SHRINKAGE OF MORTAR CONTAINING RICE HUSK ASH

By

MOHD NAZIR BIN DZULKIFLI

FINAL PROJECT REPORT

Submitted to the Civil Programme in Partial Fulfillment of the Requirements for the Degree Bachelor of Engineering (Hons) (Civil Engineering)

> Universiti Teknologi Petronas Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Civil Engineering Programme Universiti Teknologi PETRONAS in partial fulfillment of the requirement for the Bachelor of Engineering (Hons) (Civil Engineering)

Approved:

Dr. Victor Macam Jr. Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK

June 2006

CERTIFICATION OF ORIGINALITY

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

Mohd Nazir Bin Dzulkifli

ABSTRACT

Through this work, different amount of Rice Husk Ash (RHA) was employed as a partial replacement of cement with percentages of 2.5%, 5%, 7.5%, 10%, 12.5%, 15%, 17.5% and 20% in mortar mixes. The physical properties of control cement mortar, and Rice Husk Ash (RHA) mortar were tested to determine the effect of this material on mortar properties. Mortar specimens were cured in water for certain days, through which, shrinkage were tested at ages 4, 11, 18, 25, 28 and 60 days, while porosity test were determined at age 7, 28 and 60 days. All the specimens were immersed in lime saturated water for certain period. The obtained results show that the shrinkage and porosity of the mortar containing rice husk ash was better than that of the control cement mortar and the optimum amount of RHA got from this research was 7.5%. One of the main reasons for the improvement of concrete properties upon addition of RHA possibly may be attributed to the formation of more C-S-H gel and less portlandite in concrete due to the reaction occurring between RHA and the Ca^{2+} , OH⁻ ions, or Ca(OH)₂ in hydrating cement.

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LIST OF ABBREVIATIONS

=	Rice Husk Ash
=	Final Year Project
=	Ordinary Portland Cement
=	Silica Fume
=	Fly Ash
=	Filler effect
<u></u>	Loss on ignition
=	Silica fume concrete
Ŧ	American Standard Test Method
=	Weight in air
=	Weight after dried
-	Weight in water

CHAPTER 1 INTRODUCTION

1.1 Project Background

Rice husk is an agro-waste material which is produced abundantly through a year. In the majority of rice producing countries much of the husk produced from the processing of rice is either burnt or dumped as a waste [2]. Rice covers 1% of the earth's surface and is a primary source of food for billions of people. Globally, approximately 600 million tones of rice paddy are produced each year. This gives annual total production of rice husk up to120 million tones which is 20% of the rice paddy production [17]. The abundant waste of rice husk may lead to the agro-waste problem. Concerning of this, the usage of RHA had been widened and this project is also part of the way to extend the usage of RHA.

This project will undergo into details about the mechanism of mortar shrinkage that contained RHA. Before going further, this part will explain generally about the volume change (shrinkage) of mortar and concrete. Concrete is subject to several types of volume changes during its service life. The four main types of shrinkage associated with concrete are plastic, autogenous, carbonation, and drying shrinkage [11]. The volume changes in concrete due to shrinkage can lead to the cracking of the concrete. In the case of reinforced concrete, the cracking may produce a direct path for chloride ions to reach the reinforcing steel. Once chloride ions reach the steel surface, the steel will corrode, which itself can cause cracking, spalling, and delamination of the concrete.

Volume changes in concrete due to autogenous shrinkage and moisture loss can result in the early deterioration of reinforced concrete. As concrete cures and dries, tensile stresses are created due to the concrete's resistance to volume changes. Drying shrinkage is defined as the decrease in concrete volume with time due to moisture loss, whereas autogenous shrinkage is defined as the reduction in volume of the concrete due to hydration of the cement. Drying shrinkage cracking is related not only to the amount of shrinkage, but also to the modulus of elasticity, creep, and tensile strength of the concrete [11]. Further explanation about shrinkage type will be discussed in next section.

Rice husk ash has many applications due to its various properties. It is an excellent insulator, so has applications in industrial processes such as steel foundries, and in the manufacture of insulation for houses and refractory bricks. It is also highly absorbent, and is used to absorb oil on hard surfaces and potentially to filter arsenic from water. More recently, studies have been carried out to purify it and use it in place of silica in a range of industrial uses, including silicon chip manufacture. There are health issues associated with the use of crystalline ash, inhalation of which can lead to a number of diseases, the most common being silicosis. This affects the potential markets for this type of ash.

1.1.1 Potential and current uses of RHA

An extensive literature search has highlighted many uses of RHA. Two main uses have been identified, as an insulator in the steel industry and as a pozzolan in the cement industry [17].

Steel industry:

• RHA is used by the steel industry in the production of high quality flat steel. Flat steel is a plate product or a hot rolled strip product, typically used for automotive body panels and domestic 'white goods' products.

• RHA is an excellent insulator, having low thermal conductivity, high melting point, low bulk density and high porosity. It is this insulating property that makes it an excellent 'tundish powder'. These are powders that are used

to insulate the tundish, prevent rapid cooling of the steel and ensure uniform solidification in the continuous casting process.

Cement industry:

• Substantial research has been carried out on the use of amorphous silica in the manufacture of concrete. There are two areas for which RHA is used, in the manufacture of low cost building blocks and in the production of high quality cement.

• Ordinary Portland Cement (OPC) is expensive and unaffordable to a large portion of the world's population. Since OPC is typically the most expensive constituent of concrete, the replacement of a proportion of it with RHA offers improved concrete affordability, particularly for low-cost housing in developing countries.

The addition of RHA to cement has been found to enhance cement properties:

• The addition of RHA speeds up setting time, although the water requirement is greater than for OPC.

• At 35% replacement, RHA cement has improved compressive strength due to its higher percentage of silica.

• RHA cement has improved resistance to acid attack compared to OPC, thought to be due to the silica present in the RHA which combines with the calcium hydroxide and reduces the amount susceptible to acid attack.

• More recent studies have shown RHA has uses in the manufacture of concrete for the marine environment. Replacing 10% Portland cement with RHA can improve resistance to chloride penetration.

• Several studies have combined fly ash and RHA in various proportions. In general, concrete made with Portland cement containing both RHA and fly ash has a higher compressive strength than concrete made with Portland cement containing either RHA or fly ash only.

RHA can also replace silica fume in high strength concrete. Silica fume or micro silica is the most commonly used mineral admixture in high strength concrete. The major characteristics of RHA are its high water demand and coarseness compared with condensed silica fume. To solve these problems RHA needs to be ground finely into particles of 8-10 μ m and a superplasticizer added to reduce water requirement. There are two patents for a ground RHA cement additive that closely matches the performance of silica fume [17].

1.2 Problem Statement

Rice is grown on every continent except Antarctica and covers 1% of the earth's surface. It is a primary source of food for billions of people, and ranks second to wheat in terms of area and production. During growth, rice plants absorb silica from the soil and accumulate it into their structures. It is this silica, concentrated by burning at high temperatures removing other elements, which make the ash so valuable [17].

The annual production of paddy rice (*Oryza sativa*) globally was 579,500,000 tonnes in 2002.Of this, 95% was produced by 20 countries, as shown in Table 1.1.

	Rice, Paddy Production in 2002	Percentage of Total Paddy	Husk Produced	Potential Ash Production (18% of
	(t)	Production	(20% of total) (t)	husk) (t)
China	177,589,000	30.7%	35,517,800	6,393,204
India	123,000,000	21.2%	24,600,000	4,428,000
Indonesia	48,654,048	8.4%	9,730,810	1.751.546
Bangladesh	39,000,000	6.7%	7,800,000	1,404,000
Viet Nam	31,319,000	5.4%	6,263,800	1.127.484
Thailand	27,000,000	4.7%	5,400,000	972.000
Myanmar	21,200,000	3.7%	4,240,000	763,200
Philippines	12,684,800	2.2%	2,536,960	456,653
Japan	11,264,000	1.9%	2,252,800	405,504
Brazil	10,489,400	1.8%	2,097,880	377,618
USA	9,616,750	1.7%	1,923,350	346,203
Korea	7,429,000	1.3%	1,485,800	267,444
Pakistan	5,776,000	1.0%	1,155,200	207.936
Egypt	5,700,000	1%	1,140,000	205,200
Nepal	4,750,000	0.8%	950,000	171.000
Cambodia	4,099,016	0.7%	819,803	147,565
Nigeria	3,367,000	0.6%	673,400	121,212
Sri Lanka	2,794,000	0.5%	558,800	100,584
Colombia	2,353,440	0.4%	470,688	84,724
Laos	2,300,000	0.4%	460,000	82.800
Rest of the World	29,091,358	5.0%	5,818,272	1,047,289
Total (World)	579,476,722	100%	115,895,344	20,861,162

Table 1Rice paddy and potential husk and ash production in the 20 highest
producing countries, 2002.

Although the potential global estimate of RHA production is 21,000,000 tones (Table1), the actual scope for utilization is considerably less. The majorities of mills from which the husks are sourced are small and dispersed within developing countries. This makes collection of the resource logistically problematical, and currently husks are dumped and burnt in open piles. The ash produced is of poor quality and is often used domestically in small quantities for cleaning glassware and cooking utensils. Thus in rural catchments areas the collection of rice husks and security of fuel supply tends to limit the practical size of biomass power plants. It is estimated that the optimum size of power plants in such areas is between 2-5 MW, producing up to 10,000 tones of ash per year.

Larger rice mills such as the Patum rice mill in Thailand produce 320,000 tones/year, and already utilize husk for cogeneration. In developed countries, where the mills are typically larger, disposal of the husks is a big problem. Burning in open piles is not acceptable on environmental grounds, and so the majority of husk is currently going into landfill. The cost of this erodes the profit of the milling company [17]. This has led to many research programmed into potential end uses of both husk and ash.

This project is concern about the research of mortar shrinkage when RHA is added. This research is done to improve the quality of mortar or concrete and also to use the industrial waste as a partial cement replacement. Shrinkage had cause cracking problem and this is the crucial part that lead to the research. The current yearly world production of paddy rice is approximately 500 million tones that give approximately 100 million tones of rice husks as a waste product from the milling.

1.3 Objectives and Scope of Study

The objective of the project is to produce a research and study about the shrinkage of mortar that containing rice husk ash. Shrinkage is one of the overlay performance parameters. Shrinkage is a three dimensional deformation of concrete that results in an overall reduction in volume. Total shrinkage may be measured under either restrained or unrestrained conditions.

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This research examines the drying shrinkage performance of mortar that mixed with RHA. It needs to be performed due to the cracking problem of concrete and agro-waste material from the rice industries. This project will conduct the study and shrinkage experiment for mortar that contained certain amount of rice husk ash. The result of the analysis must be able to determine the percentage of RHA needed in the mortar to achieve the best result and also to produce specific procedure to prepare the mortar containing RHA. For this project shrinkage was also correlated with the porosity of mortar.

CHAPTER 2 LITERATURE REVIEW

2.1 Rice Husk Ash

Research on producing rice husk ash that can be incorporated to concrete and mortars are not recent. In 1973 Metha P.K investigated the effect of pyroprocessing on the pozzolanic reactivity of RHA [4]. Since then, a lot of studies have been developed to improve the mechanical and durability properties of concrete.

Rice husks are one of the largest readily available but most underutilized biomass resources, being an ideal fuel for electricity generation. The calorific value varies with rice variety, moisture and bran content but a typical value for husks with 8-10% moisture content and essentially zero bran is 15 MJ/kg. The unburnt rice husk contains about 50% cellulose, 25-30% lignin and 15-20% of silica [12]. The treatment of rice husk as a 'resource' for energy production is a departure from the perception that husks present disposal problems. Rice husk is unusually high in ash compared to other biomass fuels - close to 20%. The ash is 92-95% silica, highly porous and lightweight, with a very high external surface area. Its absorbent and insulating properties are useful to many industrial applications and the ash has been the subject of many research studies. The concept of generating energy from rice husk has great potential, particularly in those countries that are primarily dependent on imported oil for their energy needs. For these countries, the use of locally available biomass, including rice husks is of crucial importance.

Rice husks, an agricultural waste, constitute about one fifth of 300 million tons of rice produced annually in the world. By burning the rice husks

under a controlled temperature and atmosphere, a highly reactive rice ash is obtained. In fact the ash consists of non-crystalline silica and produces similar effects in concrete as silica fume. Nevertheless, dissimilar silica fume, the particles of rice husk ash possess a cellular structure as shown in Figure 1, which is responsible for the high surface area of the material even when the particles are not very small in size [14].



Figure 1 Rice husks ash - cellular structure responsible for the high surface area.

Rice husk is the outer covering of paddy and accounts for 20–25% of its weight. It is removed during rice milling. Before rice husk is being commercialized it is used mainly as fuel for heating in Indian homes and industries. Its heating value of 13–15 MJ/kg is lower than most woody biomass fuels. However, it is extensively used in rural India because of its widespread availability and relatively low cost. The annual generation of rice husk in India is 18–22 million tons and this corresponds to a power generation potential of 1200 MW. A few rice husk-based power plants with capacities between 1 and 10 MW are already in operation and these are based either on direct combustion or through fluidized bed combustion [16]. Both these routes are beset with technical problems because of the chemical composition of rice husk and its combustion characteristics.

Rice husk is characterized by low bulk density and high ash content (18–22% by weight). The large amount of ash generated during combustion has to be continuously removed for a smooth operation of the system. Frequently, the throat of downdraft gasifier gets clogged because of the

sintering (caking) of the ash generated in the gasifier. The rice husk gasifier virtually acts as a pyrolyzer since the residence time for the particles is often not long enough for char gasification reactions to proceed [16]. This results in poor carbon conversion efficiency, sometimes as low as 55%. It has also been reported that the main reason for a poor utilization of carbon is due to difficulties in accessing all the carbon present in the material. Some of it, trapped as chemical compounds in the ash, is not easily gasified or burnt.

Silicon oxide forms the main component (90–97%) of the ash with trace amounts of CaO, MgO, K₂O and Na₂O. The melting point of SiO₂ is $1410-1610^{\circ}$ C, while that of K₂O and Na₂O is 350 and 1275° C respectively. It has been suggested that at higher temperatures, the low-melting oxides fuse with silica on the surface of the rice husk char and form glassy or amorphous phases, preventing the completion of reaction. This places an upper limit on local temperature of the gasifier. Similar behavior was also observed in fluidized beds where the bed materials, sand or alumina react with rice husk particles forming soft agglomerates and preventing the completion of pyrolysis and combustion reactions.

From the micro-structural of RHA observations it shows that there is virtually no change in size or shape of the particle until ~ 200°C. This is followed by a rapid shrinkage between 200 and 400°C. Above 400°C, the particle shrinks at a slow rate and stops shrinking after ~ 800°C. Two distinct stages in the shrinkage of particles are clearly seen (Figure 2), when the effective radius of the particles is plotted as a function of temperature. The first stage can be explained as due to a rapid thermal degradation due to volatiles escaping from the particle. This process is complete when the temperature rises to ~ 400°C. The second stage is because of char combustion due to oxygen (present in argon) and gases generated from the first stage. This stage is sluggish and less pronounced in rice husk [16].

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Figure 2 Effective radius of particle shown in

An important feature of this shrinkage is that the size reduction along the transverse direction of the largely longitudinal husks was significantly larger than along the longitudinal axis (Figure 3). Circumscribing an ellipse around the particle and calculating the aspect ratio captures this effect. In Figure 4, the aspect ratio (L/D) increased from an initial value of 2.19 to 3.23.



Figure 3 Confocal Scanning Laser Microscope (CSLM) images of a rice husk particle heated in flowing argon at various temperatures. Particle shrinkage is much more in the transverse direction than in the longitudinal direction.



Figure 4 Aspect ratio of particle vs temperature.

From the study that has being done it was confirmed that, at temperatures around 40°C and in the presence of water, the amorphous silica contained in rice husk ash can react with Ca(OH)₂ to form one kind of C-S-H gel ($Ca_{1.5} SiO_{3.5} H_2O$). The C-S-H gel looks like flocs in morphology, with a porous structure and large specific surface. The average particle diameter of the reaction product, ranging from 4.8 to 7.9µm, varies slightly with the condition under which the reaction occurs. When the product is heated, it gradually loses the water that exists in it but it maintains an amorphous form up to 750°. Above 780°C it begins to transform to crystalline CaSiO₃. One of the main reasons for the improvement of concrete properties upon addition of RHA possibly may be attributed to the formation of more C-S-H gel and less portlandite in concrete due to the reaction occurring between RHA and the Ca²⁺, OH ions, or Ca(OH)₂ in hydrating cement [1].

Regarding the strengthening mechanism of RHA blended concrete, Sugita et al. [1] suggested that:

- the average pore size of RHA concrete compared with that of control concrete is decreased;
- (2) the practical water-to-cement (w/c) ratio of RHA concrete is less than the used one because a portion of free water has been adsorbed in the great number of mesoperes existing in RHA particles and having an average pore diameter of about 80Å;

(3) Cement hydration is improved and

(4) More C-S-H gel may be formed in RHA concrete due to the reaction that probably occurs between the silica in RHA and the Ca²⁺, OH⁻ ions, or Ca(OH)₂ in hydrating cement.

2.2 Pozzolanic Materials

A "pozzolan" is defined as "a siliceous or siliceous and aluminous material, which in itself possesses little or no cementing property, but will in a finely divided form - and in the presence of moisture - chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties."

Another definition of pozzolans:

• A pozzolanic material has to contain reactive silicates or aluminosilicates.

• The particles must be fine enough to provide a sufficient reactive surface area for the solid-state chemical reactions.

• The particles react with the alkalis and calcium hydroxide from the cement to produce cementitious compounds (calcium-silicate hydrate gel, calcium-alumino silicates, etc.).

Regarding the ambiguous words "fine enough", generally 45 μ m (micron) are specified as the maximum particle size. Realizing that these particles are too big to allow a timely reaction between the lime that is given off as the cement hardens, and the silicate or alumino-silicate of the pozzolan, fly ash producers often indicate 35 μ m as the maximum particle size. Some researchers in the field insist that even this is normally still too big, and they specify a maximum of 10 μ m.

Cement gives off lime as it hardens and this lime will inevitably react with silica (silicates or alumino-silicates). The aggregate in the concrete is basically silica, but unfortunately it reacts too slowly, due to its reduced total surface area. In a concrete without pozzolans, the lime produced in the hardening of the cement will slowly react with the aggregate, producing gels. These gels are expansive, and that is welcome as long as they just fill the voids with a slight pressure thus avoiding water penetration and leaching.

The problem starts when too large a volume of gels is produced after the concrete has hardened. That is the case if the lime only has aggregate or large pozzolanic particles to react with. Given the relatively small total surface area of the aggregate or big pozzolanic particles, as well as the slow reaction between lime and silica, most of the gels will be produced after the hardening of the cement, resulting in possible disastrous pressure build-up and a slow destruction of the concrete. This makes it clear that the pozzolan has to be very fine (<10 μ m) to ensure that most of the gels are formed before the hardening.

Pozzolans play an important role when added to Portland cement because they usually increase the mechanical strength and durability of concrete structures. The most important effects in the cementitious paste microstructure are changes in pore structure produced by the reduction grain size caused by the pozzolanic reaction, pozzolanic effect and the obstruction of pores and voids by the action of the finer grains [15].

The utilization of pozzolans in combination with Portland cement to obtain high-performance concrete principally aims at improving concrete microstructure. The small particles of pozzolans are less reactive than Portland cement. When dispersed in the paste, they generate a large number of nucleation sites for the precipitation of the hydration products. Therefore, this mechanism makes the paste more homogeneous and dense as for the distribution of the finer pores, because of the pozzolanic reactions between the amorphous silica of the mineral addition and the calcium hydroxide produced by the cement hydration reactions. In addition, the physical effect of the finer grains allows denser packing within the cement and reduces the wall effect in the transition zone between the paste and the aggregates. This weaker zone is strengthened due to the higher bond between these two phases, improving the concrete microstructure and properties [15]. In general, the pozzolanic effect (PE) depends not only on the pozzolanic reaction but also on the physical or filler effect of the smaller particles in the mixture.

Therefore, the addition of pozzolans to cement results in increased mechanical strength and durability when compared to the plain paste because of the interface reinforcement. Thus the pozzolanic effect on the paste microstructure depends not only on the pozzolanic reactions but also on the filler effect (FE) of the finer particles. The physical action of the pozzolanic effect was stronger in the binary and ternary mixtures prepared with rice husk ash in proportions of 25% higher.

Pozzolans not only strengthen and seal the concrete; they have many other beneficial features. All of the below benefits apply to fly ash and rice husk ash, and most of them to silica fume as well:

- Spherical Shape: Fly ash (FA) and rice hull ash (RHA) particles are almost totally spherical in shape, allowing them to flow and blend freely in mixtures.
- **Ball Bearing Effect**: The "ball-bearing" effect of FA and RHA particles creates a lubricating action when concrete is in its plastic state.
- Economic Savings: Pozzolans replace higher volumes of the more costly cement, with typically less cost per volume.
- **Higher Strength**: Pozzolans continue to combine with free lime, increasing structural strength over time.
- Decreased Permeability: Increased density and long-term pozzolanic action, which ties up free lime, results in fewer bleed channels and decreases permeability.
- Increased Durability. Dense pozzolan concrete helps keep aggressive compounds on the surface, where destructive action is lessened.
 Pozzolan concrete is also more resistant to attack by sulfate, mild acid, soft (lime-hungry) water, and seawater.

- **Reduced Sulfate Attack**: Pozzolans tie up free lime that otherwise could combine with sulfate to create destructive expansion
- **Reduced Efflorescence**: Pozzolans chemically bind free lime and salts that can create efflorescence. Denser concrete, due to pozzolans, holds efflorescence-producing compounds on the inside.
- <u>Reduced Shrinkage</u>: The largest contributor to drying shrinkage is water content. The lubricating action of FA and RHA reduces the need for water and therefore also drying shrinkage
- Reduced Volume: As pozzolans can in certain cases substitute for up to four times the mass of cement, besides making the same amount of concrete harder than without pozzolans, less voluminous structures are able to bear the same load.
- Reduced Heat of Hydration: The pozzolanic reaction between pozzolan and lime generates less heat, resulting in reduced thermal cracking when pozzolans are used to replace Portland cement.
- Reduced Alkali Silica Reactivity: Pozzolans combine with alkalis from cement that might otherwise combine with silica from aggregates, which would cause potentially destructive expansion.
- Workability: Concrete enhanced with FA and RHA is easier to place, with less effort, responding better to vibration to fill forms more completely.
- Ease of Pumping: Pumping of FA and RHA concrete requires less energy; therefore longer distances pumping are possible.
- Improved Finishing: Sharp, clear architectural definition is easier to achieve with FA and RHA concrete, with less worry about in-place integrity.
- **Reduced Bleeding**: Fewer bleed channels decreases porosity and chemical attack. Bleed streaking is reduced for architectural finishes.

Improved paste to aggregate contact results in enhanced bond strengths.

- Reduced Segregation: Improved cohesiveness of pozzolan concrete reduces segregation that otherwise could lead to rock pockets and blemishes.
- Reduced Slump Loss: More dependable concrete allows for longer working time especially important in hot weather.
- Very low Chloride Ion Diffusion: Pozzolans make concrete more resistant to salt water (seawater).
- Improved Water Tightness: The formation of expansive gels effectively seals the concrete.
- **Resistance to Freeze-Thaw:** As water doesn't penetrate the hardened concrete, freezing can't cause destructive expansion.
- Resistance to Adverse Chemical Reactions: The example of Dynastone shows how pozzolans can protect against strong acids

2.2.1 Fly Ash (FA) as Pozzolan

Fly ash is the most commonly known artificial pozzolan and results from the burning of pulverized coal in electric power plants. The amorphous glassy spherical particles are the active pozzolanic portion of fly ash. It is important that the coal is burnt at relatively low temperatures. At higher temperatures the glassy particles would turn crystalline, rendering them useless as pozzolans. Fly ash is 66-68% glass, on an average. Class F fly ash (see ASTM C 618) readily reacts with lime (produced when Portland cement hydrates) and alkalis to form cementitious compounds. In addition to that, Class C fly ash may also exhibit hydraulic (self-cementing) properties.

Concrete made with Type C fly ash (as opposed to Type F) has higher early strengths because it contains its own lime. This allows pozzolanic activity to begin earlier. At later ages, Type C behaves very much like Type F - yielding higher strengths than conventional concrete at 56 and 90 days.

Though fly ash is typically produced in coal-fired power plants, in reality it doesn't matter at all where the ash comes from, as long as it can produce the benefits listed above. Unfortunately that may not always be true with the kind of ash you would like to use as a pozzolan. For example, coal from the east coast tends to contain sulfur, which is still present in the ash, or the particles of an ash – regardless of its origin - might be too big or contain too much carbon. In an attempt to the classify different qualities of ash, categories have been created for coal-derived fly ash.

Fly ashes that comply with ASTM C 618 for mineral admixtures in portland cement concrete come in two classes: Class C is produced from burning sub-bituminous coal and has faster strength gain, while Class F is produced from burning bituminous coal and has higher ultimate strength.

Chemical Compositi Class C Fly Ash	on of 1
Substance or Property	Requirements (ASTM C 618) %
SiO ₂ plus Al ₂ O ₃ plus Fe ₂ O ₃ , min	50
SO ₃ , max.	5
Moisture content, max.	3
Loss on ignition ¹ , max.	6

"Loss on ignition" basically refers to the carbon content in the ash. The more carbon that is still present, the more weight will lose upon burning the ash. Ideally, there should be no weight loss at all.

In combination with portland cement, **Class C** fly ash can be used as a cement replacement, ranging from 20-35% of the mass of cementitious material. Class C fly ash must replace at least 25% of the portland cement to mitigate the effects of alkali silica reaction.

Chemical Composition	of	viziti ve ti S	1753 (WROAR)	
Class F Fly Ash	r vy Le ser ^a n ser ^a n ser an s Ser an ser an			
Substance or Property	Rec (AST	quiren 'M C (nents 518) %	, 0
SiO ₂ plus Al ₂ O ₃ plus Fe ₂ O ₃ , min		70	1994) 1994 - 1	
SO ₃ , max.		5		
Moisture content, max.		3		
Loss on ignition ¹ , max.		6		

In combination with portland cement, **Class F** fly ash can be used as a cement replacement ranging from 20-30% of the mass of cementitious material. If the fly ash has high calcium content, it should not be used in sulfate exposure or hydraulic applications.

2.2.2 Silica Fume (SF) as Pozzolan

Silica fume is a waste product of the silicon metal industry, and is a super-fine powder of almost pure amorphous silica. Though difficult (and expensive) to handle, transport and mix, it has become the chosen favorite for very highstrength concretes (such as for high rise buildings), often in combination with both cement and fly ash.

Silica fume is a by-product resulting from the production of silicon or ferrosilicon alloys or other silicon alloys. Silica fume is light or dark gray in color, containing typically more than 90% of amorphous silicon dioxide. Silica fume powder collected from waste gases and without any further treatment is generally called "undensified silica fume", to distinguish it from other forms of silica fume.

Undensified silicon fume consists of very fine vitreous spherical particles with an average diameter about $0.1 \mu m$, which is 100 times smaller than the average cement particle. The undensified silica fume is almost as fine as cigarette ash and the bulk density is only about 200 - 300 kg/m3. The

relative density of typical silica fume particles is 2.2 to 2.5. Because the extreme fineness and high silicon content, silica fumes is generally a very effective pozzolan.

High-strength silica fume concretes of up to 300Mpa have been achieved in some countries. Applying silica fume in concrete fertilizer storage silos effectively reduced calcium nitrate attack. Condensed silica fume (CSF) has been used in repairing a dam stilling basin to improve abrasion erosion resistance; it has also been employed as an essential additive to prevent alkalisilica reaction.

Though condensed silica fume is much easier to handle and transport, uncondensed silica fume (normally in the form of slurry) is more effective. The smaller, already wetted particles mix much easier and distribute better, hence reactivity is better too.

Chemical composition of SF varies depending on the nature of the the manufacture process from which the SF is collected. The main constituent material in SF is silica (SiO2), the content of which is normally over 90%. The following table lists a chemical analysis of a commercially available silica fume.

Comme Si	rcially Available lica Fume
Substance	Percentage %
SiO ₂	92.85
A1 ₂ O ₃	.61
Fe ₂ O ₃	.94
CaO	.39
MgO	1.58
K ₂ O	.87
Na ₂ O	.50

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Properties of (problems with) fresh concrete with silica fume:

- Workability, water demand: Use of silica fume in concrete usually increases water demand. The increased water demand causes an increase in water to cement ratio and could negate the benefits of adding silica fume. For this reason, silica fume concrete (SFC) normally incorporates a water reducing agent or superplasticiser.
- Stability: SFC is more cohesive than conventional concrete. This is true for SFCs both with and without superplasticiser. Increased cohesiveness reduces the likelihood of bleeding and segregation. This increased cohesiveness could however increase the required compaction energy.
- Plastic shrinkage: Increased cohesiveness of SFC encourages the potentiality of plastic shrinkage and cracking that appears when the bleeding water cannot compensate for the water loss on the surface, due to evaporation. Under conditions of fast evaporation, curing measures should be taken immediately after placing the concrete.

It should be noted that to overcome the above shortcomings, sometimes fly ash (FA) and/ or rice husk ash (RHA) are also added to the concrete, together with SF.

Combining SF with the appropriate aggregates and water-reducing agent can produce high-strength concrete with a cube compressive strength of around 100Mpa, in extreme cases up to 300 Mpa. The impermeability of SFC is higher than that of similar concrete without SF.

Tests have proven that one part of silica fume can replace up to 3-4 parts of cement without any loss of strength. Replacing 10% by weight of cement with SF is a good starting point for experiments. Unfortunately, some types of SF cannot be used in concrete. The combination of Si and FeSi-75% condensed silica fume has proven to work effectively, while mixtures of FeSi-75% with FeSi-50% and FeSi-75% with CaSi have proven to be ineffective.

The silica fume particle consists mainly of "vitreous" silica particles. It has a specific gravity of about 2.20, which happens to be the accepted value for the specific gravity of any vitreous silica. Nevertheless, it has been proven that the higher the amount of impurities in silica fume, the higher the specific value. Certain impurities such as iron, magnesium, and calcium (note: but not CaSi) have shown to increase this value.

As stated before, silica fume was first looked at as a replacement for cement, but today only a portion of the cement is replaced with a much smaller amount of condensed silica fume in the concrete mix. Besides that, silica fume - just like any other pozzolan - is not inert filler, but plays an active role in the performance of the concrete.

2.2.3 Slag as a Pozzolan

The term 'slag' defined in ASTM Specification for Ground Granulated Blast-Furnace Slag for use in Concrete and Mortars (ASTM C 989) is meant to include each modifiers used in the specification title. The slag is the nonmetallic product, consisting essentially of silicates and aluminosilicates of calcium and other bases that is developed in the molten condition simultaneously with iron in a blast furnace. The granulated material is then ground to cement fineness. According to ASTM C 125, slag is the glassy, granular material formed when molten blast-furnace slag produced as a by product in the making of iron is rapidly chilled as by immersion in water. Advantage of using slag added as a separate ingredient in concrete are as follows:

- 1. higher ultimate strengths with a tendency toward lower early strengths
- 2. higher ratio of flexural to compressive strengths
- 3. improved refractory properties
- 4. lower coefficients of variation in strengths
- 5. improved resistance to sulfates and seawater

- 6. lowered expansion from alkali-silica reaction
- 7. lower temperature rise due to lower heat of hydration
- 8. better finish and lighter color
- 9. equivalent durability in freezing and thawing
- 10. decreased porosity and chloride penetration

2.2.4 Rice Husk (Hulls) Ash as a Pozzolan

Rice hull ash (RHA) is frequently referred to as a pozzolan superior to fly ash. It also known as Super – Pozzolans because of having high silica content which is above 85%. Some people even claim that it is superior to silica fume (see above). Unfortunately, there is hardly any in-depth information available in the public domain, though there should be a lot of proprietary information. RHA does not come by nature as a "finely divided powder", one of the requirements to be a good pozzolan. So it is important to make sure that it is either already finely ground or use a suitable mill and screen.

As rice hulls are an organic product, they contain carbon. The technology for burning rice hulls has improved a lot, but that doesn't mean that each and every plant that burns these hulls is using the latest technology. Even if they do, the result will not necessarily be a suitable pozzolan. The modern furnaces for rice hulls are probably mostly designed to produce as little NO₂ emission as possible. For that the hulls would have to be burnt with the minimum possible amount of air (oxygen). That in turn would unfortunately mean that the carbon content measured in "LOI" (loss on ignition) might be high.

Given the potential of pozzolans in general - and RHA in particular - it would be good if user groups like "ferrocement" could conduct research into this matter. Among other points of research they might consider ultrasonic conditioning and wet scrubbing as a previous stage before including this pozzolan in their mixes. Rice husk ash and fly ash is pozzolanic mineral admixture with silica and silica or alumina. Silica and alumina will react with free calcium hydroxide from the hydration of portland cement and causes an increase in compressive strength at a later age. Concrete made with Portland cement containing both rice husk ash and fly ash resulted in higher compressive strength than the one made with Portland cement containing with either rice husk ash or fly ash.

2.3 Comparison for Durability of Mortar Containing Different pozzolanic Materials



2.3.1 Compressive Strength

Figure 5 Compressive strength of the investigated mortar mixes at ages 3, 7 and 28 days

Figure 5 shows the compressive strength of control, cement dust, silica fume (SF), slag, grog, fly ash (FA), and RHA mortars at ages 3, 7 and 28 days. It indicates that the ultimate compressive strength increases as the period of curing in water increases for all types of mortar mixes and using 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in

the compressive strength for all ages of testing than the control mix. The increase in the strength for 3 days curing age was about 19%, 21%, 19%, 21%, 19% and 24% than the control mortar for cement dust, SF, slag, grog, FA and RHA mortar respectively [18]. The increase in the strength for 7 days curing age was about 23%, 40%, 42%, 46%, 29% and 44% than the control mortar for cement dust, SF, slag, grog, FA and RHA mortar respectively. While the increase in the strength for 28 days curing age was about 35%, 45%, 37%, 40%, 32% and 39% than the control mortar for cement dust, SF, slag, grog, FA and RHA mortar respectively. It could be seen that the addition of a fine pozzolanic material reduces both pore sizes and porosity, and therefore raises strength.

2.3.2 Flexural Strength



Figure 6 Flexural strength of the investigated mortar mixes at age 28 days

Figure 6 shows the flexural strength of control, cement dust, SF, slag, grog, FA, and RHA mortars at age 28 days. It indicates that using 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in the flexural strength. This increase was obtained 9%, 38%, 24%, 24%, 26% and 31% respectively than the control mortar [18].



Figure 7 Tensile strength of the investigated mortar mixes at age 28 days

Figure 7 shows the tensile strength of control, cement dust, SF, slag, grog, FA, and RHA mortars at age 28 days. It indicates that using 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in the tensile strength. This increase was obtained 28%, 44%, 39%, 42%, 33% and 44% respectively than the control mortar [18].



Figure 8 Compressive strength versus period of immersion in water after the preliminary 28 days curing in water



Figure 9 Compressive strength versus period of immersion in sodium sulfate solution after the preliminary 28 days curing in water

The compressive strength versus period of immersion in water after the preliminary 28 days curing in water is shown in Figure 8. The compressive strength was obtained for ages 1, 2 and 3 months for all seven mortar mixes after the preliminary 28 days curing in water. The presence of 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in the compressive strength 31%, 40%, 32%, 34%, 27% and 33% respectively than the control mortar for age 1 month after the preliminary 28 days curing in water. For the age of 2 months, the increase in the compressive strength was 21%, 31%, 25%, 25%, 19% and 23% respectively. After age of 3 months, the increase in the compressive strength was 23%, 34%, 26%, 28%, 22% and 26% respectively than the control mortar [18]. The compressive strength versus period of immersion in sodium sulfate (Na₂SO₄) solution after the preliminary 28 days curing in water is shown in Figure 9. The compressive strength was determined for ages 1, 2 and 3 months for all mixes. Using 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in the compressive strength 8%, 41%, 28%, 34%, 2% and 39% respectively than the control mortar for curing age 1 month in the solution. For the age of 2 months, the increase in the compressive strength was 11%, 31%, 26%, 29%, 7% and 29% respectively. After age of 3 months, the increase in the compressive strength was 22%, 31%, 28%, 28%, 11% and 28% respectively than the control mortar. These results demonstrate that the replacement of 10% of the Portland cement by the different materials used in this research effectively improved the resistance of the mortar to the sulfate solution attack. This may be due to its finer pore structure and the reduced content of calcium hydroxide in the cement paste.



Figure 10 Compressive strength versus period of immersion in magnesium sulfate solution after the preliminary 28 days curing in water

The compressive strength versus period of immersion in magnesium sulfate (MgSO₄) solution after the preliminary 28 days curing in water is shown in Figure 10. The compressive strength was obtained for ages 1, 2 and 3 months for all mixes. The presence of 10% of cement dust, SF, slag, grog, FA, and RHA in the mortar causes an increase in the compressive strength 10%, 33%, 30%, 30%, 7% and 7% respectively than the control mortar for curing age 1 month in the solution. For the age of 2 months, the increase in the compressive strength was 12%, 28%, 28%, 26%, 12% and 17% respectively. After age of 3 months, the increase in the compressive strength was 21%, 24%, 24%, 23%, 16% and 24% respectively than the control mortar. It is observed that the effect of magnesium sulfate solution was found to be most severe [18]. This may be due to the reaction of the magnesium sulfate solution with C₃A as well as C₃S which is the principal cementations constituent and may also be due to the lower value of pH of the saturated solution in the case of magnesium sulfate attack and thereby the stability of C.S.H in the system is reduced.

2.4 Shrinkage

Although there has been a great deal of research conducted on the effects of shrinkage and thermal gradients on concrete performance, there is no generally accepted procedure for analyzing this problem. Most current analyses concentrate on stresses from thermal gradients (Khazanovich, 1994 and Hansen, 1997), while the modeling of the concrete drying shrinkage and thermal contraction, the interface between the slab and the base, the variation in moisture conditions in the slab, and the interaction with other slabs is less understood and accounted for.

Autogenous volumes changes are associated with cement hydration alone and do not include environmental effects due to variation in moisture and temperature. The autogenously volume change with ordinary Portland cement concrete, is usually small, that is less than 0.010% or 100 microstrain expansion or shrinkage. The magnitude is dependent on the overall effect of two opposing phenomena:

- 1) The increase in the disjoining pressure in poorly crystalline C-S-H² and ettringite due to water adsorption, and
- The reduction in the disjoining pressure due to removal of adsorbed water by desiccation.

The magnitude of autogenously shrinkage when compared to other types of shrinkage is very small; it is ignored for practical purposes except in the case of dams. This is because in the interior of mass concrete there is little likelihood of occurrence of any other type of shrinkage. The development of high strength systems with very low water cement ratio has brought the phenomenon of autogenously shrinkage to the attention of researchers again. Using cement pastes made with a 0.23 or 0.30 water cement ratio, a high early strength Portland cement (430 m²/kg) and a superplasticizer, Tazawa and Miyazawa reported that the autogenous shrinkage of sealed specimens at an age of 70 days was of the order of 1000 microstrain; this autogenous shrinkage value increased to almost double when the cement was replaced with 10 or 20% condensed silica fume by weight. This phenomenon that

caused the autogenous shrinkage is also believed to be responsible for the observation that flexural strengths of sealed beams of cement paste were much lower than those obtained for specimens cured under water when the water cement ratio was less than 0.4. it appears that, due to the desiccation phenomenon, the tensile stress generated near the surface of the specimen must have caused some micro cracking that resulted in the reduction of flexural strength [5].

Carbonation shrinkage occurs as a result of chemical interaction between atmospheric carbon dioxide (CO₂) and hydration products of cement. Since it takes place concurrently with drying shrinkage, most reported data do not distinguish between the two and designate both as drying shrinkage. This is convenient when the carbonation shrinkage is low, such as with lowpermeability concrete kept in a continually wet or dry condition. The rate of carbonation is dependent on several factors such as porosity of concrete, size of the member, relative humidity, temperature, CO₂ concentration, time of exposure, method of curing, and the sequence of drying and carbonation [5].

Drying shrinkage occurs in concrete as a result of moisture loss within the cement paste in the range of 0.04 to 0.08% (400 to 800 microstrains). The source of drying shrinkage in concrete is the adsorbed water and the water held in small capillary pores of the hydrated cement paste [5]. A portion of this drying shrinkage will be elastic (recoverable) and a portion plastic (unrecoverable). This shrinkage can cause both bending and axial stresses in concrete slabs. The drying shrinkage of concrete slabs will vary, depending on the concrete mix components and curing and environmental conditions. The shrinkage of concrete will be higher for the unrestrained case (free shrinkage) than for the partially restrained case (shrinkage restricted). The partially restrained case is further complicated by elastic deformations and creep, brought about by boundary conditions acting on the concrete specimen (Farrington, et al, 1996). The creep of concrete will reduce the stresses due to shrinkage of concrete pavement slabs.

Different models exist where the rate of concrete shrinkage can be calculated as a function of the moisture conditions, the cement shrinkage, the quantity of aggregate and the elastic properties of the concrete (Ruth, 1993). The major problem with this approach is the relationship is for calculating unrestrained concrete shrinkage, which is not the true field situation. Another problem is a large number of parameters are required, of which the relative humidity at different depths in the pavement is difficult to determine.

The drying shrinkage of a concrete slab is non-uniform because of the different moisture and evaporation conditions at the surface and base of the slab. This shrinkage gradient can have the same curling effect as the night time temperature situation where the top of the slab contracts more than the bottom of the slab, as illustrated in Figure 11. This is because the top of the slab looses moisture as it is exposed to the environment (sunlight, air, and wind) and therefore shrinks more than the bottom of the slab [9].



Figure 11 Upwards and downwards curling geometry

A model proposed by Rasmussen and McCullough assumes that the full shrinkage occurs at the surface of a concrete pavement and no shrinkage occurs below the mid-depth of the slab (Rasmussen and McCullough, 1998). The shrinkage is assumed to decrease in a linear manner between the top and center of the slab, as shown in Figure 12 [9].



Figure 12 Slab shrinkage gradient assumed by Rasmussen and McCullough (1998)

The stresses from differential shrinkage can be modeled by calculating an equivalent temperature distribution for the slab, which can then be used to determine the curling stresses. The stresses caused by axial shrinkage and base friction can be modeled in the same manner as stresses from axial thermal contraction.

Since the temperature at the top of a concrete slab varies more than that at the base, the neutral axis of bending will typically be closer to the top of the slab. During the typical night time situation, the non-linear axial and linear curling components of the temperature distribution add to each other at the top of the slab resulting in high tensile stresses.

Because of concrete shrinkage, the friction between a pavement slab and the base will always result in tensile stresses in the slab. As the top of the slab shrinks more than the bottom, upwards curling similar to nigh time temperature curling will occur, resulting in tensile stresses near the surface of the slab.

Frictional stresses caused by the concrete slab cooling from the heat of hydration will be tensile. These stresses will be highest when the slab is at its coolest (during the night) and when paving was performed during the heat of the day. As the strain magnitudes in concrete slabs are generally low, linear elasticity is assumed and the tensile stresses from temperature changes can therefore be added to those from shrinkage, taking the orientations of the stresses into account.

2.5 Water Cement Ratio (w/c)

For this project, the w/c ratio used for the specimens should be minimizing to certain amount. The value will be based on the equation below:

$$V_c = w/c - 0.36\alpha \text{ cm}^3/g$$
 (Equation 1)
 $V_c = \text{Capillary pore volume}$
 $\alpha = \text{Hydration}$

It can be seen that at low w/c ratios, there is insufficient space for the hydration products to form so that complete hydration is not possible. Using this criterion, the minimum w/c ratio that can be used and still ensure complete hydration can be determined from equation above by setting $\alpha = 1.0$. However, the hydration products must be formed with the gel pores saturated. Thus, the water required for complete hydration is [24]:

Wmin = (Wn + Wg) g/g of original cement, (Equation 2)

Or (w/c)min = 0.42 α

Wn = Non-evaporable water = 0.24 α g/g of original cement

Wg = Gel water = 0.18 α g/g of original cement

Thus for complete hydration (α =1), the w/c ratio should not fall below 0.42. The space requirements for gel are less than the water requirements, so that the available water will be used up while space is still available. This means that below a w/c ratio of 0.42, a paste will self-desiccate and the residual capillary pores will become partially empty, unless water is added during the curing period. Since water is physically lost from the paste by evaporation, absorption by formwork or subgrade, etc [24]. During actual concreting, the effective minimum w/c ratio needed to avoid self-desiccation is higher than 0.42. Figures below will show the volume relationships among constituents of hydrated cement pastes:



Figure 13 Volume relationships among constituents of hydrated cement pastes; constant w/c ratio = 0.5



Figure 14 Volume relationships among constituents of hydrated cement pastes; changing w/c ratio ($\alpha = 1.0$)

2.6 Shrinkage and Stress Relationship

Drying of concrete in air results in shrinkage, while concrete kept under water swells. When the change in volume by shrinkage or by swelling is restrained stresses develop. In reinforced concrete structures, the restraint may be caused by the reinforcing steel, by the supports or by the difference in volume change of various parts of the structure. We are concerned here with the stresses caused by shrinkage, which is generally larger in absolute value than swelling and occurs more frequently. However, there is no difference in the treatment except in the sign of the term representing the amount of volume change. The symbol ϵ_{cs} will be used for the free (unrestrained) strain due to shrinkage or swelling. In order to comply with the sign convention for other causes of strain, ϵ_{cs} is considered positive when it represents elongation. Thus shrinkage of concrete, ϵ_{cs} is a negative quantity.

Stresses caused by shrinkage are generally reduced by the effect of creep of concrete. Thus the effects of these two simultaneous phenomena must be considered in stress analysis. For this purpose, the amount of free shrinkage and an expression for its variation with time are needed. Shrinkage starts to develop at time ts when moist curing stops. The strain that develops due to free shrinkage between ts and a later instant t may be expressed as follows:

$$\epsilon_{cs}(t, t_s) = \epsilon_{cs0} \beta s(t - ts)$$
 (Equation 3)

where ϵ_{cs0} is the total shrinkage that occurs after concrete hardening up to time infinity. The value of ϵ_{cs0} depends upon the quality of concrete and the ambient air humidity. The function $\beta s(t - ts)$ adopted by MC-90 depends upon the size and shape of the element considered. The free shrinkage ϵ_{cs} (t_2 t_1) occurring between any two instant t_1 and t_2 can be determined as the difference between the two values obtained by Equation 1, substituting t_1 and t_2 for t.

2.7 Porosity

When cement is mixed with water, the chemical reactions of hydration slowly begin to produce new materials (concrete, mortar). Concrete is a random composite material with the fine and coarse aggregate acting as the inclusions and the cement paste acting as the matrix.

The properties of the aggregate are measurable and usually remain constant in time, while the properties of the cement paste depend on the original water/cement ratio, type and quantity of admixtures, hydration time, degree of hydration, and to some extent on the initial particle size distribution.

In the interface transition zone between the cement paste and the aggregates the cement paste microstructure may play a critical role in determining the bulk concrete properties. It is known that bond between cement paste and aggregate surface have higher capillary porosity and larger pores than in the bulk cement paste matrix (Maso, 1980).

The porosity of the interface transition zone and cement paste is the most important coefficient which has an influence on the strength of the concrete. Pore content of 1 V% causes 4-5% loss of strength (Woods, 1968), thus production of concrete higher than 6 V% is impractical.

The presence of capillary pores and air voids influence concrete permeability to a large extent. The ingress of aggressive agents into the pore structure is responsible for various durability problems in concrete structures (aquitardity, corrosivity and freeze-thaw resistance).

The durability of concrete depends on the type, size and distribution of pores in the concrete. Further factors are the absorption and the connection of the pores and the cracks in the concrete.

The visualization of the pore content can happen in three levels. The three levels are design level, working level and post-hydration level. Chemical reactions proceed at the most favorable rate when the environmental temperature is moderate (about 15° C). On the other hand the degree of hydration affects the pore content and micro-cracking. During hydration capillary pores are developed in those parts of the concrete which are not filled by the excess volume generated during hydration (Neville, 1995).

To minimize and control the porosity of concrete the paste-saturation and the water/cement ratio of concrete have to be optimized. The oversaturated and unsaturated concretes have higher porosities compared to those of paste-saturated concrete. In the first case the difference is caused by the capillary porosity while in the second case it is caused by the quantity of the air pores (Balázs, Erdélyi, Kovács, 1990).

2.7.1 Degree of porosity of concrete

The porosity of fresh concrete depends on the following factors:

1) Saturation of concrete,

2) Compression or the air content of the packed fresh concrete (air-bubbles),

3) Quality of aggregate (porosity of aggregate, shape and surface roughness of aggregate),

4) air-pore content imported by aggregate artificially (air-pore generator).

The porosity of the hardened concrete depends in addition to the above listed factors on the following:

5) water/cement ratio (high water content),

6) Degree of hydration (age of concrete),

7) Shrinkage of concrete.

CHAPTER 3

METHODOLOGY



3.1 Test Methods of Determining Volume Changes

For evaluation of volume changes discussed in this project, ASTM offers several methods which are listed here:

- 1. ASTM Test Method for Autoclave expansion of Portland cement (C 151)
- 2. ASTM Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete (C 157) – It may be pointed out that ASTM C 157 is intended for use under a standard laboratory environment. Much greater variability and, in some cases, higher drying shrinkage values result when specimens are cast in the field under temperature and humidity conditions that are different from the laboratory.
- 3. ASTM Test Method for Drying Shrinkage of Mortar Containing Portland Cement (C 596)
- 4. ASTM Test Method for Restrained Expansion ox Expansive Cement Mortar (C 806)
- 5. ASTM Test Method for restrained Expansion of Shrinkage-Compensating Concrete (C 878)

Tools / Equipment Used

- Rice Husk Ash, Concrete materials, Mold, Comparator, Batch furnace etc.

3.1.1 Test Method for Drying Shrinkage of Mortar Containing Hydraulic Cement

The term drying shrinkage is defined as the decrease in length of the test specimen, where the decrease is caused by any factor other than externally applied forces under stated conditions of temperature, relative humidity and evaporation rate in the environment; the term includes the net effect of a variety phenomena tending to bring about both increases and decreases in length during the period in which the test specimens under consideration are stored in the environment cement and in which a number of processes, including hydration of the cement, are taking place at a variety of rates. ASTM C 596 provides a method for determining the drying shrinkage of mortar containing hydraulic cement. The test utilizes 25 by 25 by 285 mm mortar prisms, having an effective gage length of 250mm. The sand used is graded standard sand predominantly graded between the $600\mu m$ (No. 30) sieve and the $150\mu m$ (No. 30) sieve.

The specimens are kept in molds for 23.5 ± 0.5 h, but if the strength of the specimens is insufficient to allow proper removal from the mold at 24h, moist cure in the mold for 47 ± 0.5 h. Then the specimens from the molds are removed and cured in water for 48 h. At the age of 72 ± 0.5 h, the specimens are removed from water and immediately the length comparator reading for each specimen was obtained. The specimens in air storage have a clearance of at least 25 mm on all sides. Comparator readings are taken at age 4, 11, 18, 25 and 28 days of air storage.

ASTM C 596 covers determination of the effect of Portland cement on the drying shrinkage of a graded standard sand mortar subjected to stated conditions of temperature, relative humidity, and rate of evaporation in the environment. In regard to significance and use of the method, it is stated that the drying shrinkage of mortar as determined by this method has a linear relationship to the drying shrinkage of concrete made with the same cement and exposed to same drying conditions. Since drying shrinkage of concrete is greatly influenced by the aggregate content, aggregate stiffness, and water content, many researchers question the validity of extrapolating the data on mortar shrinkage to concrete shrinkage. For instance, it is inconsistent to rely on the behavior of either neat pastes or rich mortars to predict the ultimate shrinkage of concrete, especially when the tests are concluded at early ages. The mix proportion for mortar and RHA was shown in Table 2.

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To Prepare 4 samples			
% of RHA	Cement (g)	Std Sand (g)	RHA (g)
0.00	750.00	1500.00	0.00
2.50	731.25	1500.00	18.75
5.00	712.50	1500.00	37.50
7.50	693.75	1500.00	56.25
10.00	675.00	1500.00	75.00
12.50	656.25	1500.00	93.75
15.00	637.50	1500.00	112.50
17.50	618.75	1500.00	131.25
20.00	600.00	1500.00	150.00

Table 2 Mix Proportion of Mortar for shrinkage test

3.2 Preparation of RHA

RHA used in this experiment was burnt in a batch furnace at about 600°C and ground in a ball mill for 1 hour. The RHA needed for this experiment is about 4 kg. It should have an average particle diameter of 15.4 μ m. The result of the ash produced must be grey in color otherwise it can not be used in the experiments. The chemical compositions of RHA are shown in Table 3.

2.93 %	Na ₂ O	0.05 %
91.9 %	K ₂ O	2.78 %
0.25 %	TiO ₂	0.01 %
0.41 %	P ₂ O ₅	0.36 %
0.38 %	MnO	0.16 %
0.21 %	С	0.41 %
	2.93 % 91.9 % 0.25 % 0.41 % 0.38 % 0.21 %	2.93 % Na2O 91.9 % K2O 0.25 % TiO2 0.41 % P2O5 0.38 % MnO 0.21 % C

Table 3 Chemical composition (%) of RHA used in experiment,

Time (Start)	Temperature (C°)	Time (End)	Time Range (min)
	Normal-200		
	200		10
	200-250		
	250		5
	250-280		
	280		10
	280-310		
	310		10
	310-360		
	360	2 2 2	20*
	360-480		Air entrance
	480		10
	480-520		
	520		7
	520-550		
	550		10

The rice husk burning process must follow the specific procedure and use the standard equipment, shown in Table 4 and Figure 15 and Figure 16:

Table 4Procedure of Burning the Rice Husk

*At this moment the temperature should be maintain until the entire smoke stop flowing out from the furnace.

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Figure 15 Platforms to burn the Rice Husk



Figure 16 Dimensions of the platform

Notes: For each platform the quantity of rice husk used are different.

3.3 Summary of ASTM C596, C490, C778, C305 and C157

3.3.1 Preparation of Molds (C490)

Prior to the molding of specimens, the outside joint of the mold and the contact lines of the mold and base plate shall be sealed to prevent loss of mixing water from a freshly molded specimen. The surface of the mold will be thinly covered by mineral oil. The gage studs must be taking care to keep them clean and free of oil or any foreign matter.

3.3.2 Test Specimens (C157 & C596))

The test specimens for mortar shall be a prism of 25mm square crosssection and approximately 285 in length. At least three specimens shall be prepared for each test condition. A batch of mortar shall consist cement, graded standard sand (between 600 μ m (No. 30) sieve and 150 μ m (No. 100) sieve), water (0.45w/c) and rice husk ash. Mix proportion of specimens shown in Table 2.

3.3.3 Mixing Mortars

Mortar is mix using the procedure described in Practice C305

3.3.4 Molding Specimens (C157)

The mortar will be place in the mold in two approximately equal layers. Each layer was compacted with tamper. The mortar was worked into the corners, around the gage studs, and along the surfaces of the mold with the tamper until a homogeneous specimen obtained. The surfaces need to be smooth after the top layer was compacted. After the completion of molding, immediately the device was loosen for holding the gage studs in position at each and of the mold in order to prevent any restraint of the gage studs during initial shrinkage of the specimen.

3.3.5 Use of Reference Bar (C490)

Each time the comparator reading is taken the reference bar will be placed in the same position in the instrument. The dial gage setting of the comparator by use of the reference bar must be check before and after the specimens reading was taken.

3.3.6 Obtaining Comparator Readings (C490)

The specimens was rotated slowly in the measuring instrument during the reading from the comparator is being taken. When the rotation causes a change in the dial reading the minimum reading will be recorded. The specimens must be placed in the instrument with the same end up each time reading is taken.

3.3.7 Obtaining Comparator Readings of Specimens Stored in Moist (C490)

Clean the hole in the base of the comparator into which the gage stud on the lower end of the bar fits, this is because this hole tends to collect water and sand. Record the comparator indication of the reference bar. The first bar is taken out from the immersion, the pins must be blotted. Then the bar will be putted in the comparator to record the reading of the indication. The bar is then returned to immersion and the hole in the base of the comparator was cleaned again. The same procedure will be applied to another specimen. After the last reading is done the hole in the comparator will be cleaned and the reference-bar indications will again being recorded. (Note: the blot must be only around the pins; this is to avoid drying and shrinkage of the bars)

3.4 Preparation of Porosity Test

The specimens for porosity test have the same mixtures as specimens for shrinkage test. But the amount is increased by two times as the mold is larger and more specimens needed for the test. This is important to ensure that the properties of mortar are same for both tests, so that the comparison done is adequate. The test is first start by preparing the mortar using the 50 x 220 x 300 mm wood mold. After one day the specimens are demolded and cured in water. The mortar will be cored during 7 and 28 days. For both ages 3 samples are needed. After coring, the samples will be vacuumed for 30 minutes without water and then 6 hours with water. After those processes the samples will be taken out and weight in air (W_{air}) and weight in water (W_{water}) were taken. After obtained those weights the samples will be dried in oven until the samples are fully dried and after this process, weight after dried (W_{dried}) will be taken. Same procedures go for the samples at age 28. The specimens will be only taken out from the water after it reaches the age wanted.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Shrinkage Analysis

The shrinkage of each specimen was determined using the data taken from the changes of length. The calculation for the length change at certain age was using the equation below:

$$L = (Lx - Li) \times 100$$
(Equation 4)

G

L = change in length at x age, %

Lx = comparator reading of specimen at x age minus comparator reading of reference bar at x age

Li = initial comparator reading of specimen minus comparator reading of reference bar at that same time

G = nominal gage length, 250mm

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	0.0%	2.5%	5.0%	7.5%	10.0%	12.5%	15.0%	17.5%	20.0%
1	0.0080	0.0070	0.0060	0.0070	0.0180	0.0230	0.0120	0.0020	0.0100
4	0.0127	0.0112	0.0102	0.0130	0.0251	0.0300	0.0177	0.0084	0.0195
11	0.0307	0.0493	0.0315	0.0228	0.0359	0.0511	0.0436	0.0429	0.0400
18	0.0591	0.0680	0.0490	0.0372	0.0533	0.0611	0.0480	0.0501	0.0525
25	0.0701	0.0770	0.0514	0.0391	0.0595	0.0696	0.0565	0.0558	0.0544
28	0.0809	0.0776	0.0605	0.0425	0.0643	0.0723	0.0584	0.0602	0.0560
60	0.0889	0.0922	0.0693	0.0622	0.0699	0.0769	0.0629	0.0654	0.0616

Table 5 Average length change for different percentage of RHA



Figure 17 Graph of shrinkage vs ages of specimens

This investigation is aimed at developing lowest shrinkage mortar using blends of pozzolanic by-product materials as cement replacing materials. Rice husk ash is used in this project to exploit the potential of reducing the percentage of shrinkage which can also reduce the potential of cracking. The results of shrinkage at various ages are reported. Mortar containing 0%, 2.5%, 5%, 7.5%, 10%, 12.5%, 15%, 17.5% and 20% RHA were incorporated as partial cement replacements. A water-cement ratio of 0.45 was used and it is constant for all specimens. It was found that the incorporation of 7.5% RHA as cement replacement yielded the optimum performance, resulting in the lowest shrinkage values.



Figure 18 Graph of Shrinkage versus Percent of RHA

From figure 18, the graph shows the optimum point of RHA to produce lowest shrinkage which is at 7.5% line. Using 20% RHA will also produce good result of shrinkage at day 60 but to make it economically viable 7.5% is good enough. The graph also shows that after the 7.5% line the effect of RHA to the shrinkage become lesser compared with before. So this again proved that 7.5% RHA from the cement weight is the optimum weight of RHA to reduce the shrinkage.

4.2 Porosity Analysis

This section presents experimental results on porosity of RHA modified mortars using rule of thumb;

 $Porosity = (W_{air} - W_{dried} / W_{air} - W_{water})*100$ (Equation 5)

RHA	7 days	28 days	60 days
0%	21.3558	18.5906	16.2738
5%	21.7472	18.9233	16.1537
10 %	21.5711	18.8062	16.4606
15 %	21.8965	19.5229	17.8276
20 %	22.1808	19.2266	16.3866

Table 6 Table of average porosity (%) for each specimens





The graph above shows the porosity of the specimens from day 7 to day 60 in various percentage of RHA. It shows that the addition of RHA does not influence much to the porosity. This is happened because of the properties of RHA and C-S-H gel themselves is porous. So although the RHA added reduced the capillary porosity the porous properties come from the RHA and C-S-H product had make the graph not much different.

The RHA addition will greatly reduce the capillary porosity of the interfacial zone by consuming the large crystals of CH and replacing them with a higher volume of secondary C-S-H [25]. Simulation studies have shown that a 10-20% by weight silica fume addition can produce a nearly homogeneous distribution of C-S-H (primary plus secondary) throughout the microstructure [25], including the interfacial zone, same can goes to RHA as the richness of silica content is similar.

The ability of the smaller admixture particles to pack more closely to the aggregate surface is clearly evident. But if only filling in the interfacial zone with the inert admixtures, it may be inadequate to improve microstructural homogeneity. This is where the pozzolanic reactivity of the mineral admixture becomes important. Because the reaction of a pozzolanic admixture with CH produces secondary C-S-H having a greater volume than the original solid reactants, the effect of the presence of the mineral admixture in the interfacial zone may be the production of a more homogeneous microstructure and a better bond between paste and aggregate.

4.3 Relationships between surface area, porosity and drying shrinkage

All types of drying shrinkage increase with increasing C–S–H surface area and pore volumes. The pore volume and surface area data are so highly correlated. In other words, increases in surface area are generally accompanied by increases in pore volumes of all sizes. Furthermore, increases in pore volume of one size range are generally accompanied by increases in pore volume in the other size ranges. Therefore, the effects of surface area and pore volumes on shrinkage are not independent and cannot be separated [26].

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The source of drying shrinkage in concrete is the adsorbed water and the water held in small capillary pores of the hydrated cement paste [5]. This proves that when the porosity is increase the shrinkage will also increase. So the process of shrinkage can be controlled by reducing the porosity of the concrete or mortar.

CHAPTER 5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, this project is expected to achieve the objectives and serves as a benchmark towards understanding on the shrinkage of mortar by adding the RHA. The study of shrinkage of mortar containing RHA had proved that the shrinkage mechanism can be reduced when RHA is added. The optimum amount of RHA to produced lowest shrinkage to the mortar is 7.5%. Besides, the usage of RHA in preparing a concrete or mortar is expected to widely use in the world as it increase the quality of concrete in economic way. Our country economy also can be raised up as the demand of RHA is higher.

From the literature review it is clear that the properties of RHA are necessary for cement replacement in order to produce more durable mortar and concrete. Shrinkage mechanism also shown clearly and deep understanding can be gathered from the information given above. Depending on the addition rate, RHA enhanced the compressive strength of concrete by up to 40% at 56 days and was superior to silica fume in this regard. Moreover, RHA reduced the rapid chloride penetrability of concrete from a moderate to a low or very low ASTM C1202 classification depending on the addition rate, which was comparable to improvements imparted by silica fume. RHA concrete was slightly more efficient than silica fume concrete in resisting surface scaling due to deicing salts [13].

This project also expected that mortars containing rice husk ash will increase in compressive strength, an improvement in absorption characteristics and a reduction in oxygen permeability. Considering the availability of rice husk worldwide, this study shows that economic technology can be used in rice-producing countries to produce a highly effective supplementary cementing material and reduce the environmental impact of uncontrolled burning of rice byproducts.

Based on the studies and test carried out, the results suggest the following conclusions for materials evaluated at a constant degree of hydration:

- i. Pozzolanic mineral admixtures reduce, but do not eliminate, the capillary porosity gradient in the interfacial zone in concrete.
- ii. The shrinkage of mortar will be reduced when RHA is added; the optimum amount of RHA that produce lowest shrinkage is 7.5%.
- iii. Pozzolanic mineral admixtures can greatly reduce the volume fraction of CH in both the interfacial zone and the bulk cement paste matrix in concrete.
- iv. Pozzolanic mineral admixtures improve the integrity of the interfacial zone by increasing the amount of the total C-S-H + cement phase present near the aggregate, relative to that present in the bulk paste.
- v. Both the size and the reactivity of the pozzolanic admixtures are important in producing a uniform microstructure of cement paste throughout the concrete, with small, highly reactive admixtures, such as RHA.

5.2 Recommendation

- The time constraint for this project should be longer in order to get the results of older ages for both shrinkage and porosity test.
- The equipments for both tests should be prepared earlier and more equipment is needed so that more experiments that relates to this topic can be performed.

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APPENDICES

APPENDIX A PREPARATION OF RHA



Rice Husk



Furnace



Rice Husk in Furnace







Grinder



APPENDIX B SHRINKAGE TEST



Mold for shrinkage test specimens



Specimens after cured



Comparator



Specimens cured in lime saturated water



Taking the comparator reading of the specimens



Mortar Mixer

APPENDIX C POROSITY TEST



Wood mold



Coring equipment



Saturation Vacuum



Specimens for porosity test



Specimens after cored



Buoyancy Balance (to obtain weight in air and water)