## DEVELOPMENT OF HIGHLY EFFICIENT WALL INSULATION FROM RICE HUSK FOAM REINFORCED COMPOSITE



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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"I hereby declare that knave read this thesis and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Thermal Fluid) with Honours"

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## DEVELOPMENT OF HIGHLY EFFICIENT WALL INSULATION FROM RICE HUSK FOAM REINFORCED COMPOSITE

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This thesis is submitted to Faculty of Mechanical Engineering in partial fulfillment of the requirement for the award of Bachelor's Degree in

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#### ABSTRACT

This research is focus on development of highly efficient Rice Husk Ceramic Foam as alternative wall insulation materials for structure materials. The use of insulation wall in building construction causes some problems such as having high weight, high thermal conductivity and very reflective sound. Insulation is used because the heat which impinges the wall can be absorbed efficiently so heat losses instead give high energy loss. So the objective of this project is to develop of highly efficient Rice Husk Ceramic Foam as alternate heat insulation and sound insulation material for building. To achieve this objective, rice husk waste was used as filler material combine with other engineering chemical while the manufacturing process involve were burning process, mixing process, casting method, and drying process. The raw material to develop this ceramic foam is a fine aggregates which is the mixture of binder, filler, additives and blowing agents. The research methods are based on ASTM testing standards such are Density Testing, Cold Compression Test, Optical Microscope Test, Thermal Conductivity Test and Nitrogen Gas Absorption and Desorption Test Thermal Gravimetric Analyzer was according to ISO standard. Rice Husk Foam have lower thermal conductivity and higher compressive strength with density which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity. Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times. The pore distributions showed that the distribution 20 Å-250 Å is equal to 2 nm-25 nm which are categories in mesopores. According to previous research that been done, it suggested that the type of pore which are suitable for insulation material are mesopores. So it is proven through the Specimen RH-4 have meet the criteria of in term of pore type as mesopores. The objectives of this project to are achieved and it is proven that the Rice Husk Foam have better criteria as a thermal insulation material with the current thermal insulation material such as rigid Polyurethane and Polystyrenes.

#### ABSTRAK

Penyelidikan ini berfokuskan pada pembangunan Busa Sekam Padi (Rice Husk Foam) yang berkeupayaan tinggi sebagai alternatif bahan penebatan dinding untuk struktur bangunan. Penggunaan penebat dinding dalam bidang pembinaan mempunyai masalah seperti berat bahan penebat yang tinggi, mempunyai kekonduksian terma yang tinggi dan sangat reflektif. Oleh itu objektif projek ini adalah untuk membangunkan satu alternatif Busa Sekam Padi yang cekap sebagai penebat haba dan penebat bunyi. Untuk mencapai matlamat ini, sisa sekam padi digunakan sebagai bahan utama yang bertindak selaku bahan campuran dengan pegisi bahan kimia yang lain melalui proses pembuatan merangkumi proses pembakaran, proses campuran bahan mentah, kaedah tuangan, dan proses pengeringan. Bahan mentah untuk membangunkan Busa Sekam Padi ini adalah agregat halus yang merupakan campuran pengikat, pengisi, bahan tambahan dan ejen pemangkin. Kaedah penyelidikan adalah mengikut piawaian ujian ASTM seperti Ujian Ketumpatan, Ujian Mampatan Dingin, Ujian Optik Mikroskop, Ujian Kekonduksian Termal dan Analisis Penjerapan Gas Nitrogen manakala piawaian ISO digunakan untuk Analysis Termal Gravimetrik. Busa Sekam Padi mempunyai keberkondukan haba 1.95 kali lebih rendah baik daripada Poliuretana dan 2.54 kali lebih baik daripada Polisterin Tegar. Berdasarkan kekuatan mampatan, Busa Sekam Padi mempunyai kekuatan mampatan lebih tinggi berbanding Poliuretana and Polisterin Tegar yang mana 3.08 kali lebih baik. Analisis Penjerapan Gas Nitrogen menunjukkan agihan liang berdiameter diantara 20 Å-250 Å atau 2 nm - 25 nm yang dikategorikan sebagai mesoliang. Menurut hasil penyelidikan lepas, membuktikan jenis liang paling sesuai untuk bahan penebatan ialah yang mesoliang.Kesimpulannya, Busa Sekam Padi didapati memenuhi kehendak kriteria sebagai bahan penebat haba yang lebih baik dari bahan penebat yang terdapat di pasaran seperti Polisterin Tegar dan Poliuretana.

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## LIST OF ABBREVIATIONS

UTeM	=	Universiti Teknikal Malaysia Melaka
FKM	=	Faculty of Mechanical Engineering
ASTM	=	America Standard Testing for Material
ISO	=	International Organization for Standardization
JIS	=	Japanese Industrial Standard
IUPAC	=	International Union of Pure and Applied Chemistry
IEC	=	International Electrotechnical Commission
UK	=	United Kingdom
USA	=	United State of America
		External Wall Insulation Internal Wall Insulation
RHA	=	Rice Hush Ash
CaO	=	Calcium Oxide
$Al_2O_3$	=	Aluminium Oxide
CaSO <sub>4</sub>	=	Calcium Sulfate
H <sub>2</sub> O	=	Water
SiO <sub>2</sub>	=	Silica
K <sub>2</sub> O	=	Potassium Oxide
Na <sub>2</sub> O	=	Sodium Oxide
CO	=	Carbon Monoxide
Cl	=	Chlorine
MgO	=	Magnesium Oxide
Fe <sub>2</sub> O <sub>3</sub>	=	Iron Oxide
$P_2O_5$	=	Phosphorus Pentoxide
SO <sub>3</sub>	=	Sulfur Oxide
pfa	=	pulverized-fuel ash

SEM	=	Scanning Electron Microscopy			
DSC	=	Differential Scanning Calorimetry			
ASAP	=	Accelerated Surface Area and Porosimetry analyzer			
BET	=	Brunauer–Emmett–Teller			
TGA	=	Thermo Gravimetric Analyzer			
DTG	=	Derivative Thermal Gravimetric Analysis			
RHF	=	Rice Husk Foam			
FYP	=	Final Year Project			
AASHTO	=	American Association of State and Highway Transportation			
		Officials			



## LIST OF SYMBOLS



ρ	=	Density
P/Po	=	<b>Relative Pressure</b>



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#### **CHAPTER I**

#### INTRODUCTION

#### **1.1 BACKGROUND**

Insulation is an essential engineering for energy recovering. The ideal isolation thickness is that worth at which the expense is least and it incorporates the expense of protection material and cost of energy utilization over the life time of the constructing. Insulation actions as a barricade to heat flow and is absolutely vital to hold your home moderately hot in winter and cool in summer. A well-insulated and well-designed dwelling will provide year-round comfort, chopping chilling and heating accounts by up to half (ICANNZ, 2007). This, in turn, will decrease greenhouse gas emissions.

Nowadays, wall insulation becomes important in our life where 80% domestic properties in Malaysia have solid walls. These are geographically widespread and account for a wide range of properties, from tenement flats common urban areas to stone cottages in more rural locations. Wall insulation can make a significant impact on household energy consumption by combined with the advantages in thermal comfort. Whilst solid walls are widely assumed to be less energy efficient so we have to find a solution. The solution is wall insulation so has been established in United Kingdom (UK), UK Government Flagship Energy Efficiency Policies which been introduced in autumn 2012. (Energy Saving Trust, 2011).

#### **1.2 PROBLEM STATEMENT**

At the 21<sup>st</sup> century, the whole world now concerned about energy saves, emission to environment and green technology. However there are local developer and domestic properties' owner still lacking in this transformation and information. The effects of this unawareness cause the buildings not environmental friendly where it cost a lot of operation cost and emission to the nature. This, in turn, will decrease greenhouse gas emissions the heat which impinges the wall can be absorbed efficiently so heat losses instead give high energy losses. The current wall insulation material causes some problems such as having high weight, high thermal conductivity and very reflective sound. The invention of Rice Husk Foam Reinforced Composite is viewed as a solution for these problem, as it recovered the energy saving, cost reduction, thermal comfort and emission to the environment are reduce.



#### 1.4 SCOPE

The scopes of this research are to develop highly efficient wall insulation from Rice Husk Foam Reinforced Composite that have below qualification:

- i. Efficient in thermal insulation
- ii. Excellent adhesion to any surface
- iii. Cost effective
- iv. Strong yet lightweight
- v. Environment friendly

#### **CHAPTER II**

#### LITERATURE REVIEW

#### 2.1 INTRODUCTION

This chapter describe the detail descriptions about the raw material are describe in the aspect of usage to produce insulation. Furthermore, literature of method and result that gained from previous research of wall insulation were also

## presented in this chapter.

#### 2.2 WALL INSULATION

The energy utilization is disseminated around four essential divisions: mechanical, constructing (private/ business), transportation and farming zones. The raising segment is the most elevated energy purchaser zone. Energy utilization rate is step by step expanded because of urbanization, modern development and populace development. Populace development indicates getting more structures, which expands energy use. The hotness misfortunes in edifices ordinarily happen through outer dividers, roof, floor, windows and air penetration

The first time a building was insulated was in USA in 1880 when builders installed mineral wool in houses. From the 70s new and more effective insulation materials have been discovered (Çengel, 1998).Today, in the European market inorganic fibrous materials, glass wool and stone wool account for 60% of the insulation materials, and, organic foamy materials, expanded and extruded

polystyrene and to a lesser extent polyurethane accounts for some 27% (EURIMA, 2007). In Spain the three most common insulation materials used in buildings are polyurethane, mineral wool and polystyrene.

In Malaysia the inside insulation encasing requisition are ordinarily made by the sandwiches divider sort. The structure of outer divider is made by (9 cm - 12 cm) interior mortar, distinctive divider materials (Brick), encasing material and (2 cm - 3 cm) outer mortar. In this examination, the estimations were done for a two diverse sorts of dividers, which have been developed with plaster (1 cm - 2 cm), and insulation  $(1\text{ cm} - \infty \text{ cm})$ .

There are two types of wall insulation which are External Wall Insulation (EWI) and Internal Wall Insulation (IWI) is show in Figure 2.1.



Figure 2.1: Wall structure of EWI and IWI

Table 2.1 show the summary advantages and disadvantages of External Wall Insulation (EWI) and Internal Wall Insulation (IWI). By applying wall insulation we can reducing energy consumption and have a low thermal conductivity within a property which make us more comfortable and saving more living cost.

[		External Wall Insulation	Internal Wall Insulation
		• Lower risk of moisture build-up	• Can be cheaper, particularly if
		and condensation	done on Do It Yourselves(DIY)
		• Walls retain heat so lose heat less	basis
		slowly	• Can be applied room-by-room
		• Enhance structural integrity of	or just to certain rooms
	ges	building	• Heating has faster response
	anta	• Less disruption to occupants/ no	• Can improve interior decor of
	Adva	need for decanting	property
	7	• Can enhance exterior appearance	• Fewer restrictions on where in
			what types of properties it can
			be applied (e.g. can be applied
			more easily in high-rise blocks,
			conservation areas)
		• More expensive	• Potential problems with
		• Not applicable in many properties;	moisture build-up and
		building where it is desirable to	condensation
	S	retain original appearance, multi-	• Leads to cold bridging
	tage	occupancy properties	• Issues with accessing services
	van	• Restrictions on when work can be	• Loss of room sizes (unless
	isad	carried out (e.g. due to weather)	injection method or slim line
	D	• Require neighbor's agreement if	products used)
		joined properties. Can be	• Complex cornicing or fittings
		particularly difficult in blocks of	can be an issue with fixings
		flats.	internally.

Table 2.1: Advantage and Disadvantage of EWI and IWI.

#### 2.3 RICE HUSK FOAM REINFORCED COMPOSITE RAW MATERIALS

Insulations materials are materials or combinations of materials that are used primarily to provide resistance to heat flow. One feature shared by all insulating materials used in building applications is their low thermal conductivity, usually lower than 0.1 W/m K (Çengel, 1998).

In this research, to develop highly efficient wall insulation from rice husk foam reinforced composite which consist mix binder of raw materials such as:

- i. Rice Husk Ash (RHA)
- ii. Gypsum
- iii. Hydrated Lime
- iv. Aluminum Powder
- v. Methylcellulose

vi. Cement



#### 2.3.1 Rice Husk Ash (RHA)

Rice Husk Ash can consummate by incinerate rice husk. Rice Husk has several names where the most common name is husk, hull and chaff. Rice Husk is the outmost layer of protection encasing of rice gain. It is a yellowish color and has a convex shape. It is slightly larger than a gain of rice, thus the length up to 7 mm (H .B. Mahmud, 2005). It is lightweight, and has a ground bulk density of 340 kg /m<sup>3</sup> to 400 kg /m<sup>3</sup> whereby after incineration, only about 20% weight of rice husk are transformed RHA (H. B. Mahmud, 2005). Rice husk is the outer covering of this paddy. Nowadays, rice husk is generated from rice processing industries as a major agricultural by-product in many parts of the world, especially in developing countries. About 500 million tons of paddies are produced in the world annually (Kumar, 2012). During milling of paddy about 78 % of weight is received as rice, broken rice and bran (Kumar, 2012). Rest 22 % of the weight of paddy is received as Rice Husk. Rice husk is generated from rice processing is a waste material. These wastes can be

found as natural materials, by-products or industrial wastes; these materials are also obtained with requiring low cost, energy and time. Unfortunately, having technical benefits, most of those wastes are dumped into commercial return. Thus, due to growing environmental concern and the need to conserve energy and resources, utilization of industrial and biogenic wastes as supplementary material for wall insulation. Disposal of the rice husks is a big problem and open heap burning is not acceptable on environmental grounds, and so the majority of husk is currently going into landfill. The disposal of rice husks create environmental problem that leads to the idea of substituting RHA for silica in wall insulation manufactured. We choose rice hush ash to develop wall insulation because Rice Husk Ash (RHA) is a byproduct of the agricultural industry which contains high amount of silicon dioxide (SiO<sub>2</sub>) as in Table 2.2. The content of silica in the ash is about 92 %-97 % which provides several advantages such as, very abrasive, wears conveying elements very quickly, and also improved strength and durability properties (Karim. M. R., 2012).Besides, as far as the sustainability is concerned, it will also help to solve problems otherwise encountered in disposing of the wastes (Chandra, 1997).



Figure 2.2: Rice Husk incinerate process to Rice Husk Ash (RHA)

Chemical Composition	% Decibel (Db.)
SO <sub>2</sub>	86 - 97.3
K <sub>2</sub> O	0.58 - 2.5
Na <sub>2</sub> O	0.0 - 1.75
СО	0.2 - 1.5
Cl	trace - 0.42
МО	0.12 - 1.96
Fe <sub>2</sub> O <sub>3</sub>	trace - 0.54
P <sub>2</sub> O <sub>5</sub>	0.2 - 2.85
SO <sub>3</sub>	0.1 - 1.13

Table 2.2: Chemical composition of carbon-free rice husk ash (V.Saraswathy, 2008)

The husk, if burned at high temperatures within the presence of element, yields ash with a high content of silicon dioxide (more than 90 % by weight) that is seen because the most advantageous property of rice husk ash compared to ash obtained from burning different solid fuels (Muthadhi,A, 2007). However, the burning conditions of rice husk lead to completely different kinds of rice husk ash, the crystalline and amorphous forms, different forms benefit different applications, whereas amorphous silicon dioxide had been evidenced to be helpful within the cement, construction, and rubber industries (Mehta. PK, 1976), crystalline silicon dioxide has been found to be helpful for product like steel, ceramics and refractory bricks. Nonetheless, crystalline silicon dioxide is taken into account to be carcinogenic, then this might limit its uses within the future (Bronzeoak, Ltd, 2003).

It recommended that burning time and furnace setting have an effect on the rice husk ash properties, like the silicon dioxide type and also the expanse of ash particles (Hwang.CL, 1997). It may be aforesaid that burning rice husk at temperatures below 700 °C provides amorphous silicon dioxide that features a high expanse. However, the expanse is influenced by the holding time. Moreover, it indicates that once burning rice husk at temperatures over 800 °C, the ash obtained is in crystalline type.

In addition, amorphous silicon dioxide may be obtained by burning at temperatures less than 500 °C. It been reported that the combustion of rice husk at temperatures below 500 °C with the presence of oxygen in prolonged amount yields amorphous silica (Muthadhi.A,2007). It is proven that the properties of rice husk ash depend on the burning procedures for the husk. As simply mentioned on top of, completely different characteristics of rice husk ash square measure helpful in numerous applications. Therefore, it's vital to contemplate the suitable burning conditions of rice husk for specific uses of the ash.

#### 2.3.2 Gypsum

Gypsum is an evaporate mineral most commonly found in layered sedimentary sepulture in association with halite, anhydrite, sulfur, calcite and dolomite. Gypsum as in Figure 2.2 is a naturally occurring mineral that is made up of calcium sulfate and water ( $CaSO_4 + 2H_2O$ ) that is sometimes called as hydrous calcium sulfate. Gypsum is the most widespread sulfate mineral. The usages of Gypsum are manufacture of wallboard, dement, plaster of Paris, soil conditioning, and a hardening retarder in Portland cement. Although Gypsum's low hardness limits their durability. In this research, we used Gypsum as hardening retarder. Hardening retarders are admixtures that lower the rate of hydration, distribute the heat release over time and lower maximum temperature in the foam. The potential use of hardening retarders is to lower maximum temperature in massive structures, but by doing so the early strength will necessarily be lower than for reference wall insulation. The advantage of Gypsum is that it provides economical fire protection and sound control whereby it is a green and recyclable product (Townsend & McLendon, 2000). Though, Gypsum has a superior acoustics performance in terms of insulation with insulation rating up to 74 dB (A.Wallace, 1995).



Figure 2.3: Gypsum

#### 2.3.3 Hydrated Lime

Hydrated Lime as in Figure 2.4 is a type of dry powder made from limestone. Its chemical name is Calcium Hydroxide, or Ca (OH)<sub>2</sub>. Hydrated lime is a good bonding agent and is water-tight. It is also strongly alkaline, having a pH of 12.4. It can be used to neutralize acid, in water and sewage treatment, and to stabilize soil. One of the Hydrated Lime ability is reduce stripping. Subsequent research and

experience have demonstrated that the Hydrated Lime's benefit are much broader and include:

- i. Increased mix stiffness and reduce rutting.
- ii. Reduced oxidation and age-hardening effects.
- iii. Improve low temperature cracking resistance.



Figure 2.4: Hydrated Lime

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#### 2.3.4 Aluminium Powder

Aluminium powder is a highly flammable powder which is created by grinding aluminum into fine grains. Aluminium Powder has specialty in mechanical and fatigue properties is said to combine the strength, low density, corrosion resistance, high thermal resistance, processed to eliminate porosity, good heat insulation and improve bonding yielding properties. In this research Aluminium Powder in Figure 2.5 act as blowing agents where its special characteristics of these solvent-free blowing agents are the initial delayed reaction combined with high casting stability which is created by physical processes. Appropriate proportioning of the used quantity causes the mixture to expand.



Figure 2.5: Aluminium Powder

#### 2.3.5 Methylcellulose

Methylcellulose (or methyl cellulose) as in Figure 2.6 is a chemical aggregate which is drawn from the cellulose of vegetables whereby in this research it act as additives. Methylcellulose assists command some vital properties of a formulation such as rheology, dispersion, and water demand and water retention. It has numerous functional benefits, including high consistency and workability with reduced stickiness, high standing strength and high yield. Air voids steadiness, significance that the binder maintains density and consistency during application. Air voids in the render smaller its density, making it smoother and advancing workability. Other than that, it assists to stabilizing entrained air, assisting sustains consistency of the render and prolonging the open time. This can continue the timeframe for leveling after squirting and endows users to work more effectively throughout successive spraying and leveling phases. It has good workability, so that binder can disperse and level effortlessly without sticking to devices. The binders should be very simple to spread and should adhere to walls, not devices and help to achieve an optimum balance between standing strength (which assists stops sagging) and shear stability (which support leveling). The additives furthermore support quick consistency development. Temperature tolerance, allowing binder to keep water and avoid drying out before curing is entire. In alignment to be hardened correctly, it should retain water throughout the curing process. Only when this occurs the render can come to its full power promise and ensure cement hydration. Supplementing cellulose ethers improves water retention especially critical at high temperatures. These properties support expanded open times, good workability and productive curing achieving their design flexural, tensile and adhesive strength in a kind of climates, enabling us to increase standardization of our products. It furthermore helps to negligible retardation, to reduce curing times and support effectiveness on site.



Figure 2.6: Methylcellulose

#### 2.3.6 Cement

In this research we using Portland Cement Type I where as we name it as Cement because it can provide adequate levels of strength and durability. ASTM C-150 defines Portland Cement as "hydraulic cement (cement that not only hardens by reacting with water but also forms a water-resistant product) produced by pulverizing clinkers consisting essentially of hydraulic calcium silicates, usually containing one or more of the forms of calcium sulfate as an intern ground addition. Type of Portland cement and its application as in table 2.3.ASTM C 150 and AASHTO M 85 have specified certain physical requirements for each type of cement. These properties include:

- i. Fineness
- ii. Soundness
- iii. Consistency
- iv. Setting time
- v. Compressive strength
- vi. Heat of hydration
- vii. Specific gravity
- viii. Loss of ignition

Each one of these properties has a leverage on the presentation of cement in concrete. The fineness of the cement sways the rate of hydration. Greater fineness ingreases the surface available for hydration, imitating larger early strength and more rapid generation of heat (the fineness of Type III is higher than that of kind I cement). It furthermore will outcome in higher ultimate strength and lower early-strength gain. Soundness, which is the ability of hardened cement paste to retain its volume after setting, can be characterized by assessing the expansion of mortar bars in an autoclave (ASTM C 191, AASHTO T 130). The compressive strength of 50-mm mortar cubes after 7 days (as assessed by ASTM C 109) should not be less than 19.3 MPa for Type I cement. Other physical properties included in both ASTM C 150 and AASHTO M 95 is specific gravity and untrue set. False set is a significant loss of plasticity soon after blending due to the formation of gypsum or the formation of ettringite after blending. In numerous situations, workability can be restored by remixing solid before it is cast.

	Classification	Characteristics	Applications
Туре І	General purpose	Fairly high C3S content for good early strength development	General construction (most buildings, bridges, pavements, precast units, etc.)
Туре II	Moderate sulfate resistance	Low C3A content (<8%)	Structures exposed to soil or water containing sulfate ions
Type III	High early strength	Ground more finely, may have slightly more C3S	Rapid construction, cold weather concreting
Type IV	Low heat of hydration (slow reacting)	Low content of C3S (<50%) and C3A	Massive structures such as dams. Now rare.
Type V	High sulfate	Very low C3A	Structures exposed to high levels of sulfate ions
White	White color	No C4AF, low-MgO -	properties similar to Type I)

Table 2.3: General features of the main types of Portland cement.

#### 2.3.7 Water

Water is a chemical substance with the chemical formula  $H_2O$ . Its molecule contains one oxygen and two hydrogen atoms connected by covalent bonds. Water is a polar compound and it is a good solvent. Water is odorless, tasteless, transparent liquid that is colorless in small amounts but shows a blue tinge in large quantities.

Water is a main constituent in concrete; it chemically reacts with cement with process hydration to produce the desired properties of concrete. Process hydration can be simplifying that reaction between water and cement in the mixture. Mixing water larger quantity with cement will upsetting the water cement ratio and affect the strength on concrete.

Furthermore, water used for curing and mixing shall be clean and free from injurious amounts of acids, salts, oils, alkalis, sugar, and organic materials. Water is most used material on the earth. Potable water satisfies to be used in concrete mixture.

#### 2.4 NITROGEN GAS ABSORPTION

Analysis using this technique will provide some importance parameters such as surface area, isotherms, hysteresis type, BET constant, the volume pores, the average pore diameter, BET surface area, and volume monolayer of the sample under study.





Figure 2.7: Six types of BET absorption

The Six types of BET absorption can classified as in Table 2.4. This Classification is done after IUPAC in 1984.Types II, IV and VI can be measured by BET method (interaction adsorptive-adsorbent > adsorptive-adsorbate) while Types III, V have weak interactions between gas and adsorbent.

Table	2.4:	The	Six	types	of	BET
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Types of BET	Description				
Absorption					
	Pores are typically microporus with the exposed surface				
Tuno I	residing almost exclusively inside the micropores, which				
I ype I	once filled with adsorbate, leave little or no external surface				
	for further adsorption.				
	Most frequently found when adsorption occurs on				
Tran a H	nonporous powders or powders with diameters exceeding				
I ype II	micropores. Inflection point occurs near the completion of				
	the first adsorbed monolayer.				
Type III	Characterised by heats of adsorption less than the adsorbate				
	heat of liquification, adsorption proceeds as the adsorbate				
	interaction with an adsorbed layer is greater than the				
	interaction with the adsorbent surface.				
Type IV	Occur on porous adsorbents with pores in the range of 1.5 –				
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	100nm. At higher pressures the slope shows increased				
	uptake of adsorbate as pores become filled, inflection point				
	typically occurs near completion of the first monolayer.				
Type V	Are observed where there is small adsorbate-absorbent				
	interaction potentials (similar to type III), and are also				
	associated with pores in the $1.5 - 100$ nm range.				
Type VI	Classification as the combination of mesopore and macropore				

Whereas, shows the adsorption isotherm of Type VI is multiple layers solid phases over ultra-micropore uniform. However, the isotherm Type V and VI are very difficult found.

### 2.4.2 Hysteresis

Desorption is a process contrary to the adsorption process. Desorption of adsorbed substances occurring after the saturation point is reached. Evaporation of measure usually occurs at a lower pressure than the capillary condensation which that turn to produce hysteresis. Hysteresis refers to the shape of the pores in a substance. Figure 2.8 shows four types of hysteresis which have been identified according to the IUPAC classification (G.Leofanti, 1997).

> Type H1 hysteresis demonstrate solid cylindrical open uniform. Meanwhile, the H2 type hysteresis was characterized as non-uniform open cylindrical shape. Hysteresis of H3 and H4 consists of solid through aggregation or agglomeration process which produce pores such as small cracks form either in the plate or square shape like a cube.H3 type hysteresis shows irregular interstitial form which is contra with Type H4 hysteresis (G.Leofanti, 1997).



Figure 2.8: Four Type hysteresis



Table 2.5: Pore classification based on Pore Diameter

Type of Pore	Diameter of Pore
Micropore	0 nm - 2 nm diameter
Mesopore	2 nm - 50 nm diameter
Macropore	>50 nm diameter

Material with micro-sized pores show interactions that strong in the narrow pore walls with adsorption relative pressure which cause of the volume adsorbed in the pores increases. Pore with meso-sized, showed an increase in the volume of the adsorption effect of capillary condensation hysteresis loop back through the formation. Macro-sized pores indicate the formation of a monolayer on the multiple pressure layers which relatively low and the adsorption at high relative pressures. Pore which is too wide there is a weak interaction between the material adsorbed and the adsorbent resulting in adsorption is unlikely to happen (G.Leofanti, 1997), (Bakar, 1997).

### 2.5 **PREVIOUS FINDING**

Wall insulation is any kind of insulating material that is adhered to or paste inside walls. Normally, the major function of partition insulation is to boost the weather effectiveness of the space by making it easier to heat and cool the room. Counting on the kind of wall insulation used, the product may furthermore help to soundproof the space and minimize the amount of disturbance that goes into or escapes from the room. Insulating material is a material that reduces or prevents the transmission of heat or sound.

This study that had done about New Thermal Insulation Boards Made from Coconut Husk and Bagasse (Panyakaew.S, 2011) describes the production of low density thermal insulation boards made from coconut husk and bagasse without the use of chemical binding additives. The aim of this study was to develop a thermal insulation with lower environmental footprint than conventional materials. The hot pressing method was used. Hot pressing is the process in which heat and pressure is applied to a mattress composed of fibres and resin to mould the final product (N. Norberto). In this study, binderless insulation boards made from coconut husk and bagasse were manufactured using hot pressing with pressure of 14.7 MPa. Previous studies suggested that the coconut husk and bagasse binderless boards can be made by using hot pressing method at temperatures above 180 °C for 10 minutes (Mari. E. L., 1996), (Dam, 2004), (Widyorini, 2005), (Mobarak, 1982), (Shen, 1986). Therefore, to investigate the effect of pressing temperature on physical properties of the insulation boards, three temperature settings (180 °C, 200 °C and 220 °C) were used for pressing coconut husk and three temperature settings (160 °C, 180 °C and

200 °C) for bagasse. The lower temperatures for bagasse follow previous study (Laemsak, 1996), (N. Okuda, 2004), (J. Xu, 2006) which suggested that sugar containing lignocellulosic materials such as oil palm frond and kenaf core can be made into binderless boards using lower temperatures (140 °C -180 °C) due to their high hemicellulose content. It was expected that bagasse, also with high hemicellulose content, could be made into binderless boards using the similar lower temperature setting. In order to investigate the effect of pressing duration, three pressing durations were used (7, 10 and 13 minutes) for both coconut husk and bagasse. Mechanical properties of the coconut husk and bagasse insulation boards were measured for comparison with the standard employed in Thailand: JIS A 5905: 2003 Insulation Fiberboards.

It was found that the bagasse insulation board with a density of  $350 \text{ kg/m}^3$ , using a 13 minute pressing time at a temperature of 200 °C, met all of the requirements except for swelling thickness. Thermal conductivity of the coconut husk and bagasse insulation boards was measured according to ISO 8301 and this suggested that both insulation boards have thermal conductivity values ranging from 0.046 WmK to 0.068 W/mK which were close to those of conventional insulation materials such as cellulose fibers and mineral wool. Bagasse is more porous and easily absorbs moisture during storage before the production and thus bagasse was dried to lower moisture content: firstly being sun dried for three days to a moisture content of 10 % and then further oven dried at 80 °C to a moisture content of 6 % - 7 %. The coconut fibers were cut to lengths of 8 mm -10 mm. The fiber to pith ratio for the production of coconut husk insulation boards was 80:20 by weight. For the production of bagasse insulation boards, the average length of large particles was around 20 mm – 40 mm and the average length of small particles was 50:50 by mass.

Silkworm Cocoon as Natural Material and Structure for Thermal Insulation (J.Zhang,2013). Silkworm cocoons are choose as an important biological materials that protect silkworms from environmental threat and predator attacks. Silkworm cocoons are able to provide significant buffer against temperature changes outside of the cocoon structure. The research findings are of relevance to the bio-inspired design of new thermo-regulating materials and structures. There are four types of

silkworm cocoons such are B. Mori and S. Cynthia cocoons were purchased from silk rearing houses in Northeast India: A. pernyi cocoons were collected from Northern China and A. Mylitta cocoons were collected from Central India. They were received as stifled cocoons, commonly used prior to reeling silk filament for textile applications. Calcium oxalate crystal powder was purchased from Sigma Aldrich and used as received.

The cocoon walls go through Scanning Electron Microscopy (SEM) where it is cut into square specimens with the dimension of 5 mm x 5 mm (J.Zhang,2013) which were then attached to conductive carbon tape on Aluminium stubs. The specimens were observed by a Supra 55 VP scanning electron microscope after sputter coated with gold. Both the cocoon inner and outer surfaces were investigated. The single silk fiber diameter was calculated from 50 measurements on SEM micrographs using image analysis software. The average single fiber diameter was deter- mined from the histogram graphs of size distribution. The temperature was measured both inside and outside of the cocoons (3–5 mm close to the outer surface) using two needle-type temperature probes. Each temperature probe is 1.3 mm in diameter and 35 mm in length, with two sensors located 15 mm apart (the first sensor is 7 mm from the needle tip). For each measurement, one temperature probe was placed into the cocoon through the proximal end from which the moth usually escapes and the other probe was attached onto the outer surface of the cocoon by a small piece of tape, with sensors deliberately uncovered.

Four types of thermal conditions (I–IV) were tested. For condition I, a gradual rise of temperature was introduced by placing the cocoons into an oven (Binder) with isothermal temperature setting (at 37 °C, 45 °C and 50 °C, respectively) (F.Chen,2012); for condition II, a sudden drop of temperature was introduce d by transferring he warmed cocoons that have nearly reached equilibrium from the oven to air. The other two thermal conditions were applied by heating a tube furnace (Lindberg/BlueM) with two different ramp rates (2 °C/min for thermal condition III and 4 °C/min for thermal condition IV) from ambient temperature to 50 °C, respectively, while the cocoons were located inside. The temperature profiles were established based on the average data from temperature readings (every second) recorded from two sensors (of each thermal probe) located inside and outside of

cocoons, respectively. The thermal conductivity of four types of cocoon walls was obtained using TA Q200 Differential Scanning Calorimetry (DSC) through the modulated DSC method in accordance with the procedure stated in ASTM: E 1952-11. A detailed illustration of the principle can be found in (S.M.N.Marcus & R.L.Blaine, 1994). Temperature was modulated with an amplitude of  $\pm 1^{\circ}$ C and a period of 60 s. Nitrogen gas flowed at a rate of 50 mL/min while the average test temperature and heat capacity were recorded. Silkworm cocoon walls contribute to the thermal insulation of cocoons. To further understand the thermal properties of cocoon walls, temperature modulated differential scanning calorimetry (DSC) was used to measure the thermal conductivity, thermal resistance, thermal diffusivity and thermal absorptivity at the temperature of 7°C, 20 °C, 27 °C, 47 °C, 67 °C and 87 °C as shown in Figure 2.9.



Figure 2.9: Thermal properties of silkworm cocoon walls: (a) thermal conductivity, (b) thermal resistance, (c) thermal diffusivity, and (d) thermal absorptivity

Thermal conductivity is the materials' ability to conduct heat. Dry heat transfer through insulation fabrics may involve the conduction in the solid phase constituting the insulation, the radiation in the material and the heat transfer in the air confined in the insulation (BK.Venkanna, 2010). Because the complexity of the fiber orientation, fiber construction, porosity and bulk density, the thermal conductivity of textiles can hardly be predicted and only be measured directly.

As shown in Figure 2.9. (a), the thermal conductivity values of cocoon walls are within the range from 0.0106 W/mK to 0.0653 W/mK; the calcium oxalate crystal pellet was also measured and its thermal conductivity is higher than 0.2 W/mK, which can be as high as 27 times of that of the cocoon walls. Thermal resistance is directly related to thermal insulation. In Figure 2.9. (b), it can be seen that the B. Mori cocoon wall had the highest thermal resistance thus the best thermal insulation property. Thermal resistance is directly proportional to the sample thickness and inversely proportional to thermal conductivity. Thermal absorptivity is a surface structure-related property and provides an objective measurement of the warm-cool feeling of textiles. If the thermal absorptivity is high, the textile fabric gives a cooler feeling at first contact with the skin (E.Onofrei,2011).

Although this is not directly related to cocoon biological functions, it is interesting to observe that the cocoons walls of A. Pernyi, A. Mylitta and S. Cynthia types exhibited higher thermal absorptivity values as shown in Figure. 2.9, which may correlate to the comparatively thermal conductive crystals that deposited on the outer surfaces of these cocoons. The thermal insulation properties of both domestic (B. Mori and S. Cynthia) and wild silkworm cocoons (A. Pernyi and A. Mylitta) were investigated. Under both steady and non-steady state thermal conditions, the inner temperature of silkworm cocoons showed significant thermal damping characteristics against sudden changes in outside temperature. However, the wild cocoons exhibited a higher level of thermal buffer than the domestic ones. Thermal property measurements were conducted on cocoon walls by a temperature modulated DSC method. The lower thermal diffusivity of wild cocoons partially contributes to the lower temperature changing rate and longer equilibrium time experienced by A. Pernyi and A. Mylitta cocoons. The raw materials that involved to produce the Environment-Friendly Thermal Insulation Material from Sunflower Stalk, Textile Waste and Stubble Fibers (H.Binici,2013) are:

- i. Stubble, as a result of agricultural production, which is cut out the remaining root crops, soil and deviation.
- ii. Textile wastes
- iii. Cotton waste obtained from the textile factories.
- iv. Sunflower stalks
- v. Urea-formaldehyde resins are formed by the reaction of urea and formaldehyde. The synthesis of a urea-formaldehyde resin takes place in two stages.
- vi. Plaster

Insulation boards are manufactured with plaster as a Binder. Grinding machine used in agriculture and textile waste is ground using a mixture of sunflower stalks were produced. With these materials are used as plaster binder. The mixing ratio of the insulation material is given as  $3.30 \text{ cm} \times 40 \text{ cm} \times 2.5 \text{ cm}$  in size insulation boards to according to the application, respectively. Sound and thermal insulation values of the rooms are measured. These rooms are located next to the isolation and untreated identical rooms and other rooms are created with the same points, 60 dB noise measurements were made. In addition to the rooms are identical heaters heating and cooling times were measured. Thermal conductivity coefficients of insulation material have been measured with QTM-500. Thermal conductivity of insulating material has been measured according to ASTM C 1113-09.

Ultrasonic sound penetration velocity coefficient to determine the specific relationship between the wave speed and density of the materials. The amount of space inside the material increases, the Ultrasonic sound penetration velocity coefficient decreases. In this study, tests were carried out for mixtures used as a binder in plaster and epoxy. Thermal conductivity coefficients of insulation material made with sunflower stalk fiber, sunflower stalk part sponge, cotton waste, textile waste and stubble fiber epoxy materials were 0.1642 W/mK. Water absorption and unit weight values Insulation material of unit weight and water absorption values 0.72 kg/m<sup>3</sup> and 71 % were found, respectively. Ultrasonic sound penetration velocity

coefficient Ultrasonic sound penetration velocity coefficient test, conducted in accordance with ASTM C 597. Throughout this research, it is proof that the thermal insulation material through Compressive strength 0.312 MPa, with Highest Ultrasonic penetration velocity 912.86 m/s. It Thermal Conductivity value is 0.0728 W/m K.

Based on the previous researches that have been conducted, this research was made to develop highly efficient wall insulation material from Rice Husk Foam reinforced composite. The expected result from this research are to develop Rice Husk Foam Reinforced Composite as an alternative choice wall insulation material which that have lower thermal conductivity than Silkworm cocoon as natural material and structure for thermal insulation, An environment-friendly thermal insulation material from sunflower stalk, textile waste and stubble fibers , New thermal insulation boards made from coconut husk and bagasse ,Polyurethane Foam and Polystyrene Board where it can be implement in domestic and commercial properties.

IMRANSYAKIR

### **CHAPTER III**

#### **METHODOLOGY**

### **3.1 INTRODUCTION**

The aim of this project is to to study and develop an efficient wall insulation material from Rice Husk Foam reinforced composite according to ASTM and ISO standards. This chapter demonstrated the method of how to carry out the research and experimentin order to achieve the objectives of this project.

### **3.2 RESEARCH METHOD**

This research is a lab scale study to vertex the embodiment of the constructing Rice Husk Foam. The preparations of specimens are based on International Organization for Standardization (ISO) standards for Thermal Gravimetric Analyzer (TGA/DTG)-ISO/IEC 17025:2005 to choose the most suitable temperature for the burning process of Rice Husk in furnace to gain the best quality Rice Husk Ash. Rice Husk burn to ashes at 650°C for 3600 seconds in the furnace and cooled in the furnace for 24 hours. Then the Rice Husk Ash is grind using Rotary Mill and the fineness RHA is differentiating by using Vibrato Mill Shaker. It was found out that the fineness of RHA as that retained on 45 µm sieve was about 21.87%, which conformed to grade A of dry pulverized-fuel ash (pfa) based on ASTM C430: 2008. The influence of fineness of RHA is important as it will influence the compressive strength (Shimizu.G., 1990).The Nitrogen Gas Absorption and Desorption Test ASTM D4222-03 was

conducted to determine type of pore distribution and to identify the type of pore. After that all the raw materials are hybridize well-balanced according to the composition ratio to enhance binder of Rice Husk Foam. The binder is poured into the mould that design and scorched in the oven at 60 °C for 6 hours. In this research, we investigate of properties of Rice Husk Foam such as Density Testing ASTM C380-79, Cold Compression Test ASTM C39, Optical Microscope Test ASTM E210 -63 and Thermal Conductivity Test ASTM C1470-06.Nitrogen Gas Absorption and Desorption Test ASTM D4222-03 was conducted to determine type of pore distribution and to identify the type of pore.



### 3.3 FLOW CHART



Figure 3.1: Process flow for the whole project

#### 3.4 **COMPOSITION OF RAW MATERIAL**

In Table 3.1 shows the composition of raw material in term of percentages that we used to construct Rice Husk Foam. This research is conducted according to American Society for Testing and Materials (ASTM) and International Organization for Standardization (ISO) Standards. After we fame to produce Rice Husk Foam it will be tested according to ASTM standards. The cost of raw materials to carry out this research is RM 564.20 is shown in Table 3.2.

	Sample	Gypsum	Cement	RHA	CaO	Al <sub>2</sub> O <sub>3</sub>	Methylcellulose	H <sub>2</sub> O
	Code	(%)	(%)	(%)	(%)	(%)	(%)	(%)
	RHF-4	40	25	20	10	5	20	150
	RHF-3	30	25	30	10	5	20	150
	RHF-2	20	25	40	10	5	20	150
	RHF-1	10	25	50	10	5	20	150
		RA		TS IS				
Table 3.2: Costs of raw material								

Table 3.1: Compositions of raw material

Table 3.2: Costs of raw material

No.	Raw Materials	Quantity (kg)	Price per kg (RM)	Price (RM)
1.	Hydrated Lime	10	34.80	348.00
2.	Aluminium Powder	10	10.40	104.00
3.	Rice Husk	100	0.86	86.00
4.	Gypsum	10	1.60	16.00
5.	Methylcellulose	10	0.39	3.90
6.	Cement	10	0.63	6.30
		Total Cost		564.20

### 3.5 MATERIAL PREPARATION

#### 3.5.1 Rice Husk Ash (RHA)

Rice Husk burn to ashes at 650 °C for 3600 seconds in the furnace and cooled in the furnace for 24 hours. Then the Rice Husk Ash is grind using Rotary Mill and the fineness RHA is differentiating by using Vibrato Mill Shaker. The average particle size of RHA is 45  $\mu$ m is recommended.

### **3.6 PROCEDURE TO SILHOUETTE RICE HUSK FOAM**

 Thermal Gravimetric Analyzer (TGA/DTG)-ISO/IEC 17025:2005 to choose the most suitable temperature for the burning process of Rice Husk in furnace to gain the best quality Rice Husk Ash.

ii. Rice Husk burn to ashes at 650°C for 3600 seconds in the furnace and cooled in the furnace for 24 hours. iii. Then the Rice Husk Ash is grind using Rotary Mill and the fineness

RHA is differentiating by using Vibrato Mill Shaker. The average particle size of RHA is 45µm is recommended.

- iv. After that all the raw materials are hybridize well-balanced according to the composition ratio to enhance binder of Rice Husk Foam.
- v. The binder is poured into the mould that design and scorched in the oven at 60°C for 6 hours.
- vi. After 6 hours the specimen are take out from the oven and it is ready for Density Testing ASTM C380-79, Cold Compression Test ASTM C39, Optical Microscope Test ASTM E210 -63,Thermal Conductivity Test ASTM C1470-06 and Nitrogen Gas Absorption and Desorption Test ASTM D4222-03.

### 3.7 TESTING STANDARD

All the specimens will go through the physical and mechanical properties testing such are Density Testing ASTM C380-79, Cold Compression Test ASTM C39, Optical Microscope Test ASTM E210 -63 and Thermal Conductivity Test ASTM C1470-06 with IS0 standards for Thermal Gravimetric Analyzer (TGA/DTG)-ISO/IEC 17025:2005 the most suitable temperature that suggested to burn Rice Husk into Rice Husk Ash (RHA).Nitrogen Gas Absorption and Desorption Test ASTM D4222-03 was conducted to determine type of pore distribution and to identify the type of pore.

### 3.7.1 Thermal Gravimetric Analyzer

Thermo Gravimetric Analyzer (TGA/DTG)-ISO/IEC 17025:2005 is a technique within which the mass of a substance is monitored as a function of temperature or time because, the sample specimen is subjected to a controlled temperature program in a very controlled atmosphere. The purpose of TGA is to measures a sample's weight because it is heated or cooled in a furnace. A TGA consists of a sample pan that's supported by an exactitude balance. That pan resides in a furnace and is heated or cooled throughout the experiment. The mass of the sample is monitored throughout the experiment. A sample purge gas controls the sample setting. This gas is also inert or a reactive gas that flows over the sample and exits through an exhaust. These instruments will quantify loss of water, loss of solvent, loss of plasticizer, decarboxylation, pyrolysis, oxidation, decomposition, weight % filler, amount of metallic catalytic residue remaining on carbon nanotubes,

and weight % ash. All these quantifiable applications are usually done upon heating, but there are some experiments where information may be obtained upon cooling. The temperature range from 30  $^{\circ}$ C to 800  $^{\circ}$ C. The scanning rate are 10  $^{\circ}$ C/min.

### 3.7.2 Cold Compression Test

In this Cold Compression Test which follow the ASTM C39 standard whereby the specimens will be in cylindrical size of 0.051 m diameter and the height is 0.1 m. The results of this test will provide information to check methodology square measure used as a basis for internal control of proportioning, mixing, and placing operations; determination of compliance with specifications and management for evaluating effectiveness of admixtures. The compressive strength is measure when the cracks of the Rice Husk Foam by using Universal Testing Machine as in Figure 3.2.

The compressive strength is calculated from the failure load divided by the cross sectional area resisting the load and reported in units of pound-force-per square inches (psi) in US then are convert to customary units of Mega Pascal (MPa) in SI units which are tabulated.



Figure 3.2: Universal Testing Machine

The Universal Testing Machines connected to the computer as shown in figure 3.2. The parameter of Rice Husk Foam sample such as length, width and height were entering into the program. The results that we obtained from are graphical data which is Compressive Load (N) versus Compressive Extension (mm). Auxiliary results that we gained from are maximum load, compressive strength, modulus, energy at local peak maximum, and strain 1 at yield (offset 0.2 %).

### 3.7.3 Density Test

Density Test which follow the ASTM C380-79 test whereby the mass of a substance divided by its volume. In the United States, density is accepted as the weight of a substance divided by its volume. Foam density is often expressed as pounds per cubic foot or kilograms per cubic meter according to test method ASTM C380-79.Density equation are as in equation 3.1.



Where m is the weight dry sample (kg), V is the volume cylinder (m<sup>3</sup>), and  $\rho$  is density of the Rice Husk Foam sample (kg/m<sup>3</sup>).The sample size are in 0.051m cylindrical with height 0.01 m.

### 3.7.4 Thermal Conductivity Test

The KD2 Pro as in Figure 3.3 is an absolutely portable field and research laboratory thermal properties analyzer. This experiment conducted according to Thermal Conductivity Test ASTM C1470-06 It uses the transient line heat supply methodology to measure thermal conductivity, resistivity, diffusivity, and specific heat. The KD2 Pro Thermal Properties Analyzer permits the user to settle on an automatic mode wherever the reading is displayed directly or a manual mode wherever you'll transfer the raw values from every reading for more analysis employing a program as needed by ASTM D5334-08 standards. ASTM D5334-08 is

a significantly updated version of the quality check methodology for Determination of Thermal conduction of Soils and Rock by Thermal Needle Probe Procedure. It represents the most effective practices in accordance with current analysis in heat and mass transfer. The KD2 complies with all theoretical assumptions upon that ASTM D5334-08 is predicated, however makes full use of technologically superior sensors and microprocessor-based analysis over the home-cured probes and simplistic pencil-and-paper analysis mechanism.





#### 3.7.5 Optical Microscope Test

An optical microscope creates a magnified image of an object specimen with an objective lens and magnifies the image further more with an eyepiece to allow the user to observe it by the naked eye. This Optical Microscope Test is conducted based on ASTM E210 -63 test method. Optical Microscopic as in Figure 3.4 is one of the most widely used non-destructive failure analysis tool because it is rapid and convenient in locating and identifying most external defects.



Figure 3.4: Modern Microscope component configuration

When used in conjunction with micro-sectioning, it can be a very powerful tool. But in this research the Optical Microscopic is used to identify minerals; determine rock type and crystallization sequence. This type of microscope which creates a magnified image by combining an objective lens making an inverted real image and an eyepiece making an erect virtual image is called a compound microscope. The observation optical system in an optical microscope is commonly standardized on this compound microscope. Meanwhile, such type of microscope that directly observes an inverted real image magnified with an objective lens is called a single microscope. A microscopic observation on a TV monitor, recently popularized increasingly, uses the way of directly capturing this inverted real image with a CCD camera, thereby being comprised of a simple microscope optical system.

#### 3.7.6 Nitrogen Gas Absorption and Desorption Test

The tools used are Micromeritics ASAP 2010 to analyze surface area and porosity of the samples using the technique in nitrogen gas absorption at temperature of 30 °C until 300 °C with ramp rate of 10 °C/min. Samples of 1.0 g dried in an oven used to analysis and takes 4 to 5 hours. This test was conducted according to ASTM D4222-03.Two processes are carried out in this analysis of sample preparation process and absorption - desorption of the samples. Sample preparation process requires a dry sample included into a tube which evacuated and heated to 120 °C degassing and remove moisture (Syakir, 2001). Adsorption - desorption process was done by passing the sample on a sample of helium gas at a pressure of 30 mmHg to remove moisture. Then nitrogen gas is flowed through to process Adsorption - desorption process in a period of time. Some important information is obtained as isotherms, hysteresis type, and volume monolayer, BET constant, the volume pores, the average pore diameter and BET surface area of the sample under study.



## **CHAPTER 4**

### **RESULT AND DISCUSSION**

## 4.1 THERMAL GRAVIMETRIC ANALYSIS (TGA/DTG)

In Figure 4.1 shows the Thermal Gravimetric Analysis (TGA) and Derivative Thermal Gravimetric Analysis (DTG) of the Rice Husk sample according to ISO/IEC 17025:2005. There are four types of weight-loss regions found during the analysis.



Figure 4.1: Thermal Gravimetric Analysis (TGA) and Derivative Thermal Gravimetric Analysis (DTG) of the Rice Husk sample

The first region of weight-loss occurs at 30.33 °C - 262.62 °C indicating 5.981% loss which consists of the decomposition of moisture such as water. The second weight-loss appears at 30.33 °C - 262.62 °C with 58.289% loss. The

third weight–loss appears at 262.62 °C - 393.88 °C with 23.872 % loss. The fourth weight–loss appears at 393.88 °C – 797.33 °C with 11.674 % loss However, the maximum rate of weight-loss occurred at temperature 393.88 °C which was found 23.872 % loss. There is no weight-loss observed after 445.98 °C

When rice husk is burnt at temperatures lower than 700 °C, it shows a cellular microstructure which is highly reactive (Chindaprasirt.P., 2008), (Sensale.G., 2006).RHA is a highly pozzolanic material; it contains non-crystalline silica and high specific surface area that are accountable for its high pozzolanic reactivity. By heating at higher temperatures, the unburned carbon can be removed from the ashes (Krishnarao,2001), but this leads to the crystallization of the ash from amorphous silica into cristobalite or tridymite.

At lower temperature, the amorphous nature of rice husk ash silica will be occurred (Ahmed, 2007). The most common form of crystalline silica found is quartz (Zakharov, 1993). But more research has been focused on the formation of cristobalite and tridymite (Kordatos, 2008). The transformation has been reported that  $\alpha$  -quartz can be form at below 573 °C, B-quartz at 573 °C - 870 °C, B-tridymite at 870 °C -1470 °C, and B-cristobalite at 1470 °C -1710 °C (A.R.

West, 1999). It is decided show then the temperature of 650 degrees centigrade and 60 minutes burning time are the best combination (A.A.Ramezanianpour, 2009).

### 4.2 DENSITY TESTING

Equation 4.1 is used to calculate the density according to ASTM C380-79 where m represent the weight of the specimen (kg), V is the volume of specimen in cylinder (m<sup>3</sup>), and  $\rho$  is density of the specimen (kg/m<sup>3</sup>).The highest composition of Gypsum 40% have better density compare to others where it has 400.00 kg/m<sup>3</sup>, follow by RH-3 359.4441 kg/m<sup>3</sup>, RH-2 349.8294 kg/m<sup>3</sup>, and RH-1 330.2590 kg/m<sup>3</sup>.

Density, 
$$\rho = \frac{m}{v}$$
 (3.1)

Specimen	Height (m)	Diameter (m)	Mass (x 10 <sup>-4</sup> kg)	Volume (x 10 <sup>-4</sup> m <sup>3</sup> )	Density (kg/m <sup>3</sup> )
RH-4	0.07045	0.05741	72.9468	1.82367	400.00
RH-3	0.07063	0.05740	65.6592	1.82769	359.4441
RH-2	0.07137	0.05742	64.6530	1.84813	349.8294
RH-1	0.07082	0.05741	60.5417	1.83316	330.2590

Table 4.1: Density Test Result

### 4.3 COLD COMPRESSION TEST

RH-4 have highest compressive strength and Maximum Load value which are 0.0770 MPa and 0.1500 kN compare to RH-3,RH-2 and RH-1.So whenever the composition of gypsum is higher in the mixture, it influence the compressive strength and maximum load that the Rice Husk Foam can withstand. The details of the compressive strength and maximum load as in Table 4.2.

KAN				
Sample Data	RH-4	RH-3	RH-2	RH-1
Compressive Strength (MPa)	0.0770	0.0757	0.0727	0.0621
Maximum Load (kN)	0.1500	0.1438	0.1276	0.1041

Table 4.2: Compressions Strength Test Result



Figure 4.2: Compressive Load versus Compression Extension (RHF Wall Insulation)
4.4 NITROGEN GAS ABSORPTION AND DESORPTION TEST

### 4.4.1 Nitrogen Adsorption-Desorption Isotherm Linear Plot

Figure 4.3/shows the Nitrogen Adsorption-Desorption Test ASTM D4222-03 was conducted for isotherm at 30 °C to 300 °C of the Rice Husk Foam. The effects of temperatures provided different performances of the nitrogen isotherms. All the nitrogen isotherm curves significantly increase in the low-relative pressure  $(P/P_o < 0.2)$  indicating the presence of micropore structures with the Type I isotherm. However, at higher relative pressure  $(P/P_0 > 0.2)$ , the knee of nitrogen isotherm curves became more open and steadily increased to the maximum relative pressure (P/P<sub>o</sub>  $\approx$  1.0). These results suggested that large amount of nitrogen was adsorbed during the adsorption process indicating the presence of wider porosity (mesopores) with the Type IV isotherm. The entire relative pressure ranges were suggested that the nitrogen adsorption exhibited a combination of the Type I and Type IV isotherms, indicating the attendance of micro- and mesoporosity in the Rice Husk Foam. Additionally, the presence of hysteresis loops with H3 type in the desorption isotherms clearly shown at the higher relative pressure ( $P/P_0 > 0.3$ ). This hysteresis was which indicated to the welldeveloped mesopores. These phenomena assumed may as a well micropore and mesopores.



Figure 4.3: Nitrogen adsorption-desorption isotherm linear plot

### 4.4.2 Pore Size Distributions

The distributions of mesoporosity were estimated by the BJH method. Figure 4.4 shows the plots of pore diameter distribution of the Rice Husk Foam starting from 0-20 nm. It is showed that from 20 Å-250 Å is equal to 2 nm 25 nm which are categories in mesopores where the criterion for mesopores is that the range of pore diameter must be between 2 nm-500 nm (G.Leofanti,1997). According to previous research that been done, it suggested that the type of pore which are suitable for insulation material are mesopores (Gonçalves, 2007), (S.Chandrasekhar, 2006).So it is proven through the Specimen RH-4 have meet the criteria of in term of pore type as mesopores. The specimen RH-4 surface area, pore volume and pore diameter is shown in Table 4.3.



Figure 4.4: Pore size distributions

Table 4.3: Specimen RH-4 surface area, pore volume and pore diameter

	Specimen	Surface Area( $m^2/g$ )	Pore Volume $(m^3/g)$	Pore Size( <i>nm</i> )
	RH-4	14.54	0.0221	5.547
$\sum \cup \vee$				

## 4.5 OPTICAL MICROSCOPE TEST

The Optical Microscope Test ASTM E210 -63 was carried out to gain the image by locating and identifying most external defects about the Rice Husk Foam. From Figure 4.3 and Figure 4.4, it is acknowledge that the pore at the specimen can efficiently sustain the temperature in the space that insulated and prevent it from outside temperature enter the insulated space.





Figure 4.6: Optical Microscope Test Result at 20  $\mu m$ 

### 4.6 THERMAL CONDUCTIVITY TEST

From Figure 4.5, it is show that the specimen RH-4 which contain 40% of gypsum have lower thermal conductivity value compare to other specimens. Lower thermal conductivity value influence the heat transfer across pores is ordinarily slow and inefficient. Furthermore, gaseous convection within the pores is also comparatively ineffective.



Figure 4.7: Thermal Conductivity, (W/mK) vs Specimen

# 4.7 COMPARISON BETWEEN RICE HUSK FOAM, POLYURETHANE FOAM AND POLYSTYRENE

By referring to rigid polyurethane foam data according to Federation of European Rigid Polyurethane Foam Association and Polystyrene data according to Saving Energy Trust the comparison was done to proof that the Rice Husk Foam have lower thermal conductivity and higher compressive strength with density which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity. Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times.

Table 4.4: Thermal Conductivity, Compressive Strength and Density Result of RiceHusk Foam, Polyurethane Foam and Polystyrene

Sample Data	RH-4	Rigid Polyurethane	Polystyrene (Styrofoam)	
Density (kg/m <sup>3</sup> ) Compressive Strength (MPa)	(400.0000 0.0770	62/0000 0.0250	349.8294 0,0250	$\mathbb{D}$
Thermal Conductivity (W/m.K)	0.0118	0.0230	0.0300	

### **CHAPTER 5**

#### **CONCLUSION & RECOMMENDATION**

### 5.1 CONCLUSION

In this research, it is proven that Sample RH-4 have better density, lightweight, compressive strength, maximum load and thermal conductivity compare to specimen RH-3, RH-2 and RH-1. While specimen RH-4 compare with Polyurethane Foam and Polystyrene Board in term of thermal conductivity it is proven that specimen RH-4 have lower thermal conductivity value which is 0.0018 W/mK and higher compressive strength which is 0.0770 MPa The Rice Husk Foam have lower thermal conductivity and higher compressive strength with density which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity.

Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times. When the thermal conductivity value low, the less heat the material will transfer which are one of the main criteria that needed by an insulation material. If a material density increase, the thermal diffusivity value also increases. Through Nitrogen Gas Absorption and Desorption Test, the data that gained showed the Specimen RH-4 Surface Area 14.54 m<sup>2</sup>/g and Pore Volume 0.0221 m<sup>3</sup>/g and Pore Size 5.547 nm. The pore distribution show that the RH-4 are in in mesopores where it is in 2 nm- 25 nm which meet the criteria of mesopores in the range of 2 nm-50 nm.

The objectives of this project to study and develop an efficient wall insulation material from Rice Husk Foam reinforced composite according to ASTM and ISO standards are achieved and it is proven that the Rice Husk Foam have better criteria as a thermal insulation material with the current thermal insulation material such as rigid Polyurethane and Polystyrenes.

### 5.2 **RECOMMENDATION**

It is suggested that the Rice Husk Foam use as internal wall insulation in domestic and commercial properties as alternative material thermal insulation material as show in Figure 5.1 which can benefit the consumer in term of thermal comfort ability and ergonomic so that can save the cost of operation.



Figure 5.1: Wall structure with Rice Husk Foam Reinforced Composite Insulation

Wall

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### Appendix A(Gannt Chart of PSM I)



Appendix A(Gannt Chart of PSM II)

Appendix B



Appendix C

3Flex 3.00

3Flex Version 3.00 Serial # 161 Unit 1 Port 1

Page 1

Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep:StageTemperature (°C)Ramp Rate (°C/min)Time (min)1901060230010480

#### **Summary Report**

Surface Area

Single point surface area at/R/Po = 0.268898241 13:6960 m<sup>2</sup>/g BET Surface Area: 14:5365 m<sup>2</sup>/g Pore Volume

Single point adsorption total pore volume of pores less than 460.267 Å diameter at P/Po = 0.956432262:  $0.020160 \text{ cm}^3/\text{g}$ 

Single point desorption total pore volume of pores less than 413.926 Å diameter at P/Po = 0.951356031: 0.022133 cm<sup>3</sup>/g

t-Plot micropore volume: -0.000746 cm3/g

#### Pore Size

Adsorption average pore diameter (4V/A by BET): 55.4735 Å

Desorption average pore diameter (4V/A by BET): 60.9024 Å

BJH Adsorption average pore diameter (4V/A): 94.327 Å

BJH Desorption average pore diameter (4V/A): 70.574 Å

#### **DFT Pore Size**

Volume in Pores	<	20.02 Å	:	0.00052 cm <sup>3</sup> /g
Total Volume in Pores	<=	317.92 Å	:	0.00668 cm <sup>3</sup> /g
Area in Pores	>	317.92 Å	:	6.023 m <sup>2</sup> /g
Total Area in Pores	>=	20.02 Å	:	8.166 m²/g

3Flex 3.00

3Flex Version 3.00 Serial # 161 Unit 1 Port 1 Page 2

Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

Isotherm Tabular Report					
Relative Pressure (P(Po)	Absolute Pressure (mmHg)	Quantity Ela Adsorbed (cm <sup>3</sup> /g STP)	psed Time (h.min)	Saturation Pressure (mntHg)	
			01:03	760,931213	
0.026416100	20.095617	2.2327	01:32	760.733704	
0.055722970	42.392742	2.6602	01:36	760.776794	
0.071790738	54.609550	2.8477	01:40	760.676819	
0.094735118	72.061935	3.0723	01:43	760.667603	
0.117042071	89.024033	3.2693	01:47	760.615662	
0.139582088	106.169975	3.4414	01:50	760.627502	
0.162017469	123.221237	3.6065	01:54	760.542908	
0.184569063	140.355164	3.7686	01:58	760.447937	
0.206950278	157.374634	3.9175	02:02	760.446594	
0.229377622	174.409439	4.0644	02:05	760.359436	
0.251906689	191.523392	4.1995	02:07	760.294983	
0.274423324	208.629059	4.3368	02:10	760.245361	
0.296816716	225.635071	4.4757	02:12	760.183167	
0.319443589	242.829010	4.6177	02:15	760.162415	
0.342059563	260.000885	4.7633	02:18	760.104126	
0.364192651	276.828827	4.9068	02:21	760.116455	
0.386766620	293.968079	5.0548	02:23	760.065796	
0.409267464	311.079193	5.2180	02:26	760.087769	
0.431755758	328.163574	5.3726	02:29	760.067627	
0.454072724	345.140167	5.5367	02:31	760.098877	
0.477044684	362.564209	5.7058	02:34	760.021484	
0.499321335	379.478241	5.8662	02:36	759.988037	
0.521855062	396.557770	6.0373	02:38	759.900208	
0.544281322	413.603210	6.2180	02:41	759.907043	
0.566682442	430.603912	6.4000	02:43	759.868103	
0.589356230	447.803864	6.5963	02:45	759.818665	
0.611800205	464.830627	6.7889	02:48	759.775208	
0.634162739	481.799103	6.9931	02:50	759.740479	
0.656474803	498.795197	7.2157	02:52	759.808594	
0.679222261	516.038574	7.4522	02:55	759.749207	
0.701776133	533.113037	7.7006	02:57	/59.66253/	
0.724300528	550.230652	7.9606	02:59	759.671753	
0.746383216	567.004150	8.2585	03:01	759.668945	
0.769259248	584.268127	8.5948	03:04	759.520447	

3Flex 3.00

3Flex Version 3.00 Serial # 161 Unit 1 Port 1 Page 3

Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM<br/>Completed: 2014-04-28 3:33:48 PM<br/>Report time: 2014-04-29 9:52:02 AM<br/>Sample mass: 0.4293 g<br/>Cold free space: 60.2332 cm³Analysis adsorptive: N2<br/>Analysis bath temp.: -195.803 °C<br/>Thermal correction: No<br/>Warm free space: 16.9688 cm³ Measured<br/>Equilibration interval: 5 s<br/>Sample density: 1.000 g/cm³Low pressure dose: None<br/>Automatic degas: YesSample density: 1.000 g/cm³

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

Isotherm Tabular Report						
	Relative	Absolute	Quantity	Elapsed Time	Saturation	
	Pressure (P/Po)	Pressure	Adsorbed	(h:min)	Pressure	
		(mmHg)	(cm³/g_STP)	$77 \Lambda$	( <del>mmHg) -</del>	
	0 701702007	601 252563	8 0334	02/08	750 255652	
	0.81/2000	619 365845	0.2034	03.00	750 252126	
$      \vee$	0.886501689	635 058716	9 7040	03.10	759 1840 81	
	0.859205809	652 234436	10 1566	$\int 03.10$	759 113159	
	0.881352442	669 027893	10.1000	03.12	759 092346	
	0.903833936	686.055359	11.2666	03:17	759.050232	
	0.926511245	703,199585	11,9469	03:19	758.975769	
	0.956432262	725,794495	13.0095	03:21	758.856140	
	0.983025308	745.964111	14,7702	03:24	758.845276	
	0.997814683	756.939697	20.0810	03:36	758.597473	
	0.978093077	741.902344	16.1047	03:40	758.519165	
	0.951356031	721.586304	14.2826	03:43	758.481873	
	0.920706438	698.342346	13.4033	03:45	758.485352	
	0.887562578	673.194763	12.8407	03:48	758.475830	
	0.855920386	649.179932	12.4251	03:50	758.458313	
	0.809953990	614.239563	11.8795	03:52	758.363525	
	0.784862955	595.218384	11.5958	03:54	758.372375	
	0.739012878	560.408386	11.1459	03:57	758.320190	
	0.714785272	542.002991	10.9163	04:00	758.273865	
	0.668200071	506.616455	10.5297	04:02	758.180786	
	0.621068169	470.882294	10.1622	04:05	758.181335	
	0.574425590	435.519287	9.8005	04:07	758.182251	
	0.527701229	400.078857	9.4315	04:10	758.154114	
	0.484015152	366.952820	8.1959	04:13	758.143250	
	0.461396608	349.762482	6.8438	04:18	758.051697	
	0.435282639	329.940521	6.1225	04:22	757.991455	
	0.410115674	310.853363	5.7863	04:24	757.965088	
	0.363468567	275.464783	5.4139	04:28	757.877869	
	0.315920883	239.391922	5.1005	04:31	757.759094	
	0.269076138	203.867218	4.8098	04:35	757.656250	
	0.222040519	168.215317	4.5219	04:39	757.588379	
	0.175233749	132.736923	4.2308	04:43	757.484924	
	0.131849547	99.869675	3.9406	04:47	757.451782	
	0.105847086	80.169296	3.7381	04:51	757.406738	
	0.085809801	64.982651	3.5654	04:55	757.287048	



3Flex 3.00

3Flex Version 3.00 Serial # 161 Unit 1 Port 1

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

#### BET Report

BET surface area: 14.5365 ± 0.0427 m<sup>2</sup>/g Slope: 0.293350 ± 0.000868 g/cm3 STP Y-intercept: 0.006075 ± 0.000143 g/cm3 STP -C: 49,290144 Qm: 3.3397 cm3/g 5/TP Correlation coefficient. 0.9999650 Molecy/ar cross-sectional area: 0.1620 nm 1/[Q(Po/P - 1)] Relative Quantity Pressure Adsorbed (P/Po) (cm<sup>3</sup>/g STP) 0.055722970 2.6602 0.022183 0.071790738 2.8477 0.027160 0.094735118 3.0723 0.034062 0.117042071 0.040546 3.2693 0.139582088 3.4414 0.047139 0.162017469 3.6065 0.053610 0.184569063 3.7686 0.060060 0.206950278 3.9175 0.066612 0.229377622 4.0644 0.073234 0.251906689 4.1995 0.080183

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480





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3Flex Version 3.00 Serial # 161 Unit 1 Port 1

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes

Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

Langmuir Report

Langmuir surface area: 22.7309 ± 0.6475 m<sup>2</sup>/g Slope: 0.191483 ± 0.005454 g/cm3 STP

Y-intercept: 9.834 ± 0.714 mmHg·g/cm<sup>3</sup> STP

b: 0:019472 1/mmHg Qm: 5.2224 cm3/g STP Correlation coefficient: 0.997172 Molecular cross-sectional area 0.1620 nm<sup>2</sup> Pressure P/Q Quantity (mmHg) Adsorbed (mmHg·g/cm<sup>3</sup>

	(cm³/g STP)	STP)
54.609550	2.8477	19.177
72.061935	3.0723	23.455
89.024033	3.2693	27.230
106.169975	3.4414	30.851
123.221237	3.6065	34.167
140.355164	3.7686	37.243
157.374634	3.9175	40.172
174.409439	4.0644	42.911
191.523392	4.1995	45.606

3Flex 3.00

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3



Page 10 Serial # 161 Unit 1 Port 1 Sample: Rice Husks **Operator: Pua** Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP Started: 2014-04-28 10:26:07 AM Analysis adsorptive: N2 Completed: 2014-04-28 3:33:48 PM Analysis bath temp.: -195.803 °C Report time: 2014-04-29 9:52:02 AM Thermal correction: No Sample mass: 0.4293 g Warm free space: 16.9688 cm<sup>3</sup> Measured Cold free space: 60.2332 cm<sup>3</sup> Equilibration interval: 5 s Low pressure dose: None Sample density: 1.000 g/cm3 Automatic degas: Yes Comments: 2/3 Sample prep: Stage Temperature (°C) Ramp Rate (°C/min) Time (min) 1 90 10 60 2 300 480 10 **BJH Adsorption Pore Distribution Report Faas Correction** Harkins and Jura  $t = [13.99 / (0.034 - log(P/Po))]^{0.5}$ Diameter range: 17.000 Å to 3,000.000 Å Adsorbate property factor: 9.53000 Å Density conversion factor: 0.0015496 Fraction of pores open at both ends: 0.00 Rore Diameter Average Incremental Cumulative Incremental Cumulative Range (Å) Diameter (Å) Pore Volume. Rore Volume Pore Area Pore Area  $(cm^{3}/g)$  $(cm^{3}/g)$  $(m^2/g)$ (m²/g) 8751.0 - 1148.7 0.274 0.274 1271.2 0.008709 0.008709 1148.7 - 459.0 548.2 0.219 0.003001 0.011710 0.493 459.0-277.3 323.7 0.231 0.001867 0.013577 0.724 277.3-214.0 237.1 0.001234 0.014812 0.208 0.932 214.0 - 174.7 190.1 0.001135 0.015947 0.239 1.171 174.7 - 148.0 158.9 0.000946 0.016893 0.238 1.409 148.0 - 127.9 136.3 0.000866 0.017759 0.254 1.663 127.9 - 112.9 119.4 0.000778 0.018537 0.261 1.924 112.9-100.7 106.0 0.000723 0.019260 0.273 2.197 100.7 - 90.8 95.2 0.000668 0.019928 0.281 2.477 90.8 - 82.5 86.2 0.000676 0.020604 0.314 2.791 82.5 - 75.7 0.000600 3.096 78.8 0.021204 0.305 75.7 - 69.8 72.5 0.000513 0.283 3.378 0.021717 69.8-64.6 67.0 0.000493 0.022210 0.295 3.673 64.6-60.1 0.000471 62.2 0.022680 0.303 3.976 60.1 - 56.1 0.000445 0.308 57.9 0.023126 4.283 56.1 - 52.5 54.2 0.000402 0.023528 0.297 4.580 52.5 - 49.3 0.000376 4.876 50.8 0.023904 0.296 49.3-46.4 47.7 0.000392 0.024296 0.329 5.205 46.4 - 43.7 45.0 0.000358 0.024655 0.319 5.524 43.7 - 41.3 42.4 0.000360 0.025015 0.339 5.863 41.3 - 39.1 40.1 0.000335 0.025350 0.334 6.197 39.1 - 37.0 0.000308 38.0 0.025658 0.324 6.522 37.0-35.0 36.0 0.000334 0.025991 0.371 6.893 35.0-33.2 0.000327 0.383 7.276 34.1 0.026318 33.2-31.6 32.3 0.000296 0.026614 0.366 7.642 31.6-30.0 0.000327 0.026941 0.426 8.068 30.7

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

Р	ore Diameter	Average	Incremental	Cumulative	Incremental	Cumulative
	Range (Å)	Diameter (Å)	Pore Volume	Pore Volume	Pore Area	Pore Area
			(om³/g)	(cm³/g)	(m²/g)	(m³/g)
	30.0 - 28.5	29.2	0.000275	0.027216	0.377	8.445
	28.5 - 27.1	\\ // 27. <b>?</b> \	0.000264	) 0.027480	0.381	8.826
2020	27. <del>1 - 25.7</del>	26. <del>3</del>	0.000266	0.027746		9.230
	25.7 - 24.4	25.0	0.000251	0.027996	0.401	9.631
	24.4 - 23.1	23.7	0.000240	0.028236	0.404	10.035
	23.1 - 21.9	22.5	0.000227	0.028463	0.404	10.439
	21.9 - 20.8	21.3	0.000213	0.028676	0.399	10.838
	20.8 - 19.6	20.2	0.000252	0.028928	0.501	11.339
	19.6 - 18.5	19.0	0.000248	0.029176	0.522	11.861
	18.5 - 17.4	17.9	0.000283	0.029459	0.631	12.492





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3Flex Version 3.00 Serial # 161 Unit 1 Port 1 Page 14

Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

#### **BJH Desorption Pore Distribution Report**

Faas Correction

Harkins and Jura t = [ 13.99 / ( 0.034 - log(P/Po) ) ] ^ 0.5

Diameter range: 17.000 Å to 3,000.000 Å Adsorbate property factor: 9:53000 Å Density conversion factor: 0:0015496 raction of pores open at both ends: 0.00

Pore Diameter Range (Å)	Average Diameter (Å)	Incremental Pore Volume (cm <sup>3</sup> /g)	Cumulative Pore Volume (cm <sup>3</sup> /g)	Incremental Pore Area (m²/g)	Cumulative Pore Area (m²/g)
8752.5 - 896.5	972.7	0.006645	0.006645	0.273	0.273
896.5 - 414.1	494.1	0.003165	0.009810	0.256	0.530
414.1 - 259.2	300.8	0.001558	0.011368	0.207	0.737
259.2 - 185.5	209.6	0.001008	0.012376	0.192	0.929
185.5 - 146.2	160.8	0.000762	0.013138	0.190	1.119
146.2 - 111.7	124.0	0.001055	0.014192	0.340	1.459
111.7 - 98.9	104.5	0.000568	0.014760	0.217	1.676
98.9-81.6	88.4	0.000918	0.015678	0.415	2.091
81.6-74.6	77.8	0.000481	0.016159	0.248	2.339
74.6-63.8	68.3	0.000815	0.016974	0.477	2.816
63.8 - 55.5	59.0	0.000795	0.017769	0.539	3.355
55.5-48.9	51.7	0.000813	0.018582	0.629	3.984
48.9-43.4	45.8	0.000862	0.019444	0.753	4.738
43.4 - 39.1	41.0	0.003756	0.023200	3.663	8.401
39.1 - 37.1	38.1	0.004386	0.027586	4.607	13.008
37.1 - 35.0	36.0	0.002098	0.029683	2.330	15.338
35.0-33.1	34.0	0.000724	0.030408	0.852	16.190
33.1 - 29.9	31.3	0.000432	0.030840	0.552	16.742
29.9-27.0	28.3	0.000205	0.031045	0.290	17.032
27.0-24.4	25.5	0.000138	0.031183	0.217	17.250
24.4-21.9	23.0	0.000113	0.031296	0.197	17.446
21.9-19.6	20.6	0.000093	0.031390	0.182	17.628
19.6 - 17.4	18.3	0.000101	0.031491	0.221	17.848





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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

#### Porosity Distribution by Model: N2 - DFT Model Method: Non-negative Regularization: 0.03160 Standard Deviation of Fit: 0.02405 cm<sup>3</sup>/g STP

Volume in Pores	<	20.02 Å	:	0.00052 cm³/g
Total Volume in Pores	<=	317.92 Å	:	0.00668 cm <sup>3</sup> /g
Area in Pores	>	317.92 Å	:	6.023 m²/g
Total Area in Pores	>=	20.02 Å	:	8.166 m²/g

Pore Size Table					
Pore Width	Cumulative	Incremental	Cumulative	Incremental	
(A)	Volume	Volume	Area	Area	
	(cm³/g)	tcm?#gt ~	(m²/g)	-{m²/g)	
20.02	0.00052	0.00000	0.000	0.000	
	0.00052	0.00000	0.000	0.000	
		0.00016		$\sim$	
20.20	0.00072	0.00010	0.102	0.120	
27.34	0.00090	0.00024	0.337	0.175	
23.43	0.00110	0.00022	0.400	0.145	
34 31	0.00150	0.00020	0.010	0.120	
36.99	0.00174	0.00017	0.813	0.092	
40.03	0.00193	0.00019	0.909	0.096	
43.25	0.00216	0.00023	1.016	0.107	
46.64	0.00246	0.00029	1.142	0.126	
50.40	0.00277	0.00031	1.265	0.123	
54.33	0.00305	0.00028	1.368	0.103	
58.80	0.00332	0.00028	1.462	0.094	
63.44	0.00357	0.00025	1.540	0.078	
68.45	0.00381	0.00024	1.611	0.071	
73.99	0.00408	0.00026	1.682	0.071	
79.88	0.00437	0.00029	1.755	0.073	
86.32	0.00469	0.00032	1.830	0.075	
93.11	0.00499	0.00030	1.894	0.064	
100.61	0.00521	0.00022	1.938	0.044	
108.66	0.00540	0.00019	1.973	0.035	
117.23	0.00557	0.00017	2.001	0.028	
126.53	0.00572	0.00016	2.026	0.025	
136.71	0.00590	0.00017	2.052	0.025	

3Flex 3.00	3Flex Version 3.0 Serial # 161 Unit	0 t 1 Port 1		Page 18
Sample: Rice Hus Operator: Pua Submitter: Thayalla File: C:\3Flex Started: 2014-04-28 1 Completed: 2014-04-28 3 Report time: 2014-04-29 9 Sample mass: 0.4293 g Cold free space: 60.2332 cm <sup>3</sup> Low pressure dose: None Automatic degas: Yes	ks h UTeM data\000-201.SMP 0:26:07 AM ::33:48 PM 1:52:02 AM	Analysis adsorptiv Analysis bath ter Thermal correctio Warm free spa Equilibration inte Sample den	ve: N2 np.: -195.803 ° on: No ace: 16.9688 ci rval: 5 s sity: 1.000 g/cr	°C m³ Measured n³
Comments: 2/3				
Sample prep: Stage Te 1 2	mperature (°C) 90 300	Ramp Rate (°C/ 10 10	/min)	Time (min) 60 480
	Pore Siz	ze Table		
Pore Width C (Å)	Cumulative Incren Volume Volu (cm³/g) (cm³	nental Cumula ime Area <sup>3</sup> /g) (m²/g	ative Incre a A g) (m	mental rea <sup>12</sup> /g)
147.61 159.41 172.10 185.86 200.69 216.60 233.93 252.52 272.71 294.51 317.92	0.00609 0.00627 0.00641 0.00649 0.00656 0.00663 0.00668 0.00668 0.00668 0.00668	0.00019 0.00018 0.00014 0.00006 0.00007 0.00005 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000	2.077 2.100 2/117 2(126 2.132 2.138 2.143 2.143 2.143 2.143 2.143 2.143	0.026 0.023 0.016 0.009 0.006 0.006 0.004 0.000 0.000 0.000 0.000 0.000

3Flex 3.00	3Flex Ve Serial #	rsion 3.00	I	Page 19
Sample: Rice Operator: Pua Submitter: Thay File: C:\3F Started: 2014-04- Completed: 2014-04- Report time: 2014-04- Sample mass: 0.4293 g Cold free space: 60.2332 Low pressure dose: None Automatic degas: Yes Comments: 2/3	Husks allan UTeM Flex\data\000-207 28 10:26:07 AM 28 3:33:48 PM 29 9:52:02 AM cm <sup>3</sup>	1.SMP Analys Analys Therm War Equilibr Sa	is adsorptive: N2 is bath temp.: -195.80 nal correction: No m free space: 16.968 ration interval: 5 s imple density: 1.000 g	)3 °C 8 cm³ Measured g/cm³
Sample prep: Stage	Temperature (°(	C) Ram	ip Rate (°C/min)	Time (min) 60
2	300		10	480
		Porosity Distribut	ion by	
	Method: No	Model: N2 - DFT n-negative Regula	Model arization: 0.03160	
	Standard [	Deviation of Fit: 0.	02405 cm³/g STP	
		Isotherm Table		
Relative Pressure (P/Po)	Experimental Quantity Adsorbed (cm <sup>3</sup> /g STP)	Fitted Quantity Adsorbed (cm³/g STP)	Absolute Residual (cm³/g STP)	Relative Residual
0.073040441 0.082588248 0.092847057 0.103808001 0.115456402 0.127772301 0.140730694 0.154301897 0.168452203 0.183144197 0.198337302 0.213988706	2_8605 2.9560 3.0546 3.1562 3.2566 3.3527 3.4499 3.5500 3.6536 3.7588 3.8608 3.9642	28247 2.9346 3.0487 3.1620 3.2704 3.3723 3.4675 3.5685 3.6528 3.7839 3.8612 3.9368	0.0358 0.0214 0.0068 -0.0058 -0.0138 -0.0196 -0.0176 -0.0186 0.0007 -0.0251 -0.0004 0.0274	0.012515 0.007247 0.001914 0.001914 0.004231 -0.005843 -0.005096 -0.005232 0.000200 -0.006666 -0.000111 0.006912
0.230053306	4.0686	4.0895	-0.0209	-0.005133
0.263235897 0.280259013 0.297506303 0.314930797 0.332486212 0.350127310 0.367810607 0.385494202 0.403138310 0.420704991 0.438158900	4.2683 4.3728 4.4800 4.5890 4.7014 4.8156 4.9303 5.0464 5.1736 5.2958 5.4197	4.2386 4.3884 4.4667 4.6167 4.7014 4.7905 4.9505 5.0474 5.1474 5.3089 5.4090	0.0298 -0.0156 0.0133 -0.0276 0.0000 0.0251 -0.0202 -0.0011 0.0262 -0.0132 0.0106	0.006971 -0.003572 0.002963 -0.006016 0.005209 -0.004091 -0.000210 0.005066 -0.002490 0.001961
0.455466807	5.5470	5.5774	-0.0304	-0.005486
0.472598106 0.489524394 0.506219923	5.6731 5.7957 5.9170	5.6721 5.7638 5.9387	0.0010 0.0319 -0.0217	0.000179 0.005512 -0.003667

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Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

3Flex 3.00

Sample prep: 1 2	Stage	Temperature (°0 90 300	C) Ram	p Rate (°C/min) 10 10	Tim	e (min) 60 480
			Isotherm Table			
	Relative Pressure (P/Po)	Experimental Quantity Adsorbed (cm³/g STP)	Fitted Quantity Adsorbed (cm³/g STP)	Absolute Residual (cm <sup>3</sup> /g STP)	Relative Residual	
	0.522661209 0.538827300 0.554699600 0.570261598 0.585499227 0.600400090	6.0437 6.1739 6.3019 6.4302 6.5628 6.6911	6.0243 6.2200 6.3018 6.3831 6.5852 6.6668	0.0195 -0.0461 0.0001 0.0471 -0.0223 0.0243	0.003219 -0.007467 0.000021 0.007328 -0.003402 0.003626	
	0.614954293 0.629153311 0.642990828	6.8164 6.9455 7.0795	6.8622 6.9468 7.0340	-0.0458 -0.0013 0.0455	-0.006718 -0.000182 0.006420	
	0.656461716 0.669562697	7.2156 7.3505	7.2364 7.3285	-0.0209 0.0220	-0.002890 0.002995	
	0.682291925 0.694648683	7.4852 7.6211 7.7540	7.5257 7.6204 7.7151	-0.0406 0.0007	-0.005419 0.000096	
	0.718248010	7.8883	7.9115	-0.0232	-0.002937	
	0.740377605 0.750900388	8.1726 8.3243	8.2052 8.2916	-0.0326 0.0327	-0.003988 0.003933	
	0.761068285	8.4736 8.6190	8.5051 8.5880	-0.0316 0.0310	-0.003728 0.003600	
	0.780362606	8.7606 8.8985 9.0350	8.8161 8.8977 8.9801	-0.0555 0.0008 0.0549	-0.006331 0.000093 0.006079	
	0.806798697 0.814971626	9.1737 9.3121	9.2001 9.2853	-0.0264 0.0268	-0.002874 0.002883	
	0.822837889 0.830405474	9.4508 9.5896	9.4757 9.5654	-0.0249 0.0242	-0.002630 0.002523	
	0.837682605	9.7264 9.8621 9.9966	9.7475 9.8411 10.0158	-0.0210 0.0210 -0.0192	-0.002163 0.002128 -0.001923	
	0.857852995 0.864050388	10.1286 10.2587	10.1095 10.2791	0.0191 -0.0203	0.001820	
	0.869998574 0.875705481	10.3896 10.5213	10.3695 10.5441	0.0201 -0.0229	0.001938 -0.002173	

3Flex 3.00	3Flex V Serial #	ersion 3.00 # 161 Unit 1 Port 1		Page 21
Oper Subm Started: Completed Report time Sample mass: Cold free space Low pressure dose: Automatic degas:	ator: Pua ator: Pua itter: Thayallan UTeM File: C:\3Flex\data\000-20 2014-04-28 10:26:07 AN I: 2014-04-28 3:33:48 PM I: 2014-04-29 9:52:02 AM I: 2014-04-29 9:52:02 AM I: 0.4293 g I: 60.2332 cm <sup>3</sup> None Yes	01.SMP I Analysis Analysi Therma Warn Equilibr Sa	adsorptive: N2 s bath temp.: -195. I correction: No n free space: 16.96 ation interval: 5 s mple density: 1.000	803 °C 688 cm³ Measured 0 g/cm³
Comments: 2/3				
Sample prep: Stage 1 2	e Temperature (* 90 300	°C) Ramp	Rate (°C/min) 10 10	Time (min) 60 480
		Isotherm Table		
Rela Pres (P/ 0.88 0.89 0.90 0.90 0.90 0.90 0.90 0.90	ative Experimental Quantity   Adsorbed (am³/g STP)   1179392 10.6536   6428118 10.7871   1459525 10.9212   6281302 11.0536   0900900 11.1835   5325770 11.3087   9563184 11.4273   3620114 11.6552   1219707 11.7695   4430817 12.1216   4542298 12.2321   7517405 12.3378   0361619 12.4388   3080306 12.5353   5678592 12.6276   8161721 12.7158	Fitted Quantity Adsorbed (cm <sup>3</sup> /g STP) 10.6308 10.8117 10.8966 11.0756 11.1618 11.3228 11.4133 11.5505 11.6466 11.7782 11.8779 12.0148 12.1130 12.2378 12.3321 12.4255 12.5175 12.6100 12.7035	Absolute Residual (cm³/g STP) 0.0227 -0.0246 0.0245 -0.0218 0.0217 -0.0141 0.0140 -0.0087 0.0085 -0.0087 0.0085 -0.0087 0.0086 -0.0087 0.0086 -0.0057 0.0057 0.0057 0.0133 0.0178 0.0176 0.0122	Relative Residual 0.002134 0.002245 -0.001971 0.001940 -0.001246 0.001228 -0.000751 0.000751 0.000742 0.000721 -0.000721 -0.000721 -0.000721 -0.000721 -0.000721 -0.000721 -0.000721 0.000711 -0.000721 0.000711 -0.000466 0.000461 0.001068 0.001418 0.001393 0.000962
0.95 0.95 0.95	0534225 12.8000 2800930 12.8805 4966187 12.9574	) 12.7996 5 12.8987 4 13.0018	0.0005 -0.0182 -0.0444	0.000035 -0.001413 -0.003424



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Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3



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Serial # 161 Unit 1 Port 1 Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM	Analysis adsorptive: N2
Completed: 2014-04-28 3:33:48 PM	Analysis bath temp.: -195.803 °C
Report time: 2014-04-29 9:52:02 AM	Thermal correction: No
Sample mass: 0.4293 g	Warm free space: 16.9688 cm <sup>3</sup> Measured
Cold free space: 60.2332 cm <sup>3</sup>	Equilibration interval: 5 s
Low pressure dose: None	Sample density: 1.000 g/cm <sup>3</sup>
Automatic degas: Yes	

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

**Goodness of Fit** 







Appendix C

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Sample: Rice Husks Operator: Pua Submitter: Thayallan UTeM File: C:\3Flex\data\000-201.SMP

Started: 2014-04-28 10:26:07 AM Completed: 2014-04-28 3:33:48 PM Report time: 2014-04-29 9:52:02 AM Sample mass: 0.4293 g Cold free space: 60.2332 cm<sup>3</sup> Low pressure dose: None Automatic degas: Yes Analysis adsorptive: N2 Analysis bath temp.: -195.803 °C Thermal correction: No Warm free space: 16.9688 cm<sup>3</sup> Measured Equilibration interval: 5 s Sample density: 1.000 g/cm<sup>3</sup>

Comments: 2/3

Sample prep: Stage	Temperature (°C)	Ramp Rate (°C/min)	Time (min)
1	90	10	60
2	300	10	480

		Sample log
Date	Time	Log Message
2014-04-27 2014-04-27 2014-04-27 2014-04-27 2014-04-28	12:32:26 PM 1:42:26 PM 10:06:26 PM 10:26:07 AM	Start degas stage 1 of 2: ramp to 90 °C at 10 °C/min/min and hold for Start degas stage 2 of 2: ramp to 300 °C at 10 °C/min/min and hold for Start degas cooldown. Start degas cooldown. Started analysis of file 900 201.SMP on port 1.
2014-04-28 2014-04-28	10:26:07 AM 10:28:04 AM	System volume: 35.4569 cm <sup>3</sup> Port 1 1000 mmHg transducer scale changed from 516.4686 to 516.4911 mmHg (fraction of paminal: 1.01)
2014-04-28	10:57:09 AM	Ambient free-space measurement on sample port 1 complete (elapsed: 1859 s, qty in free-space: 16.9688 cm <sup>3</sup> ).
2014-04-28	11:09:42 AM	Analysis free-space measurement on sample port 1 complete (elapsed: 2612 s, qty in free-space: 60.2332 cm <sup>3</sup> ).
2014-04-28	11:30:03 AM	Psat port is charged with N2 at 760.6202 mmHg
2014-04-28	11:45:45 AM	Port 1 vacuum level is 3.83e-005 mmHg
2014-04-28	11:45:50 AM	Data collection started on sample port 1 (gas: N2).
2014-04-28	3:21:40 PM	Analysis termination started.
2014-04-28	3:33:48 PM	Finished a sample analysis for C:\3Flex\data\000-201.SMP on port 1.

Appendix D

### DEVELOPMENT OF HIGHLY EFFICIENT WALL INSULATION FROM

### **RICE HUSK FOAM REINFORCED COMPOSITE**

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**Keywords:** Rice Husk Ceramic Foam, wall insulation materials, manufacturing Process, ASTM AND ISO Standards

Abstract. This research is focus on development of highly efficient Rice Husk Ceramic Foam as alternative wall insulation materials for structure materials. The use of insulation wall in building construction causes some problems such as having high weight, high thermal conductivity and very reflective sound. So the objective of

this project is to develop of highly efficient Rice Husk Ceramic Foam as alternate heat insulation and sound insulation material for building. To achieve this objective, rice husk waste was used as filler material combine with other engineering chemical while the manufacturing process involve were burning process, mixing process, casting method, and drying process. The research methods are based on ASTM testing standards such are Density Testing, Cold Compression Test, Optical Microscope Test, Thermal Conductivity Test and Nitrogen Gas Absorption and Desorption Test Thermal Gravimetric Analyzer was according to ISO standard. Rice Husk Foam have lower thermal conductivity and higher compressive strength with density which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity. Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times. The pore distributions showed that the distribution 20 Å-250 Å is equal to 2 nm-25 nm which are categories in mesopores. It suggested that the type of pore which are suitable for insulation material are
mesopores. The objectives of this project to are achieved and it is proven that the Rice Husk Foam have better criteria as a thermal insulation

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material with the current thermal insulation material such as rigid Polyurethane and Polystyrenes.

# Introduction

Insulation is an essential engineering for energy recovering. The ideal isolation thickness is that worth at which the expense is least and it incorporates the expense of protection material and cost of energy utilization over the life time of the constructing. Insulation actions as a barricade to heat flow and is absolutely vital to hold your home moderately hot in winter and cool in summer. A well-insulated and well-designed dwelling will provide year-round comfort, chopping chilling and heating accounts by up to half [1]. This, in turn, will decrease greenhouse gas emissions. The solution is wall insulation so has been established in United Kingdom (UK), UK Government Flagship Energy Efficiency Policies which been introduced in autumn 2012. [2]

Nowadays, wall insulation becomes important in our life where 80% domestic properties in Malaysia have solid walls. At the 21<sup>st</sup> century the whole world now concerned about energy saves emission to environment and green technology. However there are local developer and domestic properties' owner still

lacking in this transformation and information. The effects of this unawareness cause the buildings not environmental friendly where it cost a lot of operation cost and emission to the nature. The invention of Rice Husk Foam Reinforced Composite is viewed as a solution for these problem, as it recovered the energy saving, cost reduction, thermal comfort and emission to the environment are reduce.

## **Rice Husk**

Rice Husk Ash can consummate by incinerate rice husk. Rice Husk has several names where the most common name is husk, hull and chaff. Rice Husk is the outmost layer of protection encasing of rice gain. It is a yellowish color and has a convex shape. It is slightly larger than a gain of rice, thus the length up to 7mm [3].It is lightweight, and has a ground bulk density of 340 kg /m3 to 400 kg /m3 whereby after incineration, only about 20% weight of rice husk are transformed RHA [3].Rice husk is the outer covering of this paddy. About 500 million tons of paddiesareproducedintheworld

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annually [4]. During milling of paddy about 78 % of weight is received as rice, broken rice and bran [4].Rest 22 % of the weight of paddy is received as Rice Husk

Rice husk is generated from rice processing is a waste material. Disposal of the rice husks is a big problem and open heap burning is not acceptable on environmental grounds, and so the majority of husk is currently going into landfill. The disposal of rice husks create environmental problem that leads to the idea of substituting RHA for silica in wall insulation manufactured. We choose rice hush ash to develop wall insulation because Rice Husk Ash (RHA) is a by-product of the Agricultural Industry which contains high amount of silicon dioxide (SiO2). The content of silica in the ash is about 92 %-97 % which provides several advantages such as, very abrasive, wears conveying elements very quickly, and also improved strength and durability properties [5]. Besides, as far as the sustainability is concerned, it will also help to solve problems otherwise encountered in disposing of the wastes [6].



Figure 1: Rice Husk incinerate process to Rice Husk Ash (RHA)

It recommended that burning time and furnace setting have an effect on the rice husk ash properties, like the silicon dioxide type and also the expanse of ash particles (Hwang). It is proven that the properties of rice husk ash depend on the burning procedures for the husk. As simply mentioned on top of, completely different characteristics of rice husk ash square measure helpful in numerous application [7]. Therefore, it's vital to contemplate the suitable burning conditions of rice husk for specific uses of the ash.

### Methodology

Table 1 shows the composition of raw material in term of percentages that weused to construct Rice Husk Foam. This research is conducted according toAmerican Society for Testing and Materials (ASTM) and International OrganizationforStandardizationStandardization(ISO)

Sample Code	Gypsum (%)	Cement (%)	RHA (%)	CaO (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Methylcellulose (%)	H₂O (%)
RHF-4	40	25	20	10	5	20	150
RHF-3	30	25	30	10	5	20	150
RHF-2	20	25	40	10	5	20	150
RHF-1	10	25	50	10	5	20	150

Table 1: Compositions of raw material

# **Procedure to Silhouette Rice Husk Foam**

- Thermal Gravimetric Analyzer (TGA/DTG)-ISO/IEC 17025:2005 to choose the most suitable temperature for the burning process of Rice Husk in furnace to gain the best quality Rice Husk Ash.
- Rice Husk burn to ashes at 650°C for 3600 seconds in the furnace and cooled in the furnace for 24 hours as recommended from TGA Test.
- iii. Then the Rice Husk Ash is grind using Rotary Mill and the fineness RHA is differentiating by using Vibrato Mill Shaker. The average particle size of RHA is 45µm is recommended.

iv. After that all the raw materials are hybridize well-balanced according to the composition ratio to enhance binder of Rice Husk Foam. V The binder is poured into the mould that design and scorched in the oven at 60°C for 6 hours.

vi. After 6 hours the specimen are take out from the oven and it is ready for Density Testing ASTM C380-79, Cold Compression Test ASTM C39, and Thermal Conductivity Test ASTM C1470-06

vii. Optical Microscope Test ASTM E210 -63(2010), and Nitrogen Gas Absorption and Desorption Test ASTM D4222-03.

# Result

# Nitrogen Gas Absorption and Desorption Test

# Nitrogen Adsorption-Desorption Isotherm Linear Plot

The effects of temperatures provided different performances of the nitrogen isotherms. All the nitrogen isotherm curves significantly increase in the low-relative pressure

(P/Po < 0.2) indicating the presence of micropore structures with the Type I isotherm.



Figure 2: Nitrogen adsorption-desorption isotherm linear plot

Figure 2 shows the Nitrogen Adsorption-Desorption Test ASTM D4222-03 was conducted for isotherm at 30 °C to 300 °C of the Rice Husk Foam. All the nitrogen isotherm curves significantly increase in the low-relative pressure (P/Po = 0.2) indicating the presence of micropore structures with the Type I isotherm. However, at higher relative pressure (P/Po = 0.2), the knee of nitrogen isotherm curves became more open and steadily increased to the

nitrogen isotherm curves became more open and steadily increased to the maximum relative pressure (P/Po  $\approx$  1.0). These results suggested that large amount of nitrogen was adsorbed during the adsorption process indicating the presence of wider porosity (mesopores) with the Type IV isotherm. The entire relative pressure ranges were suggested that the nitrogen adsorption exhibited a combination of the Type I and Type IV isotherms, indicating the attendance of micro- and mesoporosity in the Rice Husk Foam. Additionally, the presence of hysteresis loops with H3 type in the desorption isotherms clearly shown at the higher relative pressure (P/Po > 0.3). This hysteresis was which indicated to the well-developed mesopores.

### Pore size distributions

The distributions of mesoporosity were estimated by the BJH method. Figure 3 shows the plots of pore diameter distribution of the Rice Husk Foam starting from 0-20 nm. It is showed that from 20 Å-250 Å is equal to 2 nm-25 nm which are categories in mesopores where the criterion for mesopores is that the range of pore diameter must be between 2 nm-500 nm [8].



Figure 3: Pore size distributions

According to previous research that been done, it suggested that the type of pote which are suitable for insulation material are mesopores [9], [10]. So it is proven through the Specimen RH-4 have meet the criteria of in term of pore type as mesopores. The specimen RH-4 surface area, pore volume and pore diameter is shown in Table 3.

Table 5: Specimen RH-4 surface area, pore volume and pore diameter

Specimen	Surface Area( $m^2/g$ )	Pore Volume $(m^3/g)$	Pore Size( <i>nm</i> )
RH-4	14.54	0.0221	5.547

# Comparison between Rice Husk Foam, Polyurethane Foam and Polystyrene



Figure 6: Thermal Conductivity and Compressive Strength Result of Rice Husk Foam, Polyurethane Foam and Polystyrene



By referring to rigid polyurethane foam data according to Federation of European Rigid Polyurethane Foam Association and Polystyrene data according to Saving Energy Trust the comparison was done to proof that the Rice Husk Foam have lower thermal conductivity and higher compressive strength with density which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity. Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times.

### Summary

In this research ,it is proven that Sample RH-4 have better density, lightweight, compressive strength, maximum load and thermal conductivity compare to specimen RH-3, RH-2 and RH-1.While specimen RH-4 compare with Polyurethane Foam and Polystyrene Board in term of thermal conductivity it is proven that specimen RH-4 have lower thermal conductivity value which is 0.0018 Wnn.K and higher compressive strength which is 0.0770 MPa The Rice Husk Foam have lower thermal conductivity and higher compressive strength with density

which are 1.95 times better than rigid Polyurethane and 2.54 time better that Polystyrenes in term of thermal conductivity. Based on compressive strength, the Rice Husk Foam have higher compressive strength rigid Polyurethane and Polystyrenes which are 3.08 times. When the thermal conductivity value low, the less heat the material will transfer which are one of the main criteria that needed by an insulation material. If a material density increase, the thermal diffusivity value also increases. Through Nitrogen Gas Absorption and Desorption Test, the data that gained showed the Specimen RH-4 Surface Area 14.54 m<sup>2</sup>/g and Pore Volume 0.0221 m<sup>3</sup>/g and Pore Size 5.547 nm. The pore distribution show that the RH-4 are in in mesopores where it is in 2 nm- 25 nm which meet the criteria of mesopores in the range of 2 nm-50 nm. The objectives of this project to study and develop an efficient wall insulation material from Rice Husk Foam reinforced composite according to ASTM and ISO standards are achieved and it is proven that the Rice Husk Foam have better criteria as a thermal insulation material with the current thermal insulation material such as rigid Polyurethane and Polystyrenes.

## Acknowledge

The authors wish to his parents, friends and lecture together with Universiti Teknikal Malaysia Melaka (UTeM) for providing them the financial, infrastructure and support to this research.

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Appendix E