Effect Burning Temperature and Soaking Time of Pre-treatment hydrochloric acid toward the Characteristic of Ultra-Fine Silica Obtained From rice husk Ash

By

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15540

Dissertation submitted in partial fulfilment of

the requirements for the

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(Civil Engineering)

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CERTIFICATION OF APPROVAL

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(CIVIL ENGINEERING)

Approved by,

____

(Prof .Dr Nasir shafiq)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

____

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ABSTRACT

Rice husk ash is one of the material that can producing silica (SiO_2) and it is one of the important chemical composition Portland cement. The objective of this research to study the effect of soaking time and burning temperature toward physical and chemical characteristic silica obtained from the pre-treatment process. Hydrochloric acid (HCI) with 0.1N concentration was used to remove alkaline impurities on the surface of rice husk ash. Five different soaking time were used which are 1h,2h,3h,4h and 5h. For the burning process, three different temperature were used which are 600°C, 700°C and 800°C. Rice husk ash will be grinded using planetary ball mill (mechanical activation) at maximum of 350rpm for 15 minutes. The sample will be tested using five different machine which are X-ray fluorescence (XRF), scanning electron microscopy (SEM), particle size analysis (PSA), atomic absorption spectroscopy (AAS), and Brunauer–Emmett–Teller test (BET) to study chemical composition, removal particle during soaking time, shape of particle and size of particle. Based the research it shown the 4 h soaking time with burning temperature 600°C temperature produced the highest content of silica which is 97.6%. In the future, the different type of acid and concentration will be used as chemical solution for pre-treatment process.

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CHAPTER 1

INTRODUCTION

Malaysia is also one of the country that produced paddy every year, usually the harvest season start from November to march. At 2011 the production of paddy at Malaysia is almost 2500 metric tons and it still can be considered as large ("(Paddy Statistics of Malaysia)," 2011). Rice husk is waste product from the process of paddy and don't has any economic value. Usually the farmer just left it abundantly or burning it in open area to save the disposal cost of this material. The different chemical content of rice husk is depend on the type of paddy, geographical condition, climate, crop of the year, type of soil ,procedure of preparation and method of analysis (Rafiee, Shahebrahimi, Feyzi, & Shaterzadeh, 2012).

Constituent	% composition
Fe_2O_3	1.32
SiO_2	54.8
CaO	2.6
Al_2O_3	1.29
MgO	0.674
K_2O	28.1
P_2O_5	3.86
SO_3	2.24
Other constituent	5.116

Table1: chemical content of raw rice husk.

From the table above it shown the highest chemical content of rice husk is silica (SiO_2) and with using proper method the quantity of silica from the rice husk can be increased. Lots of researchers have wrap up that rice husk are good source of high-quality of silica(Yalc & Sevinc, 2001). Acid leaching is a simple method to obtain silica from rice husk and using this method also silica > 99 % purity can be achieved by burning the rice husk at 600°C temperature.(Yalc & Sevinc, 2001). Silica is the basic raw material used in many industry. For instance in electronic industry, cement and material polymer industries (Liou, 2004).

1.1 BACKGROUND OF STUDY

Malaysia is one country that produces paddy every year and taking this opportunity, the study is to find the best pre-treatment process of rice husk ash as one of the material that has a big potential as cement material replacement (CRM).

Figure 1: rice husk ash after the burning process

1.2 PROBLEM STATEMENT

Before this, there are many researches have done the study to find best pre-treatment process for rice husk and the problem is, there is no standard way to treat the rice husk. The HCI acid were used to clean the rice husk ash from any unwanted material, Other than that sodium hydroxide (NaOH) and acid sulfuric($H2SO_4$) also were used for acid treatment process. The time for soaking process also playing important role in other to get the good silica.

1.3 THE OBJECTIVE OF THIS STUDY

The objectives of this study are:

- ✓ To determine the effect of pre-treatment soaking time to the physical and chemical characteristics of ultra-fine silica from rice husk.

- ✓ To determine the effect of pre-treatment temperature to the physical and chemical characteristics of ultra-fine silica from rice husk.

1.4 SCOPE OF STUDY

For this research we only use rice husk ash as the experimental material and the concentration of the HCI acid used is only 0.1M. To treat the rice husk, the ration 1: 20 was used, which means 1g of rice husk equals to 20 ML of HCI. For the raw sample material, it was taken from BERNAS factory at Sg Ranggam. Below are the parameters of this research;

- ➢ Soaking time
- ➢ Temperature

1.5 LAB TESTING

To achieve the objective for this research, several tests have been conducted. The test is important in other to support the objective of this research and the test are atomic absorption microscopy (AAS), X-ray fluoresces (XRF), particle size analysis (PSA), scanning electron microscope (SEM) and lastly Brunauer Emmat Teller Nitrogen adsorption method test (BET).

I. Atomic absorption spectrophotometer (AAS)

Figure 2: atomic absorption machine

AAS is a method used to identify the presence and concentration in solutions. The aim of this test to see the chemical content inside the solution (HCI concentration mix with rice husk) after the soaking process. There are four chemical elements that we will look into for this test and they are magnesium (Mg), calcium (Ca), potassium (K) and sodium (Na). All of these elements are inside the rice husk.

II. X-ray fluorescence (XRF)

XRF is the fastest, effective, and the best way to measure the major oxide and trace the element abundance in the loose powder, solid, and liquid. The way XRF work is the X-rays characteristic wavelength are emitted from the sample when the sample is ionized by a stream of X-rays. Based on the result from this test, the highest content of silica can be obtained.

Figure 3: X-ray fluorescence machine

III. Particle size analysis

Matersizer 2000 has been used to conduct this analysis. This machine is one of the most user friendly and flexible particle size measurement until now. The result from this test will be determined either the size of silica obtained from rice husk ash are nano silica or ultra-fine silica.

Figure 4: Mastersizer 2000

IV. Scanning electron microscope (SEM).

Used effectively in microanalysis and failure analysis of solid material. During the analysis, the signal that was generated by SEM will produced two-dimensional image and produce the information about sample regarding the texture, chemical composition and arrangement of material that making up the sample.

Figure 5; scanning electron microscopic machine

V. Brunauer Emmett Teller Nitrogen adsorption method test (BET).

The aim to conduct this experiment is to obtain the surface area of rice husk ash by using nitrogen multilayer measured. Surface area is important factor in other to determine the reaction of silica. The biggest surface area will produce fast reaction compared to the small surface area.

Figure 6: BET machine

CHAPTER 2

LITERATURE REVIEW

2.1 STUDY ON SILICA OBTAINED FROM RICE HUSK

Different concentration acid were used as chemical and post-treatment. Three types of acid that are used for treatment process are hydrochloric acid (HCI), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH). (Yalc & Sevinc, 2001). 78% of mass rice husk were lost at temperature 500°C, which can be described 22% mass of silica content (Yalc & Sevinc, 2001). The huge different content of silica can be observed between the treated and untreated rice husk. One of the way to reduce the carbon content is by increasing the oxygen pressure, but this procedure not really effective to solve this problem. Type of chemical used to treat rice husk was influenced the content of silica of rice husk ash. High surface area of amorphous and pure silica can be obtained from rice husk. The homogeneous particle size distribution were obtain at 600°C temperature burning process after leaching process with hydrochloric (HCI) solution. The other burning process and leaching process did not shown the good result (Yalc & Sevinc, 2001).

2.2 EFFECT OF RICE HUSK ASH ON THE STRENGTH AND DURABILITY CHARACTERISTIC OF CONCRETE.

This research has used the rice husk from South Vietnam. Rice husk ash was ground for 1 h to ensure the pozzolanic activity become well. It will be added with the concrete mixture as cement material replacement material. Based on the research, with adding 20% of ground rice husk ash into concrete mixture, the result shown same compressive strength with control concrete. The control concrete for this test is 28 days. The study shown the compressive strength of concrete of the compound and the indicator were similar, even the rice husk ask has high carbon content. This study was shown the possibility to use ground rice husk ash as partial Portland cement replacement material and also will reducing the pollution toward the environment (Chao-Lung, Anh-Tuan, & Chun-Tsun, 2011).

2.3 PREPARATION AND CHARACTERIZATION OF NANO-STRUCTURED SILICA FROM RICE HUSK

Silica is an important material, usually used in the ceramic, electronic and polymer material. At this moment it was prepared using a few methods and they are vapour-phase reaction, sol-gel and thermal decomposition technique. The main problem for all methods above is the cost too high. Under controlled condition very fine particle size, very high surface area and purity silica can be obtained (Liou, 2004). Hydrochloric acid (HCI) were used to remove alkaline impurities on the surface of the rice husk. In this study burning temperatures adopted were between 600°C – 800°C. High specific area of silica can be obtained after the heating process at temperature of 700°C.(Liou, 2004). . The heating rate plays an important role towards the properties of silica. Almost 95% of impurities were extracted after thermal decomposition process of sample.(Liou, 2004). The early and final reaction temperatures, and reaction space range increases when the temperature increase (Liou, 2004)

2.4 OPTIMIZATION OF SYNTHESIS AND CHARACTERIZATION OF NANO SILICA PRODUCED FROM RICE HUSK (COMMON WASTE MATERIAL)

Adopted acid treatment and thermal combustion under controlled conditions produce 22.50% of rice husk ash and out of that 90.469% was silica(Rafiee, Shahebrahimi, Feyzi, & Shaterzadeh, 2012). Surface area will be increased when the strong interaction happened between silica and metallic ion. The effective procedure to dismantle and to produce highly purified silica powder is using acid solution which are hydrochloric acid (HCI), ammonium Nitrate ($NaOH_3$), or acetic acid (CH_3COOH). Several factors was detected that was influenced the quality of rice husk ash such as soaking time, burning temperature and rate of heating (Rafiee et al., 2012). Adopted 0.1N hydrochloric acid (HCI) solution to removing the metallic material shown the best result in terms of high surface area and pore volume. The highest impurities of rice husk ash is at combustion temperature 500°C, meanwhile at combustion temperature 1000°C the crystallization of silica increase. The combustion temperature at 700°C will produced amorphous silica relative high surface and high of purity (Rafiee et al., 2012). From the research, it shown highly effective silica can be produced using economic technology and at the same time can reduce the impact toward the environment pollution (Rafiee et al., 2012).

2.5 SYNTHESIS AND CHARACTERIZATION OF RICE HUSK SILICA, SILICA-CARBON COMPOSITE AND H_3PO_4 ACTIVATED SILICA.

Ultra- fine silica has many potential applications in industrial sector. For example, it is an important material for sorption media, glass, cement manufacturing and dehydration system. To treat rice husk, complicated chemical process need to be used. Nano silica also can be obtain from rice husk ash by burning process under appropriate condition which means low cost needed (Singh et al., 2008). Out of total, 20% of rice husk still remain as ash and this ash consists 95% of silica. (Singh at. el, 2008). One method was found to improve the process the silica with lower sodium content with adding silicate to pH 1.5 hydrochloric acid (HCl), oxalic acid (H2C2O4), or citric (C6H7O8) solution until the pH become 4.0. (Singh at. el, 2008). The active silica with high specific area can be produced using heat-treating process at temperature 700°C in air (Singh at. el, 2008). Based on the process and experiment that has been done, it shown the surface diffusion at low temperature and bulk diffusion at high temperature rate will derive activation barrier for densification (surface area decreasing) (Singh at. el, 2008).

2.6 EFFECT OF TEMPERATURE ON NANO-CRYSTALINE SILICA AND CARBON COMPESITE OBTAINED FROM RICE HUSK ASH

Ultra-fine silica powder has many potential applications in industry, for example as material of high surface area catalyst, cement manufacturing, glass and many more. The silica that is produced from rice husk is easy to process and can save the cost of production. At present, there are lots of technique to produce silica including vapor phase, sol-gel and thermal decomposition (Sarangi, Nayak, & Tiwari, 2011). However, the cost is expensive. By applying optimum burning of the rice husk, high grade and economical silica can be produced, at the same time it needs minimum grinding. (Sarangi et al., 2011). In the burning process, almost 20% mass of rice husk remaining and its contents 95 wt.% of silica.(Sarangi et al., 2011). For the leaching process with HCI, it showed that with 75°C and 1 H soaking time the product of amorphous silica is completely white color.

2.7 THE EFFECT OF HYDROCHLORIC ACID PRE-TREATMENT ON THE PHYSIOCHEMICAL PROPERTIES AND POZZOLANIC PERFOMANCE OF RICE HUSK ASH

Every day the world is growing, many construction works need to be completed. This will drive the demand of the material in other to ensure the construction works going well. For every year, approximately millions of cement are produced and China leads the production of Portland cement in the world.(Gholizadeh Vayghan, Khaloo, & Rajabipour, 2013). This will lead the pollution to the environment. The research had been conducted to find the alternative way to produce silica by using other waste material like rice husk, coal fly ash, sugar cane and many more. Rice husk is one of the good materials to produce silica. Acid leaching treatment increase silica (SiO_2) content in ashes and at the same time reduces the alkali contaminant likes potassium (K_2O) and sodium (Na_2O)(Gholizadeh Vayghan et al., 2013). The result shows rice husk with acid treatment process gives better improvement in term of silica content, alkalinity content, surface area and many more compared to the rice husk without acid treatment process. To improving the rice husk reactivity, 0.01N hydrochloric acid (HCI) is already sufficient enough (Gholizadeh Vayghan et al., 2013).

2.8 STUDY ON EFFECT OF BURNING CONDITIONS AND RICE HUSK ASH (RHA) BLENDING AMMOUNT ON MECHANICAL BEHAVIOUR CEMENT.

Rice husk is one of the waste of agricultural and it has its own uniqueness which is content of silica that is useful for industry like glass, sorption media and many more. With using low-temperature at 600°C the amorphous silica can be obtained and some analysis tell that silica inside the rice husk form of nano particle.(Bie, Song, Liu, Ji, & Chen, 2015). There are two factors that influence the strength of rice husk blended with cement and they are mixture proportion and burning condition. From the energy dispersive energy test (EDX) shown the outer and the inner space of rice husk ash is formed by dense silica (SiO2), and the main metal element is potassium (K). The structural porosity, carbon removal and few metal impurity are the main factor that influenced the high active and surface area of silica (SiO2). Surface area from 700°C burning temperature are greater than 600°C burning temperature and at the same time was found that the potassium content influenced the specific surface are. Small content potassium will be produced high specific surface are compared to the high content of potassium. Temperature giving huge impact toward the residual carbon content of rice husk ash. The result show 600°C is the optimum temperature to obtained large specific surface area and rice husk ash can be used as cement additive at the same time could improve the strength of the mortar.(Bie et al., 2015)

2.9 NANO SILICA FROM RICE HUSK: EXCTRACTION AND CHARACTERIZATION

Every year abundant of rice husk did not disposal in the proper way, the worst comes when some of the farmers throw their rice husk into the river. This will drive the pollution toward our environment especially water pollution. The study regarding rice husk has been done before this and has suggested a several methods to treat the rice husk for getting the silica on the surface of rice husk ash. One of the method is to boiled rice husk with acid solution under the pressure prior to the burning process. The rice husk ash with acid treatment produced amorphous and white silica compared with the untreated rice husk ash that produced silica and impurities(Carmona, Oliveira, Silva, Mattoso, & Marconcini, 2013) . Acid treatment were capable to remove manganese, potassium and calcium from rice husk ash. Potassium and manganese could partially be removed using water treatment process. Based on the result, it shown that using mild acid solution the amorphous and white silica can be obtained with micro and nanometric particle size (Carmona et al., 2013).

2.10 SYNTHESIS AND SURFACE CHARACTERISTIC OF NANO SILICA PRODUCED FROM ALKALI-EXTRACTED RICE HUSK ASH.

Having high porosity and surface area, nano silica become important material for pharmaceuticals, as a catalyst, and many more. For the industry, the way to produce silica is using sodium silicates, and this process required lot of energy. Hydrochloric acid (HCI) pre-treatment process produced the maximum surface area of silica from rice husk (Liou & Yang, 2011). Different type of acid treatment has huge effect on the pore volume and surface area of sample. From the study it shown that hydrochloric acid

(HCI) is the optimum nominee for acid precipitation(Liou & Yang, 2011). Acid-leached rice husk hands to clear away settled impurities in rice husk and also produced high purity of silica. Treating the rice husk with an alkali extraction process will gain the pore volume and surface area. Various type of acid showed important role in differences mass loss(Liou & Yang, 2011). Based on the result, the surface area of the sample depends on the gelation pH, time and aging temperature. Other than that, concentration of sodium silicate also influence the surface area of the sample(Liou & Yang, 2011).It shown hydrochloric acid (HCI) is the best acid solution for pre-treatment process which are produced higher surface area and pore volume compared to other type acid solution(sulfuric, citric and oxalic acid). The author has proven that nano silica can be produced from rice and at the same time can reduce the pollution toward the environment(Liou & Yang, 2011).

CHAPTER 3

METHODOLOGY

This chapter will describe the methodology that has been used for this research and also the previous methodology from other researches that related to this topic. In this study, the methodology include:

- Research Methodology
- Project Experiment
- Gantt chart

3.1 RESEARCH METHODOLOGY

For this research the methodology that has been used is, experimental. The previous research also had been used as reference in other to ensure the experiment is going well. The sampling and characterizing also used as methodology for this research.

3.2 PROJECT EXPERIMENT

STEP 1:

- using the tap and distilled water to wash rice husk

Figure 7: washing the rice husk ash with water tap

STEP 2:

-Soaking the rice husk with hydrochloric acid (HCI) into two liters biker with the ratio 1: 2 (1g rice husk/ 20ml hydrochloric acid)

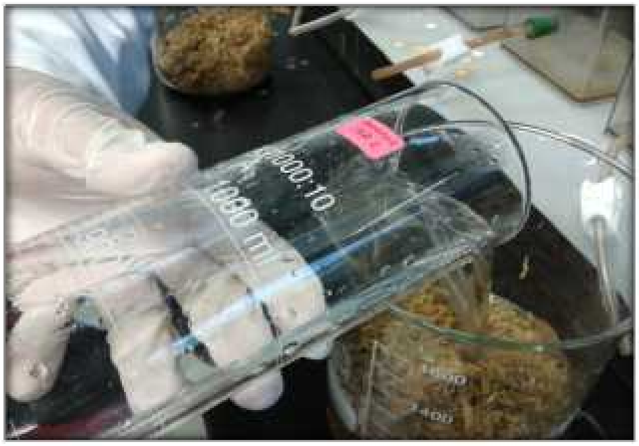

Figure 8: mix the HCI (0.1M) acid with the rice husk ash

STEP 3:

-Put the two liter biker on the top surface of hot plate and heat it until the temperature reach 80°C temperature. Maintain the temperature follow based on soaking time.

Figure 9: rice husk mix with hydrochloric acid (HCI) on the top surface of hot plate.

STEP 4:

-using the thermometer to ensure the temperature of solution is at 80°C

Figure 10: thermometer was used to measure the temperature of the solution.

STEP 5:

-Take the water sample from the soaking process for atomic absorption spectroscopic (AAS) test.

Figure 11: water sample for atomic absorption spectroscopic (AAS) test.

STEP 6:

-wash rice husk using the tap water followed by distilled water. This to ensure the rice husk is in natural condition after treated with acid solution.

Figure12: was the rice husk with tap and distilled water

<u>STEP 7:</u>

- measure the pH rice husk using pH meter.

Figure13: measuring the rice husk pH

<u>STEP 8:</u>

-Insert the treated rice husk into the oven at 110^{o}C for 24 hours to remove excess water and moisture in the RH.

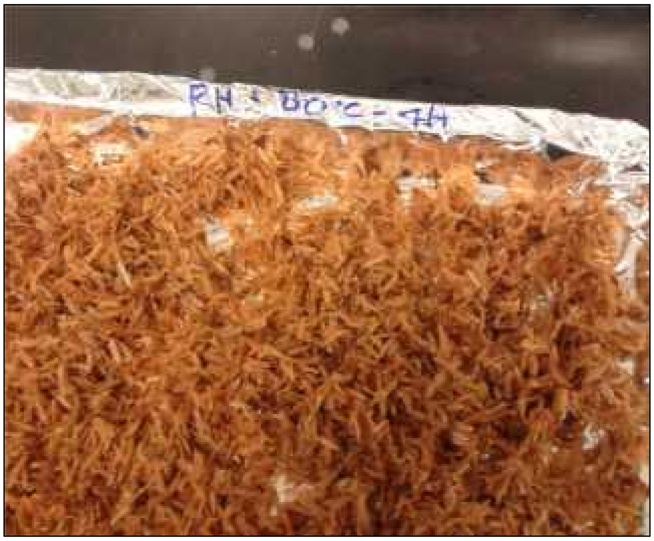

Figure14: rice husk ready for dryer process

Figure15: dryer that was used to dried rice husk

STEP 9:

-Completed with drying process, burn rice husk using chamber furnace.

-Burn the treated RH in muffle furnace at varies temperatures (600^{o}C, 700°C 800^{o}C).

Figure16: chamber furnace

Figure17: rice husk after completed burning process

STEP 10:

-Grind the rice husk ash using planetary ball mill (mechanical activation) at maximum of 350rpm for 15 minutes.

-Possible parameter variation:-

- -BPR (Ball to Powder ratio)
- -Speed (RPM)
- -Duration of grinding

Figure18: grinding machine

Figure19: rice husk ash inside the mould of grinding machine

STEP 11:

-Continue with physical characterization particle size analysis (PSA), X-ray fluorescence (XRF), atomic absorption spectroscopic (AAS), Brunauer Emmat Teller Nitrogen adsorption method test (BET), and scanning electron microscope (SEM).

Temperature (^oC)	Grinding using Planetary Ball Mill			
	BPR	RPM	Grinding Duration (minute)	PSA, XRF, SEM, BET, AAS
600	Y	350	15	
700	Y	350	15	
800	Y	350	15	

Table 2: grinding information for rice husk ash

3.3 PROJECT GANT CHART

To ensure the study being run smoothly and on track, the author has prepared a Gantt chart which lists all activities that need to be completed in a specific time frame.

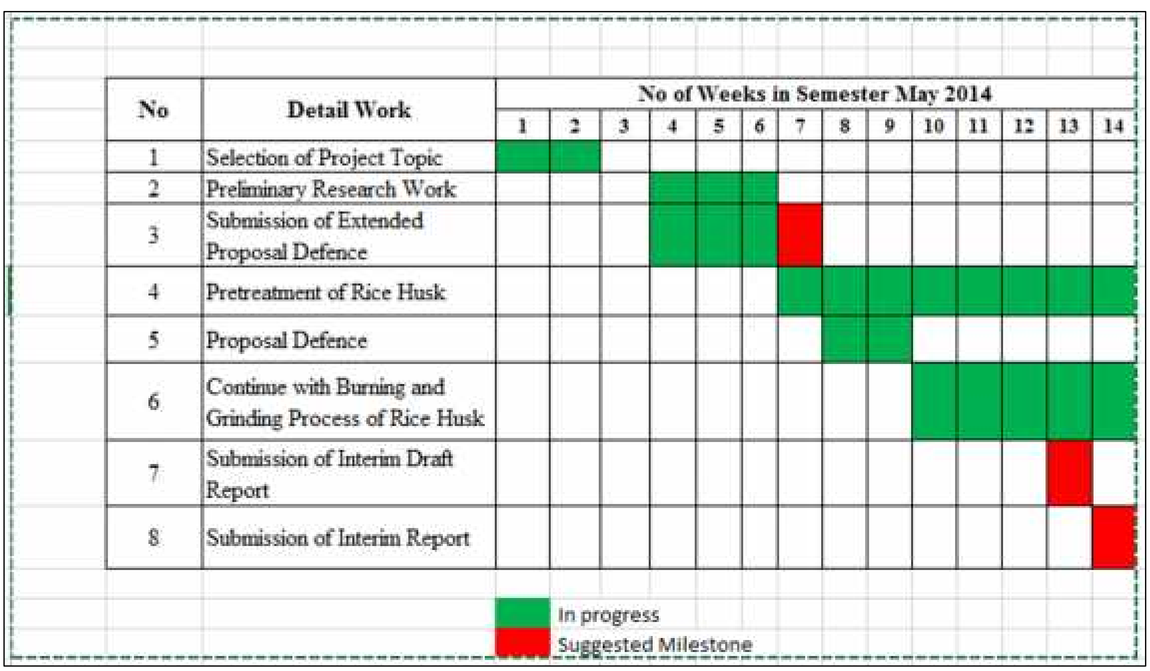

Figure 20; Gantt chart for Final year project 1

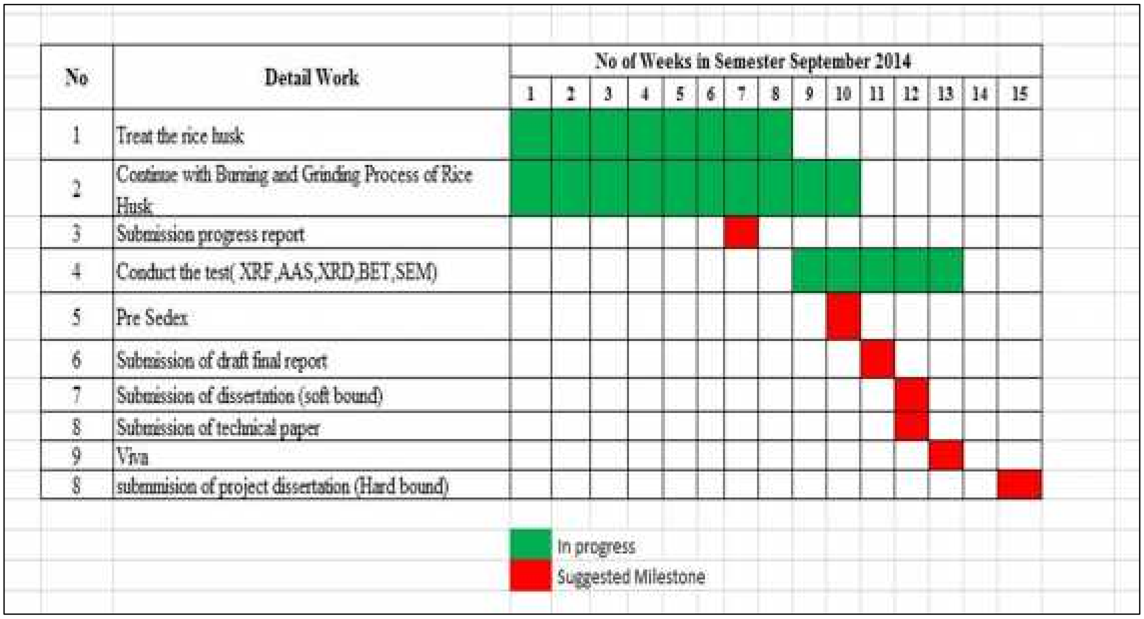

Figure 21; Gantt chart Final year project 2

CHAPTER 4

RESSLUT AND DISCUSSION

Five types of test has been conducted in other to find the result for this study which are, X-ray fluorescence (XRF), particle size analysis (PSA), scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET) and atomic absorption spectroscopy (AAS) . This chapter also will include the discussion of this study based on the result of the test that we conducted.

4.1 ATOMIC ABSORPTION SPECTROSCOPIC (AAS) TEST RESULT.

The objective of this test to trace the alkaline impurities (Mg (magnesium), K (potassium), Na (sodium), and Ca (calcium)) that was removed during the soaking process. The alkaline impurities give huge impact toward the characteristic of ultra-fine silica obtained from rice husk ash.

Figure 22: water sample for 0.1N and 5 hours soaking time

Figure 23: water sample for 0.1M and 1 hour soaking time.

Soaking time(H)	Magnesium:	Potassium:	Sodium:	calcium: flame actual

	flame actual (ppm)	flame actual (ppm)	flame actual (ppm)	(ppm)
1	10.568	49.156	2.154	21.534
2	11.04	54.054	1.808	22.284
3	11.138	55.348	1.588	21.95
4	11.426	51.492	1.114	22.366
5	14.412	63.172	1.244	24.122

Below is the result of AAS test;

Table 3: the quantity of removal particle based on AAS test

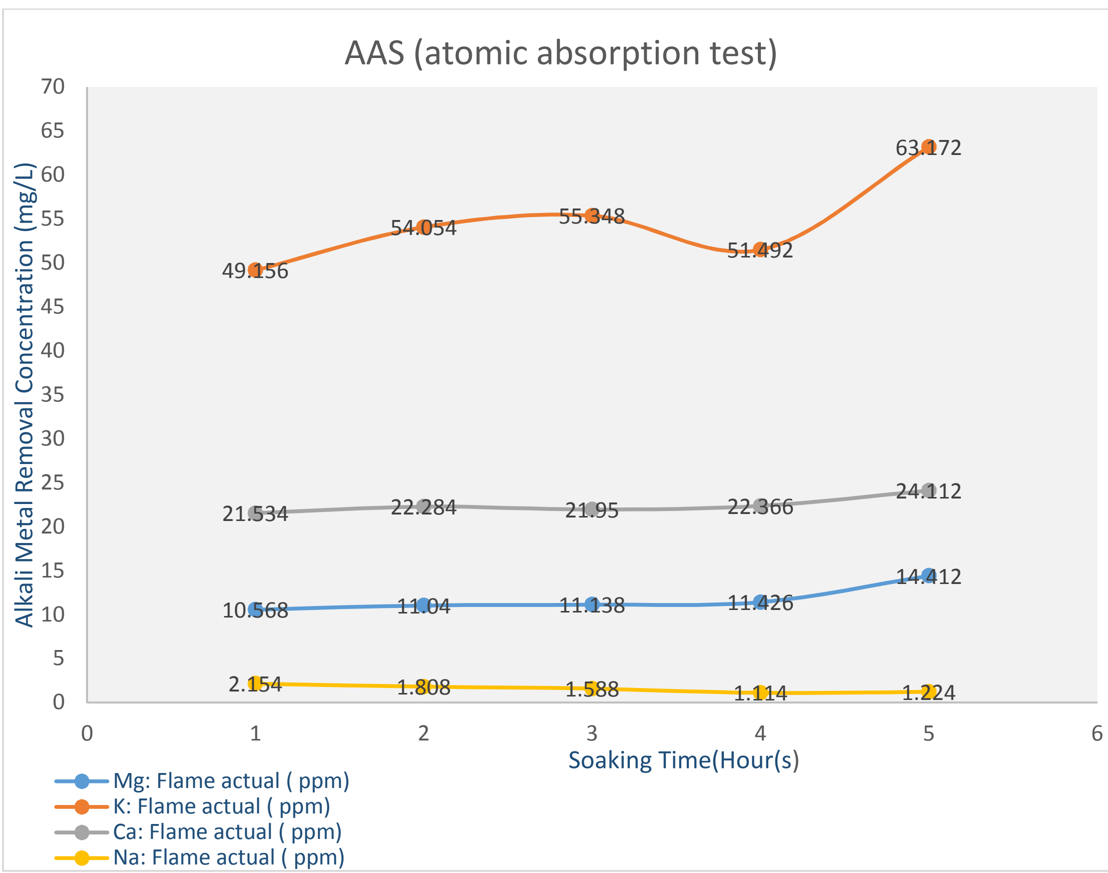

Figure 24: result for atomic absorption microscopy (AAS) test

Based on the graph above, it shown the five hours soaking time remove the highest contains of alkaline impurities. Even the value of sodium (Na) is small at five hours soaking time, it is not become the problem toward the durability of concrete. The important thing is to remove the potassium, this because it involved the arrangement of rectangular structure and also precisely affect the carbon elimination process in rice husk combustion. The amount of carbon removal has direct reaction toward the specific surface area of rice husk ash. Therefore the important thing to produced rice husk ash with high specific surface area is to ensure the structural porosity of silica oxide (SiO_2) and to dismantle carbon and some metal impurities.

4.2 X-RAY FLOURESCNENCE (XRF) TEST RESULT

The XRF test was conducted to see the chemical composition on the rice husk ash. This test is very important to find best pre- treatment process (temperature and soaking time) for rice husk. Below is the result for the X-ray fluorescence (XRF) test in the variation temperature which are, 600°C, 700°Cand 800°C. The highest content of silica come four hours soaking time with the burning temperature 600°C which the value is 97.6% and the lowest is 70.8% from 800°C with four hours soaking time. The value of silica from raw material is 54.9 %. From the XRF test we can see the highest value of silica that increased after the treatment process is 42.7 %. From the X-ray fluorescence test (XRF) the best temperature for burning process is 600°C. At this temperature rice husk could burn more completely compare to the other temperature (700°C and 800°C).

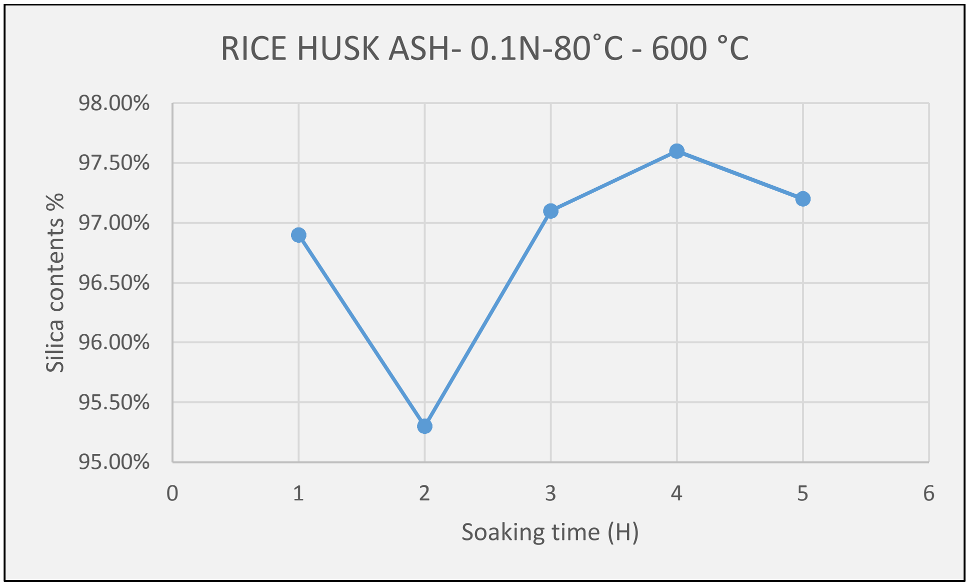

Figure 25: silica content for 600°C

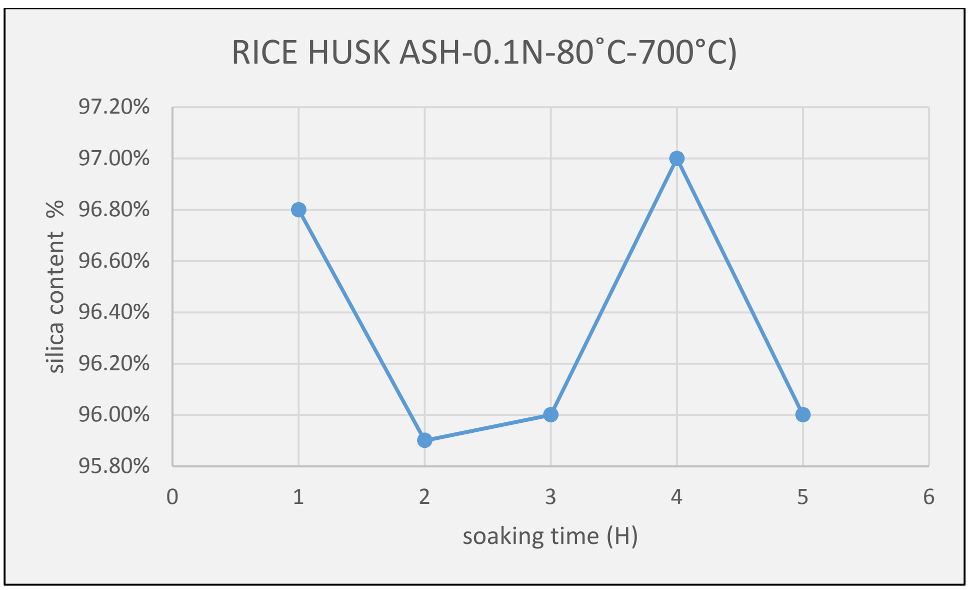

Figure 26: silica content for 700°C

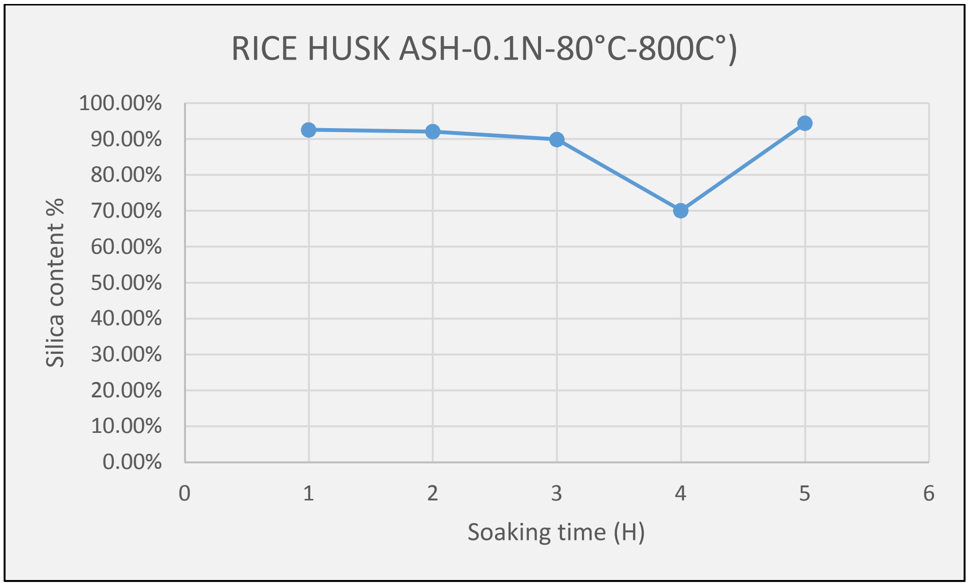

Figure 27: silica content for 800°C

Constituent	% composition
Sio_2	97.6
P_2O_5	1.38
CaO	0.364
ZrO_2	0.218
NaO_2	0.209
K_2O	0.0624

Fe_2O_3	0.0575

Table 4: chemical composition for rice husk ash 4hour soaking time with 600°C burning temperature.

Constituent	% composition
Sio_2	97.2
P_2O_5	1.59
CaO	0.410
ZrO_2	0.215
Al_2O_3	0.209
K_2O	0.144
Fe_2O_3	0.240

Table 5: chemical composition for rice husk ash 5hour soaking time with 600°C burning temperature.

Constituent	% composition
Sio_2	54.8
Cl	3.97
CaO	2.6
P_2O_5	3.86
Al_2O_3	1.29
K_2O	28.1

Fe_2O_3	1.32
SO_3	2.24

Table 6: chemical composition for rice husk ash without acid treatment process with 700°C burning temperature.

4.3 SCANNIG ELECTRON MICROSCOPIC (SEM) TEST RESULT

Scanning electron microscopy (SEM) test is to find the chemical composition, crystalline of structure and the orientation of material that make up the sample. Three different magnification were used, which are X 500, X 1000, and X3000. Below is the best result of scanning electron microscopy (SEM) which come from 4 and 5 hour soaking time and burned under 600°C temperature.

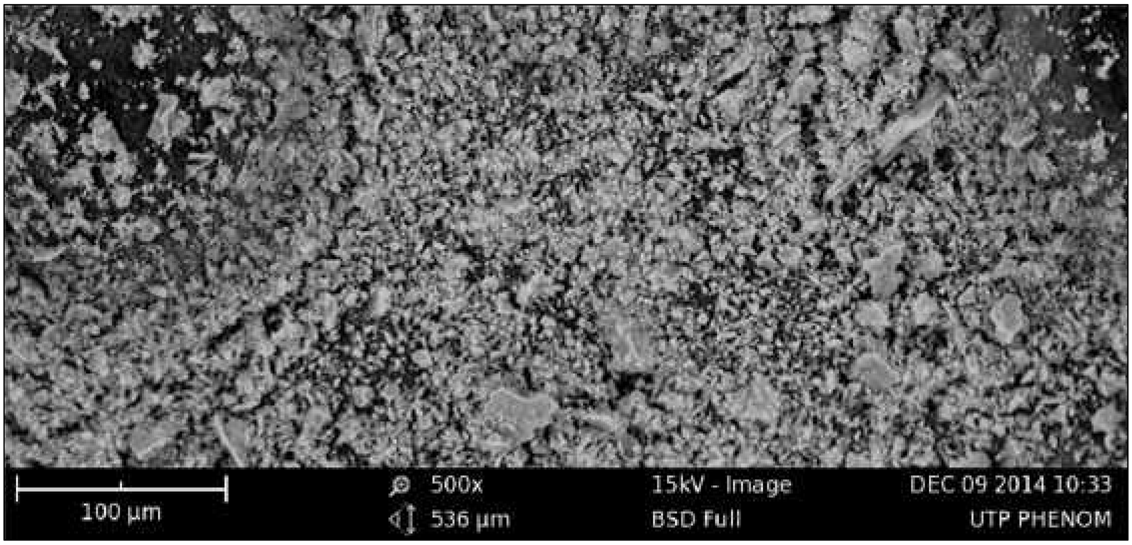

Figure 28: SEM result for X 500 magnification for 4 hour soaking time and burned with 600°C temperature.

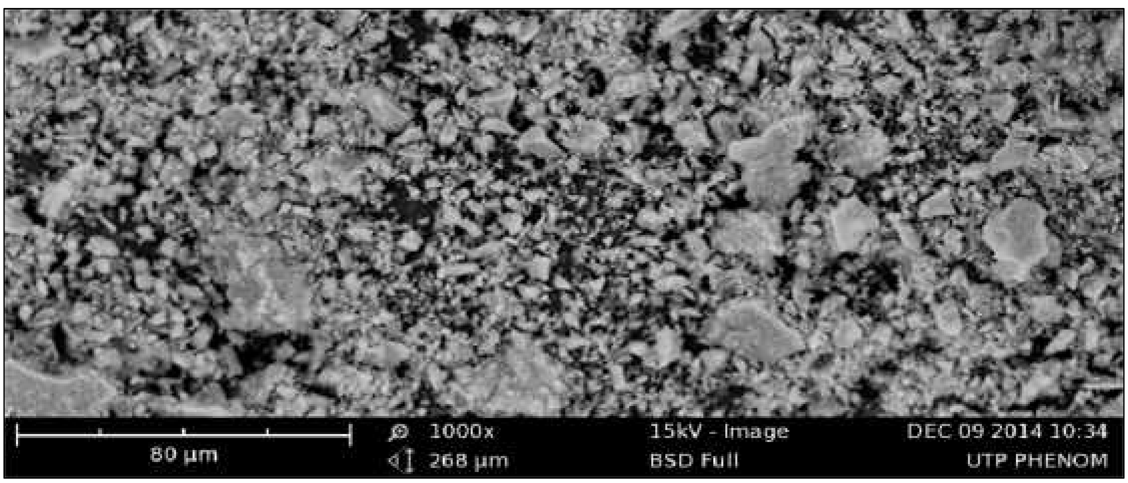

Figure 29: SEM result for X 1000 magnification for 4 hour soaking time and burned with 600°C temperature.

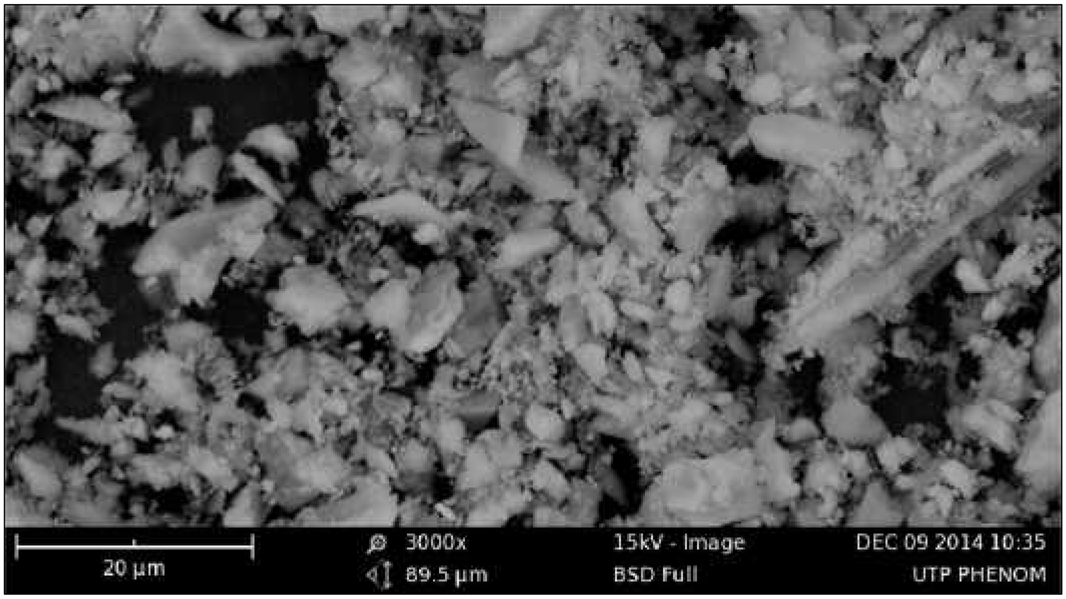

Figure 30: SEM result for X 3000 magnification for 4 hour soaking time and burned with 600°C temperature.

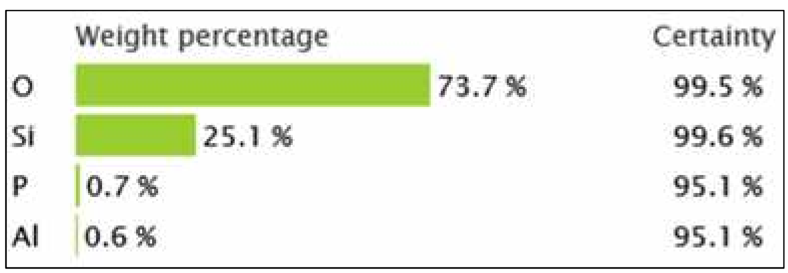

Figure 31: weight percentage for 4 hour soaking time and burned with 600°C temperature.

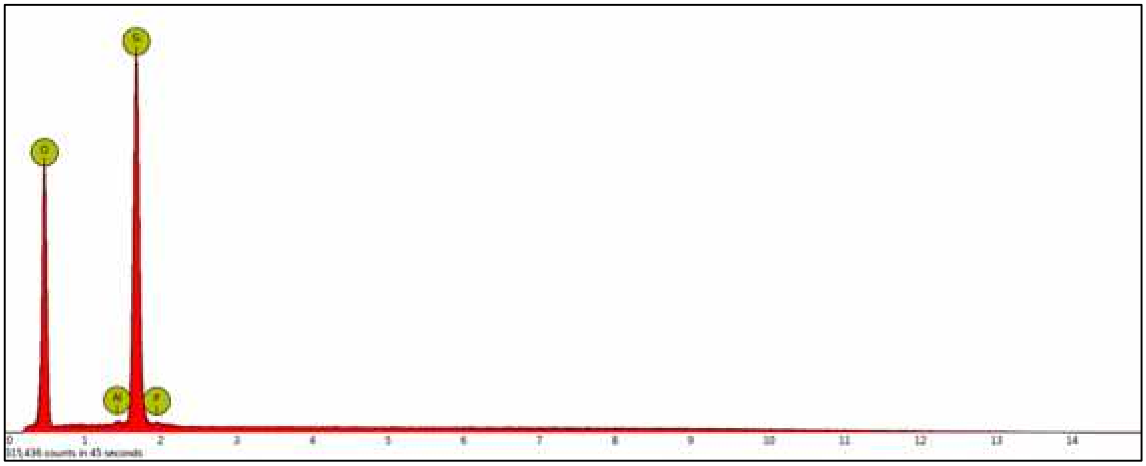

Figure 32: the graph element for 4 hour soaking time and 600°C burning temperature

14	Si	Silicon	100.0	25.1	0.4
8	O	Oxygen	100.0	73.7	0.5
13	Al	Aluminum	100.0	0.6	4.9
15	P	Phosphorus	100.0	0.7	4.9

Table 7: element number, element symbol, and element name confidence concentration error.

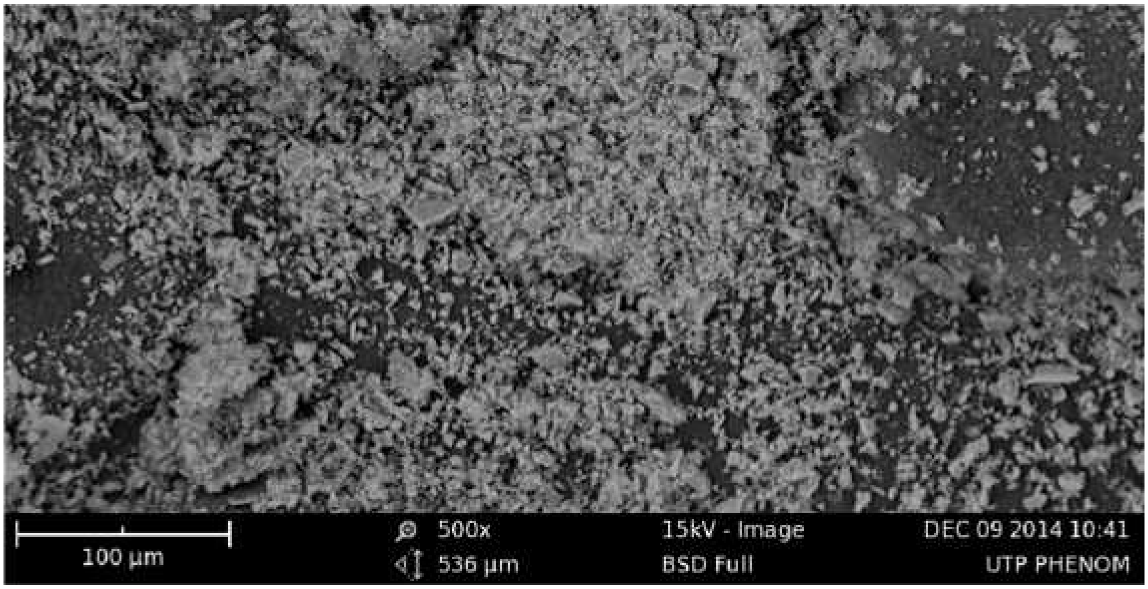

Figure 33: SEM result for X 500 magnification for 5 hour soaking time and burned with 600°C temperature.

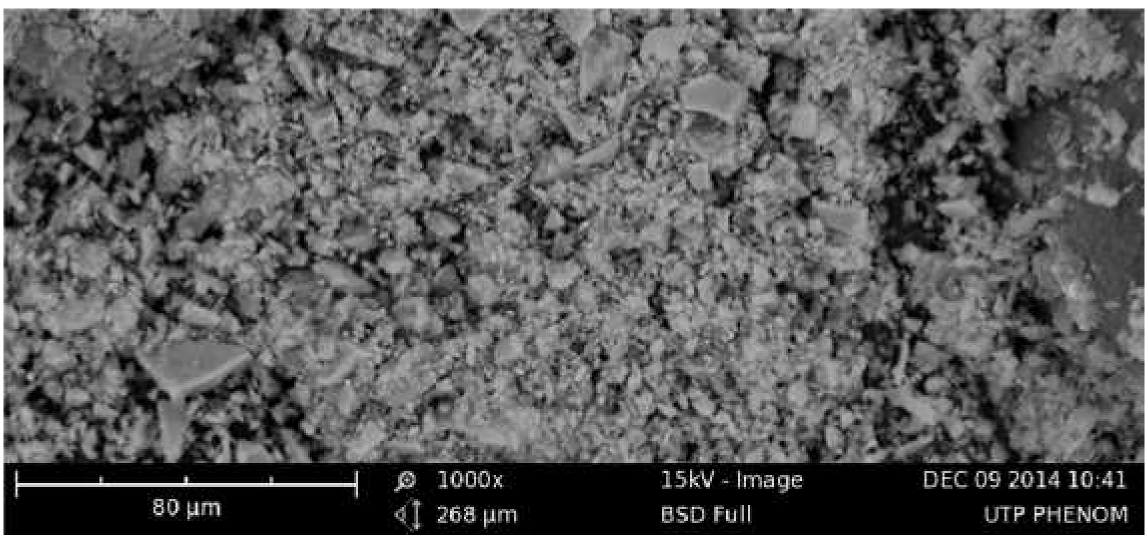

Figure 34: SEM result for X 1000 magnification for 5 hour soaking time and burned with 600°C temperature.

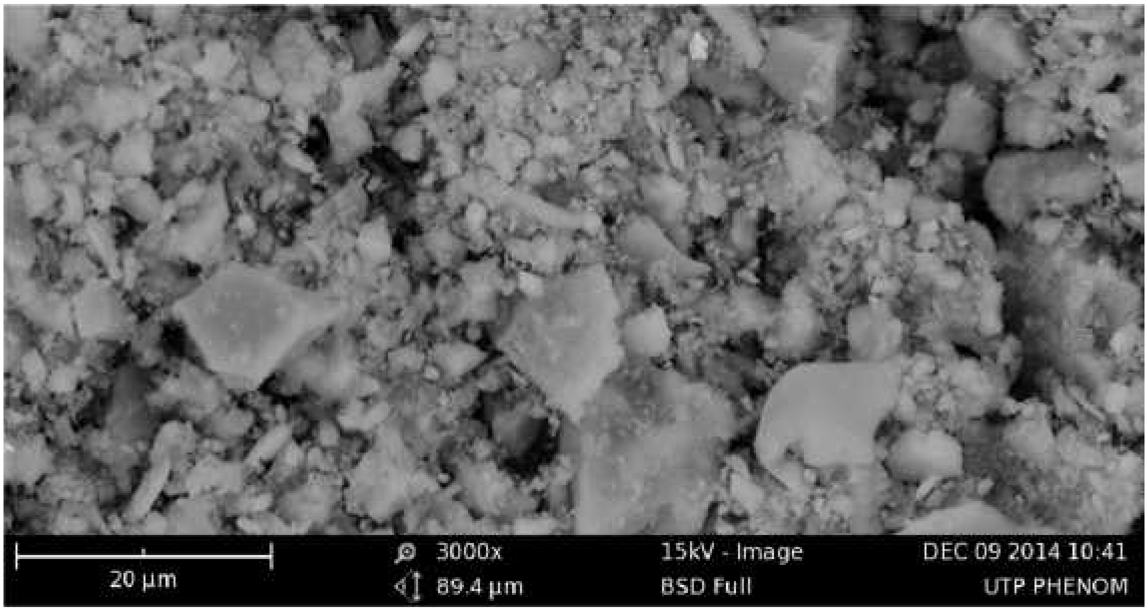

Figure 35: SEM result for X 3000 magnification for 5 hour soaking time and burned with 600°C temperature.

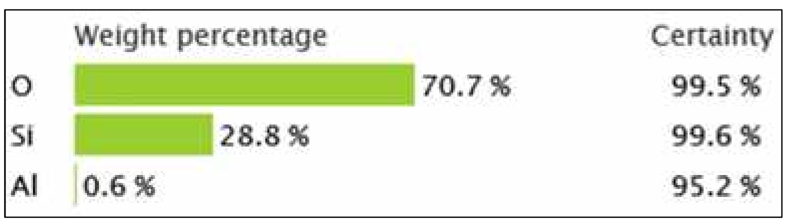

Figure 36: weight percentage rice husk ash for 5 hour soaking time and burned with 600°C temperature.

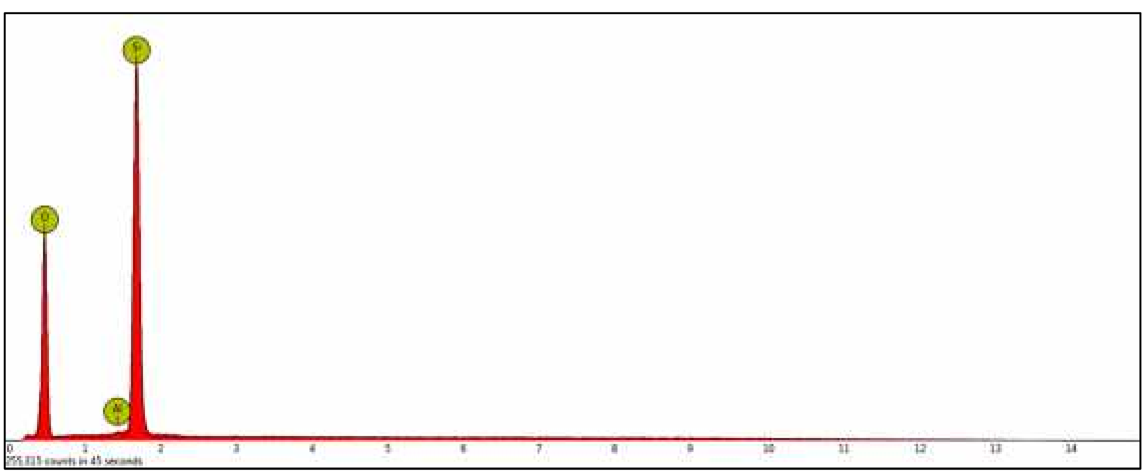

Figure 37: the graph element for 4 hour soaking time and 600°C burning temperature

14	Si	Silicon	100.0	28.8	0.4
8	O	Oxygen	100.0	70.7	0.5
13	Al	Aluminium	100.0	0.6	4.8

Table 8: element number, element symbol, and element name confidence concentration error for 5 hour soaking time with 600°C burning temperature.

The result from scanning electron microscopy (SEM) shown two main chemical elements are silica (Si) and oxygen (O) which are the total of both them contribute more than 90% weight of rice husk ash. Other chemical elements are aluminum (Al) and phosphorus (P). From scanning electron microscopy test it shown the highest silica come from 5 hour soaking time with 600°C burning temperature. The result was proven that 5 hour soaking time will produce high contain of silica.

4.4 BRUNAUER EMMAT TELLER (BET) TEST

Burning temperature °C	Soaking time (H)	Sample name	Specific surface are (m^2/g)	Total pore volume (cm^3/g)	Adsorption average pore width (4V/A by BET)
600	4	A	205.1350	0.292876	57.1089
600	5	B	219.5759	0.293704	53.5038
700	4	C	187.9673	0.262920	55.9501
700	5	D	186.7230	0.255594	54.7537
800	4	E	133.9083	0.182734	54.5849
800	5	F	146.0750	0.231666	63.4375
700(raw)	-	G	4.2286	0.016052	151.8449

Table 9: specific surface area, total pore volume and adsorption average pore with result from BET test.

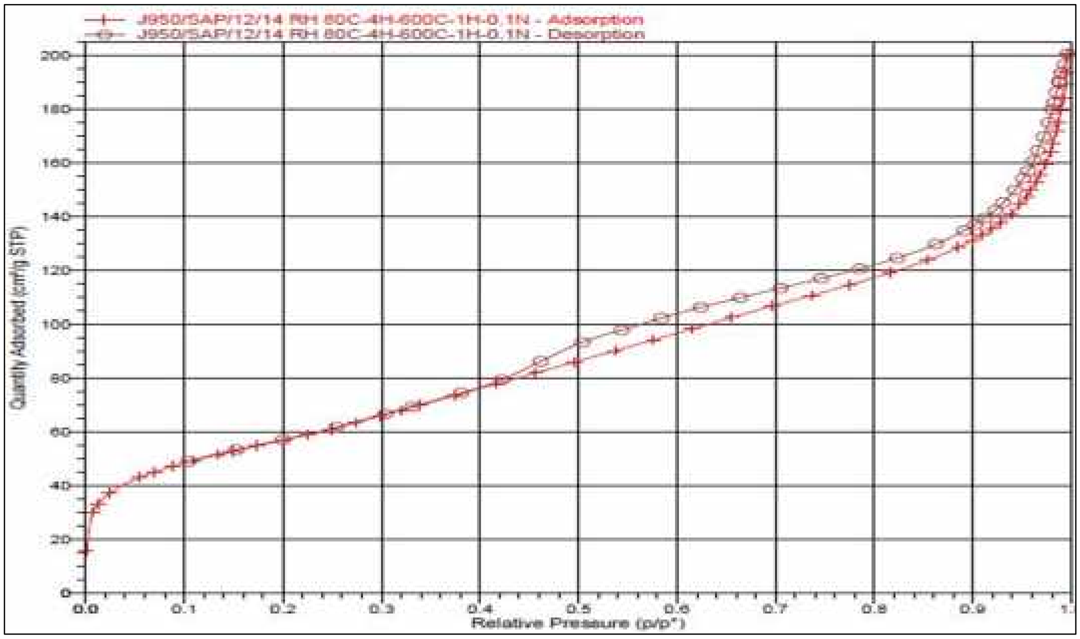

Figure 38: adsorption and desorption nitrogen for 4 hour soaking time and burning 600°C temperature

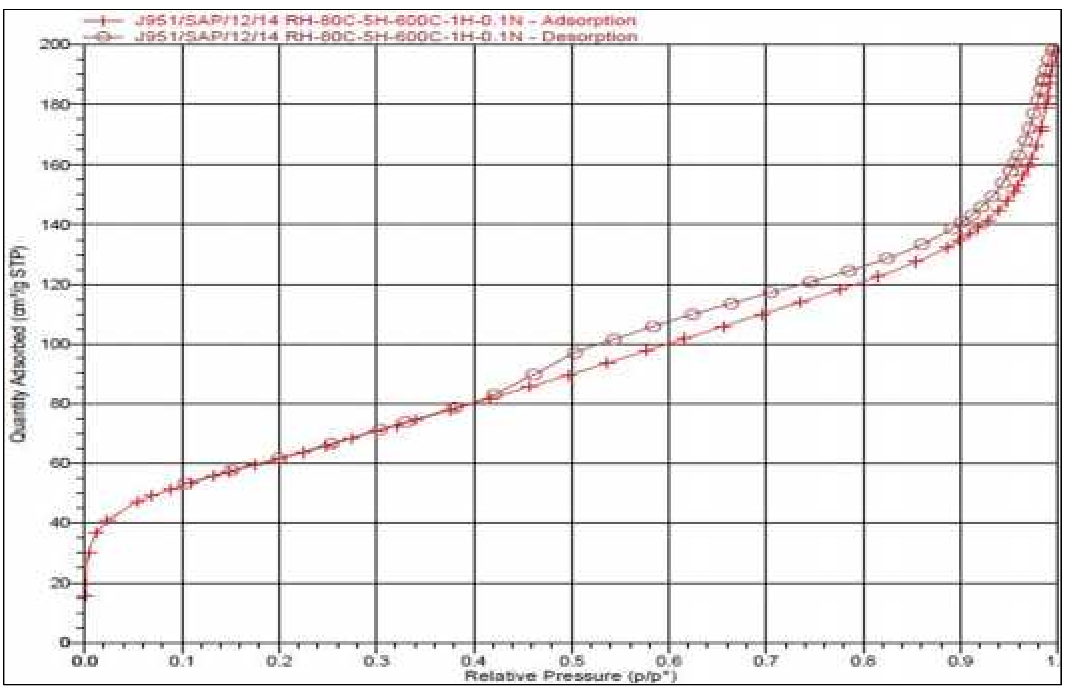

Figure 39: adsorption and desorption nitrogen for 5 hour soaking time and burning 600°C temperature

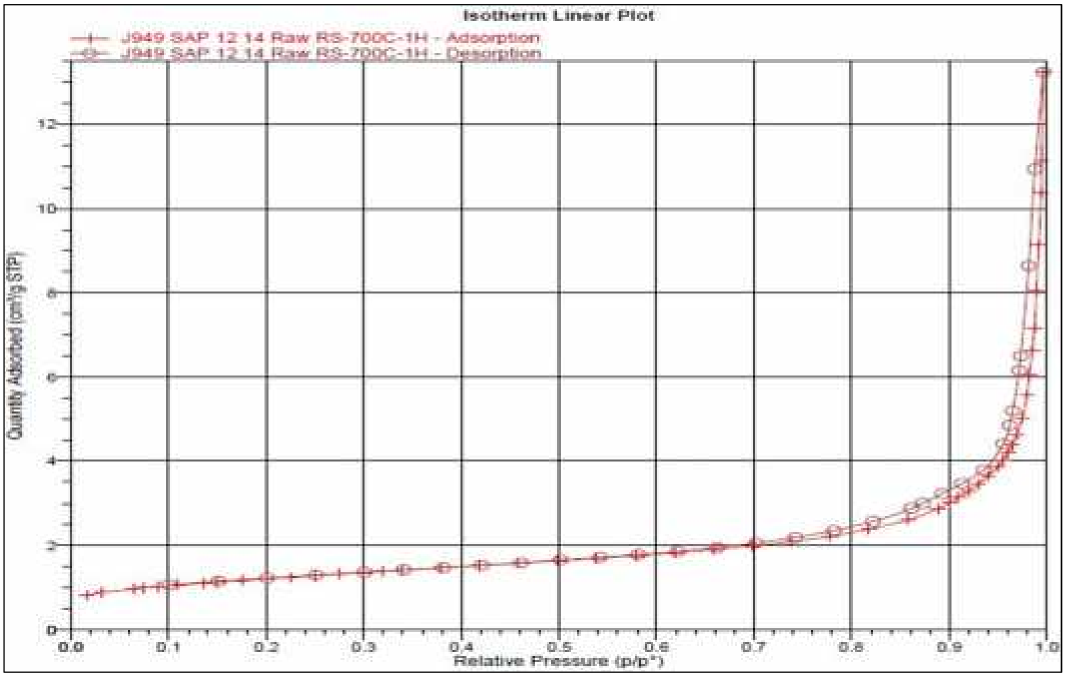

Figure 40: adsorption and desorption nitrogen for raw rice husk ash 700°C temperature

Brunauer–Emmett–Teller (BET) specific surface area, total pore volume and Adsorption average pore width of rice husk ash are shown in table 4.The highest specific surface area is 219.5759 (cm^2/g) which come from sample A. Compared to raw rice husk ash the specific surface area is 4.2286 (cm^2/g). As shown in table 4, the total pore volume for sample A is 0.293704 (cm^3/g), much bigger than 0.016052 (cm^3/g) from sample G which is untreated rice husk ash. The adsorption average pore width (4V/A by BET) for sample A is 53.5038 Å smaller than sample G which is the value 151.8449 Å. The result shown with increasing the temperature for burning process does not help too much in increasing the surface area of rice husk, but the Soaking time giving huge impact toward the increasing of surface area. The surface area also depend on the content of potassium (K), the small content of potassium will bring the big surface area. The figure 33 and 34 has shown the rate adsorption and desorption for sample B and sample A, where the lower portion of the loop is detected out on adsorption loop and the uppermost portion on desorption. The adsorption process for sample A is faster compared to sample B, which indicate the condensation take place at sample A is early compared to sample B.

For the sample G which is raw rice husk, the forces adsorption and desorption almost weak. These results shown that silica obtained from rice husk is porous material.

4.5 PARTICLE SIZE ANALYSIS (PSA) TEST RESULT.

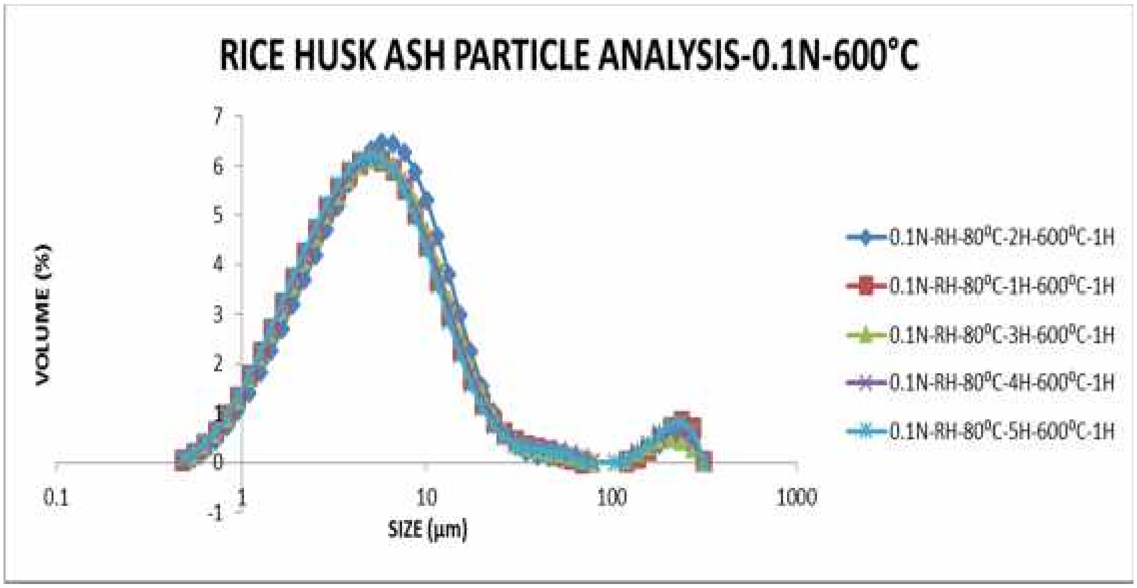

Figure 41: rice husk ash particle analysis for 600°C temperature

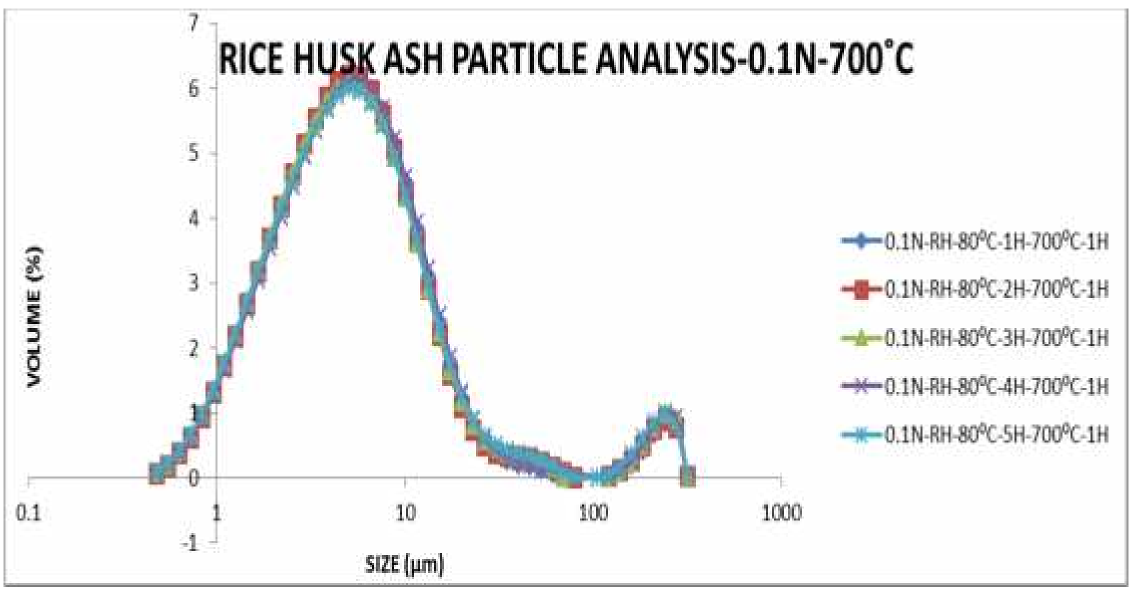

Figure 42: rice husk ash particle analysis for 700°C temperature

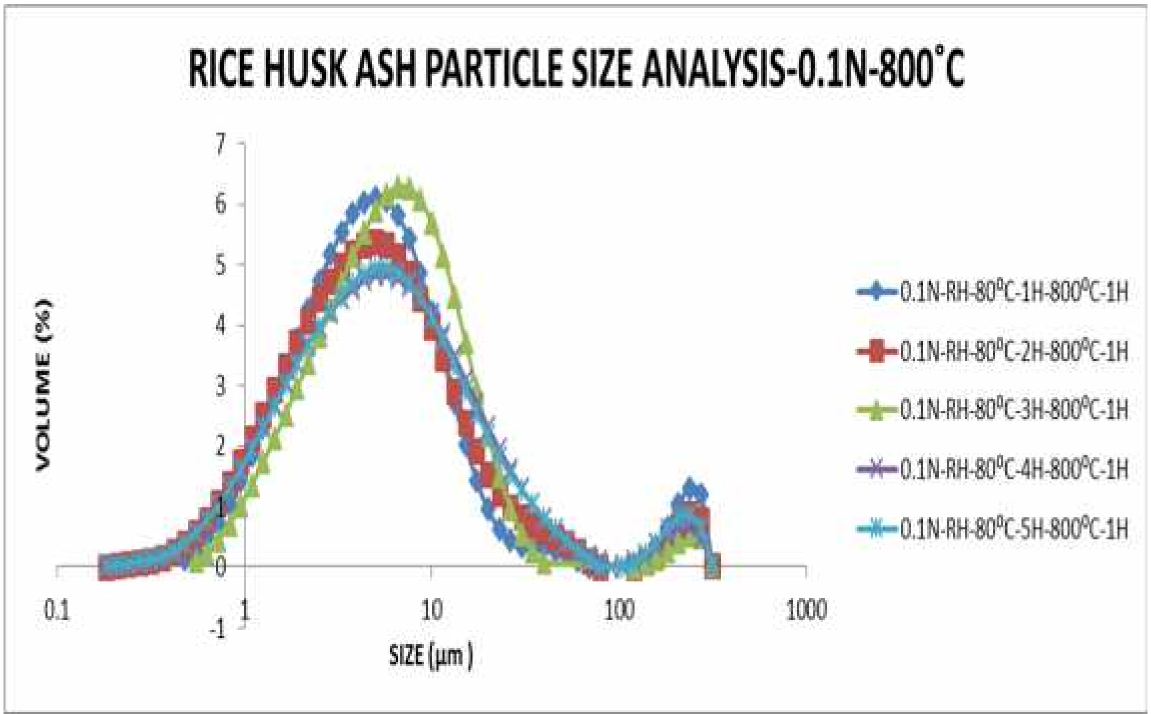

Figure 43: rice husk ash particle size analysis 800°C temperature

Figure 36-38 shown the logarithm graph for particle size analysis of rice husk ash with different adopted burning temperature (600˚C, 700˚C and 900˚C).The grinding time is 15 minutes and 350 revolution per minute (RPM). From the graph shown more than 50 % of particle size is d (0.5) which mean equal to 5 μm and it is the size of ultra-fine silica. The particle size give huge impact toward porosity of concrete. The smaller size would decrease the porosity of concrete.

CHAPTER 5

CONCLUSSION AND RECOMMENDATION

5.1 Conclusion

The conclusion is five hours is the best soaking time where the highest alkaline particle were removed. With adopted 600°C temperature for burning rice husk, it produced high content of silica and also high surface area. The X-ray fluorescence (XRF) test shown the huge different in term of silica contain between the treated and treated of rice husk. From the atomic absorption microscopy (AAS), it shown more higher time for soaking process will remove more alkaline impurities. The soaking time will influenced the surface area and silica content on the rice husk ash. For the particle size, there is no huge different between 600°C, 700°C and 800°C. More than 50 % of particle size is d (0.5) which mean equal to 5 μm and it is the size of ultra-fine silica. For the Brunauer–Emmett–Teller (BET) test, it shown treating rice husk with hydrochloric acid (HCI) the surface area was increased compared with untreated rice husk.

5.2 Recommendation

- To prevent the weight loss during the acid treatment, rice husk should clean with tap and distilled water first and dried it. Just after that, the rice husk will be weighing followed by the acid treatment process. Using the current method, the weight of rice husk will decrease after wash with tap and distilled water after the acid treatment process.
- Ensure the temperature of acid solution is 80°C prior mix it with rice husk. For this research the acid and rice husk was mixed before the temperature of solution (acid) reach 80°C. This will influence the result for atomic absorption spectroscopic (AAS) and also for surface area for silica.

- For the burning process, the time for all sample to burn is homogenous. But the confinement time after the burning process ending is different for each sample. This also will influenced the result for surface area and silica content.
- For the burning process we using tray as mold to put the rice husk inside the furnace. For every time finish the burning process, ensure clean and dried the tray first prior to use it back for new sample to burning.
- Increase the time and revolution per minute (rpm) for grinding process. Currently the time for grinding process is 15 minutes and revolution per minute (rpm) is 350.

REFFERENCE

[1]. Bie, R.-S., Song, X.-F., Liu, Q.-Q., Ji, X.-Y., & Chen, P. (2015). Studies on effects of burning conditions and rice husk ash (RHA) blending amount on the mechanical behavior of cement. *Cement and Concrete Composites*, *55*, 162–168. doi:10.1016/j.cemconcomp.2014.09.008

[2] .Carmona, V. B., Oliveira, R. M., Silva, W. T. L., Mattoso, L. H. C., & Marconcini, J. M. (2013). Nanosilica from rice husk: Extraction and characterization. *Industrial Crops and Products*, *43*, 291–296. doi:10.1016/j.indcrop.2012.06.050

[3]. Gholizadeh Vayghan, a., Khaloo, a. R., & Rajabipour, F. (2013). The effects of a hydrochloric acid pre-treatment on the physicochemical properties and pozzolanic performance of rice husk ash. *Cement and Concrete Composites*, *39*, 131–140. doi:10.1016/j.cemconcomp.2013.03.022

[4]. Liou, T.-H. (2004). Preparation and characterization of nano-structured silica from rice husk. *Materials Science and Engineering: A*, *364*(1-2), 313–323. doi:10.1016/j.msea.2003.08.045

[5]. Liou, T.-H., & Yang, C.-C. (2011). Synthesis and surface characteristics of nanosilica produced from alkali-extracted rice husk ash. *Materials Science and Engineering: B*, *176*(7), 521–529. doi:10.1016/j.mseb.2011.01.007

[6].Rafiee, E., Shahebrahimi, S., Feyzi, M., & Shaterzadeh, M. (2012). Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material). *International Nano Letters*, *2*(1), 29. doi:10.1186/2228-5326-2-29

[7].Sarangi, M., Nayak, P., & Tiwari, T. N. (2011). Effect of temperature on nano-crystalline silica and carbon composites obtained from rice-husk ash. *Composites Part B: Engineering*, *42*(7), 1994–1998. doi:10.1016/j.compositesb.2011.05.026

[8].Singh, D., Kumar, R., Kumar, A., Rai, K. N., Programme, M. S., & Engineering, M. (2008). Synthesis and characterization of rice husk silica , silica-carbon composite and H 3 PO 4 activated silica, *54*, 203–212.

[9].Yalc, N., & Sevinc, V. (2001). Studies on silica obtained from rice husk, *27*, 219–224.

[10]. Chao-Lung, H., Anh-Tuan, B. Le, & Chun-Tsun, C. (2011). Effect of rice husk ash on the strength and durability characteristics of concrete. *Construction and Building Materials*, *25*(9), 3768–3772. doi:10.1016/j.conbuildmat.2011.04.009

[11]. Ceram. (n.d.). Retrieved from ceram: http://www.ceram.com/testing-analysis/techniques/brunauer-emmett-teller-surface-area-analysis-barrett-joyner-halenda-pore-size-and-volume-analysis/

[12]. http://www.labtesting.com/services/materials-testing/metallurgical-testing/sem-analysis/ Lab testing. (n.d.). Retrieved from labtesting:

[13] Ceram. (n.d.). Retrieved from ceram: http://www.ceram.com/testing-analysis/techniques/x-ray-diffraction-xrd

[14] Bruker. (n.d.). Retrieved from bruker: http://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/x-ray-fluorescence.html

[15] *easychem.* (n.d.). Retrieved from easychem: http://www.easychem.com.au/monitoring-and-management/manufactured-products/atomic-absorption-spectroscopy-aas

[16] Cienec. (2012, Sept/Oct). *scielo.* Retrieved from http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1413-70542012000500010

[17] Rubenstein, M. (2012, september). *blogs.ei.* Retrieved from ee5the-cement-Industry/

[18] 2012, Disember). Retrieved from vodppl: http://www.vodppl.upm.edu.my/uploads/docs/Perangkaan%20Padi%20Malaysia%202011.pdf

APPENDICES

Figure 25: water sample for 0.1M and 4 hours soaking time.

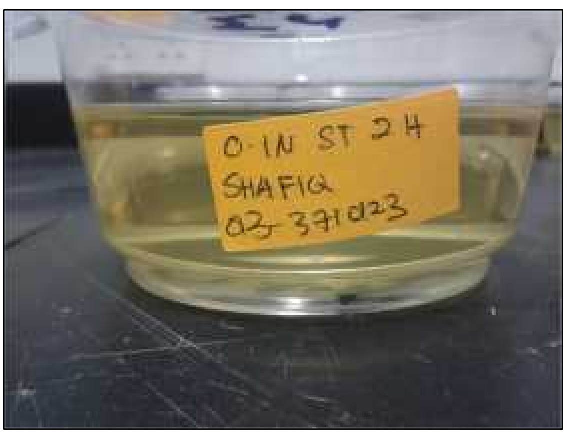

Figure 26: water sample for 0.1M and 2hours soaking time.

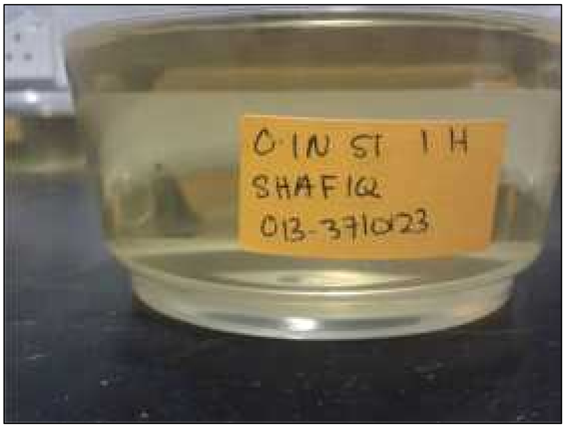

Figure 27: water sample for 0.1M and 1 hours soaking time.