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THESIS

DEVELOPMENT OF NATURAL RUBBER FROM WATER HYACINTH FOR ALTERNATIVE SOUND ABSORPTION MATERIAL

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Sound absorptive materials are produced from solid dry natural rubber (NR) with the addition of 4,4’-oxybis (benzene sulfonyl) hydrazide (OBSH) as a blowing agent and were expanded using compression molding technique with utilization of heat transfer process. The cure behavior, physical, mechanical and acoustic absorption properties of the NR foams were investigated at three different foaming temperatures, i.e. 150, 160, and 170°C and different feeding ratio of the water hyacinth (0, 5, 10 phr) including blowing agent (0, 2, 4, 6 phr). Acoustics determination of sound absorption coefficient (α) in impedance tube according to ISO 10534-2 at thickness of 13 mm was used at two different ranges of frequency. This research is aim to study the influence of foaming temperature, water hyacinth and blowing agent content and the resultant NR foam cell structure on the acoustic properties called the noise reduction coefficient (NRC). This study focuses on the factors that affect the acoustic and mechanical properties of the foams that are related to changes in the cure behavior, morphology and foam physical properties. The results showed that the morphology, physical and compressive properties of the NR foams can be controlled closely by the foaming temperature. The increase of the foaming temperature led to different results compared to those from the increase of the water hyacinth and blowing agent content. Furthermore, it can be clearly observed that the NR foam filled with 10 phr of water hyacinth containing 4 and 6 phr of blowing agent at thickness of 13 mm foamed at 170°C, exhibits superior sound properties than NR foam produced at lower foaming temperatures. The results suggested that the suitable condition gave NR foams with bigger cell size, better cell distribution, and good compression deflection test, resulting in, the increase in α-value and NRC supporting the foaming efficiency results.

Student’s signature	Thesis Advisor’s signature	___ / ____ / _____

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LIST OF ABBREVIATIONS

ASTM = American Society of Testing and Materials
ADC = Azodicarbonamide
BET = Brunauer-Emmett-Teller
CBS = N-cyclohexyl-benzothiazole-2-sulphenamide
CBAs = Chemical blowing agents
DSC = Differential Scanning Calorimetry
DTA = Derivative Thermogravimetric Analysis
FTIR = Fourier transform infrared spectrometer
IPPD = N-isopropyl-N’-phenyl-p-phenylenediamine
MDR = Moving Die Rheometer
MPa = Mega Pascal
µm = Micrometer
NR = Natural rubber
NF = Natural rubber foam
NRC = Noise Reduction Coefficient
phr = Parts per hundred parts of rubber
rpm = rounds per minute
OBSH = 4,4’oxybis(benzenesulfonylhydrazide)/ blowing agent
PBAs = Physical blowing agents
SEM = Scanning Electron Microscope
STR = Standard Thai Rubber
α = sound absorption coefficient
TMQ = 2,2,4-trimethyl-1,2-dihydroquinoline
TTR = Thai Tested Rubber
TGA = Thermogravimetric analysis
ZnO = Zinc Oxide

DEVELOPMENT OF NATURAL RUBBER FROM WATER HYACINTH FOR ALTERNATIVE SOUND ABSORPTION MATERIAL

INTRODUCTION

The global concern on the problem of the infestation of water sources by water hyacinth (*Eichhornia crassipes* (Mart.) Solms), affect man and his well-being by impeding water flow in irrigation and drainage canals, altering the oxygen level of the water, thereby affecting fish and the quality of water for domestic and commercial use has provided the impetus for researchers and other conceited bodies to exploit ways of controlling its spread. Generally, efforts in this study have been directed at the complete elimination of this weed from our water sources, additionally, water hyacinth cover the water surface, so much that it obstructs water transportation. It is believed that water hyacinth is potentially beneficial to man, hence, this review was undertaken to emphasize its possible application in aqua-culture, bio-gas production, livestock feed, bin-fertilizer, waste water treatment and as raw material for industries. It is suggested that cottage industries be established in the affected communities to translate these possibilities into income earning sources. Thus, with institutional support from government and non-governmental organisations, the water hyacinth menace could be transformed from nuisance to wealth generation, employment and poverty alleviation.

Meanwhile, Thailand is the largest producer of natural rubber in the world. Despite its economic importance, its price has rised dramatically during indeed few years. More research and development in this area should be carried out to increase the value of natural rubber products by finding new applications, in particular for industries. This leads to more fuel consumption and more exhaust to be developed of alternative materials. This research is an attempt to make use of water hyacinth as filler by mixing natural rubber with ground fiber and various chemicals. The use of natural rubber in industrial and commercial applications continues to have great demand; one of the major areas is rubber foam, which is also known as cellular, sponge or expanded rubber. In these types of foamed polymers, bending of the cell edges and stretching of the cell faces are the primary mechanisms that control their structural response to mechanical deformation. The ability to perform these mechanical movements strongly depends on the foam density, cell microstructure such as cell size, shape and type (open or closed) and solid polymer properties by Klempner and Sendijarevic (2004).

Cellular rubbers or expanded rubbers are composite materials made up of a rubber matrix and a gas phase. Such materials have been used commercially in a wide range of applications including thermal insulations, gaskets, buoyancy, and impact sound deadening products. The mechanical properties of cellular rubbers are related to both the foam cells and the properties of the rubbers used. Structural variables include rubber crosslink density, foam cell density, cell structure, average cell size, and the cell geometry (open or closed cell). The processing of rubber foams involves curing an unvulcanized rubber compound that contains a blowing agent in a mould under high temperature and pressure until the blowing agent has decomposed on opening the mould, allowing expansion of the rubber compound (Roberts, 1990).

In recent years, a significant amount of interest has been shown in the potential of natural fiber to replace glass fibers in materials. This is the alternative way which is more economical and can be very cost-effective than using synthetic fibers. Although these fibers may not be as strong as carbon and aramid, their main advantages are low cost and biodegradability. Among the advantages of these fibers are: renewable, nonabrasive, cheaper, abundance and show less health and safety concern during handling and processing (Zulkifli *et al*., 2008). Mineral fibers such as glass fiber, rockwool, or asbestos have been used extensively in acoustical applications. However, the health hazard associated with asbestos fibers is now well known and this type of material is no longer widely used. Other man-made mineral fibers are not known to be as hazardous as asbestos but there are concerns with possible health effects, and it is standard practice to take safety precautions when installing or handling fiberglass or rockwool. Agricultural lignocellulosic fibers such as rice straw, wheat straw or oil palm frond can be easily crushed to chips particles and may be used as substituted for wood-based raw materials (Khedari *et al*., 2003; Wambua *et al*., 2003) their explore the use of local natural fiber as filler for composite system in the noise control application. Sound absorption coefficient and acoustic impedance were tested using impedance tube method. The tube was fabricated using standard ISO 10534-2. Foam has a wide range of applications, such as the use of foam as an acoustic absorbing material for sound. Sound absorption is the most important acoustic properties in engineering applications (Jaouen *et al*., 2008; Lee *et al*., 2006). Sound absorption can be explained by two mechanisms. The first mechanism is the viscous losses due to the friction between the air and solid phase. These two phases are elastically coupled, and a relative motion between the two phases exists. The second mechanism is the thermal losses due to heat transfer of the matrix (Hong *et al*., 2007).

Because undesirable and potentially hazardous noise is considered to be serious environmental pollution, various studies concerning acoustic properties have been performed. Regarding the influence of the cell characteristics, the sound absorption characteristics of flexible natural rubber foams were investigated by Adachi *et al*. (1997). For this study, NR foams were produced using solid dry NR foam that contained a vulcanization agent and a blowing agent that were added to the formulation during the compounding process using a two-roll mill. The use of dry rubber eliminates complexities in the process that can occur in the NR latex (liquid form of NR), which is often used for foam production. Liquid latex requires chemical stabilization for the colloidal stability, the critical storage time and environmental issues. This study uses 4,4’oxybis(benzenesulfonyl hydrazide); OBSH as a blowing agent, which releases nitrogen gas during its decomposition and is able to produce foam. This blowing agent system has a low foaming activity and non-toxicity; it sufficiently decomposes at a relatively low temperature range of 150-170°C. To compensate for its low ability of gas formation, a two-stage heat transfer process is proposed that involves an initial pre-vulcanization at different temperatures and a subsequent foaming in an air-circulating oven at the desired foaming temperature. Moreover, the cellular structures of the plant fibers also provide excellent insulation against heat and noise. In addition, most cellulosic fibers are harvested yearly and the supply should be inexhaustible compared to the limited supply of the oil reserve from which many synthetic fibers are derived. It was found that these could be achieved by the use of natural fibers. The use of cellulosic fibers in plastic composites is of particular interest because such fibers can serve as a good reinforce and/or filler for synthetic polymers to enhance certain properties while reducing material cost. The water hyacinth fibers have many advantages, such as low density, round plump twig, swelled in the middle of twig and air gap inside like sponge which can float in the water. It is sticky, strong, ease of fiber surface modification and wide availability. In addition, the use of fiber-reinforced plastic composite arises due to the increasing environmental consciousness such as the problem of convenient removal after the end of life time which is relatively stable and difficult to separate and recycle. Fillers can be classified into two types, namely, reinforcing and inert fillers. Reinforcing fillers, in the proper amount, enhance the strength of product. However, if the amount of the fillers is excessive, the rubber might lose its strength. Most of reinforcing fillers are in the form of small particles, such as carbon black or silica. In 1975, Anthoine *et al.* proposed that cellulose short fiber could be used as filler in natural rubber compounds. Its anisotropic characteristics cellulose short fiber increases the tensile strength and modulus of the natural rubber product but decrease elongation at break. It was also reported by Richard (1979) that, in order to maintain the elasticity characteristic of the product, the amount of short fiber should not exceed 10 phr.

Profound research has to be done to discover the suitability of water hyacinth fiber as filler in natural rubber foam in order to produce more economical sound absorption materials. The purpose of this research is to find the alternative uses of water hyacinths and natural rubber by exploring the possibility of using the fiber as filler in NR foam to produce sound absorption materials. Effects of the amount of water hyacinth fiber including factors that control the foaming process such as the change amount of blowing agent, and foaming temperature on noise reduction coefficient (NRC) of NR foams were investigated.

OBJECTIVES

1. To study the influences of the foaming temperature, water hyacinth, and blowing agent content on the cure behavior, cell morphology and physical properties of the natural rubber (NR) foams.
2. To investigate the factors affecting the mechanical properties of NR foams at different foaming temperatures and feeding ratios of the water hyacinth and blowing agent.
3. To examine the possibility of using water hyacinth fiber as sound absorption material in NR foams from the influences of the foaming temperature, water hyacinth, and blowing agent content on the acoustic properties.

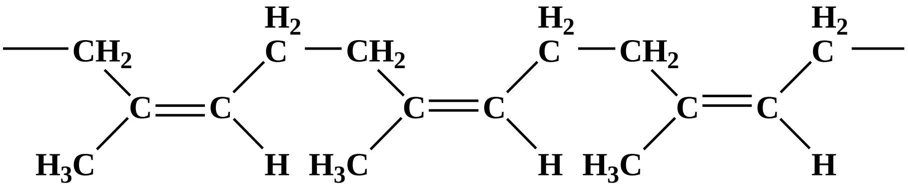
LITERATURE REVIEW

1. Natural Rubber (NR)

1.1 Introduction of natural rubber

It is estimated that there are more than 2000 species of trees, shrubs, or vines from mostly tropical and also temperate climate regions that produce milky fluid that congeals when exposed to air or known as latex from which natural rubber or a closely related material can be obtained. Nevertheless, latex from the trees of Hevea Brasiliensis is the only important commercial source of natural rubber. (Blow and Hepburn, 1982)

Natural rubber is a hydrocarbon consisting solely of carbon and hydrogen with an empirical formula of C_5H_8 with high molecular weight polymer of isoprene (Stern, 1982). Structurally, cis-polyisoprene is a highly stereoregular polymer with CH_3 group at the alpha terminal and three to four trans units at the omega end of the molecule (Figure 1). Molecular weight distribution of Hevea Brasiliensis rubber shows considerable variation from clone to clone, ranging from 100,000 to over 1,000,000. Natural rubber has a broad bimodal molecular weight distribution. The polydispersity or ratio of weight-average molecular weight to number-average molecular weight, M_w/M_n, can be as high 9.0 for some variety of natural rubber. This tends to be of considerable significance in that the lower molecular weight fraction will facilitate ease of processing in end product manufacturing, while the higher molecular weight fraction contributes to high tensile strength, tear strength, and abrasion resistance (Rodgers, 2004).



cis-Polyisoprene (repeat units, n = 1,500 to 15,000: M_w = 100,000 to 1,000,000)

Figure 1 Straight chain polymer structure of cis-1, 4 polyisoprene.

Source: Rodgers (2004)

1.2 Properties of natural rubber

Strength

Natural rubber is well-known for the strength properties of its vulcanizates. The tensile strength of gum vulcanizates ranges from 17 to 24 MPa while those of black filled vulcanizates range from 24 to 32 MPa. Strength can also be characterized as tear resistance, in both of which natural rubber is excellent. This high strength of natural rubber is certainly due to its ability to undergo strain-induced crystallization. The strength drops rapidly with increase in temperature but is still better than in other elastomers.

Elongation at break

The ultimate elongation depends, naturally, very much on the nature and amount of fillers in the compound, and on the degree of vulcanization. In general, it is about 500 to 1000%, or even greater.

Abrasion and wear

Natural rubber has excellent abrasion resistance, especially under mild abrasive conditions. Below about 35°C, natural rubber shows better wear than styrene butadiene rubber (SBR), while above 35°C, SBR is better.

Dynamic properties

Natural rubber has high resilience, with values exceeding 90% in well-cured gum vulcanizates. At large strain, the fatigue life of natural rubber is superior to that of SBR, the reverse is true for small strains. Good resistance to flexing and fatigue together with high resilience makes natural rubber useful in applications where cyclic stressing is involved.

Compression set

Compression set and related processes, such as creep, are poorer in natural rubber than in synthetic polyisoprene. At ambient and slightly elevated temperature, the compression set of NR vulcanizates is relatively low. At lower temperatures the compression set appears to be poor. Heat resistance of the NR vulcanizate has a detrimental effect on the compression set.

Aging resistance

Natural rubber vulcanizates can be given adequate heat-aging resistance by a suitable choice of vulcanization system and by use of amine or phenolic antioxidants. The heat-aging resistance of NR vulcanizates is insufficient for many technical applications.

Weather and Ozone resistance

Even after vulcanization, the NR has double bonds in the polymer chain. Therefore, it has an insufficient weather and ozone resistance, particularly in light-colored vulcanizates.

Solvents and Oils

Solvents will attack the rubber more rapidly with actual rates dependent on the types of solvent and rubber. Attack by contact with oils is usually restricted to a thin surface layer due to slow diffusion rate.

2. Water Hyacinth

2.1 Introduction of water hyacinth

Water hyacinth or *Eichhornia crassipes* (Mart.) Solms is a non-native, invasive, free-floating aquatic macrophyte. With board, thick and glossy ovate leaves, water hyacinth may rise some 1 meter in height. The leaves are 10-20 cm across, supported above the water surface by long, spongy and bulbous stalks. The feathery, freely hanging roots are purplish black. As much as 50 percent of a single water hyacinth's biomass can be roots, which extend to a depth of up to 2 feet in the water. Moreover, its dramatic lavender flowers and shiny green leaves make it highly prized as an ornamental plant. Water hyacinth is known as one by way of runners, eventually forming daughter plants. The growing season for water hyacinth is typically from March to early December. However, the majority of plants do not die and carry over plants begin to grow in spring as the weather warms. Plants can tolerate extremes of water level fluctuation and seasonal variations in flow velocity, extremes of nutrient availability, pH, temperature and toxic substances. It spreads and grows rapidly under favorable temperature and nutrient condition (warmer temperatures and higher nutrient levels).

Water hyacinth is thought to have originated in the Amazon basin and the extensive lakes and marshes of the Pantanal region of western Brazil. Water hyacinth has invaded freshwater systems in over 50 countries on five continents; it is especially pervasive throughout Southeast Asia, the southeastern United States, central and western Africa, and Central America as shown in Figure 2. Management notes for this location: the mottled water hyacinth weevil has been established in Australia, Fiji, Honduras, India, Malaysia, Papua New Guinea, South Africa and Thailand for control of water hyacinth.

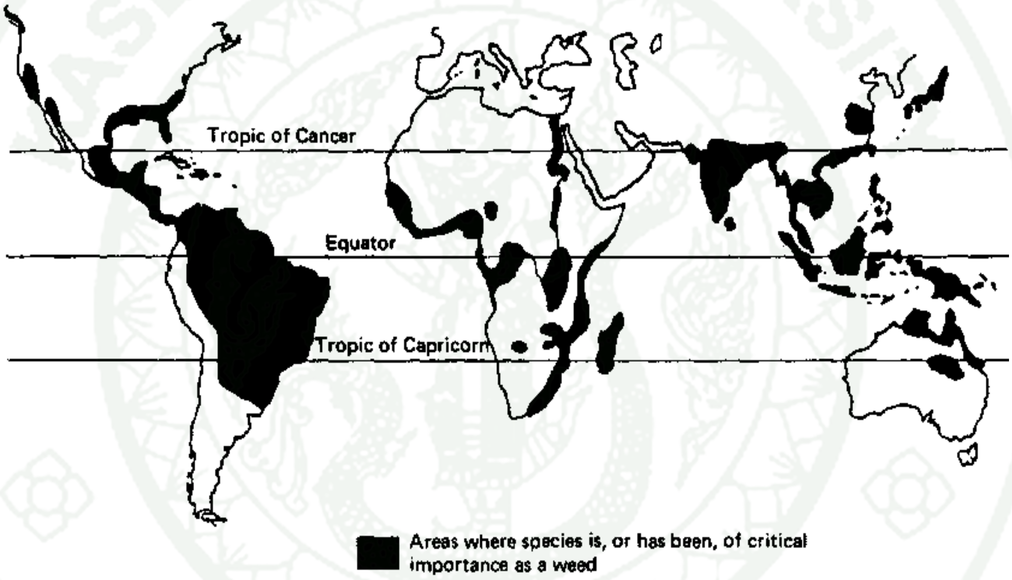


Figure 2 The spreading of water hyacinth which took photo by air.

Source: Anh (2007)

2.2 Properties of water hyacinth

Habit:

- water hyacinth is a floating, flowering perennial
- floating, "obligate" (requiring a wet habitat)
- linked plants form dense rafts in the water and mud

– will halt boat traffic on rivers; will cover a lake surface from shore to shore

Habitat:

– an aquatic weed worldwide; in lakes, rivers, ponds, ditches of temperate climates

– temperature tolerance: water hyacinth is not winter-hardy; its minimum growth temperature is 12°C (54°F); its optimum growth temperature is 25-30°C (77-86°F); its maximum growth temperature is 33-35°C (92-95°F) (Kasselmann, 1995)

Identification:

– monocot, perennial

– free-floating, except when stranded in the mud; mother plants and daughter plants attached by floating stolons

– leaves formed in rosettes rise to three feet above the water; leaves entire, ovate, rounded, circular, or broadly elliptic, to 6 in. wide; thick, glossy, waxy green, waterproof; sides gently incurved and often undulate; leaf base hearshaped, squared or rounded; veins dense, numerous, fine, longitudinal

– petioles (leaf stems) floating, creeping; inflated, bulbous, spongy, to 12 in. long

– multiple (8 to 15) flowers in a single very showy, spike (spathe) to 12 in. long; spike at top of erect thick stalk to 20 in. long, rising above the leaves; each flower in the flower-spike with six lavender-blue petals (perianths), petal tips slightly 2-lipped; uppermost petal somewhat larger, lavender, having a bright yellow, blue-bordered central oval splotch; stamens 6, stigmas 3

– roots hanging submersed beneath floating leaves, dark purplish to black, feathery, tips with long root caps

– fruit a capsule, 3-celled, with many seeds; seeds ribbed, formed in submerged, withered flower; fruit and seeds are rarely observed; seeds may produce many seedlings in moderate climates

Temi *et al.* (2007) investigated the biomass from water hyacinth, an abundant, low-cost, and undesired plant material, was used for the purification of aqueous systems polluted with Methyl Red (1). Herein, they reported the adsorptive capacity of this biomass for dye elimination from aqueous systems, elaborating optimal conditions for the batch removal of 1. Some surface properties of the biomass will also be covered. Reactive dyes, which represent the largest class of dyes used in textile-processing industries, typically contain azo compounds. The dyes used by the textile industry are required to show a high degree of chemical and photolytic stability to fulfill the fastness requirements of consumers. One consequence of this stability is that these dyes are not readily degradable under aerobic conditions prevailing in the biological-treatment unit at a sewage-treatment plant. Hence, the release of these compounds into the environment is undesirable, not only for aesthetic reasons, but also because many azo dyes and their by-products are either toxic or mutagenic (Jadhav *et al*., 2004).

2.3 Problem/Effects of water hyacinth

– *Eichhornia crassipes* mats clog waterways, making boating, fishing and almost all other water activities, impossible

– water flow through water hyacinth mats is greatly diminished

– an acre of water hyacinth can weigh more than 200 tons; infestations can be many, many acres in size; mats may double their size in as little as 6-18 days (Mitchell, 1976);

– water hyacinth mats degrade water quality by blocking the air-water interface and greatly reducing oxygen levels in the water, eliminating underwater animals such as fish (Penfound and Earle, 1948)

– water hyacinth greatly reduces biological diversity: mats eliminate native submersed plants by blocking sunlight, alter emersed plant communities by pushing away and crushing them, and also alter animal communities by blocking access to the water and/or eliminating plants the animals depend on for shelter and nesting (Gowanloch, 1944)

– in Florida, millions of dollars a year used to be spent on water hyacinth control; finally getting the plant under "maintenance control" has greatly reduced that expenditure

Control ; Due to decades of university, state and federal research and experience with *Eichhornia crassipes* in the U.S., several methods have been developed to help in its management:

– the action of mechanical harvestors and chopping machines remove water hyacinth from the water and transport it to disposal on shore; chopping machines grind the plant into bits and spray the slurry across the water

– years of research to find insect biocontrols has resulted in the successful introduction of two water hyacinth weevils, which are believed to be keeping water hyacinth under maintenance control in many places; however biocontrol fish which are able to control submersed plants are ineffective against the floating water hyacinth

– registered aquatic herbicides do provide temporary control of water hyacinth

Mahmood *et al*. (2005) reported that the water hyacinth (*Eichhornia crassipes* (Mart.) Solms) is a prolific free floating aquatic macrohpyte found in tropical and subtropical parts of the earth. The effects of pollutants from textile wastewater on the anatomy of the plant were studied. Water hyacinth exhibits hydrophytic adaptations which include reduced epidermis cells lacking cuticle in most cases, presence of large air spaces (7~50 µm), reduced vascular tissue and absorbing structures. Textile waste significantly affected the size of root cells. The presence of raphide crystals was noted in parenchyma cells of various organs in treated plants.

Water hyacinth has long been used commercially for cleaning wastewater. The luxuriant plant's tremendous capacity for absorbing nutrients and other pollutants from wastewater has long been overlooked by many wastewater engineers. In recent years, the plant has been used to treat a variety of wastewaters and to produce high protein cattle food, pulp, paper, fiber, and more importantly, biogas as energy source

(Agency for International Development, 1976; Bates and Hentges, 1976; Kojima, 1986).

Amy (2009) reviewed studies conducted on water hyacinth worldwide and found that the effects of water hyacinth on water quality are similar but the magnitude of effects is dependent on the percent cover and potentially the spatial configuration of water hyacinth mats. Water hyacinth’s effect on aquatic invertebrates, fish, and waterbirds is less predictable and dependent on conditions prior to invasion.

Pranut *et al.* (2001) studied about using water hyacinth fiber as a filler in natural rubber, which there was mixing natural rubber (TTR 20) with ground fiber and various chemicals. Effects of the amount (5 and 10 phr) and size (fine, medium and coarse) of the fiber on the mechanical properties of the products were investigated. It was found that the hardness and modulus of the products were higher than those of the unreinforcing compound whereas the tensile strength of the product was lower than that of the unreinforcing compound. As the amount and size of the fiber were increased, the hardness and modulus of the products would increase whereas the tensile strength would decrease. However, the abrasion resistance of the products was lower than that of the unreinforcing compound and the resistance would decrease as the amount and size of the fiber were increased. Better mechanical properties of the product could be obtained by using suitable coupling agent which would enhance the adhesion between Water hyacinth fiber and natural rubber.

3. Rubber Compounding

Processing is termed as the complete sequence used in industry to convert raw rubber into a useful rubber product. There are usually three steps: mixing, forming/ shaping and vulcanizing.

The mixing stage is generally understood as the mastication and compounding of the rubber compound. The reason for mixing is to incorporate the compounding ingredients into the rubber and distribute it as uniformly as possible within the rubber compounds. With NR, it may be necessary to add a preliminary stage: in which NR on its own or with the addition of peptizers is subjected to a shearing process to reduce its viscosity prior to mixing.

The forming or shaping stage came subsequently after mixing. This is to ensure that the mixed rubber compounds are converted or processed into the shape and form of the final finished products. Most of the time, shaping and vulcanization stage is carried out as one operation. Lastly, vulcanization process takes place. Vulcanization is the heating of the shaped article to cause cross linking to take place. This then will produce useful elastic products by means of cross linking agents. The vulcanized rubber product made then has to meet various physical properties such as hardness, modulus, tear strength, abrasion resistance etc.

Materials used in dry rubber compounding can be classified into eight major categories, namely: rubber, processing aids, curing agents, accelerators, activators, antidegradant, fillers, miscellaneous ingredients which include retarder, inhibitors, pigments and blowing agents. In conclusion, Table 1 shows the materials required for compounding, the functions of the materials and the common proportions used in rubber compounding.

Table 1 A general rubber formulation.

Material Property Required	**Compounding Ingredient Function**	**Common Proportions**
Non thermoplastic properties i.e. Elastic behaviour	Basic Additive Crosslinking or vulcanization reagents. Reaction catalysts or accelerators.	Parts by weight 1 -3 0.5 - 3
Protection from degradation by O_2, O_3	Antidegradants i.e. antioxidant additives	1 - 4

Table 1 (Continued)

Material Property Required	**Compounding Ingredient Function**	**Common Proportions**
Ability to design for a specific stiffness or hardness	Use of fillers especially small particles size powders: Examples are carbon blacks, silicas, calcined clays.	10 - 100
Processability i.e. shaping before crosslinking	Processing Aids: Oils, Waxes, Soaps etc.	1 - 10
Adhesion to textile, metal, ceramic etc. substrates	Integral bonding additives and surface treatments	2 - 10
Colour changes	Pigments as organic Pigments as inorganic	0.1 – 3.0 5 - 20
Cost Reduction	Extender fillers, Depolymerized reclaimed rubber, Ground vulcanized rubber	10 – 200 10 – 100 5 - 50
Cellular structures	Blowing agents e.g. organic, inorganic	0.5 – 2.0 ; 5 - 30
Reduced fire hazard (self extinguishing)	Flame retardants e.g. phosphates, antimony salts, halogenated organics, borates (Antimonytrioxide and chlorinated wax – common for NR)	1 - 20
Magnetism	Magnetic inorganic salts e.g. iron oxides	5 - 100
High energy radiation absorption	High energy absorbers e.g. heavy metal salts, lead oxides	10 - 100
Electrical insulation	Non conductive ingredients e.g. mineral fillers, hydrocarbon oils	5 – 50

Table 1 (Continued)

Material Property Required	**Compounding Ingredient Function**	**Common Proportions**
Electrical anti-static	Anti-static additives, polar esters, soaps; small amount of black	0.1 – 2.0
Electrical conductivity	Conductive ingredients e.g. carbon black, metal particles and their salts	10 - 50
Bacteria resistance	Fungicides e.g. chlorinated phenols.	0.5 – 5.0

Source: Blow and Hepburn (1982)

3.1 Mixing

Compounding - that is, mixing together a variety of components such as different polymeric materials, solid, and liquid additives, sometimes accompanied by a chemical reaction - is often used to produce a polymeric material with new and improved properties. In fact, compounding actually offers a far less expensive and more practical route for creating a wide variety of new, improved products than the development of chemically new polymers (Tadmor *et al.* 2006). The objectives of mixing are to obtain uniformed ingredients with good dispersion of each ingredient and to produce consistent batches which are uniform in viscosity and in degrees dispersion.

The two basic machines for this process are the two-roll mill and the internal mixer. Rubber mixing is a partly physical, partly chemical process. For mixing, there are two stages i.e. the mastication and compounding of the rubber compound. The purpose of mastication is to reduce the viscosity and increase the plasticity of natural rubber and some synthetic rubbers brought about by mechanical milling. Decrease in viscosity for rubber is also achieved by heat generated through rubber shearing between rubber - rubber and rubber - mixing machine. This directly will improve the processability of the material for next stage. The purpose of compounding is to incorporate, dissolute or partially dissolute and physically disperse compounding ingredients and reduce their particle size.

Mixing should be optimized with respect to time, temperature, and energy. Compound viscosity should be reduced only to that level which allows acceptable processing in the ongoing manufacturing stages. Uniform distribution and optimum dispersion of all compounding materials should be achieved, and the influence on scorch time has to be minimal and/or controllable. If possible, the tackiness of the compound should be controlled. Both excessive sticking to the machines and bagging on the mill due to a lack of stickiness must be avoided (Rodgers, 2004).

3.2 Forming

Rubber compound that has been put through the mixing stage generally requires some kind of forming or shaping into blanks of suitable dimensions before it can be vulcanized. At this stage the stock will retain the shape imposed on it because it is predominantly plastic (Gent, 1992). Ideally rubber compounds should have a smooth surface, uniform shrinkage, and freedom from blisters. Last but not least, bloom should be avoided. The majority of products are produced by combined forming or shaping and vulcanization operation known as moulding. Three most widely used moulding methods are compression, transfer and injection moulding. A steel or aluminium mould having a cavity of the product shape required is heated to the vulcanization temperature (140-200°C). The differences arise in the method of introducing the unvulcanized rubber into the mould (Hofmann, 1989).

For compression moulding, a pair heated platens mounted on hydraulic press is used. A shape and weighed of rubber blanks is inserted into the mould which is then reassembled and placed between the platens of the hydraulic press. The heat and compression force generated by the hydraulic press causes the rubber to flow and takes shape according to mould. The compression force generated by the hydraulic press also helps to contain the expansion of volatiles within the rubber mix until dimensional stability of the rubber mix is achieved. The vulcanization time is highly dependent upon this three factors i.e. temperature, size of product and their heat transfer.

For transfer moulding, the heated hydraulic press used is similar as used in compression moulding. The principle is the same in both instances; the mould cavity is closed before the moulding operation starts, the rubber is then introduced into a secondary cavity adjacent to the shaped product-forming cavity and is transferred by hydraulic ram pressure to the primary mould cavity through channels known as runners. The advantage of this system is twofold; the rubber received considerable fractional heating in transfer, shortening the vulcanization time; and metal inserts used in rubber to metal bonding may be positively located. The majority of engineering rubber components is produced by this method (Morton, 1987).

For injection moulding, it is a bit more complicated compared to compression and transfer moulding. Processing cycle wise, it involves automatic feeding, heating and plasticization of a rubber mix and its high pressure in measured quantity, through a narrow orifice into a tightly closed mould in which vulcanization takes place.

The advantage of this type of machine is in the heat generated by thermal conduction through the mechanical work done by the screw. Heat can be carefully regulated by the screw speed, back pressure and barrel temperature. As the screw plasticizes, the rubber mix progresses along the retractable screws and fills up the required preset volume in front of the screw. This volume of rubber mix is then injected into the mould. Heat generated as the mix is forced through the mould gates (Blow and Hepburn, 1982).

3.3 Vulcanization

After the rubber compound has been formed into the desired shape, it needs to be converted to an elastic material. This is achieved through subjecting the rubber compounds through vulcanization process, which usually takes place at a high temperature under elevated pressure, using different techniques, such as press vulcanization, open vulcanization, continuous vulcanization and cold vulcanization. Chemically, this process is about introducing crosslinks between polymer macromolecules through the action of vulcanizing ingredients. Without these chemical bonds, no improvements in the physical properties of the rubber mix can take place. The crosslinks tie the macromolecules together in such a way that the whole mass becomes a single interconnected three dimensional molecule. When rubber is vulcanized, sulphur molecules crosslink the polymer strands. This crosslinking reaction helps to strengthen the vulcanizates and allows it to retain its natural elastic disposition that is their ability to undergo large elastic deformations, that is, to stretch and return to their original shape in a reversible way (Morton, 1987).

4. Vulcanizing Ingredients

Vulcanizing agents are chemicals which are incorporated in order to insert cross-linking between the polymer chains when the compounded stock is heated to an appropriate temperature. A cross-linking is formation of chemical bonds between polymer chains to give a network structure. The main purpose of vulcanization is to convert the essentially plastic raw rubber into an elastic and dimensionally stable material and also to render its physical properties temperature independent to a greater extent while making it insoluble in liquids which would have dissolved the unvulcanized rubber (Freakly and Payne, 1978). The type of cross-linking agent required will vary with the type of rubber used; however, they can usually be grouped in the following categories:

– Sulfur and related elements: the most common agent used is sulfur, as it enters into reactions with the majority of the unsaturated rubber to produce vulcanizates. In addition, two other elements in the same periodic family, namely selenium and tellurium, are capable of producing vulcanization. Selenium and tellurium are used in place of sulfur where excellent heat resistance is required. They generally shorten cure time and improve some vulcanizate properties.

– Sulfur-bearing chemicals: accelerators and similar compounds can be used as a source of sulfur for the vulcanization of natural rubber and styrene-butadiene rubbers in recipes using very small amount of elemental sulfur.

– Nonsulfur vulcanization: most nonsulfur vulcanization agents belong to one of three groups: metal oxides, difunctional compounds, and peroxides. Carboxylated nitrile, butadiene, and styrene-butadiene rubbers may be crosslinked by the reaction of zinc oxide with the carboxylated groups on the polymer chains. This involves the formation of zinc salts by neutralization of the carboxylate groups. Other metal oxides are also capable of reacting in the same manner. Certain difunctional compounds form crosslinks with rubbers by reacting to bridge polymer chains into three-dimensional networks. Organic peroxides are used to vulcanize rubbers that are saturated or do not contain any reactive groups capable of forming crosslinks. This type of vulcanization agent does not enter into the polymer chains but produces radicals which form carbon-carbon linkages with adjacent polymer chains (Morton, 1973).

4.1 Accelerators

The function of an accelerator is to increase the rate of vulcanization. Accelerators can cut the vulcanization time from hours to minutes or seconds at high temperatures; and at lower temperatures the vulcanization time may be reduced from months to hours or minutes. This reduction is of a great importance as this result in very high production rates and reduction in capital investment. The main reason for using accelerators is to aid controlling the time and/or temperature required for vulcanization and thus improves properties of the vulcanizate. The reduction in the amount of time required for vulcanization is generally accomplished by changing the amounts and/ or types of accelerators used (Morton, 1973). At one time, basic oxides such as lime, litharge, and magnesia were widely used as accelerators.

Today, accelerators are almost always organic compounds containing either nitrogen or sulfur or both. Very few accelerators, known as sulfur donors, such as tetramethylthiuram disulphide, can be used as vulcanizing agents without the addition of elemental sulfur. According to speed of action, accelerators are sometimes described as slow, moderately fast, fast and ultra accelerators (Nagdi, 1993).

4.2 Activators

These components are used to increase the vulcanization rate by activating accelerators so that it performs more effectively. It is believed that they react in some manner to form intermediate complexes with the accelerators. The complex thus formed is more effective in activating the sulfur present in the mixture, thus increasing the cure rate. Accelerators are grouped as follows:

– Inorganic compounds (mainly metal oxides): zinc oxide, red lead, white lead, magnesium oxide, alkali carbonate, etc. Zinc oxide is the most common and it is often used in combination with a fatty acid to form a rubber-soluble soap in the rubber matrix.

– Organic acids: are normally used in combination with metal oxides; they are generally high molecular weight monobasic acids or mixtures of the following types: stearic, oleic, lauric, palmitic, etc.

– Alkaline substances will increase the pH of a rubber compound and in most instance increase the cure rate. As a rule of thumb, in the majority of recipes, any material which makes the compound more basic will increase the cure rate since acidic materials tend to retard the effect of accelerators (Morton, 1973).

4.3 Antioxidants

Antioxidants design to inhibit oxidative and ozone-caused deterioration, but ultraviolet light protectors and antiflex agents are included as well. The results of oxidative attack depend on the polymer, like NR, become soft and sticky. Ozone attack is manifested by cracking at the surface perpendicular to the stress. In the selection of antioxidant, the following factors must be considered: type of protection desired, chemical activity, discoloration, staining and cost. *N*-isopropyl-*N'*-phenyl-*p*-phenylenediamine (IPPD) is one of the most popular antioxidants being used in compounding; the chemical structure is shown in Figure 3.

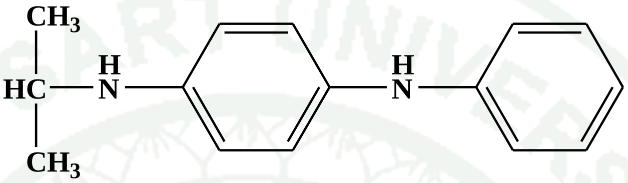


Figure 3 *N*-isopropyl-*N'*-phenyl-*p*-phenylenediamine (IPPD).

Source: Roff & Scott (1971)

4.4 Special-Purpose Ingredients

Certain ingredients are added for special purposes, but these ingredients are not normally required in the majority of rubber compounds. Examples include coloring pigments, blowing agents, flame retardants, odorants, antistactic agents, retarders and peptizers.

Coloring pigments are substances added for coloring nonblack rubber goods. It is important to note that only the nonstaining grades of crude rubbers, age resistors, accelerators and other ingredients should be used for colored compounds. Coloring pigments are usually divided into two groups: inorganic or mineral pigments and organic dyes.

Blowing agents are gas-generating chemicals that are necessary for manufacturing sponge and microporous rubber products. Suitable agents that are capable of releasing gas during the vulcanization period include sodium bicarbonate, ammonium carbonate and certain nitrogen-bearing compounds. The released gas brings about a cellular or spongelike structure.

Flame retardants are chemicals added to reduce the flammability of the end product. Materials used extensively for this purpose include, for example, chlorinated hydrocarbons, certain phosphates and antimony compounds. Odorants or odor improvers are strongly scented substances added in very small amounts (about 0.1 phr) that are capable of masking the characteristic odor of some rubber compounds or imparting a scent. Vanillin is frequently used for this purpose.

Antistatic agents are sometimes added to reduce the accumulation of dust or dirt on the surface of the elastomeric part during service and also to minimize the possibility of sparking resulting from discharge of accumulated static electricity. Typical antistatic agents include certain esters, fatty amines and amides. Retardants are substance used to reduce the tendency of a rubber mix to scorch, that is, to avoid premature vulcanization during factory processing. Peptizers are compounding ingredients used in small proportions to accelerate the softening crude rubber under the influence of mechanical action, generally induced on open roll mills or in internal mixers (Nagdi, 1993).

5. Fillers

Since the early days of the rubber industry, fillers in the form of fine particulates have been used in rubber compounding. Particulate fillers are usually divided into two groups, inert fillers and reinforcing fillers. Inert fillers are added to the rubber to increase the bulk and reduce costs. In contrast, reinforcing fillers such as carbon black and silica are incorporated in the rubber to enhance the mechanical properties, to change the electrical conductivity, to improve the barrier properties or to increase the resistance to fire and ignition (Alexandre & Dubois, 2000, Nugay & Erman, 1999). Reinforcement of elastomeric compounds, defined as the simultaneous enhancement of the elastic modulus and the elongation at break, by addition of particulate filler, is probably one of the most important phenomena in material science and technology. The increase in stiffness imparted by the filler particles involves a hydrodynamic effect, which depends on the filler volume fraction, but the occlusion of rubber by the aggregate and the rubber trapped within the filler agglomerates may increase the effective filler volume. On the other hand, the shape factor (anisometry) also contributes to the increase in moduli (Bokobza & Chauvin, 2005).

6. Principles of foam formation

The most widely used system of producing foamed polymers involves dispersing a gas throughout a fluid polymer phase and stabilizing the resultant foam. In most of these systems the foam is expanded by increasing the bubble size before stabilizing the system. Foams produced by this dispersion process include several major types (Klempner and Frisch, 1991):

(a) Thermoplastic polymers, which are foamed first as solids, then melted to provide the fluid phase, foamed, and then cooled to solidify and thus stabililize the foam.

(b) Thermoset foam systems, in which the reactants are foamed while only partially reacted and are still fluid, followed by curing to the thermoset state to stabilize the foam

(c) A latex that is foamed and then stabilized by phase inversion, achieved by lowering the pH, freezing, or both, frequently with additional curing in the foamed state.

There are three steps of the fundamentals of foam formation (i) the nucleation of gas bubbles in a liquid polymer, (ii) the growth and stabilisation of these bubbles, and (iii) the solidification of the polymeric phase by crosslinking or cooling to give a structurally stable cellular system (Duanghathai and John, 2005).

6.1 Bubble formation

In the general case described above, the first step in producing foam is the initiation of gas bubbles in a liquid system. This process is known as nucleation. In many cases, it is necessary to add nucleation agents such as talc and chemical silica to control cell size and cell distribution. Several techniques are used to entrain the gas in the liquid polymer before expansion.

Bubbles nucleation is affected by a number of conditions. Physically the effects of temperature, pressure, and in some cases humidity are fairly obvious. Other important parameters are surface smoothness of the substrate, surface characteristics of filler particles, presence and concentration of certain surfactants or nucleators, size and amount of second-phase liquid bubbles, and the rate of gas generation.

6.2 Bubble growth

Saunders (1960) reported that a better bubble stability, a small average cell size, and a more even bubble size distribution is produced at low interfacial surface tension. Schematic representations of idealised cellular structures at different stages of bubble expansion are shown in Figure 4. The foam depicted in Figure 4(a) shows the bubbles uniformly dispersed throughout the liquid medium and if stablilized in this condition the foam would clearly have a high density. As the gas volume fraction increases, Figure 4(b), the foam density decreases. For spherical bubbles of uniform size, the lowest density is achieved when the bubbles are close packed.

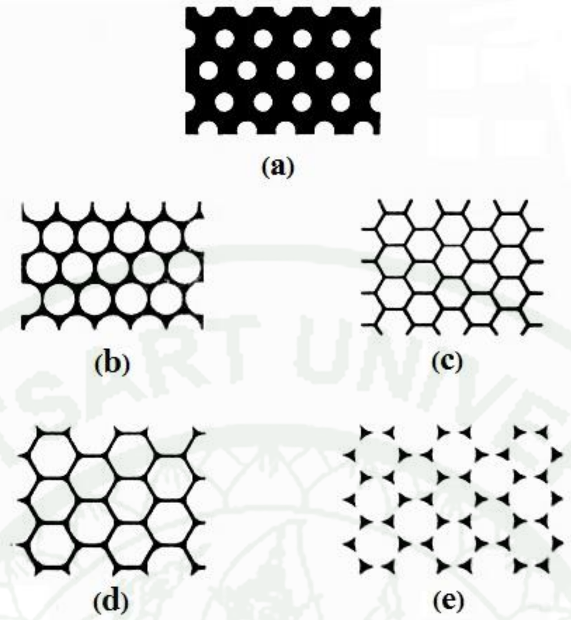


Figure 4 Schematic representations of cellular structures at different stages of foam expansion.

Source: Duanghathai and John (2005)

On further expansion, the bubbles eventually touch each other and the internal gas pressure presses the cells together causing the polymer to fill the interstices (available spaces). Thus, the cells become bounded by polyhedra, often represented in an idealized form as a pentagonal dodecahedron with plane surfaces of uniform thickness, but simplistically shown in two dimensions as a hexagonal honeycomb (Figure 4(c)). Depending on the physiacl state of the liquid system, i.e. its viscosity and surface energy, gravitaytional and other forces may cause the liquid material to concentrate at the intersection of the elements of the associated cells as depicted in Figure 4(d). At this stage the foam is of low density and is closed cell. However, the membranes between the cell struts, which represent the interface between the gas bubbles, may become ruptured and give varying degrees of connectivity between the cells. This gives rise to an open cell structure as represented in Figure 4(e).

Bubble stability during growth is a function of type and concentration of surfactant present, the rate of bubble growth, the viscosity of the fluid medium, any differential gas pressure variations, and the presence of cell-disrupting agents such as solid particles, liqiuds, or gases. As the cell walls are squeezed into polyhedra, a wall-thinning effect takes place, and liquid is drained from cell wall faces into the cell intersection regions to form ribs or struts, which are typically triangular in cross section. If allowed to, this cell wall membrane thinning process can continue to the point where the cell walls collapse and the cells become interconnecting. This mode of cell formation and development is a very important characteristic of most plastic foams and will affect thermal conductivity, moisture absorption, breathability, and load bearing properties.

The extent of drainage of liquid polymer from the membrane into the struts will be influences by capillary action and gravity. Capillary drainage is proportional to the square of the distance between rib junctions. Increasing the viscosity of the fluid reduces the drainage effect. Viscosity increase may be brought about by (i) chemical reactions to increase molecular wieght through polymerisation or crosslinking or (ii) temperature reduction (thermoplastics).

6.3 Bubble stabilisation

Ultimate stabilisation is due to either chemical reaction continuing to complete gelation or the physical effect of cooling below melting point or softening point to prevent polymer flow. As complete solidification is approached the previously formed spherical bubbles may become distorted by the flow of foam or by gravity, with resultant anisotropy of cellular structure. This effect must be considered when evaluating the physical properties of polymeric foams. Also, there is a tendency for the gas within the cell to equilibrate over a period of time at ambient temperature and pressure. This can result in changing properties with time as the balance between gas diffusion out of the cell and air diffusion into the cell takes place as the foam ages. For obvious reasons the “ageing” stage is minimised when the expanding gas within the cell is the same as, or similar to, air. For example, nitrogen blown foams have no apparent ageing problems whereas hydrocarbon blown foams present significant ageing difficulties due to the diffusion rate of gas out of the cell being significantly greater than the diffusion rate of air into the cell. Consequenly the foam is prone to collapse after manufacture, recovering slowly with ageing as air diffuses back into the cells. This problem is overcome in the manufacture of expanded polystyrene (EPS) moulding by first preparing pre-expanded beads. These beads are equilibrated before being charged into a steam chest for moulding.

Foams prepared by the dispersion process generally have three steps involved: bubble formation, bubble growth, and bubble stabilization. A surface tension depressant is very helpful and often essential for each of these steps. Bubble formation normally requires the presence of a large number of microvoids or a finely divided nucleating agent of some sort. Self-nucleation is unlikely. Bubble growth results primarily from the diffusion of gas from the solution into the microvoids or foam bubbles (Klempner and Frisch, 1991).

Four effects, in addition to the use of a surface tension depressant, promote stabilization of foam bubbles.

1. An increase in viscosity of the polymer phase, as by cooling or further polymerization, is the major stabilizing influence.
2. A reduction in the surface excess of the adsorbed component (the surface tension depressant), as bubbles grow and films become thinner, increases the surface tension at the point; flow of more component from other surfaces of higher concentration brings some liquid polymer with it, thus providing self-healing.
3. An increase in surface area requires an increase in free energy; a very small increase in area is necessary for rupture to occur, so there is an activation energy of rupture.
4. An electric double-layer effect can cause the two surfaces of a film to repel each other.

Four other factors promote the thinning of membranes, and hence rupture and collapse.

1. Drainage of fluid from the membranes to the ribs, due to capillary action and gravity, is the most important factor.
2. Excessive expansion, so there is not enough polymer to provide adequate cell walls and ribs, is also an important factor limiting the degree of expansion that can be obtained.
3. Van der Waals attractions between the surfaces of a thin film may lead to further thinning.
4. Any local depression of surface tension (e.g., due to excessive heat or antifoam agents) may cause voids or collapse.

Two additional factors make foaming difficult. An increase in the free energy of the system occurs on foaming, in direct proportion to the increase in overall area. The thermodynamics thus favor collapse. The second is the delicate balance usually required between the strength of membranes and ribs and the time of reaching maximum foam rise. The ribs, at least, must be strong enough to maintain the foam volume at that time. The membranes must break at that point for the production of open-cell foams, and they must not break for the production of closed-cell foams.

7. Cellular materials

A cellular material is defined as ‘A generic term for materials containing many cells (either open, closed, or both) dispersed throughout the mass’ (ASTM D1056). Cellular polymers, or polymeric foam, are multi-phase materials consisting of a minimum of two phases; a contiuous polymer matrix phase and a gaseous phase. There may be more than one solid phase present, as in the case of a blend or alloy of polymers. Other solid phases may be present in the form of fillers, either fibrous or other shapes, e.g. glass, ceramic, metal, or other polymers (Duanghathai and John, 2005).

7.1 Cellular rubber

Cellular rubber is prepared from solid rubber containing vulcanizing agents and a blowing agent, usually one that generates nitrogen or carbon dioxide. Bascom (1964) described the relationships among temperature, viscosity, decomposition temperature of the blowing agent, and whether open or closed cell result. Initial heating reduces the viscosity, of course, until the temperature is reached at which vulcanization starts, when the viscosity rises. The use of a blowing agent that decomposes just before the minimum viscosity is reached gives open cells, because the expansion will occur rapidly and membranes will rupture before cross-linking stabilizes them. For closed-cell foams one should use a blowing agent that decomposes as the viscosity begins to rise, so the cell membranes will be sufficiently cured that they do not rupture when the foam rise is completed.

ASTM D1056 pointed out that the expanded rubbers or closed-cell rubbers are made by incorporating gas-forming ingredients in the rubber compound, or by subjecting the compound to high-pressure gas such as nitrogen. Expanded rubbers are manufactured in sheet, strip molded, and special shapes by molding or extruding. Unless otherwise specified, the presence of skin on the top or bottom surfaces of sheet and strip expanded rubber shall be optional. Extruded shapes have skin on all surfaces except cut ends. On the other hand, sponge rubbers are made by incorporating into the compound an inflating agent, such as sodium bicarbonate, that gives off a gas which expands the mass during the vulcanization process. Sponge rubbers are manufactured in sheet, strip, molded, or special shapes. Unless otherwise specified, sheet and strip sponge rubber shall have a natural skin on both the top and bottom surfaces. Fabric surface impressions are ordinarily not objectionable. The coarseness of the impressions shall be agreed upon by the parties concerned.

Cellular rubber is a generic term that includes both synthetic and natural materials. Natural rubber includes urethane foam, latex foam, expanded rubber and sponge rubber. Cellular rubber commonly refers to latex foam rubber, an invention of the Dunlop Rubber Company (UK) in 1929. Alternatively, rubber foams can also be produced using the solid form of NR or often called dry NR. Cellular rubber was manufactured using natural latex initially, but latex was replaced with synthetic rubber owing to its disadvantages (bad odor, high shrinkage and poor aging). Such materials have been used commercially in a wide range of applications including thermal insulations, gaskets, buoyancy, and impact sound deadening products. The mechanical properties of cellular rubbers are related to both the foam cells and the properties of the rubbers used. Structural variables include rubber crosslink density, foam cell density, cell structure, average cell size, and the cell geometry (open or closed cell). The processing of rubber foams involves curing unvulcanized rubber compound that contains a blowing agent in a mould under high temperature and pressure until the blowing agent has decomposed on opening the mould, allowing expansion of the rubber compound (Roberts, A. D. 1990).

Ariff (1998) purposed that the processing of rubber foam from dry rubber involved compounding rubber with vulcanizing agents and blowing agent. The compounded rubber will be cured and simultaneously foamed at different temperatures to produce foams with range of end properties. Dry rubber foaming can be implemented through two processes which are called single stage process and heat transfer process. The expansion of the compound for the single stage process occurs while opening the mold. While in the two-stage heat transfer process, the first stage is identical to the single stage process but using lower temperature. On opening of compression molding platen, the pre-expanded rubber compound is immediately transferred to a circulating hot air oven at a higher temperature.

7.2 Open cell structure

The voids (gas cells) coalesce so that the combined solid and gaseous phases are continuous. Therefore, the gas is able to flow through the polymer matrix under the action of some driving force. Open cell foams are generally formed during processing under ambient, i.e. low-pressure, atmospheric conditions.

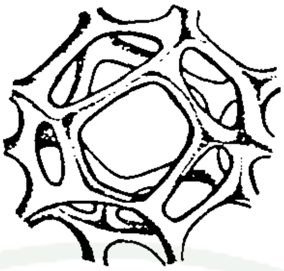


Figure 5 The open cell structure.

Source: Klempner and Frisch (1991)

7.3 Closed cell structure

The gas is dispersed in the form of discrete gas bubbles and the polymer matrix exists as the only continuous phase. Gas transport takes place by diffusion through the cell wall. Closed cell foams are generally formed by high-pressure techniques.

Figure 6 The closed cell structure.

Source: Klempner and Frisch (1991)

In practice, the two cellular morphological forms co-exist so that a polymer foam does not always have completely open or completely closed cells. The volume fraction of closed cells has a considerable influence on the mechanical behaviour of these systems, i.e. a high volume fraction of closed cells (low volume fraction of open cells) will lead to higher stiffness and strength. The main physical difference between the two types of cells, i.e. open and closed cells, is depicted in Figure 5-6.

The open cell structure, Figure 5, consists of a lattice of struts, whereas for the closed cell structure, Figure 6, in addition to the struts there are windows or membranes of base polymer between the strut faces. These membranes are usually significantly thinner than the dimensions of the struts themselves but provide addition support and therefore stiffness and strength to the cell.

Cellular polymers can also be classified according to their stiffness, the two extremes being rigid and flexible depending on the chemical composition, the rigidity of the polymer backbone, the degree of crystallinity and the degree of crosslinking (if any). Skochdopole and Rubens have defined each term as follows:

(1) **Rigid foams** The polymer matrix exists in the crystalline state, or if amorphous, below its glass transition temperature (T_g). Typical examples include most polyolefins, polystyrene (PS), phenolics (PFs), polycarbonate (PC), and some polyurethane (PU) foams.

(2) **Flexible foams** The polymer matrix is always above its T_g. Typical examples include rubber foams, flexible PU foam, and plasticised poly(vinyl chloride) (pPVC) foam.

Intermediate between these two extremes is a class of cellular polymer known as a semi-rigid. Although these materials have an elastic modulus higher than that of flexible foams, the stress-strain behaviour is nevertheless closer to that of flexible systems than that exhibited by rigid foams (Duanghathai and John, 2005).

8. Blowing agent

For the production of sponge-(open cell), closed cell sponge (small, closed and open cells) or micro cellular rubber (small closed cells with thin cell walls) inorganic and organic blowing agents are used. Two processes are mainly used: the blowing process for the production of sponge-rubber and the expansion process for the production of micro cellular rubber (closed cell).

Blowing agents are substances that are worked into the rubber and are stable at room temperature, but decompose at higher temperature with gas release before or during vulcanization. The gas that is formed, nitrogen or carbon dioxide, gives rise to the formation of pores.

Inorganic blowing agents are difficult to work into the rubber, their dispersion is primarily poor, causing irregular pore structure. The storage stability of such mixtures, especially those containing ammonium bicarbonate, is not very high. For that reason, nitrogen releasing organic blowing agents were developed, that have better properties and have, in spite of their higher price, largely replaced the inorganic blowing agents. A good blowing agent should fulfill the following requirements:

- contain a large amount of releasable gas
- be toxicologically “clean”
- decomposition products should not have a bad odor
- should not discolor the vulcanizate
- should disperse well in the rubber
- should not influence the vulcanization and not worsen aging properties
- have a suitable decomposition range (varies for different uses)
- be low-priced

Since it is difficult to combine all these properties in one product, special blowing agents have been developed for different areas of applications, that produce vulcanizates with optimum properties combined with good processing safety. For example, in some cases (hard micro cellular rubber soles) a high decomposition temperature is beneficial; in other cases (for example for sponge rubber production) a low decomposition temperature of the blowing agent is necessary (Hofmamm, 1989).

Blowing agents, sometimes refered to as foaming agents, are used to generate cells in polymeric materials. They are generally classified based on the mechanism by which gas is created (Duanghathai and John, 2005).

i) Physical blowing agents (PBAs) are gases or compounds that produce gases as a result of physical properties such as evaporation, desorption at elevated temperatures, or reduced pressures.

ii) Chemical blowing agents (CBAs) are individual compounds or mixtures of compounds that produce gas as a result of a chemical reaction. The chemical change is ususally brought about by thermal decomposition or as a result of chemical reaction with other components of the formulation.

Both PBAs and CBAs should possess the following qualities.

(a) Long-term storage stability (shelf life) under ordinary conditions.

(b) Gas release over a controlled time and temperature range.

(c) The gas liberated and the products must be noncorrosive, nonflamemable, and incombustible; compounds that liberate N_2 and CO_2 are preferable.

(d) Low toxicity, odour, and colour of both the blowing agent and its decomposition products.

(e) No deleterious effects on the stability and processing characteristics of the polymer.

(f) The liberated gas must have a low rate of diffusion through the polymer

(g) The ability to produce cells of uniform size.

(h) The ability to produce a stable foam. The stability of the foam depends on the solubility and diffusivity of the gas in the polymer matrix.

(i) Good cost-performance relation and availability.

(j) Environmentally acceptable.

8.1 Physical blowing agents

PBAs are low boiling point liquids which are dispersed in the material to be foamed. In addition to the desirable properties requires of as mentioned above, other requiredments should be taken into account when selecting a suitable physical blowing agent. The PBAs should:

(a) have no effect on the physical and chemical properties of the polymer or on other components of the formulation.

(b) be soluble in or mix readily with other foaming ingredients.

(c) be thermally stable and chemically inert in gaseous form.

(d) have low vapour pressure at room temperature.

(e) be highly volatile under the action of either external heat or reaction (exothermic) heat, and have low heat capacity and low latent heat of gas formation.

8.2 Chemical blowing agents

Two principal advantages of using CBAs are (i) they are easy to introduce into the composition to be foamed and (ii) they are easily processed with ordinary equipment. There are additional requirements to be taken into account when selecting a CBA.

(a) The decomposition temperature of CBA must be compatible with the processing temperature of the polymer, i.e. be close to the melting point or the hardening temperature of the polymer.

(b) Gas must be liberated within a certain narrow temperature range.

(c) The rate of gas liberation must be adequately high (but not violently so), and controllable by temperature, pressure, and the decompostion of activators (kickers).

(d) The CBA and gas(es) liberated must readily disperse or dissolve in the molten polymer compound.

(e) The decomposition of CBA must not be accompanied by the release of so much heat that the polymer matrix will be thermally destroyed.

(f) The CBA and the products of its decomposition must not affect the rate of polymerisation (hardening) or cause destruction of the matrix.

(g) The rate of gas liberation must not decrease appreciable during the process of thermal decompostion of the CBA as the internal pressure in the system increases.

8.2.1 Inorganic blowing agents

These types of BA liberate gaseous products as a result of reversible equilibrium thermal decomposition. The reversible character of the reaction may result in a decrease in the gas content in the system, which can lead to a pressure drop in the foam cells causing shrinkage of the material. Examples of these compounds are carbonates of alkali metals (such as sodium carbonate and bicarbonate) and ammonium salts of inorganic and organic acids (such as ammonium carbonate, $(NH_4)_2CO_3$). These CBAs are not suited for producing flexible cellular materials, but can be used successfully to produce microcellular rigid foams such as PVC and PS foams.

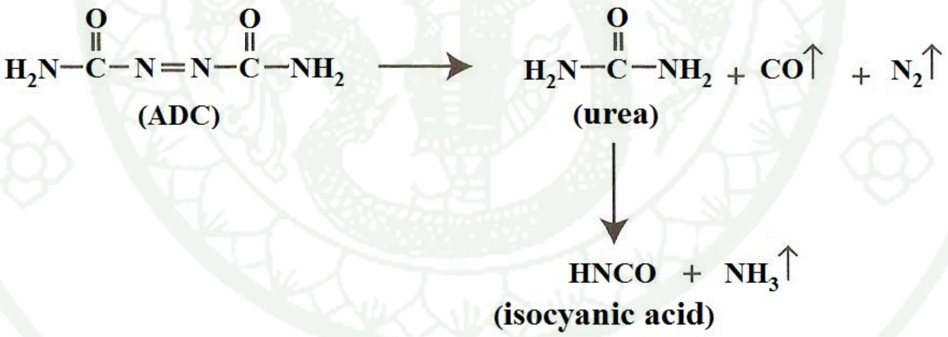
8.2.2 Organic blowing agents

In contrast to inorganic BAs these compounds liberate gaseous products as a result of irreversible thermal decomposition reactions. The advantages of these organic CBAs are (i) the reaction is irreversible (ii) they can be made to liberate the maximum amount of gas at temperatures close to the melt temperature range of the polymer composition and (iii) they can easily be mixed uniformly with the polymer and other compounding ingredients. Their main disadvantage is their high cost, and in some cases their toxicity.

Most of the known organic CBAs fall into one of the following classes: azo and diazo compounds. N-nitroso compounds, sulfonylhydrazides, azides, triazines, triazoles and tetraazoles, sulfonyl semicarbazides, urea derivatives, guanidine derivatives, and esters. The two most widely used organic BAs are azodicarbonamide and 4,4'-oxybis(benzenesulfonyl hydrazide); their main characteristics are as follows:

8.2.2.1 Azodicarbonamide (ADC)

This is one of the most effective high temperature CBAs and consequently the most widely used. It is a yellow powder that decomposes at 195-216 °C depending on its method of preparation. It evolves 220 $cm^3\ g^{-1}$ of gas and leaves a white residue that is odourless, nontoxicity, and nondiscolouring. The thermal decomposition of ADC is as follows:



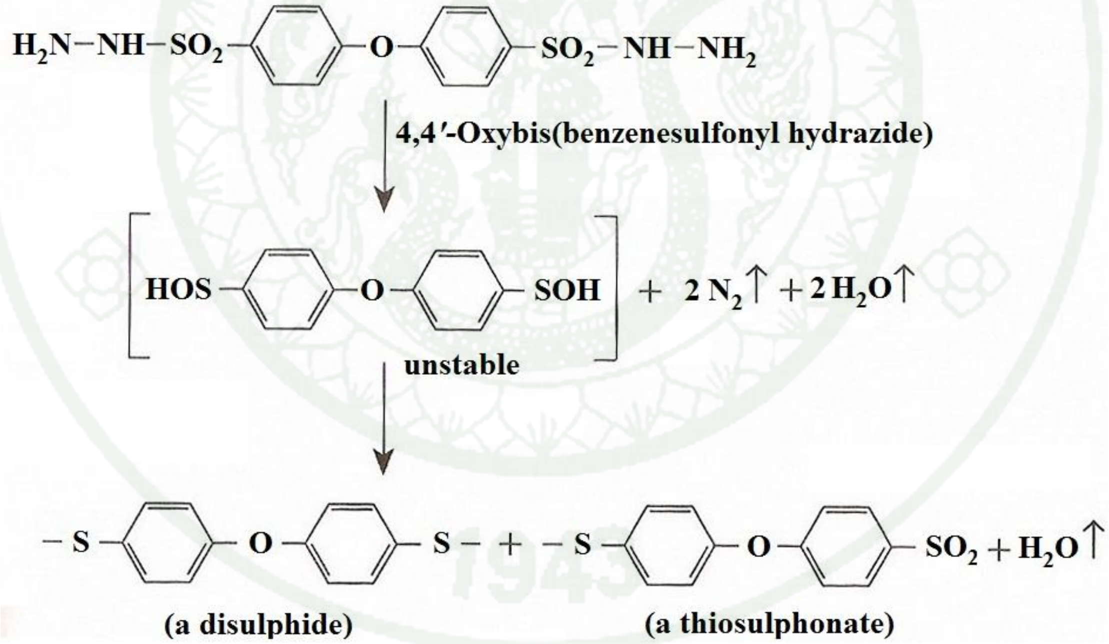
$$H_2N-\overset{O}{\overset{\|}{C}}-N=N-\overset{O}{\overset{\|}{C}}-NH_2 \longrightarrow H_2N-\overset{O}{\overset{\|}{C}}-NH_2 + CO\uparrow + N_2\uparrow$$

Note: Several other reactions take place during thermal decomposition of the ADC, but overall CO, CO_2, N_2, and NH_3 are the main gases liberated.

Unlike many other organic blowing agents, ADC does not support combustion and is self-extinguishing. The decomposition rate of ADC can be adjusted by using activators, or 'kickers', and a wide range of lower decomposition temperature can be atttained. This range of useful temperature and high gas efficiency are the main advantage of ADC. Suitable activators include transition metal salts (especially those of lead, cadmium, and zinc), polyols, urea, alcohol amines, and some organic acids. The decomposition temperature of ADC can be reduced to 130°C. The residual solids, which are not decomposed under the decompostion condition, consist of urazole, hydrazodicarbonamide, cyanuric acid, etc. ADC is generally used for the foaming of thermoplastic polymers such as PE, PS, PVC, and ABS (acrylonitrile-butadiene-styrene terpolymer). It is also used for foaming certain rubber compositions.

8.2.2.2 4,4'-oxybis(benzenesulfonyl hydrazide) (OBSH)

OBSH is a white, crystalline solid that decomposes at 160°C and evolves 125 cm^3g^{-1} of N_2 and a small amount of water vapour as expressed by the following schemes:



OBSH produces a polymeric, snakelike residue when decomposed in air. This residue remains colourless and odourless unless it is heated above 170-180°C. This nonpolar residue has made OBSH an ideal choice for the production of LDPE and PVC foamed insulation for wire on account of its non-interference with electrical properties. The application of an ultrahigh alternating electric field (UHF), which is also used to crosslink PE, can decompose OBSH.

The total (maximum) gas number is 313 cm^3/g (which includes water vapor). Thus, in paraffin the thermal decomposition accelerates, whereas in plasticizers of the phthalate or phosphate type it goes down (Klempner and Frisch, 1991).

9. Cellular structure

A typical foam structure contains cells of many shapes and sizes. Two dimensional cell representations are assemblies of triangles, squares and, more commonly, pentagons or hexagons, whereas there are a wide variety of cell shapes in three dimensions, most commonly pentagonal dodecahedrons (twelve pentagonal faces per cell) as illustrated in Figure 7.

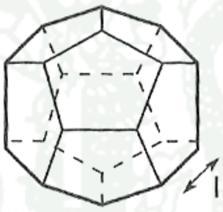


Figure 7 A pentagonal dodecahedron cell structure.

Source: Duanghathai and John (2005)

From a fundamental point of view, a liquid foam is most stable when the gas bubbles are perfectly spherical in shape because the interfacial area and the capillary pressure are at their minimum values. For a monodispersed spherical cellular structure, the closest packing pattern is obtained when each sphere contacts 12 neighbours; the gas then occupies 74% of the total volume of the foam. If the gas volume ratio is further increased, the spheres have to deform to accommodate the higher gas volume and cells become polyhedral in shape,

It is commonly found that cellular polymers are anisotropic, that is, the cells are elongated in the direction of foaming as shown in Figure 8.

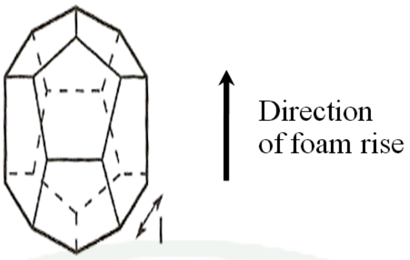


Figure 8 Representation of cell elongation during foam rise.

Source: Duanghathai and John (2005)

The mechanical stresses generated during foaming are not distributed uniformly throughout the volume of foam, hence the gas bubbles tend to expand along the directions of minimum local stress. In free-rise PU foaming, the extent of elongation of the cells decreases from the bottom to the top layers of the block, with the cells in the uppermost layers being substantially spherical. When foam expands in a closed space, the cellular anisotropy is always lower than for free-rise foaming, and cell elongation occurs only near the mould walls. The higher the pressure developed in the mould, the closer the cells approach a spherical shape. However, the outermost layers of the foam article usually contain very small cells, and under high pressure the surface layer of a block of foam is relatively smooth. Usually, where a foam contacts a mould surface the cells collapse to give a solid, smooth skin on the surface of the foam.

Anisotropy is commomly found in PU foams whereas thermoplastic foams such as polyethylene (PE), Polypropylene (PP), PS, and PVC tend to be more isotropic.

10. Mechanical properties of cellular polymers

The mechanical properties of the rigid foams differ markedly from those of flexible foams. The tests used to characterize both types of foam therefore differ, as do their application areas.

Compressive properties are perhaps the most important mechanical properties for cellular polymers. Compressive energy absorption characteristics and deformation characteristics of a foam depend mainly on density, type of base polymer, and the predominance of either open or closed cells.

In simple terms, open cell foam (invariably flexible) relies on cell walls bending and buckling, which is essentially a reversible process (Figure 9).

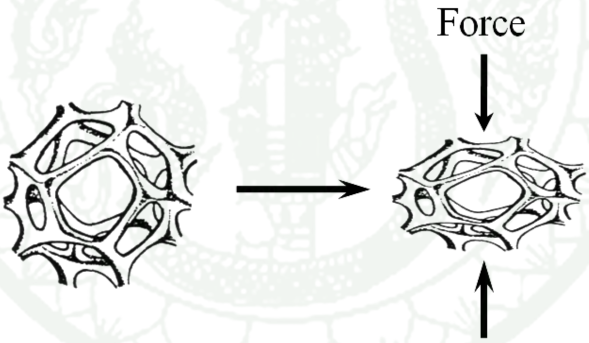


Figure 9 Schematic representation of open cell deformation.

Source: Duanghathai and John (2005)

In addition, as the cells become more compacted during compression, the escape of air through and out of the foam will become increasingly more difficult. The entrapped air will therefore offer some resistance to foam deformation during the final stages of compression.

On the other hand, air flow is not a consideration with closed cell foams. In this case (Figure 10) deformation involves cell wall bending/buckling (reversivle), gas compression, cell wall stretching/yielding (non-reversible). Severe compression causes cell rupture.

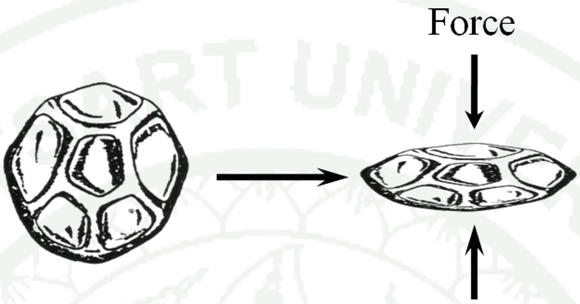


Figure 10 Schematic representation of closed cell deformation.

Source: Duanghathai and John (2005)

Closed cell rigid foams (e.g. PS and PU foams) exhibit from very limited to no yielding behaviour. Consequently, gas compression and matrix strength play important roles during the mechanical deformation of rigid foam. In addition, cell rupture often occurs during the energy absorption process.

The energy absorbing characteristics of foam can be represented in terms of compression stress-strain curves. Figures 11 and 12 show typical compression stress-strain curves of flexible and rigid cellular polymers, respectively.

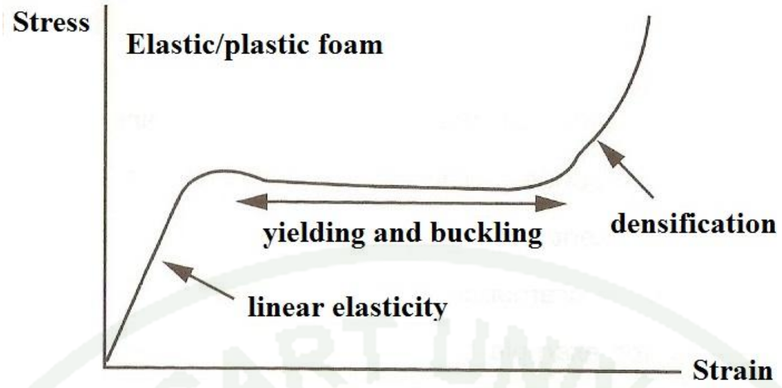


Figure 11 Typical compression stress-strain curve for flexible foams.

Source: Duanghathai and John (2005)

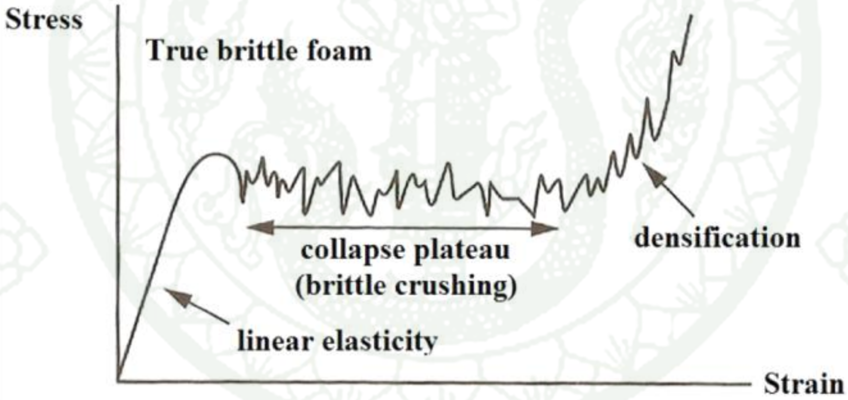


Figure 12 Typical compression stress-strain curve for rigid foams.

Source: Duanghathai and John (2005)

In the case of an elastic/plastic foam, e.g. a flexible closed cell foam, deformation at small strains shows classical elastic response corresponding to cell wall stretching. A long collapse plateau follows this as gas compression occurs accompanied by cell wall yielding and buckling. When the cells have almost completely collapsed, opposing cell walls start to touch each other and further strain results in densification of the base polymer as the material approaches a completely solid state. Open cell foams exhibit a more gradual increase in the stress-strain curve until the onset of the collapse plateau; moreover, densification occurs at lower stress values than for closed cell foams.

For rigid foams, elements of true brittle crushing are superimposed on the elastic/plastic response. The erratic nature of the collapse plateau corresponds to intermittent rupturing of individual cells. Due to cell rupture in rigid foams, resilience is dramatically affected. Foams can generally withstand only single impacts, for example the liners used inside riding or cycle helmets.

11. Applications of cellular polymers

Cellular polymers have been commercially accepted for a wide variety of applications since the 1940's. Approximately 90% of total foam usage derives from their excellent thermal insulation and energy absorbing characteristics. Some examples of typical applications of polymeric foams are (Duanghathai and John, 2005):

11.1 Thermal insulation

Low density polymeric foams have thermal conductivities in the order of 10^{-2} W/m K. Thermal insulation embraces all classes of materials for both heat/cold retention and protection against freezing. This is the single largest application for polymeric foams, with uses in a number of industries such as: building and construction, transportation, business machine housing, and food and drink containers. Rigid closed cell foams are particularly suitable for thermal insulation because in addition to their low thermal conductivity they are easy to dispense (on-site application) and have low moisture absorption. PS and PU foams are extensively used for these applications.

11.2 Cushioning

Flexible foams play an important role in this field. The important properties of cellular polymers in this category are suitable compression deflection behaviour, high resilience, low compression set, good tensile strength, and elongation. The broad range of compressive behaviour of flexible foam is one of their major advantages. It allows the requirements of any cushioning application to be melt by changing either the chemical or physical structure of the foam. Major cushioning applications include automobile padding, furniture, seating, flooring, carpet underlay, and athletic support. In many instances, cushioning applications also involve energy absorption and in this respect there are certain similarities between cushioning and packaging applications. The majority of cushioning materials is manufactured from flexible PU foams, e.g. for seat cushions, pillow and mattresses. Polyolefins or plasticised PVC foams are used for more severe conditions such as sports surface underlay and gymnasium mats.

11.3 Sound absorption and insulation

Open and closed cell foams are used for sound absorption and sound insulation respectively. Open cell flexible PU is used to absorb sound by lining walls and ceilings in music studios to prevent echo and feedback. Similarly, the judicious use of foam in concert halls is carried out in order to achieve the required sound balance. Surface area is increased by using profiled or convoluted surfaces to absorb sound at all angles of incidence. However, open cell foams are not as efficient in terms of sound insulation. Closed cell PS, PE or PU foams are used in building for the sound insulation of walls, floors, amd ceilings.

12. Sound Absorptive Materials

Materials that reduce the acoustic energy of a sound wave as the wave passes through it by the phenomenon of absorption are called sound absorptive materials (Lewis, 1994). They are commonly used to soften the acoustic environment of a closed volume by reducing the amplitude of the reflected waves. Absorptive materials are generally resistive in nature, either fibrous, porous or in rather special cases reactive resonators (Leo, 1988). Classic examples of resistive material are nonwovens, fibrous glass, mineral wools, felt and foams. Resonators include hollow core masonry blocks, sintered metal and so on. Most of these products provide some degree of absorption at nearly all frequencies and performance at low frequencies typically increases with increasing material thickness (Lewis, 1994).

Porous materials used for noise control are generally categorized as fibrous medium or porous foam. A particular interest of this research is to conduct a systematic study on fibrous sound absorbing materials. Fibrous media usually consists of glass, rock wool or polyester fibers and have high acoustic absorption. Sometimes fire resistant fibers are also used in making acoustical products (Claudio and Andrea, 1998).

Often sound barriers are confused with sound absorbing materials. Generally materials that provide good absorption are poor barriers. Unlike, barriers and damping materials, the mass of the material has no direct effect on the performance of the absorptive materials (Timothy *et al*., 1999). The performance of absorptive materials depends on many parameters, which are explained in the latter part of this chapter. Absorptive materials are almost always used in conjunction with barriers of some type since their porous construction permits some noise to pass through relatively unaffected (Price and Mulholland, 1968). An absorber, when backed by a barrier, reduces the energy in a sound wave by converting the mechanical motion of the air particles into low-grade heat. This action prevents a buildup of sound in enclosed spaces and reduces the strength of reflected noise (Lewis, 1994). The porous nature of absorptive materials renders them susceptible to contamination, moisture retention and deterioration due to physical abuse. To avoid these problems, facings may be attached to at least one side of the absorber. The addition of a facing to acoustical foam has the effect of increasing the lower frequency absorption at the expense of the higher frequencies (David, 1988).

12.1 Mechanism of Sound Absorption in Fibrous Materials

The absorption of sound results from the dissipation of acoustic energy to heat. Many authors have explained this dissipation mechanism in the past (Leo, 1971). Fridolin *et al*. (1992) described the mechanism of sound dissipation as: when sound enters porous materials, owing to sound pressure, air molecules oscillate in the interstices of the porous material with the frequency of the exciting sound wave. This oscillation results in frictional losses. A change in the flow direction of sound waves, together with expansion and contraction phenomenon of flow through irregular pores, results in a loss of momentum.

Owing to exciting of sound, air molecules in the pores undergo periodic compression and relaxation. This results in change of temperature. Because of long time, large surface to volume ratios and high heat conductivity of fibers, heat exchange takes place isothermally at low frequencies. At the same time in the high frequency region compression takes place adiabatically. In the frequency region between these isothermal and adiabatic compression, the heat exchange results in loss of sound energy. This loss is high in fibrous materials if the sound propagates parallel to the plane of fibers and may account up to 40% sound attenuation. So, altogether the reasons for the acoustic energy loss when sound passes through sound absorbing materials are due to:

- Frictional losses
- Momentum losses
- Temperature fluctuations

12.2 Types of Sound Absorption Materials

The main aim of using sound absorption material is to reduce Sound Pressure Level (SPL) within the spaces to an acceptable level. High SPL could create listening discomfort inside the spaces. Further, sound absorption material is able to prevent undesirable reflection from surfaces. Undesirable reflections are particularly detrimental to speech intelligibility. Undesirable reflections, which are delayed in the region of 30 ms to 50 ms as compare to direct sound results in echo. The availability of echo results in poor speech intelligibility (Maekawa and Lord, 1994). Finally, the use of sound absorption material in an enclosed space is able to control reverberation level within the spaces. Suitable level of reverberation would result in good speech intelligibility (Yerges, 1978). There are mainly four types of sound absorption materials available that can be used to achieve the above as discussed below namely porous absorber, Helmholtz resonator, membrane absorber and perforated panel absorber.

12.2.1 Porous Absorber

Porous absorber is the most common material used in an enclosed room acoustic treatment. These materials which could be from mineral or cellulose fiber are good sound absorber in the mid to high frequency range. Typical porous absorbers are carpets, acoustic tiles, acoustic (open cell) foams, curtains, cushions, cotton and mineral wool. The porosity created by the fiber interstices can trap and dissipates the sound energy. The frictional resistance change impinges sound energy into frictional heat and viscous losses within the pores due to small fiber vibrations. Energy conversion into heat is the most important mechanism of sound absorption for a porous material. The energy intensity would decrease over the distance travel through the material. The porous fiber sound absorption is affected by the density, the thickness, the airspace behind the material and the surface treatment (Everest, 1994).

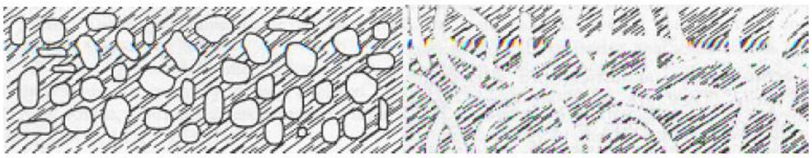


Figure 13 Illustration of porous absorber.

Source: Cox and Antonio, (2004)

Density changes are known to have little effect on porous material sound absorption performance. The thicker the porous absorbers, the higher the sound absorption at lower frequencies, particularly at below 500 Hz. The material thickness of $\lambda/4$ should result in higher sound absorption performance at the respected frequency (Maekawa and Lord, 1994). This explains why porous material has low sound absorption at lower frequency range and become higher at higher frequency range. Additional sound absorption for a porous material can be achieved whenever the porous material is position with an air-gap distance where the particle velocity of the sound waves is maximum, such as at $\lambda/4$. Acoustically transparent surface treatment such as acoustic fabric, tissue lamination or water based paint should be able to preserve the sound absorption of the porous fiber.

12.2.2 Helmholtz Resonator

Sound absorption at lower frequency range could be achieved particularly using Helmholtz resonator. The quality of this resonator is indicated by its quality factor (Q). This indicates the bandwidth of its tuning which is –3dB from its resonance frequency (Everest, 1994). The quality factor of the Helmholtz resonator is formulated as

$$Q = \frac{f_{rev}}{\Delta f} \qquad 1)$$

Where

Q = quality factor

f_{res} = resonance frequency

Δf = bandwidth

Helmholtz resonator consists of a volume of air contained in a cavity where there is a neck of small opening. When there is no porous fiber in the cavity, the Helmholtz resonator is considered undamped. The undamped resonance frequency of a Helmholtz resonator could be estimated using the following equation (Lawrence, 1970).

$$f_{rev} = \frac{1}{2\pi\sqrt{MC}} \tag{2}$$

Where, no porous fiber

M = the inertance

C = the acoustical capacitance

$$M = \frac{\rho(L + 1.7R)}{\pi R^2} \tag{3}$$

Where

ρ = Density of the air, kg/m^3

(L+1.7R) = Effective length of the neck, m

πR^2 = Area of the opening, m^2

R = Radius of the hole, m

$$C = \frac{V}{\rho c^2} \tag{4}$$

Where

ρ = Density of the air, kg/m^3

C = Acoustical capacitance

V = Volume of the chamber, m^3.

c = Speed of sound in air, m/sec.

Alternatively, the approximate resonance frequency (no porous fiber) could be calculated using the following equation (Lawrence, 1970).

$$f_{rev} = \frac{cA}{2\pi\sqrt{vV}} \tag{5}$$

Where

c = Speed of sound, m/sec

A = Area of the neck, m^2
v = A (L + 1.7R) Effective volume of the neck, m^3
V = Volume of the chamber, m^3

The sound absorption performance of the Helmholtz resonator at its resonance frequency can be estimated using the following equation

$$A = 0.159\left(\frac{c}{f_{rev}}\right)^2 \qquad (6)$$

Where

c = Speed of sound in air, m/sec
A = Sound absorption, m^2 Sabine

12.2.3 Membrane Absorber

A Membrane absorber or diaphragmatic absorber is used to absorb low frequencies sound energy. Membrane absorber works by vibrating at these low frequencies and turning the sound energy into heat conversion to heat takes place through the resistance of the membrane to rapid flexing and due to the resistance of the enclosed air to compression. Practically, a membrane absorber constructed by flexible sheets made from plywood or rubber is stretched over supports or rigid panels mounted at some distance from the front of a solid wall as shown in Figure 14 below.

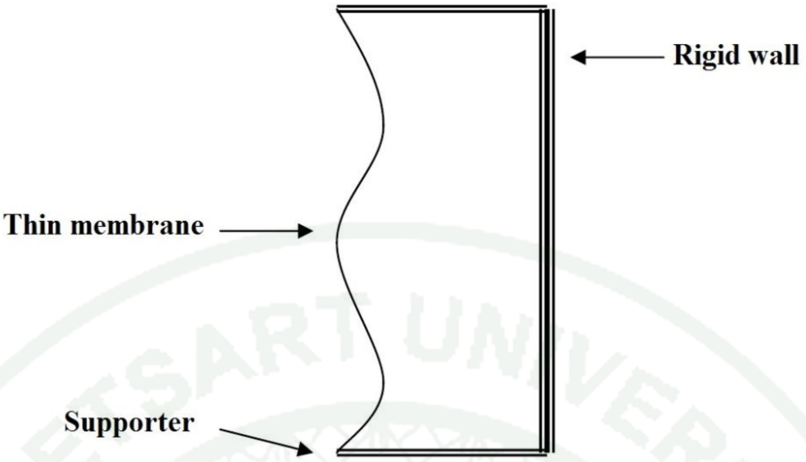


Figure 14 Membrane absorber.

Source: Marsh, (1999)

In practice, the method of fixing and the stiffness of the panels will also have some effect as the panel itself will tend to vibrate.

12.2.4 Perforated Panel Absorber

Perforated panel is typically made from rigid thin sheet of metal perforated with circular aperture. The perforated panel is normally mounted over some distance from the wall. There is an air cavity behind the wall and this air cavity could be filled with porous fiber to result in broader frequency range of sound absorption.

The resonance frequency of perforated panel can be estimated using the following equation (Lawrence, 1970).

$$f_{rev} = \frac{c}{2\pi}\left(\frac{P}{d(L + 1.7R)}\right) \tag{7}$$

Where

c = Speed of sound in air, m/sec
P = Perforation ratio (hole area/plate area)
d = Distance of the perforated panel from the wall, m
L = Perforated panel thickness, m
(L+1.7R) = Effective length of the neck, m
R = Radius of the hole, m.

To broaden the frequency range of sound absorption performance of the perforated panel, the air cavity behind the wall should be filled with porous fiber. An air-gap between the porous fiber and the wall should be maintained. Increasing the thickness of the perforated panel and also the depth of the air gap would lower its resonance frequency. Therefore, by varying the depth of the air space and the thickness of the perforated panel, broader frequency range of sound absorption performance could be achieved.

A perforation ratio of more than 20% with small aperture would not affect the sound absorption of porous fiber. However, a smaller perforation ratio would reduce higher frequency sound absorption performance of porous fiber (Maekawa and Lord, 1994).

12.3 Performance of Sound Absorbing Materials

For porous and fibrous materials, acoustic performance is defined by a set of experimentally determined constants namely: absorption co-efficient, reflection co-efficient, acoustic impedance, propagation constant, normal reduction coefficient and transmission loss (www.bksv.com, 2011). There are different methods available to determine these acoustical parameters but all of these methods mainly involve exposing materials to known sound fields and measuring the effect of their presence on the sound field.

The performance of sound absorbing materials in particularly is evaluated by the sound absorption co-efficient (α) (Horoshenkov and Swift, 2001 and Lewis, 1994). Alpha (α) is defined as the measure of the acoustical energy absorbed by the material upon incidence and is usually expressed as a decimal varying between 0 and 1.0. If 55 percent of the incident sound energy is absorbed, the absorption coefficient of that material is said to be 0.55. A material that absorbs all incident sound waves will have a sound absorption coefficient of 1. The sound absorption coefficient (α) depends on the angle at which the sound wave impinges upon the material and the sound frequency. Values are usually provided in the literature at the standard frequencies of 125, 250, 500, 1000 and 2000 Hertz (Warnock, 1980; Takahashi *et al*., 2005). Other important acoustic parameters that need to be considered while studying the acoustical absorptive properties are:

– Sound Reflection Coefficient: Ratio of the amount of total reflected sound intensity to the total incident sound intensity.

– Acoustic Impedance: Ratio of sound pressure acting on the surface of the specimen to the associated particle velocity normal to the surface.

In comparing sound absorbing materials for noise control purposes, the noise reduction co-efficient (NRC) is commonly used. NRC is the average usually stated to the nearest multiple of 0.05, of the co-efficient at four frequencies 250, 500, 1000 and 2000 HZ (Harris, 1979). It is intended for use as a single number index of the sound absorbing efficiency of a material. This NRC values provides a decent and simple quantification of how well the particular surface will absorb the human voice (http://www.absoluteastronomy.com/, 2011).

The sound absorption for a sample of material or an object is measured sometimes in sabins or metric sabins. One sabin may be thought of as the absorption of unit area (1 m^2 or 1 ft^2) of a surface that has an absorption coefficient of 1.0 (100 per cent). When areas are measured in square meters, the term metric sabin is used. The absorption for a surface can be found by multiplying its area by its absorption coefficient. Thus for a material with absorption coefficient of 0.5, 10 sq. ft has a sound absorption of 5 sabins and 100 m^2 is 50 metric sabins (Warnock, 1980). Harris (1979) gives four factors that affect the sound absorption co-efficient. They are:

- Nature of the material itself
- Frequency of the sound
- The angle at which the sound wave strikes the surface of the material
- Air gap

More fundamentally, all sound absorptive materials can be characterized by two basic parameters namely: characteristic impedance and complex propagation constant (Faulkner, 1976). Characteristic impedance is the measure of wave resistance of air. It is the ratio of sound pressure to particle velocity. Attenuation and phase constant which are included in the propagation constant are the measure of how much sound energy is reduced and the speed of propagation of sound respectively. Even other parameters were tried by researchers (Voronina, 1996; Kirby and Cummings, 1999) in order to include various effects like material internal structure, viscous and thermal loss, which are not discussed here.

12.4 Acoustic property

12.4.1 Definition

Acoustic property is termed as sound absorption, which is an important property of automotive interior components because it measures how effectively sound is dissipated once it enters the interior, which affects the overall sound level. When a sound wave strikes a surface, a fraction of the acoustic energy is absorbed and the remainder is reflected (Figure 15). The ratio of absorbed energy to incident energy average over all possible angles of incidence is the absorption coefficient of surface. The absorption coefficient (α), for material, is defined as the ratio of energy entering a material (absorbed or transmitted energy), to the incident energy in relation below (Maekawa and Lord, 1994):

$$\alpha = \left(\frac{E_i - E_r}{E_i}\right) = \left(\frac{E_\alpha + E_t}{E_i}\right) \quad (8)$$

Where

E_i = incident acoustic energy
E_r = reflected acoustic energy
E_t = transmitted acoustic energy
E_α = absorbed acoustic energy

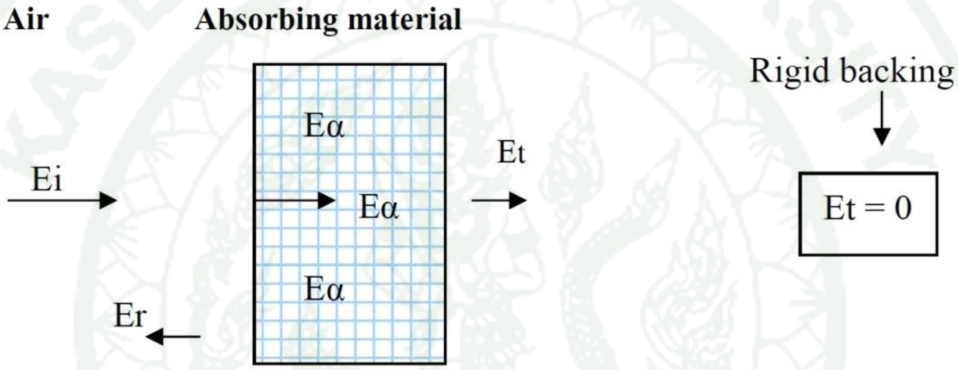


Figure 15 The breakdown of incident sound energy striking on acoustic layer.

Source: Maekawa and Lord, (1994)

12.4.2 Effect of parameter on acoustic property

Normally, the sound absorption ability of polymer foams depends on two major factors. The first is the macroscopic physical state of the foam, such as cell morphology, porosity, thickness and density (Figure 16), and the second is the molecular structure of the polymer, such as flexibility and crosslinking density. In principle, the noise contacts the open-celled foam structure in the form of a sound pressure wave within the porous structure. It is partially converted to heat energy due to the effect of air friction, and part of the sound energy is dissipated in the form of heat energy due to the molecular chain damping effect of the polymer. Examples of mainly polyols based polyurethane foams that were claimed such as polyether, polyester, polycaprolactone, and polybutadienes with phenolic resin were used for acoustic absorber hybrid foams (Chen *et al*., 1990).

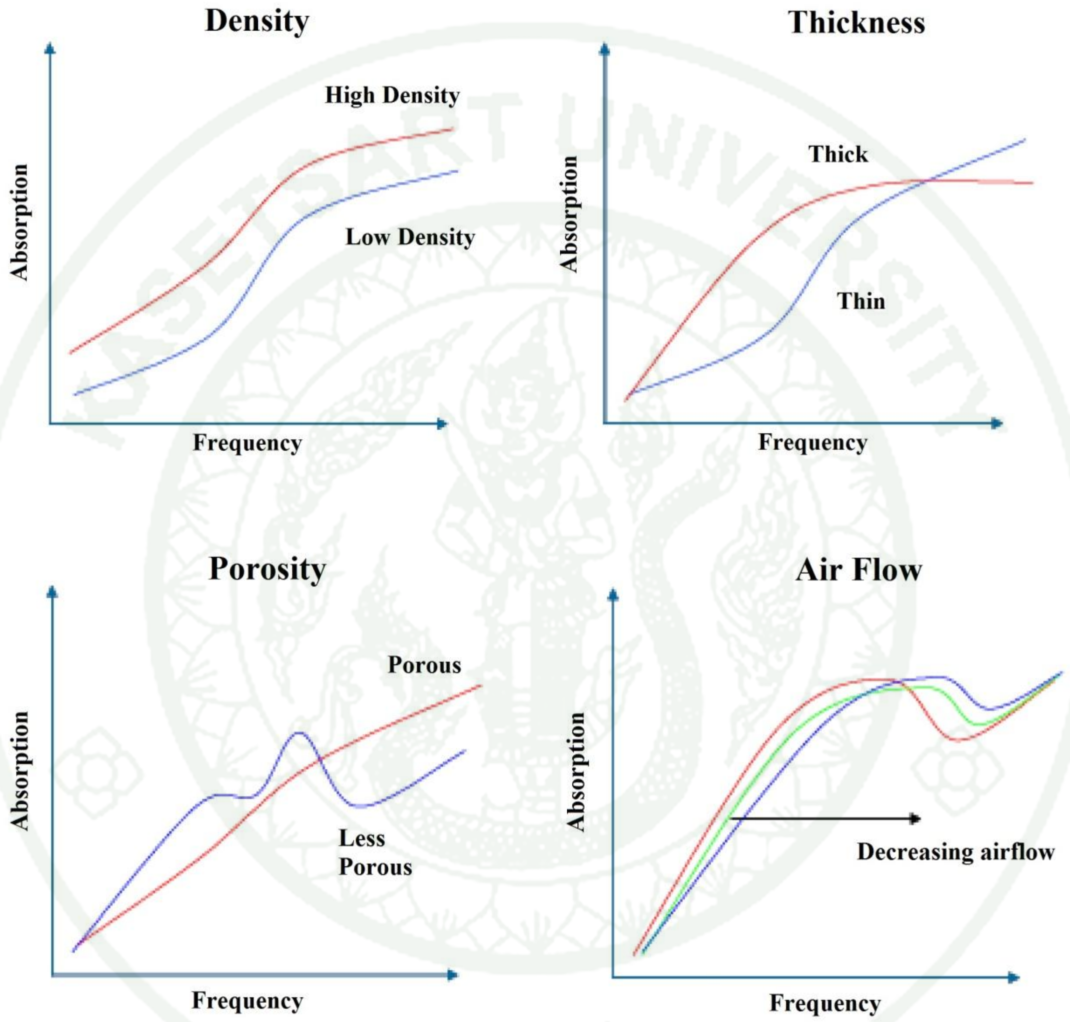


Figure 16 The key features driving absorption performance.

Sourec: Skinner *et al*., (2006)

The physical effects highlighted in the graphs (increasing density, thickness, porosity and airflow) will result in increased level of absorption performance and then potential to improve the acoustic comfort of these materials.

12.5 Measurement of Sound Absorption Coefficient

A number of measurement techniques can be used to quantify the sound absorbing behavior of porous materials. In general one is interested in one of the following properties: sound absorption coefficient (α), reflection coefficient (R), or surface impedance (Z). Detailed description of the measurement technique used in this research is given below.

12.5.1 Acoustic Measurements

Measurement techniques used to characterize the sound absorptive properties of a material are (Takahashi *et al.*, 2005):

- Reverberant Field Methods
- Impedance Tube Methods
- Steady State Methods

Reverberant Field Method for measuring sound absorption is concerned with the performance of a material exposed to a randomly incident sound wave, which technically occurs when the material is in diffusive field (Faulkner, 1976). However creation of a diffusive sound field requires a large and costly reverberation room. A completely diffuse sound field can be achieved only rarely. Moreover, an accurate value of complex impedance cannot be derived from the absorption coefficient alone (Takahashi *et al*., 2005). Since sound is allowed to strike the material from all directions, the absorption coefficient determined is called random incidence sound absorption coefficient, RAC. This method is clearly explained in ASTM C 423-72.

Impedance Tube Method uses plane sound waves that strike the material straight and so the sound absorption coefficient is called normal incidence sound absorption coefficient, NAC (www.tcnind.com, 2011). This research uses impedance tube method which is faster and generally reproducible and, in particular, requires relatively small circular samples, either 35 or 100 mm in diameter (according to the type of impedance tube). In the impedance tube method, sound waves are confined within the tube and thus the size of the sample required for test needs only be large enough to fill the cross-section of the tube (Kin *et al*., 2005). Thus this method avoids the need to fabricate large test sample with lateral dimensions several times the acoustical wavelength. The impedance tube method employs two techniques to determine NAC, namely:

1. Movable microphone which is one-third-octave frequencies technique (ASTM C 384) is based on the standing wave ratio principle and uses an audio frequency spectrometer to measure the absorption coefficients at various centre frequencies of the one-third-octave bands.

2. Two-fixed microphone impedance tube or transfer function method (ASTM E 1050), which is relatively recent development. In this technique, a broadband random signal is used as a sound source. The normal incidence absorption coefficients and the impedance ratios of the test materials can be measured much faster and easier compared with the first technique (Mueller *et al*., 2002).

The final method of measuring the sound absorption coefficient is known as *Steady State Method*. This method is mostly used when the other will not work. This particular method is described in ASTM E336-71. To measure the transmission coefficient of the materials, a third microphone or even a second pair of microphone can be placed behind the test sample in a second impedance tube.

All the materials in this work were tested by using the two microphone impedance tube method (ASTM E 1050), which is described below in section 12.5.2

12.5.2 Two Microphone Impedance Tube Technique (Transfer Function Method)

The transfer function method (ASTM E 1050) covers the use of an impedance tube, with two microphone locations and a digital frequency analysis system for the determination of normal incidence sound absorption coefficients (NAC) and normal specific acoustic impedance ratios of materials. This test method is similar to test method (ASTM C 384) in that it also uses an impedance tube with a sound source connected to one end and the test sample mounted at the other end. The measurement techniques for the two methods are fundamentally different, however. First microphone tube method (standing wave method) is quite cumbersome since a probing of the sound field has to be carried for each frequency.

Theoretical Background:

Rather than probing the sound field to determine sound maxima and minima pressure level as in standing wave tube method, in the two microphone method the ratio between the sound pressure amplitudes at two-fixed microphone positions is measured. Quantities are determined as a function of frequency with a resolution determined by the sampling rate of a digital frequency analysis system (Uno, 1994). The usable frequency range depends on the diameter of the tube and the spacing between the microphone positions. An extended frequency range may be obtained by using tubes with various diameters and microphones spacing. By this method acoustical parameters like absorption coefficient, reflection coefficient and surface admittance for a small samples exposed to plane waves can be determined (Yunseon, 2002). The reflection co-efficient (*R*) of the sample can be obtained from the equation (9).

$$\mathrm{R} = \left(\frac{\mathrm{H}_{\mathrm{l}} - \mathrm{H}_{\mathrm{i}}}{\mathrm{H}_{\mathrm{r}} - \mathrm{H}_{\mathrm{l}}}\right) e^{j2k(l+s)} \tag{9}$$

Where

H_l = Frequency Response Function (FRF) of the impedance tube
H_i = FRF associated with the incident wave components
H_r = FRF associated with the reflected wave components
k = Wave number
l = Distance between the microphone and the sample
s = Spacing between the microphone (Yunseon, 2002)

By using equation (10), normal sound absorption coefficient, NAC (α) can be determined.

$$\alpha = |1 - R|^2 \tag{10}$$

Where

R = The reflection co-efficient

Also normalized surface impedance (*Z*) can be calculated using the equation (11):

$$\frac{Z}{\rho c} = \frac{1 + R}{1 - R} \tag{11}$$

Where

ρ = Air density, kg/m^3
c = Sound velocity in air, ms^{-1}

Outline of the theory behind the calculation of sound absorption coefficient by using transfer function method is given by Frank (2001) and many others.

The results of tests by both methods (reverberation and impedance tube) on sound absorption coefficient of a number of materials have been reported and show that a consistent comparison is not always obtainable (Faulkner, 1976). The relation will vary depending on the physical properties, thickness and mounting conditions. However, it may be stated to a rough approximation that random incidence values are twice the normal incidence values in low range, higher by 0.25 to 0.35 in the middle range and approximately equal in the high range (Harris, 1979). Moreover, paper by Yoshio and Tsuneo, (1982) indicated that, though the results of NAC and RAC are different, the absorption peak positions were the same.

12.6 Applications of Absorbers

Acoustical material plays a number of roles that are important in acoustic engineering such as the control of room acoustics, industrial noise control, studio acoustics and automotive acoustics. Sound absorptive materials are generally used to counteract the undesirable effects of sound reflection by hard, rigid and interior surfaces and thus help to reduce the reverberant noise levels (Bruce, 1981; Leo, 1960). They are used as interior lining for apartments, automotives, aircrafts, ducts, enclosures for noise equipments and insulations for appliances (Knapen *et al.*, 2003 and Youn and Chang, 2004). Sound absorptive materials may also be used to control the response of artistic performance spaces to steady and transient sound sources, thereby affecting the character of the aural environment, the intelligibility of unreinforced speech and the quality of unreinforced musical sound (Frank, 2001). Combining absorptive materials with barriers produces composite products that can be used to lag pipe or provide absorptive curtain assemblies. Therefore, sound absorbing materials are chosen in terms of material types and dimension, and also based on the frequency of sound to be controlled (Francisco and Jaime, 2004).

12.6.1 Reverberation Control

One of the main applications of sound absorber material is to reduce reflected sound energy in the room and to reduce the reverberance and sound level. The amount of reverberation in a space depends on the size of the room and the amount of sound absorption. This kind of absorber is commonly used in restaurants or railway stations to improve the speech intelligibility by reducing the noise.

12.6.2 Noise Control in Factories and Large Rooms

The other application of sound absorber is to control the noise level in working environment or factory to protect the hearing lost of the workers. The treatment method to solve this problem is to reduce the reverberance sound level within the space. By adding sound absorption materials on the wall, the noise exposure is decreased to save level for human ear.

12.6.3 Modal Control in Critical Listening Space

Bass trap or bins absorption is commonly applied for controlling low frequency problem in recording studio, home theatre and conference rooms. Resonant and Helmholtz absorber are usually used for mass spring system with damping to provide absorption at the resonant frequency of the system.

12.6.4 Echo Control in Auditorium and Lecture Theatres

Sound absorption material are commonly used to absorb the late arriving reflected sound in large auditorium, a late arriving reflection appears as an echo if its level is significantly above the general reverberation level. By adding sound absorption material at rear wall, it can optimally reduce the echo in audience areas near to the stage.

12.6.5 Absorption in Sound Insulation

The other applications of porous absorbers materials are in sound insulation building system, to protect the noise entering the room. The porous absorber is commonly used to prevent a resonance of air cavity of light weight construction based on leaf partition with an air gap.

MATERIALS AND METHODS

Materials

1. Natural Rubber (NR); STR5L, S.M.P. RUBBER, Thailand.

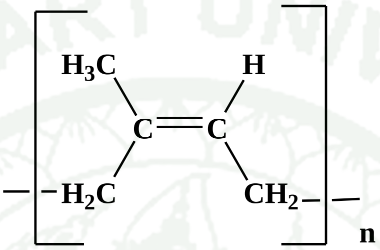


Figure 17 The structure of natural rubber.

2. Fillers

- Water hyacinth was supplied by Living museum of Thai farmers, Ban Lan Laem, Nakornchaisri-Don Toom Road. Nakhon Pathom, Thailand.

3. Reagents

- ZnO, an activator, Global Chemistry, Thailand.
- Stearic acid, an activator, Imperial Industrial Chemical, Thailand.
- Sulfur, a crosslink agent, Sahapaisal Industry, Thailand.
- 4,4’- Oxybis (benzene sulfonyl) hydrazide (OBSH), blowing agent was supplied by A.F. Goodrich Chemical Co., Ltd., Bangkok, Thailand.

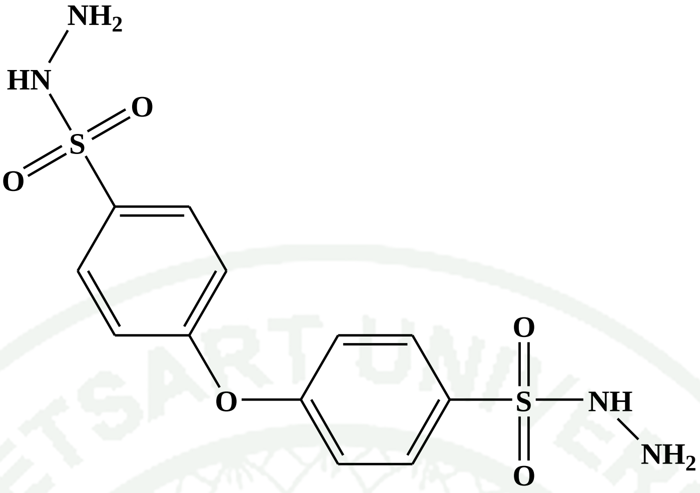


Figure 18 Structure of 4,4'- Oxybis (benzene sulfonyl) hydrazide (OBSH).

- 2,2,4-Trimethyl-1,2-dihydroquinoline polymer (TMQ), an antioxidant, Eliokem, USA.

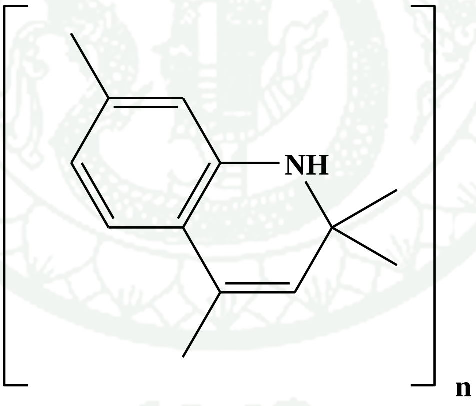


Figure 19 Structure of 2,2,4-Trimethyl-1,2-dihydroquinoline polymer (TMQ).

- N-cyclohexyl-2-benzothiazole sulphenamide (CBS), an accelerator, Flexsys, Germany.

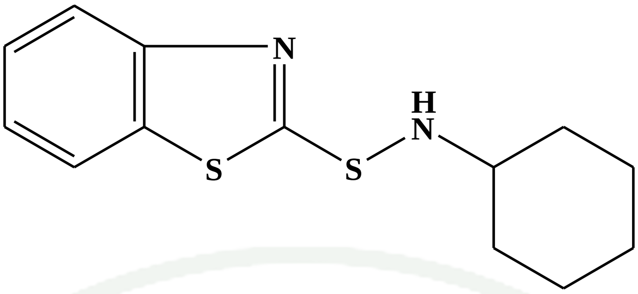


Figure 20 Structure of N-cyclohexyl-2-benzothiazole sulphenamide (CBS).

- Toluene, Analytical reagent grade, Fisher

4. Equipments

- Two-roll-mill, model YFCR 6, Chor.Sri-Anan Co., Ltd.
- Moving Die Rheometer (MDR), A022S rheoTECH MD+, USA *Department* of *Materials Science*, *Faculty* of *Science*, *Chulalongkorn* University
- Compression testing, Materials Testing Machine by Testometric Company Ltd., United Kingdom
- Compression molding, G30H-15-CX, Wabash, USA
- Acoustic testing, The testing apparatus was part of a complete acoustic material testing system, featuring Brüel&Kjaer PULSE^{TM} interface, Denmark
- Sieving Machine, Retsch Muhle rotary mill, type SR2, Germany
- Thermogravimetric analysis (TGA), Perkin-Elmer7, USA
- Differential scanning calorimetry (DSC), Perkin–Elmer DSC7, USA
- FT-IR spectroscopy, Perkin-Elmer, FT-IR 2000 spectrometer, USA
- Oven, Binder
- Electronic balance, Precisa 1600 C
- Electronic Densimeter, MD-200S, Mirage

Methods

1. Fiber preparation

Water-hyacinth fiber was taken from stems which are approximately 1 m in length and have a diameter of approximately 2.5-3.5 cm. The stems were cut into small pieces 2 cm long, and then they were dried in direct sunlight. Dried stems were ground with a Retsch Muhle rotary mill, type SR2. Finally, the water-hyacinth was sieved through a 100-mm mesh size to obtain finely or ground fiber. The fine fiber was mixed with the rubber compound in the amount of 5 and 10 phr. The rubber compound without fiber was also prepared.

2. Sponge Rubber Preparation and Formulation

The compounding process was commenced by masticating the NR on a laboratory two-roll mill for 5 min, followed by adding the required vulcanizing agents and blowing agents on the two-roll mill for a further 35 min, according to the recipe shown in Table 2. The ingredients were added in the same order as presented in Table 2. The compounded blend was then passed endwise ten times through the mill at the 1 mm opening, after which time it was sheeted off at 2 mm thick. The specifications of the mill are as follows: roll diameter, 160 mm; roll width, 380 mm; speed ratio, 1:1.22, a circulating water temperature of 50°C. The compounded rubber was left for at least 24 h before foaming. To investigate the effects of the foaming temperature and filler content (water hyacinth content) on the physical properties and morphology of NR foam, the foaming was performed at three different foaming temperature (150, 160, and 170°C). The compounded NRs were then compression molded along the mill grain direction by using an electrically heated hydraulic press at different temperatures under a pressure of 2500 psi, after removal of the pressure, expansion occurred immedialtely; the foamed samples were shelf-aged for at least 48 h before testing. Efficient blowing agent (OBSH) was carefully selected by TGA analysis.

Table 2 Formulation of the rubber compounds.

Ingredients	Function	Content (phr)
NR	Matrix	100
ZnO	Activator	5
TMQ	Antioxidant	1
Water Hyacinth	Filler	Varied; 0, 5, and 10
OBSH	Blowing agent	Varied; 0, 2, 4, and 6
Stearic acid	Activator	3
CBS	Accelerator	1
Sulfur	Cross-linker	1.5

Note: phr refers to parts per hundred rubber.

3. Characterization

3.1 Physical and chemical properties of fillers

Physical properties and surface area of fillers (fine water hyacinth fiber) were determined by the Brunauer-Emmett-Teller (BET) technique using nitrogen, around 2.5 m^2/g; Model: Autosorb 1, surface analyzer at 77.40 K. Particle size analyzer (wet sieve) was used to measure the particle size of the fillers.

The FT-IR spectra were collected on a Perkin-Elmer, FT-IR 2000 spectrometer from 4000 to 450 cm^{-1} with a resolution of 2 cm^{-1} with a five times scan per sample. All spectra were measured with air as background and were background subtracted to make them directly comparable. 5 mg of water hyacinth fibers was mixed with 200 mg of analytical grade KBr and the resulting mixture ground and pressed in order to obtain pellets (disc diameter 10 mm).

Detailed chemical composition of the water hyacinth fibers was determined as explained below using oven dried fibers: Specific gravity following

ASTM 845-83, Dry matter according to the standard methods (no. 35 LMBG). Moisture content of the fibers was determined according to ABNT NBR 9656, which consists of determining the percentage difference between the initial weight of the sample (1.0 g) and after 4 h drying at 105°C. Ash content was measured by considering the percent difference between the initial weight of dried fiber of the sample and that after calcination for 4 h at 800°C. Lignin content was evaluated following the TAPPI T13M- 54 method, which is based on isolation of lignin after polysaccharide hydrolysis by concentrated sulfuric acid (72%). The samples (1.0 g, previously dried) were macerated using 72% H_2SO_4 (15 mL) at room temperature and kept under these conditions for 24 h. After that, the material impregnated with sulfuric acid was transferred to an appropriate vessel, and 560 mL of distilled water was added. The system was then kept under reflux for 4 h. The remaining solid (insoluble lignin and ash) was filtered (sintered glass funnel, number 4, previously weighed), dried (105°C, 24 h), and then weighed. The acid-insoluble lignin content was calculated by considering the weight difference between the initial sample (fibers) and that of the filtered solid and subtracting the ash content from the weight of this last sample.

The holocellulose content was determined according to TAPPI T19m-54, which consists of a selective degradation of the lignin polymer. Sodium hypochlorite (2.5 g) and glacial acetic acid (1 mL) were added to an aqueous suspension (120 mL put in an Erlenmeyer flask) of previously dried and milled LC fibers (3 g). The mixture was covered with an inverted Erlenmeyer flask and kept at 70°C, under magnetic stirring for 1 h. This sequence was repeated twice. Then, after 3 h (3×1 h), the system was cooled to nearly 5°C, and the holocellulose was filtered and washed exhaustively with water and methanol and then dried at 40°C, in a vacuum oven, until constant weight was observed. For the determination of α-cellulose content, sodium hydroxide solution (10 mL, 17.5%) was added to cellulose (1.0 g) at room temperature. Then, the mixture was ground for 8 min, and sodium hydroxide (10 mL, 17.5%) was added to the mixture, which was kept at least for 20 min. If the alkaline solution is kept at room temperature longer than 20 min, as usually described for wood, cellulose can also degrade, leading to erroneous results for hemicellulose content. Then, water (40 mL) was added, and the solid residue was filtered and washed exhaustively with aqueous acetic acid and water. This remaining solid, considered α-cellulose, was dried at 40°C, in a vacuum oven, until constant weight was obtained. An average of three repetitions was taken for all mentioned analyses. The hemicellulose content was obtained by subtracting the α-cellulose part from the holocellulose content.

3.2 Relative density measurement

The density of the foam was measured by a buoyancy method using a densimeter (SD-200 L,MIRAQE). A 2×2 cm sample was cut from foam and measured; the expansion ratio was calculated from the following equation (Uejyukkoku and Nakatsu, 1996):

$$\text{Expansion ratio} = [(2\times 2\times t_1)/W_2] \qquad (12)$$

Where

t_1 = thickness, cm

W_2 = weight of the sample, g

The relative density of the foam was measured in accordance with ASTM D3575-93 by using equation (13) given below:

$$\text{Relative density} = \frac{\text{Foam density (g/cm}^3)}{\text{Solid density (g/cm}^3)} \qquad (13)$$

3.3 Crosslink density determination

The determination of the crosslink density was performed at room temperature in accordance with ASTM D471. For this measurement, different shapes of vulcanized test pieces were cut and weighed using an analytical balance and were considered to be the original weight. Then, the samples were immersed for 6 h in a glass vessel that contained toluene. The samples were then removed from the solvent, wiped thoroughly to remove excessive solvent and reweighed; this value was used as the swollen weight. The crosslink density of the sample was determined by using the following Flory–Rehner equation:

$$-\{\ln(1 - V_r) + V_r + \chi V_r^{\,2}\} = \rho V_0 M_c^{-1} V_r^{\,1/3} \qquad (14)$$

Where

χ = the interaction constant characteristic between rubber and toluene, which is 0.42,

ρ = the density of rubber, g cm^{-3}

V_o = the molar volume of toluene, cm^3 mol^{-1}

V_r = the volume fraction of the rubber in the swollen sample

M_c = the physical crosslink concentration, mol cm^{-3}

To solve equation (14), the above parameters should be calculated as follows. The volume fraction of rubber in the swollen sample, V_r, is given by the following equation:

$$V_r = \frac{(X_r/\rho_r)}{(X_r/\rho_r) + (X_s/\rho_s)} \qquad (15)$$

Where

ρ_s = the density of toluene, g cm^{-3}

ρ_r = the density of rubber, g cm^{-3}

X_s = the mass fraction of toluene, g which can be obtained as follow;

$$X_s = \frac{(\text{Weight of swollen sample (g)} - \text{Original weight (g)})}{\text{Weight of swollen sample (g)}} \qquad (16)$$

and X_r = the weight of rubber, g which is given as;

$$X_r = 1 - X_s \qquad (17)$$

Therefore, the value of M_c can be used to calculate the physical crosslink density, $[X]_{phys}$, using the following equation:

$$[X]_{phys} = \frac{1}{2M_c} \tag{18}$$

3.4 Foam cell morphology

Morphology study was carried out using scanning electron microscope (SEM) instrument. The surface morphology of water hyacinth fiber was examined using a SEM. The foam structures and distributions of samples prepared by razor cut surfaces were viewed using a JEOL (JSM-5800LV) SEM at 15 kV accelerating voltage. The SEM micrographs were then analyzed using ImagePro Plus software to determine the cell size. The results of average cell size were determined from measurement at 25 different locations of the captured image.

3.5 Thermal analysis of water hyacinth fiber and blowing agent

Thermogravimetric analysis (TGA) was used to measure the thermal stability, moisture and volatile content in water hyacinth fiber and blowing agent. Thermal decomposition patterns of water hyacinth and blowing agent (OBSH) were obtained from a thermogram and derivative thermogravimetric (DTG) using thermogravimetric analyzer (model PERKIN-ELMER7). Each sample was heated at a heating rate of 5°C/min from 50-800°C under a nitrogen atmosphere and switched over to an oxygen atmosphere from 600-800°C. The decomposition temperatures of the water hyacinth and blowing agent were characterised as the peak position of the decomposition.

Differential scanning calorimetry (DSC) was carried out on a Perkin–Elmer DSC7 equipment, fitted with a cooler system using liquid nitrogen to study the decomposition temperatures of the OBSH. Small samples (about 2-3 mg), were encapsulated in aluminum pans and analyzed in a temperature range from 30°C to

250°C at the heating rate 10°C/min in nitrogen atmosphere. The melting temperature (T_m) was taken as the peak temperature of the melting endotherm.

3.6 Cure characteristic

Cure characteristic was evaluated using a Moving Die Rheometer (MDR) according to ASTM D224. Each sample containing 4 g respective compound was tested at different temperatures (150, 160, and 170°C), curing time (t_{c90}) was determined from the plotted rheograph of MDR. As the heat transfer foaming process was implemented throughout the study, the cure characteristic evaluation need to be as close as possible to the actual process. Because of that fact, each sample needs to be heated for 10 min in an air-circulated oven which was set at 100°C before charging into the rheometer.

4. Mechanical Properties

4.1 Compression deflection

The mechanical test method used was ASTM D1056 standard test method, specification for flexible cellular materials-sponge or expanded rubber. This test method provides information regarding the behavior of cellular materials under compressive loads. The samples of NR foam were cut without any skin layer exists on the top and bottom of the rubber foam by a sharp blade into square blocks with 50 mm×50 mm×25 mm dimensions. Three specimens were used for the compression tests. The stress–strain diagrams of the samples were obtained using Universal Testing Machine (Testometric instrument) 2,500 kgf and the cross-head speed was 20 mm/min downwards to measure its compression stress, (Test specimens were deformed at cross head speed and compressed up to 75% of their original thickness). The test was stopped when machine gives overloading alarm. Deformation data and complete load deformation curve were taken from Wintest software program supplied from Testometric Co.

The compression deflection was obtained from the average value of three specimens and was reported in unit of kilopascal (kPa) as written in equation 19.

$$\text{Compressive deflection} = \frac{F}{A} \qquad (19)$$

Where

F = Final force at 75% compression of thickness of the specimen, N

A = Cross section area of specimen, mm^2

Figure 21 Materials Testing Machine by Testometric.

5. Acoustic properties

5.1 Sound absorption measurement

The material measurements were based on a two-microphone transfer-function method according to ISO 10534-2 (The frequency was range from 50 to 6400 Hz.), which is for horizontally mounted orientation-sensitive materials. The testing apparatus was part of a complete acoustic material testing system, featuring Brüel&Kjaer PULSE^{TM} interface, as it is seen in Figure 22.

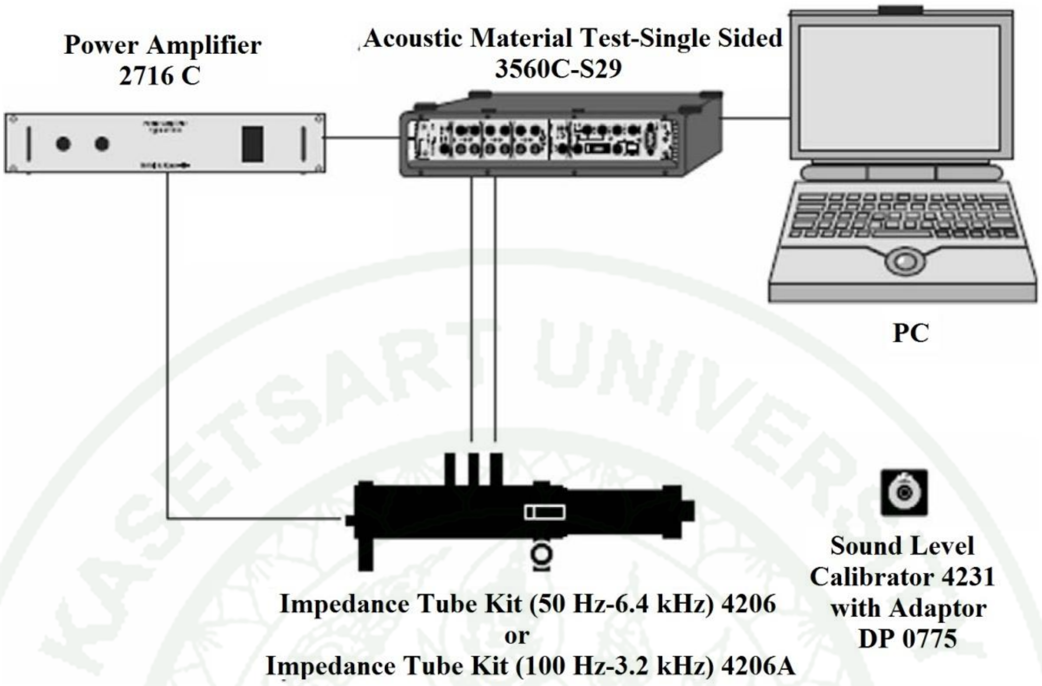


Figure 22 Impedance tube kit (courtesy of Brüel&Kjaer).

Acoustics determination of sound absorption coefficient in impedance tube have two different sizes of specimens in form of dish: diameter 29 mm and diameter 100 mm were used at two different frequency ranges: high-frequency (2000 and 4000 Hz) and low frequency (125, 250, 500 and 1000 Hz), respectively. A small and large tube setup was used to measure different acoustical parameters for the frequency range of 500-6400 and 50-1600 Hz, respectively. Small and large impedance tube kit from Bruel&Kjaer Type 4206 consists of a 29 and 100 mm diameter tube, sample holder and an extension tube at the same diameter. A frequency-weighting unit is also provided within the tube, in which different types of weighting are available; highpass, for high frequency measurements in the small tube, linear for measurements in the large tube, and low-pass for additional measurement accuracy below 100 Hz. At one end of the tube, a loudspeaker is situated to act as a sound source. At the other end of the tube, the test material is placed to measure sound absorption properties, as shown in Figure 23. For proper fitting of samples into the measurement tube, an aluminum rod was machined to a length of 40 mm and it was utilized to push the material into a pre-adjusted depth.

Measurements are done with two different tubes in order to cover a wide frequency range: a 100 mm diameter tube (50Hz - 1600Hz) and a 29 mm diameter tube (500Hz - 6400Hz). The absorption coefficient is physically limited to a value that lies between 0 and 1.

In this experiment, all cylindrical samples were cut off each formulation foams with thickness 13 mm.

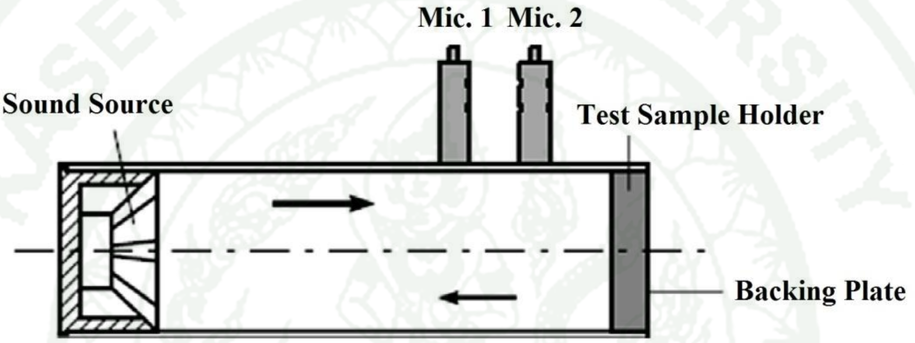


Figure 23 Impedance tube setup for two-microphone transfer function method (courtesy of Brüel&Kjaer).

The Noise Reduction Coefficient (NRC) is the single figure index, indicating the quality of the sound absorption material tested. NRC is calculated using the average of the tested sound absorption coefficient at 250 Hz, 500 Hz, 1000 Hz, and 2000 Hz (Cowan, 2000).

RESULTS AND DISCUSSION

1. Characterization of water hyacinth and blowing agent

1.1 Water hyacinth characterization

Thailand is facing serious water pollution problems, especially in natural water sources such as rivers and canals. One of the main reasons is the widespread of the water hyacinth (*Eichhorinia Crassipes*) plant. Since it can grow and reproduce very fast, it covers the water surface in natural water sources. This prevents oxygen from penetrating and dissolving into the water. A water source can be considered polluted when the dissolved oxygen in the water is lower than a standard limit. Additionally, water hyacinth covers the water surface, so much that it obstructs water transportation. This leads to more fuel consumption and more exhaust to be produced. This research is an attempt to make water hyacinth fibers useful by mixing them with natural rubber and various chemicals to prepare natural rubber foams.

The proximate chemical analysis showed that the water hyacinth fiber contained holocellulose, hemicellulose, and lignin. The data for proximate analyses, physical properties, and some surface characteristics are presented in the Table 3. The proximate analyses showed a low amount of moisture, ash, and dry matter, indicating that the particle density is relatively small. In 2003, Horsfall and Abia proposed that biomaterial should be an excellent raw material for adsorbents to be used in column or fixed-bed reactors. Surface functional groups have been reported (Machida *et al.*, 2006) to have a very important specific influence on the removal of organic and inorganic contaminants in liquid-phase adsorption. The acidity or basicity of an adsorbent surface may give an indication of the ease of quantitative removal of either organic or inorganic pollutants.

Table 3 Proximate properties analyses of water hyacinth.

Chemical compositions	Constituent (%)
Ash	4.14
Moisture content	8.44
Lignin	6.70
Holocellulose	54.80
Hemicellulose	21.00
Dry matter	6.2
Physical properties	**Value**
Diameter, mm	0.80-1.20
Specific gravity (ASTM845-83)	0.428
Water absorption, %	388
Particle density, g cm^{-3}	1.12
Average pore diameter, Å	$1.85x10^{2}$
Pore volume, cm^{3} g^{-1}	0.99427
Surface area, m^{2} g^{-1}	2.44

The FTIR absorption bands of the water hyacinth fiber are presented in Figure 24. The FTIR spectra of the water hyacinth could be assigned as follows. The OH band of water appears at 3388.3 cm^{-1} while the CH of CH_2 and CH_3 form two bands at 2922.6 and 2852.2 cm^{-1}, respectively. The stretching band of C=O at 1738.4 cm^{-1} is for the carboxyl group. Both the symmetric and asymmetric bands of COO at 1640.4 cm^{-1} and 1590.0 cm^{-1}, are presented in the IR spectrum. The C-O band of CHO is located at 1516.7 cm^{-1}. The OH band can overlap with the N-H band which appears at 1422.2 cm^{-1}. The CH of CH_2 band is seen again at 1371.1 cm^{-1}. The C-O band of the cellulose backbone appears at 1035.6 cm^{-1}.

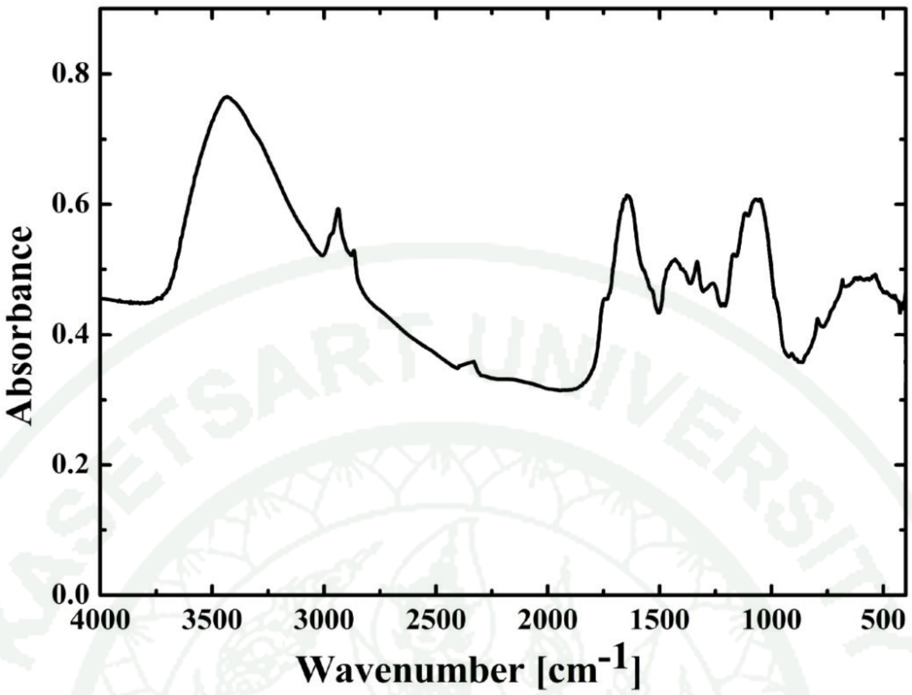


Figure 24 FTIR absorption spectra of water hyacinth fiber.

Very few studies report on the morphology for the water hyacinth fibers. In Figure 25, the scanning electron micrograph (SEM) images of water hyacinth at different magnifications are shown, probably revealing the thin-walled and weak, microscopic pores, in crossed-section of fine fiber. The plants used for the experiments were between two and three months old. Using younger plants with less fiber could possibly ease the processing.

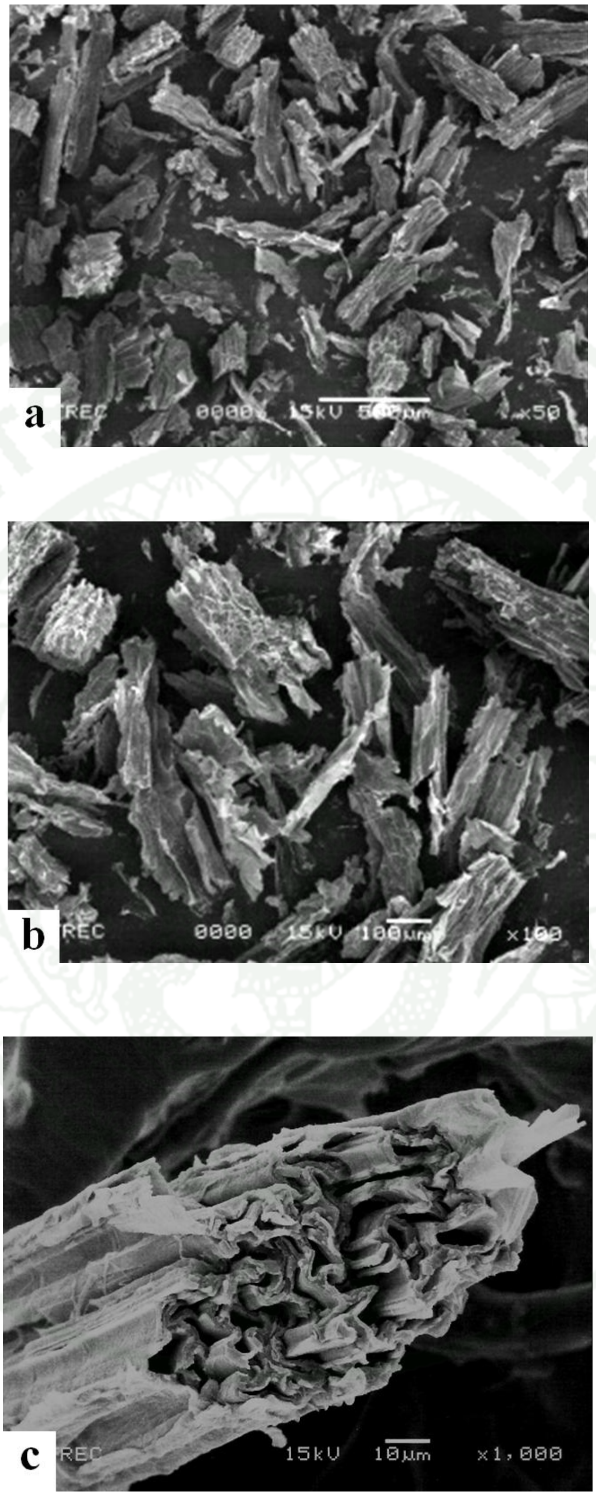


Figure 25 SEM micrographs of fine water hyacinth fiber at different magnifications: (a) longitudinal axis X50, (b) longitudinal axis X100, and (c) crossed-section X1000.

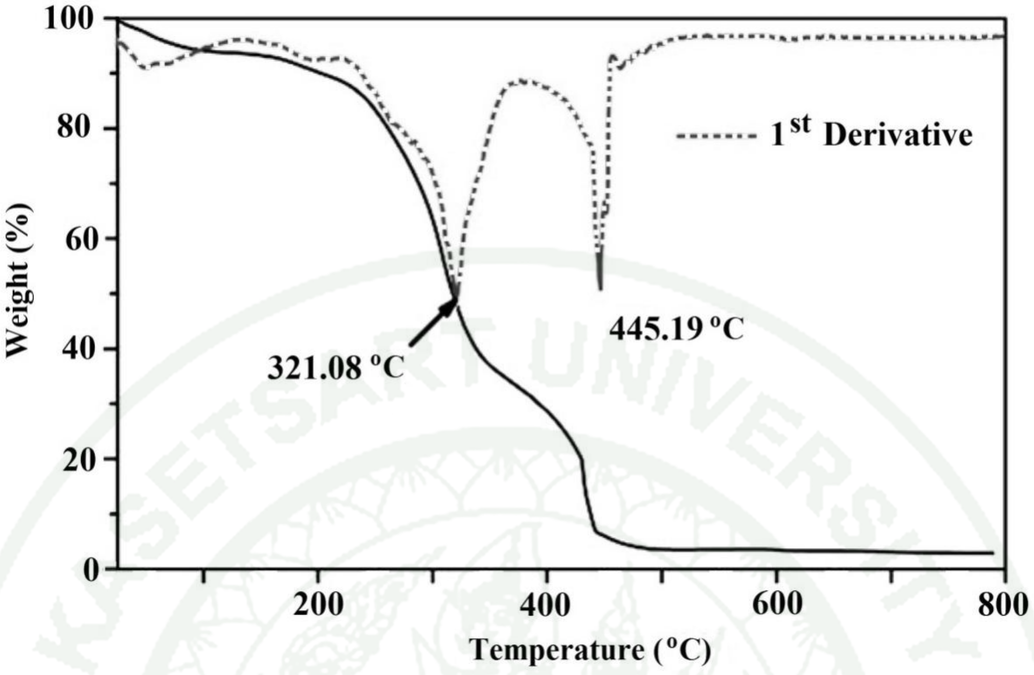


Figure 26 TGA/DTG curves of water hyacinth.

Thermogravimetric analysis (TGA) and Derivative thermogravimetric (DTG; 1st derivative) curves of water hyacinth fiber are shown in Figure 26. It can be seen that the curves in Figure 26 show behavior with three main mass loss steps. The first one up to 100°C representing the mass loss in the water hyacinth fibers could be related to water loss associated with moisture present in the fibers (Table 3). Although the water hyacinth fibers was dried before the analysis, total elimination of water was difficult due to the hydrophilic nature of the fibers, which is presented even as structurally bound water molecules. Then, between 100 and 200°C, the fibers present thermal stability. One may consider 200°C for water hyacinth as the maximum temperature up to which these fibers can be used since above this temperature mass loss is high. From 300°C and above, the water hyacinth fibers show considerable mass loss due to the decomposition of both cellulose and hemicellulose in the fibers. The decomposition temperatures of hemicellulose and α-cellulose of water hyacinth are in the similar range as those of jute, hemp, and sisal (Ray *et al.*, 2002, Albano *et al.*, 1999, and Joseph *et al.*, 2003). Further, above 400°C, degradation of fibers can be seen as a result of the break of bonds of the lignin (Paiva *et al*., 2004; Rohella *et al*., 1996). In both steps the mass loss is attributed to the oxidation (burning) of the fragments in the presence of air. After 500°C only the ashes of water hyacinth can be observed around 4.14%.

1.2 Blowing agent characterization

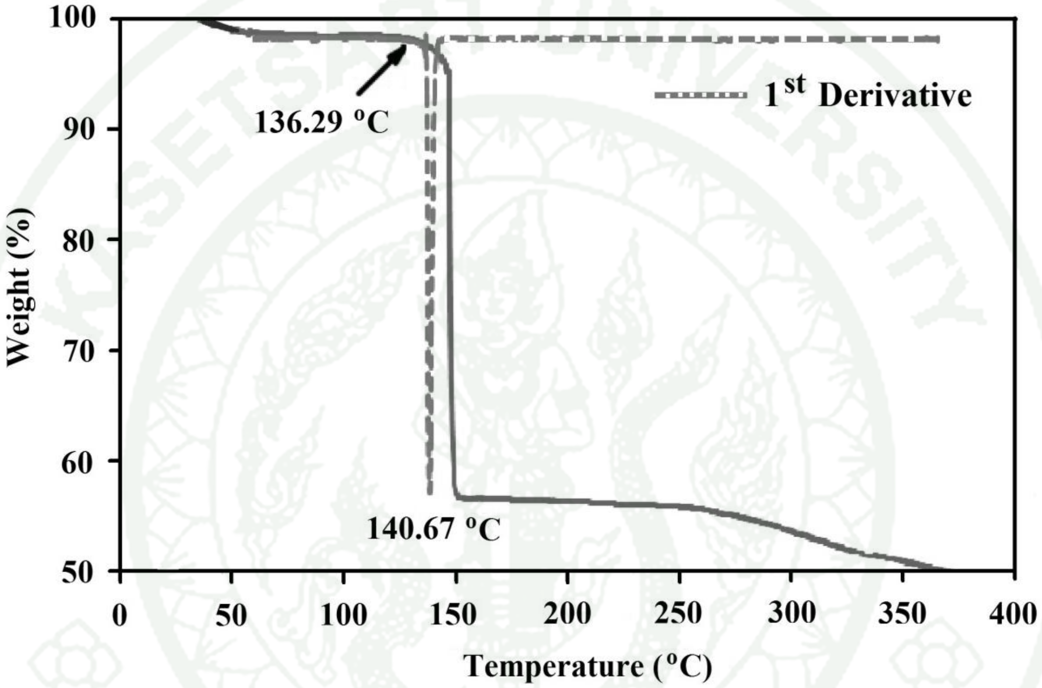


Figure 27 TGA curves of blowing agent (OBSH).

Thermogravimetric analysis (TGA) is used for rapid determination of the thermal stability of substances. Essentially, TGA measures weight loss as a function of temperature under controlled environmental conditions. TGA was used for studying the decomposition temperature of blowing agent or Oxybis (benzene sulfony) hydrazide (OBSH). The thermal decomposition behaviours of OBSH (i.e., decomposition temperature and decomposition rate) were characterised using a TGA at a heating rate of 5°C/min in a nitrogen atmosphere. Figure 27 shows TGA curves during decomposition of OBSH, the weight losses were observed. At temperature of 136.29°C represented an onset of the decomposition, while at 140.67°C indicated the point at which the maximum rate of the decomposition occurred. OBSH, supplied by

A.F. Goodrich Chemical Co., Ltd., (Bangkok, Thailand) was used as the chemical blowing agents. According to the suppliers, blowing agents had the decomposition temperature of around 140-170°C. Work on the effects of blowing agents on the foaming characteristics of cellular plastic and rubbers were carried out and reviewed by Rowland (1993).

2. Factors affecting the cure characteristic of NR foams

The influences of the foaming temperature, water hyacinth, and blowing agent content on the cure behavior and cell morphology of the solid dry natural rubber (NR) foams were investigated at three different foaming temperatures, i.e. 150, 160, and 170°C and different feeding ratios of the water hyacinth (0, 5, 10 phr) and blowing agent (0, 2, 4, 6 phr). The study was carried out using formulated compositions containing oxybis (benzene sulfonyl) hydrazide (OBSH), which releases nitrogen gas during its decomposition and is able to produce foam with small, uniform cells, and an open cell structure as the chemical blowing agent and was expanded using conventional compression molding technique.

2.1 Effect of foaming temperature

Dry rubber foaming can be implemented through two processes which are called single stage process and heat transfer process. The expansion of the compound for the single stage process occurs while opening the mold. While in the two-stage heat transfer process, the first stage is identical to the single stage process but using lower temperature. On opening of compression molding platen, the pre-expanded rubber compound is immediately transferred to a circulating hot air oven at a higher temperature (Ariff, 1998). Temperature is one of the important processing parameters that could yield different foam properties.

The cure characteristics of the NR foams with and without water hyacinth and blowing agent at various contents were determined by using the Moving Die Rheometer (MDR) at 150, 160, and 170°C as presented in Table 4. The scorch time

(t_{s2}) or time to incipient cure is a measure of the time taken for the minimum torque value to increase by two units. It can be seen that the scorch time decreased with increasing foaming temperature. This observation may be attributed to the viscosity decrease upon prevulcanization along with the rising foaming temperature. The optimum cure time (t_{c90}), time for vulcanization where t_{c90} denotes 90% of maximum torque, decreased with increasing foaming temperature. Increasing the foaming temperature subsequently causes fast cure rate as depicted in Table 4. The minimum torque (M_L) is a measure of stiffness of the unvulcanized test specimen taken at the lowest point of the cure curve. The minimum torque decreased with increasing foaming temperature. This is because homogenous mastication and high temperature could reduce the viscosity of rubber compound. Meanwhile, the maximum torque (M_H) is a measure of stiffness or shear modulus of the fully vulcanized test specimens at the vulcanization temperature. It can be seen that the maximum torque also decreased when the foaming temperature was increased. This is due to the fact that crosslink density decreased with increasing foaming temperature, which is in agreement with a decrease in delta torque after increasing the foaming temperature as shown in Table 4. This was also reported by Ismail and Hashim (1998) where the increase in maximum torque was due to the presence of higher crosslink density level in the compound. Presence of higher crosslinking will subsequently increase the viscosity and reduce the capability of expansion due to rigidities of the compound, resulting in the decrease in expansion ratio with decreasing foaming temperature (Table 5).

Table 5 shows the cure rate index of the foamed NRs with respect to various foaming temperatures. The cure rate index is calculated as follows.

$$\text{Cure rate index} = \frac{100}{(t_{c90}-t_{s2})} \qquad (20)$$

The cure rate index is a measure of the rate of vulcanization based on the difference between the optimum vulcanization time, t_{c90}, and incipient scorch time, t_{s2}. Vulcanization, as well as scorch, is very sensitive to the temperature conditions (Lee *et al*., 2007). As presented in Table 5, it can be seen that the increase of the foaming temperature accelerated the cure rate index in which the cure rate index at 150°C was smaller than those at 160 and 170°C.

2.2 Effect of water hyacinth loading

The influence of the water hyacinth loading on the cure behavior of NR foams was investigated at different foaming temperature and different feeding ratios of the water hyacinth. It can be seen that the scorch time decreased with the increasing water hyacinth loading. This may be attributed to the viscosity increased with the increase of water hyacinth content. Therefore, a shorter time was required for the beginning of the vulcanization process for the NR foam compounds. A decreasing trend of the cure time was also observed with increasing the foaming temperature and the water hyacinth content. The reduction in cure time of the filled vulcanizates was attributed to the higher time the rubber compounds remain on the mill during mixing. As the water hyacinth fiber loading increases, the time of incorporation also increases and consequently generates more heat due to friction. At the same time, the variation of maximum torque and minimum torque values in different water hyacinth contents was also given in Table 5. Both the maximum torque and minimum torque are also increased after the addition of the water hyacinth fiber into the NR matrix. The presence of water hyacinth fibers generates an increase in viscosity of the NR compounds. The increment in torque values with increasing water hyacinth fiber loadings indicated that as more and more water hyacinth fiber gets into the rubber matrix, the mobility of the macromolecular chains of the rubber reduces resulting in more rigid vulcanizates. Increase of maximum torque is due to the presence of crosslink density that caused restriction to flow hence increasing the viscosity (Ismail and Hashim, 1998).

The effects of the water hyacinth content compared to the influence of foaming temperature on the cure rate index were different as depicted in Table 5. Cure rate index gradually increases upon increasing the water hyacinth content, but there was no significant rise. It is obvious that the cure rate index is more sensitive to the vulcanizing temperature than to filler content. In 2004, Lin *et al*. proposed that the short fibers have no influences on the cure process of microcellular rubber foams, nor on the main foaming dynamics of the composites.

2.3 Effect of blowing agent loading

The foam structure can be controlled by the proper selection of blowing agents and curatives to achieve the correct balance between the gas generated and the degree of curing (Egli, 1972 and Ariff *et al*., 2007). Oxybis (benzene sulfonyl) hydrazide: OBSH, which was used in this study, is an organic chemical blowing agent that releases nitrogen gas during decomposition. In this study, natural rubber foams were prepared by varying the content of OBSH (0, 2, 4, and 6 phr) at different foaming temperature and water hyacinth content.

The cure characteristics of NR foams produced at various blowing agent content with/without water hyacinth content and different foaming temperature are shown in Tables 4-5. The minimum torque indicates that the blowing agent content did not significantly affect the compound viscosity prior to crosslinking. It can also be seen that as the blowing agent content increased, the value of the maximum torque decreased. The decrease in the maximum torque value results from the fact that higher blowing agent content generates more nitrogen gas in the rubber phase, simultaneously producing more microvoids. These microvoids reduce the shearing force; therefore, the torque began to decrease at the onset of the blowing agent decomposition and reached an equilibrium state (Ariff *et al*., 2007). The scorch time is the induction time experienced by a rubber compound before vulcanisation initiates. Table 4 illustrates a decreasing trend in scorch time as the blowing agent content increases. This may be attributed to the decrease in compound viscosity. A decrease in the cure time was also observed. Strauss and D’Souza, (2004) claimed that carbon dioxide gas can act as an efficient solvent in most polymers; the gas molecules accumulate interstitially between the polymer chains, thus increasing the free volume and mobility of the chain. Normally for blowing agent, majority of the gas release is nitrogen. Nitrogen consists of small molecules which allowed it to be absorbed into the compounded rubber and reduce the matrix viscosity. As a result, it directly affects the crosslinking rate due to higher volumetric gas decomposition in the vulcanized rubber. The data is supported by the crosslink density which will be discussed in the next section**.**

Table 4 Cure characteristics of the NR foams.

Water Hyacinth Content (phr)	Formula	Scorch time, t_{s2}			Cure time, t_{90}			Minimum torque, M_L (dNm)			Maximum torque, M_H (dNm)		
	Foaming Temp* (°C)	150	160	170	150	160	170	150	160	170	150	160	170
0	NF-0	4.53	2.53	1.48	7.25	4.06	2.29	0.41	0.39	0.35	5.45	5.42	5.14
	NF-2	4.85	3.39	2.46	8.67	4.33	2.84	0.43	0.38	0.36	4.39	4.33	3.80
	NF-4	4.38	2.68	2.42	8.44	4.28	2.72	0.44	0.41	0.39	3.23	3.21	2.87
	NF-6	3.83	2.18	2.14	8.35	4.24	2.67	0.43	0.34	0.31	3.18	2.78	2.54
5	NF-0	3.07	1.84	1.15	5.62	3.31	2.20	0.49	0.44	0.42	6.07	5.93	5.56
	NF-2	4.83	3.26	2.37	7.36	4.04	2.78	0.44	0.40	0.39	4.46	4.38	3.87
	NF-4	4.33	2.62	2.31	7.21	4.01	2.69	0.43	0.43	0.40	4.13	3.79	3.35
	NF-6	3.68	2.20	2.11	7.20	3.92	2.63	0.42	0.39	0.34	3.23	2.82	2.58
10	NF-0	2.81	1.71	1.07	5.61	3.21	2.15	0.66	0.56	0.52	6.29	5.72	5.39
	NF-2	4.42	3.18	2.27	7.30	3.83	2.70	0.51	0.47	0.46	4.78	4.55	4.37
	NF-4	4.34	2.28	2.23	7.23	3.45	2.61	0.52	0.46	0.45	4.32	3.75	3.55
	NF-6	3.42	2.12	2.05	7.17	3.39	2.51	0.50	0.45	0.43	3.28	2.99	2.60

Note: NF-0, 2, 4, and 6; N refers to NR, F refers to foam and 0, 2, 4, and 6 refers to blowing agent (OBSH) content (0, 2, 4, 6, respectively), and Temp* refers to temperature.

Table 5 Rheometric characteristics and expanding properties of the NR foams.

Water Hyacinth Content (phr)	Formula / Foaming Temp* (°C)	Delta torque (dNm)			Cure rate index			Density (g/ml)			Expansion ratio		
		150	160	170	150	160	170	150	160	170	150	160	170
0	NF-0	5.04	5.03	4.79	36.76	65.36	123.46	0.95	0.93	0.94	1.05	1.07	1.06
	NF-2	3.96	3.95	3.44	26.18	106.38	263.16	0.93	0.90	0.90	1.08	1.10	1.11
	NF-4	2.79	2.80	2.48	24.63	62.50	333.33	0.82	0.86	0.80	1.14	1.20	1.21
	NF-6	2.75	2.44	2.23	22.12	48.54	188.68	0.69	0.74	0.69	1.16	1.21	1.24
5	NF-0	5.58	5.49	5.14	39.22	68.03	95.24	0.95	0.92	0.94	1.05	1.09	1.06
	NF-2	4.02	3.98	3.48	39.53	128.21	243.90	0.90	0.86	0.80	1.10	1.16	1.25
	NF-4	3.70	3.36	2.95	34.72	71.94	263.16	0.84	0.78	0.72	1.19	1.27	1.37
	NF-6	2.81	2.43	2.24	28.41	58.14	192.31	0.80	0.76	0.67	1.24	1.30	1.48
10	NF-0	5.63	5.16	4.87	35.71	66.67	92.59	0.98	0.97	0.93	1.02	1.02	1.07
	NF-2	4.27	4.08	3.91	34.72	153.85	232.56	0.88	0.85	0.79	1.13	1.17	1.25
	NF-4	3.80	3.29	3.10	34.60	85.47	263.16	0.78	0.74	0.66	1.27	1.35	1.50
	NF-6	2.78	2.54	2.17	26.67	78.74	217.39	0.73	0.70	0.60	1.35	1.42	1.66

Note: NF-0, 2, 4, and 6; N refers to NR, F refers to foam and 0, 2, 4, and 6 refers to blowing agent (OBSH) content (0, 2, 4, 6, respectively), and Temp* refers to temperature.

3. Factors affecting the morphology and physical properties of NR foams

The influence of various water hyacinth and blowing agent contents at different foaming temperature on the cell morphology, foam characteristics, and physical properties of NR foams was presented in this section. In 1991, Klempner and Frisch reported that cell size and cell size distribution depend on filler loading, particle size and the type of blowing agent used. Furthermore, foam properties will also depend on the density and the mechanical properties of the base polymer (Sims *et al*., 1995). Thus, this study focuses on the factors that affect the morphology and physical properties, including the relative density, crosslink density and average cell size of NR foams. Furthermore, the density and expansion ratio of the foamed NRs were also studied. SEM analysis was used to investigate the effects of the foaming temperature, water hyacinth and blowing agent content on the morphologies of the foamed NRs.

3.1 Effect of foaming temperature

The effects of the foaming temperature, water hyacinth, and blowing agent content on the morphology and physical properties of NR foams were shown in Table 6. The apparent relative foam density and crosslink density of the NRs foamed at 150, 160, and 170°C with/without water hyacinth contents including various blowing agent were described in Figures 28-31. A decreasing trend of the relative foam density was observed upon increasing the foaming temperature. The NRs with and without blowing agents foamed at 170°C show lower relative foam densities than those foamed at 150 and 160°C, as seen in Figure 28, Figures 29(b)-31(b). The density of gas cells inside the rubber matrix increases upon increasing the foaming temperature, whereas their volume is enlarged by the thermal expansion effect. This situation is also supported by the expansion ratios observed in Table 5, which can be closely related with the time difference between the decomposition temperature of the blowing agent and curing temperature. It has been reported that the curing temperature should not be prior to and not be too close to or too far from the decomposition temperature of the blowing agent. Therefore, control of the curing temperature is very important for foaming. As a result, in this study, the lowest foam density for the foamed NRs was achieved at 170°C.

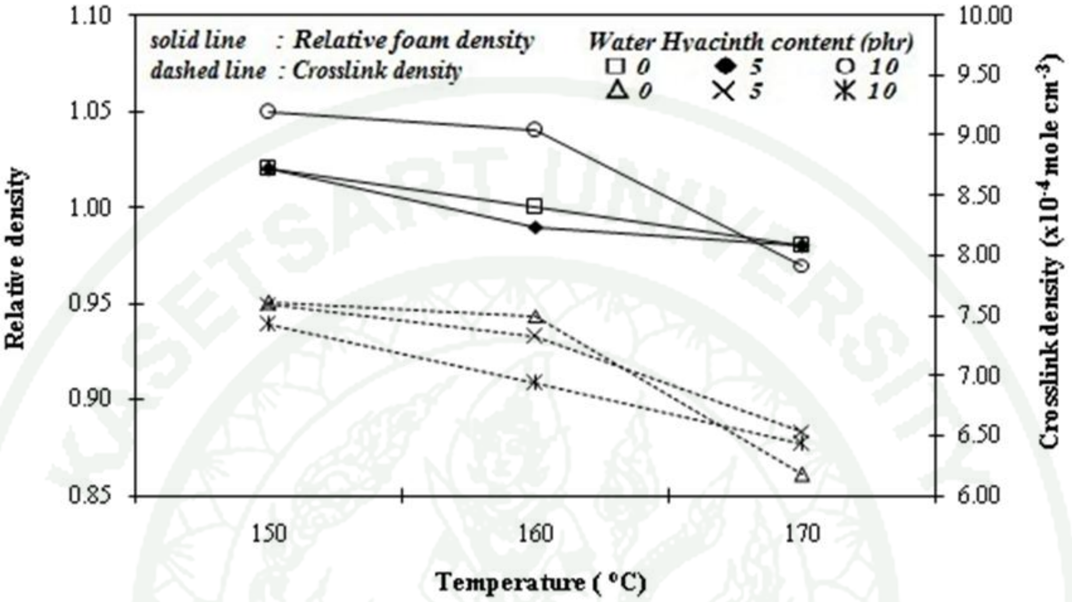
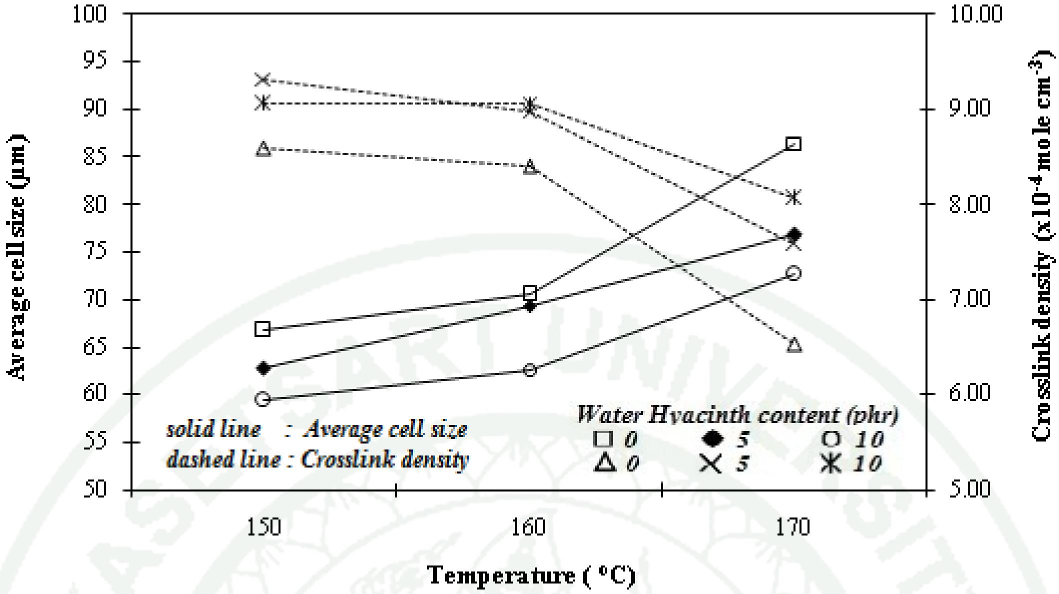


Figure 28 The effect of foaming temperatures on relative foam density and crosslink density for NR vulcanizates without blowing agent at various water hyacinth content.

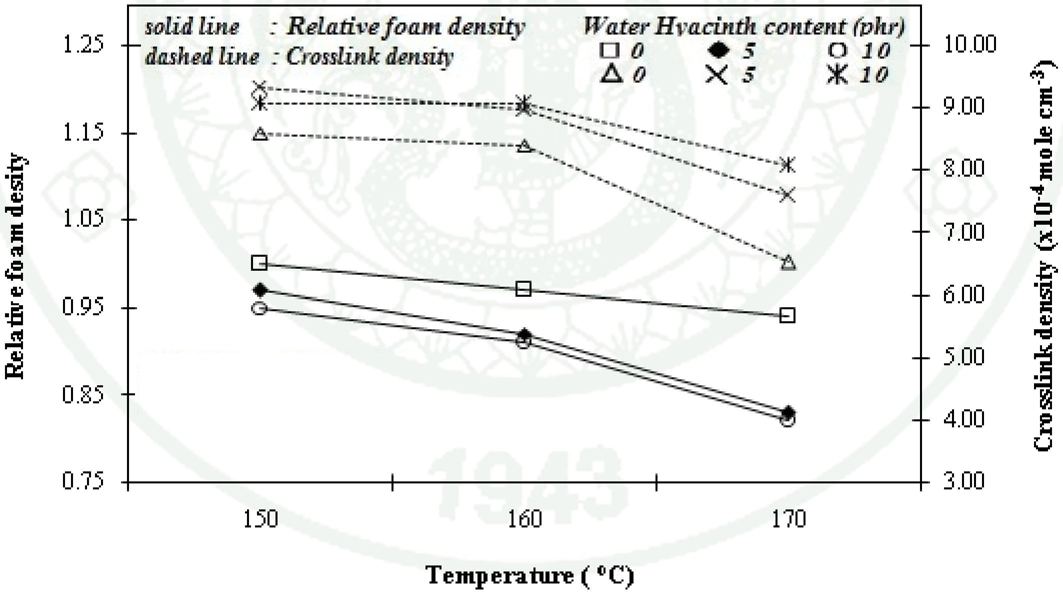
Table 6 Effect of foaming temperature on average cell size, relative foam density and crosslink density of the NR foams.

Water Hyacinth Content (phr)	Formula / Foaming Temp* (°C)	Average cell size (μm)			Relative foam density			Crosslink density ($\times 10^{-4}$ mole cm^{-3})		
		150	160	170	150	160	170	150	160	170
0	NF-0	-			-			7.61	7.50	6.18
	NF-2	66.76	70.51	86.48	1.00	0.97	0.94	8.59	8.40	6.53
	NF-4	65.40	87.25	113.70	0.92	0.88	0.83	8.56	8.38	7.36
	NF-6	106.96	114.61	120.85	0.80	0.74	0.72	8.50	9.40	7.29
5	NF-0	-			-			7.60	7.32	6.54
	NF-2	62.77	69.37	76.93	0.97	0.92	0.83	9.32	8.98	7.58
	NF-4	63.83	70.10	85.75	0.90	0.84	0.75	9.17	8.68	7.30
	NF-6	87.74	94.59	95.92	0.86	0.82	0.70	9.40	9.60	8.70
10	NF-0	-			-			7.42	6.93	6.43
	NF-2	59.39	62.60	72.71	0.95	0.91	0.82	9.05	9.07	8.07
	NF-4	63.51	82.36	91.17	0.84	0.80	0.69	8.85	8.44	7.53
	NF-6	84.12	89.67	94.90	0.78	0.75	0.63	8.94	9.08	8.90

Note: NF-0, 2, 4, and 6; N refers to NR, F refers to foam and 0, 2, 4, and 6 refers to blowing agent (OBSH) content (0, 2, 4, 6, respectively), and Temp* refers to temperature.

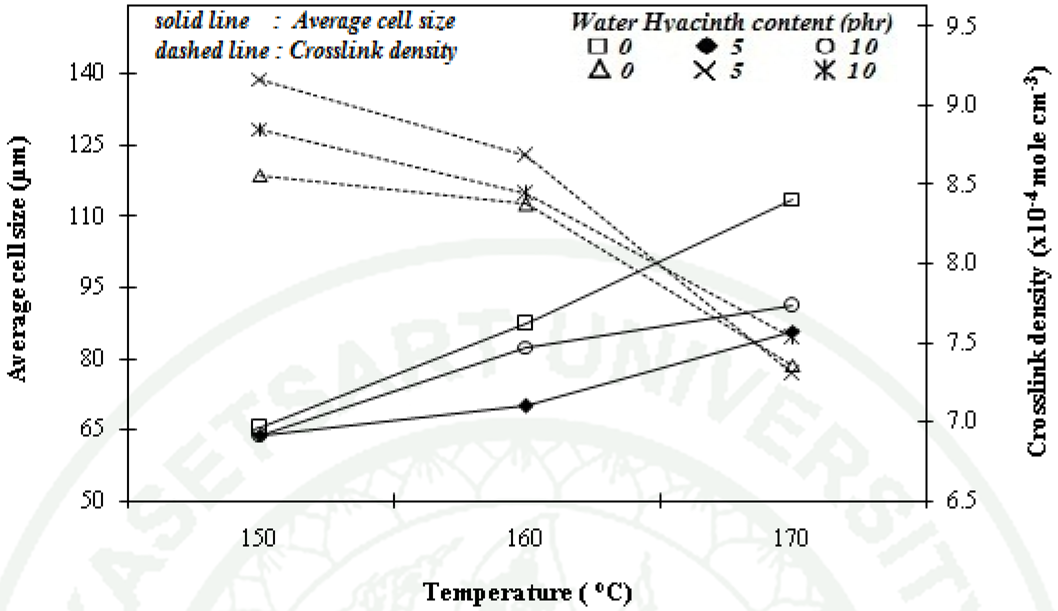


(a)

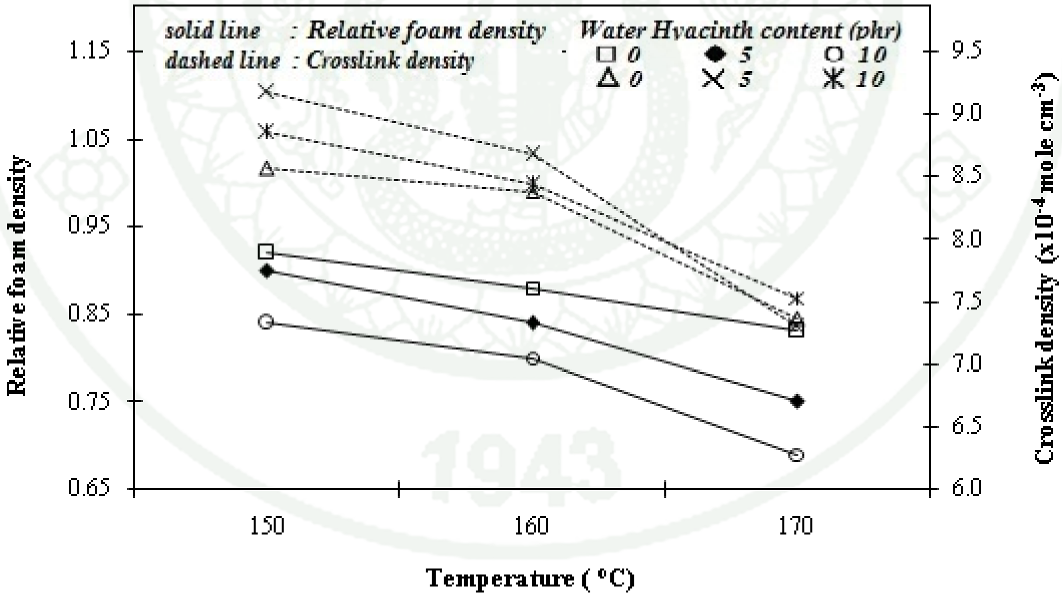


(b)

Figure 29 The effect of foaming temperatures on (a) average cell size and crosslink density and (b) relative foam density and crosslink density for NR foams containing 2 phr of blowing agent at various water hyacinth content.

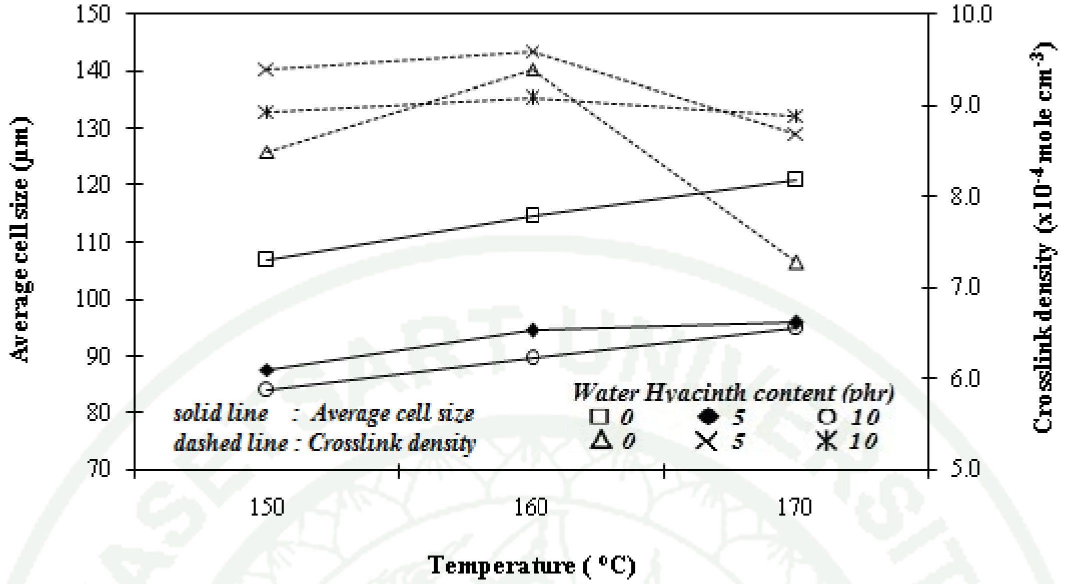


(a)

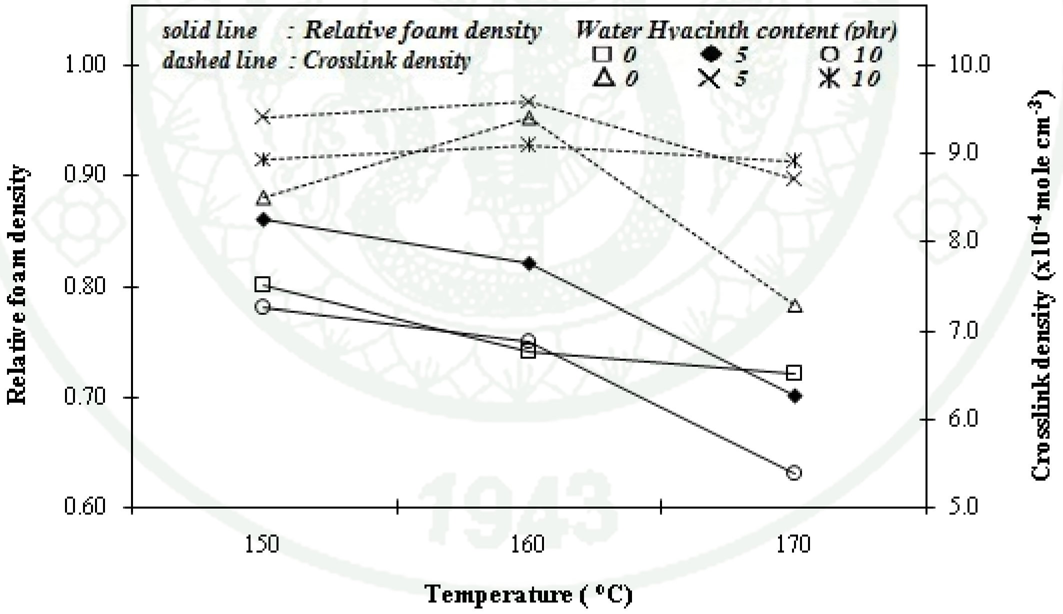


(b)

Figure 30 The effect of foaming temperatures on (a) average cell size and crosslink density and (b) relative foam density and crosslink density for NR foams containing 4 phr of blowing agent at various water hyacinth content.



(a)



(b)

Figure 31 The effect of foaming temperatures on (a) average cell size and crosslink density and (b) relative foam density and crosslink density for NR foams containing 6 phr of blowing agent at various water hyacinth content.

Figure 28 and Figures 29(b)-31(b) reveal quantitatively that relative density and crosslink density of rubber foam decreased with increasing foaming temperature, whereas the average cell size increased. Higher rigidities of the vulcanisates and greater restriction to expansion occurred at lower foaming temperature. It was supported by the SEM results shown in Figures 33-35, where the cells produced at lower temperature were slightly smaller compared to the cells produced at higher temperature. This is because the vulcanisate was more rigid and difficult to expand. However, higher foaming temperature would give greater expansion due to the presence of higher gas volume which would result in decreasing relative foam density.

Nevertheless, using results of minimum torque (Table 4) and qualitatively evidence of Figures 33-35, it was obvious that less restriction to expansion was achieved at higher foaming temperature. In this condition, as seen in Figures 29(a)-31(a) the existence of greater gas volume and lower crosslink density, will increase the average cell size and subsequently decrease foam density and this can be related to the recorded lower minimum torque (Table 4) causing the flexiblities (i.e. low viscosity). This subsequently gave supplement to gas expansion to cell growth and cause increasing in cell size. Increase the cell size adversely affects the stability of the cell foam and is a possible cause of the eventual fracture of the cell walls (Klempner and Frisch, *et al*., 1991). The decreased in crosslink density would probably caused by the breakdown of strained crosslink in the polymer network as it swelled (Sombatsompop and Lertkamolsin, 2000). The peculiar occurrence of decrease in crosslink density at higher temperature would be probably caused by the breakdown of strained crosslink in the polymer network (Ariff *et al*., 2008).

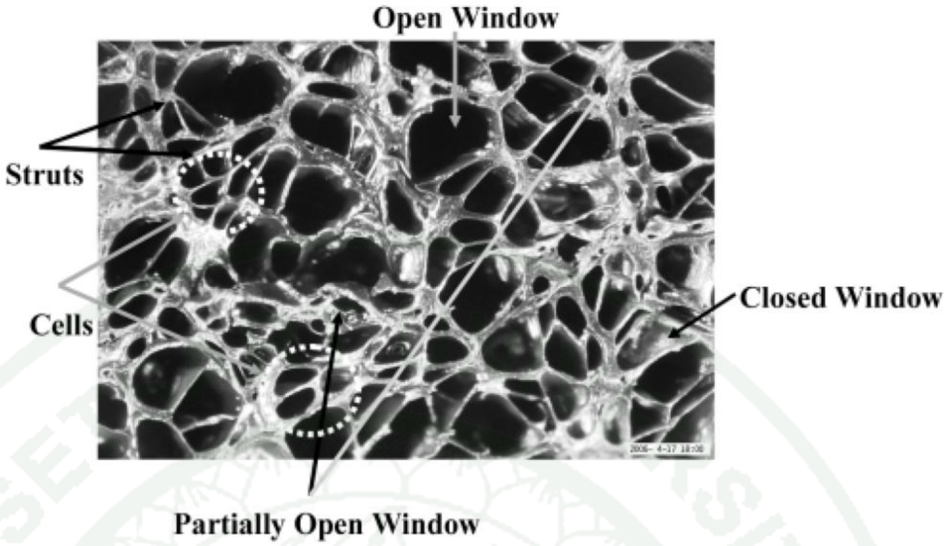


Figure 32 Morphology of flexible NR foam at 100× magnification.

Sourece: Kim *et al*., (2006)

Foams are three-dimensional agglomerations of gas bubbles, separated from each other by thin sections of polymer (Kim *et al*., 2007). For a finished flexible NR foam, as shown in Figure 32, the void areas as “cells” are artifacts of gas bubbles that were introduced into the reacting mixture early in its existence. The thin, geometrical structures separating the void areas are made up of the polymer formed from the various reactions.

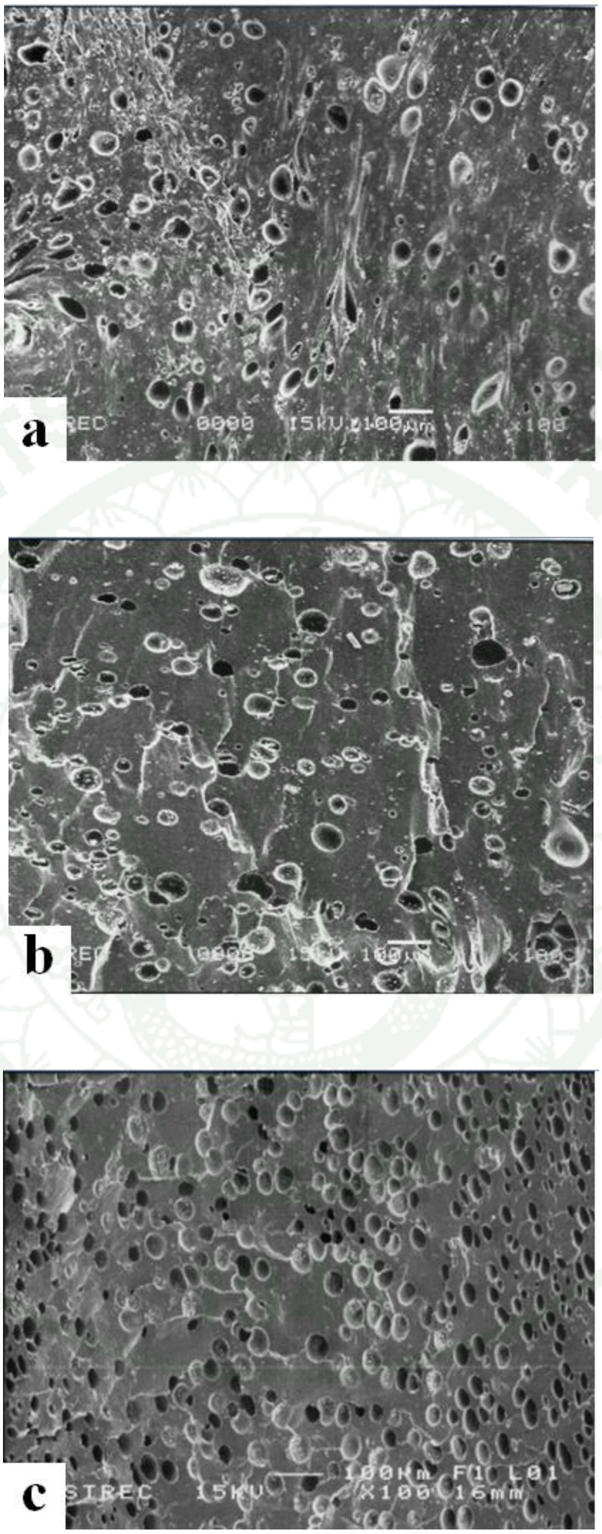


Figure 33 SEM micrographs of the NR foams without water hyacinth containing 2 phr of blowing agent at different foaming temperatures: (a) 150°C, (b) 160°C, and (c) 170°C.

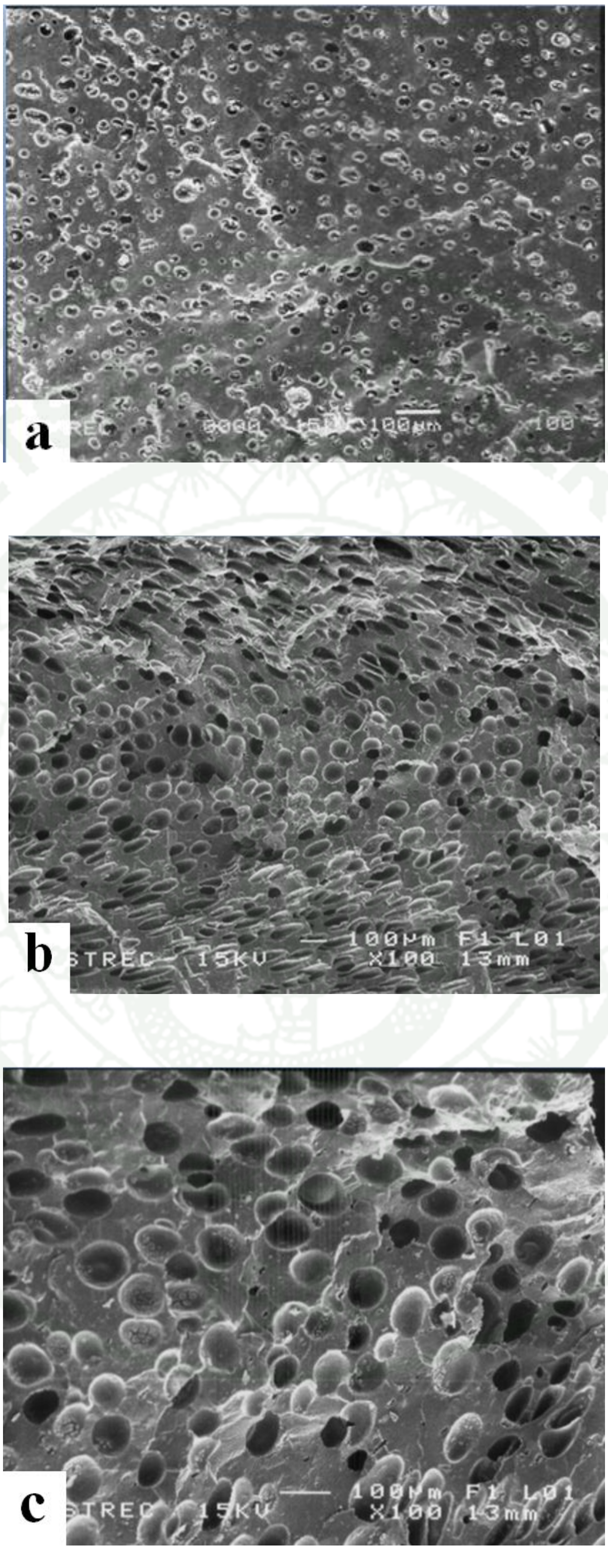


Figure 34 SEM micrographs of the NR foams without water hyacinth containing 4 phr of blowing agent at different foaming temperatures: (a) 150°C, (b) 160°C, and (c) 170°C.

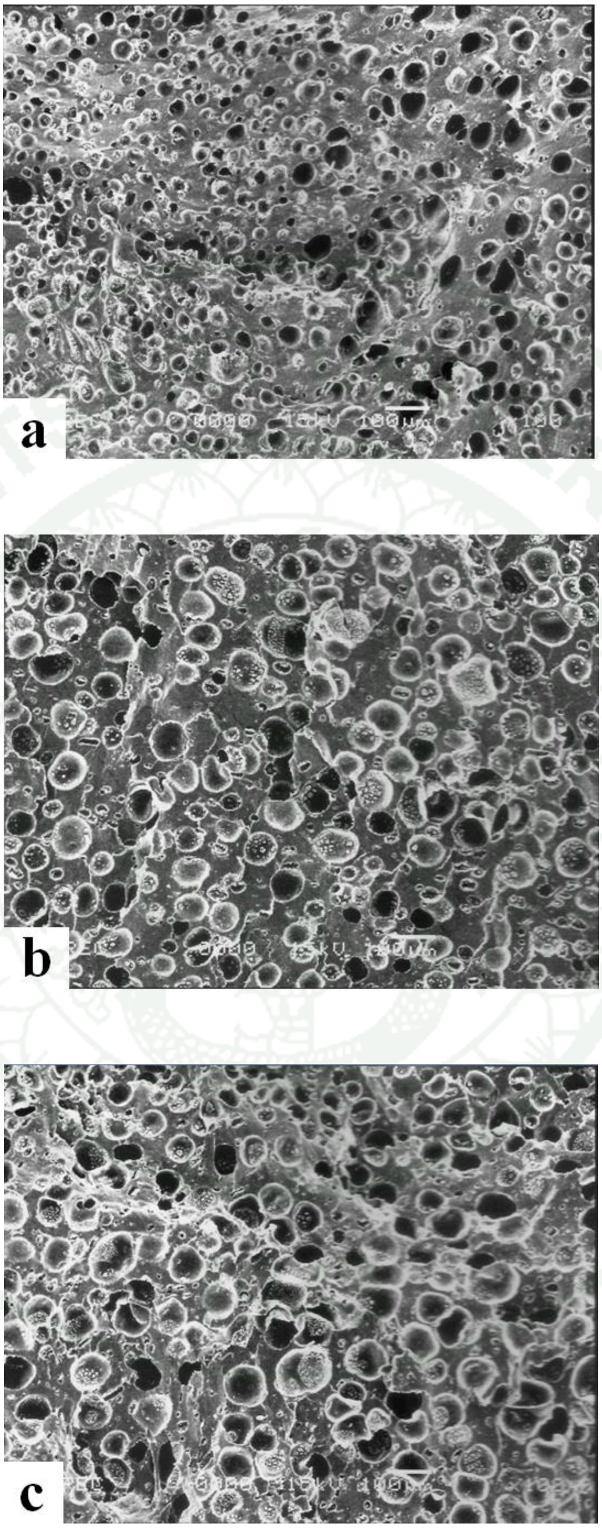


Figure 35 SEM micrographs of the NR foams without water hyacinth containing 6 phr of blowing agent at different foaming temperatures: (a) 150°C, (b) 160°C, and (c) 170°C.

SEM micrographs of NR foam samples at three different foaming temperatures are shown in Figures 33-35. The thickness of each of the struts (thickness cells wall) formed inside the rubber matrix decreased with increasing foaming temperature. Considering the shapes of the bubbles with foaming temperature, they are almost spherical at high temperature but show irregular spherical shapes with decreasing foaming temperature. In the early stages of foaming, when the gas volume is small, bubbles are spherical. As the bubble volume grows, however, the fluid phase becomes insufficient to maintain the spherical shape, so that the bubbles take on polyhedral shapes, with the fluid distributed in thin membranes between two adjacent bubbles. In other words, it is difficult for spherical bubbles to maintain their shape because gas tends to diffuse from the smaller bubble into the larger one, resulting in the destruction of spherical shape. Therefore, some of the spherical bubbles changed into a distorted spherical shape. The increase of the foaming temperature, made foaming efficiency higher, leading to the thin struts. It was clearly from the SEM micrographs that the NRs foamed at 170°C had a higher foaming efficiency, supporting by the density and expansion ratio results shown in Table 5. It can be explained that the crosslink rate would significantly increase at lower foaming temperature and thus avoid the expansion listed in Figures 29(a)-31(a). At higher foaming temperature, higher gas is generated by the blowing agent 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. Generally, the foaming mechanism of rubber foam is exactly similar to plastic foaming mechanism (Lin *et al*., 2004).

When temperature increases, thermal decomposition occurs and releases gas, nitrogen. The decomposition behavior of OBSH is one function of particle size, heating rate, activator type and concentration and uniformity of dispersion, which affect the total gas yields and the rate of gas evolution of OBSH to eventually change the properties of foams. Temperature is very crucial for producing rubber foams because it can affect the decomposition of the chemical blowing agent (OBSH), which influences cell nucleation, growth, stabilization, density, and cell size including retention of the foam. For a successful foaming process, the temperature should give a sufficiently low viscosity to allow cell formation and expansion and sufficiently high viscosity to prevent cell collapsing (Zunaida *et al*., 2007).

3.2 Effect of water hyacinth loading

SEM micrographs of the fractured surfaces of the NR vulcanizates (without blowing agent) filled with and without water hyacinth fiber (0, 5, 10 phr) were cured at various temperatures from 150°C-170°C depicted in Figures 36-39. It was clearly noticed that the distribution of water hyacinth fiber in the rubber matrix was quite poor, which may be one of the reasons for the regression in some mechanical properties which will be discussed in the next section.

Anthoine *et al*., (1975) proposed that cellulose short fiber could be used as filler in natural rubber compounds. In 2004, Lin *et al*., pointed out that for application, it is necessary for short fibers to stay in the rubber matrix to guarantee the reinforcement and, hence, suggesting short fibers is mainly responsible for the great enhancement of most mechanical properties of the rubbers.

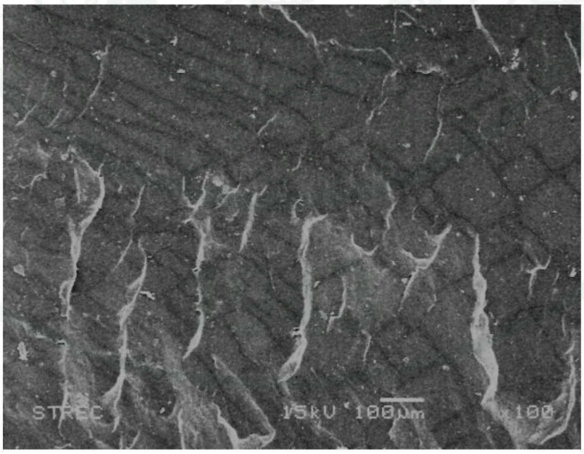


Figure 36 SEM micrographs of the NR vulcanizates without water hyacinth and blowing agent at curing temperatures of 160°C.

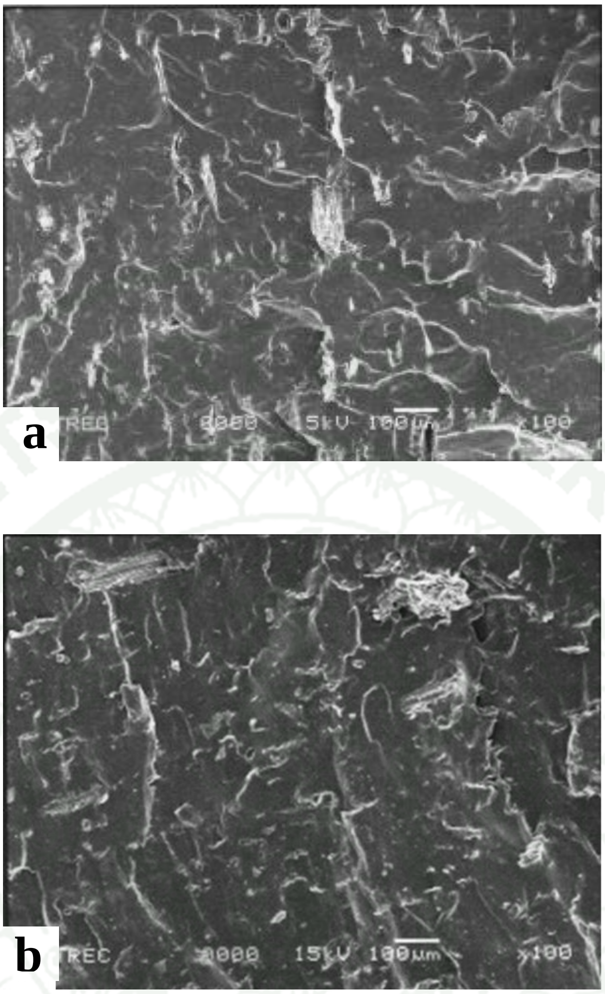


Figure 37 SEM micrographs of the NR vulcanizates without blowing agent at curing temperatures of 150°C filled with various water hyacinth content: (a) 5 phr, and (b) 10 phr.

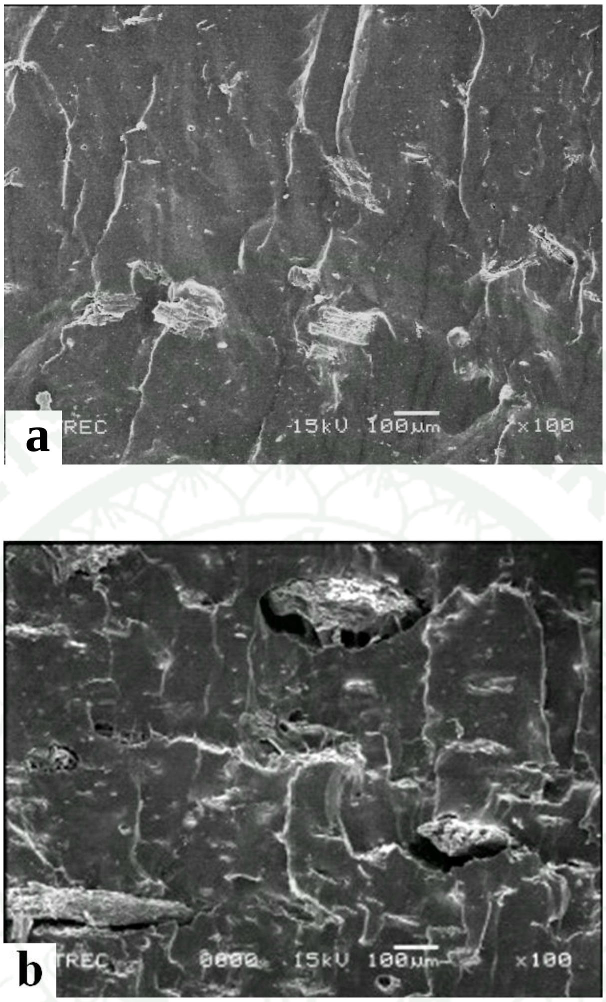


Figure 38 SEM micrographs of the NR vulcanizates without blowing agent at curing temperatures of 160°C filled with various water hyacinth content: (a) 5 phr, and (b) 10 phr.

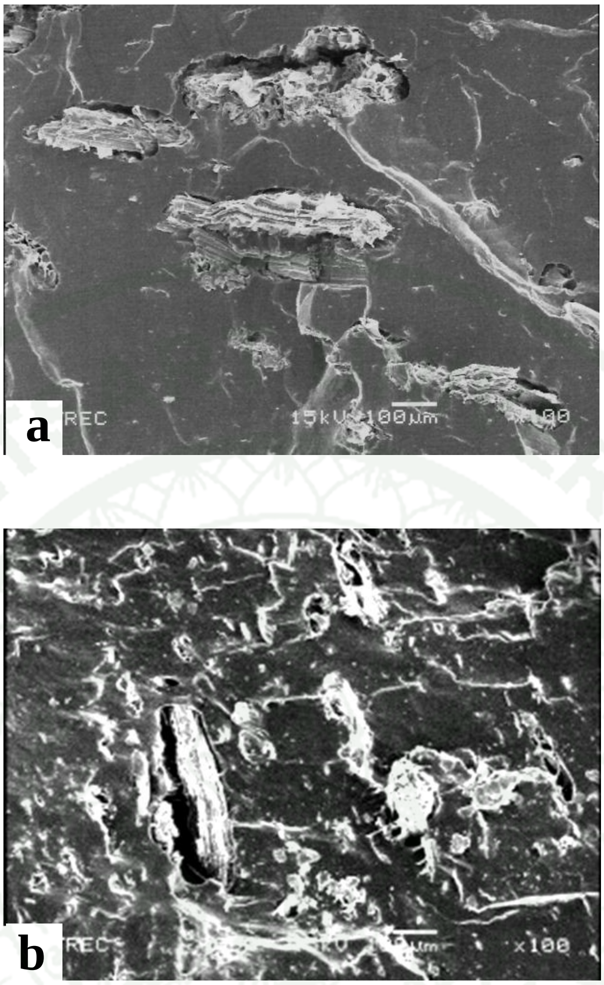


Figure 39 SEM micrographs of the NR vulcanizates without blowing agent at curing temperatures of 170°C filled with various water hyacinth content: (a) 5 phr, and (b) 10 phr.

It can be noted here from the results shown in Figures 36-39 that when the amount of water hyacinth was increased, fractured surface of NR vulcanizates could become more irregular.

Figure 40 shows the SEM micrographs of the NR foams filled with 10 phr of water hyacinth and 4 phr of blowing agent at foaming temperatures of 170°C (100× magnification). It can be demonstrated that the morphology of the cells structure is affected by the presence of the water hyacinth fibers. It is tempting to speculate that when the NRs foam are vulcanized, blowing agent generates gas and gas cells' expansion may be anisotropic because of the incorporation of water hyacinth fibers to the rubber matrix. However, for NR foams without water hyacinth fiber containing different blowing agent content, the gas liberate anisotropically in NR foam because of the homogenous matrix as seen in Figures 33-35, which is basically the same as the foaming mechanism of plastic foams and sponges.

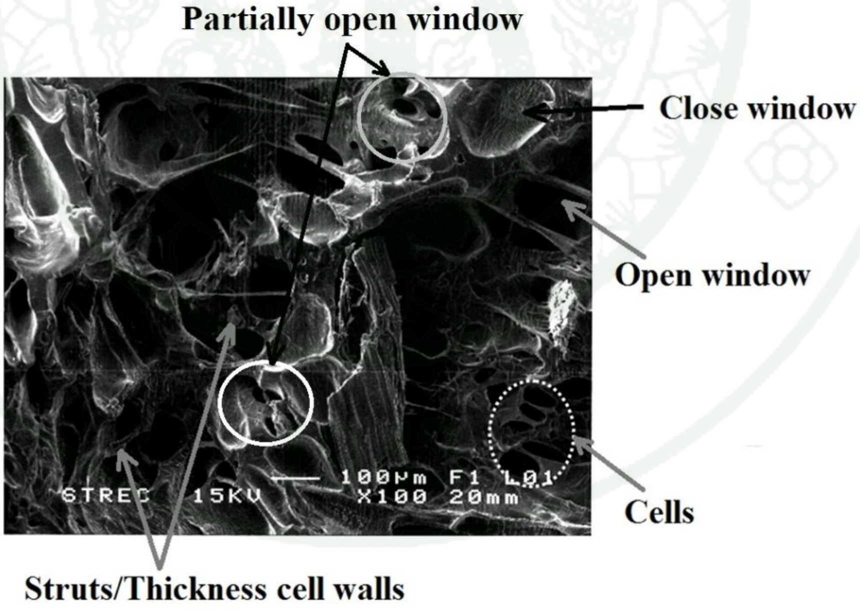


Figure 40 SEM micrographs of the NR foams filled with 10 phr of water hyacinth and 4 phr of blowing agent at foaming temperatures of 170°C (100× magnification).

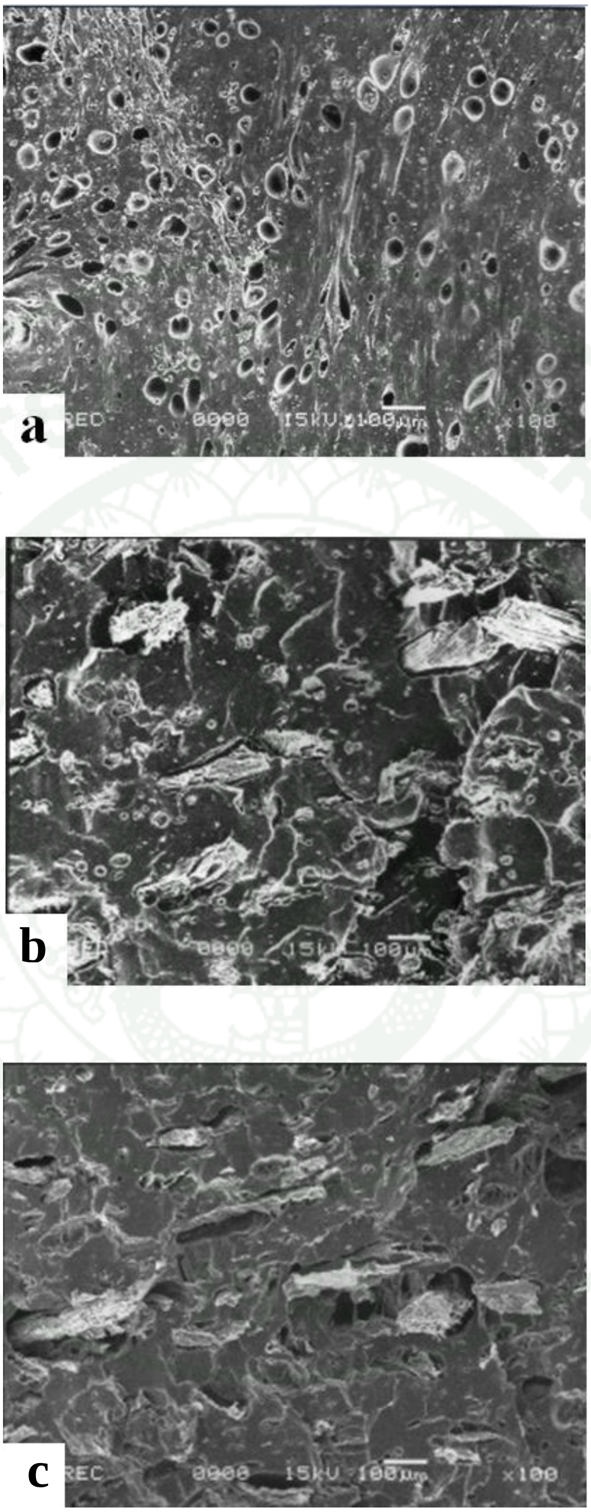


Figure 41 SEM micrographs of the NR foams at foaming temperatures of 150°C filled with 2 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

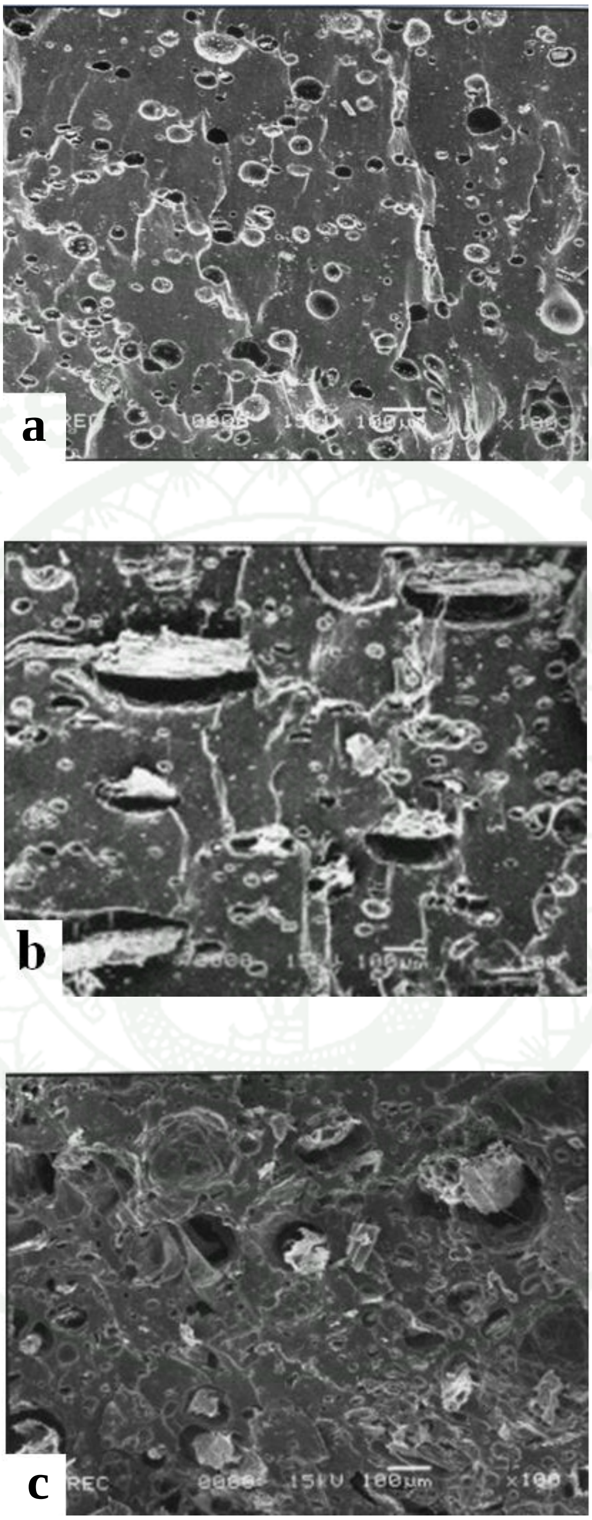


Figure 42 SEM micrographs of the NR foams at foaming temperatures of 160°C filled with 2 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

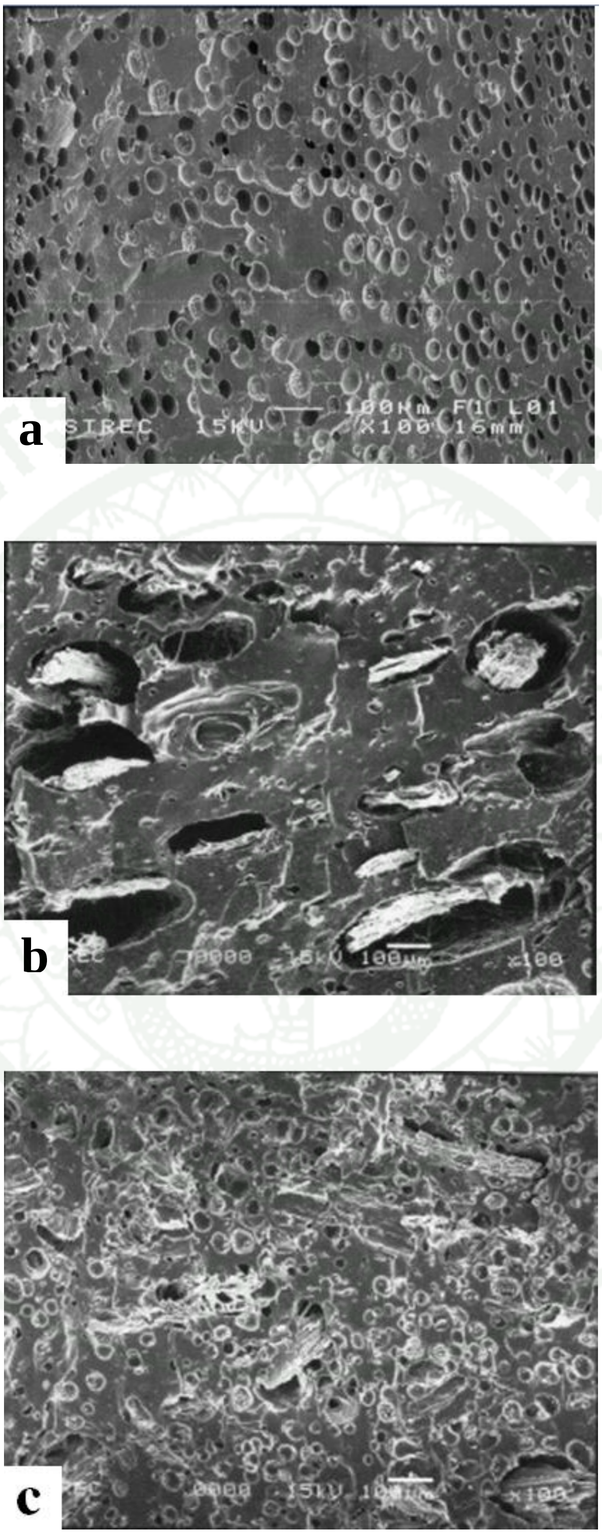


Figure 43 SEM micrographs of the NR foams at foaming temperatures of 170°C filled with 2 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

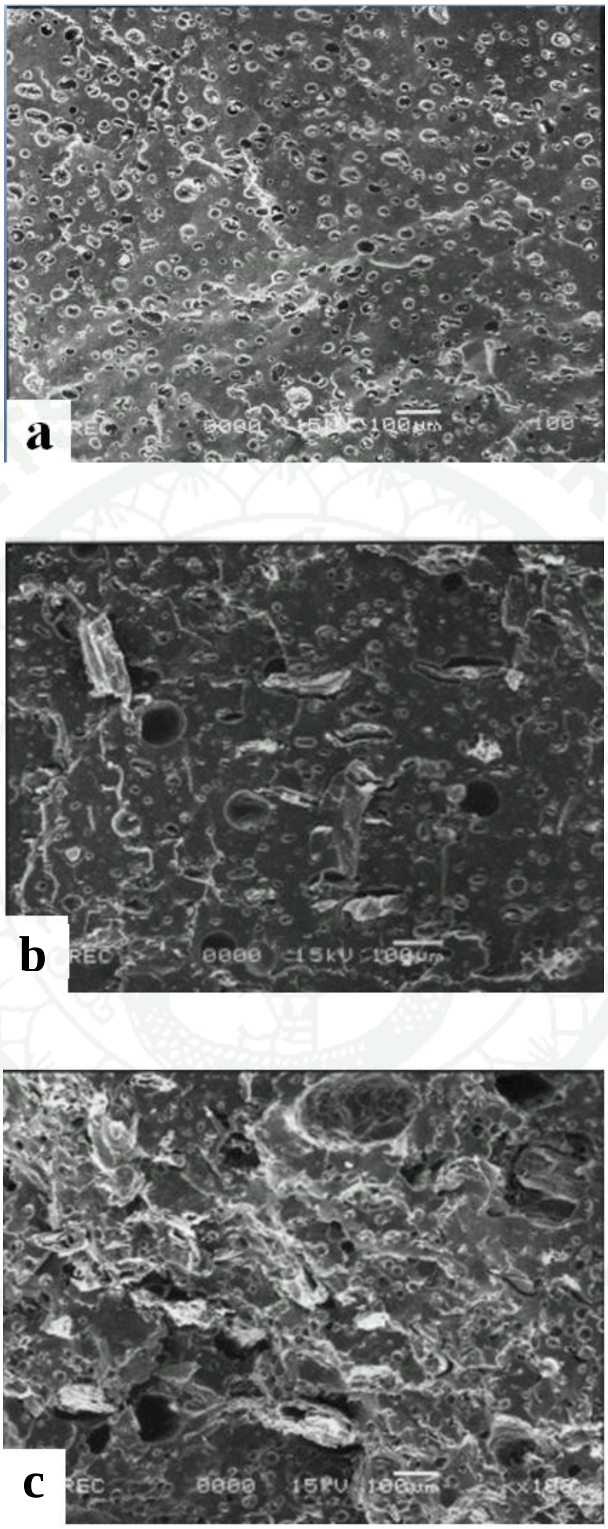


Figure 44 SEM micrographs of the NR foams at foaming temperatures of 150°C filled with 4 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

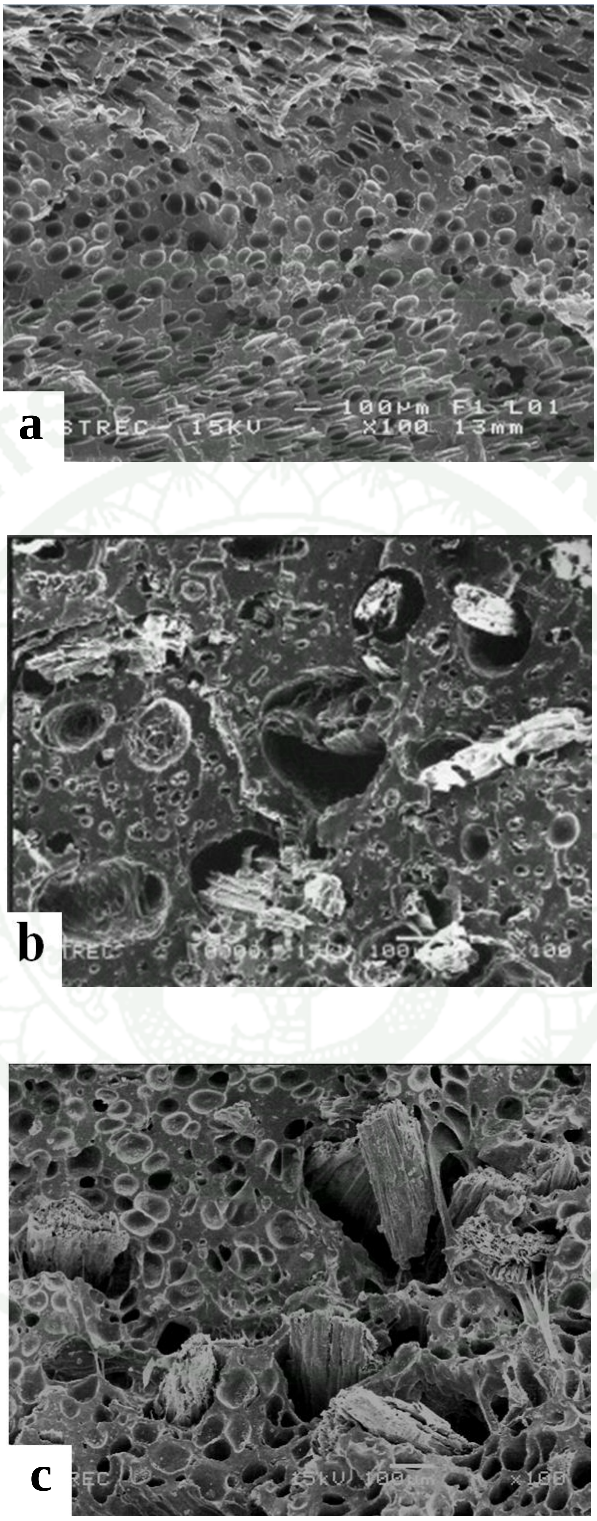


Figure 45 SEM micrographs of the NR foams at foaming temperatures of 160°C filled with 4 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

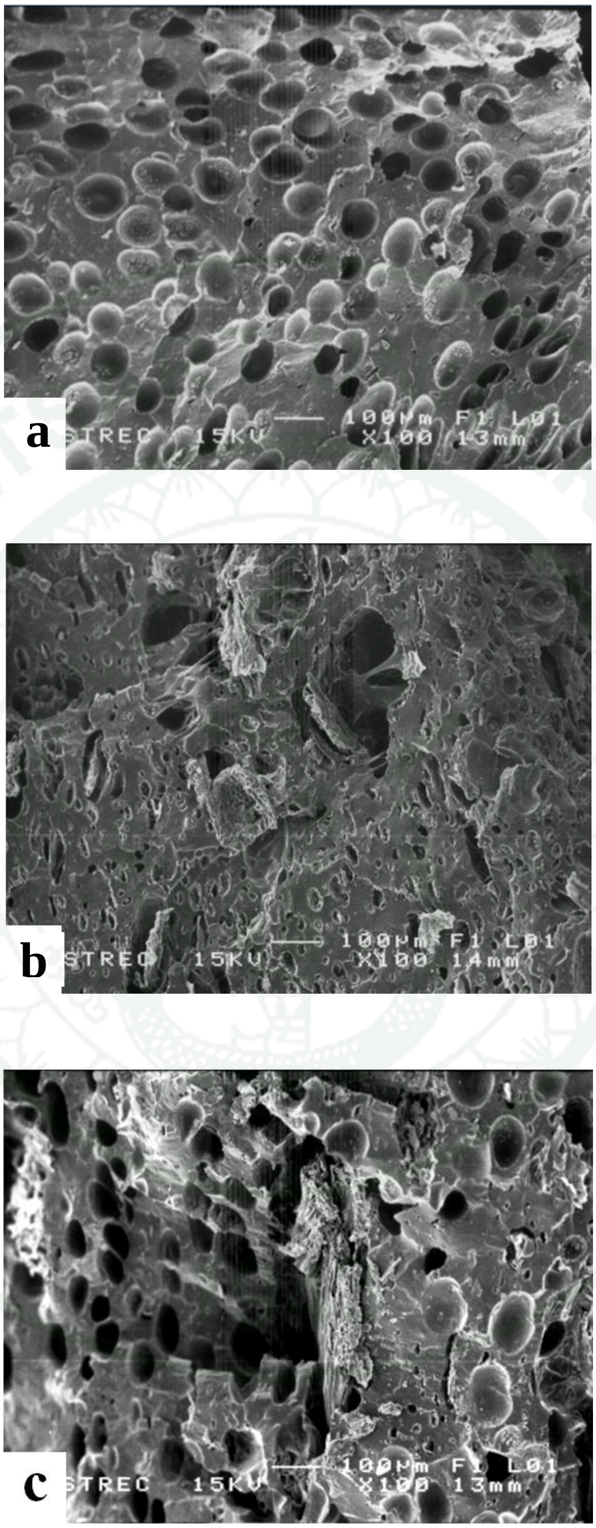


Figure 46 SEM micrographs of the NR foams at foaming temperatures of 170°C filled with 4 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

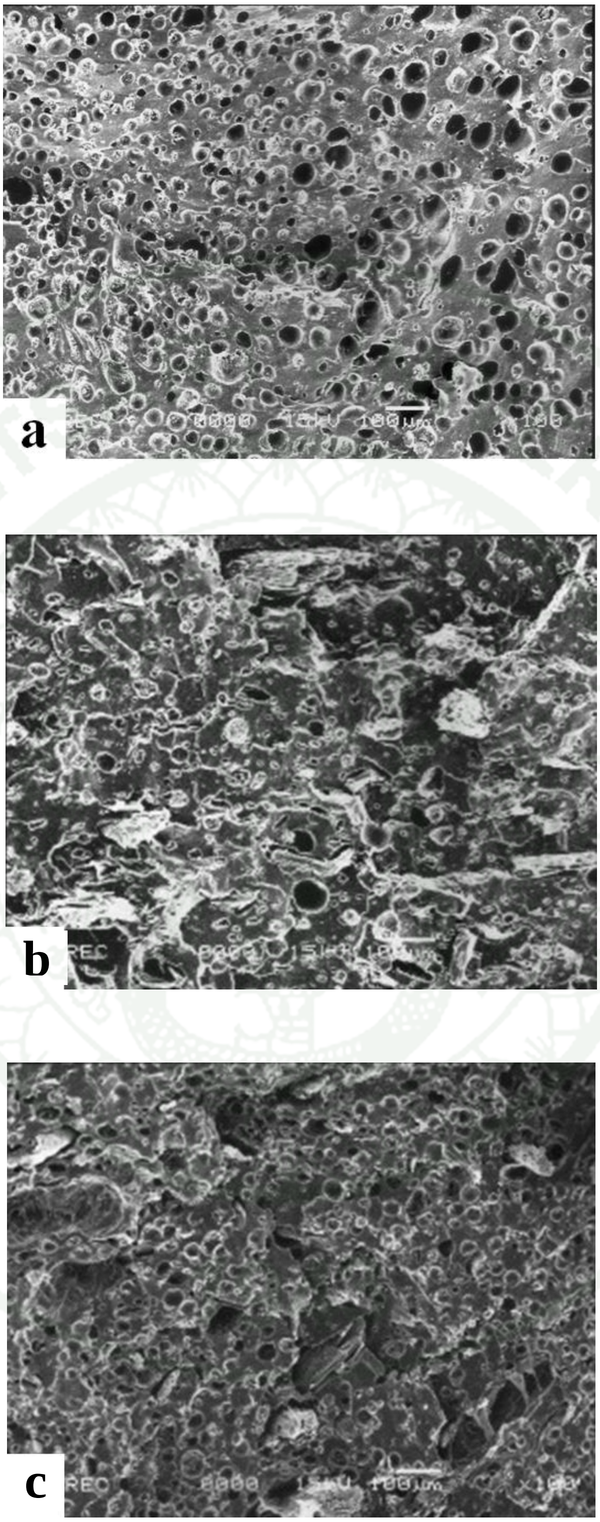


Figure 47 SEM micrographs of the NR foams at foaming temperatures of 150°C filled with 6 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

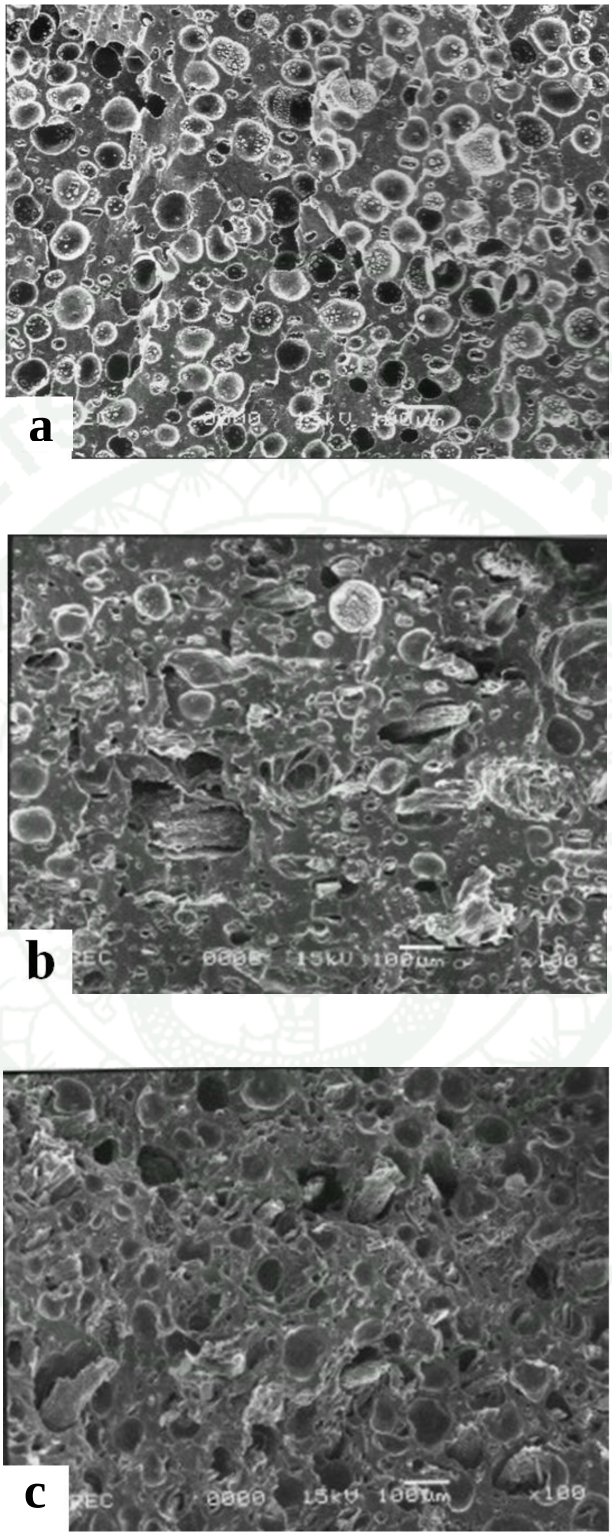


Figure 48 SEM micrographs of the NR foams at foaming temperatures of 160°C filled with 6 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

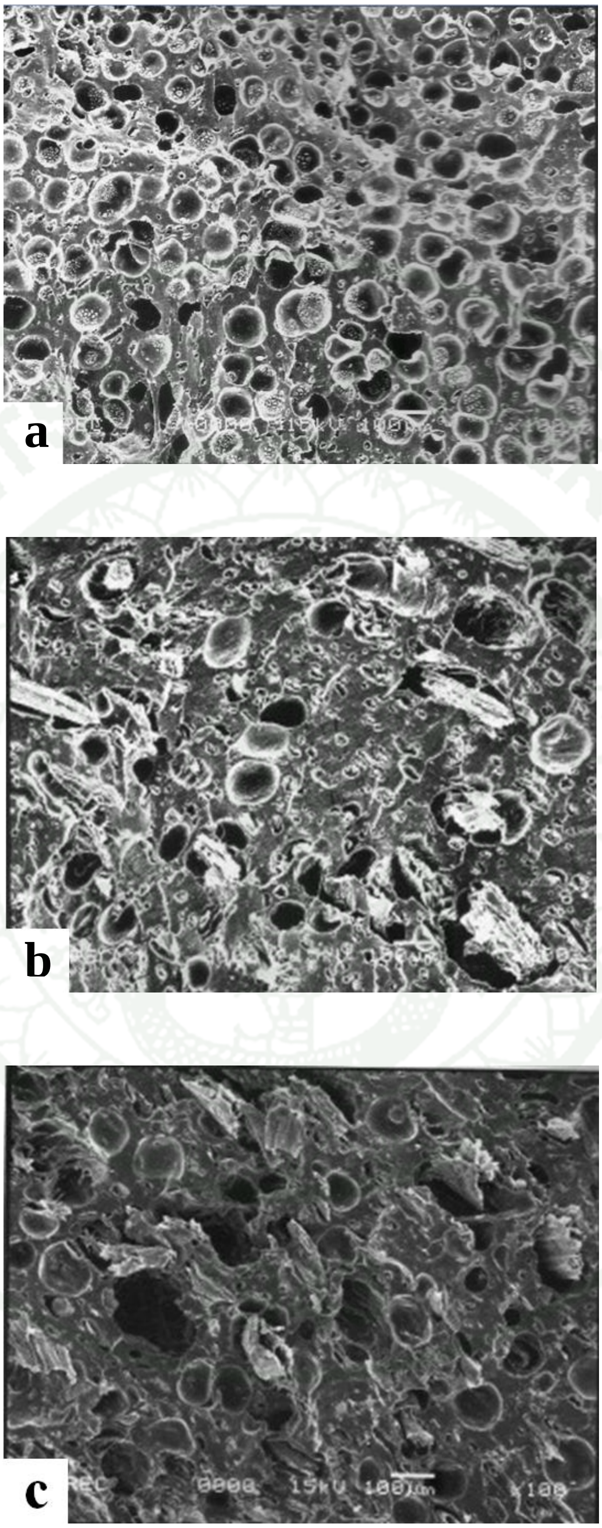


Figure 49 SEM micrographs of the NR foams at foaming temperatures of 170°C filled with 6 phr of blowing agent and various water hyacinth content: (a) 0 phr, (b) 5 phr, and (c) 10 phr.

SEM microscopy studies were performed for the NRs foamed at various water hyacinth contents as seen in Figures 37-39 and Figures 41-49. SEM micrograph of the NR foam samples at three different foaming temperatures (150, 160, and 170°C) depicts the influence of water hyacinth fiber on the foam formation. The cell size and shapes of NR foam becomes more or less irregular and it can be said that with the addition of water hyacinth fiber, the structure of the foam gets poorer. The water hyacinth fibers are detached into singles, distributing evenly in the NR foams; most of the water hyacinth fibers are in bidimensional orientation because of the process molding technique. Later, the topologies of the crossed-section planes show that the water hyacinth fibers are surrounded with gas cells. Besides, Figures 47(c)-49(c) also suggests that almost all gas cells distribute uniformly in the NR foams filled with 10 phr of water hyacinth and 6 phr of blowing agent at different foaming temperatures. Moreover, the thickness of cell walls is very uniform. All these illustrate that the curing characteristic of vulcanizates well matches the decomposition characteristic of the blowing agent. The SEM results illustrated in Figures 41-49 obviously show that the thickness of cells wall formed inside the rubber matrix increased upon increasing the water hyacinth content. With the increase of water hyacinth, the rubber matrix did not have a sufficient volume fraction for foaming, resulting in the formation of thickness of cells wall. This subsequently resulted from amount of gas presence in foam cell. The gas generated by blowing agent will promote the cell walls to expand further and eventually these walls will coalesce or may even rupture to form bigger cell. It is clear from the SEM micrographs that the NRs foamed at 170°C had a higher foaming efficiency. Again, the results of the expansion ratio and SEM micrographs of the foamed NRs supported the density characteristics which are tabulated in Table 5.

Figures 29(a)-31(a) reveal quantitatively that the average cell size of NR foam decreased with increasing water hyacinth content, which is in agreement with qualitative findings of Figures 37-39 and Figures 41-49. Moreover, from Figure 28 and Figures 29(b)-31(b), It can be seen that the relative foam density and crosslink density slightly decreased with increasing water hyacinth contents. We can explain such behavior in the following way. It is common that the relative foam density decreases with the increasing filler content because of the lower density of filler (water hyacinth) as depicted in the Table 5. Moreover it would be expected that similar crosslink densities should be obtained for all the samples because the same amount of blowing agent was used.

3.3 Effect of blowing agent loading

Foamed polymers can be produced by utilizing either one kind of chemical or physical blowing agent. The most popular chemical blowing agent, OBSH or 4,4’oxybis (benzenesulfonyl hydrazide), was of interest in this work. Liu (1998) studied the effect of blowing agent contents and processing conditions on the characteristics of linear low-density polyethylene (LLDPE) foam. They found that the decomposition rate of the blowing agent was the critical parameter for selecting the optimum blowing agent. Their findings also suggested that OBSH was the most suitable blowing agent for LLDPE. Fine cell morphology in the composite foam samples could be obtained as a result of good dispersion of active blowing agent in the matrix (Bledzki *et al*., 2006 and Shenton *et al*., 2002).

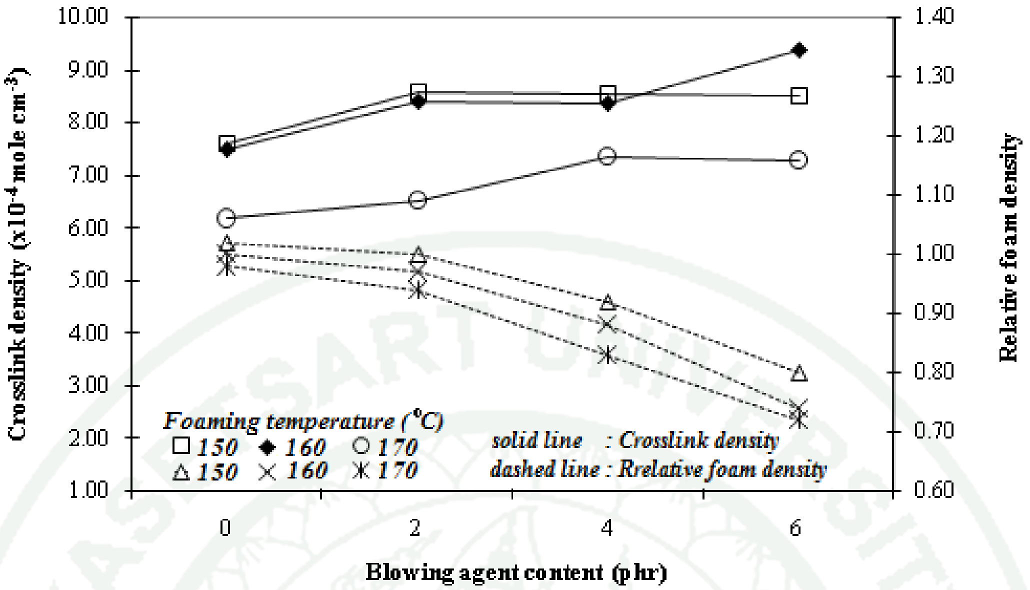


Figure 50 The effect of blowing agent content on relative foam density and crosslink density of NR foam without water hyacinth at different foaming temperature.

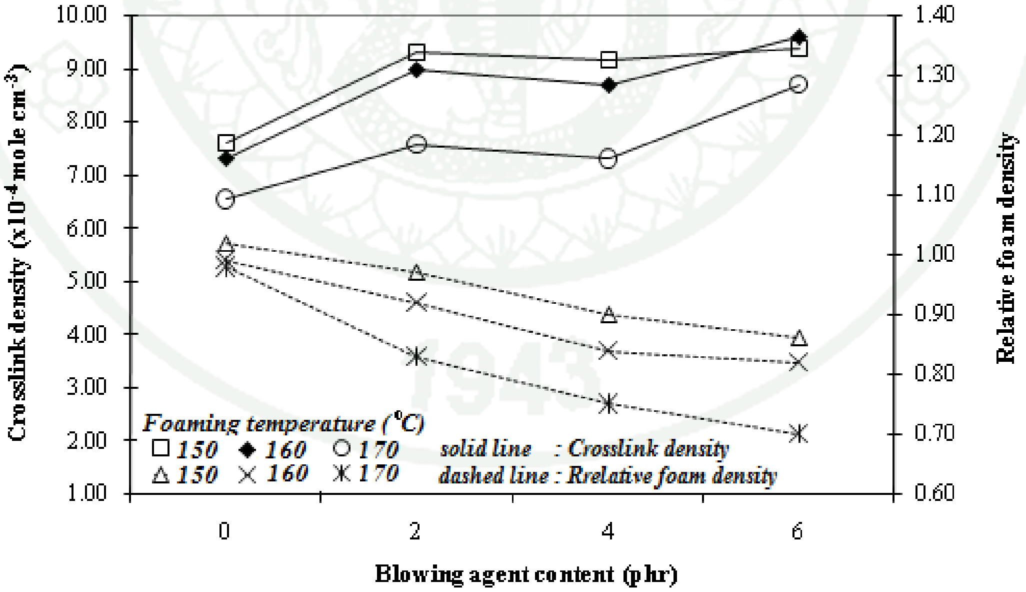


Figure 51 The effect of blowing agent content on relative foam density and crosslink density of NR foam filled with 5 phr of water hyacinth at different foaming temperature.

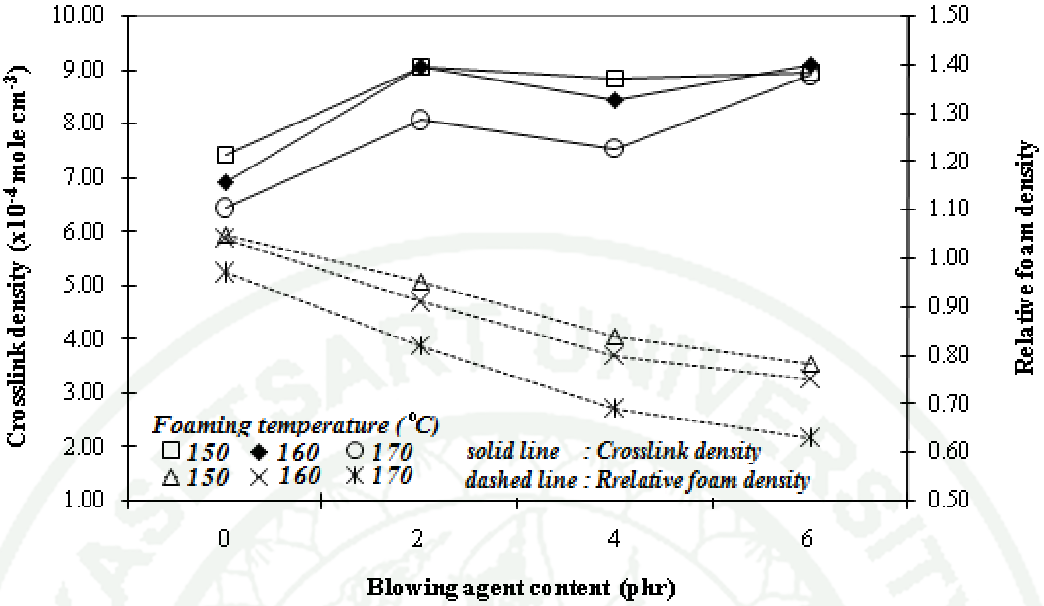


Figure 52 The effect of blowing agent content on relative foam density and crosslink density of NR foam filled with 10 phr of water hyacinth at different foaming temperature.

In this study, cell morphology of NR foam was considered in terms of average cell size which can be used to represent the number of cellular structure and bulk density of the foam. Figures 50-52 illustrated the loading effect of the blowing agent on relative foam density and crosslink density of the NR foams at various water hyacinth contents. As greater content of blowing agent was used, more gas was subsequently generated, causing the reduction in the relative foam density. Zakaria (2007) reported that higher blowing agent concentrations shorten the growth time of the foam, thus restricting the gas from escaping through the foam surface, allowing the foam to expand more, and consequently, producing foam with a lower relative density. Furthermore the crosslink density increased with increasing blowing agent content. This is due to the fact that crosslinking and decomposition occur simultaneously; at high blowing agent contents, more nitrogen gas is present; thus, the gas phase will be more prominent than the solid phase. Hence, thinner cell walls are formed, and consequently, more crosslinking occurs. It would be expected that trend to increase crosslink densities would be obtained for all the samples because OBSH was not use only as the blowing agent but also as a crosslinking agent (Klempner and frisch *et al*., 1991). However, the OBSH used in this study decomposed exothermically; this may result in crosslinking excess as the blowing agent content increases. At high contents of OBSH, more heat was absorbed from the system, hence, interrupting the crosslinking process (Guriya, 1996). Furthermore, Sombatsompop and Lertkamolsin, (2000) suggested in his study that 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. There are various factors influencing the foam morphology such as temperature, pressure, type of polymeric materials and the formulation used. The effect of chemical blowing agent introduced will definitely effect the cell distribution and the cell size depending on the type of blowing agent used, loading amount and the blowing agent particle size (Klempner and frisch *et al*., 1991).

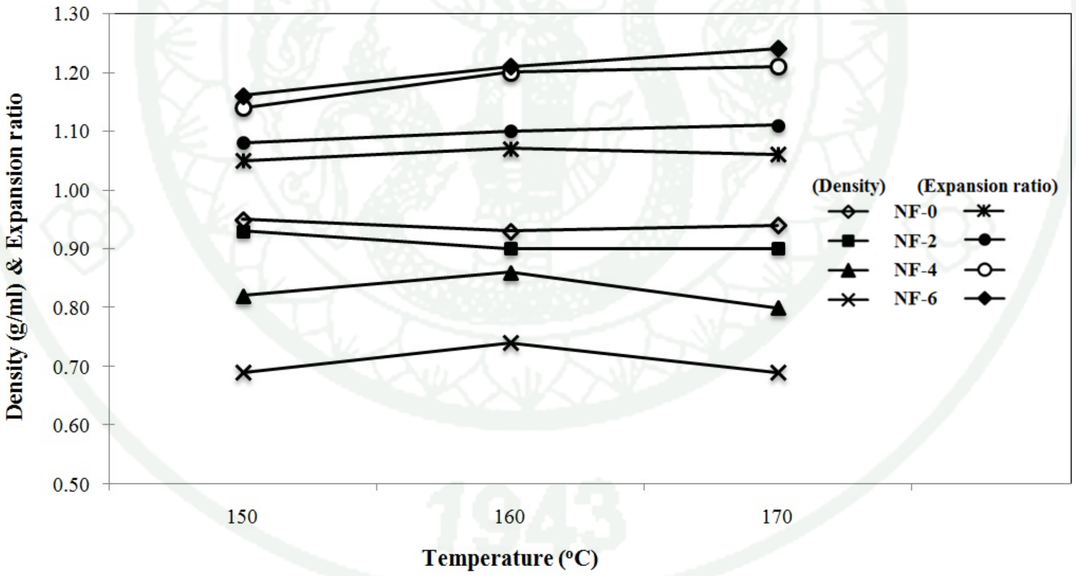


Figure 53 Variation of the density and expansion ratio of the foamed NR without water hyacinth as a function of the foaming temperature and various blowing agent content.

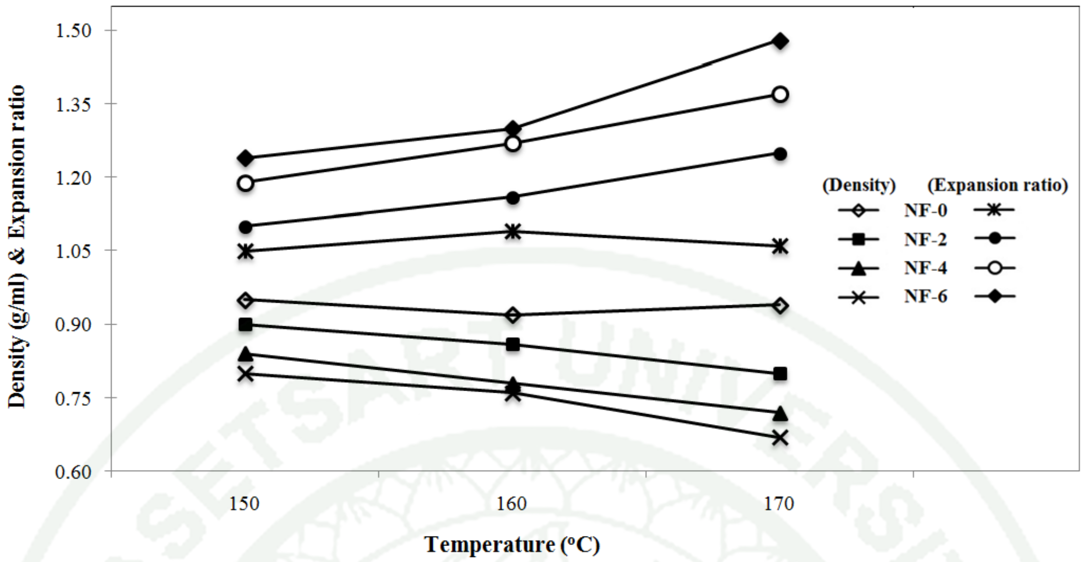


Figure 54 Variation of the density and expansion ratio of the foamed NR filled with 5 phr of water hyacinth as a function of the foaming temperature and various blowing agent content.

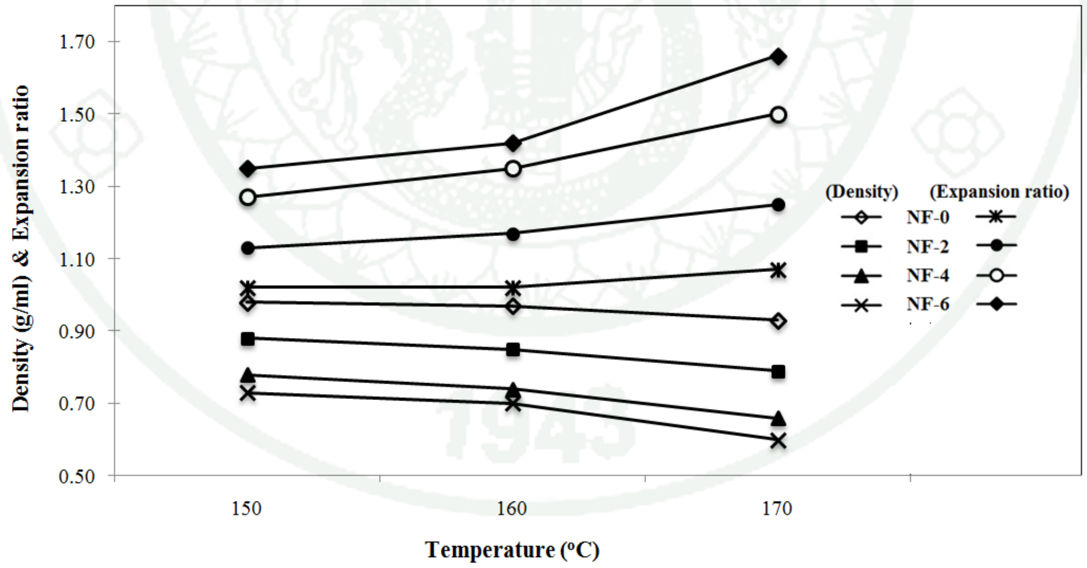


Figure 55 Variation of the density and expansion ratio of the foamed NR filled with 10 phr of water hyacinth as a function of the foaming temperature and various blowing agent content.

From these previously, the effect of blowing agent content on average cell size can be calculated and is tabulated in Table 6. The results reveal that the average cell size increased with increasing blowing agent content. The SEM micrograph analysis (Figures 41-49) shows that there is a systematic correlation between the various water hyacinth and average cell size of NR foam at different foaming temperature. Consequently, an increase in the blowing agent content resulted in bigger, and more uniform cells. The decomposition of high amount of nitrogen gas occurs simultaneously for a given time; thus, more cells formed at that difference time. The cause can be related to reduction in matrix viscosity due to partially complete crosslinking and consequently produced larger cells with increasing amount of blowing agent. Increasing cell size and thinning effect of the cell wall will directly alter the foam density as well as foam expansion ratio. Relatively, the foam density is reduced and the ratio of foam expansion will also increase as shown in Figures 53-55. On the other hand, the foam relative density is highly influenced by the chemical crosslinking generated among the polymer chain. The crosslinking will form a network consisting of higher packing polymer chain and results in increasing relative foam density (refer Figures 50-52). The result shows that the amounts of cell increase when more open cells are produced. The data are strongly connected to Figure 40 which showed the foam morphology presenting more open cell existed as increasing amount of blowing agent.

4. Factors affecting the mechanical properties of NR foams

4.1 Effect of foaming temperature

As mentioned earlier in Figures 28-31, the relative foam density of NR foams progressively increased with a decrease in foaming temperature and it was proportional to crosslink density or degree of crosslink. Higher rigidities of the vulcanisates and greater restriction to expansion occurred at lower foaming temperature. It was supported by the results in Figures 41-49 where the cells produced at lower temperature were slightly smaller compared to the cells produced at higher temperature. This is because the vulcanisate was more rigid and difficult to expand. However, higher foaming temperature would give greater expansion due to the presence of higher gas volume which would result in decreasing relative foam density. This subsequently affects the mechanical properties of the foams where NR foams with lower densities tend to have lower compression deflection which was observed in Tables 7, it can be related to the recorded lower relative foam density (Table 6). The foam density is directly related to cell wall thickness and therefore, resistance to the cell wall bending and cell collapse increases with foam density which attributed to higher plateau stresses (Lin, 1996).

Table 7 Effect of foaming temperature on compression deflection of the NR foams with and without water hyacinth loading at various blowing agent content.

Water Hyacinth Content (phr)	Formula / Foaming Temp* (^oC)	Compression Deflection (kPa)		
		150	160	170
0	NF-0	532.00 ± 3.61	469.33 ± 11.59	383.67 ± 26.73
	NF-2	365.67 ± 32.13	309.00 ± 33.51	254.67 ± 2.08
	NF-4	207.67 ± 23.59	237.67 ± 4.16	159.00 ± 27.73
	NF-6	140.33 ± 11.59	73.33 ± 8.50	50.00 ± 2.00
5	NF-0	534.67 ± 26.31	383.00 ± 15.13	276.00 ± 22.72
	NF-2	241.33 ± 9.07	169.33 ± 10.97	124.67 ± 3.21
	NF-4	145.33 ± 12.22	128.00 ± 14.00	103.67 ± 9.24
	NF-6	131.67 ± 12.50	87.00 ± 38.04	41.67 ± 5.13
10	NF-0	550.67 ± 37.02	495.50 ± 40.31	368.00 ± 28.28
	NF-2	275.00 ± 15.13	226.67 ± 13.87	88.67 ±17.01
	NF-4	131.67 ± 8.33	92.00 ± 3.00	56.33 ± 3.79
	NF-6	86.33 ± 5.51	85.00 ± 28.58	30.00 ± 1.73

Note: NF-0, 2, 4, and 6; N refers to NR, F refers to foam and 0, 2, 4, and 6 refers to blowing agent (OBSH) content (0, 2, 4, 6, respectively), and Temp* refers to temperature.

Table 7 shows the effect of foaming temperature on compression deflection of the NR foams with and without water hyacinth loading at various blowing agent content in which compression deflection value of NR foam was considered in terms of compression stress-strain curve. It can be seen that the compression deflection of NR foams produced at 150°C was higher followed by 160°C and 170°C. This is due to the fact that at lower foaming temperature, the cell wall became thicker which subsequently provided greater resistance to buckling and collapse than at higher foaming temperature. Therefore the cell size, the crosslink density, the relative foam density and the cell wall thickness were different and become more dominant factor in progressive cell collapse. This indicates that irreversible damage has taken place, apparently caused by rupture of the cell wall and fracture of the cell edge (Shen *et al*., 2001).

4.2 Effect of water hyacinth loading

The effects of water hyacinth content on the mechanical properties of the foamed NRs are shown in Figures 56-59. In this study the behavior of NR foams containing 5 and 10 phr of untreated water hyacinth fibers was analyzed. It was found from Figure 56, showing the influence of water hyacinth content on the compression deflection of vulcanized NR (without blowing agent) that the compression deflection showed little effect as the water hyacinth content increased.

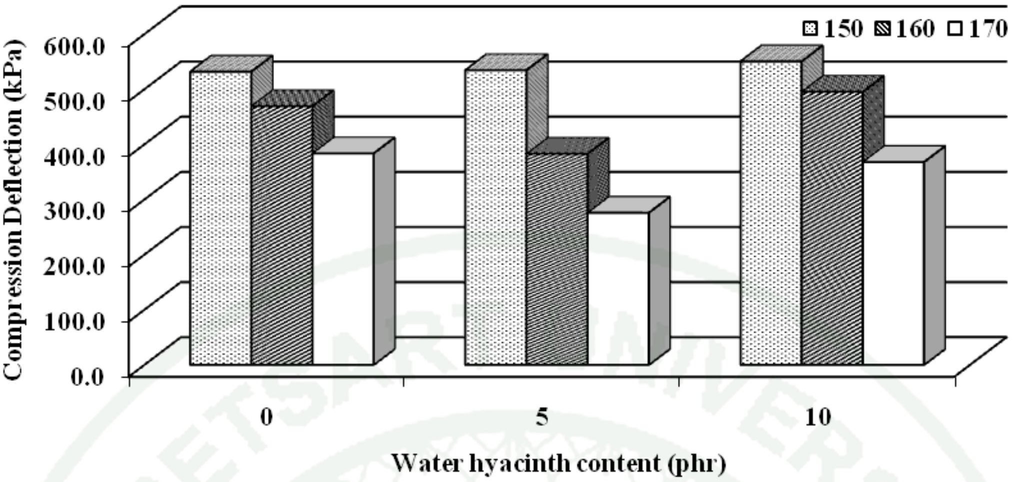


Figure 56 The effect of water hyacinth content on compression deflection of the NR vulcanizates without blowing agent at different foaming temperature.

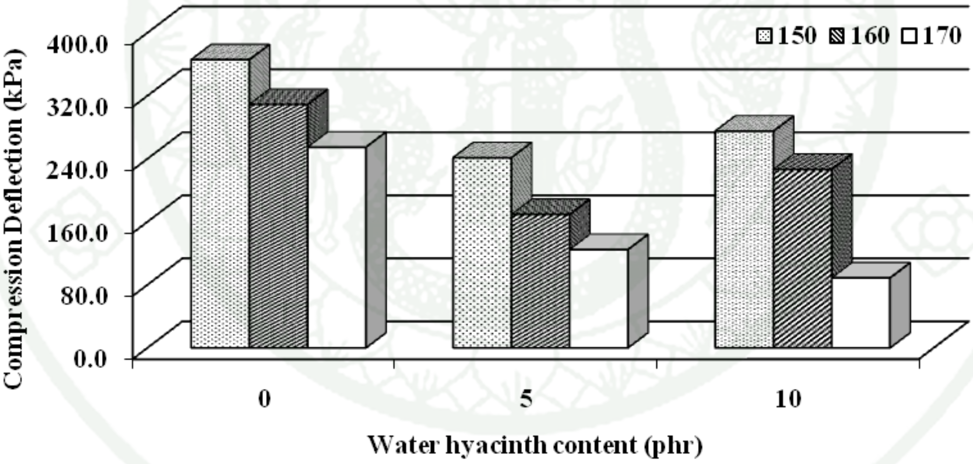


Figure 57 The effect of water hyacinth content on compression deflection of the NR foams containing 2 phr of blowing agent at different foaming temperature.

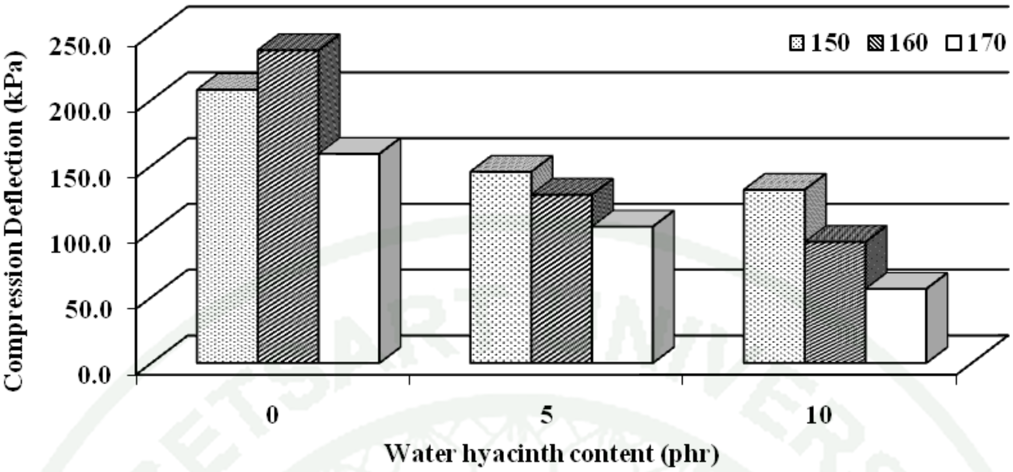


Figure 58 The effect of water hyacinth content on compression deflection of the NR foams containing 4 phr of blowing agent at different foaming temperature.

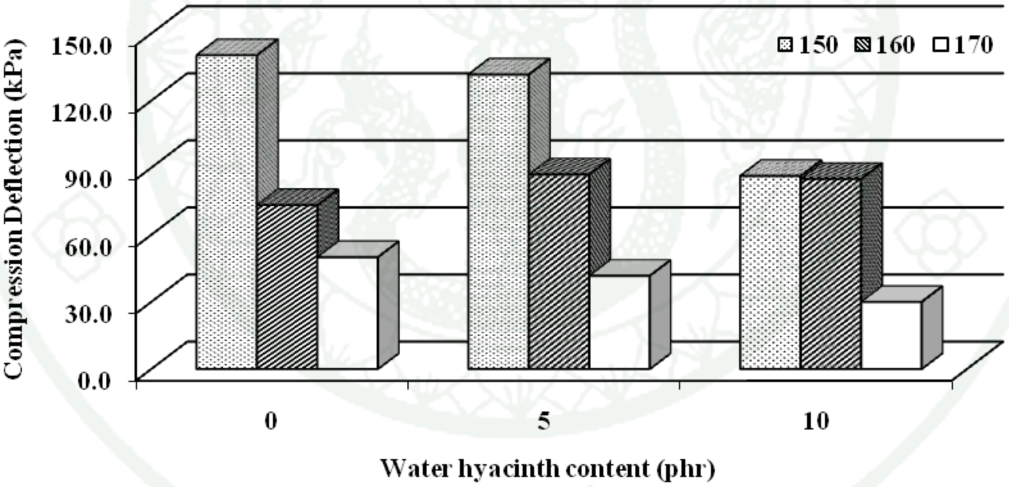


Figure 59 The effect of water hyacinth content on compression deflection of the NR foams containing 6 phr of blowing agent at different foaming temperature.

The nature of the deformation of the NR foams under an applied load can be understood from the stress-strain curves. It can be seen that NR foams can accommodate more stress, while exhibiting lower compression deflection with the increasing water hyacinth content. The compression deflection dropped continuously with the addition water hyacinth content as compared with increasing foaming temperature in NR foams as seen in Figures 57-59. This trend may be attributed to the partially water hyacinth fiber-rubber interface upon increasing content of water hyacinth. This is a similar case as increasing particulate filler in the vulcanized as seen in Figure 56. It was found that the increment in filler content resulted in a reduction of the deformability of a partially interface between the filler and the rubber matrix. There is, however, a different phenomenon exerted by the foaming temperature. This situation is also supported by the expansion ratios, which can be closely related with the decomposition temperature of the blowing agent. Moreover, the decreases in the compression deflection of the NR foams by the addition of water hyacinth content were attributed to three possible reasons which include; (a) an increase of interfacial defects or debonding between NR foams and water hyacinth phases due to the use of untreated water hyacinth fibers, (b) a poor dispersion or an agglomerate of the water hyacinth fibers throughout the NR foam due to the incompatibility between strongly polarized water hyacinth fibers and hydrophobic NR foams, and (c) the presence of voids or cells of NR foams–water hyacinth fiber interface, this resulting from different thermal contractions of these two incompatible materials. The two latter reasons can be substantiated using SEM micrographs as shown in Figures 41-49, which depicted the agglomerate and insertion of the water hyacinth fibers and large voids around the NR foams.

Fiber reinforced rubber composites are of tremendous importance both in end-use applications and the area of research and development. These composites exhibit the combined behavior of the soft, elastic rubber matrix and the stiff, strong fibrous reinforcement. The development of fiber reinforced rubber composites has made available polymers that are harder than aluminum and stiffer than steel. Generally short fiber reinforced rubber composites has become popular in industrial fields because of the processing advantages and increase in strength, stiffness, modulus and damping (Goettler and Shen, 1983; Setua and De, 1984). The design of a short fiber reinforced rubber composite depends on several factors such as the aspect ratio of the fiber, control of fiber orientation and dispersion and existence of a strong interface between fiber and rubber.

Cellulosic fibers are derived from many renewable resources and have many desirable properties for reinforcement of thermoplastics such as low density, high stiffness and low cost (Maya *et al*., 2004). Various natural fibers have been used as reinforcement in NR viz. Recently studies relating to the incorporation of bamboo fiber in natural rubber have been investigated by Ismail *et al*. (2002). In 2001, Iannace *et al*. discovered that sisal fiber is one of the strongest fibers, which can be used for several applications. Oil palm empty fruit bunch (OPEFB) and oil palm mesocarp fibers are two important types of fibrous materials left in the palm oil mill. Oil palm fibers are hard and tough and have found to be a potential reinforcement in phenol-formaldehyde resin (Sreekala et al., 1997).

4.3 Effect of blowing agent loading

As discuss earlier in section 3, it was expected that the morphological and physical properties including cell size, cell structure and cell distribution of NR foams will directly or indirectly affect the mechanical properties. As for compression deflection, it is determined that by adding more blowing agent (OBSH), less value of compression deflection are obtained as illustrated in Figures 60-62.

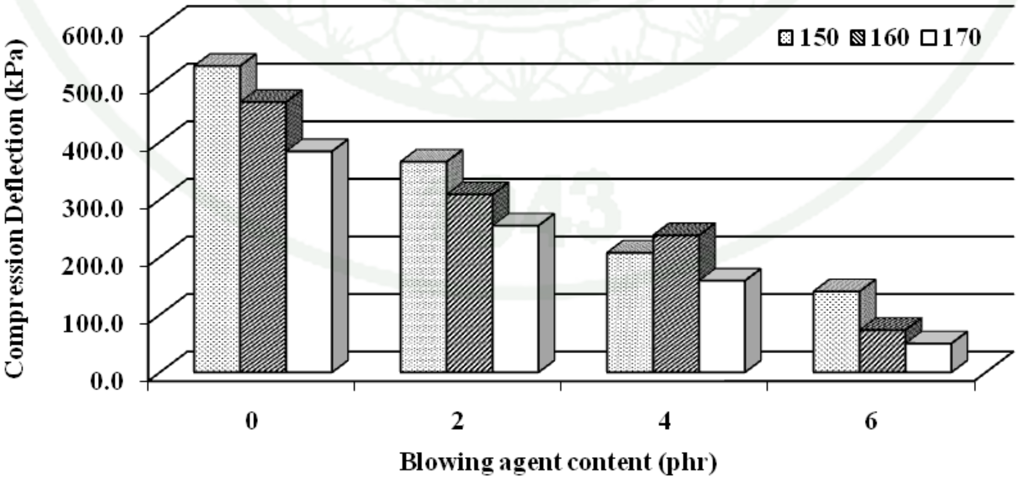


Figure 60 The effect of blowing agent content on compression deflection of the NR foams without water hyacinth at different foaming temperature.

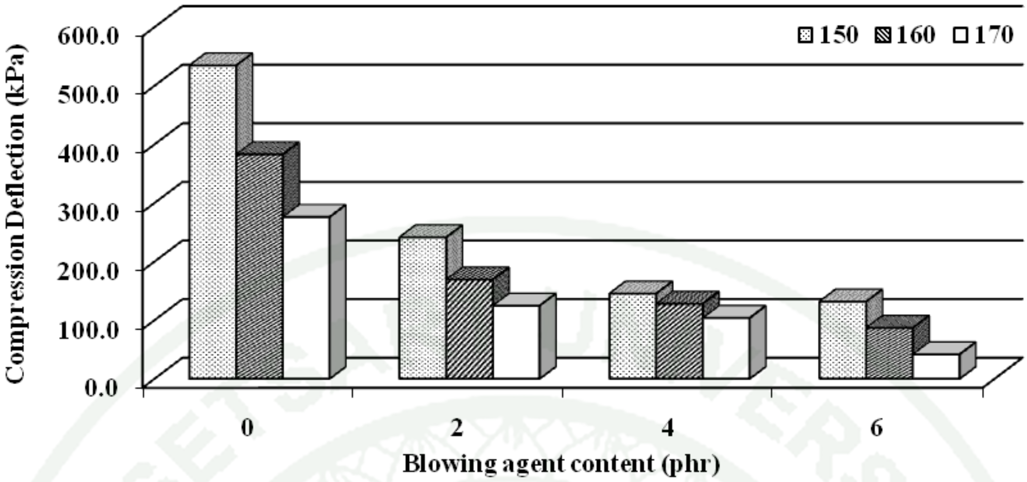


Figure 61 The effect of blowing agent content on compression deflection of the NR foams filled with 5 phr of water hyacinth at different foaming temperature.

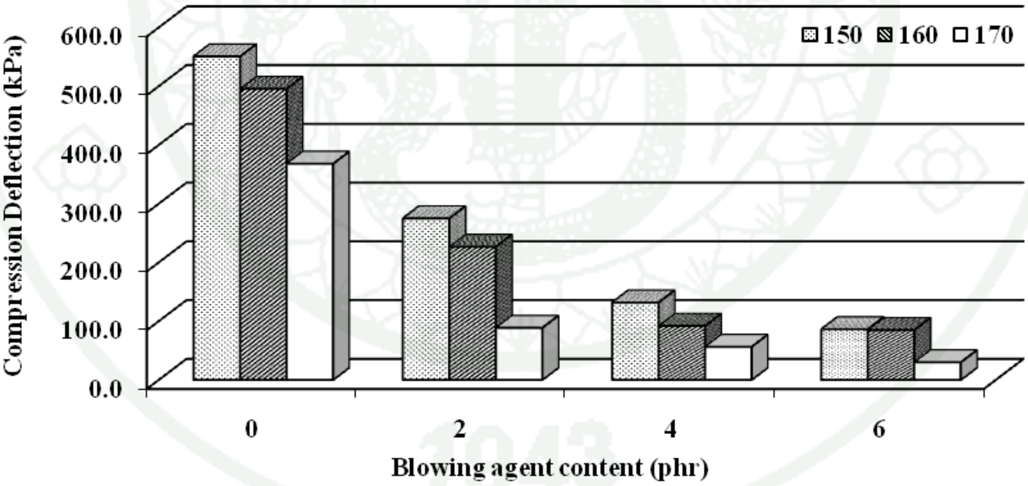


Figure 62 The effect of blowing agent content on compression deflection of the NR foams filled with 10 phr of water hyacinth at different foaming temperature.

Generally, as the cell morphology changes, the foam will also give different value of compression stress. It is discovered that the compression stress is dropped drastically with increasing content of blowing agent because the rubber matrix phase to resist the compression deflection had been reduced or replaced by the presence of gas phases as shown in Table 7. Bigger cell size and thinner cell wall will reduce the resistant towards bending. As a consequence, it is observed that NR foam produced with 6 phr (NR-6) of blowing agent displayed the lowest compression deflection value followed by 4 phr and 2 phr as shown in Figures 60-62, respectively. As shown in Figure 62, the compression deflection of the NR foams containing 10 phr of water hyacinth foamed at temperature of 170°C decreased from 368.00 to 30.00 kPa as the amount of blowing agent increased from 0 to 6 phr, respectively. From the results of compression deflection, it is suggested that the compression deflection of the NR foams decreased with the increase of the blowing agent content. This is because that the average cell size of the NR foams increased with the increase of the blowing agent, therefore, the density of the NR foams decreased. It is generally known that the mechanical properties of a cellular material mainly depend on its density and expansion ratio of NR foams as shown in Figures 53-55. The results of the density measurement demonstrated that the relative foam density of the NR foams decreases with the increase of blowing agent contents and foaming temperature which are shown in Figures 50-52.

This statement was substantiated by ASTM D 1056; standard specification for flexible cellular materials-sponge or expanded rubber of the NR foams. It can be seen that the NR foam exhibited lower compression deflection with the increasing blowing agent and water hyacinth content at higher foaming temperture. It can be noted here that cellular NR filled with 10 phr of water hyacinth and 6 phr of blowing agent foamed at 170°C exhibits a better behavior which it can be closely related to the cellular rubber made from NR or rubber-like materials alone or in combination. That is to say NR foams at this particular condition meet specific requirements for medium mass showing the compression deflection values of approximately 30.84 kPa (reference). In this study, the highest blowing efficiency, the lowest foam density for the foamed NRs at these conditions was achieved at 170°C. This could be explained by the cell structures as depicted in Figures 41-49. This was because the cell size had reached equilibrium, this being known as the gas containment limit (Thomas *et al*., 1997), where the cell walls made contacts to each other during their expansion. It is also indicated that at 6 phr of blowing agent, there were a loss of N_2 gas by diffusing through the cell wall as a result of the gas containment limit. At these excess loadings (9-11 phr), there may be a loss of N_2 gas either by diffusing through the cell wall or by destroying the cell structure (Li and Matuana, 2003). Besides, the increase in cell size which is parallel to the foaming direction will cause longer distance for the upper and lower cell wall to be in contact resulting higher maximum strain. A sudden drastic change in the compression stress value for all the foam is caused by the effect of compression stress on the matrix phase (Gibson *et al*., 1987). Also in this study for compression deflection, foams with high blowing efficiency were seen to be inferior to those with low blowing efficiency.

The morphological and mechanical properties of cellular rubbers are known to be affected by the type and content of blowing agents used. Sombatsompop and Lertkamolsin (2000) investigated the effects of additions of oxybis (benzene sulfonyl) hydrazide (OBSH) and azo dicarbonamide (ADC) blowing agents on density, degree of crosslinking, and swelling behaviour in cellular natural rubber (NR) vulcanizates. The rubber swelling greatly changed with OBSH, but unaffected by ADC content. Similar behavior was also observed for synthetic rubbers by Liu (1998); Bledzki and Faruk (2006). In rubber composites, Kim *et al*., (2007) discovered that the mechanical properties of the foam vulcanizates were dependent on the type, and concentration of filler, fiber orientation, and adhesion between the fiber (or filler) and the rubber matrix. Lin *et al*. (2004) studied microstructure and mechanical properties of nylon 6,6-fiber/NR foam composites with and without surface treatment of the fiber. The results suggested that the addition of nylon-fibers improved the overall mechanical properties of the rubber foams. The mechanical properties of the rubber composite foams were strongly dependent on the density of the microcellular foam, fiber orientation. The relationship between the foam density and mechanical properties was also studied by Kim *et al*. (2007) who examined the influences of foaming temperature and carbon black (CB) content on cure behavior and mechanical properties for the NR foams. They found that the densities and tensile strength of the NR foam decreased with increasing curing temperature. Increasing

CB content increased the tensile modulus, tensile strength and tear strength, but decreased the elongation at break.

5. Factors affecting the acoustic properties of NR foams

Acoustic absorbing foam materials are produced from dry natural rubber (NR) with the addition of 4,4'oxybis(benzenesulfonylhydrazide) (OBSH) as a blowing agent. The ability of material to absorb sound can be measured using the sound absorption coefficient (α). In this study, the sound absorption of the NR foams was measured under two different ranges of frequency:

(1) low-frequency (125, 250, 500, and 1,000 Hz) and
(2) high-frequency (2000 and 4000 Hz).

Acoustic determinations of sound absorption coefficient in impedance tube with two different sizes of specimens in form of dish: diameters of 29 mm and 100 mm were used at two different frequency ranges. In this experiment, all cylindrical samples were cut off each formulation foams with thickness of 13 mm.

The objective of this research was carried out to study the potential use of water hyacinth fiber in replacing synthetic-based fibers for sound absorption applications. This part investigated the sound absorption coefficient of natural rubber foam with and without water hyacinth content at various blowing agent contents of 0, 2, 4, and 6 phr at different foaming temperature in order to understand their acoustic characteristics. The main critical parameters in the determination of the acoustic properties are the sound absorption coefficient. These properties are related to the changes in the physical properties of NR foams, including the relative density, morphology, crosslink density and average cell size.

5.1 Effect of foaming temperature

Table 8 shows the effect of foaming temperature on acoustical properties of the NR foams with and without water hyacinth at various blowing agent content. Acoustical properties can be reported as the sound absorption coefficients (α) as well as the values of noise reduction coefficient (NRC). However, indicating the sound properties of materials using the α-values at different frequency ranges would be complex as it involved calculations over several frequencies. To solve this problem, the ability of material to absorb sound can be indicated using one single value so called the noise reduction coefficient (NRC). The NRC can be calculated using the following equation (Thuman and Miller, 1986):

$$\text{NRC} = \frac{(\alpha_{250} + \alpha_{500} + \alpha_{1000} + \alpha_{2000})}{4}$$

As presented in Table 8, it can be seen that the NR foams at the highest foaming temperature (170°C) recorded the highest NRC. As the foaming temperature decreased, NRC tends to be lower and a lower value of the sound absorption coefficient, α can be observed. These distinct α-values indicate that the samples can be used as a sound-absorbing material within a certain frequency range. Overall, the NR foams have superior absorption capabilities at low frequency regions (1000 Hz) and at high frequency regions (4000 Hz). This observation is influenced by the structural properties of the foam, which are controlled by the cell size, relative density and crosslink density. The physical properties of the NR foam at different foaming temperatures were previously presented in Tables 6 and 7.

Results from previous data clearly indicated the relationship between these properties and foaming temperature in that a higher foaming temperature generates a higher pressure of nitrogen gases and thus stretches the cell wall the most. This expansion of the cell walls resulted in a relatively larger cell size and thus resulted in foam with a lower relative density. Hence, the amount of solid phase is reduced, and less crosslinking occurs. Variations in the foam morphology and average cell size are supported in Figures 41-49. The average cell size increases as the foaming temperature increased. The NR foam at higher foaming temperature exhibits a better cell-size homogeneity compared to the NR foams at lower foaming temperature.

Numerous types of sound absorption mechanisms have been previously reported. Sound is primarily dissipated due to viscous flow, thermal damping and the Helmholtz resonance effect. The efficiency of sound absorption is a compromise between a small amount of sound reflection and large amount of sound dissipation. When the sound wave strike the foam surface, the air constituents flow in and/or out of the cell due to pressure or disturbances produced by the sound. These frictional forces convert the sound energy into heat. The sound pressure will also force the cell wall to stretch, bend and buckle. These deformations occur when sound energy is converted into kinetic energy. The capability of the NR foam at higher foaming temperature to dissipate sound compared to the other two types of foam is primarily due to the conversion of more sound energy into dynamic energy. The amount of solid phase in the foam with smaller cells is more prominent, and the cell walls are thicker and stiffer and can thus withstand extra cell stretching, bending and buckling as sound energy strikes, which results in a lower amount of sound reflection. The sound absorption capability of the NR foam is dependent on the stiffness of the solid portion, i.e., it is controlled by the viscoelastic properties of the base material (Najib *et al.*, 2011).

For this study, the acoustical results of the NR foam at 170°C with the bigger average cell size is in agreement with the observations of Bonfiglio and Pompoli (2008). They suggested that the large cells allow for a considerable amount of sound wave dissipation via friction due to a significant increase in the air velocity if the air travels from a large area to small area. Therefore, there are two primary factors that affect the sound absorption capabilities of the NR foams. The first factor is the matrix properties with respect to the viscoelasticity; the foam with a larger viscoelastic property exhibits a larger sound absorption coefficient. The second factor is the cell size; a bigger cell size exhibits a larger sound absorption coefficient value.

Table 8 Effect of foaming temperature on acoustical properties of the NR foams with and without water hyacinth loading at various blowing agent content.

Water Hyacinth Content	Formula	*Temp	Absorption coefficient (α)						NRC
			125	250	500	1000	2000	4000	
0	NF-0	150	0.18	0.10	0.10	0.20	0.12	0.16	0.128
		160	0.05	0.10	0.19	0.22	0.21	0.16	0.180
		170	0.05	0.07	0.30	0.15	0.16	0.13	0.171
	NF-2	150	0.19	0.07	0.19	0.26	0.20	0.42	0.180
		160	0.03	0.15	0.20	0.29	0.19	0.50	0.207
		170	0.01	0.18	0.64	0.41	0.28	0.47	0.375
	NF-4	150	0.08	0.04	0.27	0.37	0.26	0.45	0.233
		160	0.24	0.16	0.21	0.38	0.27	0.51	0.256
		170	0.00	0.16	0.18	0.44	0.35	0.39	0.281
	NF-6	150	0.21	0.19	0.12	0.42	0.15	0.44	0.222
		160	0.11	0.10	0.25	0.39	0.33	0.44	0.268
		170	0.03	0.08	0.16	0.55	0.42	0.42	0.302
5	NF-0	150	0.00	0.06	0.08	0.17	0.12	0.11	0.106
		160	0.05	0.08	0.12	0.20	0.13	0.13	0.131
		170	0.08	0.09	0.15	0.19	0.23	0.18	0.168
	NF-2	150	0.05	0.08	0.15	0.29	0.22	0.47	0.188
		160	0.02	0.09	0.28	0.39	0.24	0.38	0.251
		170	0.18	0.12	0.37	0.41	0.24	0.49	0.286
	NF-4	150	0.01	0.12	0.15	0.31	0.23	0.46	0.200
		160	0.04	0.12	0.20	0.44	0.33	0.60	0.274
		170	0.11	0.13	0.51	0.69	0.34	0.39	0.416
	NF-6	150	0.10	0.12	0.14	0.60	0.26	0.47	0.279
		160	0.08	0.13	0.18	0.69	0.26	0.55	0.316
		170	0.07	0.03	0.18	0.68	0.46	0.40	0.339

Table 8 (Continued)

Water Hyacinth Content	Formula	*Temp	Absorption coefficient (α)						NRC
			125	250	500	1000	2000	4000	
10	NF-0	150	0.07	0.07	0.09	0.17	0.14	0.14	0.117
		160	0.07	0.03	0.07	0.31	0.11	0.16	0.132
		170	0.04	0.08	0.07	0.20	0.18	0.14	0.134
	NF-2	150	0.12	0.12	0.15	0.27	0.21	0.45	0.190
		160	0.03	0.10	0.16	0.64	0.41	0.40	0.329
		170	0.01	0.16	0.19	0.63	0.44	0.49	0.354
	NF-4	150	0.12	0.13	0.15	0.58	0.22	0.51	0.269
		160	0.11	0.17	0.15	0.67	0.28	0.54	0.319
		170	0.04	0.12	0.16	0.69	0.44	0.42	0.353
	NF-6	150	0.09	0.12	0.16	0.52	0.40	0.48	0.298
		160	0.05	0.12	0.15	0.71	0.32	0.49	0.327
		170	0.22	0.15	0.15	0.67	0.32	0.52	0.321

Note: NF-0, 2, 4, and 6; N refers to NR, F refers to foam and 0, 2, 4, and 6 refers to blowing agent (OBSH) content (0, 2, 4, 6, respectively), and Temp* refers to temperature.

5.2 Effect of water hyacinth loading

The effect of water hyacinth loading on the sound absorption properties of foamed NRs with various blowing agents performed at different foaming temperatures can be determined via one single value so called NRC as shown in Figures 63-66. Apparently, the NR foam seemed to have superior sound absorption properties to that of NR vulcanizates without blowing agent, although results were inconclusive at the lower frequency range. As seen in Table 8, it was found that the increment in the water hyacinth content resulted in higher NRC percentage and developed more efficient the overall NRC percentage of the NR foams. However, at high frequency of 4000 Hz, the ability to absorb sound of NR foam filled with water hyacinth at 5 and 10 phr, respectively was found to be much better than that of NR without water hyacinth as shown in Table 8.

The ability to absorb sound at both frequency ranges of NR foam are given in Figures 63-66 and Table 8. As seen in Table 8, at low frequency ranges of 125, 250 and 500 Hz, both NR foam with and without water hyacinth exhibited unstable α-values. Moreover, at paticular frequency (1000 Hz), the NR foam began to show slightly higher α-values while increased the water hyacinth content. At high frequency regions (4000 Hz), the ability to absorb sound at this frequency of almost NR foam filled with water hyacinth except for NR vulcanizates (without blowing agent; NF-0) was found to be better than that of the NR foam without water hyacinth. At 6 phr of blowing agent, the NR foam filled with 10 phr of water hyacinth exhibited a maximum sound absorption coefficient (α-value) of 0.71 at low frequency regions (1000 Hz) than NR foam filled with 5 phr of water hyacinth and without water hyacinth showing with α-value of 0.69, 0.55, respectively. That is to say NR foams containing 10 phr of water hyacinth have superior sound absorption capacity at low frequency regions (1000 Hz). Whereas, at high frequency regions (4000 Hz) NR foams filled with 5 phr of water hyacinth showed the highest α-value of 0.60. The sound absorption of NR vulcanizates (without blowing agent) was found to be poorer than that of NR foam at every frequency. This is due to lower densities of the NR foam compared with the NR vulcanizates (NF-0) as seen in Table 5.

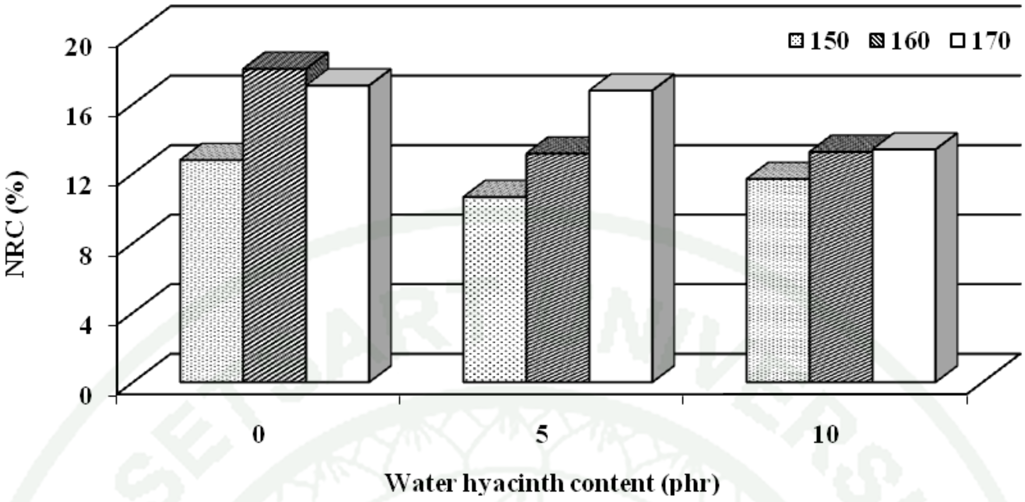


Figure 63 The effect of water hyacinth content on percentage noise reduction coefficient (NRC) of the NR vulcanizates without blowing agent at different foaming temperature.

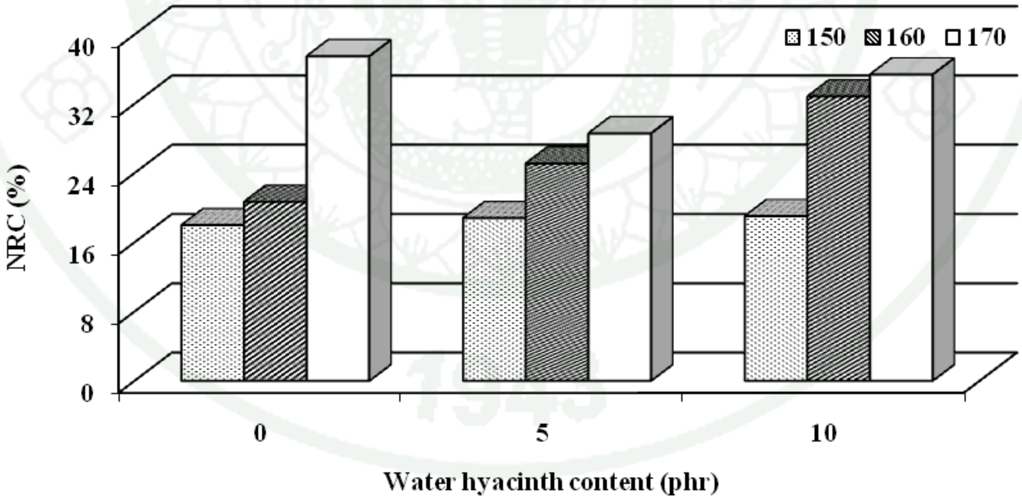


Figure 64 The effect of water hyacinth content on percentage noise reduction coefficient (NRC) of the NR foams containing 2 phr of blowing agent at different foaming temperature.

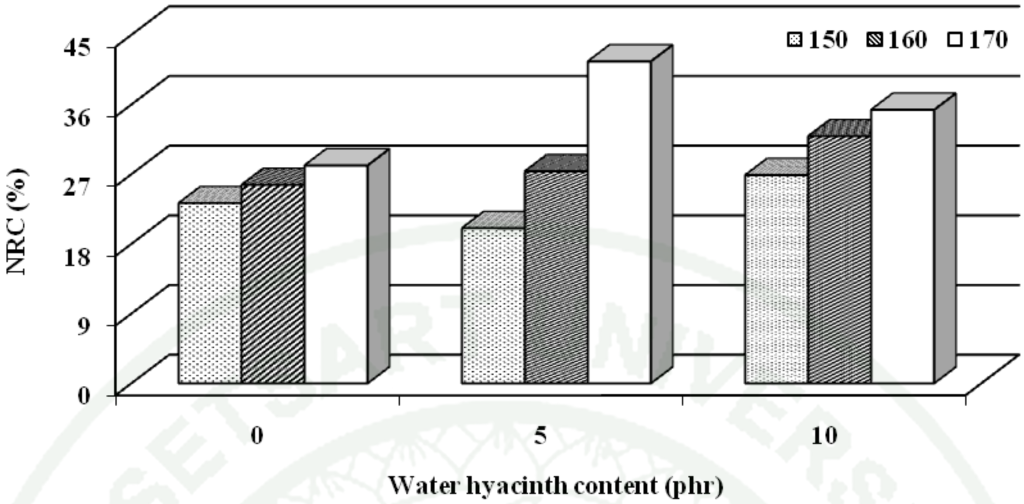


Figure 65 The effect of water hyacinth content on percentage noise reduction coefficient (NRC) of the NR foams containing 4 phr of blowing agent at different foaming temperature.

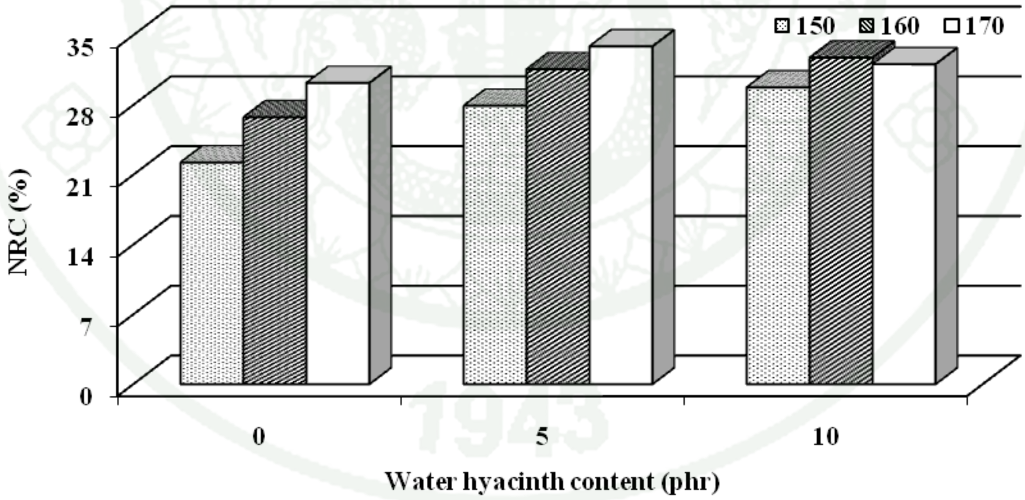


Figure 66 The effect of water hyacinth content on percentage noise reduction coefficient (NRC) of the NR foams containing 6 phr of blowing agent at different foaming temperature.

This is in agreement with the results of Suhawati *et al*., (2008) which reported a sound absorption of foamed rubber composites from wood and calcium carbonate. Their results revealed that the low sound absorption coefficient of foam rubber was observed, with increasing the high degree of hardness at a very high filler loading. Sound coefficient increased with the decreasing fiber diameter (Lee *et al*., 2001). With the impermeable size much smaller compare to sound wavelength, the change from sound energy to heat energy will increase because of friction air particle that vibrate. Both of the materials are porous material. The rate of sound absorption will be affected with the number of air space that exists in the material. Wassilieff, (1996) included the other parameters such as porosity and tortuosity to define acoustic properties for impermeable material.

5.3 Effect of blowing agent loading

The sound absorption properties of NR foam filled with and without water hyacinth content at various blowing agent loading at different foaming temperature were determined for 13 mm of the NR foam sample and for the frequency of low and high range. This is an important parameter, because it influences mainly the foaming temperature and the NR foam samples' structural characteristics. The effect of blowing agent will definitely affect the acoustic properties depending on the type of blowing agent used and loading of blowing agent used.

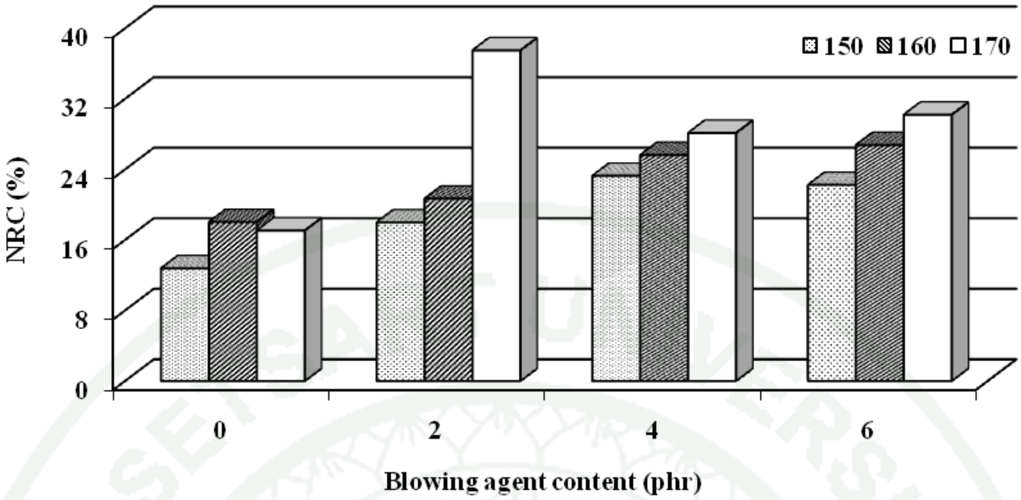


Figure 67 The effect of blowing agent content on percentage noise reduction coefficient (NRC) of the NR foams without water hyacinth at different foaming temperature.

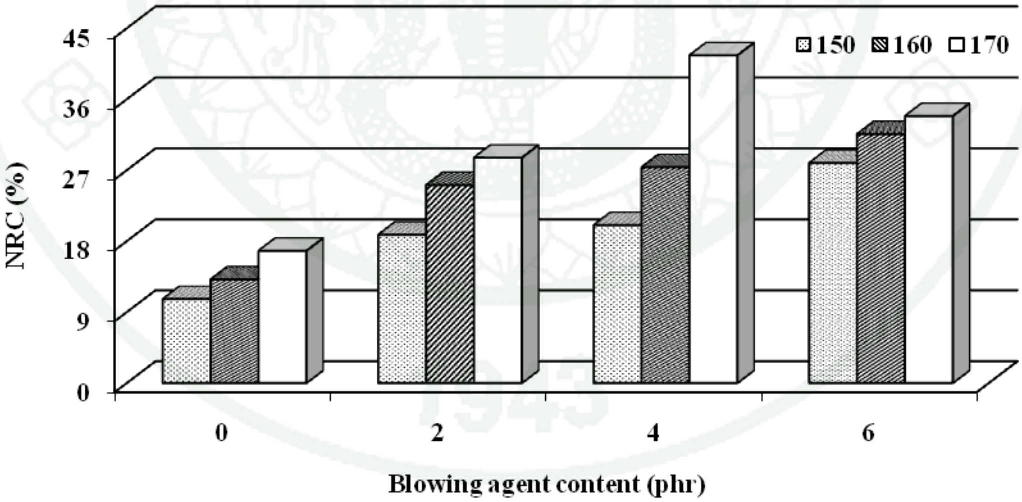


Figure 68 The effect of blowing agent content on percentage noise reduction coefficient (NRC) of the NR foams filled with 5 phr of water hyacinth at different foaming temperature.

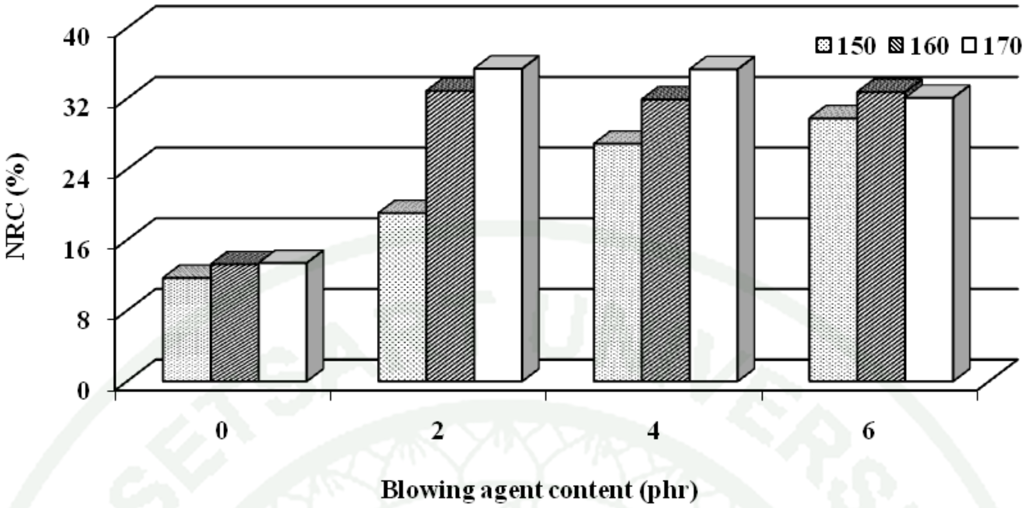


Figure 69 The effect of blowing agent content on percentage noise reduction coefficient (NRC) of the NR foams filled with 10 phr of water hyacinth at different foaming temperature.

The blowing agent content affects both the cell structure and physical properties of NR foams. Also the effect of blowing agent loading on the acoustic properties of NR foams is depicted in Table 8. The results for the 13 mm thick NR foam sample show that the increment in blowing agent yields a gradual increase in α-value with a maximum at 0.69 at low frequency regions (1000 Hz) when compared to that of the NR vulcanizates without blowing agent. Whereas, at high frequency regions (4000 Hz) NR foams filled with 6 phr of blowing agent showed the highest α-value of 0.52. The sound absorption of NR vulcanizates (without blowing agent) was found to be poorer than that of NR foam at every frequency. Furthermore, it can seen from Figures 67-69 that the NRC percentage tended to increase appreciably with increasing blowing agent loading due to the quanlity of gas released. Moreover, the thickness of the cell walls is very uniform when compared to NR foam filled with 4 phr of blowing agent, as seen in Figure 40. Likewise, the average cell size of foams increased with increasing the blowing agent loading.

Therefore, the suitable blowing agent loading to be employed was depends on the desired physical properties and application of NR foam. In this study, we can concluded that the optimum amount of water hyacinth and blowing agent including foaming temperature to produce an appropriate NR foams were 10 phr of water hyacinth, 4 and 6 phr of blowing agent at foaming temperature of 170°C. The NRC percentage of NR foam filled with blowing agent at this optimum condition at both frequency ranges gave NRC percentage around 0.321-0.416. Therefore NR foams filled with higher water hyacinth content has a better sound absorber, at a fixed blowing agent loading at 4 and 6 phr for foaming temperature of 170°C in which it could be can appropriate for acoustic absorption material at both frequency ranges about 1,000 and 4,000 Hz with thickness of 13 mm.

The sound absorptive of NR foam at this optimum condition was found to be comparable to acoustical product of M-Sorb (sound insulation and acoustic PE board) produced by M-PE Insulation Co., Ltd. (Thailand) which NRC was reported to be 0.45 at thickness of 40 mm. NR foams at this optimum condition can be used for sound absorptive protection and echo in which it is capable to effectively control echo level and direction of sound reflection for sound frequency at low frequency (1,000 Hz) and high frequency (4,000 Hz). These studied NR foam is a best suitable for installation with ceilings or room walls which require to control echo level which it can be easily installed on wall/ ceiling in auditorium, movie room, karaoke, music practical room and used for other functions of sound absorption. NR foam at this optimum condition give more good absorption value (α-value) and NRC percentage compared to NR vulcanizates (without blowing agent), but the capabilities depend on a lot of sound absorption factors such as thickness, fiber diameter, bulk density and other. Future work could be done to standardize some parameter like thickness and density including with blowing agent type to produce a better understanding results.

CONCLUSION

The purpose of this study was to verify the possibility of using water hyacinth fiber in replacing synthetic-based fibers as alternative fiber for rubber foam in industrial and commercial applications, especially in sound absorption applications. The natural rubber and water hyacinth fiber is an environmentally friendly product, being biodegradable, abundant and cheap. Sound absorbing foam materials are produced from solid dry natural rubber (NR) with the addition of 4,4’-oxybis (benzene sulfonyl) hydrazide (OBSH) as a blowing agent, which releases nitrogen gas during its decomposition and is able to produce foam with an partially open cell structure and were expanded using compression molding technique with utilization of heat transfer process.

The results suggested that the cure behavior, physical and mechanical properties of the NR foams tended to decrease with increasing foaming temperature, water hyacinth and blowing agent content. The resultants at 170°C and higher blowing agent including more water hyacinth content gave NR foams with bigger cell size, better cell distribution, and good compression deflection test which are closely related to the cellular rubber made from NR or rubber-like materials alone or in combination followed by ASTM D 1056 than the NR foams produced at 150 and 160°C, supporting the foaming efficiency results. It was comfirmed that the optimum amount of water hyacinth and blowing agent including foaming temperature to produce an appropriate NR foams were 10 phr of water hyacinth, 6 phr of blowing agent at foaming temperature of 170°C. The results showed that the morphology, physical and compressive properties of the NR foams can be controlled closely by the foaming temperature. The increase of the foaming temperature led to different results relative to those from the increase of the water hyacinth and blowing agent content. As a consequence, it is observed that blowing agent contents influenced the cell morphology of NR foam, thus simultaneously affecting the mechanical properties of the NR foam. As the blowing agent content increases, more nitrogen gas decomposes, resulting in a bigger average cell size. This simultaneously causes a decrease in the relative foam density and slightly decreases the crosslink density. It is proven from the result of this study that using OBSH is capable in exerting enough nitrogen to produce NR foam.

Sound absorbing foam materials are produced from solid NR with the addition of OBSH at foaming temperatures of 170°C, and feeding ratio of the water hyacinth (10 phr) including blowing agent (4 and 6 phr) at thickness of 13 mm, exhibits superior sound properties than NR foam produced at foaming temperatures of 150 and 160°C, which is reflected by the uniform average cell size as measured by the increase in sound absorption coefficient (α) and noise reduction coefficient (NRC). The sound absorptive of NR foam at this optimum condition was found to be comparable to acoustical product of M-Sorb (sound insulation and acoustic PE board) produced by M-PE Insulation Co., Ltd. (Thailand) which NRC was reported to be 0.45 at thickness of 40 mm. NR foams at this optimum condition can be used for sound absorptive protection and echo in which it is capable to effectively control echo level and direction of sound reflection for sound frequency at low frequency (1,000 Hz) and high frequency (4,000 Hz). These studied NR foam is a best suitable for installation with ceilings or room walls which require to control echo level which it can be easily installed on wall/ ceiling in auditorium, movie room, karaoke, music practical room and used for other functions of sound absorption. NR foam at this optimum condition give more good absorption value (α-value) and NRC percentage compared to NR vulcanizates (without blowing agent), but the capabilities depend on a lot of sound absorption factors such as thickness, fiber diameter, bulk density and other. Future work could be done to standardize some parameter like thickness and density including with blowing agent type to produce a better understanding results.

LITERATURE CITED

Agency for International Development. 1976. **Making Aquatic Weeds Useful, Some Perspectives for Developing Countries**. National Tech. Inf. Ser. Washington D.C., USA.

Adachi, H., T. Hasegawa and T. Asano. 1997. Cell distributions in and sound absorption characteristics of flexible polyurethane foams. **Journal of Applied Polymer Science**. 65: 1395-1402.

Albano, C., J. Gonzalez, M. Ichazo and D. Kaiser. 1999. Thermal stability of blend of polyolefins and sisal fiber. **Polymer Degradation and Stability**. 66: 179-190.

Alexandre, M. and P. Dubois. 2000. Polymer-Layered Silicate Noanocomposites: preparation, properties and uses of a new class of materials. **Materials Science and Engineering**. 28: 1-63.

Amy, M. 2009. **Ecological effects of water hyacinth (Eichhornia crassipes) on Lake Chapala, Mexico**. Ph.D. Thesis, Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

Anh, H.M. and B.T. Huyen. 2007. **Possible utilization of the water hyacinth in nutrition and industry in Vietnam**. Horizon International Bilingual School Press. (International Environmental Project Olympiyad, Bronze medals)

Anthoine, G., R. Arnold, K. Boustany and J. Campbell. 1975. Regenerated cellulose in elastomer compounds. **European Rubber Journal**. 157: 28.

Ariff, Z.M., Z. Zakaria, L.H. Tay and S.Y. Lee. 2008. Effect of foaming temperature and rubber grades on properties of natural rubber foams. **Journal of Applied Polymer Science**. 107: 2531-2538.

Ariff, Z.M. 1998. **Foaming Behavior Dependence on Base Polymer Parameter**. M.Sc. Dissertation, University of Manchester Institute Science and Technology, England.

ASTM International. 2003. **Standard test method for impedance and absorption of acoustical material using a tube, two microphones and a digital frequency analysis system**. ASTM E 1050-03, Vol.04.06.

ASTM International. 2000. **Standard test method for vulcanized rubber and thermoplastic elastomers-Tension**. ASTM D 412-00, Vol. 09.01.

Bascom, R.C. 1964. Cellular Elastomers. **Rubber Age Journal**. 95: 576-578.

Bates, R.P. and J.F. Hentges. 1976. Aquatic weeds-eradicate or cultivate. **Journal Economic Botany**. 30: 39-50.

Bledzki, A.K. and O. Faruk. 2006. Injection moulded microcellular wood fibre-polypropylene composites. **Journal of Composites Part A**. 37(9): 1358-1367.

Blow, C.M. and C. Hepburn. 1982. **Rubber Technology and Manufacture**. 2nd ed. Butterworth Scientific, London.

Bokobza, L. and J.P. Chauvin. 2005. Reinforcement of natural rubber: use of in-situ generated silicas and nanofibres of sepiolite. **Polymer Journal**. 46(12): 4144-4151.

Bonfiglio, P. and F. Pompoli. 2008. A single measurement approach for the determination of the normal incidence transmission loss. **Journal of the Acoustical Society of America**. 124: 1577-1583.

Lee, F.C. and W.H. Chen. 2001. Acoustic transmission analysis of multi-layer absorbers. **Journal of Sound and Vibration**. 4: 621.

Rodgers, B. 2004. **Rubber compounding; Chemistry and Applications**. Marcel Dekker, Inc., New York.

Bruce, F. 1981. **Industrial Noise Control**. Interscience publication, John Wiley and Sons, NewYork.

Bruel and Kajer. 2009. **Product data sheet**. Available Source: http://www.bksv.com, November 20, 2009.

Chen, J.H., S.J. Tsai, S.N. Tong and P.T.K. Wu. 1990. **Production of hybrid foam for sound absorption**. U.S. Patent 4,916,167.

Claudio, B. and B. Andrea. 1998. Least squares estimation of main properties of sound absorbing materials through acoustical measurements. **Applied Acoustics**. 54(1): 59-70.

Cowan, J. 2000. **Architectural Acoustics-Design Guide**. McGraw-Hill, New York.

Cox, T.J. and P. D'Antonio. 2004. **Acoustic Absorbers and Diffusers**. Spon Press, Taylorand Francis Group, London and New York.

David, F. 2009. **The Four-Fold Method of Noise and Vibration Control**. McGraw-Hill, Inc., New York.

Duanghathai, P. and W.E. John. 2005. **An Introduction to Plastics Foams**. Chulalongkorn University Press, Bangkok, Thailand.

Egli, E.A. 1972. Design properties of structural foam. **Journal of Cellular Plastics**. 8: 245-249.

Everest, F.A. 1994. **The Master Handbook of Acoustics**. 3rd ed. TAB Books, New York.

Freakly, P.K. and A.R. Payne. 1978. **Theory and Practice of Engineering with Rubber**. Applied Science Publishers Ltd., London.

Faulkner, L.L. 1976. **Handbook of Industrial Noise Control**. Industrial Press, New York.

Francisco, S. and P. Jaime. 2004. Guidelines for the Acoustic Design of Absorptive Devices. **Noise and Vibration worldwide**. 35(1):12-21.

Frank, F. 2001. **Foundations of Engineering acoustics**. Academic Press, San Diego, Califonia.

Gent, A.N. 1992. **Engineering with rubber: How to design rubber components**. Hanser Publishers, Munich.

Gent, A.N. and D.A. Tompkins. 1968. Nucleation and growth of gas bubbles in elastomers. **Journal of Applied Physics**. 40(6): 2520-2525.

Gibson, L.J. 2005. Biomechanics of cellular solids. **Journal of Biomechanics**. 38: 377-399.

Gibson, L.J. and M.F. Ashby. 1988. **Cellular Solids: Structure and Properties**. Pergamon Press, Oxford.

Gibson, L.J. and M.F. Ashby. 1997. **Cellular Solids, Structure and Properties**. 2nd ed. Cambridge University Press, Cambridge.

Goettler, L.A. and K.S. Shen. 1983. Short fiber reinforced elastomers. **Rubber Chemistry and Technology**. 56(3): 619-620.

Gowanloch, J.N. 1944. The econimc status of water-hyacinth in Louisiana. **The Los Angeles Conservancy**. 2: 3-8.

Guriya, K.C. and D.K. Triphthy. 1996. Morphology and Physical Propeties of Closed-Cell Mircorcellular Ethylene-Propylene-Diene Terpolymer (EPDM) Rubber Vulcanizates: Effect of Blowing Agent and Carbon Black Loading. **Journal of Applied Polymer Science**. 62: 117-127.

Iannace, S., R. Ali and L. Nicolais. 2001. Effect of processing conditions on dimensions of sisal fibers in thermoplastic biodegradable composites. **Journal of Applied Polymer Science**. 79(6): 1084-1091.

Ismail, H.S. and A.S. Hashim. 1998. **Pengenalan Penyebatian dan Pemprosesan Getah**. Pulau Pinang, Penerbit University Sains, Malaysia.

Ismail, H.S., S. Shuhelmy, and M.R. Edyham. 2002. The effect of a silane coupling agent on curing characteristics and mechanical properties of bamboo fiber filled natural rubber composites. **European Polymer Journal**. 38: 39-47.

Harris, C.M. 1979. **Handbook of noise control**. 2nd ed. McGraw-Hill, New York.

Hofmamm, W. 1989. **Rubber Technololog Handbook**. Oxford University Press, New York.

Hong, Z., L. Bo, H. Guangsu and H. Jia. 2007. A novel composite sound absorber with recycled rubber particles. **Journal of Sound and Vibration**. 304: 400-406.

Horoshenkov, K.V. and M.J. Swift. 2001. The effect of consolidation on the acoustic properties of loose rubber granulates. **Applied Acoustics**. 62(6): 665-690.

Horsfall, M.Jr. and A.A. Abia. 2003. Sorption of cadmium(ii) and zinc(ii) ions from aqueous solutions by cassava waste biomass (manihot sculenta cranz). **Water Research**. 37(20): 4913-4923.

Jadhav, D.N. and A.K. Vanjara. 2004. Removal of phenol from wastewater using sawdust, polymerized sawdust and sawdust carbon. **Indian Journal of Chemical Technology**. 11(1): 35-41.

Jaouen, L., A. Renault and M. Deverge. 2008. Elastic and damping characterizations of acoustical porous materials: available experimental methods and applications to a melamine foam. **Applied Acoustics**. 69: 1129-1140.

Joseph, P.V., K. Joseph, S. Thomas, C.K.S. Pillai, V.S. Prasad and G. Groeninckxl. 2003. The thermal properties and crystallisation studies of short sisal fibre reinforced polypropylene composites. **Journal of Composites Part A**. 34: 253-266.

Kasselmann C. 1995. **Aquarienpflanzen**. Egen Ulmer GMBH and Co., Stuttgart, German.

Khedari, J., S. Charoenvai and J. Hirunlabh. 2003. New insulating particleboards from durian peel and coconut coir. **Building and Environment.** 38: 435-441.

Kim, J.H., J.S. Koh, K.C. Choi, J.M. Yoon and S.Y. Kim. 2007. Effects of foaming temperature and carbon black content on the cure characteristics and mechanical properties of natural rubber foams. **Journal of Industrial and Engineering Chemistry**. 13(2): 198-205.

Kin, M.H., Z. Yang, X.X. Zhang and P. Sheng. 2005. Measurements of sound transmission through panels of locally resonant materials between impedance tubes. **Applied Acoustics**. 66: 751-765.

Kirby, R. and A. Cummings. 1999. Prediction of the bulk acoustic properties of fibrous materials at low frequency. **Applied Acoustics**. 56: 101-125.

Klempner, D. and K.C. Frisch. 1991. **Handbook of Polymeric Foams and Foam Technology**. Hanser Publishers, New York.

Klempner, D. and V. Sendijarevic. 2004. **Handbook of Polymeric Foams and Foams Technology**. Hanser Publishers, Munich.

Knapen. E., R. Lanoye, G. Vermeir and G.D. Van. 2003. **Sound absorption by polymer-modified porous cement mortars**, pp. 347-358. *In* 6th International Conference on Materials Science and Restoration. MSR-VI Aedificatio Publishers.

Kojima, T. 1986. Generation of methane gas from water hyacinth (Eichhornia crassipes), production of methane gas from combination of water hyacinth and fowl droppings. **Bulletin of the faculty of Agriculture, Saga University, Japan**. 61: 1-8.

Lawrence, A. 1970. **Architectural Acoustics**. Applied Science Publishers, London.

Lee, C.M. and Y.S. Wang. 2006. A prediction method of the acoustical properties of multilayered noise control materials in standing wave-duct systems. **Journal of Sound and Vibration**. 298: 350-365.

Lee, E.K. and S.Y. Choi. 2007. Preparation and characterization of natural rubber foams: Effects of foaming temperature and carbon black content. **Korean Journal of Chemical Engineering**. 24(6), 1070-1075.

Leo, L.B. 1960. **Noise reduction, prepared for a special summer program at MIT**. McGraw-Hill, New York.

Leo, L.B. 1971. **Noise and Vibration control**. McGraw-Hill, New York.

Leo, L.B. 1988. **Noise and Vibration Control (Revised Edition)**. Institute of Noise Control Engineering, Washington, DC, USA.

Leo, L.B. 1992. Noise and Vibration Control Engineering, Principles and Applications, Chapter 8. *In* P.M. Fridolin and L.V. Istvan, eds. **Sound-Absorbing Materials and Sound Absorbers**. Wiley, New York.

Lewis, H.B. 1994. **Industrial noise control, Fundamentals and applications**. 2nd ed. M. Dekker, New York.

Li, Q. and L.M. Matuana. 2003. Foam extrusion of high density polyethylene/wood-flour composites using chemical foaming agents. **Journal of Applied Polymer Science**. 88: 3139.

Liu, G. 1998. Production of low-density LLDPE foams in rotational molding. **Polymer Engineering Science**. 38(12): 1997-2009.

Lin, G., X.J. Zhang, L. Liu, J.C. Zhang, Q.M. Chen, and L.Q. Zhang. 2004. Study on microstructure and mechanical properties relationship of short fibers/rubber foam composites. **European Polymer Journal**. 40(8): 1733-1742.

Lin, H.R. 1996. Material properties: The structure and property relationships of commercial foamed plastics. **Polymer Testing**. 16: 429-433.

Livestock Ayutthaya, office. 1991. **Water Hyacinth**. Livestock Ayutthaya, office, Phra Nakhon Si Ayutthaya, Thailand. (Mimeographed)

Machida, M., T. Mochimard and H. Tatsumoto. 2006. Lead(II) adsorption onto the graphene layer of carbonaceous materials in aqueous solution. **Carbon**. 44(13): 2681-2688.

Maekawa, Z and P. Lord. 1994. **Environmental and Architectural Acoustics**. E and FN SPON, London.

Mahmood, Q., P. Zheng, M.R. Siddiqi, E.U. Islam, M.R. Azim and Y. Hayat. 2005. Anatomical studies on water hyacinth (Eichhornia crassipes (Mart.) Solms) under the influence of textile wastewater. **Journal of Zhejiang University Science B**. 6: 991-998.

Marsh, A.U. 1999. **Membrane Absorber**. The School of Architecture and Fine Arts, The University of Western Australia. (Lecturer note)

Maya, J., S. Thomasa and K.T. Varugheseb. 2004. Mechanical properties of sisal/oil palm hybrid fiber reinforced natural rubber composites. **Composites Science and Technology**. 64: 955-965.

Michael, H.Jr. and J.L. Vicente. 2007. Adsorption of Methyl Red by Water-Hyacinth (Eichornia crassipes) Biomass by Temi Tarawoua. **Chemistry and biodiversity**. 4: 2236-2245.

Mitchell, D.S. 1976. The growth and management of Eichhornia crassipes and Salvinia spp. in their native environment and in alien sitautions. **Aquatic weeds in Southeast Asia**. 396.

Morton, M. 1987. **Rubber Technology**. 3rd ed. Van Nostrand Reinhold, New York.

Mueller, D.H., K. Andreas and S. Heidrun. 2002. **Acoustical properties of reinforced composite materials and layered structures basing on natural fibers**. *In* Proceedings of the INTC; International Nonwovens Technical Conference, September 24-26th. Atlanta, Georgia/USA.

Nagdi, K. 1993. **Rubber as an Engineering Material: Guideline for User**. Hanser, Munich.

Najib, N.N., Z.M. Ariff, A.A. Bakar and C.S. Sipaut. 2011. Correlation between the acoustic and dynamic mechanical properties of natural rubber foam: Effect of foaming temperature. **Materials and Design Journal**. 32: 505-511.

Nugay, N. and B. Erman. 2001. Property optimization in nitrile rubber composites via hybrid filler systems. **Journal of Applied Polymer Science**. 79: 366-374.

Penfound W.T. and T.T. Earle. 1948. The biology of the water hyacinth. **Ecological Monographs**. 18: 449-472.

Potiyaraj, P., P. Panchaipech and S. Chuayjuljt. 2001. Using Water-Hyacinth Fiber as a Filler in Natural Rubber. **Journal of Science Research Chulalongkorn University.** 26(1): 12-19.

Price, A.J. and K.A. Mulholland. 1968. The effect of surface treatment on sound-absorbing materials. **Applied Acoustics**. 1(1): 67-72.

Ray, D., B.K. Sakar, R.K. Basak and A.K. Rana. 2002. Study of the thermal behavior of alkali treated jute fibers. **Journal of Applied Polymer Science**. 85: 2594-2599.

Richard, T.G. 1979. The mechanical behavior of a solid microsphere filled. **Journal of Composite Materials**. 9: 710.

Roberts, A.D. 1990. **Natural Rubber Science and Technology**. Oxford University Press, New York.

Roff, W.J. and J.R. Scott. 1971. **Fibres, films, plastics and rubbers: a handbook of common polymers**. Butterworths, London.

Saunders, J.H. 1960. The Formation of Urethane Foams. **Rubber Chemistry and Technology**. 33: 1293-1322.

Setua, D.K. and S.K. De. 1984. Short silk fibre reinforced nitrile rubber composites. **Journal of Materials Science**. 19: 983-999.

Shen, Y., F. Golnaraghi and A. Plumtree. 2001. Modelling compressive cyclic stress-strain behaviour of structural foam. **International Journal of Fatigue**. 23: 491-497.

Shenton, H.W., R.P. Wool, B. Hu, A. O'Donnell, L. Bonnaillie, and E. Can. 2002. **An allnatural composite material roof system for residential construction**, pp. 255-262. *In* Proceedings of the international conference on advances in building technology, Hong Kong.

Sims, G.L.A and C. Khunniteekool. 1995. **Cell structure development in compression moulded crosslinked polyethylene and ethylene-vinyl acetate foam**, pp 1-6. *In* Cellular polymers III: 3rd International Conference. Rapra technology, Coventry, England.

Skinner, C., J. Peters and J. Vandenbroeck. 2006. **Acoustic absorber: A third way for the management of sound in automobiles**. pp. 1-14. *In* UTECH 2006, Maastricht, Netherland.

Sombatsompop, N. and P. Lertkamolsin. 2000. Effects of chemical blowing agents on swelling properties of expanded elastomers. **Journal of Elastomers and Plastics**. 32: 311-326.

Sreekala, M.S., S. Thomas and N.R. Neelakantan. 1997. Utilization of short oil palm empty fruit bunch fibre (OPEFB) as a reinforcement in phenol-formaldehyde resin: Studies on mechanical properties. **Journal of Polymer Engineering**. 16: 265-294.

Stern, H.J. 1955. **Practical Latex Work**. 3rd ed. The Blackfriars Press Ltd., Leicester, UK.

Stern, A.S., K.B. Li and J.C. Hoch. 2002. Modern spectrum analysis in multidimensional NMR spectroscopy: comparison of linear prediction extrapolation and maximum entropy reconstruction. **Journal of the American Chemical Society**. 124: 1982-1993.

Strauss, W. and N.A. D'Souza. 2004. Supercritical CO_2 processed polystyrene nanocomposites foams. **Journal of Cellular Plastics**. 40: 229-241.

Suhawati, I., K. Shamsul, I. Ismaliza and A.M. Kamarudin. 2008. **Sound absorption analysis of foamed rubber composites from wood fibre and calcium carbonate**. *In* Proceeding of International Rubber Conference and Exhibition, Convention Center, Kuala Lumpur, Malaysia.

Tadmor, Z. and C.G. Gogos. 2006. **Principles of Polymer Processing**. 2nd ed. Wiley-Interscience, New Jersey, USA.

Takahashi, Y., T. Otsuru and R. Tomiku. 2005. In Situ Measurements of surface impedance and absorption coefficients of porous materials using two microphones and ambient noise. **Applied Acoustics**. 66: 845-865.

Thomas, N.L., R.P. Eastup and J.P. Quirk. 1997. Aspects of formulation technology of rigid poly(vinyl chloride) foam. **Plastics Rubber and Composites Processing Applications**. 26(2): 47-54.

Thuman, A. and R.M. Miller. 1986. **Foundation of noise control engineering**. Fair Mart Inc., Georgia.

Timothy, H., J. David, M. Robert, G. Rebandt, R. Phillip and S. Pranab. 1999. Automotive Noise and Vibration Control Treatments. **Sound and Vibration**. 3: 22-32.

Uejyukkoku, N. and Y. Nakatsu. 1996. **Polyolefin based crosslinked foam**. U.S. Patent 5,786,406.

Uno, I. 1994. **Notes on Sound Absorption Technology**. Poughkeepsie, Noise Control Foundation, New York.

Voronina, N. 1996. Improved empirical model of sound propagation through a fibrous material. **Applied Acoustics**. 48(2): 121-132.

Wambua, P., J. Ivens and I. Verpoest. 2003. Natural fibres: can they replace glass in fibre reinforced plastics? **Composites Science and Technology**. 63(9): 1259-1264.

Warnock, A.C.C. 1980. **Reverberant noise control in rooms using sound absorbing materials.** Press by Institute for Research in Construction, National Research Council of Canada.

Wassilieff, C. 1996. Sound absorption of wood-based materials. **Applied Acoustics**. 48(4): 339-356.

Yerges, L.F. 1978. **Sound, Noise and Vibration Control.** 2nd ed. Van Nostrand Rainhold Campany, New York.

Yoshio, I. and A. Tsuneo. 1982. Studies of acoustical absorption of flexible polyurethane foam. **Journal of Applied Polymer Science**. 27: 183-195.

Youn, E.L. and W.J. Chang. 2004. Sound absorption properties of thermally bonded nonwovens based on composing fibers and production parameters. **Journal of Applied Polymer Science**. 92: 2295-2302.

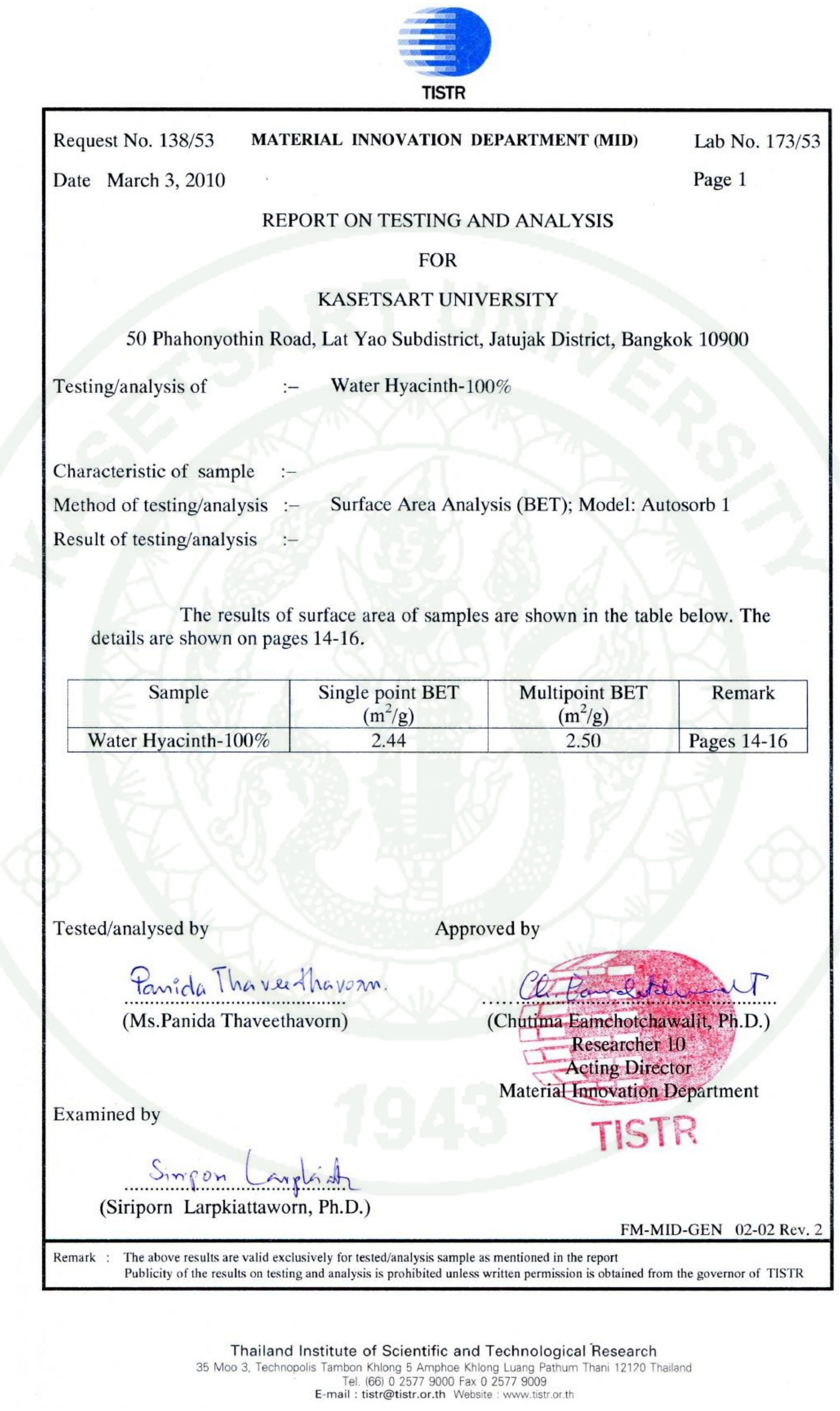
Yunseon, R. 2002. Technical Note: Test procedure and user interface for determination of the acoustic properties of materials using the two-microphone transfer function method. **Building Acoustics**. 9(1): 73-79.

Zakaria, Z., Z.M. Ariff, L.H. Tay and C.S. Sipaut. 2007. Effect of foaming temperature on morphology and compressive properties of ethylene-propylene-diene terpolymer (EPDM) foam. **Malaysian Polymer Journal**. 2(2): 22-30.

Zakaria, Z. 2007. **Characterization of polyethylene foam and its structure-properties relationship in shock absorbing application**. M.Sc. Dissertation, Universiti Sains Malaysia, Pulau Pinang, Malaysia.

Zulkifli, R., M.J. Mohd Nor, M.F. Mat Tahir, A.R. Ismail and M.Z. Nuawi. 2008. Acoustic properties of multi-layer coir fibers sound absorption panel. **Journal of Applied Science**. 8(20): 3709-3714.

APPENDIX



TISTR

Request No. 138/53 **MATERIAL INNOVATION DEPARTMENT (MID)** Lab No. 173/53

Date March 3, 2010 Page 1

REPORT ON TESTING AND ANALYSIS

FOR

KASETSART UNIVERSITY

50 Phahonyothin Road, Lat Yao Subdistrict, Jatujak District, Bangkok 10900

Testing/analysis of :– Water Hyacinth-100%

Characteristic of sample :–

Method of testing/analysis :– Surface Area Analysis (BET); Model: Autosorb 1

Result of testing/analysis :–

The results of surface area of samples are shown in the table below. The details are shown on pages 14-16.

Sample	Single point BET (m^2/g)	Multipoint BET (m^2/g)	Remark
Water Hyacinth-100%	2.44	2.50	Pages 14-16

Tested/analysed by

Panida Thaveethavorn.

(Ms.Panida Thaveethavorn)

Approved by

(Chutima Eamchotchawalit, Ph.D.)
Researcher 10
Acting Director
Material Innovation Department

TISTR

Examined by

(Siriporn Larpkiattaworn, Ph.D.)

FM-MID-GEN 02-02 Rev. 2

Remark : The above results are valid exclusively for tested/analysis sample as mentioned in the report
Publicity of the results on testing and analysis is prohibited unless written permission is obtained from the governor of TISTR

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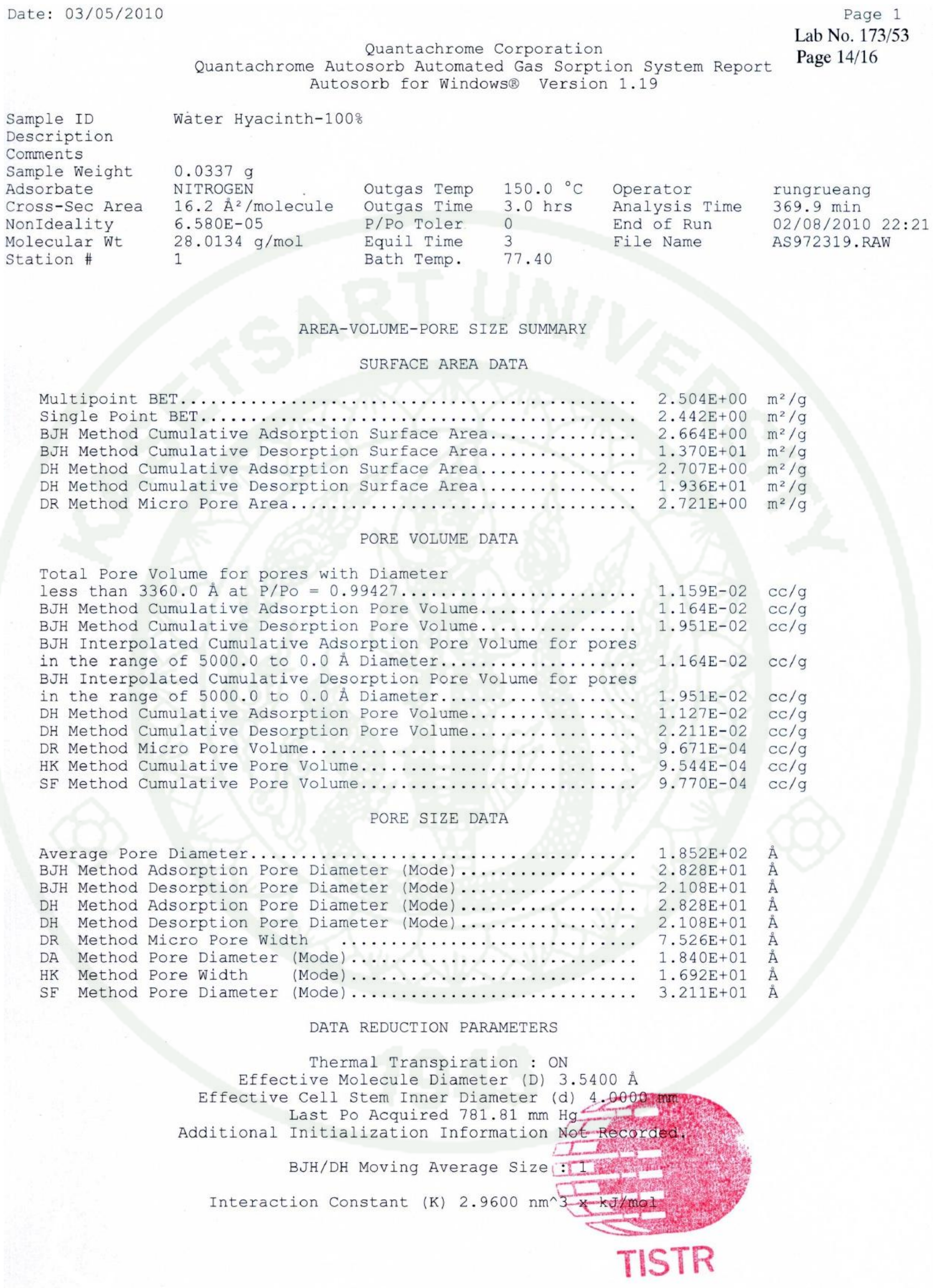
Appendix Figure 1 Report on Surface area of water hyacinth fiber



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Date: 03/05/2010                                                              Page 1
                                                                        Lab No. 173/53
                        Quantachrome Corporation                        Page 14/16
          Quantachrome Autosorb Automated Gas Sorption System Report
                    Autosorb for Windows®  Version 1.19

Sample ID        Water Hyacinth-100%
Description
Comments
Sample Weight    0.0337 g
Adsorbate        NITROGEN           Outgas Temp   150.0 °C   Operator         rungrueang
Cross-Sec Area   16.2 Å²/molecule   Outgas Time   3.0 hrs    Analysis Time    369.9 min
NonIdeality      6.580E-05          P/Po Toler    0          End of Run       02/08/2010 22:21
Molecular Wt     28.0134 g/mol      Equil Time    3          File Name        AS972319.RAW
Station #        1                  Bath Temp.    77.40


                       AREA-VOLUME-PORE SIZE SUMMARY

                             SURFACE AREA DATA

   Multipoint BET.............................................  2.504E+00  m²/g
   Single Point BET...........................................  2.442E+00  m²/g
   BJH Method Cumulative Adsorption Surface Area..............  2.664E+00  m²/g
   BJH Method Cumulative Desorption Surface Area..............  1.370E+01  m²/g
   DH Method Cumulative Adsorption Surface Area...............  2.707E+00  m²/g
   DH Method Cumulative Desorption Surface Area...............  1.936E+01  m²/g
   DR Method Micro Pore Area..................................  2.721E+00  m²/g

                             PORE VOLUME DATA

   Total Pore Volume for pores with Diameter
   less than 3360.0 Å at P/Po = 0.99427.......................  1.159E-02  cc/g
   BJH Method Cumulative Adsorption Pore Volume...............  1.164E-02  cc/g
   BJH Method Cumulative Desorption Pore Volume...............  1.951E-02  cc/g
   BJH Interpolated Cumulative Adsorption Pore Volume for pores
   in the range of 5000.0 to 0.0 Å Diameter...................  1.164E-02  cc/g
   BJH Interpolated Cumulative Desorption Pore Volume for pores
   in the range of 5000.0 to 0.0 Å Diameter...................  1.951E-02  cc/g
   DH Method Cumulative Adsorption Pore Volume................  1.127E-02  cc/g
   DH Method Cumulative Desorption Pore Volume................  2.211E-02  cc/g
   DR Method Micro Pore Volume................................  9.671E-04  cc/g
   HK Method Cumulative Pore Volume...........................  9.544E-04  cc/g
   SF Method Cumulative Pore Volume...........................  9.770E-04  cc/g

                              PORE SIZE DATA

   Average Pore Diameter......................................  1.852E+02  Å
   BJH Method Adsorption Pore Diameter (Mode).................  2.828E+01  Å
   BJH Method Desorption Pore Diameter (Mode).................  2.108E+01  Å
   DH  Method Adsorption Pore Diameter (Mode).................  2.828E+01  Å
   DH  Method Desorption Pore Diameter (Mode).................  2.108E+01  Å
   DR  Method Micro Pore Width   .............................  7.526E+01  Å
   DA  Method Pore Diameter (Mode)............................  1.840E+01  Å
   HK  Method Pore Width    (Mode)............................  1.692E+01  Å
   SF  Method Pore Diameter (Mode)............................  3.211E+01  Å

                        DATA REDUCTION PARAMETERS

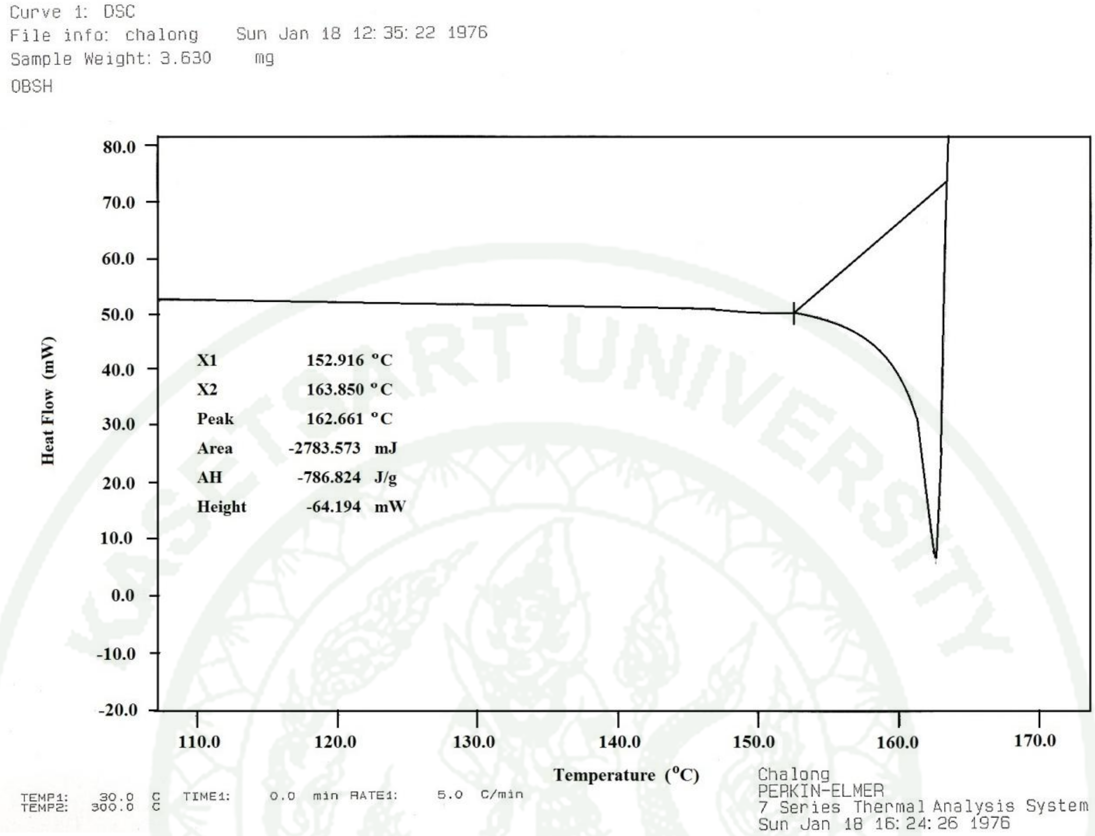
                       Thermal Transpiration : ON
                Effective Molecule Diameter (D) 3.5400 Å
            Effective Cell Stem Inner Diameter (d) 4.0000 mm
                       Last Po Acquired 781.81 mm Hg
          Additional Initialization Information Not Recorded.

                      BJH/DH Moving Average Size : 1

             Interaction Constant (K) 2.9600 nm^3 x kJ/mol

                                                       TISTR
```

Appendix Figure 2 Report on Pore volume of water hyacinth fiber



Curve 1: DSC
File info: chalong   Sun Jan 18 12:35:22 1976
Sample Weight: 3.630   mg
OBSH

X1 152.916 °C
X2 163.850 °C
Peak 162.661 °C
Area -2783.573 mJ
AH -786.824 J/g
Height -64.194 mW

TEMP1: 30.0 C TIME1: 0.0 min RATE1: 5.0 C/min
TEMP2: 300.0 C

Chalong
PERKIN-ELMER
7 Series Thermal Analysis System
Sun Jan 18 16:24:26 1976

Appendix Figure 3 Report on DSC of Blowing agent (OBSH)

CIRRICULUM VITAE

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BIRTHDAY PLACE : Trang, Thailand

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