POZZOLANIC EFFECT OF RICE HUSK ASH IN PASTE

by

CHIA SETT HAN

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University Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

POZZOLANIC EFFECT OF RICE HUSK ASH IN THE PASTE

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 acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Your faithfully,

(Chia Sett Han)

Approved by,

(Dr. Victor R.Macam)

ABSTRACT

Objectives

The research of this final year project is to investigate the pozzolanic effect of rice husk ash in the cement/binder paste. In order to attain this main objective, there are several tasks need to be carried out. In other word, there are several sub-objectives need to be attained to achieve the main objective of this research. The sub-objectives of this research will be discussed clearly in the introduction.

Problem Statement

The production of rice husk ash is the main topic concerning in this research. Rice husk ash produced must fulfill two basic requirements, the ash must contain at least 80% of silica and it must attain amorphous phase so that the ash is chemical reactive in the mixture. Thus, the high quality ash can be obtained by controlling the burning process of the rice husk. A standard burning procedure had been fixed and the detail of the burning procedure is discussed in detail in the methodology section.

The fines of the rice husk ash will be determined by using Blaine Air Permeability test and Le Chatelier flask test. However, the equipment of Le Chatelier flask is not available in the university. Thus, the test had to be carried out by using ordinary conical flask. The concept and procedure for Le Chatelier Flask test will maintain the same for ordinary conical flask test but the result obtained for Le Chatelier Flask test will be more accurate than the ordinary conical flask due to the different equipment calibration between both tests.

Water/cement-binder ratio is supposed determining by using the flow tablet but due to unavailability of flow table in the university, Vicat test had been chosen to determine the water/cement-binder ratio. The water/cement-binder ratio is maintained constant previously in this research but the selected water/cement-binder ratio is not suitable for all the mixtures especially mixtures that incorporate higher percentage of RHA. Therefore, water/cement-binder ratio for all the mixtures will be determined by carrying out the Vicat test to ensure that the mixtures will undergo complete hydration process. Thus, we had fixed one water/cement-binder ratio for every mixture, but one

value of W/C ratio for each mixture cannot be justified as the optimum W/C ratio that gives the optimum compressive strength for the each mixture. Thus, we choose a few more water/cement-binder ratio for each mixture so that we can obtain a curve that will give the optimum compressive strength for all the mixtures.

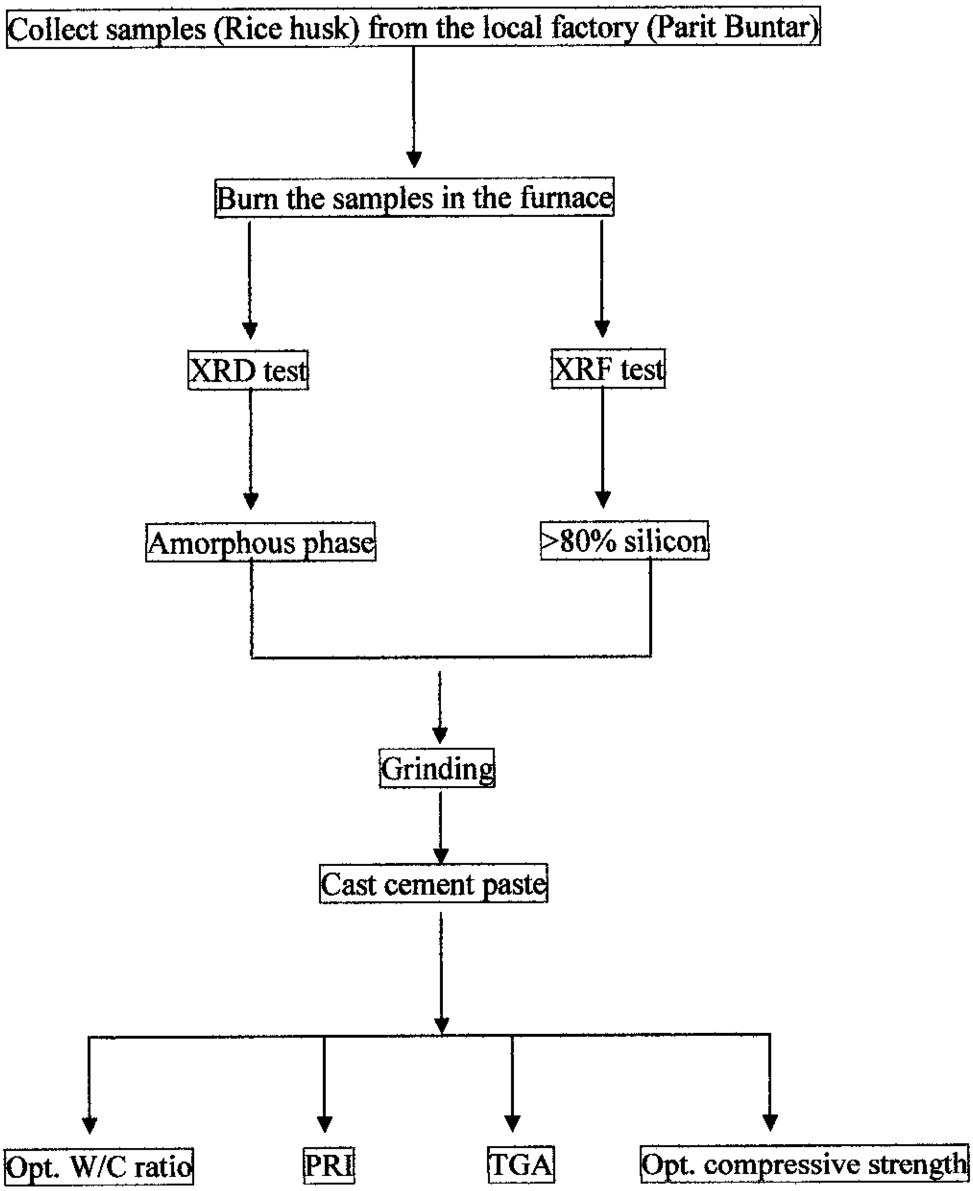
The degree of hydration of the paste can be obtained by carrying out the TGA test. The paste at the age of 28 days will be grinded and will be tested in the TGA machine. Since there are a lot samples to be tested, the optimum compressive strength for each samples will be chosen to undergo the TGA test.

Scope of Study

The scope of studies for this final year project is stated as below:

- X-ray diffraction and X-ray fluorescent
- Finesses of rice husk ash
- Pozzolanic reactivity index
- Thermal analysis (TGA)
- Compressive strength of cement-binder paste

Methodology and Findings



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CHAPTER 1

INTRODUCTION

1.1 Background of study

Concrete is the main construction material in the world nowadays and researchers are trying to improve the properties of concrete so that the structure can last longer and can sustain heavier load. Various kind of methods had been investigated by researcher to improve the properties of concrete and one of the method that intensively carried out by the researchers are adding pozzolanic materials into the concrete to increase the cement gel in the concrete. For instance, fly ash, silica fume, GGBS and so forth had been successfully proven can improve the properties of the concrete. These materials are selected as the admixture in the concrete due to the major content of silica in the materials. Silica is important for the production of extra cement gel in the concrete.

The reactions occur inside the concrete is showing as below:

$$2CaO.SiO_2 + 6H_2O \rightarrow 3CaO.SiO_2.3H_2O + 3Ca(OH)_2$$
$$2SiO_2 + 3Ca(OH)_2 \rightarrow 3CaO.SiO_2.3H_2O$$

From the equation, we can observe that RHA will react with calcium hydroxide to perform the pozzolanic reactivity, which will contribute to the formation of extra cement gel in the concrete. Thus, the higher the composition of silica inside the RHA, more cement gel will be generated.

Since years ago, researchers had investigated agriculture materials such like rice husk, oil palm husk, coconut husk etc. because these materials having the cementitious properties which potential to improve the properties of the concrete. However, rice husk is the most favorite topic discussed among the researchers because it contains very high amount of silica and the physical and chemical characteristic of rice husk are approximate the same as silica fume. Silica fume had been used in many constructions especially high rise building and offshore structure. Anyway, the research done for rice husk is still carrying on in order to have more compatible result among all the researchers.

1.2 Problem statement

Burning procedure

Burning process is a very important procedure that needs to be considered in order to produce a high quality ash. Temperature and time are the main factors that governed the whole process of the burning. For your information, the rice husk ash required after burning must fall in amorphous phase and the ash must contains more than 80% of silica, so that, the ash will have sufficient silica to form the cement gel with highly chemical reactivity. To achieve these criteria, the temperature and duration for the burning must be totally control so that the rice husk can me homogenously burnt into high quality ash.

According to the guideline given by the master student, Mr. Kassim, he recommends that the burning process have to divide into few stages. Each stage serves different kind of purposes and the main purpose to divide the burning process into few stages is to get rid organic material and carbon from the rice husk. By having organic material and carbon in the rice husk ash, it will not improve the property of concrete instead; it will have inversely effect on the concrete. For instance, carbon is an unreactive material in the concrete so, it will not react with any components in the concrete to produce useful material.

Other than that, the rice husk cannot be burnt exceed than certain temperature – 600^0C because it will change the ash into quartz. When the ash is changed into quartz condition, the ash will not chemical reactive in the concrete. In this project, a standard procedure had been developed and this procedure is being verified as the best procedure for producing the high quality ash. The burning procedure is discussed clearly in the methodology section.

Finesses

Finesses of rice husk ash is important in determining the property and characteristic of concrete. The finer the particle of rice husk ash, the chemical reaction in the concrete will be increasing. The finer particle will fill in the pore of concrete and make the concrete less penetration from chemical component like chloride and sulphate. Beside that, the concrete will gain higher density and it will increase the compressive strength of the concrete. Thus, the process for grinding will become an important factor in determining the finesses of the RHA.

In this project, the equipment used to grind the RHA is Los Angelas machine. The detail procedure for the grinding process will be discussed in the methodology section. For the fines test of RHA, the equipment used is Blaine Air Permeability test. This test requires the value of density of RHA in order to calculate the fines of RHA. Thus, the density of RHA will be determined by using the Le Chatelier test. The equipment for Le Chatelier test is unavailable in the university so the test will carry out by using the normal conical flask. The concept is the same as Le Chatelier test, the major different is equipment calibration for Le Chatelier test is better than normal conical flask.

Water/cement-binder ratio

According to the researchers, mixture with the incorporation of RHA is demand higher content of water to undergo complete hydration process. Thus, the water/cement-binder ratio is various for each mixture with different content of RHA in order to attain the complete hydration process. To determine the optimum water/cement-binder ratio, a graph of compressive strength against water/cement-binder ratio had to be plotted for each mixture. Thus, we decided to choose at least five different water/cement-binder ratios for each mixture to obtain the curve.

The water/cement-binder ratios actually are chosen according to the Vicat test result. The Vicat test result approximately will tell us the optimum water/cement-binder ratio, which give the optimum compressive strength of the paste. Therefore, a nice curve will create by using the randomly chosen water/cement-binder ratios. In this project, we manage to carry out the casting up to mixture with 7.5% incorporation of RHA due to lack of time and also insufficient RHA. The analysis for this topic will be discussed in detail in the discussion section.

1.3 Objective and scope of study

The objective of this project is listing as below:

a) To produce a high quality rice husk ash to incorporate in the paste.
b) To determine the finesses of rice husk ash.
c) To find out the optimum water/cement-binder ratio for the optimum compressive strength of cement/binder paste.
d) To determine the pozzolanic reactivity index for the optimum compressive strength of the paste.
e) To find out the degree of hydration of paste at the age of 28 days.

The scope of studies for this final year project is stated as below:

- X-ray diffraction and X-ray fluorescent
- Finesses of rice husk ash
- Pozzolanic reactivity index
- Thermal analysis (TGA)
- Compressive strength of cement-binder paste

CHAPTER 2

LITERATURE REVIEW

2.1 Burning for rice husk ash

According to the website published by [Uwe Brunjes], the world's primary staple crop which is rice had generated 100 millions tons of hulls or chaff annually. Rice farmer utilized the rice husk to generate power by burning and by this way of burning; it can generate the desired quality of ash for pozzolanic use. Therefore, for every 1 ton of paddy milled, about 200kg of husk is produced and when the husk is burnt in the kiln, the production of RHA in kiln is approximately 50kg. This kind of burning may not able to produce a high quality ash and the ash may contain many unburned substances such like carbon, which is not chemical reactivity in the concrete. If the ash is totally out of carbon, the ash may be crystallized which it is not chemically reactivity in the concrete.

According to research done by [Mazlum, F., and Uyan, M, 1992], they found that the burning rice husk at 600^0C produces an ash with an optimum composition for pozzolanic materials. They comment that the silica formed is of pozzolanic nature below the temperature and above the temperature, the silica partially changes to cristobalite, quartz and tridymite, which require fine grinding to exhibit pozzolanicity. Thus, it is suggestible to burn the ash into amorphous phase (below 600^0C) rather than in crystallize phase so that all the silica inside the ash can be chemical reactivity in the concrete. [Qijun yu[b], K. Sawayama[a], S. Sugita[a], M. Shoya[a], Y. Isojima[a], 2003] mention that when the rice husk is heated, it gradually loses the water it contains, but it will keep an amorphous form up to 750^0C. above 780^0C it will be transform to crystalline $CaSiO_3$ (wollastonite).

[Metha, P.K, 1979] mention there are over 400 million tones vast quantities of rice husk annually produced from around 16 million tones of pozzolanic ash could be potentially be produced. Relative to other organic, rice husk yields a high percentage of ash (22%) with a very high silica content, typically 93%. Under controlled combustion conditions the form of the ash varies depending on the combustion conditions. From 400 to 500 ^{0}C the silica is amorphous, at around 600^0C quartz may occur and above this temperature, other crystalline forms are produced, such as crystobalite. Combustion time also influences the final form. Under certain conditions an active form of silica can be produced and the silica activity index has been shown to be related to the amorphousness of the silica in the ash. [P R S Speare, K Eleftheriou and S Siludom] obtain more than 70% for silica activity index.

[M. Nehdi*, J. Duquette, A. El Damatty, 2003] found out that a new technology for the production of RHA based on a Torbed reactor allowed producing highly reactive RHA with much lower carbon conent than that of RHA produced using fluidized beds. The technique was apply from Egypt, and the performance of the resulting RHA was compared to those of silica fume and RHA produced in the United States using fluidized bed technology. The RHA produced using the new technique required less grinding time than that produced in a fluidized bed and did not substantially increase the water demand and the superplasticizer requirement, which is an advantage compared to silica fume.

[R. Jauberthie[a,*], F. Rendell[a,1], S. Tamba[b,2], I. Cisse[b,3], 2000] conclude that the role of rice husk ash as a filler and its pozzolanic properties have been reported by several authors; we have verified the pozzolanic property of an ash from Senegal and identified the origin of the amorphous silica. In the light of this study one can see that the rice husk and the rice husk ash contains strong concentration of silica occurring in a crystalline quartz and amorphous forms. The amorphous silica occurs principally on the external face of the husk and to a lesser concentration on the inner surface. This amorphous silica and to a lesser extent the fine grained quartz explains the previously observed pozzolanic role. The crystalline silica found on the husk before calcinations indicates that its presence, in the ash, is probably due to contamination by sand.

2.2 Finesses of rice husk ash

[I K Cisse, R Jauberthie M Temimi and J P Camps, 1999] found out the following characteristic of ground rice husk ash:

- The maximum particles size is 100micrometer
- 50% of the particles have a size smaller than 10micrometer
- the volume percentage of particles smaller than 7 micrometer is 35%
- The specific area is 6960 cm^2/g

Researchers [I K Cisse and M Laquerbe, 2000] conclude that the rice husk ash had high absorption of water. The pozzolanic reaction can be achieved highly if the particle size of the rice husk is fine where the size is smaller than 7 micron. According to researcher, [M. H. Zhang, R.Lastra and V. M. Malhotra, 1996], in general the average particle size ranges from 5 to 10 micrometer and the specific surface are ranges from 20 to 50m^2/g. From here, we know that the particle size for rice husk ash is lower than the cement and the low surface area of the rice husk ash will increase the density of the concrete and increase the rate of chemical reaction in the concrete.

2.3 Water/cement-binder ratio

According to [Muhammad Shoaib Ismail and A. M. WaliuddintS, 1996], RHA demand more water for workability. This mean that, higher water/cement-binder ratio have to adjust to accommodate the higher water demand from the RHA unless there is superplasticizer added into the sample to adjust the flow for paste and workability for concrete. From the literature studies, no author is carrying out the experiment without the using of superplasticizer such as naphthalene. The water/cement-binder ratio normally used by the researchers are 0.3 and above depending the flow of the paste and the workability of the concrete.

From here, we know that the water demand for different amount of RHA incorporate into the paste would need different water/cement-binder ratio. In addition, higher water/cement-binder ratio will slightly decrease the compressive strength of the sample. Thus, there must have an optimum water/cement-binder ratio for all the mixtures, which will give the optimum compressive strength. Superplasticizer might can solve this problem but to minimize the factors that will affect the compressive strength, superplasticizer is not used in the research we done.

2.4 Optimum amount of RHA incorporation in concrete

From the journal 'Effect of rice husk on high strength concrete' written by [Muhammad Shoaib Ismail* and A. M. Wailiuddin, 1996], they comment that the possibility optimum amount replacement of cement by RHA will be around 10% to 20% with fine ground RHA but base on their compressive strength result, they find out that the optimum amount of RHA is 10%. From journal 'Concrete incorporating rice husk ash : compressive strength and chloride ion penetrability' written by [N. Bouzoubaa and B. Fournier, 2001] they found out that the optimum compressive of concrete is with the RHA incorporation of 10% which is same with the previous researchers.

However, there is one journal name 'Physical and pozzolanic action of mineral additions on the mechanical strength of high performance concrete' written by [G.C. Isaia, A.L.G. Gastaldini, R. Moraes, 2001] showing different comment. They find out that the optimum amount of RHA incorporation in the concrete is falling at 25% content of RHA. The author also recommends the compressive strength of concrete may get higher if the amount of RHA increases more than 25%. In my opinion, the incompatible results obtain by these researchers may cause by the different water/cement ratio. Up to this moment, all the authors believe that the higher the water/cement ratio may give the lower compressive strength but with the incorporation of RHA in the concrete, the effect of water/cement-binder ratio may be changing due to the higher absorption water of RHA.

2.5 The correlation between unitary compressive strength, unitary calcium hydroxide and unitary combined water contents.

According to [G.C. Isaia, A.L.G. Gastaldini, R. Moraes, 2001], the correlation between unitary compressive strength, unitary calcium hydroxide and unitary combined water contents show that when concrete has to reach a given level of compressive strength, it is necessary to achieve a minimum unitary strength with a give minimum content of combined water and/or remaining calcium hydroxide. The unitary compressive strengths increased sharply as the pozzolan content in the concrete mixtures did. On average, they grew from 22%, for 12.5% addition content, to 79%. For 50% addition content, when compared to the reference mixture. The physical effect contribution to these values was less significant (+2% and +34% respectively) than that of the PE (+20% and +45%, respectively).

[M.H.Zhang*, R. Lastra*, and V.M. Malhotra**,1996)] found out that for ordinary portland cement paste, the calcium hydroxide and calcium silicates hydrates were the major hydration and reaction products in the RHA paste. because of the pozzolanic reaction, the paste incorporating RHA had lower calcium hydroxide content than the control portland cement paste. The cement paste incorporating RHA did not show any increase in the compressive strength compared with that of the control Portland cement paste. the higher compressive strength of the RHA concrete compared with that of the control concrete is due to probably to its reduced porosity, reduced $Ca(OH)_2$ content, and the reduced width of the interfacial zone between the paste and aggreagate.

[Muhammad Shoaib Ismail* and A. M. Wailiuddin, 1996] found out that their experiment result possible to get a strength of 70 MPa in Pakistan with locally available ingredients of concrete. The result also confirms that it is possible to get high strength concrete economically using RHA by burning locally available rice husk. They also observed that even the RHA is in crystalline formation, good compressive strength results may be obtained by fine grinding.

CHAPTER 3

METHODOLOGY

3.1 Burning procedure

Burning procedure is a very important step towards obtaining the high quality RHA. The rice husk is subjected to thermal combustion in the furnace, which is based on the conduction and convention theory. The burning procedure is developed by having trials and errors burning and the quality of ash can be examined by physically and chemically. The ash is physically examined by looking at the ash right after the burning. If the ash contain carbon (Unburnt rice husk), the ash is considered fail as the high quality ash.

Chemically examination can be done by X-ray diffraction test and X-ray fluorescent test. These tests are done to ensure the ash is falling under amorphous phase and to ensure the ash contains more than 80% of silica. After numerous trials and errors, a standard procedure had been developed base on the trial burning procedure and the standard procedure developed for all the burning is shown in the following section. The burning guideline is provided by Mr. Kasim (master student) who assists us to carry out this project.

Preparation of sample on the platform

The platform of the rice husk had been constructed by using the BRC materials so that homogenous burning of the samples can be achieved. The platform is specially designed to suit the size of the furnace available in the university. The size of the furnace can fit two platforms and each platform is separating into two layers. Each layer can accommodate maximum 500g of rice husk or 1.5 – 2 inches from the bottom of each layer.

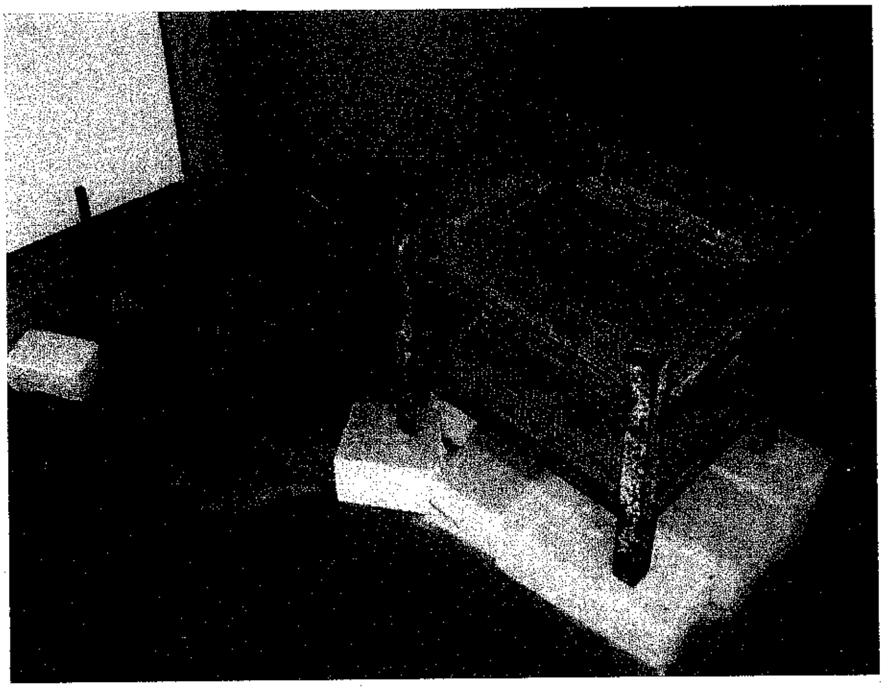


Figure 7: The picture above shows the platform for the rice husk.

Figure 3: The picture above shows some samples of rice husk.

First stage

The main purpose of this stage is to remove the moisture and volatile matters in the samples. The samples will be burnt below the ignition point, which is 380^0C and the temperature increment is approximately 10 – 20^0C/min from the room temperature. The presence of air is essential in this stage because the diffusion of hot airs appears to be dominant mode of the heat transfer. This stage will be last until all the black smoke subsides. At this stage, the cover of the furnace needs not to be slightly opened.

Step	**Temperature (^{0}C)**	**Duration (min)**
1	24(room temperature) – 200	10 – 15
2	200	10
3	200 – 250	5
4	250	5
5	250 – 280	4 – 5
6	280	10
7	280 – 310	3 – 4
8	310	10
9	310 – 360	5
10	360 (duration is depend on the observation of the smoke; when the smoke is finish flowing out, straight away go to the next stage)	15 – 20
		Total = 77 min – 89 min

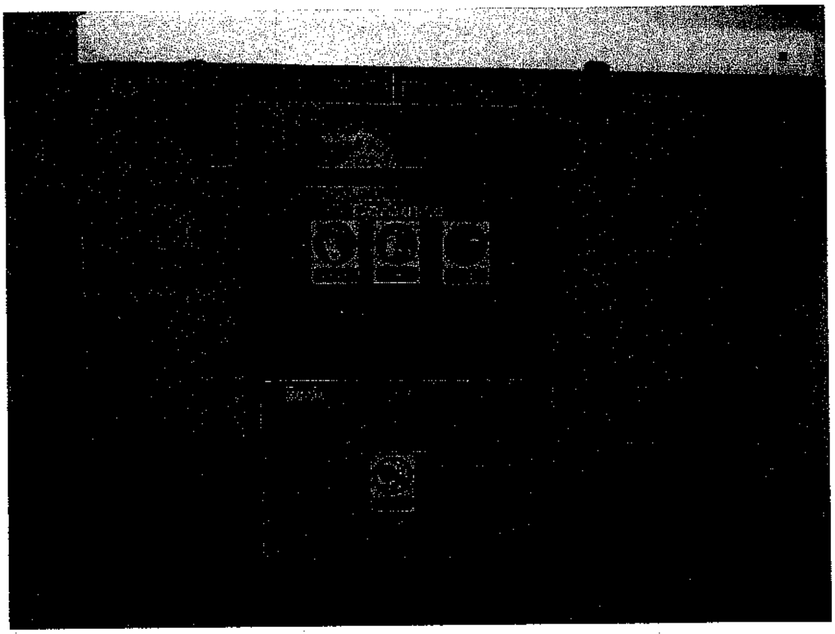


Figure 1: The picture above shows the big furnace used to burn the rice husk.

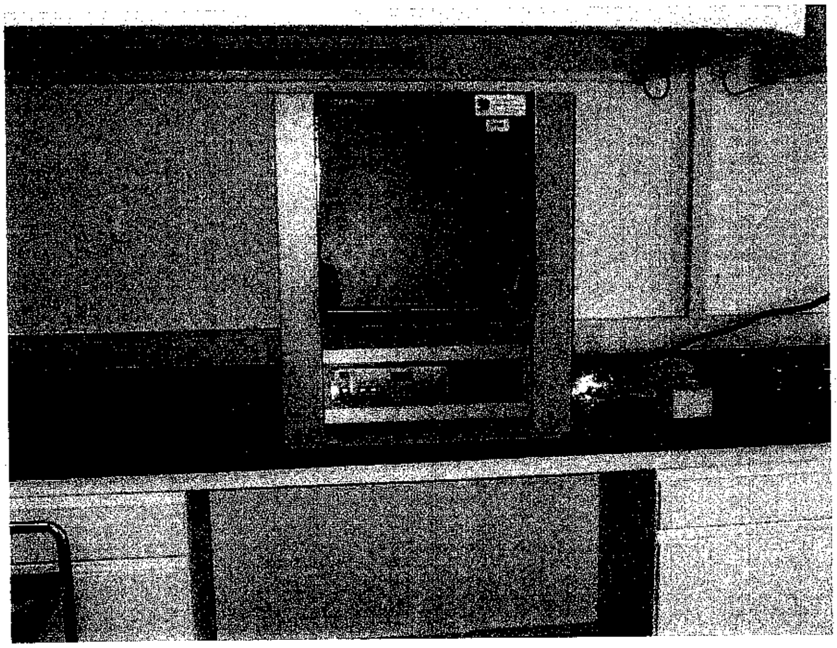


Figure 2: The picture above shows the small furnace used to burn the rice husk.

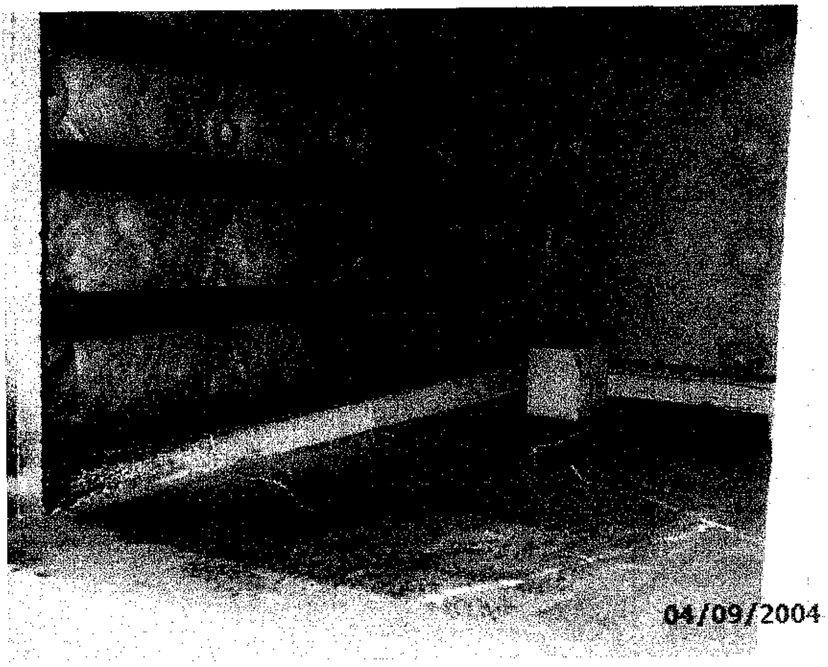


Figure 6: The picture above shows the internal view of the big furnace.

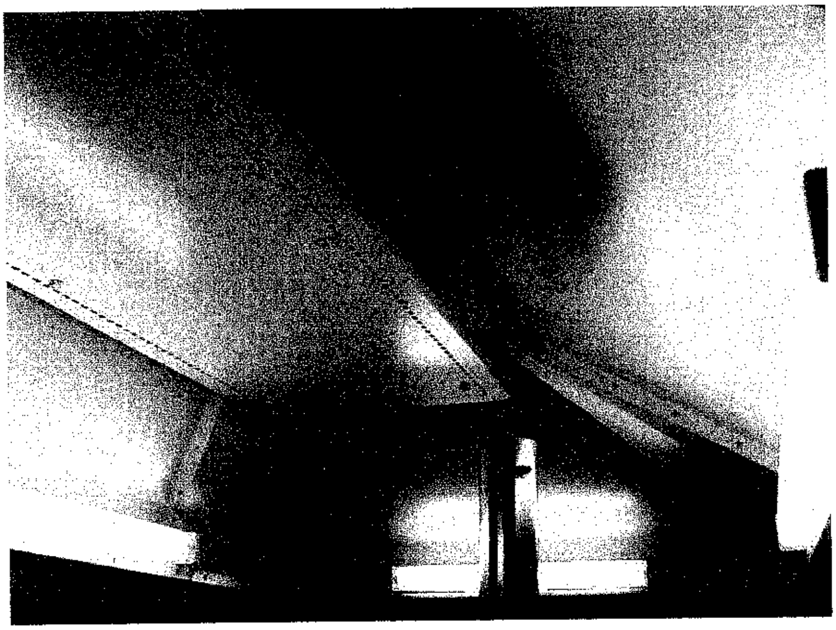


Figure 5: The picture above shows the smoke release from furnace during burning.

Second stage

At this stage, the temperature is increased and maintained at 480^0C – 520^0C. The presence of air is essential to oxidize the residual carbon from rice husk and palm oil husk. It is because air is an oxidizing medium. At this stage, the cover of the furnace needs to be opened slight to allow the air to flow in.

Step	Temperature (^{0}C)	Duration (min)
1	360 – 480 (the temperature must be adjusted to 550 at the beginning and adjust down to 530 when the temperature reach 480, the temperature will maintain at 480 - 490)	10 - 15
2	480	30 - 35
		Total = 40 min – 50 min

Third stage

At the last stage, the temperature is increased to 550^0C in the absence of air. It is due to the exothermic reaction in the furnace where the flow of cool air in the previous stage may cool down the husk. Thus, the temperature is increased to heat up the rice husk for the final burning. The temperature must not exceed 600^0C.

Step	Temperature (^{0}C)	Duration (min)
1	(480 – 490) – 520	5 - 8
2	520	5
3	520 - 550	5 – 7
4	550	10
		Total = 25 min – 30 min

**The duration for burning the rice husk ash is 142 min – 169min. The weight of the final product (RHA) is within 200 g – 250 g or 10 % - 12.5 % of the weight of the rice husk ash.*

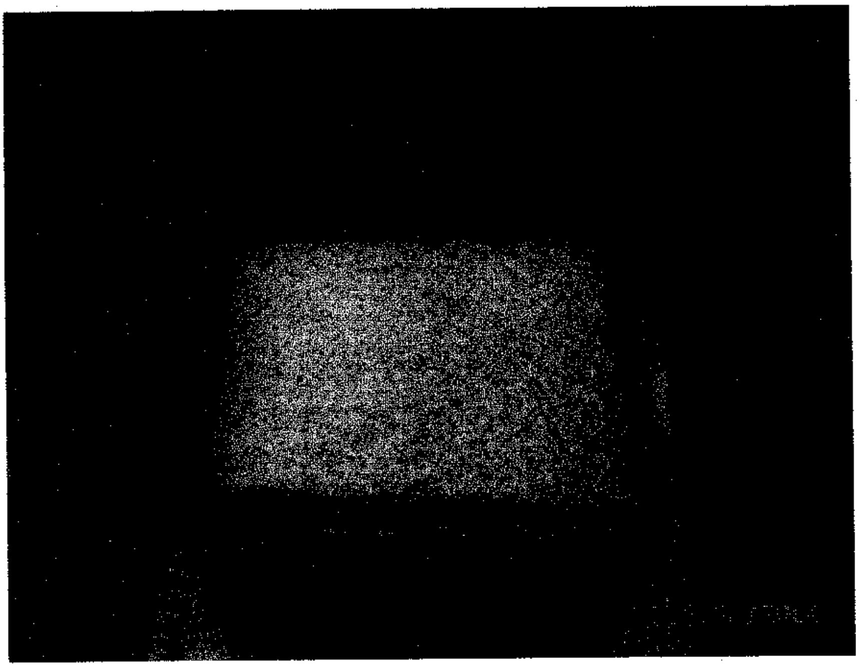


Figure 4: The picture above shows the production of rice husk – rice husk ash.

3.2 Particle density (specific gravity).

a) Objectives: to determine the density and water absorption of RHA and cement.

b) Equipment:

1) Incubator
2) 100ml conical flask
3) 100 g and 500g cement, 100g RHA
4) Kerosene
5) A sample tray
6) 100ml cylinder

c) Procedure (Refer to ASTM C188 – **Appendix 1**):

1. A sample (cement or RHA) about 100g in the tray is weighted.
2. The flask was filled in with 100 ml of kerosene.
3. The flask then will be put into the incubator with the temperature at 23±2^{0}C to cool down the sample.

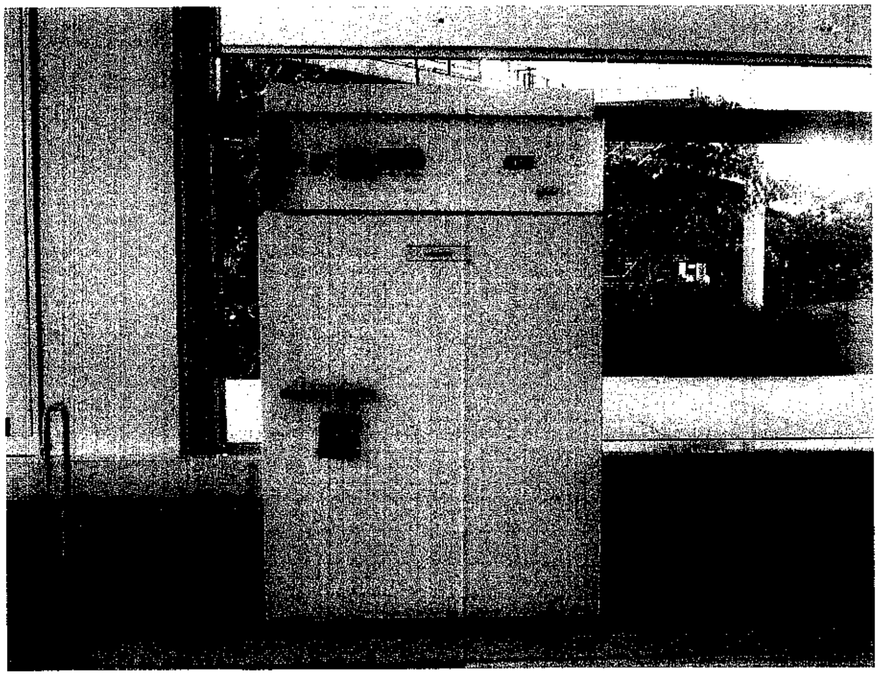


Figure 9: The picture above shows the incubator use to cool down the temperature of sample inside the conical flask that fill with cement. RHA and kerosene.

4. The kerosene level will drop from the final position (100ml) and refill the flask with kerosene and repeat the step until the level of kerosene maintain at the final position.
5. After the final position had achieved, take out kerosene inside the flask (remain portion of kerosene to avoid sample sticking on the flask) into a cylinder and fill the flask with cement weighted 100g.
6. Refill the kerosene into the flask, shake the flask thoroughly to ensure the sample is fully immersed in the kerosene, and ensure that the bubbles do not exist in the flask.
7. After the kerosene had filled into the flask up to the final position, install the flask into the incubator to cool down the temperature to 23^0C. If the level of the kerosene is dropping, instantly refill the kerosene back into the flask and cool down the flask again.
8. After the final position is set, the volume of kerosene in the cylinder will be recorded as Vm^3.
9. Calculate particle densities as follows:

Particle density = Weight of sample (g)/Volume of displaced kerosene (m^3)

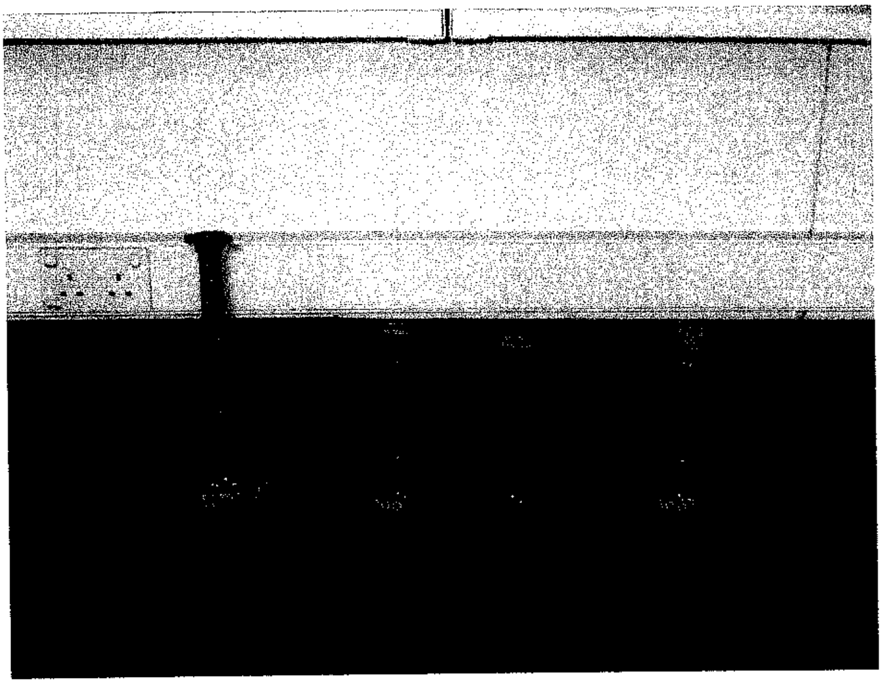


Figure 8: The picture above shows conical flask fill with cement, RHA and kerosene.

3.3 Determination of the fineness

a) Objectives: To determine the fineness of cement and RHA using air permeability method.

b) Equipment:

1) Blaine air permeability apparatus
2) Manometer liquid
3) Timer
4) Balance

c) Procedure (Refer to ASTM C204 – **Appendix 2**):

1. Place the perforated disc on the ledge at the bottom of the cell and place on it with a new filter paper disc. Ensure that the filter paper disc fully covers the perforated disc and is flat by pressing with a clean dry rod.
2. Place the weighted quantity of sample in the cell with care to avoid loss. Tap the cell to level the sample. Place a second new filter paper disc. Press the plunger gently but firmly until the lower face of the cap is in contact with the cell. The bed is now compacted and ready for the permeability test. Slowly withdraw the plunger.
3. Insert the conical surface of the cell into the socket at the top of the manometer, Close the top of the cylinder with a suitable plug. Open the stopcock and with gentle aspiration raise the level of the manometer liquid to that of the highest etched line. Close the stopcock. Remove the plug from the top of the cylinder. The manometer liquid will begin to flow. Start the timer as the liquid reaches the second etched line and stop it when the liquid reaches the third etched line. Record the time, t, to the nearest 0.2s.
4. Expression of the result:

   The specific surface, S, is expressed as:

$$S = \frac{524.2K \times \sqrt{t}}{\rho}$$

where K is the apparatus constant.

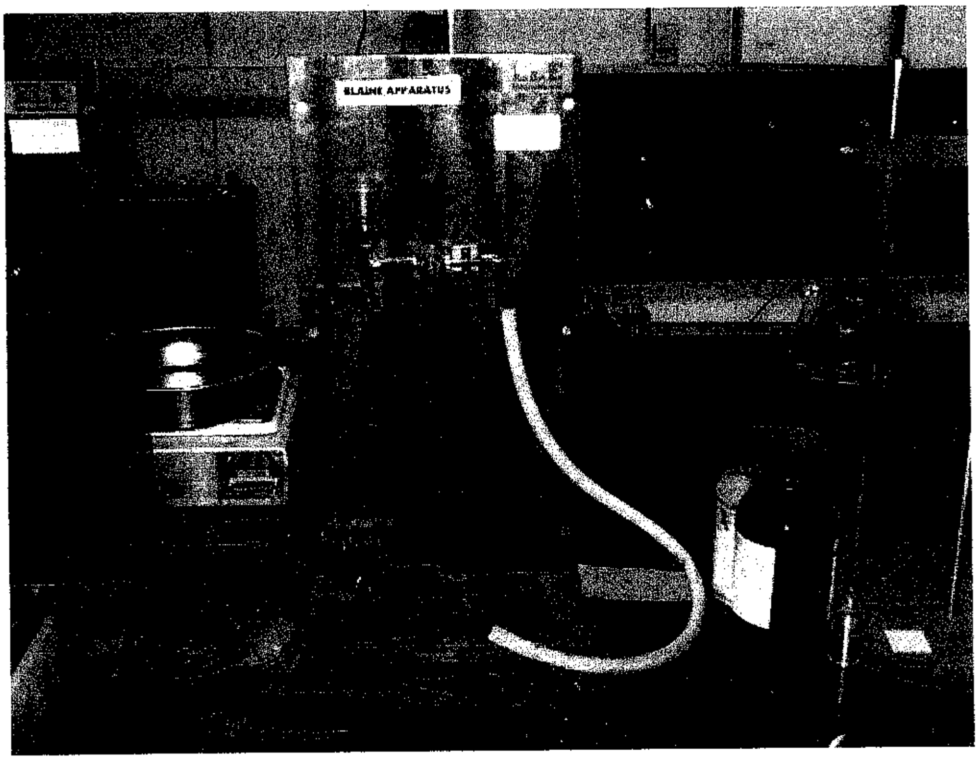


Figure 10: The picture shows the Blaine air apparatus use to determine the finesses of the cement and RHA.

3.4 Grinding of the sample and mixture

a) Objective: To grind the sample to the desired finesses and to grind the mixture to increase the rate of reaction in the mortar and paste.

b) Equipment:

1) Los Angela grinding machine
2) 15 steel balls

c) Procedure:

1. Open the cover of the grinding machine and clean the grinding properly before use.
2. Put the sample or mixture inside the grinding machine and screw up the cover. The weight of each sample (RHA) is 1 kg.
3. Set the number of rotation for sample (the numbers of rotation required for grinding sample is 1500 rotations).
4. On the switch and turn the grinding machine to start grinding.

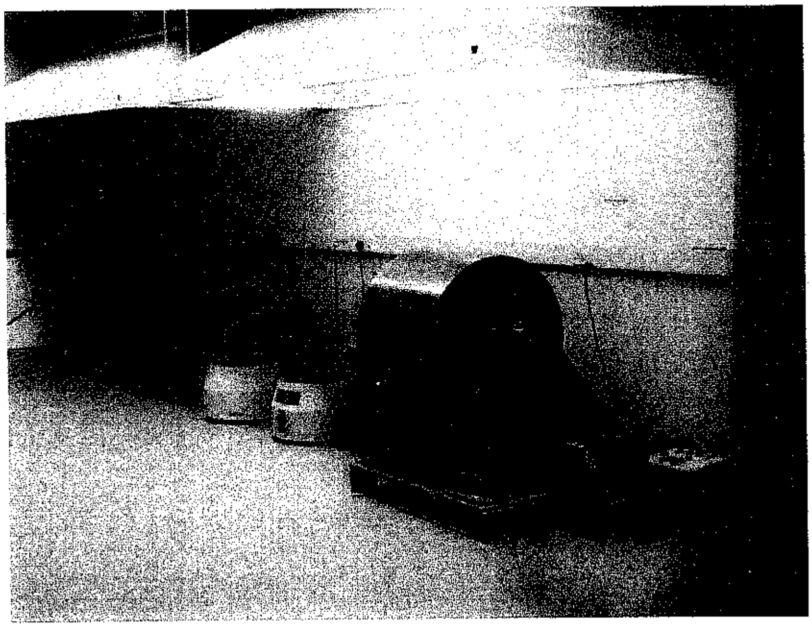


Figure 11: The picture shows the Los Angelas grinding machine used to grind the RHA.

3.5 Determination of water/cement ratio by carrying out Vicat test

a) Objective: To determine the water/cement ratio for each type of mixture

b) Equipment:

1) Vicat apparatus – a movable rod, the plunger end, rigid conical ring
2) Mixture – sample paste
3) Timer

c) Procedure (Refer to ASTM C187 – **Appendix 3**):

1. Mix 750g of mixture with a measured quantity of water following the procedure prescribed in the next section.
2. Quickly form the paste into the approximate shape of a ball with gloved hands.
3. Toss six times through a free path of about 150mm from one hand to another to produce a nearly spherical mass that may be easily inserted into the Vicat ring with a minimum amount of additional manipulation.
4. Press the ball, resting in the palm of one hand into the larger end of the conical ring.
5. Remove the excess at the larger end by a single movement of the palm hand.
6. Place the ring on its larger end on the base plate and slice off the excess paste at the smaller end at the top of the ring by a single oblique stroke of a sharp-edged trowel held at a slight angle with the top of the ring, and smooth the top. If necessary, with a few light touches of the pointed end of the trowel.
7. During these operations of cutting and smoothing, take care not to compress the paste.
8. Center the paste confined in the ring, resting on the plate, under the rod, the plunger end of which shall be brought in contact with the surface of the paste, and tighten the set-screw.
9. Then set the movable indicator to the upper zero mark if the scale, or take an initial reading, and release the rod immediately. This must be not exceeded 30s after completion of mixing.

10. The paste shall be free of all vibrations during the test and shall be of normal consistency when the rod settles to a point (h = 10±1mm) below the original surface in 30s after being released.
11. Make trial pastes with varying percentages of water until the normal consistency is obtained. Make each trial with fresh mixture.

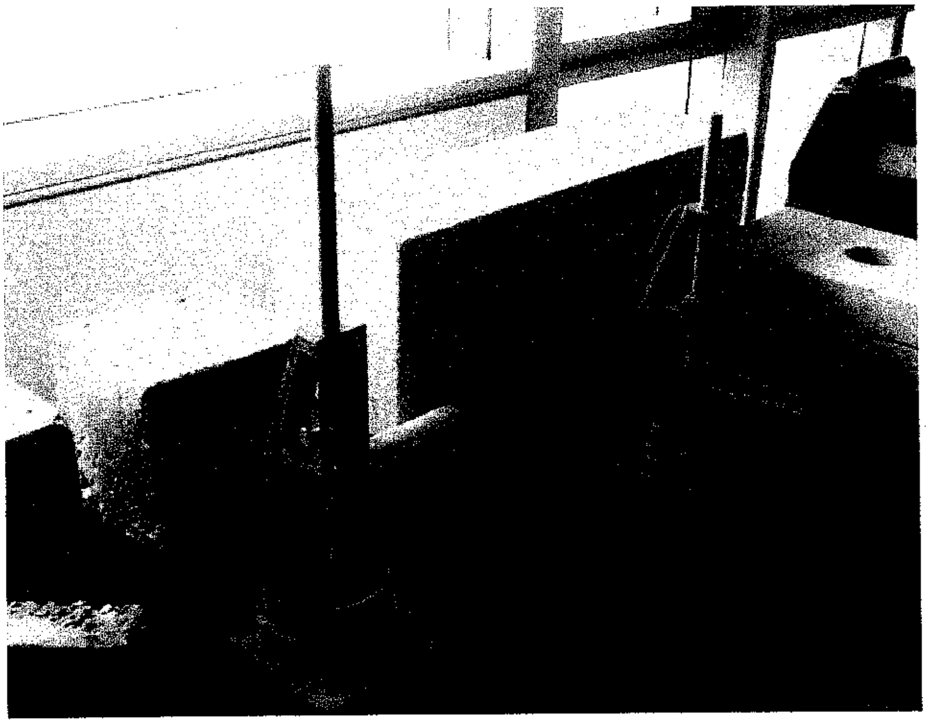


Figure 12: The picture shows the Vicat test apparatus used to determine the water/cement-binder ratio.

3.6 Mechanical Mixing of pastes.

a) Objectives: To prepare the mixture of pastes.

b) Equipment:

1) Mixer
2) Mixing bowl
3) Timer
4) Balance
5) Cylinder
6) A sample tray

c) Procedure (Refer to ASTM C305 – **Appendix 4**):

1. Prepare the required amount of sample and water.
2. Place all the mixing water in the bowl.
3. Add the sample to the water and allow 30s for the absorption of the water.
4. Start the mixer and mix at low speed (140±5r/min) for 30s.
5. Stop the mixer for 15s and during this time scrape down into the batch any paste that may have collected on sides of the bowl.
6. Start the mixer at medium speed (285±10r/min) and mix for one minute.
7. After one minute, the paste is ready to cast and do not wait too long to cast the paste.

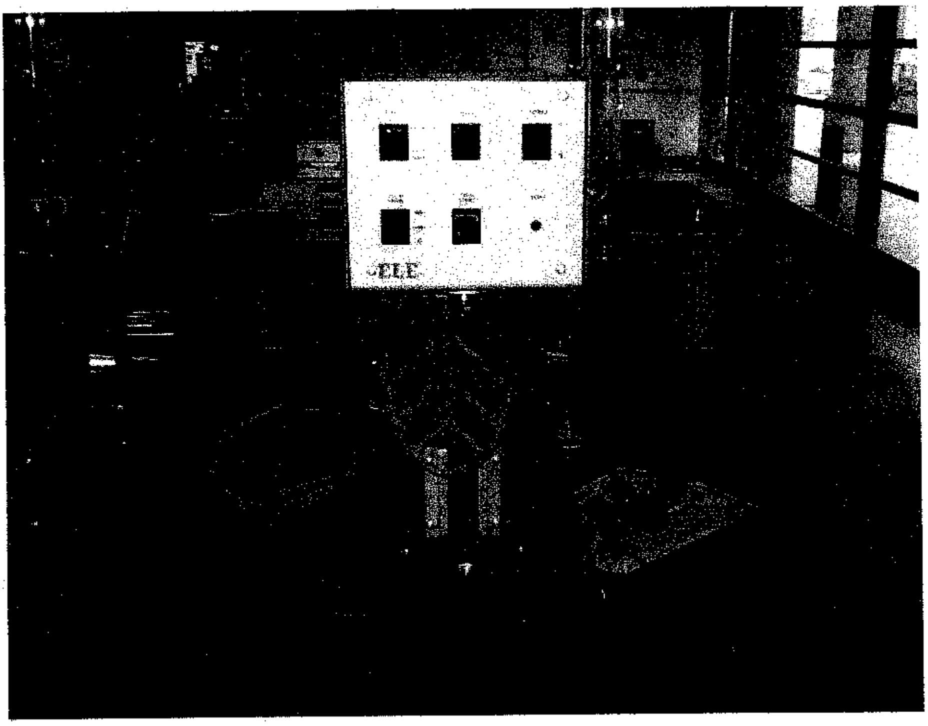


Figure 13: The picture above shows the mixing machine used to mix the paste.

3.7 XRD, XRF and TGA tests.

XRD

Objective – to examine the rice husk ash whether the ash is in the amorphous phase.

Equipment – XRD machine and grinded sample

Procedure – The procedure is very simple, the prepared sample will put on the small plate which is used to insert in the XRD machine. After preparing the sample, insert into the XRD machine and set the parameter (the degree of shooting). The machine can start now and the duration of this test is depending on the degree of shooting.

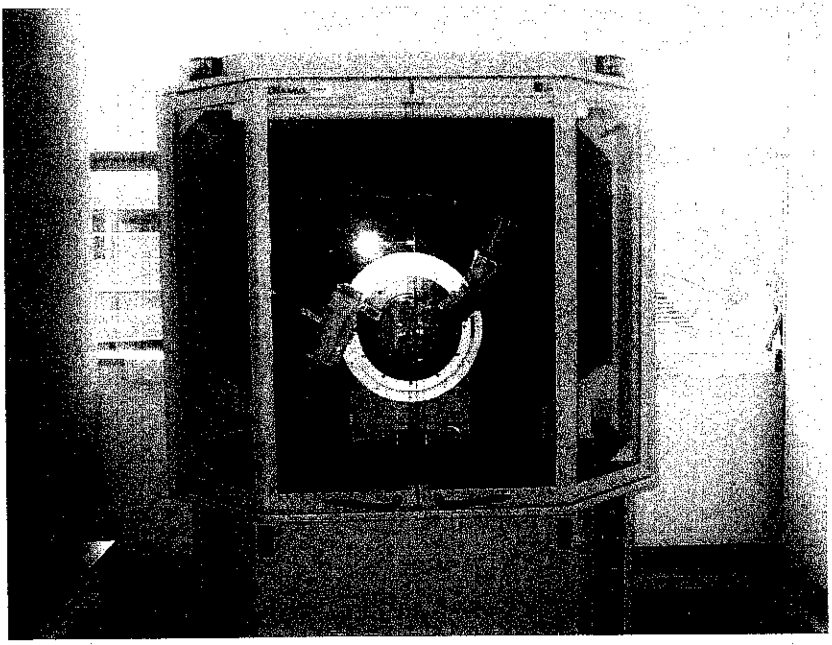


Figure 14: The picture above shows the XRD machine.

XRF

Objective – to examine the silica content in the rice husk ash.

Equipment – XRF machine, compressor, acid boric and grinded sample.

Procedure – Prepare the sample in the form of pill shape by using the compressor. The sample are mixed with acid boric which had no chemical reaction with the sample. Insert the sample into the XRF machine and the examination can begin now.

Figure 15: The picture above shows the XRF machine.

TGA

Objective – to examine the degree of hydration of the paste at the age of 28 days.

Equipment – TGA machine, sample (paste) and grinding machine (Rocklabs).

Procedure – The sample (paste at the age of 28 days) will be used to test the degree of hydration. The sample is grind in the grinding machine name Rocklabs. The duration of grinding is 2 minutes. The machine use nitrogen to burn the sample and the temperature is starting from 50^0C to 900^0C with increasing rate of 40^0C/min. The result with all the data will be instantly shown in the computer.

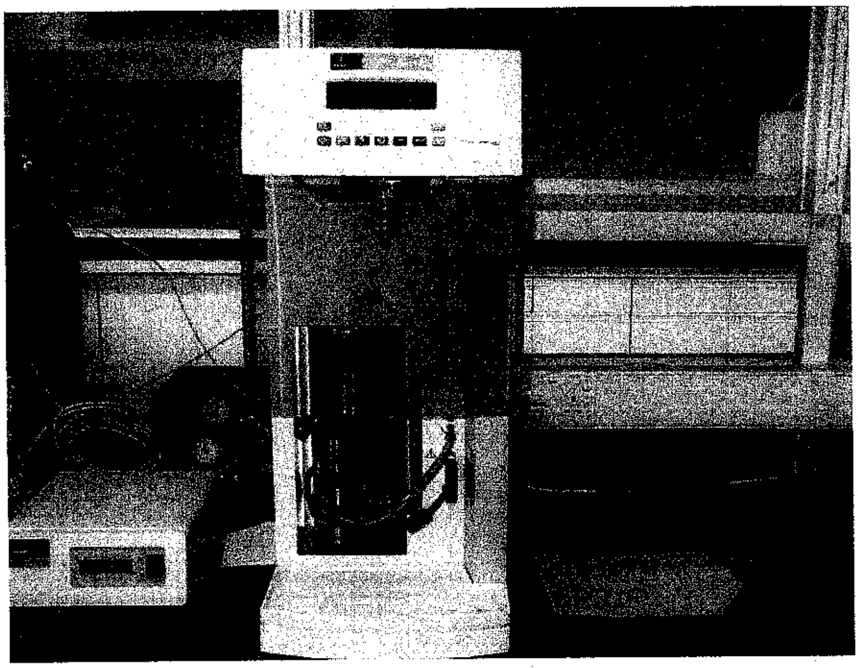


Figure 16: The picture above shows the TGA machine.

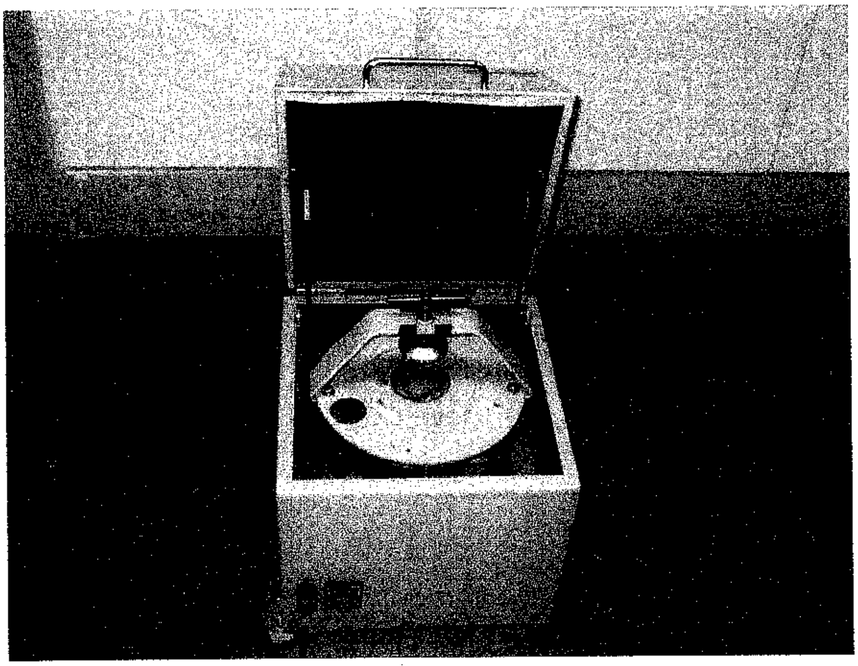


Figure17: The picture above shows the grinding machine 'Rockslabs' use to grind sample for TGA.

3.8 Mixtures proportion

Table 1: The table shows the mixtures proportion for all the samples.

Percentage of RHA	W/C	Cement (gram)	RHA (gram)	Water (ml)
0%	0.2350	750.00	0.00	191.95
	0.2500	750.00	0.00	187.50
	0.2625	750.00	0.00	196.88
	0.2800	750.00	0.00	210.00
	0.3200	750.00	0.00	240.00
	0.3500	750.00	0.00	262.50
2.50%	0.2350	731.25	18.75	171.84
	0.2500	731.25	18.75	182.81
	0.2800	731.25	18.75	204.75
	0.3000	731.25	18.75	219.38
	0.3250	731.25	18.75	237.66
5.00%	0.2350	712.50	37.50	167.44
	0.2500	712.50	37.50	178.13
	0.2800	712.50	37.50	199.50
	0.2950	712.50	37.50	210.19
	0.3200	712.50	37.50	228.00
7.50%	0.2350	693.75	56.25	163.03
	0.2800	693.75	56.25	194.25
	0.3300	693.75	56.25	228.94
	0.3100	693.75	56.25	215.06
	0.3500	693.75	56.25	242.81
10.00%	0.3250	675.00	75.00	219.38
12.50%	0.3500	656.25	93.75	229.69
15.00%	0.3600	637.50	112.50	229.50
17.50%	0.3719	618.75	131.25	230.11
20.00%	0.3930	600.00	150.00	235.80

CHAPTER 4

RESULTS AND DISCUSSIONS

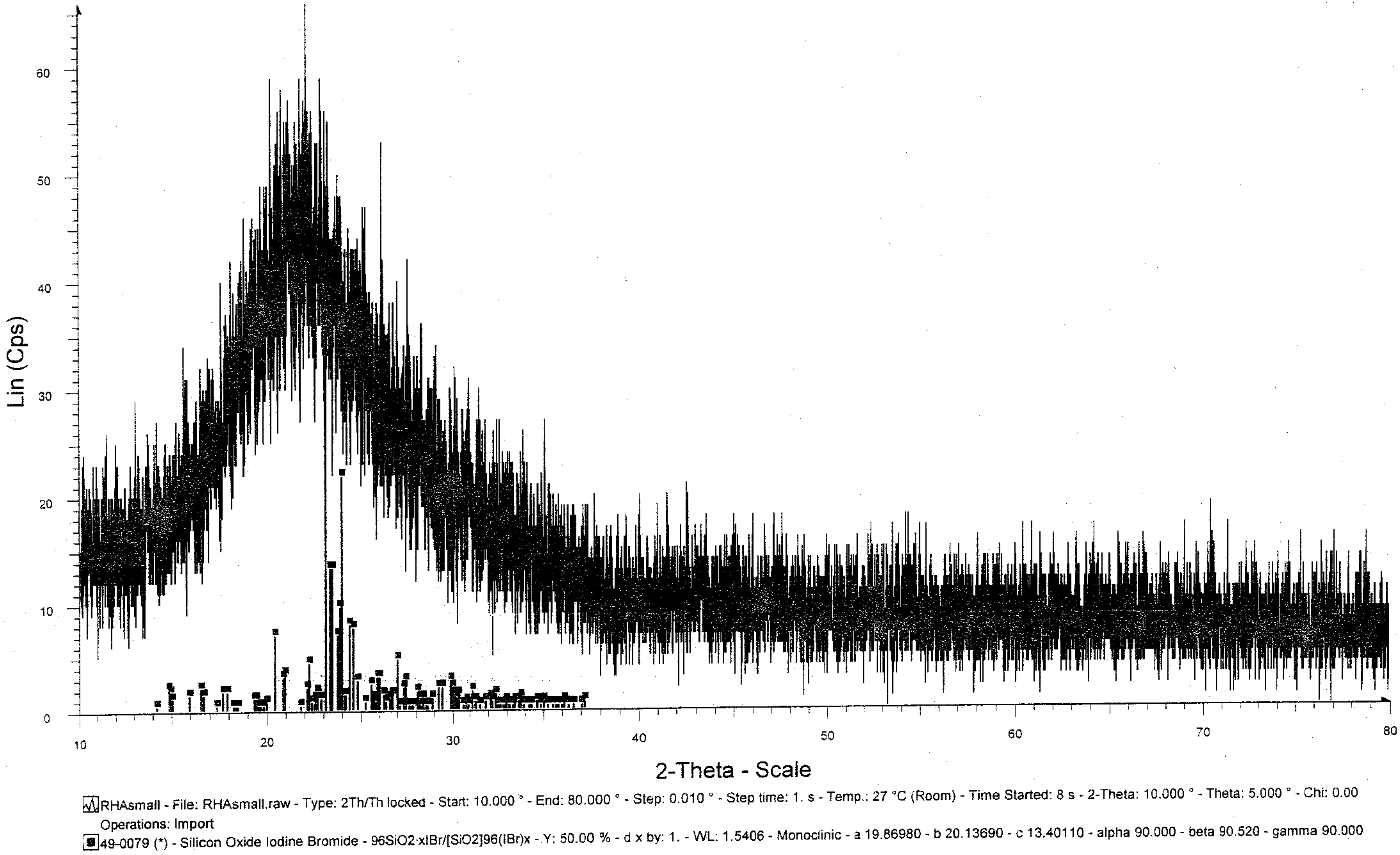
4.1 XRD and XRF result

The tables above are the burning trials make for rice husk. There are two trials among the numerous trials had been make by the author and the rice husk ashes for these two trial are used as the samples for XRD test and XRF test. The result of the test is shown in **Figure 18** – XRD test on the rice husk ash (first trial), **Table 2** – XRF test on the rice husk ash (first trial) and **Table 3** – XRF test on the rice husk ash (second trial).

4.1.1 Discussion - XRD test on the rice husk ash (first trial)

Refer to the figure 18, the result shows that there is quartz inside the samples and most of the ashes are in amorphous phase. The existing of quartz in the sample may due to the over burning of the ashes especially for the surface of samples. The surface of the samples will be heated first before the center samples are heated. Thus, in order to burn the middle samples, the temperature for the burning must be set higher than the favorable temperature in order to achieve homogenous burning in the samples. When the temperature is getting higher, the surface samples will be over burnt and the ash will turn into quartz at the end of the burning process.

re 18 - XRD test result on the rice husk ash (first trial)



4.1.2 Discussion – XRF test on the rice husk ash (first trial and second trial)

Refer to the table 2 and table 3, the result shows that the compound material – Silicon oxide is the highest composition among the oxide compounds. This can prove that by using this burning process, the composition percentage of Silicon oxide in the ash is achievable.

Table 2: The table shows the XRF result for the first trial burning of rice husk.

MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	MnO	Rb_2O	Y_2O_3	Compton	Rayleigh	Norm
11.6KCps	0.9 KCps	**1279.0 KCps**	12.1 KCps	7.2 KCps	2.0 KCps	108.2KCps	17.4 KCps	8.9 KCps	23 KCps	5.7 KCps			
0.657%	0.0481%	**94.3%**	1.22%	0.437%	0.108%	2.63%	0.481%	0.0518%	0.0102%	0.000405%	1.57	1.79	100%

Table 3: The table shows the XRF result for the second trial burning of rice husk.

MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	ZnO	Rb_2O	Y_2O_3	Compton	Rayleigh	Norm
12.1KCps	0.8 KCps	**1265.2 KCps**	13.2 KCps	19.7 KCps	3.4 KCps	113.5KCps	17.7 KCps	1.3 KCps	9.6 KCps	57.4KCps	22.9 KCps	6.5 KCps			
0.682%	0.0403%	**93.1%**	1.32%	1.19%	0.185%	2.78%	0.495%	0.0290%	0.0574%	0.102%	0.0104%	0.000803%	1.49	1.70	100%

4.2 Particle density for cement

First trial for cement – Using 500ml flask

Weight of cement – 500g

Displace volume of kerosene – 175ml

Particle density = Weight of cement/Displace volume of kerosene

= 500/175

= ***2.86g/cm^3***

Second trial for cement – Using 500ml flask

Weight of cement – 500g

Displace volume of kerosene – 172ml

Particle density = Weight of cement/Displace volume of kerosene

= 500/172

= ***2.91g/cm^3***

Third trial for cement – Using 100ml flask

Weight of cement – 100g

Displace volume of kerosene – 33ml

Particle density = Weight of cement/Displace volume of kerosene

= 100/33

= ***3.03g/cm^3***

**Standard particle density for cement is 3.15g/cm^3*

Average particle density of cement = 2.93g/cm^3

4.3 Particle density for RHA

First trial for RHA – Using 100ml flask

Weight of RHA – 66g

Displace volume of kerosene – 29ml

Particle density = Weight of cement/Displace volume of kerosene

= 66/29

= ***2.28g/cm³***

Second trial for RHA – Using 100ml flask

Weight of RHA – 70g

Displace volume of kerosene – 28ml

Particle density = Weight of cement/Displace volume of kerosene

= 70/28

= ***2.5g/cm³***

Third trial for RHA – Using 100ml flask

Weight of RHA – 63g

Displace volume of kerosene – 28ml

Particle density = Weight of cement/Displace volume of kerosene

= 63/28

= ***2.25g/cm³***

Average particle density of RHA = 2.34g/cm³

4.4 Determination of finesses

The results obtain from the experiment – Blaine Air permeability Test is showing as below:

Time (t) for cement

1) 18s
2) 19s
3) 20s

Average = 19s

Time (t) for RHA

1) 38.3
2) 36.2
3) 36.7

Average = 37.1s

4.5 Calculation for specific surface,

Specific surface (S) $= \frac{524.2 \times K \times \sqrt{t}}{P}$ **K = 1**

Specific surface for cement,

$$S = \frac{524.2 \times 1 \times \sqrt{19}}{2.93}$$

$$= \mathbf{779.84\ cm^2/g}$$

Specific surface for RHA,

$$S = \frac{524.2 \times 1 \times \sqrt{37.1}}{2.34}$$

$$= \mathbf{1364.5\ cm^2/g}$$

4.6 Vicat test for cement-binder paste – Water/cement-binder ratio

In this project, the author intend to maintain the water/cement ratio for all the mixture but due to the higher water requirement for paste with RHA, the water/cement-binder ratio is determined base on the percentage of RHA incorporate in the paste. Thus, base on the result of this vicat test, a few more W/C is selected for each particular percentage of RHA so that a curve can be plotted and the optimum W/C that gives the optimum compressive strength can be obtained.

Table 4 shows the water/cement ratio for control mix and this water/cement ratio is used for all the mixtures. Since the water/cement ratio can not be maintained for all the mixture due to the higher demand water for RHA, thus optimum water cement ratio have to be chosen for all the mixture to give optimum compressive strength. Vicat test had been done for all the mixtures to know approximately the minimum water requirement. Table 5 shows the water/cement-binder ratio for all the mixtures.

Table 4: Water/cement-binder ratio used previously for all the mixtures:

Percentage of RHA	First trial (Water/cement ratio)	Second trial (Water/cement ratio)	Third trial (Water/cement ratio)
0.0%	***0.235 (h = 10mm)***	-	-

Table 5: Water/cement-binder ratio determined for all the mixtures:

Percentage of RHA	First trial (Water/cement ratio)	Second trial (Water/cement ratio)	Third trial (Water/cement ratio)
0.0%	0.2500 (h = 5mm)	0.2750 (h = 20mm)	***0.2625 (h = 10mm)***
2.5%	***0.2800 (h = 9mm)***	-	-
5.0%	***0.2950 (h = 9mm)***	-	-
7.5%	***0.3100 (h = 9.5mm)***	-	-
10.0%	0.4000 (h = 30mm)	0.3500 (h = 25mm)	***0.3250 (h = 10mm)***
12.5%	***0.3500 (h = 11mm)***	-	-
15.0%	0.3563 (h = 5mm)	0.3656 (h = 20mm)	***0.3600 (h = 10mm)***
17.5%	***0.3719 (h = 10mm)***	-	-
20.0%	0.3875 (h = 5mm)	0.3900 (h = 7mm)	***0.3930 (h = 10mm)***

4.7 Compressive strength results for pastes

Table 6: The table summarized the results of compressive strength.

Percentage of RHA	W/C	Compressive strength (N/mm^2)			
		Age – 1 day	Age – 3 days	Age – 7 days	Age – 28 days
0.0%	0.2350	51.48	58.51	79.65	99.25
	0.2500	-	60.64	88.04	81.04
	0.2625	-	57.93	58.79	76.71
	0.2800	-	69.14	86.58	70.66
	0.3200	-	52.94	52.54	57.88
	0.3500	-	52.00	66.38	59.14
2.5%	0.2350	41.40	46.57	61.35	78.43
	0.2500	-	58.88	56.14	80.26
	0.2800	-	36.03	43.07	64.26
	0.3000	-	45.12	50.42	73.28
	0.3250	-	47.28	52.70	57.62
5.0%	0.2350	47.89	41.24	79.65	66.85
	0.2500	-	14.36	54.34	98.70
	0.2800	-	18.36	58.66	105.64
	0.2950	-	38.28	47.48	63.86
	0.3200	-	42.80	54.98	101.62
7.5%	0.2350	5.19	7.75	9.43	9.85
	0.2800	-	28.62	70.88	85.26
	0.3100	-	37.02	51.84	69.40
	0.3300	-	45.88	40.31	87.20
	0.3500	-	38.60	64.22	67.14
10.0%	0.3250	-	27.29	39.21	60.76
12.5%	0.3500	-	24.45	35.44	40.45
15.0%	0.3600	-	20.80	33.03	35.44
17.5%	0.3719	-	22.41	30.51	41.21
20.0%	0.3930	-	17.39	20.84	32.88

7: The table shows the detail of compressive strength result.

CONTROL MIX (W/C = 0.235)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	15/02/2005	16/02/2005	1	147.2	58.88	51.48
RC-2	15/02/2005	16/02/2005	1	104.9	41.96	
RC-3	15/02/2005	16/02/2005	1	134	53.60	
RC-4	15/02/2005	19/02/2005	3	140.6	56.24	58.51
RC-5	15/02/2005	19/02/2005	3	144.3	57.72	
RC-6	15/02/2005	19/02/2005	3	153.9	61.56	
RC-7	15/02/2005	23/02/2005	7	186.7	74.68	79.65
RC-8	15/02/2005	23/02/2005	7	205.4	82.16	
RC-9	15/02/2005	23/02/2005	7	205.3	82.12	
RC-10	15/02/2005	15/03/2005	28	260.1	104.04	99.25
RC-11	15/02/2005	15/03/2005	28	246.1	98.44	
RC-12	15/02/2005	15/03/2005	28	238.2	95.28	

CONTROL MIX (W/C = 0.25)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	124.40	49.76	60.64
RC-4	22/04/2005	25/04/2005	3	178.80	71.52	
RC-5	22/04/2005	29/03/2005	7	232.90	93.16	88.04
RC-6	22/04/2005	29/03/2005	7	207.30	82.92	
RC-7	22/04/2005	20/05/2005	28	213.00	85.20	81.04
RC-8	22/04/2005	20/05/2005	28	192.20	76.88	

CONTROL MIX (W/C = 0.2625)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	18/03/2005	19/03/2005	1	-	-	-
RC-2	18/03/2005	19/03/2005	1	-	-	
RC-3	18/03/2005	19/03/2005	1	-	-	
RC-4	18/03/2005	21/03/2005	3	145.20	58.08	57.93
RC-5	18/03/2005	21/03/2005	3	144.00	57.60	
RC-6	18/03/2005	21/03/2005	3	145.30	58.12	
RC-7	18/03/2005	25/03/2005	7	123.10	49.24	58.79
RC-8	18/03/2005	25/03/2005	7	143.80	57.52	
RC-9	18/03/2005	25/03/2005	7	174.00	69.60	
RC-10	18/03/2005	14/04/2005	28	122.00	48.80	76.71
RC-11	18/03/2005	14/04/2005	28	221.00	88.40	
RC-12	18/03/2005	14/04/2005	28	232.30	92.92	

CONTROL MIX (W/C = 0.28)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	189.40	75.76	69.14
RC-4	22/04/2005	25/04/2005	3	156.30	62.52	
RC-5	22/04/2005	29/03/2005	7	207.3	82.92	86.58
RC-6	22/04/2005	29/03/2005	7	225.6	90.24	
RC-7	22/04/2005	20/05/2005	28	209.50	83.80	70.66
RC-8	22/04/2005	20/05/2005	28	143.80	57.52	

CONTROL MIX (W/C = 0.32)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	134.30	53.72	52.94
RC-4	22/04/2005	25/04/2005	3	130.40	52.16	
RC-5	22/04/2005	29/03/2005	7	110.4	44.16	52.54
RC-6	22/04/2005	29/03/2005	7	152.3	60.92	
RC-7	22/04/2005	20/05/2005	28	167.90	67.16	57.88
RC-8	22/04/2005	20/05/2005	28	121.50	48.60	

CONTROL MIX (W/C = 0.35)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	134.00	53.60	52
RC-4	22/04/2005	25/04/2005	3	126.00	50.40	
RC-5	22/04/2005	29/03/2005	7	162.6	65.04	66.38
RC-6	22/04/2005	29/03/2005	7	169.3	67.72	
RC-7	22/04/2005	20/05/2005	28	151.00	60.40	59.14
RC-8	22/04/2005	20/05/2005	28	144.70	57.88	

RHA - 2.5% (W/C = 0.235)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	16/02/2005	18/02/2005	1	114	45.60	41.4
RC-2	16/02/2005	18/02/2005	1	95.1	38.04	
RC-3	16/02/2005	18/02/2005	1	101.4	40.56	
RC-4	16/02/2005	19/02/2005	3	114.4	45.76	46.57
RC-5	16/02/2005	19/02/2005	3	115.4	46.16	
RC-6	16/02/2005	19/02/2005	3	119.5	47.80	
RC-7	16/02/2005	23/02/2005	7	140.6	56.24	61.35
RC-8	16/02/2005	23/02/2005	7	182	72.80	
RC-9	16/02/2005	23/02/2005	7	137.5	55.00	
RC-10	16/02/2005	15/03/2005	28	176.1	70.44	78.43
RC-11	16/02/2005	15/03/2005	28	188	75.20	
RC-12	16/02/2005	15/03/2005	28	224.1	89.64	

RHA - 2.5% (W/C = 0.25)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	143.40	57.36	58.88
RC-4	22/04/2005	25/04/2005	3	151.00	60.40	
RC-5	22/04/2005	29/03/2005	7	136.6	54.64	56.14
RC-6	22/04/2005	29/03/2005	7	144.1	57.64	
RC-7	22/04/2005	20/05/2005	28	202.70	81.08	80.26
RC-8	22/04/2005	20/05/2005	28	198.60	79.44	

RHA - 2.5% (W/C = 0.2800)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	6/04/2005	7/04/2005	1	-	-	-
RC-2	6/04/2005	7/04/2005	1	-	-	
RC-3	6/04/2005	7/04/2005	1	-	-	
RC-4	6/04/2005	8/04/2005	3	94.70	37.88	36.03
RC-5	6/04/2005	8/04/2005	3	107.10	42.84	
RC-6	6/04/2005	8/04/2005	3	68.40	27.36	
RC-7	6/04/2005	12/04/2005	7	110.30	44.12	43.07
RC-8	6/04/2005	12/04/2005	7	138.20	55.28	
RC-9	6/04/2005	12/04/2005	7	74.50	29.80	
RC-10	6/04/2005	2/05/2005	28	213.90	85.56	64.26
RC-11	6/04/2005	2/05/2005	28	107.40	42.96	
RC-12	6/04/2005	2/05/2005	28	-	-	

RHA - 2.5% (W/C = 0.30)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	119.40	47.76	45.12
RC-4	22/04/2005	25/04/2005	3	106.20	42.48	
RC-5	22/04/2005	29/03/2005	7	120.8	48.32	50.42
RC-6	22/04/2005	29/03/2005	7	131.3	52.52	
RC-7	22/04/2005	20/05/2005	28	180.60	72.24	73.28
RC-8	22/04/2005	20/05/2005	28	185.80	74.32	

RHA - 2.5% (W/C = 0.325)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	102.70	41.08	47.28
RC-4	22/04/2005	25/04/2005	3	133.70	53.48	
RC-5	22/04/2005	29/03/2005	7	140.3	56.12	52.7
RC-6	22/04/2005	29/03/2005	7	123.2	49.28	
RC-7	22/04/2005	20/05/2005	28	174.30	69.72	57.62
RC-8	22/04/2005	20/05/2005	28	113.80	45.52	

RHA - 5.0% (W/C = 0.235)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	16/02/2005	18/02/2005	1	123.3	49.32	47.89
RC-2	16/02/2005	18/02/2005	1	119.2	47.68	
RC-3	16/02/2005	18/02/2005	1	116.7	46.68	
RC-4	16/02/2005	19/02/2005	3	124.3	49.72	41.24
RC-5	16/02/2005	19/02/2005	3	85.9	34.36	
RC-6	16/02/2005	19/02/2005	3	99.1	39.64	
RC-7	16/02/2005	23/02/2005	7	186.7	74.68	79.65
RC-8	16/02/2005	23/02/2005	7	205.4	82.16	
RC-9	16/02/2005	23/02/2005	7	205.3	82.12	
RC-10	16/02/2005	15/03/2005	28	167.4	66.96	66.85
RC-11	16/02/2005	15/03/2005	28	158.1	63.24	
RC-12	16/02/2005	15/03/2005	28	175.9	70.36	

RHA - 5.0% (W/C = 0.25)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	42.10	16.84	14.36
RC-4	22/04/2005	25/04/2005	3	29.70	11.88	
RC-5	22/04/2005	29/03/2005	7	118.00	47.20	54.34
RC-6	22/04/2005	29/03/2005	7	153.70	61.48	
RC-7	22/04/2005	20/05/2005	28	224.00	89.60	98.7
RC-8	22/04/2005	20/05/2005	28	269.50	107.80	

RHA - 5.0% (W/C = 0.28)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	56.60	22.64	18.36
RC-4	22/04/2005	25/04/2005	3	35.20	14.08	
RC-5	22/04/2005	29/03/2005	7	151.00	60.40	58.66
RC-6	22/04/2005	29/03/2005	7	142.30	56.92	
RC-7	22/04/2005	20/05/2005	28	256.70	102.68	105.64
RC-8	22/04/2005	20/05/2005	28	271.50	108.60	

RHA - 5.0% (W/C = 0.2950)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	6/04/2005	7/04/2005	1	-	-	-
RC-2	6/04/2005	7/04/2005	1	-	-	
RC-3	6/04/2005	7/04/2005	1	-	-	
RC-4	6/04/2005	8/04/2005	3	94.40	37.76	38.28
RC-5	6/04/2005	8/04/2005	3	93.40	37.36	
RC-6	6/04/2005	8/04/2005	3	99.30	39.72	
RC-7	6/04/2005	12/04/2005	7	96.50	38.60	47.48
RC-8	6/04/2005	12/04/2005	7	141.70	56.68	
RC-9	6/04/2005	12/04/2005	7	117.90	47.16	
RC-10	6/04/2005	2/05/2005	28	166.60	66.64	63.86
RC-11	6/04/2005	2/05/2005	28	152.70	61.08	
RC-12	6/04/2005	2/05/2005	28	-	-	

RHA - 5.0% (W/C = 0.32)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	101.80	40.72	42.8
RC-4	22/04/2005	25/04/2005	3	112.20	44.88	
RC-5	22/04/2005	29/03/2005	7	129.60	51.84	54.98
RC-6	22/04/2005	29/03/2005	7	145.30	58.12	
RC-7	22/04/2005	20/05/2005	28	274.20	109.68	101.62
RC-8	22/04/2005	20/05/2005	28	233.90	93.56	

RHA - 7.5% (W/C = 0.235)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	16/02/2005	18/02/2005	1	11.3	4.52	5.19
RC-2	16/02/2005	18/02/2005	1	14.4	5.76	
RC-3	16/02/2005	18/02/2005	1	13.2	5.28	
RC-4	16/02/2005	19/02/2005	3	16.6	6.64	7.75
RC-5	16/02/2005	19/02/2005	3	24.7	9.88	
RC-6	16/02/2005	19/02/2005	3	16.8	6.72	
RC-7	16/02/2005	23/02/2005	7	23.1	9.24	9.43
RC-8	16/02/2005	23/02/2005	7	23.3	9.32	
RC-9	16/02/2005	23/02/2005	7	24.3	9.72	
RC-10	16/02/2005	15/03/2005	28	26.4	10.56	9.85
RC-11	16/02/2005	15/03/2005	28	26.7	10.68	
RC-12	16/02/2005	15/03/2005	28	20.8	8.32	

RHA - 7.5% (W/C = 0.28)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	67.20	26.88	28.62
RC-4	22/04/2005	25/04/2005	3	75.90	30.36	
RC-5	22/04/2005	29/03/2005	7	185.20	74.08	70.88
RC-6	22/04/2005	29/03/2005	7	169.20	67.68	
RC-7	22/04/2005	20/05/2005	28	211.60	84.64	85.26
RC-8	22/04/2005	20/05/2005	28	214.70	85.88	

RHA - 7.5% (W/C = 0.3100)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	6/04/2005	7/04/2005	1	-	-	-
RC-2	6/04/2005	7/04/2005	1	-	-	
RC-3	6/04/2005	7/04/2005	1	-	-	
RC-4	6/04/2005	8/04/2005	3	109.30	43.72	45.88
RC-5	6/04/2005	8/04/2005	3	115.40	46.16	
RC-6	6/04/2005	8/04/2005	3	119.40	47.76	
RC-7	6/04/2005	12/04/2005	7	88.70	35.48	40.31
RC-8	6/04/2005	12/04/2005	7	121.10	48.44	
RC-9	6/04/2005	12/04/2005	7	92.50	37.00	
RC-10	6/04/2005	2/05/2005	28	144.30	57.72	69.40
RC-11	6/04/2005	2/05/2005	28	202.70	81.08	
RC-12	6/04/2005	2/05/2005	28	-	-	

RHA - 7.5% (W/C = 0.33)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	76.50	30.60	37.02
RC-4	22/04/2005	25/04/2005	3	108.60	43.44	
RC-5	22/04/2005	29/03/2005	7	136.50	54.60	51.84
RC-6	22/04/2005	29/03/2005	7	122.70	49.08	
RC-7	22/04/2005	20/05/2005	28	215.40	86.16	87.2
RC-8	22/04/2005	20/05/2005	28	220.60	88.24	

RHA - 7.5% (W/C = 0.35)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average Stress
RC-1	22/04/2005	22/04/2005	1	-	-	-
RC-2	22/04/2005	22/04/2005	1	-	-	
RC-3	22/04/2005	25/04/2005	3	99.40	39.76	38.6
RC-4	22/04/2005	25/04/2005	3	93.60	37.44	
RC-5	22/04/2005	29/03/2005	7	155.2	62.08	64.22
RC-6	22/04/2005	29/03/2005	7	165.9	66.36	
RC-7	22/04/2005	20/05/2005	28	181.70	72.68	67.14
RC-8	22/04/2005	20/05/2005	28	154.00	61.60	

RHA - 10.0% (W/C = 0.3250)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	6/04/2005	7/04/2005	1	-	-	-
RC-2	6/04/2005	7/04/2005	1	-	-	
RC-3	6/04/2005	7/04/2005	1	-	-	
RC-4	6/04/2005	8/04/2005	3	78.40	31.36	27.29
RC-5	6/04/2005	8/04/2005	3	31.80	12.72	
RC-6	6/04/2005	8/04/2005	3	94.50	37.80	
RC-7	6/04/2005	12/04/2005	7	102.70	41.08	39.21
RC-8	6/04/2005	12/04/2005	7	106.80	42.72	
RC-9	6/04/2005	12/04/2005	7	84.60	33.84	
RC-10	6/04/2005	2/05/2005	28	134.90	53.96	60.76
RC-11	6/04/2005	2/05/2005	28	168.90	67.56	
RC-12	6/04/2005	2/05/2005	28	-	-	

RHA - 12.5% (W/C = 0.3500)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	18/03/2005	19/03/2005	1	-	-	-
RC-2	18/03/2005	19/03/2005	1	-	-	
RC-3	18/03/2005	19/03/2005	1	-	-	
RC-4	18/03/2005	21/03/2005	3	59.10	23.64	24.45
RC-5	18/03/2005	21/03/2005	3	62.60	25.04	
RC-6	18/03/2005	21/03/2005	3	61.70	24.68	
RC-7	18/03/2005	25/03/2005	7	96.60	38.64	35.44
RC-8	18/03/2005	25/03/2005	7	82.70	33.08	
RC-9	18/03/2005	25/03/2005	7	86.50	34.60	
RC-10	18/03/2005	14/04/2005	28	89.40	35.76	40.45
RC-11	18/03/2005	14/04/2005	28	113.00	45.20	
RC-12	18/03/2005	14/04/2005	28	101.00	40.40	

RHA - 15.0% (W/C = 0.3600)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	21/03/2005	22/03/2005	1	-	-	-
RC-2	21/03/2005	22/03/2005	1	-	-	
RC-3	21/03/2005	22/03/2005	1	-	-	
RC-4	21/03/2005	24/03/2005	3	61.00	24.40	20.80
RC-5	21/03/2005	24/03/2005	3	48.90	19.56	
RC-6	21/03/2005	24/03/2005	3	46.10	18.44	
RC-7	21/03/2005	28/03/2005	7	84.70	33.88	33.03
RC-8	21/03/2005	28/03/2005	7	84.40	33.76	
RC-9	21/03/2005	28/03/2005	7	78.60	31.44	
RC-10	21/03/2005	17/04/2005	28	102.30	40.92	35.44
RC-11	21/03/2005	17/04/2005	28	81.70	32.68	
RC-12	21/03/2005	17/04/2005	28	81.80	32.72	

RHA - 17.5% (W/C = 0.3719)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	21/03/2005	22/03/2005	1	-	-	-
RC-2	21/03/2005	22/03/2005	1	-	-	
RC-3	21/03/2005	22/03/2005	1	-	-	
RC-4	21/03/2005	24/03/2005	3	57.10	22.84	22.41
RC-5	21/03/2005	24/03/2005	3	49.80	19.92	
RC-6	21/03/2005	24/03/2005	3	61.20	24.48	
RC-7	21/03/2005	28/03/2005	7	74.80	29.92	30.51
RC-8	21/03/2005	28/03/2005	7	82.10	32.84	
RC-9	21/03/2005	28/03/2005	7	71.90	28.76	
RC-10	21/03/2005	17/04/2005	28	97.20	38.88	41.21
RC-11	21/03/2005	17/04/2005	28	113.80	45.52	
RC-12	21/03/2005	17/04/2005	28	98.10	39.24	

RHA - 20.0% (W/C = 0.3930)

No	Date of cast	Date of test	Age	Load(kN)	Stress (N/mm2)	Average stress
RC-1	21/03/2005	22/03/2005	1	-	-	-
RC-2	21/03/2005	22/03/2005	1	-	-	
RC-3	21/03/2005	22/03/2005	1	-	-	
RC-4	21/03/2005	24/03/2005	3	45.50	18.20	17.39
RC-5	21/03/2005	24/03/2005	3	42.20	16.88	
RC-6	21/03/2005	24/03/2005	3	42.70	17.08	
RC-7	21/03/2005	28/03/2005	7	47.40	18.96	20.84
RC-8	21/03/2005	28/03/2005	7	58.50	23.40	
RC-9	21/03/2005	28/03/2005	7	50.40	20.16	
RC-10	21/03/2005	17/04/2005	28	81.60	32.64	32.88
RC-11	21/03/2005	17/04/2005	28	75.60	30.24	
RC-12	21/03/2005	17/04/2005	28	89.40	35.76	

At the beginning of this project, the author proposes that the w/c is maintained for all the mixtures so that comparison within each mixture can be done. However, the mixture with RHA demand higher water requirement than mixture with cement only. During the mixing period, the paste with RHA is very dry especially for paste with 7.5% RHA. Mixture is too dry to mix and cast in the cube. Thus, the casting is instantly stopped to avoid the wastage of RHA.

After obtaining the compressive strength result, the compressive strength for paste with 7.5% RHA is extremely low compare to the others paste result. Thus, the author decides to carrying out the vicat test for all the mixtures so that the water/cement-binder ratio for all the mixture can be obtained. After that, a few more w/c ratio are chosen for each mixture so that a graph of compressive strength versus w/c can be plotted and from the graph, a constant value of w/c can be determined. In this project, the casting is done up to 7.5% RHA due to lack of time and material (RHA). However, the result obtain is good enough for author to carrying out the analysis.

4.8 Effect of Water/cement-binder ratio to Compressive strength

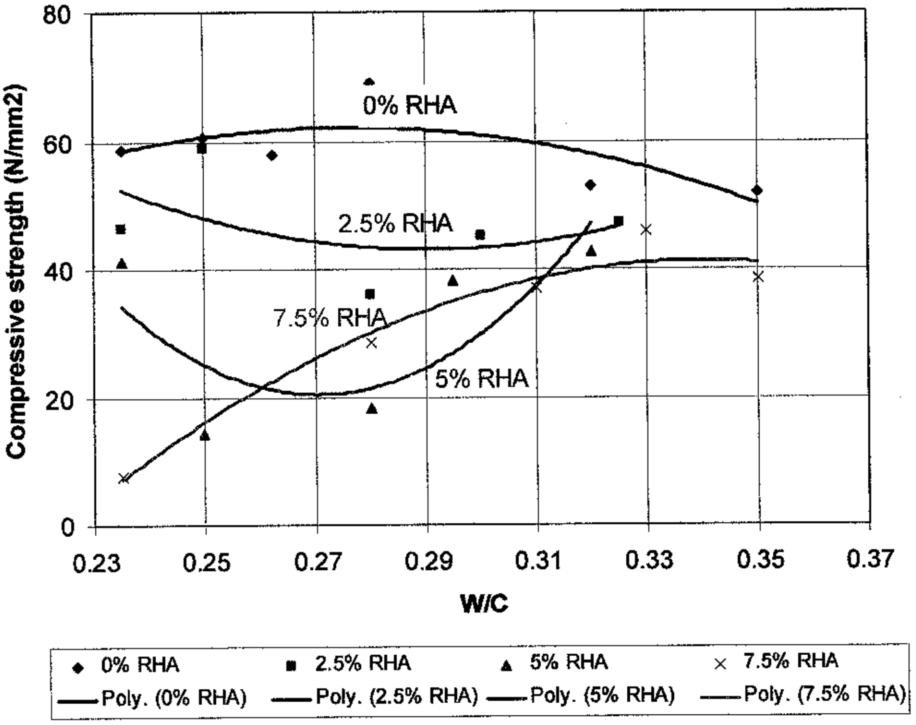


Figure 19: The graph shows the correlation between compressive strength and W/C at the age of 3 days.

From the graph above, it shows that the pastes with incorporation of RHA achieve lower compressive strength than the control paste. For paste with 5% RHA, the second sample and third sample is found defect after opening the cube mould. There are many voids on the samples and even cracks are found on the samples. This is due to the inadequate vibration during the casting period. Thus, these values can be ignored.

Paste with 2.5% and 5% RHA achieve almost the same compressive strength for each w/c but for paste with 7.5% RHA, the compressive strength is increasing as the w/c increasing. The preliminary conclusion can be made is the control paste can achieve higher early compressive strength than the pastes with RHA. Paste with 2.5% RHA gain higher ealry strength than 5% RHA and 7.5% RHA and same to paste with 5% RHA that gain higher early strength than 7.5% RHA.

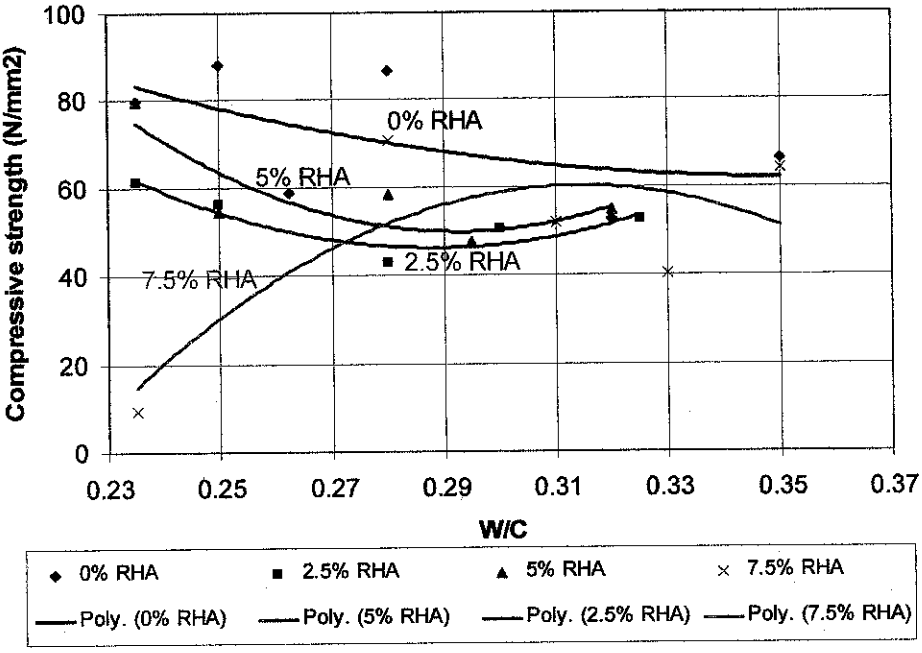


Figure 20: The graph shows the correlation between compressive strength and W/C at the age of 7 days.

From the graph above, it shows that the pastes with incorporation of RHA achieve lower compressive strength than the control paste. The compressive strength for 2.5% RHA and 5% RHA decreasing as the w/c is increasing but for paste with 7.5% RHA, the compressive strength is increasing as the w/c increasing. The compressive strength for 7.5% RHA will start dropping when the w/c exceeded 0.32. The preliminary conclusion can be made is the control paste still achieving higher early compressive strength than the pastes with RHA. Paste with 2.5% RHA which gain higher early strength than 5% RHA at the age of 3 days achieve lower compressive strength than 5% RHA at the 7 days.

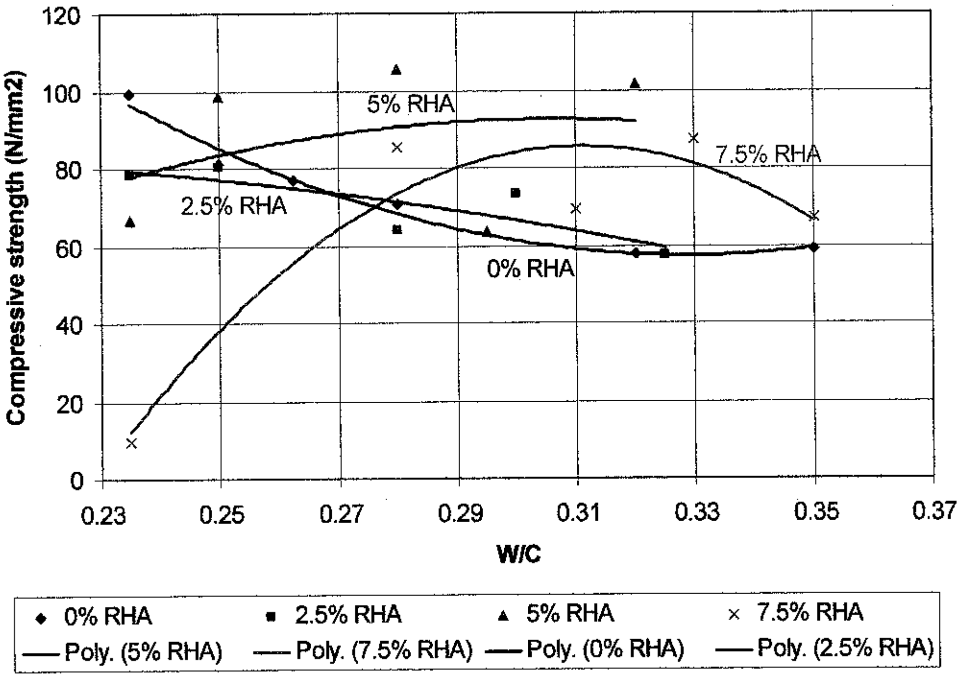


Figure 21: The graph shows the correlation between compressive strength and W/C at the 28 days.

Figure 21 shows that the optimum compressive for 0% RHA and 2.5% RHA is falling below the W/C = 0.24 and the compressive strength is continuing dropping as the W/C is increasing. Conversely, compressive strength for 5% RHA and 7.5% RHA that initially low are getting higher as the W/C is increasing. Thus the optimum W/C that give the optimum compressive strength is within W/C = 0.30 and W/C 0.32. However, the samples will obtain almost the same compressive strength when the W/C is more than 0.35.

For your information, the compressive strength for paste with 0% RHA will be decreasing as the W/C is increasing and the result obtain is justified this statement. The author having the same thought for the paste with RHA. However, paste with RHA show the different trend compare to the paste with 0% RHA. From the graph above, the curve of 2.5% RHA shows almost the same trend as the paste with 0% RHA curve trend, this may due to lower percentage of RHA incorporate in the paste. Thus, there is not much effect different between paste with 0% RHA and 2.5% RHA. Nevertheless, the 2.5%

RHA curve shows that when the W/C ratio exceeded w/c = 0.27, the compressive strength is slightly higher than the 0% RHA compressive strength.

When the percentage incorporation of RHA increased to 5% and 7.5%, there is an obvious change to the curve. The curve shows that optimum compressive strength is obtained when the W/C is falling within 0.30 and 0.32. The first thing proofing here is more water is demanded for the RHA to achieve the optimum compressive strength and to complete the hydration process. The second thing is the compressive strength does not dropping down as the W/C ratio increasing instead it increasing as the w/c is increasing. However, the trend of the curve after the W/C = 0.32 will be the same as the 0% RHA and 2.5% RHA curve where the curve will slide down and approach the 0% RHA and 2.5% RHA curve. Thus, the conclusion is the optimum water/cement-binder ratio that will give the optimum compressive strength for the paste with the incorporation of RHA is within 0.30 and 0.32 and below 0.24 for 0% RHA and 2.5% RHA.

4.9 The optimum percentage of RHA incorporation in the paste

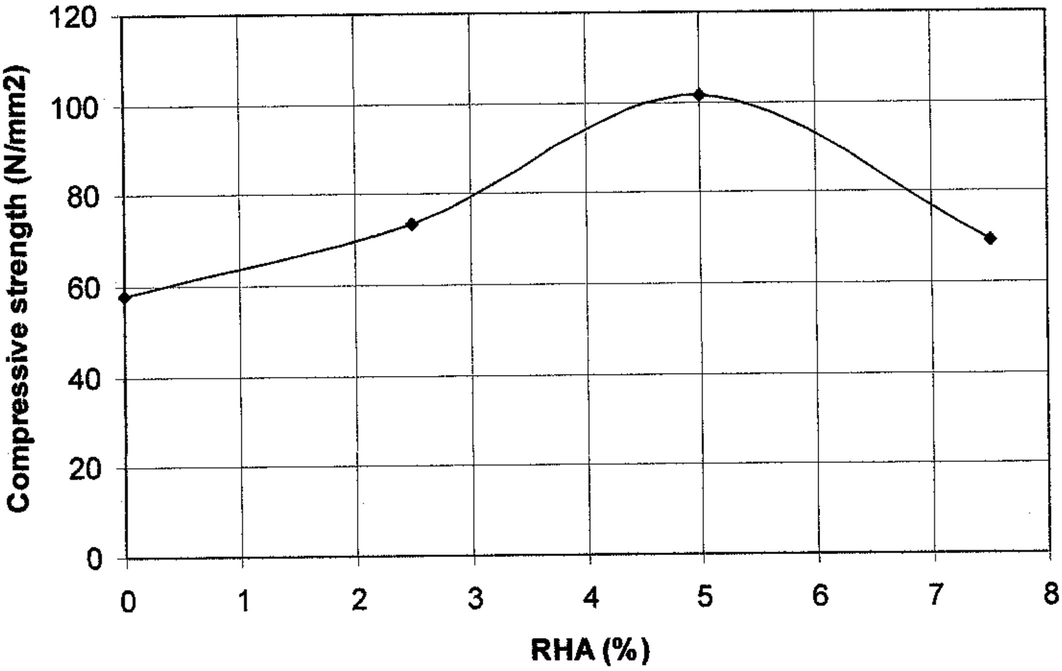


Figure 22: The graph shows the correlation between compressive strength and % RHA at the 28 days for 0.3<W/C<0.32.

The graph above shows the relation of compressive strength to the percentages of RHA incorporates in the paste. Since from the previous discussion reveals that optimum compressive strength is falling within 0.30 and 0.32, thus, the compressive strength for each percentage of RHA with the W/C within 0.30 and 0.32 is chosen to plot this graph in order to obtain the optimum percentage of RHA. According to the graph, the compressive strength is increasing with the percentages of the RHA. However, the optimum percentage of RHA is falling within 4.5% to 5.5%. The compressive strength will be sliding down when the incorporation of RHA is more than 5.5 %.

4.10 The effect of compressive strength against the ages of paste for 0.30<w/c<0.32

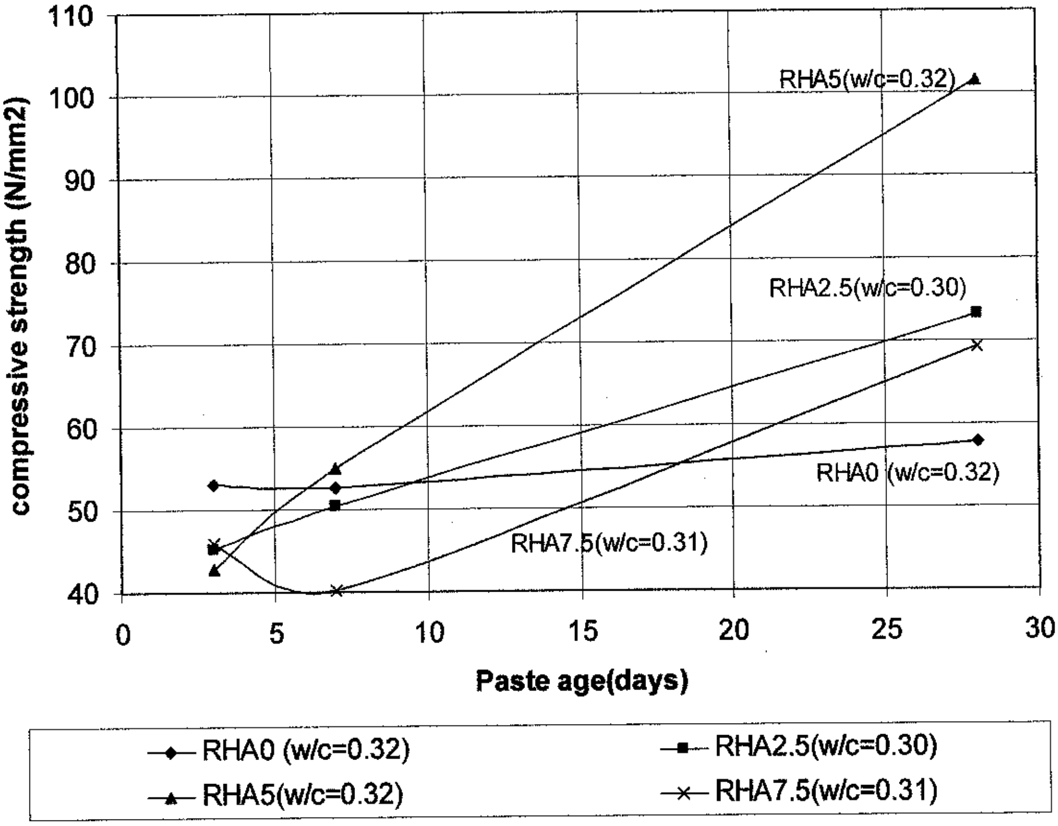


Figure 23: The graph shows the correlation between compressive strength and paste age.

The graph above shows that the relation between compressive strength and the age of the paste. From the graph, it is known that the paste with RHA has low compressive strength at the age of 3 days. This proofs that the paste with RHA will have low rate of hydration at the age of 3 days. However, 2.5% RHA and 5% RHA attain almost the same compressive strength with the control mix (0% RHA) at the age of 7 days. For 7.5% RHA at the age of 7 days, the compressive strength is dropping even lower than the 3 days compressive strength and it may due to the improper mixing that causing the sample to have honeycomb and crack. Thus, the compressive strength value may be affected by this factors and this value can be considered neglected.

At the ages of 28 days, the compressive strength for paste with RHA showing obvious changes compare to the control mix. The pastes with RHA attain higher compressive strength than the control. This proofs that RHA in the paste react slowly at the younger ages but will become very active at the higher ages. In other words, the hydration process will not active after the age of 7 days. Other than that, the graph also shows that when the paste is mixing with RHA more than 5 %, the rate of hydration of the paste will decrease. Therefore, it is advisable to mix not more than 5% to obtain higher compressive strength.

4.11 Pozzolanic reactivity index

The calculation for the pozzolanic reactivity index is showing as below:

Pozzolanic activity index with Portland cement = A/B x 100

A = average compressive strength of paste with RHA at the age of 28 days, psi(Mpa)

B = average compressive strength of control paste at the age of 28 days, psi (Mpa)

**The compressive strength for each sample is chosen base on the w/c within 0.30 and 0.32 that give the optimum compressive strength.*

Table 8: The table shows the result for pozzolanic reactivity index

Percentage of RHA	Compressive strength, N/mm^2 (B)	Compressive strength, N/mm^2 (A)	Pozzolanic Reactivity Index
0.0%	57.88	57.88	100%
2.5%	57.88	73.28	127%
5.0%	57.88	101.62	176%
7.5%	57.88	69.40	120%

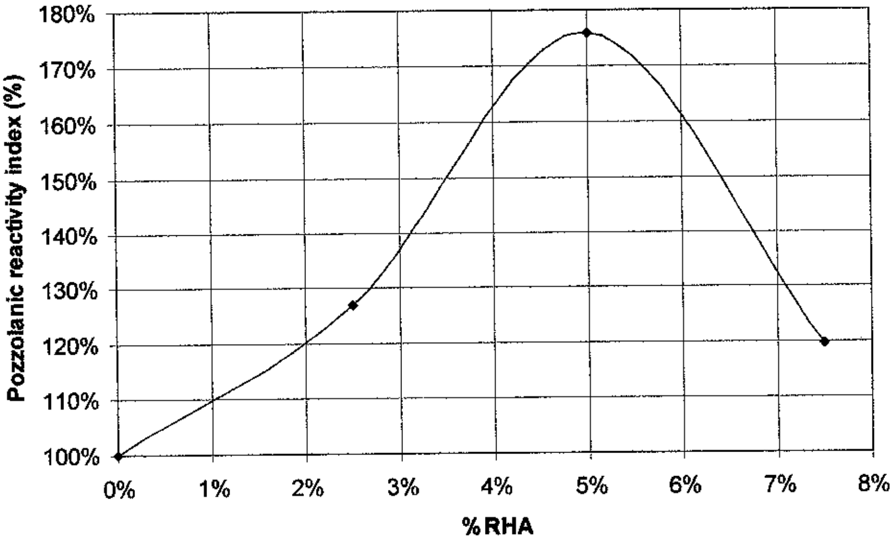


Figure 24: The graph shows the correlation between pozzolanic reactivity index and %RHA

From the graph above, the author, analyze the correlation between pozzolanic reactivity index and the percentage of RHA. The pozzolanic action in the paste is at the optimum when the paste is added with 5% of RHA. Pozzolanic action in the paste is mainly depending on the calcium hydroxide released by the hydration process of calcium silicates. At the age of 28 days, Silica in the RHA will be actively reacted with the released calcium hydroxide and 5% incorporation of RHA will successfully maximize the pozzolanic reaction.

Pozzolanic action in the paste with 7.5% RHA achieved lower pozzolanic reactivity index than paste with 5% RHA. The author predicts that there is insufficient of calcium hydroxide to react with the RHA to form the cement gel. Thus, the remaining RHA will become unhydrated substances in the paste and this will decrease the compressive strength of the paste. The author suggests higher age of paste should be used investigate the effect of pozzolanic reactivity index in the paste with RHA higher than 5%. The paste may produce more calcium hydroxide to react with RHA at the higher age and the higher amount of cement gel produce will increase the compressive strength of the paste.

4.12 TGA test result

The process for dehydration of $Ca(OH)_2$, carbonation and decarbonation is showing as below:

Dehydration of $Ca(OH)_2$,

$Ca(OH)_2$ (54g/mol) $\longrightarrow$ CaO + H_2O (18g/mol)

Carbonation and decarbonation,

$Ca(OH)_2$ (54g/mol) + CO_2 $\longrightarrow$ $CaCO_3$ (80g/mol) + H_2O (18g/mol)

$CaCO_3$ (80g/mol) $\longrightarrow$ CaO + CO_2 (44g/mol)

a) Calculation for total amount of $Ca(OH)_2$,

$$Ca(OH)_2 = \frac{54}{18}(A) + \frac{54}{44}(B)$$

where:

A = area above the TGA curve corresponding to the total mass lost due to the dehydroxylation of calcium hydroxide.

B = area above the TGA curve corresponding to the total mass lost due to the decarbonation reaction

b) Calculation for degree of hydration,

$\alpha_{CH} = [Ca(OH)_{2(i)}/Ca(OH)_{2(fh)}]*100$

i = paste age

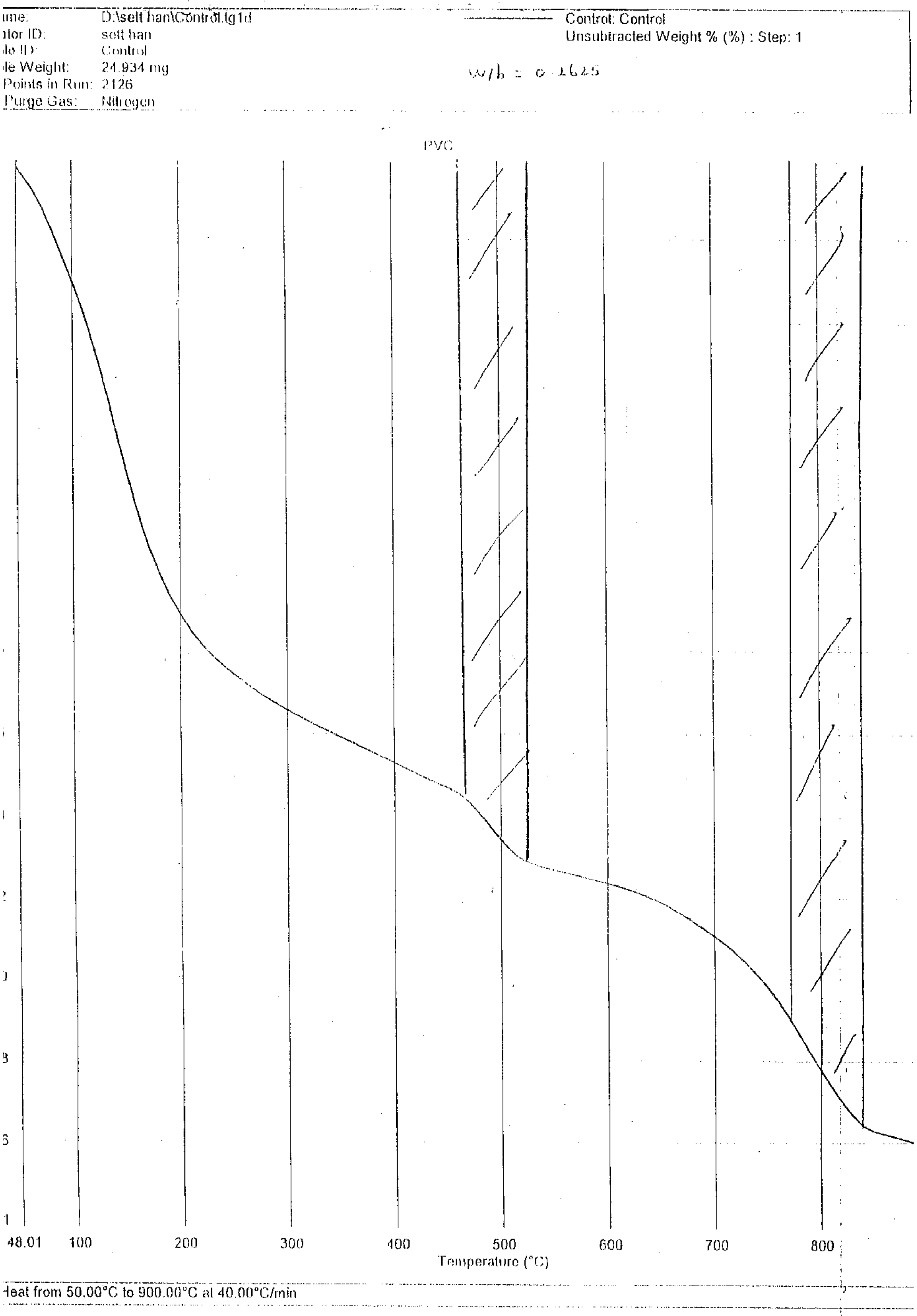
fh = paste full hydration age
= 26.69% (This value is taken from the thesis done by Dr. nasir shafiq)

Table 9: The table shows the result for degree of hydration.

RHA(%)	W/C	Total amount(%) of $Ca(OH)_2$	Degree of hydration (%)
0.0	0.2625	7.94	29.73
2.5	0.2800	10.09	37.79
5.0	0.2950	7.91	29.63
7.5	0.3100	7.62	28.54

The results of TGA are showing at the following pages. The degree of hydration should be calculated from the curve of DTG but the author is using the curve for TG to calculate the data. Thus, the value calculated out is wrong and can not be used to do the analysis. However, the information can be analyze from the graph is the dehydration of calcium hydroxide is at 450 – 525 ^{0}C and the decarbonation process is at the temperature of 740 – 840 ^{0}C.

ure 25: The graph shows the TGA result for control paste.



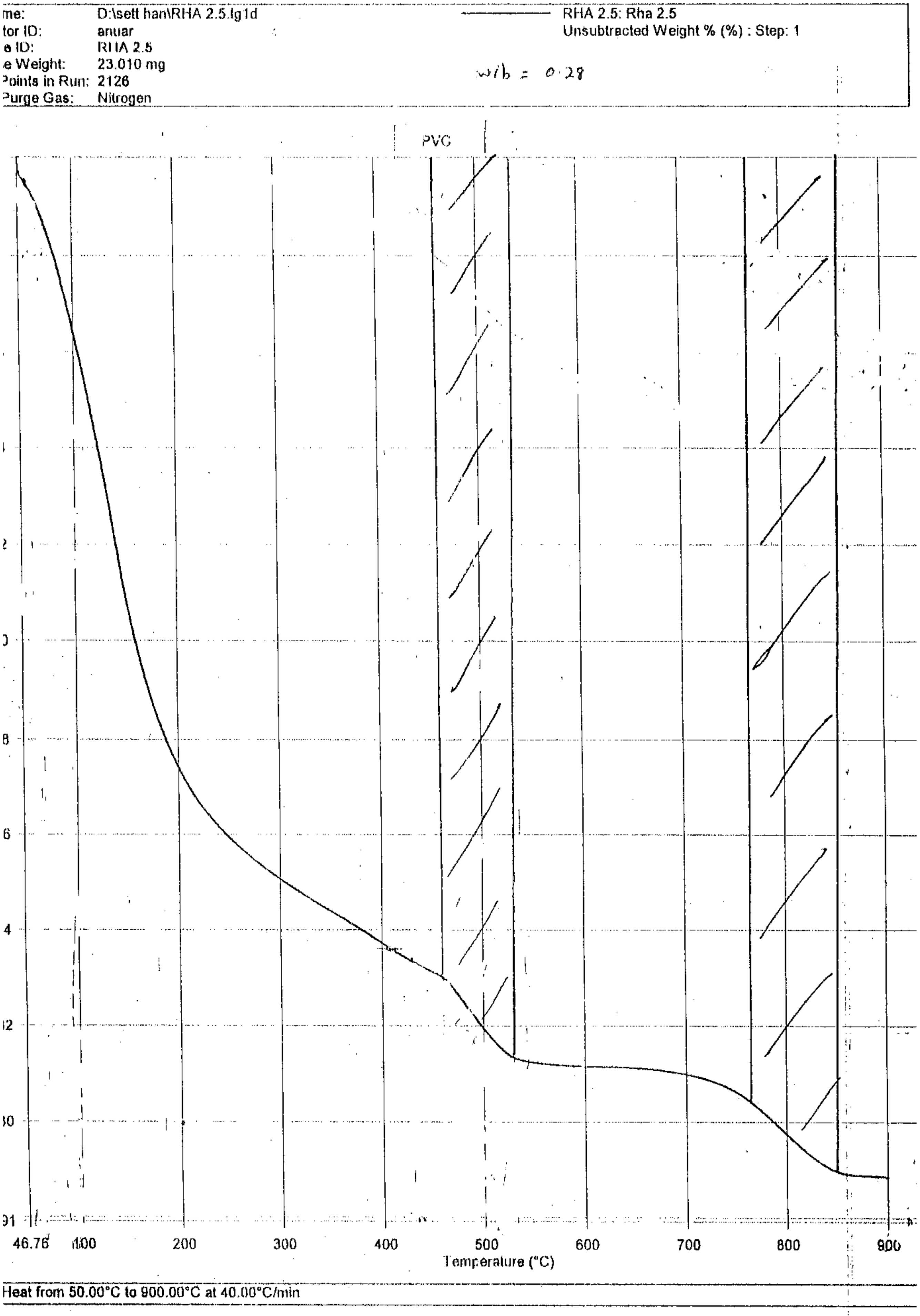
ume: D:\selt han\Control.tg1d
ıtor ID: selt han
ıle ID: Control
ıle Weight: 24.934 mg
Points in Run: 2126
Purge Gas: Nitrogen

Control: Control
Unsubtracted Weight % (%) : Step: 1

w/b = 0·2625

Heat from 50.00°C to 900.00°C at 40.00°C/min

re 28: The graph shows the TGA result for paste with 2.5% RHA.



me: D:\sett han\RHA 2.5.tg1d
tor ID: anuar
e ID: RHA 2.5
e Weight: 23.010 mg
Points in Run: 2126
Purge Gas: Nitrogen

RHA 2.5: Rha 2.5
Unsubtracted Weight % (%) : Step: 1

w/b = 0.28

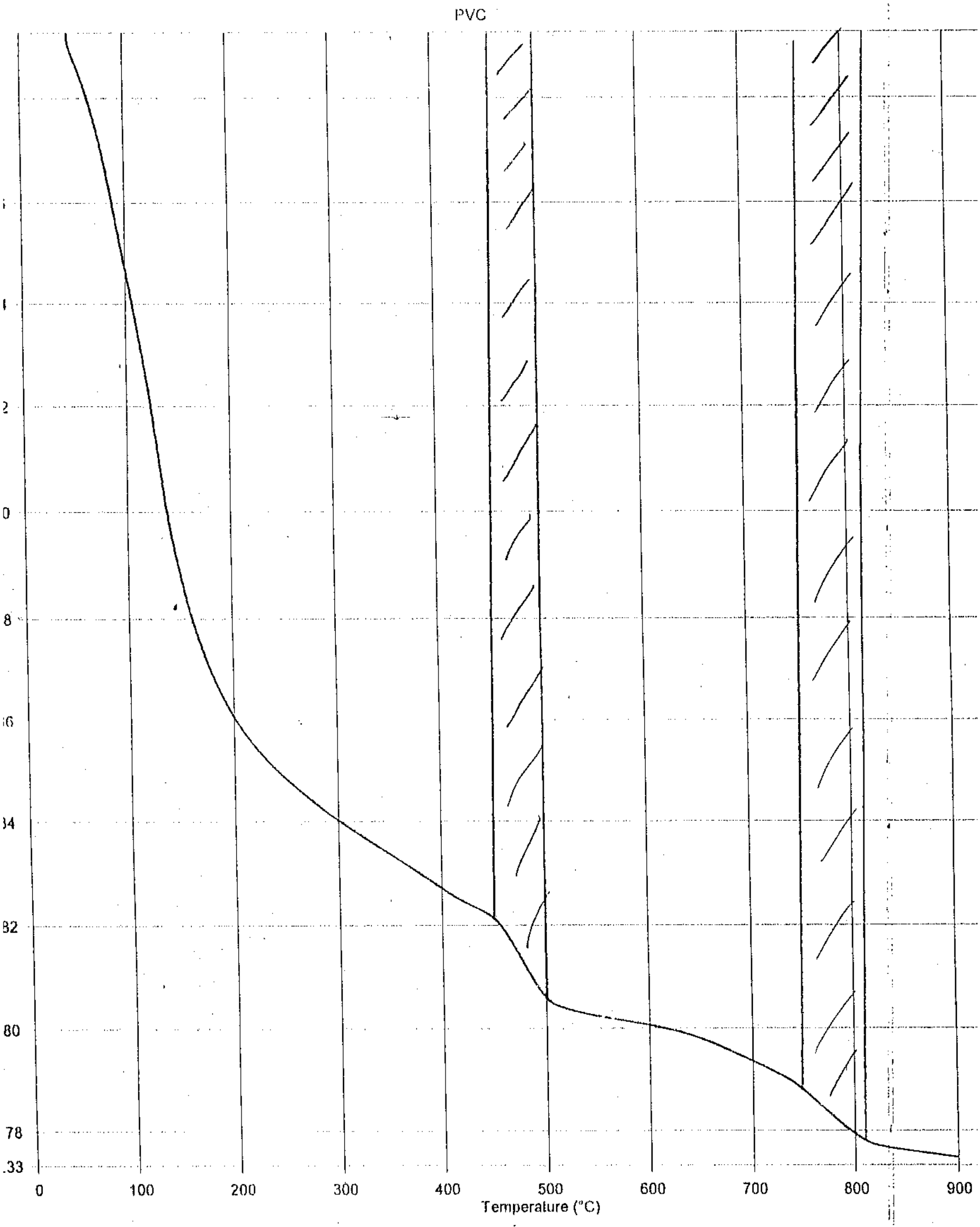
Heat from 50.00°C to 900.00°C at 40.00°C/min

e 27: The graph shows the TGA result for paste with 5% RHA.

me: D:\sett han\RHA 5.tg1d
tor ID: anuar
le ID: RHA 5
le Weight: 18.519 mg
Points in Run: 2126
Purge Gas: Nitrogen

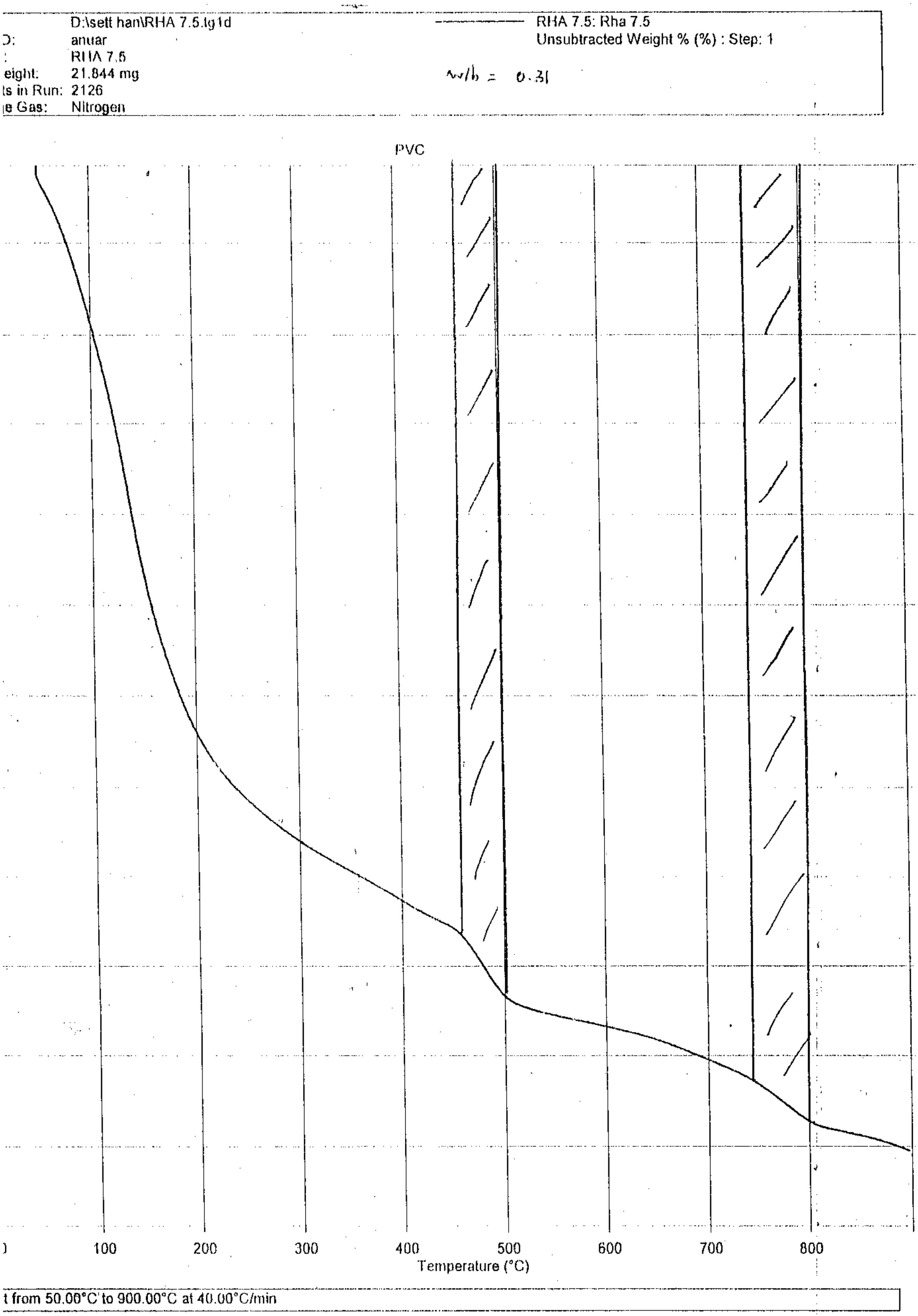
RHA 5: Rha 5
Unsubtracted Weight % (%) : Step: 1

W/b = 0.295



Heat from 50.00°C to 900.00°C at 40.00°C/min

26. The graph shows the TGA result for paste with 7.5% RHA.



D:\sett han\RHA 7.5.tg1d
): anuar
: RHA 7.5
eight: 21.844 mg
ts in Run: 2126
e Gas: Nitrogen

RHA 7.5: Rha 7.5
Unsubtracted Weight % (%) : Step: 1

w/b = 0.31

t from 50.00°C to 900.00°C at 40.00°C/min

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

Following are the conclusions and recommendations the author makes for this research:

1) The author successfully generates a standard burning procedure for rice husk. The rice husk ash can be categorized as the high quality ash after base on the analysis done on the XRD test and XRF test.
2) The fineness of the rice husk ash is determined by using the conical flask and from the result; it is proving that the fineness of rice husk ash is almost double the fineness of cement.
3) The optimum water/cement-binder ratio of the paste with the incorporation of RHA had been determined, there is within 0.30 and 0.32. By using this water/cement-binder ratio, the optimum compressive strength of the paste with RHA can be obtained.
4) The optimum percentage of RHA suitable to incorporate in the paste which gives the optimum compressive strength is approximately 5%.
5) Pozzolanic reactivity of the paste is at the optimum when the paste is mixed with 5% of RHA.
6) The author recommends that the age of the paste should extend up to 90 days so that the growing of the paste strength can be view explicitly.
7) The finding of the density of RHA should be redone by using the proper experiment equipment.
8) The TGA result is not favorable due to insufficient data provided on the graph. Thus, TGA or this project should be redone to check the degree of hydration for the RHA.

CHAPTER 6

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Designation: C 188 – 95

American Association State
Highway and Transportation Officials Standard
AASHTO No.: T133

Standard Test Method for
Density of Hydraulic Cement[1]

This standard is issued under the fixed designation C 188; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

pe

'his test method covers determination of the density of ic cement. Its particular usefulness is in connection : design and control of concrete mixtures.

'he density of hydraulic cement is defined as the mass it volume of the solids.

'he values stated in SI units are to be regarded as the d.

'his standard does not purport to address all of the concerns, if any, associated with its use. It is the ibility of the user of this standard to establish appro- afety and health practices and determine the applica- f regulatory limitations prior to use.

erenced Documents

4*STM Standards:*

4 Test Methods for Chemical Analysis of Hydraulic ment[2]

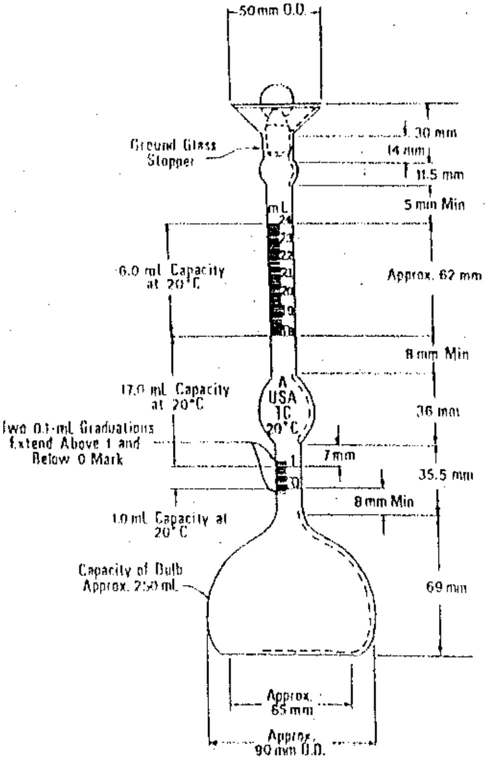
0 Practice for Preparing Precision and Bias Statements Test Methods for Construction Materials[3]

aratus

Le Chatelier flask—The standard flask which is circular s section with shape and dimensions conforming essen- to Fig. 1 (Note 1). The requirements in regard to ce, inscription and length, spacing, and uniformity of tion will be rigidly observed. There shall be a space of t 10 mm between the highest graduation mark and the point of grinding for the glass stopper.

ı The material of construction shall be best quality transparent and free of striae. The glass shall be ally resistant and shall have small thermal hysteresis. asks shall be thoroughly annealed before being gradu- 'hey shall be of sufficient thickness to ensure reasonable nce to breakage.

2 The neck shall be graduated from 0 to 1 mL and from 24 mL in 0.1-mL graduations. The error of any indicated ty shall not be greater than 0.05 mL.



NOTE 1—Variations of a few millimetres in such dimensions as tot height of flask, diameter of base, etc., are to be expected and will not b considered sufficient cause for rejection. The dimensions of the flas shown in Fig. 1 apply only to new flasks and not to flasks in use whic meet the other requirements of this test method.

FIG. 1 Le Chatelier Flask for Density Test

3.1.3 Each flask shall bear a permanent identification num ber and the stopper, if not interchangeably ground, shall bea the same number. Interchangeable ground-glass parts shall b marked on both members with the standard-taper symbo followed by the size designation. The standard temperatu shall be indicated, and the unit of capacity shall be shown b the letters "mL" placed above the highest graduation mark.

3.2 Kerosine, free of water, or naphtha, having a densit greater than 0.73 g/mL at 23 ± 2° C shall be used in the densit determination.

3.3 The use of alternative equipment or methods for dete mining density is permitted provided that a single operator ca obtain results within ± 0.03 Mg/m^3 of the results obtaine using the flask method.

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s test method is under the jurisdiction of ASTM Committee C-1 on Cement, ie direct responsibility of Subcommittee C01.25 on Fineness.

ent edition approved Sept. 10, 1995. Published November 1995. Originally :d as C 188 – 44. Last previous edition C 188 – 89(1995)$^{\epsilon 1}$.

ual *Book of ASTM Standards*, Vol 04.01.

ual *Book of ASTM Standards*, Vol 04.02.

1—The design is intended to ensure complete drainage of the en emptied, and stability of standing on a level surface, as well as and precision of reading.

cedure

Determine the density of cement on the material as d, unless otherwise specified. If the density determina- a loss-free sample is required, first ignite the sample as ed in the test for loss on ignition in section 16.1 of Test ds C 114.

Fill the flask (Note 2) with either of the liquids specified o a point on the stem between the 0 and the 1-mL mark. e inside of the flask above the level of the liquid, if ary, after pouring. Record the first reading after the flask en immersed in the water bath (Note 3) in accordance 4.

2—It is advisable to use a rubber pad on the table top when filling g the flask.

3—Before the cement has been added to the flask, a loose-fitting, g weight around the stem of the flask will be helpful in holding the an upright position in the water bath, or the flask may be held in er bath by a buret clamp.

Introduce a quantity of cement, weighed to the nearest (about 64 g for portland cement) in small increments at ne temperature as the liquid (Note 2). Take care to avoid ng and see that the cement does not adhere to the inside flask above the liquid. A vibrating apparatus may be used elerate the introduction of the cement into the flask and vent the cement from sticking to the neck. After all the t has been introduced, place the stopper in the flask and e flask in an inclined position (Note 2), or gently whirl it orizontal circle, so as to free the cement from air until no air bubbles rise to the surface of the liquid. If a proper t of cement has been added, the level of the liquid will its final position at some point of the upper series of ations. Take the final reading after the flask has been sed in the water bath in accordance with 4.4.

Immerse the flask in a constant-temperature water bath ficient periods of time in order to avoid flasktemperature variations greater than 0.2°C between the initial and the final readings.

5. Calculation

5.1 The difference between the first and the final readings represents the volume of liquid displaced by the mass of cement used in the test.

5.2 Calculate the cement density, ρ, as follows:

$$\rho(\mathrm{Mg/m^3}) = \rho(\mathrm{g/cm^3}) = \text{mass of cement, g/displaced volume, cm}^3$$

NOTE 4—The displaced volume in millilitres is numerically equal to the displaced volume in cubic centimetres.

NOTE 5—Density in megagrams per cubic metre (Mg/m^3) is numeri- cally equal to grams per cubic centimetre (g/cm^3). Calculate the cemen density, ρ, to three decimal places and round to the nearest 0.01 Mg/m^3

NOTE 6—In connection with proportioning and control of concret mixtures, density may be more usefully expressed as specific gravity, th latter being a dimensionless number. Calculate the specific gravity a follows: Sp gr = cement density/water density at 4°C (at 4°C the densit of water is 1 Mg/m^3(1g/cm^3)).

6. Precision and Bias

6.1 The single-operator standard deviation for portlan cements has been found to be 0.012.[4] Therefore, the results o two properly conducted tests by the same operator on the sam material should not differ by more than 0.03.

6.2 The multilaboratory standard deviation for portlan cements has been found to be 0.037.[4] Therefore, the results o two properly conducted tests from two different laboratories o samples of the same cement should not differ by more tha 0.10.[4]

6.3 Since there is no accepted reference material suitable f determining any bias that might be associated with this te method, no statement on bias is being made.

7. Keywords

7.1 density; hydraulic cement; specific gravity

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[4] These numbers represent the 1s and d2s limits described in Practice C 670.

Designation: C 204 – 00

American Association S
Highway and Transportation Officials Stan
AASHTO No.: T

Standard Test Method for Fineness of Hydraulic Cement by Air-Permeability Apparatus[1]

This standard is issued under the fixed designation C 204; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ope

This test method covers determination of the fineness of
lic cement, using the Blaine air-permeability apparatus,
ns of the specific surface expressed as total surface area
are centimetres per gram, or square metres per kilogram,
ient. Although the test method may be, and has been,
or the determination of the measures of fineness of
s other materials, it should be understood that, in
l, relative rather than absolute fineness values are
ed.

. This test method is known to work well for portland
s. However, the user should exercise judgement in
ining its suitability with regard to fineness measure-
of cements with densities, or porosities that differ from
ssigned to Standard Reference Material No. 114.

The values stated in SI units are to be regarded as the
d.

*his standard does not purport to address all of the
concerns, if any, associated with its use. It is the
ibility of the user of this standard to establish appro-
afety and health practices and determine the applica-
regulatory limitations prior to use.*

renced Documents

STM Standards:

/A 582M Specification for Free-Machining Stainless
Heat-Resisting Steel Bars[2]

Practice for Preparing Precision and Bias Statements
Test Methods for Construction Materials[3]

Specification for Laboratory Filter Papers[4]

ther Document:

14 National Institute of Standards and Technology
ndard Reference Material[5]

ratus

ature of Apparatus—The Blaine air-permeability apparatus consists essentially of a means of drawing a defini quantity of air through a prepared bed of cement of defini porosity. The number and size of the pores in a prepared bed definite porosity is a function of the size of the particles ar determines the rate of airflow through the bed. The apparatu illustrated in Fig. 1, shall consist specifically of the par described in 3.2-3.8.

3.2 *Permeability Cell*—The permeability cell shall consi of a rigid cylinder 12.70 ± 0.10 mm in inside diamete constructed of austenitic stainless steel. The interior of the ce shall have a finish of 0.81 μm (32 μin.). The top of the cell sha be at right angles to the principal axis of the cell. The lowe portion of the cell must be able to form an airtight fit with th upper end of the manometer, so that there is no air leakag between the contacting surfaces. A ledge ½ to 1 mm in widt shall be an integral part of the cell or be firmly fixed in the ce 55 ± 10 mm from the top of the cell for support of th perforated metal disk. The top of the permeability cell shall b fitted with a protruding collar to facilitate the removal of th cell from the manometer.

NOTE 1—ASTM A 582 Type 303 stainless steel (UNS designatio S30300) has been found to be suitable for the construction of th permeability cell and the plunger.

3.3 *Disk*—The disk shall be constructed of noncorrodin metal and shall be 0.9 ± 0.1 mm in thickness, perforated wit 30 to 40 holes 1 mm in diameter equally distributed over its area. The disk shall fit the inside of the cell snugly. The center portion of one side of the disk shall be marked or inscribed in a legible manner so as to permit the operator always to place that side downwards when inserting it into the cell. The marking or inscription shall not extend into any of the holes, nor touch their peripheries, nor extend into that area of the disk that rests on the cell ledge.

3.4 *Plunger*—The plunger shall be constructed of austenitic stainless steel and shall fit into the cell with a clearance of not more than 0.1 mm. The bottom of the plunger shall sharply meet the lateral surfaces and shall be at right angles to the principal axis. An air vent shall be provided by means of a flat 3.0 ± 0.3 mm wide on one side of the plunger. The top of the plunger shall be provided with a collar such that when the plunger is placed in the cell and the collar brought in contact with the top of the cell, the distance between the bottom of the plunger and the top of the perforated disk shall be 15 ± 1 mm.

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st method is under the jurisdiction of ASTM Committee C01 on Cement
irect responsibility of Subcommittee C01.25 on Fineness.
edition approved July 10, 2000. Published September 2000. Originally
C 204 – 46 T. Last previous edition C 204 – 96a.

Book of ASTM Standards, Vol 01.03.

Book of ASTM Standards, Vol 04.02.

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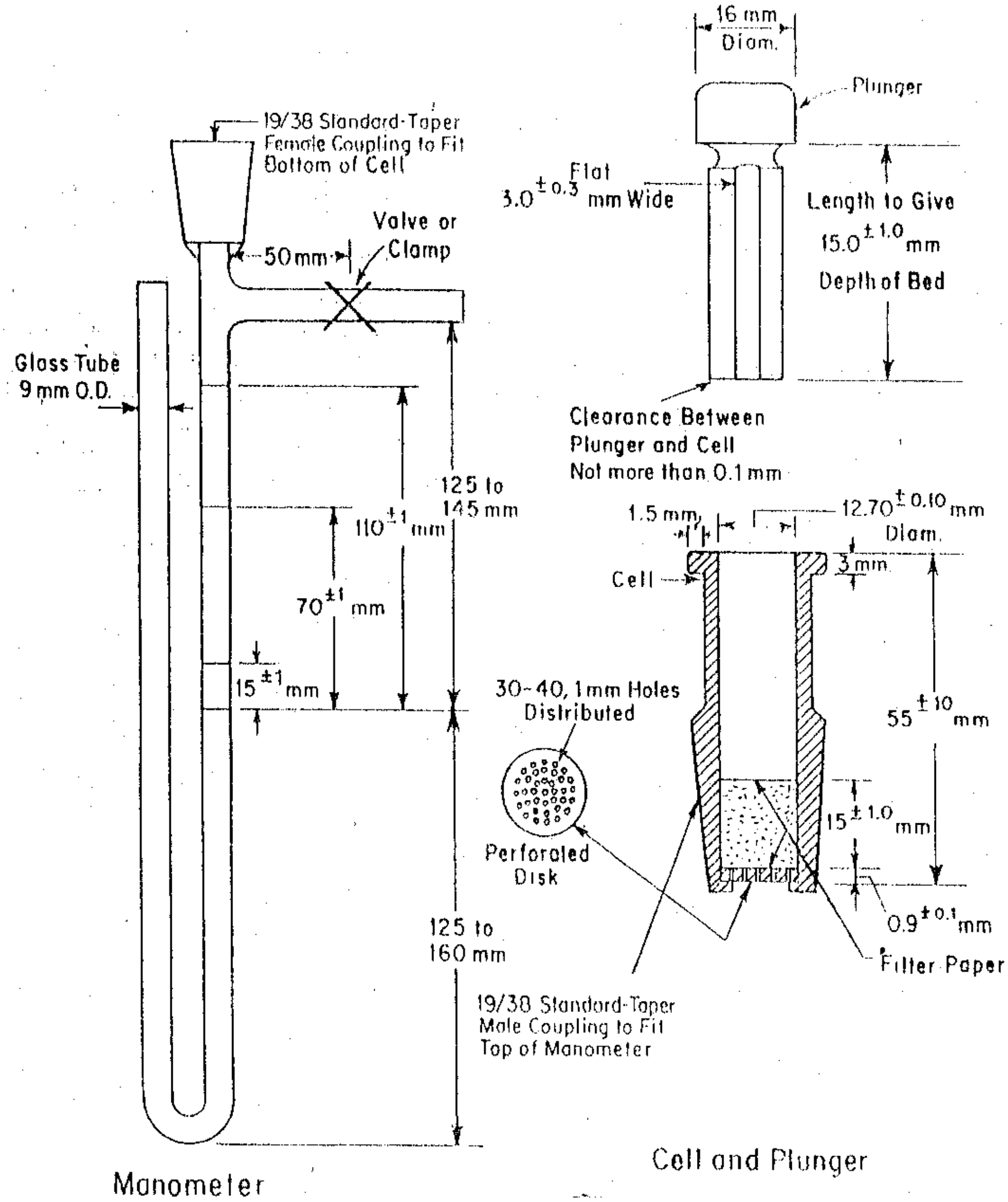


FIG. 1 Blaine Air-Permeability Apparatus

er *Paper*—The filter paper shall be medium reten-
sponding to Type 1, Grade B, in accordance with
on E 832. The filter paper disks shall be circular,
th edges, and shall have the same diameter (Note 2)
de of the cell.

Filter paper disks that are too small may leave part of the
ring to the inner wall of the cell above the top disk. When too
meter, the disks have a tendency to buckle and cause erratic

nometer—The U-tube manometer shall be con-
ccording to the design indicated in Fig. 1, using
-mm outside diameter, standard-wall, glass tubing.
one arm of the manometer shall form an airtight
with the permeability cell. The manometer arm
to the permeability cell shall have a line etched
tube at 125 to 145 mm below the top side outlet and also others at distances of 15 ± 1 mm, 70 ± 1 mm, and 110 ± 1 mm above that line. A side outlet shall be provided at 250 to 305 mm above the bottom of the manometer for use in the evacuation of the manometer arm connected to the permeability cell. A positive airtight valve or clamp shall be provided on the side outlet not more than 50 mm from the manometer arm. The manometer shall be mounted firmly and in such a manner that the arms are vertical.

3.7 *Manometer Liquid*—The manometer shall be filled to the midpoint with a nonvolatile, nonhygroscopic liquid of low viscosity and density, such as dibutyl phthalate (dibutyl 1,2-benzene-dicarboxylate) or a light grade of mineral oil.

3.8 *Timer*—The timer shall have a positive starting and stopping mechanism and shall be capable of being read to the nearest 0.5 s or less. The timer shall be accurate to 0.5 s or less for time intervals up to 60 s, and to 1 % or less for time of 60 to 300 s.

ration of Apparatus

mple—The calibration of the air permeability apparall be made using the current lot of NIST Standard e Material No. 114. The sample shall be at room ure when tested.

ulk Volume of Compacted Bed of Powder—Determine volume of the compacted bed of powder by the displacement method as follows:

Place two filter paper disks in the permeability cell, down the edges, using a rod having a diameter slightly han that of the cell, until the filter disks are flat on the d metal disk; then fill the cell with mercury, ACS grade or better, removing any air bubbles adhering to of the cell. Use tongs when handling the cell. If the cell of material that will amalgamate with mercury, the f the cell shall be protected by a very thin film of oil to adding the mercury. Level the mercury with the top ll by lightly pressing a small glass plate against the surface until the glass is flush to the surface of the and rim of the cell, being sure that no bubble or void etween the mercury surface and the glass plate. the mercury from the cell and measure and record the he mercury. Remove one of the filter disks from the ng a trial quantity of 2.80 g of cement (Note 3) the cement (Note 4) in accordance with 4.5 with one above and one below the sample. Into the unfilled he top of the cell, add mercury, remove entrapped air, off the top as before. Remove the mercury from the neasure and record the mass of the mercury.

alculate the bulk volume occupied by the cement to st 0.005 cm^3 as follows:

$$V = (W_A - W_B)/D \qquad (1)$$

bulk volume of cement, cm^3,

grams of mercury required to fill the cell, no cement being in the cell,

grams of mercury required to fill the portion of the cell not occupied by the prepared bed of cement in the cell, and

density of mercury at the temperature of test, Mg/m^3 (see Table 1).

lake at least two determinations of bulk volume of sing separate compactions for each determination.

Density of Mercury, Viscosity of Air (η), and $\sqrt{\eta}$ at Given Temperatures

, °C	Density of Mercury, Mg/m^3	Viscosity of Air, η µPa·s	$\sqrt{\eta}$
	13.55	17.98	4.24
	13.55	18.08	4.25
	13.54	18.18	4.26
	13.54	18.28	4.28
	13.53	18.37	4.29
	13.53	18.47	4.30
	13.52	18.57	4.31
	13.52	18.67	4.32
	13.51	18.76	4.33

The bulk volume value used for subsequent calculations shall be the average of two values agreeing within ±0.005 cm^3. Note the temperature in the vicinity of the cell and record at the beginning and end of the determination.

NOTE 3—It is not necessary to use the standard sample for the bulk volume determination.

NOTE 4—The prepared bed of cement shall be firm. If too loose or if the cement cannot be compressed to the desired volume, adjust the trial quantity of cement used.

4.3 *Preparation of Sample*—Enclose the contents of a vial of the standard cement sample in a jar, approximately 120 cm^3 (4 oz), and shake vigorously for 2 min to fluff the cement and break up lumps or agglomerates. Allow the jar to stand unopened for a further 2 min, then remove the lid and stir gently to distribute throughout the sample the fine fraction that has settled on the surface after fluffing.

4.4 *Mass of Sample*—The mass of the standard sample used for the calibration test shall be that required to produce a bed of cement having a porosity of 0.500 ± 0.005, and shall be calculated as follows:

$$W = \rho V(1 - \epsilon) \qquad (2)$$

where:

W = grams of sample required,

ρ = density of test sample (for portland cement a value of 3.15 Mg/m^3 or 3.15 g/cm^3 shall be used),

V = bulk volume of bed of cement, cm^3, as determined in accordance with 4.2, and

ϵ = desired porosity of bed of cement (0.500 ± 0.005) (Note 5).

NOTE 5—The porosity is the ratio of volume of voids in a bed of cement to the total or bulk volume of the bed, V.

4.5 *Preparation of Bed of Cement*—Seat the perforated disk on the ledge in the permeability cell, inscribed or marked face down. Place a filter paper disk on the metal disk and press the edges down with a rod having a diameter slightly smaller than that of the cell. Measure the mass to the nearest 0.001 g the quantity of cement determined in accordance with 4.4 and place in the cell. Tap the side of the cell lightly in order to level the bed of cement. Place a filter paper disk on top of the cement and compress the cement with the plunger until the plunger collar is in contact with the top of the cell. Slowly withdraw the plunger a short distance, rotate about 90°, repress, and then slowly withdraw. Use of fresh paper filter disks is required for each determination.

4.6 *Permeability Test:*

4.6.1 Attach the permeability cell to the manometer tube, making certain that an airtight connection is obtained (Note 6) and taking care not to jar or disturb the prepared bed of cement.

4.6.2 Slowly evacuate the air in the one arm of the manometer U-tube until the liquid reaches the top mark, and then close the valve tightly. Start the timer when the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and stop when the bottom of the meniscus of liquid reaches the third (next to the bottom) mark. Note the time interval measured and record in seconds. Note the temperature of test and record in degrees Celsius.

4.6.3 In the calibration of the instrument, make at least three ons of the time of flow on each of three separately :ds of the standard sample (Note 7). The calibration ıde by the same operator who makes the fineness on.

\ little stopcock grease should be applied to the standard tion. The efficiency of the connection can be determined by cell to the manometer, stoppering it, partially evacuating the ıe manometer, then closing the valve. Any continuous drop in cates a leak in the system.

[he sample may be refluffed and reused for preparation of the vided that it is kept dry and all tests are made within 4 h of of the sample.

ılibration—The apparatus shall be recalibrated

periodic intervals, the duration of which shall not years, to correct for possible wear on the plunger or ty cell, or upon receipt of evidence that the test is not data in accordance with the precision and bias :n Section 8.

any loss in the manometer fluid occurs, recalibrate th 4.5, or

a change is made in the type or quality of the filter for the tests.

It is suggested that a secondary sample be prepared and used s standard for the check determinations of the instrument ular calibrations with the standard cement sample.

ure

ıperature of Cement—The cement sample shall be at ›erature when tested.

: of Test Sample—The weight of sample used for the ›e the same as that used in the calibration test on the ample, with these exceptions: When determining the f Type III or other types of fine-ground portland hose bulk for this mass is so great that ordinary ssure will not cause the plunger collar to contact the cell, the weight of the sample shall be that required a test bed having a porosity of 0.530 ± 0.005. When ıg the fineness of materials other than portland r if for a portland cement sample one of the required cannot be attained, the mass of the sample shall be o that a firm, hard bed is produced by the compacting n no case, however, shall more than thumb pressure secure the proper bed, nor shall such thumb pressure hat the plunger "rebounds" from the cell top when s removed.

ıparation of Bed of Cement—Prepare the test bed of accordance with the method described in 4.5.

rmeability Tests—Make the permeability tests in e with the method described in 4.6, except that only of-flow determination need be made on each bed.

lation

lculate the specific surface values in accordance with ying equations:

$$S = \frac{S_s\sqrt{T}}{\sqrt{T_s}} \qquad (3)$$

$$S = \frac{S_s\sqrt{\eta_s}\sqrt{T}}{\sqrt{T_s}\sqrt{\eta}} \qquad (4)$$

$$S = \frac{S_s(b - \epsilon_s)\sqrt{\epsilon^3}\sqrt{T}}{\sqrt{\epsilon_s^3}\sqrt{T_s}(b - \epsilon)} \qquad (5)$$

$$S = \frac{S_s(b - \epsilon_s)\sqrt{\epsilon^3}\sqrt{\eta}\sqrt{T}}{\sqrt{\epsilon_s^3}\sqrt{T_s}\sqrt{\eta}(b - \epsilon)} \qquad (6)$$

$$S = \frac{S_s\rho_s(b_s - \epsilon_s)\sqrt{\epsilon^3}\sqrt{T}}{\rho(b - \epsilon)\sqrt{\epsilon_s^3}\sqrt{T_s}} \qquad (7)$$

$$S = \frac{S_s\rho_s(b_s - \epsilon_s)\sqrt{\eta_s}\sqrt{\epsilon^3}\sqrt{T}}{\rho(b - \epsilon)\sqrt{\epsilon_s^3}\sqrt{T_s}\sqrt{\eta}} \qquad (8)$$

where:

S = specific surface of the test sample, m^2/kg,

S_s = specific surface of the standard sample used in calibration of the apparatus, m^2/kg (Note 9),

T = measured time interval, s, of manometer drop for test sample (Note 10),

T_s = measured time interval, s, of manometer drop for standard sample used in calibration of the apparatus (Note 10),

η = viscosity of air, micro pascal seconds (µPa·s), at the temperature of test of the test sample (Note 10),

η_s = viscosity of air, micro pascal seconds (µPa·s), at the temperature of test of the standard sample used in calibration of the apparatus (Note 10),

ϵ = porosity of prepared bed of test sample (Note 10),

$\epsilon\epsilon_s$ = porosity of prepared bed of standard sample used in calibration of apparatus (Note 10),

ρ = density of test sample (for portland cement a value of 3.15 Mg/m^3 or 3.15 g/cm^3 shall be used),

ρ_s = density of standard sample used in calibration of apparatus (assumed to be 3.15 Mg/m^3 or 3.15 g/cm^3),

b = a constant specifically appropriate for the test sample (for hydraulic cement a value of 0.9 shall be used), and

b_s = 0.9, the appropriate constant for the standard sample.

NOTE 9—Upon purchase of SRM 114 series samples, a certificate comes with them that indicates the proper specific surface value.

NOTE 10—Values for $\sqrt{\eta}$, $\sqrt{\epsilon 3}$, and $\sqrt{T}$ may be taken from Tables 1-3, respectively.

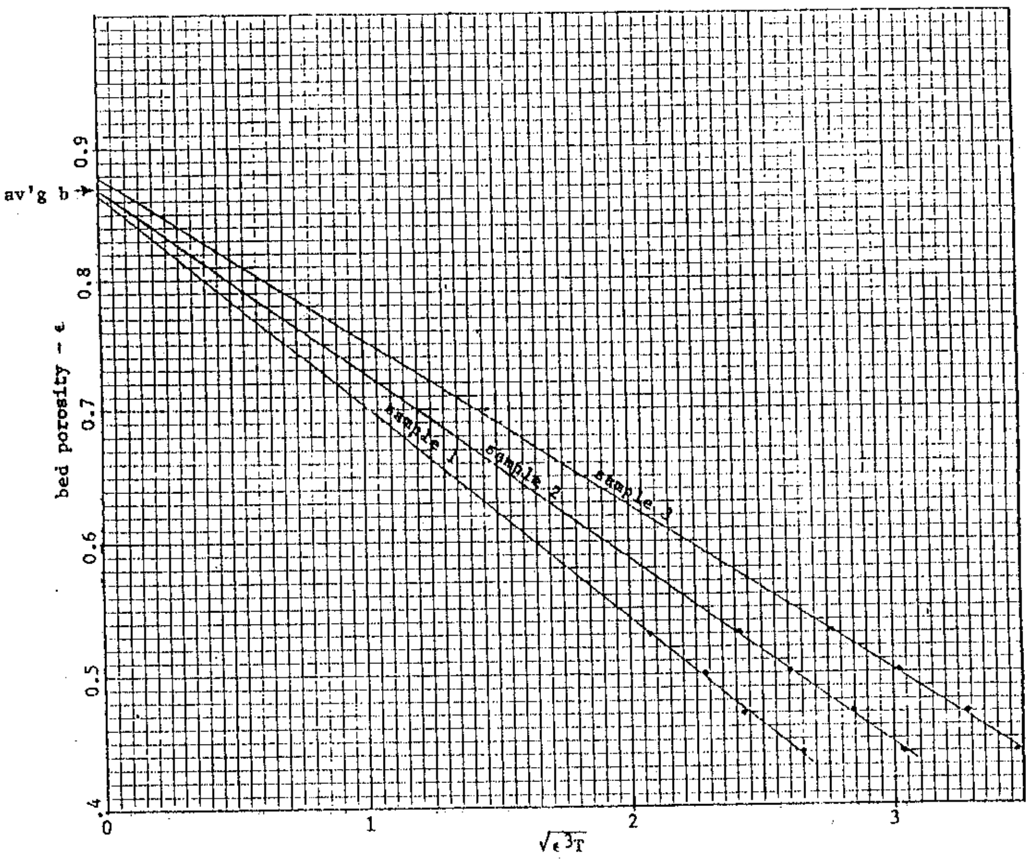
6.1.1 Eq 3 and 4 shall be used in calculations of fineness of portland cements compacted to the same porosity as the standard fineness sample. Eq 3 is used if the temperature of test of the test sample is within ±3°C of the temperature of calibration test, and Eq 4 is used if the temperature of test of the test sample is outside of this range.

6.1.2 Eq 5 and 6 shall be used in calculation of fineness of portland cements compacted to some porosity other than that of the standard fineness sample used in the calibration test. Eq 5 is used if the temperature of test of the test sample is within ± 3°C of the temperature of calibration test of the standard fineness sample, and Eq 6 is used if the temperature of test of the test sample is outside of this range.

6.1.3 Eq 7 and 8 shall be used in calculation of fineness of materials other than portland cement. Eq 7 shall be used when the temperature of test of the test sample is within ±3°C of the temperature of calibration test, and Eq 8 is used if the temperature of test of the test sample is outside of this range.

(Nonmandatory Information)

X1.



ϵ	W	T	$\sqrt{\epsilon^3 T}$
		Sample 1	
0.530	2.350	29.0	2.078
0.500	2.500	42.0	2.291
0.470	2.650	57.5	2.443
0.440	2.800	82.5	2.651
		Sample 2	
0.530	2.350	39.0	2.410
0.500	2.500	55.5	2.634
0.470	2.650	79.0	2.864
0.440	2.800	108.5	3.040
		Sample 3	
0.530	2.350	51.5	2.769
0.500	2.500	73.0	3.021
0.470	2.650	104.0	3.286
0.440	2.800	141.5	3.472

rial: Silica flour
density of test sample = 2.65 $\mathrm{Mg/m^3}$
bulk volume of sample bed = 1.887 $\mathrm{cm^3}$
desired porosity of test
= grams of sample required = $\rho V(1 - \epsilon)$
= measured test time interval, seconds

puted values of b by linear regression:
imple 1 b = 0.863 (correlation coefficient = 0.9980)
imple 2 b = 0.869 (correlation coefficient = 0.9993)
imple 3 b = 0.879 (correlation coefficient = 0.9973)
verage b = 0.870

G. X1.1 Illustrative Method for the Determination of the Value for the Constant b (for use in fineness calculations of materials o than portland cement)

Designation: C 187 – 98

American Association Stat
Highway and Transportation Officials Standar
AASHTO No.: T 12

Standard Test Method for Normal Consistency of Hydraulic Cement[1]

This standard is issued under the fixed designation C 187; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

pe

This test method covers the determination of the normal ency of hydraulic cement.

The values stated in SI units are to be regarded as the d. The values given in parentheses are for information.

This standard does not purport to address all of the concerns, if any, associated with its use. It is the sibility of the user of this standard to establish appro- safety and health practices and determine the applica- f regulatory limitations prior to use.

erenced Documents

ASTM Standards:

5 Practice for Mechanical Mixing of Hydraulic Cement stes and Mortars of Plastic Consistency[2]

0 Practice for Use of Apparatus for the Determination Length Change of Hardened Cement Paste, Mortar, and ncrete[2]

05 Specification for Weights and Weighing Devices fo in the Physical Testing of Hydraulic Cements[2]

93 Specification for Reagent Water[3]

7 Practice for Use of the Terms Precision and Bias in TM Test Methods[4]

ificance and Use

This test method is intended to be used to determine the of water required to prepare hydraulic cement pastes ing.

aratus

Weights and Weighing Devices—The weights and g devices shall conform to the requirements of Speci- C 1005. The weighing device shall be evaluated for n and accuracy at a total load of 1000 g.

Glass Graduates, 200 or 250-mL capacity, and conform- he requirements of Practice C 490.

4.3 *Vicat Apparatus*—The Vicat apparatus shall consist of a frame *A* (Fig. 1) bearing a movable rod *B*, weighing 300 g, one end *C*, the plunger end, being 10 mm in diameter for a distance of at least 50 mm, and the other end having a removable needle *D*, 1 mm in diameter and 50 mm in length. The rod *B* is reversible, and can be held in any desired position by a set screw *E*, and has an adjustable indicator *F* which moves over a scale (graduated in millimetres) attached to the frame *A*. The paste is held in a rigid conical ring *G*, resting on a plane nonabsorptive square base plate *H*, about 100 mm on each side. The rod *B* shall be made of stainless steel having a hardness of not less than 35 HRC (Note), and shall be straight with the plunger end which is perpendicular to the rod axis. The ring shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 70 mm at the base and 60 mm at the top, and a height of 40 mm. In addition to the above, the Vicat apparatus shall conform to the following requirements:

Weight of movable rod	300 ± 0.5 g (0.661 lb ± 8 grains) in
Diameter of plunger end of rod	10 ± 0.05 mm (0.394 ± 0.002 in.)
Diameter of needle	1 ± 0.05 mm (0.039 ± 0.002 in.)
Inside diameter of ring at bottom	70 ± 3 mm (2.75 ± 0.12 in.)
Inside diameter of ring at top	60 ± 3 mm (2.36 ± 0.12 in.)
Height of ring	40 ± 1 mm (1.57 ± 0.04 in.)
Graduated scale	The graduated scale, when compared with a standard scale accurate to within 0.1 mm at all points, shall not show a deviation at any point greater than 0.25 mm.

NOTE 1—The requirement that the rod be made of stainless steel shall apply only to new Vicat apparatus or replacement rods and not to equipment in use which meets the other requirements of this test method.

5. Temperature and Humidity

5.1 The temperature of the air in the vicinity of the mixing slab, the dry cement, molds, and base plates shall be maintained between 20 and 27.5°C (68 and 81.5°F). The temperature of the mixing water shall not vary from 23.0°C (73.5°F) by more than ±2.0°C (3.5°F).

5.2 The relative humidity of the laboratory shall be not less than 50 %.

6. Procedure

6.1 *Preparation of Cement Paste*—Mix 650 g of cement with a measured quantity of water following the procedure prescribed in the Procedure for Mixing Pastes of Practice C 305. The water shall conform to the numerical limits of

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est method is under the jurisdiction of ASTM Committee C-1 on Cement direct responsibility of Subcommittee C01.22 on Workability.

edition approved July 10, 1998. Published December 1998. Originally as C 187 – 44. Last previous edition C 187 – 86 (1991).

al *Book of ASTM Standards*, Vol 04.01.

al *Book of ASTM Standards*, Vol 11.01.

al *Book of ASTM Standards*, Vol 14.02.

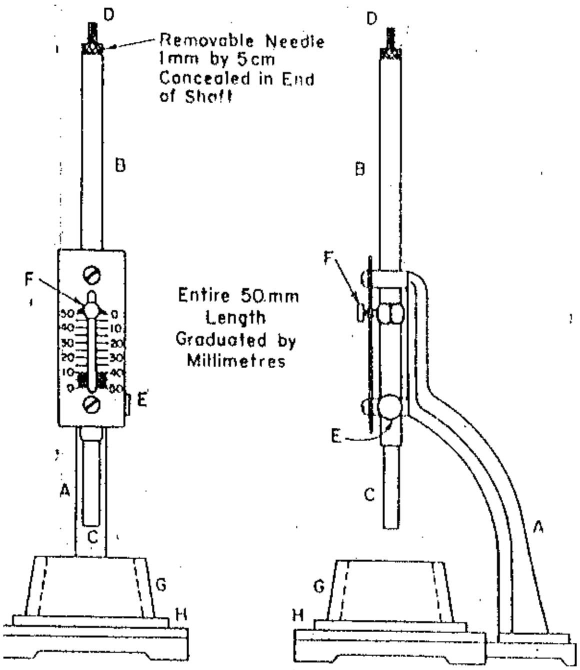

FIG. 1 Vicat Apparatus

fication D 1193 for Type III or Type IV grade of reagent

Molding Test Specimen—Quickly form the cement prepared as described in 6.1, into the approximate shape all with gloved hands. Then toss six times through a free of about 150 mm (6 in.) from one hand to another so as to ice a nearly spherical mass that may be easily inserted into icat ring with a minimum amount of additional manipu- . Press the ball, resting in the palm of one hand, into the end of the conical ring *G*, Fig. 1, held in the other hand, letely filling the ring with paste. Remove the excess at the end by a single movement of the palm of the hand. Place ng on its larger end on the base plate *H*, and slice off the excess paste at the smaller end at the top of the ring by a sing oblique stroke of a sharp-edged trowel held at a slight ang with the top of the ring, and smooth the top, if necessary, w a few light touches of the pointed end of the trowel. Duri these operations of cutting and smoothing, take care not compress the paste.

6.3 *Consistency Determination*—Center the paste confin in the ring, resting on the plate, under the rod *B*, Fig. 1, t plunger end *C* of which shall be brought in contact with t surface of the paste, and tighten the set-screw *E*. Then set t movable indicator *F* to the upper zero mark of the scale, or ta an initial reading, and release the rod immediately. This mu not exceed 30 s after completion of mixing. The apparatus sh be free of all vibrations during the test. The paste shall be normal consistency when the rod settles to a point 10 ± 1 n below the original surface in 30 s after being released. Ma trial pastes with varying percentages of water until the norn consistency is obtained. Make each trial with fresh cement.

7. Calculation

7.1 Calculate the amount of water required for norn consistency to the nearest 0.1 % and report it to the near 0.5 % of the weight of the dry cement.

8. Precision and Bias

8.1 The single operator-instrument precision has been fou to be 0.25(1S), and the multilaboratory precision has be found to be 0.35(1S) as defined in Practice E 177; therefo the results of two properly conducted tests by the sa operator in a laboratory should agree within 0.7 percenta point, and test results between two laboratories should ag with 1.0 percentage point 95 % of the time.

9. Keywords

9.1 consistency; normal consistency; Vicat needles

additional useful information on details of cement test methods, reference may be made to the "Manual of Cement Testing," which appears in the *Annual Book of ASTM Standards*, Vol 04.01.

Designation: C 305 – 99

American Association
Highway and Transportation Officials Sta
AASHTO No.:

Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency[1]

This standard is issued under the fixed designation C 305; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

.1 This practice covers the mechanical mixing of hydraulic nent pastes and mortars of plastic consistency.

.2 *This standard does not purport to address all of the ety concerns, if any, associated with its use. It is the ponsibility of the user of this standard to establish appro- ate safety and health practices and determine the applica- ity of regulatory limitations prior to use.*

Referenced Documents

2.1 *ASTM Standards:*

C 778 Specification for Standard Sand[2]

Significance and Use

3.1 This practice is intended for use in the mechanical xing of pastes and mortars for the testing of hydraulic nents.

Apparatus

4.1 *Mixer*—The mixer shall be an electrically driven me- anical mixer of the epicyclic type, which imparts both a netary and a revolving motion to the mixer paddle. The xer shall have a minimum of two speeds, controlled by finite mechanical means. (Rheostat adjustment of speed will t be acceptable.) The first, or slow speed shall revolve the ddle at a rate of 140 ± 5 r/min, with a planetary motion of proximately 62 r/min. The second speed shall revolve the ddle at a rate of 285 ± 10 r/min, with a planetary motion of proximately 125 r/min. The electric motor shall be at least 4 W (⅙hp).[3] The mixer shall be equipped with either an justment screw which is an integral part of the mixer or a earance adjustment bracket such as the one shown in Fig. 1 Note 1) to provide clearance between the lower end of the ddle and the bottom of the bowl that is not greater than 2.5 m but not less than 0.8 mm (Note 2) when the bowl is in the ixing position.

NOTE 1—When the bracket is in the proper position beneath the housing, the lugs are to the front and facing upward and the heads adjustment screws are to the rear and facing downward in the path sliding frame that holds the bowl. It is intended that the brack fastened at the front housing connection by inserting replacement s on an appropriate size upward through the opening in each lug and in existing threaded holes in the bottom of the motor housing. The or stops for the sliding frame are to be filed down if they prevent the from coming in contact with the adjustment screws.

NOTE 2—This is the approximate diameter of a grain of 20-30 s described in Specification C 778.

4.2 *Paddle*—The paddle shall be readily removable, of stainless steel, and shall conform to the basic design sl in Fig. 2.[2] The dimensions of the paddle shall be such when in the mixing position the paddle outline conforms contour of the bowl used with the mixer, and the clea between corresponding points on the edge of the paddle a side of the bowl in the position of closest approach sh approximately 4.0 mm but not less than 0.8 mm.

4.3 *Mixing Bowl*—The removable mixing bowl shall nominal capacity of 4.73 L, shall be of the general shap comply with the limiting dimensions shown in Fig. 3, shall be made of stainless steel. The bowl shall be so equ that it will be positively held in the mixing apparatus in a position during the mixing procedure. There shall be pro a lid, made of a nonabsorbing material not attacked cement.

4.4 *Scraper*—The scraper shall consist of a semirigid blade attached to a handle about 150 mm long. The blad be about 75 mm long, 50 mm wide, and tapered to a thi about 2 mm thick.

NOTE 3—A kitchen tool known as a plate and bowl scraper conf these requirements.

4.5 *Supplementary Apparatus*—The balances, w glass graduates, and any other supplementary apparatus measuring and preparing the mortar materials prior to shall conform to the respective requirements for such app as specified in the method for the particular test for whi mortar is being prepared.

5. Temperature and Humidity

5.1 The temperature of the room shall be maintain tween 20 and 27.5°C (68 and 81.5°F), and the tempera the dry materials, paddle, and bowl shall be within the range at the time of test. The temperature of the mixing

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[1] This practice is under the jurisdiction of ASTM Committee C-1 on Cement and the direct responsibility of Subcommittee C01.22 on Workability.

Current edition approved June 10, 1999. Published June 1999. Originally blished as C 305 – 53 T. Last previous edition C 305 – 94.

[2] *Annual Book of ASTM Standards*, Vol 4.01.

[3] The Model N-50 Mixer, manufactured by the Hobart Corporation, Troy, OH, ed in conjunction with paddle, special stainless steel "B" flat beater, part No. 4688 and the mixing bowl part No. 78575-2, is considered to conform to these quirements.

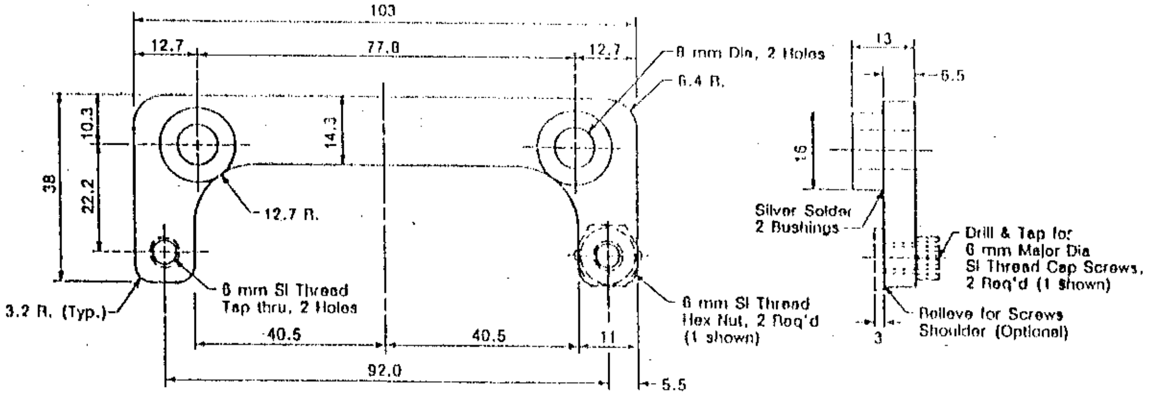

Plate and Bushings—Brass
Screws and Nuts—Steel
All Dimensions in mm

FIG. 1 Clearance Adjustment Bracket

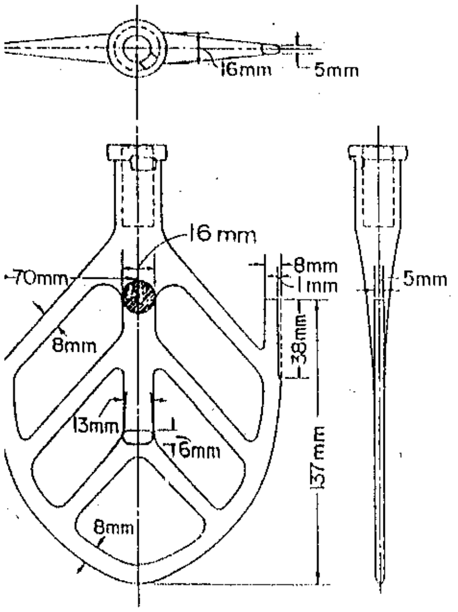

FIG. 2 Paddle

ry from 23°C (73.4°F) by more than ±1.7°C (3°F). elative humidity of the laboratory shall be not less

ls, Proportioning, and Consistency

materials and their proportions and quantities shall the requirements contained in the particular method he paste or mortar is being prepared.

ıre for Mixing Pastes

: the dry paddle and the dry bowl in the mixing the mixer. Then introduce the materials for a batch wl and mix in the following manner:

.ce all the mixing water in the bowl.

d the cement to the water and allow 30 s for the of the water.

.rt the mixer and mix at slow speed (140 ± 5 r/min)

)p the mixer for 15 s and during this time scrape he batch any paste that may have collected on sides l.

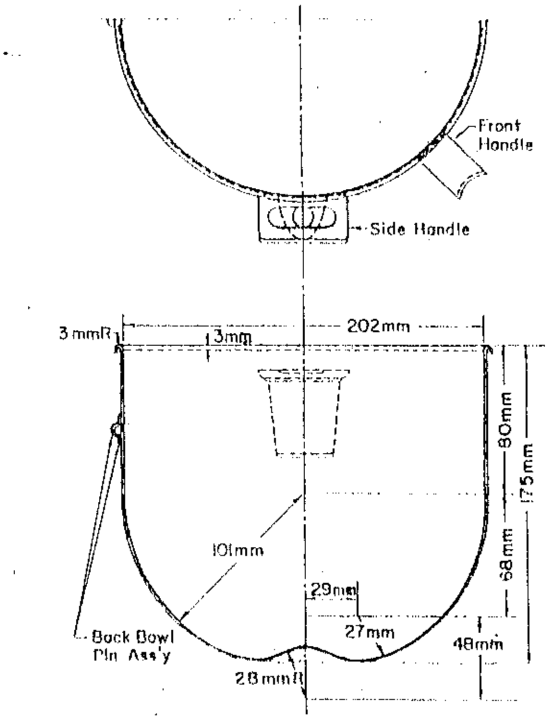

FIG. 3 Mixing Bowl

7.1.5 Start the mixer at medium speed (285 ± 10 r/min) and mix for 1 min.

8. Procedure for Mixing Mortars

8.1 Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for a batch into the bowl and mix in the following manner:

8.1.1 Place all the mixing water in the bowl.

8.1.2 Add the cement to the water; then start the mixer and mix at the slow speed (140 ± 5 r/min) for 30 s.

8.1.3 Add the entire quantity of sand slowly over a 30-s period, while mixing at slow speed.

8.1.4 Stop the mixer, change to medium speed (285 ± 10 r/min), and mix for 30 s.

8.1.5 Stop the mixer and let the mortar stand for 1½ min. During the first 15 s of this interval, quickly scrape down into batch any mortar that may have collected on the side of the wl; then for the remainder of this interval, cover the bowl h the lid.

3.1.6 Finish by mixing for 1 min at medium speed (285± 10 in).

3.1.7 In any case requiring a remixing interval, any mortar ering to the side of the bowl shall be quickly scraped down o the batch with the scraper prior to remixing.

NOTE 4—**Caution:** The clearances between the paddle and the specified in this practice are suitable when using mortar made standard sand as described in Specification C 778. To permit the mi operate freely and to avoid serious damage to the paddle and bowl coarser aggregates are used, it may be necessary to set the clea adjustment bracket to provide greater clearances than those specifi 4.1.

r additional useful information on details of cement test methods, reference may be made to the "Manual of Cen Testing," which appears in the *Annual Book of ASTM Standards*, Vol 04.01.