sodium bicarbonate concentration as blowing agent. The length size of foam is depending on the concentration of sodium bicarbonate.

There are various factors influencing the foam morphology such as temperature, pressure, polymeric materials and the formulations used. The effect of chemical blowing agent introduces the effect of cell distribution and the cell size depending on the concentrations of blowing agent. From the figure 4.8, it observed that, the cell size grow larger when higher amount of sodium bicarbonate regardless of foaming direction such as perpendicular or parallel direction of foam. Figure 4.8(a) show the smallest cells foam with lowest concentration of sodium bicarbonate where figure 4.8(e) show the largest cells foam due to the largest concentration of sodium bicarbonate.

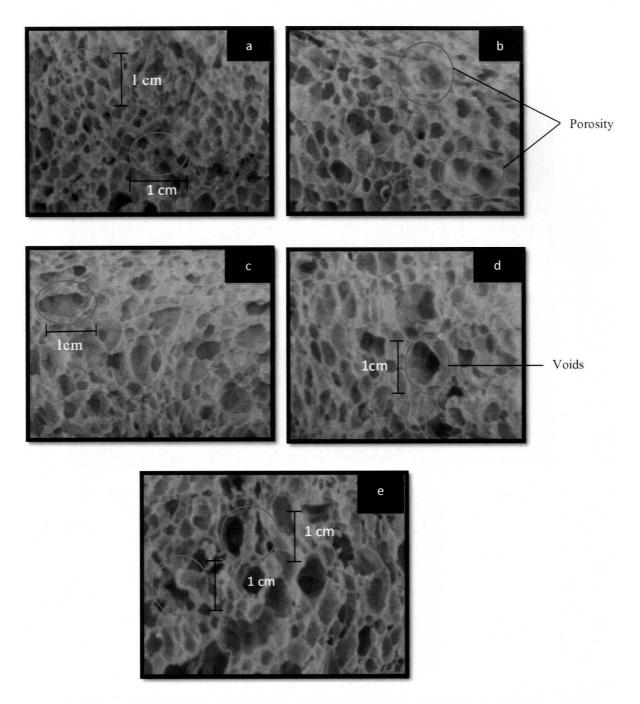
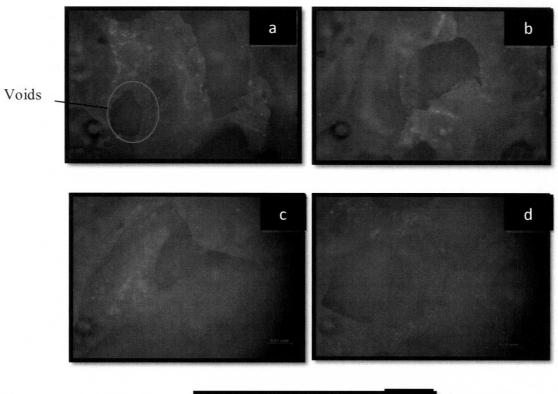


Figure 4.8: (a) Micrograph of 4phr of sodium bicarbonate. (b) Micrograph of 6phr of sodium bicarbonate. (c) Micrograph of 8 phr of sodium bicarbonate. (d) Micrograph of 10phr of sodium bicarbonate. (e) Micrograph of 12 phr of sodium bicarbonate.

4.3.2 Optical microscope



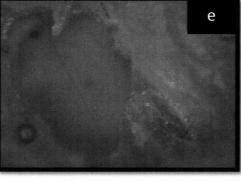
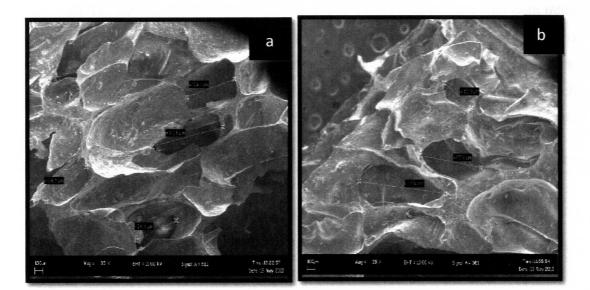


Figure 4.9: (a) Optical micrograph of 4phr of sodium bicarbonate at magnification of 5x. (b)
Optical micrograph of 6phr at magnification of 5x. (c) Optical micrograph of 8 phr at magnification of 5x. (d) Optical micrograph of 10phr at magnification of 5x. (e) Optical micrograph of 12 phr at magnification of 5x.

Figure 4.9(a) show the smallest cells foam whereby figure 4.9(e) show the highest cells foam. This is due to the that increase the concentration of blowing agent produced the larger cells. Increasing the cell size and thinning effect of the cell wall directly alter the foam height as well as foam expansion ratio. At higher concentration of blowing agent, higher gas was generated by the blowing agent and thus it will promote the cell foam to expand more and subsequently the cell will combine and contact with each other to produce the bigger cell.

4.3.3 Scanning Electron Microscope (SEM)

Figure 4.10 shows the micrographs of average cell sizes of rubber foam at different sodium bicarbonate concentration as blowing agent. The length size of foam is depending on the concentration of sodium bicarbonate.



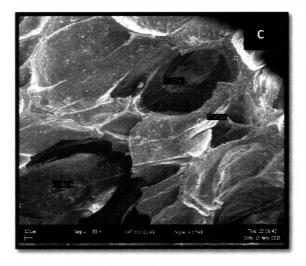


Figure 4.10: (a) Scanning electron micrograph of 4phr of sodium bicarbonate at magnification of 35x. (b) Scanning electron micrograph of 8phr at magnification of 35x. (c) Scanning electron micrograph of 12 phr at magnification of 35x.

The micrograph of the 4phr of sodium bicarbonate shows the characteristics of smallest average size of foam cells. In Figure 4.10(b), the average size of foam cells larger with incresing amount of sodium bicarbonate with 8phr. In Figure 4.10(c), the average size of foam cells largest with 12phr of sodium bicarbonate. At high concentration of sodium bicarbonates, higher gas is generated by the blowing agent from sodium bicarbonate and thus it will promote the cell foam to expand further and subsequently the cell will combine or contact with each other to produce a bigger cell. Then its affects the mechanical properties of the foams where foams with largest average size tend to have lower compression stress. This is because, the sample with high foam has a lot of empty space which contain more porosity and voids and the stress need to compress the sample was lower.

CHAPTER 5

CONCLUSION AND FUTURE WORKS

5.1 Conclusion

This conclusion is according to research that seen from revenue and discussion were made previously. In this study, the different blowing agent concentrations which are 4 phr, 6phr, 8phr, 10 phr and 12 phr were shown to influence the cell morphology of rubber foam, thus simultaneously affecting the physical and mechanical properties of the foam. As the blowing agent concentration increases, more carbon dioxide gas decomposes, resulting in a smaller relative density, higher expansion ratio, lower crosslink density, and higher water absorption rate, lower reading for compression testing and increasing the number of cells per unit volume.

It is proven from the result of this study that using sodium bicarbonate was capable in exerting enough carbon dioxide to produce rubber foam. From the study that was conducted, it was found that the lower amount of sodium bicarbonate with percentage concentration of 4phr was generate smaller cell size, lower value of expansion ratio, higher value of relative density and higher reading for compression stress. This subsequently affects the mechanical properties of the foams where foams with lower densities tend to have lower compression stress. This was contributed by the smaller cells exists in between the cell wall which minimize the microscopic collapsing and promote the recovery to the rubber foam.

At the end of this study, three objective of this research has been achieved which are to prepare rubber foam from reclaimed rubber glove through melt compounding using internal mixer, to study the effect of sodium bicarbonate loading as blowing agent in physical properties and mechanical properties and to characterize the physical properties and mechanical properties of rubber foam made from reclaimed rubber glove. Sodium bicarbonate loading as blowing agent was affect the physical properties and mechanical properties of the rubber foam. Several futures work can be done to further study on the properties of rubber foam such as

- 1. The effects of different ratio of accelerator between TMTD and CBS to the physical properties and mechanical properties of rubber foam.
- 2. The effects of different ratio of reclaimed rubber blend to the physical properties and mechanical properties of rubber foam.
- The effects of different type of blowing agent to the physical properties and mechanical properties of rubber foam.

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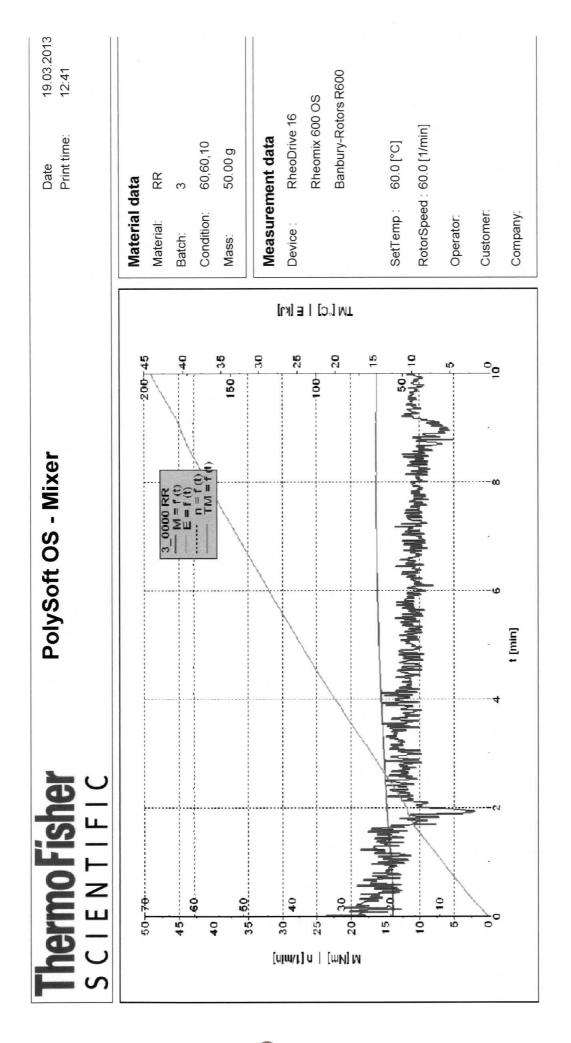
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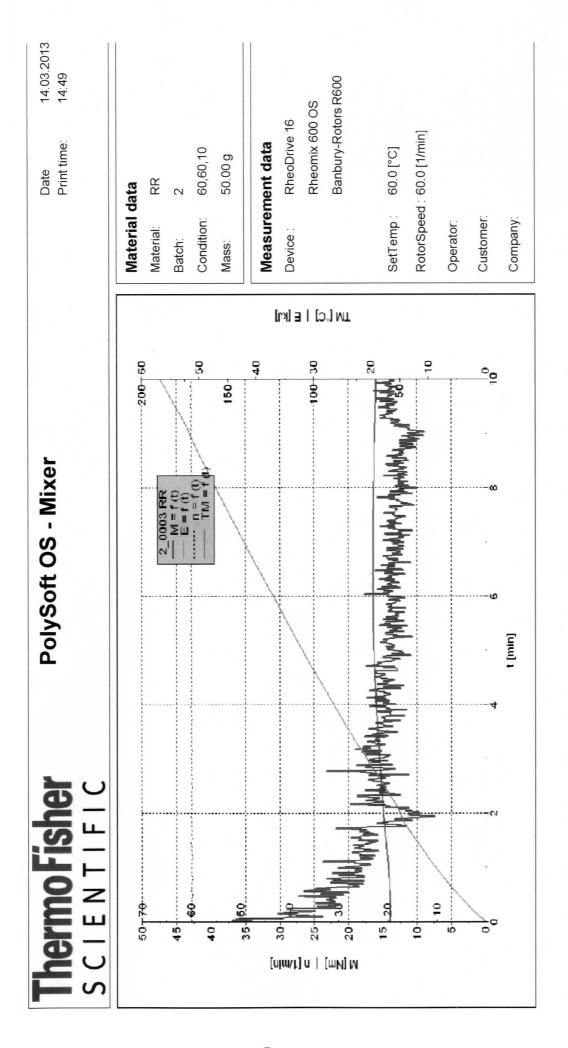
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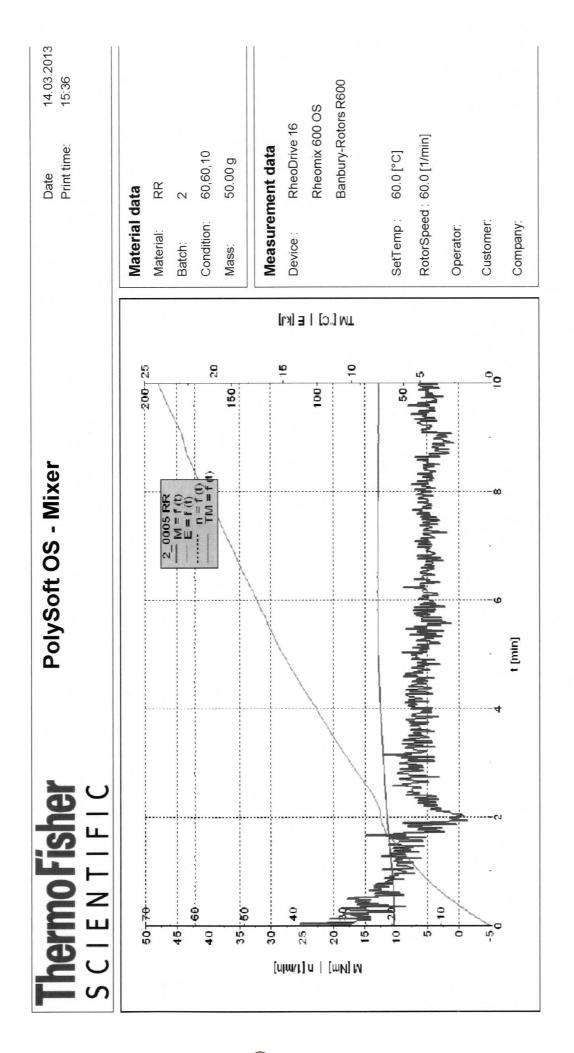
No	Task								Week	X							
		1 2	3		4	5	9	2	8	6	10	11	12	13	14	15	16
-	Selection of PSM title																
5	PSM title selected and filled the form																
33	Research, study and understand the synopsis of the title								-								
4	Find all the related information, journal																
	and references book															The second second	
5	Discuss the objective, scopes and problem statement with supervisor						0										
9	Rework and make correction for																
	objective, scopes and problem statement	-						_	_								
7	Meet and discuss the introduction with																
	supervisor																
8	Implement the introduction and																
	literature review				_					-							
6	Review the introduction and literature review																
10	Implement the methodology chapter			-													
11	Review the methodology by supervisor			-													
12	Complete the report																
13	Make a correction and submit to the														単語		
	supervisor	_	_	_	_	_	_	-	_								
14	Preparation for oral presentation (Power point slide)																
15	Presentation PSM 1																

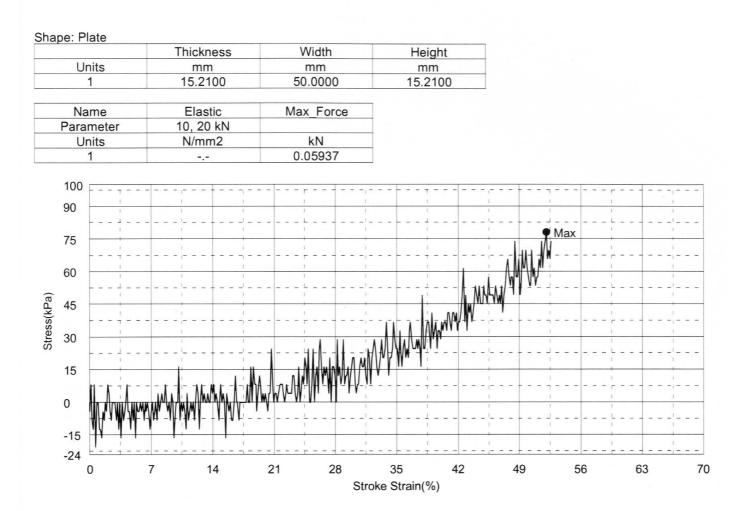
Appendix A

									Week	~						
		 2	Э	4	5	9	2	8	9 1	10	11	12	13	14	15	16
_	Sample preparation															
5	Internal mixer/Mixing parameter															
3	Hot compress															
4	Foaming process															
5	Density test and relative foam density test															
9	Crosslink density test															
9	Water absorption test															
~	Retention test															
10	Compression test															
=	OM and SEM															
12	Data analysis and result															
13	Literature review and result comparison															
14	Draft report submission and correction															
15	Submission of report															
16	Presentation preparation															
17	Presentation															

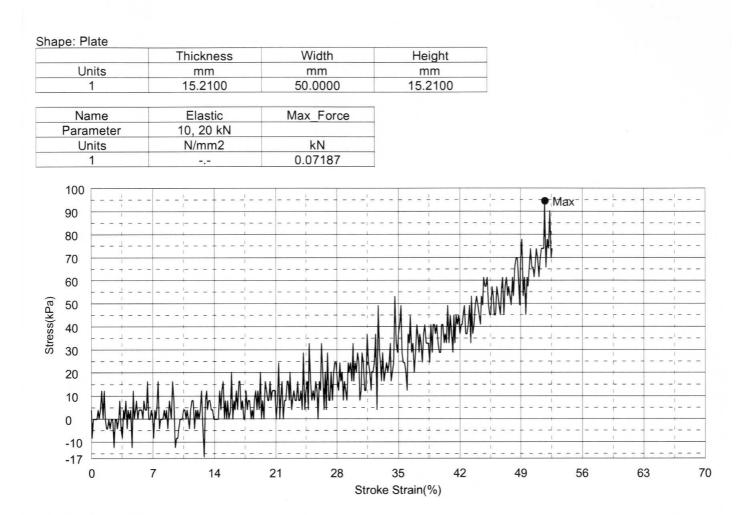




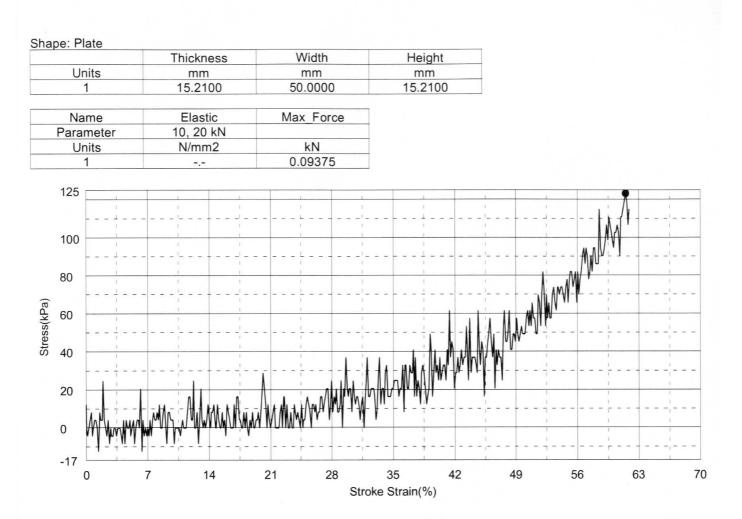




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