### University of Southern Queensland

### Faculty of Health, Engineering & Sciences

# Utilisation of Rice Husk Ash and Fly Ash as a Partial Replacement for Cement in Self-Consolidating Concrete

A dissertation submitted by

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### Abstract

The production of rice on a global scale results in significant quantities of agricultural waste. A leading constituent of this waste is rice husk. On combustion, it has been identified that the cellulose-lignin matrix of rice husk burns away leaving only a silica skeleton, along with small quantities of trace elements. In response to the continual evolution towards an environmentally aware society, there is increasing demand for the application of sustainable building materials and minimising of waste to landfill. Therefore it has been identified that the practical application of Rice Husk Ash (RHA) as a Supplementary Cementitious Material (SCM) is capable of reducing both the quantity of Portland cement used in the construction industry, and also the quantity of rice husk contributing to landfill. Previous research has identified that RHA is a suitable pozzolanic material, indicating that it is able to react with cement in the hydration reaction. Therefore it is possible to utilise RHA in a similar application to that of Fly Ash, a common pozzolanic material, to create a cost effective sustainable building material.

Application of Self-Consolidating Concrete (SCC) in the construction industry provides a potential solution to the limitations facing the use of traditional concrete in densely reinforced structural members. As SCC can flow and consolidate under its own weight, it is able to pass through extensive layers of reinforcement and settle more effectively without the need for vibration.

The aim of this research is to determine the optimum quantity of Rice Husk Ash (RHA) and Fly Ash that can be incorporated as a supplementary cementitious material for partial replacement of Portland cement in a self-consolidating concrete. This will be conducted through testing of the Compressive and Flexural strength of 6 test samples that differ in amounts of RHA and Fly Ash ranging from 20% to 50% total replacement percentage.

The outcomes identified suggest that RHA burnt under the proposed experimental conditions has the potential to be applied as a pozzolanic material. Likely applications include any works requiring a low strength concrete with high flowability. Factors limiting the use of SCC in construction were also identified, with one key issue being the presence of air voids indicating potential for premature failure. Further development of this material has the potential to mitigate this issue.

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## Chapter 1

## Introduction

The following chapter provides an introduction to Rice Husk Ash (RHA) and how it can be effectively incorporated as a supplementary cementitious material (SCM) for practical use in self-consolidating concrete (SCC). It also further analyses the background aims of this project and identifies specific objectives that will be the central focus of this research.

#### 1.1 Project Background

Rice is currently the second most widely produced agricultural product globally, and with a demand of 470 million tonnes per year it generates a significant amount of agricultural waste. In response to the continual evolution towards an environmentally aware society, there is demand for the reduction of non-renewable resources and minimising of waste to landfill. One of the major challenges that negatively affect the rice industry focuses on the appropriate use or disposal of agricultural by-products (Moraes, et al, 2014).

A main constituent of the agricultural waste resulting from rice production develops during the milling process in the form of rice husk, a shell that grows around the grain during growth. The husk is a raw material considered problematic for reuse due to many unfavourable properties. These include characteristics such as high abrasion, low nutritional properties, very low density, and resistance to degradation which in turn results in a large volume of waste. Because of the materials low nutritional properties it is deemed to be unfit for animal feed, unlike many other agricultural waste products.

Effective management of this waste has become an increasingly significant issue. This is primarily apparent in developing countries where due to high disposal costs the husk is deposited along roads, in agricultural soil, and near water sources, which increases its potential for pollution (Lim, et al. 2012). Currently the most effective use for rice husk is fuel for biomass power generation. This application not only reduces the negative environmental impact associated with the disposal of this waste, but also contributes to the reduction of dependencies on non-renewable energy resources.

The controlled burning of rice husk to produce energy results in the production of RHA. Disposal of this ash directly into the environment is considered detrimental due to the presence of residual carbon, fine particulate matter and high silica content. This can cause soil acidification and changes in soil and water chemistry (Moraes, et al. 2014). As such, it is imperative that an environmentally beneficial method of utilising this waste product is determined.

In addition to the above, there is a growing environmental awareness throughout society as a whole. The pollution and health hazards associated with the concrete and cement industries are coming under increasing scrutiny due to their adverse impact on the environment. RHA has been found to contain a high silica content which can potentially be substituted as a raw material in cement manufacturing (Khan, et al, 2012). As illustrated in research conducted by Meryman (2009) the most effective way of reducing the environmental impact of the construction industry is by replacing a large amount of Portland cement with a carbon neutral alternative. The utilisation of RHA in concrete construction will not only reduce its use in landfills and the associated environmental impact but also reduce the carbon footprint of concrete manufacturing through minimising the quantity of cement required (Gursel, et. al, 2015).

A study conducted by Komnitsas (2011) identified that with continuous increasing of population densities at an exponential rate it is necessary to focus on creating sustainable developments. This would take into account the energy consumption, waste production and Green House Gas emissions that are a by-product of the construction industry. The study concluded that in order to promote sustainable development it was essential to focus on strategies that improve the management of waste and resources.

The utilisation of self-consolidating concrete (SCC) provides a more versatile construction material than a regular concrete mix. Because SCC can flow and consolidate under its own weight, it is able to pass through extensive layers of reinforcement and settle more effectively without the need for vibration (Tahn Le, 2016). Through development of a SCC mix design and incorporation of additives to the mix it is possible to create a product that maintains the necessary strength requirements whilst remaining cohesive enough to be handled without segregation or bleeding (Shi, 2015). This also results in a faster construction time whilst maintaining the quality and strength properties of the finished product. SCC, particularly with the inclusion of Supplementary Cementitious Materials (SCM), is a relatively new innovation within the construction

sector, and as such requires further research and development to establish a universally accepted mix design methodology.

### 1.2 Research Aims

The application of RHA in the construction industry is not currently practiced within Australia. This is largely due to the extensively researched and accepted methodology of incorporating Fly Ash (FA) as a pozzolanic material. However in recent years there has been a decline in the availability of FA due to factors such as general economic stagnation, decreasing coal use and regulatory uncertainties which inadvertently support the growth and application of RHA (Gursel, 2015). Additionally, due to the negative environmental impact associated with burning of coal for energy, it is important for Australia to consider not only alternative construction material options but also alternative energy production methods to create a sustainable environment for the future.

On this basis, the aim of the project is to provide evidence supporting the utilisation of RHA as a partial replacement of Portland cement in concrete mix design for application in the construction industry. Because FA is widely accepted and utilised as a Pozzolanic material in replacement percentages ranging as high as 50% it was deemed appropriate to incorporate this material into the initial mix design parameters of the research testing procedure and SCC mix design.

In addition to this, SCC is also an underutilised resource within the Australian construction industry, with the primary application being for pre-cast structural elements. This is largely due to the undeveloped nature of this material, however with increasing urbanisation and population density it is expected that an increasing demand for high load bearing infrastructure will require a form of concrete capable of adapting to a multitude of uses and design requirements.

Through determining whether RHA can be incorporated into a SCC mix design this research aims to provide suitable evidence in support of utilising RHA more widely as a construction material and more importantly develop a new method of concrete construction with potential to not only lower costs and the environmental impact of current methods but also utilise the Pozzolanic effects of RHA to increase the characteristics and physical properties of SCC.

In summary, this research report has identified the potential to capitalise on an agricultural waste product in an industry that would significantly benefit from the addition of a new cost effective SCM. The implication of utilising this product in SCC also identified a knowledge gap in current research, which was to develop an appropriate mix design that incorporated higher percentages of both RHA and FA. Through analysis of similar research it is evident that both these materials have the potential to contribute to concrete strength through supplementing the chemical and physical characteristics of cement.

### 1.3 Research Objectives

This study shall utilise agricultural waste produced within Australia to build upon comparable research developed over seas, similar to the study conducted by Rodriguez de Sensale (2006). In this instance, although it is expected that a significant increase in SCM will negatively impact the strength of concrete, it is also anticipated that suitable applications within the construction industry can be identified. A control specimen containing 20% FA is prepared along with a number of specimens with 5% increasing percentages of RHA starting at 10%. The specimens are exposed to compressive and flexural strength tests whereby the strength characteristics of each sample is determined and collated for analysis. Methodology discussed further in Chapter 3.

The following objectives will be addressed to an extensive degree in order to provide justification for the incorporation of RHA as a construction material here in Australia:

- Research background information on the physical and chemical properties of RHA that contribute to the properties of concrete
- Determine the most effective method of burning Rice Husk to develop a suitable RHA product, ideally with an Amorphous Silica content greater than 90%
- Formulate an appropriate SCC mix design through analysis of previous research methods and current global standards
- Determine the passing requirements for fresh SCC
- Conduct compressive and flexural strength testing in accordance with AS1012

- Determine the most effective RHA blended concrete mix composition based on compressive and flexural strength
- Compare this data with previous research reports which will assist in providing credibility for experimental results
- Provide a cost analysis comparing a Portland cement mix design to the most effective experimental mix proportion and provide suitable uses for each mix design within the construction industry

Refer to the project specification located in Appendix A for further details.

### 1.4 Conclusion

This research is intended to provide a comprehensive review of RHA and SCC in order to develop a suitable end product with the capacity to be utilized within Australia as a sustainable building material. Results are obtained through application of an appropriate SCC mix design, development of various samples with increasing amounts of RHA and application of destructive testing on each SCC mix to determine their compressive and flexural strength properties. Also included in this research is an in-depth experimental analysis of RHA and a summary of the specific material properties required to achieve a suitable SCM.

The most likely outcome of this project is that there will be a quantitative result which identifies the optimum percentage of RHA and FA that can be incorporated into a mix design. Furthermore, a direct relation will be identified between an increase in RHA and an associated decrease in strength. Economic analysis of the mix designs will show a cost comparison of the utilisation of these materials. These results will further support justification primarily for the use of RHA in the construction sector, and furthermore support the further acceptance of SCC.

## Chapter 2

## Literature Review

### 2.1 Chapter Overview

The following chapter provides a review of existing academic research undertaken in the field of both Rice Husk Ash development and Self-consolidating Concrete. Information discussed below provides the necessary background detail to accompany following methodology and data analysis. The information analysed was obtained through review of published works, SCC guidelines and specifications, and relevant technical papers on this field of research. The concept of RHA as a SCM is explained, including the mechanisms that contribute to its pozzolanic activity. Also analysed is the ideal combustion conditions to produce suitable RHA and the optimum replacement percentages based on previous research. The methods of elemental analysis for RHA are also identified and a general understanding of SCC is provided, which includes a review of the relevant guidelines required for the production of this concrete type.

The purpose of a literature review is to attain a thorough understanding of previous research in order to not only determine opportunities for research development but also attain the key understanding required to adequately formulate tangible results from the following research methodology.

### 2.2 Rice Husk Ash

Rice Husk Ash is a fine agricultural waste material that results from the burning of Rice Husk, either through uncontrolled and controlled burning, fluidization or gasification. Rice husk contributes to approximately 20% of dried rice paddy weight, and the ash resulting from burning rice husk accounts for approximately 20% of the husk weight (Meryman, 2009). One of the primary consequences of commercial food production is the

generation of high volumes of agricultural waste. When this waste is poorly managed it can result in extensive environmental and health risks (Moraes, 2015). Figure 2.1 provides a visual representation of rice husk in raw condition, and also its appearance following combustion and grinding. As shown below, the grinding process transforms what is essentially a rice husk ash skeleton comprising primarily of silica into a fine homogenous material, which is much more effective as a SCM.



Figure 2.1 - Development of Rice Husk to Ash (Aprianti et al. 2014)

As awareness of global CO2 emissions increase due to the production of Portland cement, it is necessary to determine sustainable methods of construction. It has been extensively criticized that the production of cement consumes high amounts of energy, is costly, emits large amounts of greenhouse gases and depletes natural resources. More accurately with reference to a study conducted by Khan (2011), it was reported that the production of 1 ton of cement required approximately 1.7 tons of raw materials and produced roughly 1 ton of CO<sub>2</sub>.

Rice husk is considered problematic to recycle commercially in any significant quantity due to its negative characteristics. This primarily includes its low nutritional properties, low density and resistance to degradation. However as the husk contains a very high calorific value, it is predominantly utilised as a renewable biomass source for power generation in nearby mills. The consumption of rice husk for power generation not only reduces the negative environmental impacts resulting from its disposal, but also contributes to the reduction in dependency on non-renewable resources (Moraes, 2015).

In addition to the production of energy, suitable controlled burning or gasification of rice husk produces an ash with considerably high amorphous silica  $(SiO_2)$  content with proportions usually ranging between 85-95% (Meryman, 2009). Currently a vast proportion of RHA goes unused, further contributing to environmental contamination in

areas where it is discarded. Through provision of a large scale application Sua-Iam (2014) postulated that the practice of dumping RHA would be essentially eliminated, and  $CO_2$  emissions resulting from cement production would be reduced.

#### 2.2.1 Cementitious Contribution of Pozzolans

A Pozzolan is a SCM, which in itself possesses little or no cementing property, but in the presence of cement and water chemically reacts with calcium hydroxide (hydrated lime) to form compounds possessing cementitious properties (Agarwal, SK, 2006). It has been well established that the incorporation of pozzolanic materials in partial replacement cement in concrete significantly increased the materials durability and strength characteristics (Meryman, H, 2009). Furthermore, although the inclusion of pozzolans in concrete generally reduces the materials early strength properties, there are significant long term benefits to its application. As identified by Chindaprasirt (2007) these include higher strength at later stages, sulfate resistance, acid resistance, low porosity and an increase in the concretes durability.

Research conducted by Jamil (2014) discussed the principles behind pozzolanic reactions. It was identified that initially calcium silicates and aluminates in cement react with water to produce Calcium-Silicate-Hydrated (C-S-H) gel. This gel is predominantly responsible for the hardening of concrete, as it forms the component that binds all aggregates together. Following this initial reaction there is an excess amount of Calcium Hydroxide (Ca(OH)<sub>2</sub>). This remaining Ca(OH)<sub>2</sub> reacts with the silica and alumina components found in pozzolanic materials in the presence of water to produce additional quantities of C-S-H gel (Jamil, 2014).

Due to its high amorphous silica content, it is determined that RHA is an effective Pozzolanic material for the production of concrete. It has also been noted that the Pozzolanic reactivity of RHA can be improved by grinding to create a finer particle size (Jamil, M, et al. 2013). In a study conducted by M. Jamil et al. (2013) it was determined that the optimised RHA which has been produced through controlled burning could be used as a pozzolanic material in cement and concrete, which would result in the reduction of the environmental impact associated with the manufacture of cement and disposal of waste materials, a key beneficial factor supporting the use of RHA in concrete.

In order for RHA to react with Portland cement effectively it must be burnt at appropriate conditions to develop reactive amorphous silica, this is further analysed in section 2.2.2. The amorphous silica, which comprises roughly 90% of the total RHA material content, reacts with the calcium hydroxide as discussed previously to produce C-S-H gel. The microstructure created with C-S-H gel reduces the porosity of concrete leading to an increase in compressive strength (Chopra, D, 2015).

As RHA is a porous material, water added to the concrete mix will be absorbed by the ash particles during the mixing process in small quantities. It was determined in a study by Ki-Bong Park (2015) that due to the self-desiccation of hydrating blended cement, the water absorbed by the RHA will be later released. This process further contributes to the overall hydration process of the SCC over time. Research conducted by Chopra (2015) identified that although SCC mixes containing higher percentages of RHA showed lower compressive strength initially in comparison to a control specimen with no SCM, there was an increased rate in the development of strength after a period of 60 days. This can largely be attributed to an increase in the rate of pozzolanic reactions from RHA over time. In order to determine whether the rice husk ash will be appropriate as a supplementary cementitious material it is vital that a sample of the product be analysed to identify all properties. Further details regarding the tests that are required for this can be found in section 2.3.

#### 2.2.2 Ideal Combustion Conditions

In order to successfully produce an adequate pozzolanic material from existing rice husk it is essential to understand all contributing factors that affect the burning requirements and resulting end product. There are external influences that affect the quality of RHA that are somewhat uncontrollable, such as differences in paddy type, crop year, climate and geographical conditions (Khan, 2012). However these factors do not contribute significantly to the end product. The most significant influence on the final quality of RHA as a pozzolan is burning temperature. On combustion, the cellulose-lignin matrix of rice husk burns away and only a silica skeleton remains, along with a small amount of carbon and other trance elements. Less significant contributing factors include particle size, surface area and carbon content, however these can also be mitigated effectively.

A study conducted by Xiong (2009) provided an initial insight into the effects burning temperature has on the final quality and characteristics of Rice Husk Ash. In this study

the rice husk was burnt at a firing rate of  $10^{\circ}$ /minute for 2 hours to maximum temperatures of 400°C through to 900°C. Through utilisation of an X-ray Diffraction (XRD) test it was possible to ascertain the amount of amorphous silica (SiO<sub>2</sub>) that transferred to crystalline form, and proportion of silica in the sample by analysing the level and location of peaks on the final XRD output. In this study it was determined that temperatures above 750°C produced higher levels of crystalline silica and therefore further burning should not be conducted above this temperature, this result was also attained in research conducted by Fernandes (2016). Crystalline silica is a non-reactive material and therefore it is essential not to burn the rice husk at levels high enough to obtain this in the final product. This study also analysed the use of a Hydrogen Chloride (HCl) solution to soak the rice husk as a pre-firing treatment measure, which resulted in the end product having a slightly higher percentage of SiO<sub>2</sub> with minimal trace elements.

This observation was further confirmed in a study by Abu Bakar (2015), which analysed the combustion of rice husk over an identical temperature range and time. The results of this study concluded that the ideal firing condition that created the highest percentage of SiO<sub>2</sub> without transition to the crystalline form was to burn at 600°C for 2 hours. This method achieved a silica content of 95%. This study also included the addition of hydrochloric and sulfuric acid leaching of the husk however the difference in the final quality was not significant enough to warrant this method. Overall there was an increase of 4% in SiO<sub>2</sub> content, with minimal differences in particle size, surface area and pore diameter. An XRD analysis was conducted on the samples to confirm whether the silica formed for each sample was predominantly SiO<sub>2</sub>.

Abu Bakar (2015) also conducted an X-Ray Fluorescence (XRF) analysis of the final material to attain the overall chemical composition and percentage of each major component. This test was supplemented with a Scanning Electron Microscopy (SEM) analysis to provide a definitive understanding of the materials overall composition. In this particular test the resulting purity reached 95%, with small amounts of metallic impurities present. It was noted that the level of residual Carbon present in the ash was observable by colour analysis, whereby higher percentages of carbon would result in a much darker product and lower percentages result in a light grey colour. As illustrated in Table 2.1 below, the effects of temperature between 600 and 700°C is minimal, and with an increased occurrence of crystalline silica above 700°C this study concluded that the optimum burning condition for this particular sample was 650°C for 60 minutes.

Table 2.1: XRF Results at various Temperatures (Ramezanianpour, 2009)

						El	ements (ª	%)					
		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	SO3	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	$P_2O_5$	TiO <sub>2</sub>	LOI	
	550°C	60min	75.22	0.05	0.14	0.57	0.37	0.36	0.07	1.47	0.51	0.01	21.01
_		90min	80.76	0.03	0.09	0.66	0.23	0.43	0.05	1.72	0.79	0.01	14.95
tior	600°C	60min	80.55	0.02	0.24	0.59	0.34	0.39	0.06	1.65	0.44	0.02	15.33
ura		90min	85.60	0.06	0.15	0.87	0.22	0.41	0.06	1.53	0.48	0.02	9.81
e di		30min	76.21	0.08	0.22	0.86	0.21	0.31	0.08	1.69	0.52	0.01	19.53
re and tim	650°C	60min	89.61	0.04	0.22	0.91	0.15	0.42	0.07	1.58	0.41	0.02	5.91
		90min	90.21	0.06	0.27	0.85	0.25	0.49	0.08	1.51	0.56	0.02	5.48
	a 700°C	30min	81.35	0.09	0.15	0.77	0.18	0.33	0.08	1.72	0.53	0.02	14.53
atu		60min	89.93	0.06	0.11	0.88	0.14	0.39	0.09	1.48	0.55	0.02	6.01
per		90min	92.19	0.09	0.10	0.71	0.09	0.41	0.05	1.64	0.41	0.01	4.14
tem	750°C	30min	84.22	0.09	0.18	0.54	0.17	0.38	0.06	1.35	0.61	0.02	12.09
		60min	93.11	0.08	0.27	0.67	0.11	0.44	0.06	1.69	0.63	0.02	2.67
	1100°C	a few min	95.31	0.04	0.11	0.78	0.11	0.41	0.09	1.61	0.45	0.01	0.84
Cement type I		21.50	3.68	2.76	61.5	2.5	4.8	0.12	0.95	0.23	0.04	1.35	

Similar studies by Tahn Le (2016) and Ramexanianpour (2009) further confirmed the optimum temperature range of 600°C and also established that grinding of the final RHA product using a Los Angeles machine for 30 minutes further contributed to the materials physical properties and improved its performance. Meryman (2009) also noted that in order to improve the reactivity of RHA and its ability to act as a pozzolanic material, the raw ash should be ground to a fine particle size. This practice is confirmed by Jamil (2013) who confirmed the previous observation that the grinding of partially crystalline RHA in a Los Angeles machine using steel rods will produce an optimum quality RHA. This practice should be taken into consideration when acquiring the material, as all three of the studies mentioned above have determined that this is a contributing factor to the effectiveness of RHA.

Upon review of the above research documents it was determined that overall the most appropriate firing method was to burn the rice husk in a kiln at a firing rate of 200°C per hour and hold at 600°C for one hour upon reaching this point. Further details regarding the results for this firing method will be available in the results section of the report.

#### 2.2.3 Optimum Replacement Percentage

In order to achieve an overall reliable experimental method it is essential to critically review outcomes from previous studies and determine where there are suitable research opportunities and also which methods were most effective. Comparison of three experimental designs provided an overall representation of this. This included a review of the compressive strength of a 10% and 20% concrete mix up to 91days (Rodriguez De Sensale, 2006), the use of both RHA and admixture to determine the 7 and 28 day

compressive strength of 5% and 15% mix designs (Khatri, 2014), and a study of both the compressive and flexural strength of RHA mix designs ranging between 5% and 15% (Padma Rao, et. al, 2014). An evaluation of all three experiments produced results that suggested both advantages and disadvantages of the incorporation of RHA in concrete mix design.

Rodriguez De Sensale (2006) determined that the inclusion of higher percentages of RHA resulted in a lower early age strength, however at higher ages (91 days) concrete with a high percentage of RHA achieved the highest values of compressive strength overall. This reiterates that the pozzolanic activity of RHA continues to enhance the performance of concrete over time. Table 2.2 illustrates the results from this research. In the table it is evident that the incorporation of RHA up to 20% provides a higher compressive strength, in addition Table 2.2 also compares various W/C ratios to identify their associated compressive strength characteristics.

w/(c + RHA)	RHA		f <sub>c</sub> (MPa)			
	Туре	%	7d	28d	91d	
0.32		0	48.4	55.5	60.6	
	UY	10	51.1	60.4	64.3	
		20	44.3	54.8	62.7	
	USA	10	39.5	51.4	64.5	
		20	30.5	47.4	68.5	
0.40		0	35.8	42.3	45.6	
	UY	10	41.1	50.4	54.9	
		20	27.9	40.7	51.4	
	USA	10	29.7	40.8	51.5	
		20	23.6	39.4	57.3	
0.50		0	24.6	32.9	35.9	
	UY	10	24.1	31.5	35.5	
		20	24.9	34.9	37.9	
	USA	10	22.7	34.5	44.4	
		20	20.8	35.9	52.9	

Table 2.2: Compressive Strength of RHA Samples (Rodriguez de Sensale, 2006)

Results attained by Rodriguez de Sensale (2006) were confirmed in a similar study conducted by Khatri (2014) which concluded that the input of 15% RHA as a pozzolanic material resulted in a higher compressive strength at 7 and 28 days. Although this result identifies the suitability of RHA inclusion in concrete mix design, research discussed by Padma Rao et.al (2014) identified that the flexural strength of concrete samples is negatively affected by the incorporation of RHA. This study determined that with increase in percentage replacement of RHA the flexural strength subsequently decreases.

More specifically it was determined that as the age of the concrete advances, there is a significant decrease in the flexural strength of the RHA samples.

Through analysis of the above reports it was determined that in order to achieve an overall understanding of the samples properties it is critical for this research project to analyse both the compressive and flexural strengths of various RHA concrete samples. Taking into account all three similar research examples it is understood that the most effective experimental analysis will involve the testing of two of each sample to account for unforeseen errors, testing will be conducted after a 7 and 28 day curing time and also the range of samples to be used will extend from 0% RHA to 30%, as previous studies suggest the most effective mix design falls between 10 and 20% RHA replacement.

#### 2.2.4 Application in the Construction Industry

The building and construction industry is a significant contributor to the global increase of carbon emissions and waste material production to landfill. The manufacture of cement alone accounts for 10% of global CO<sub>2</sub> emissions (Zhong & Wu, 2015). Through limiting the amount of Ordinary Portland Cement (OPC) used in concrete construction and focusing on waste management and recycling practices, it is possible to minimise the construction industry's environmental impact. The most effective means of reducing the CO<sub>2</sub> output of construction is to replace large quantities of OPC with a carbon neutral alternative. RHA is a readily available sustainable resource that is considered carbon neutral, as carbon emitted in the burning of rice husk has been identified as equal to the amount absorbed by new crops (Meryman, 2009).

Due to RHA's beneficial properties as a Pozzolanic material, its application in the construction industry has the potential to complement its utilisation as a supplementary cementitious material. In doing so this application would also assists in alleviating a proportion of the current landfill and pollution control issues resulting from increasing infrastructure development. A study conducted by Huntzinger (2009) confirmed that in order to reduce global CO<sub>2</sub> emissions from the cement manufacturing process it would be a suitable option to replace cement with industrial waste that contains Pozzolanic properties, which would also allow for a reduction in the costs associated with raw materials.

Zhong et al. (2015) conducted a lifecycle analysis of RHA's application in the building sector. This study determined that an understanding of the constructability performance of economic sustainable materials such as RHA is essential in order for construction and infrastructure to evolve towards being a sustainable industry. In terms of economic requirements, over an extended period of time it can be more lucrative and sustainable to invest in technology that converts wastes material such as rice husk into a value-added product (Moraes, et al. 2014). Meryman (2009) identified that the development of a market for rice husk would provide rice farmers with an additional incentive against crop burning and waste dumping, both of which result in extensive environmental damage.

#### 2.3 Elemental Analysis of RHA

The primary purpose for conducting elemental analysis of a sample is to determine its overall structure, particle size and composition of various compounds. This analysis can either be quantitative, where the total amount of each element is determined; or qualitative, in which all elements in a sample are identified. By applying qualitative methods of elemental analysis the total composition of each element within a RHA sample can be identified. There are three key methods of analysis applied in order to determine the adequacy of a sample; these include X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), and X-ray Fluorescence (XRF). A collaboration of these methods provides both an initial representation of the samples composition, and also further analyses the quantities of each element.

#### 2.3.1 X-ray Diffraction Analysis (XRD)

XRD analysis provides a valuable basis in determining the quality of RHA following the required firing procedure. XRD is a rapid analytical technique that is primarily used for phase identification of materials in crystalline form. This method of examination determines the crystalline nature of a sample by measuring diffraction of x-rays from the planes of atoms of each component. XRD analysis is primarily utilised in this instance to determine the existence of amorphous silica (SiO<sub>2</sub>), which is evident through the existence of a broad peak with an equivalent Bragg angle at  $2\Theta = 21.8^{\circ}$  (Music, 2011).



Figure 2.2: XRD pattern of Amorphous SiO2 (Music, 2011).

The maximum value, equivalent to roughly 22°, was determined by Music (2011) as a suitable reference point in determining amorphous SiO<sub>2</sub> particles. The accuracy of this result was subsequently confirmed in similar studies analysing the components of RHA. Abu Bakar (2016) most recently achieved a result of  $2\Theta = 22^{\circ}$  in a study analysing the impacts of firing temperature on amorphous SiO<sub>2</sub>. Results from this study, with reference to Figure 2.2, illustrate the existence of sharp peaks with increasing temperature. This indicates that existing SiO<sub>2</sub> has transitioned from amorphous state to crystalline.



Figure 2.3: RHA XRD Pattern at Various Temperatures (Abu Bakar, 2016)

The above XRD analysis for RHA illustrates the existence of a primary proportion of  $SiO_2$ , which is consistent with current research on the material. Further analysis of the results provided by Abu Bakar (2016) confirm that with increasing temperature the XRD

peak increases in intensity due to the increased existence of crystalline particles. By comparison, Figure 2.4 illustrates an XRD analysis for FA which contains oxide compounds such as Aluminium and Iron as well as Silica.



Figure 2.4: Fly Ash XRD pattern (Criado et al. 2007)

Through comparison of Figures 2.3 and 2.4 it is evident the overcall composition of FA varies considerably with that of RHA. Although both results identify critical phases of  $SiO_2$ , the sharp peaks displayed in the FA XRD pattern suggest the existence of crystalline particles in greater percentages than that of the RHA pattern. Although FA contains a larger range of compounds, both materials are able to act as pozzolans in the presence of a hydration reaction.

#### 2.3.2 Scanning Electron Microscope (SEM) Analysis

A SEM produces a high resolution, high depth of field surface image of a sample through application of a focused beam of electrons. This procedure is most effective for analysis of samples with a particle size less than 100 micron. In order to accurately determine the suitability of a RHA for application as a pozzolan it is essential to take into account factors such as particle size, roughness and porosity (Meryman, 2009). In addition to retaining an image of the particle structure, SEM analysis can utilise an Energy Dispersive X-ray (EDS) analyser to identify the existence of various base elements, including Silica and Oxides, which are the main components of amorphous SiO<sub>2</sub> (Music, 2011). Although this is not a necessary test concerning the elemental analysis of RHA it

does provide further evidence confirming the existence of specific major elements within a sample. With reference to Figure 2.5, Sua-Iam (2014) identified that OPC and RHA both have an irregular shaped particle size, whilst FA particles are predominantly rounded. This plays a significant role in the effect each material has on an overall concrete mix.



Figure 2.5: SEM analysis of (a) OPC, (b) Fly Ash & (c) RHA, (Sua-Iam, 2014)

Sua-Iam (2014) provided further research indicating that the rounded shape of FA particles directly affect the workability of a SCC mix by reducing the friction between individual particles in a mix. When the replacement percentage of FA is increased, the workability subsequently increases also. In comparison, when OPC and RHA particles are added to a SCC mix their angular shape results in greater friction between each particle. Thus the angular shape and higher porosity of RHA results in not only increased water retention, but also less free water and decreased flowing ability of the SCC. The potential consequence of this is that the SCC mixture may display some segregation and decreased workability. As shown in the above SEM analysis, the particle size of RHA is much larger than that of FA and OPC. As a large particle size of RHA is not suitable for a pozzolanic material, it is essential the ash is ground to a finer, homogenous product. Acceptance of the final powder can be achieved through passing the powder through a fine sieve, preferably less than 75 micron.

Research published by Tahn Le (2016) identified many of the same particle characteristics as those detailed above; however this study also concluded that the addition of RHA in SCC would in turn result in a significant increase in plastic viscosity and segregation resistance. This was attributed as a result of suction forces leading to cluster formation, or more accurately an increase in interparticular bonds. As there is an identified variance in resulting SCC characteristics between these two research papers, it is essential that further experimental work provides evidence in order to identify the correct outcome under the proposed experimental conditions.

#### 2.3.3 X-ray Fluorescence (XRF) Spectrometry

XRF analysis provides a detailed output of the chemical composition of a material. This occurs through measuring the fluorescent X-ray emitted from a sample following exposure to a primary X-ray. Each element within a sample produces a unique set of results. This process is carried out in order to identify and quantify the major and minor elements that are present in a sample (Ramezanianpour, 2009). In this instance not only are components such as Silica and Oxides identified, but also various trace elements that are also present, typically through environmental conditions specific to an area. Refer to Table 2.3 for a further comparison between the elemental analyses of RHA, FA and OPC. This table identifies the chemical composition and physical properties of each material to provide further experimental details pertaining to differences in their cementitious properties. An optimum RHA sample would display a very similar chemical composition to that of the sample below, noted with a Silica (SiO<sub>2</sub>) content above 90%, few trace elements and a relatively low Loss on Ignition.

Chemical	Mass percent composition					
	OPC	FA		RHA		
Calcium oxide (CaO)	68.48	13.	99	0.76		
Silicon dioxide (SiO <sub>2</sub> )	16.39	40.	51	93.44		
Aluminium oxide (Al <sub>2</sub> O <sub>3</sub> )	3.85	21.	52	0.21		
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> )	3.48	13.4	41	0.18		
Magnesium oxide (MgO)	0.64	2.10		0.43		
Sulphur trioxide (SO3)	4.00	4.00		0.16		
Sodium oxide (Na <sub>2</sub> O)	oxide (Na <sub>2</sub> O) 0.06 1.4		44	0.05		
Potassium oxide (K <sub>2</sub> O)	0.52	2,20		1.98		
Loss on Ignition (LOI)	1.70	0.49		1.27		
Physical property		Value				
Specific gravity		3.15	2.22	2.18		
Specific surface area, Brunauer-Emmett-Teller (BET) method (m <sup>2</sup> /kg)		610	480	370		

Table 2.3: Properties of OPC, Fly Ash & RHA (Sua-Iam, 2014)

The provision of accurate experimental analysis data has the potential to provide defined quality measures. These quality measures may be utilised for mass production of a RHA product for use as a construction material. Through continued research development a quantitative measure of RHA quality for application as a pozzolanic material can be achieved.

### 2.4 Self-Consolidating Concrete

Self-Consolidating Concrete (SCC) is a relatively recent innovation in concrete technology, first developed at the University of Tokyo, Japan in 1986 by Okamura (Shi, 2015). This type of concrete provides a material which can be placed and consolidated under its own weight. This allows SCC to flow through and fill the gaps in densely packed reinforcement and corners of moulds without the need for compaction and vibration during placement whilst providing excellent resistance to segregation or bleeding (Su, 2001). The resulting hardened SCC has the same durability and engineering properties as traditional concrete, and due to its fluidity and segregation resistance it provides a superior finish. Defects are a particular issue in precast panels and in-situ concrete walls due to difficulty in adequate vibration. The development of structural defects often impacts on project completion in time and under budget. The ability to utilise SCC in this application has the potential to negate this issue.

Through utilisation of SCC in construction it is possible to capitalise on numerous advantages. These include; a decrease in construction time and labour costs, reduction in noise pollution, improving the filling capacity of congested structural members, eliminates the need for vibration, reduced risk of defects resulting from segregation, decreased permeability and improved durability (Shi, 2015). Furthermore due to SCC's low water / cement ratio it has the potential to develop high early strength, shorter formwork stripping times and therefore faster development of structural members.

Although the construction sector has seen an increase in the use of SCC over the past decade, predominantly in the manufacture of large precast members, there is no systematic design standard or specification pertaining to the mix proportion requirements (Guru Jawahar, J et.al, 2012). As such there is a multitude of varying design methods reported in existing literature, most with only general guidelines and mix proportions. A thorough review of existing methods is essential in order to determine the most appropriate method for the inclusion of RHA. When identifying a preferred method accurate repetition will be a determining factor. Analysis of existing literature conducted by Shi (2015) determined that there are five basic design methods of determining an SCC mix design, based on a review of 19 academic reports. These include the empirical design method, compressive strength method, closes aggregate packing method, statistical factorial modelling and rheology of paste modelling. Of these procedures it was determined that the most suitable was the compressive strength method.

#### 2.4.1 Compressive Strength Mix Design Method

The method of proportioning SCC based on compressive strength requirements was developed by Ghazi (2010). This method has been generated to allow for a mix design composition relevant to any specified compressive strength, in comparison alternate current methods emphasise on the fulfilment of fresh concrete properties over strength. In addition this proposed method adopts guidelines discussed in the European Guideline for SCC (EFNARC, 2005), which has been determined as an essential research for experimental development of SCC, discussed further in section 2.4.2.

The compressive strength method, analysed by Shi (2015), provides a clear, comprehendible methodology that specified each material quantity required and minimised the need for multiple trial mixes. This method also accounted for any contribution of pozzolanic materials and allowed for direct adaption of all components for varying levels of SCM's (Ghazi, 2010). Overall, an ideal mix design should be considered as suitable for a wide range of applications, with a strong robustness for variables in raw materials and an emphasis on sustainability and costs (Shi, 2015).

#### 2.4.2 European Guidelines for SCC

The requirements for testing of fresh SCC differ from that of ordinary concrete. SCC is not common in Australia and therefore testing methods cannot be found in the Australian Standards. As such this section of the project will refer to the European Guidelines for Self-Compacting Concrete (EFNARC, 2005). This document is widely accepted as a general guideline for implementation of SCC, and is most recently intended to facilitate standardisation at a European level. This guideline identifies many of the technical properties and requirements that are required to procure a suitable SCC mix design. As such it is referred to in numerous research papers regarding SCC mix design methods, including the primary technical paper previously referred to for the compressive strength method (Ghazi, 2010), and an overall review conducted by Shi (2015) pertaining to the various SCC mix design methods.

The EFNARC guidelines (2005) identify that the filling ability and stability of SCC in fresh concrete state are defined by four key characteristics. These characteristics identify whether a concrete mix is acceptable for use, in a similar way to that of the slump test for

ordinary concrete. The four key parameters are flowability, viscosity, passing ability and resistance to segregation. Further details regarding these testing methods are provided in Chapter 3. Guidelines regarding the constituent materials required for SCC are also provided in this document, however the materials required do not differ largely in type from that of traditional concrete.

In order to develop concrete that is self-consolidating in nature the material constituents vary in percentage, primarily this includes a lower percentage of coarse aggregate and higher quantities of fines, cementitious paste and superplasticiser. As mentioned previously there is no standard method for SCC mix design, with many academic institutions and SCC suppliers developing their own proportioning methods. Although the information provided in the EFNARC guidelines (2005) are intended only as technical advice, the proportions provided in Table 2.4 below have been provided as an indication of typical ranges of constituent materials.

Constituent	Typical range by mass (kg/m³)	Typical range by volume (litres/m³)		
Powder	380 - 600			
Paste		300 - 380		
Water	150 - 210	150 - 210		
Coarse aggregate	750 - 1000	270 - 360		
Fine aggregate (sand)	Content balances the volume of th	e other constituents, typica		
	48 – 55% of total aggregate weight.			
Water/Powder ratio by Vol		0.85 - 1.10		

Table 2.4: Typical SCC mix design composition (EFNARC, 2005)

Future experimental SCC mix design proportions developed in this report will refer back to Table 2.4 for confirmation of its general acceptance. In the event a trial mix that meets the above proportions does not eventuate to a mix design that passes the required fresh concrete testing a trouble shooting guide is provided in the 2002 edition of the EFNARC guidelines. Further guidance in the event of unsatisfactory performance is also provided in Annex C of the EFNARC guideline (2005). Overall, through utilising the advice conveyed in this document it is possible to develop a high quality end product with a significantly reduced risk of defects. The checklists provided specify methods of resolving material quality issues identified at each stage of development.

### 2.5 Chapter Summary

Understanding of previous research has identified specific challenges that will impact the final quality of SCC. The most significant of these being the necessity to develop RHA with amorphous silica content above 85% as a minimum. This ensures the pozzolanic material will provide sufficient C-S-H gel to bond with aggregates and effectively contribute to the hardness and associated physical properties of SCC. Following the firing of rice husk it is therefore imperative that experimental analysis be conducted on the RHA to ensure it possesses a negligible quantity of crystalline particles, high percentage of amorphous  $SiO_2$  and that the particle size, roughness and porosity is acceptable.

In addition attaining an acceptable RHA product, the mix design composition and methodology for SCC is to be based on the compressive strength method (Ghazi, 2010). Through comparison of multiple mix design methods it was determined that this methodology provided the most clear and precise procedure in order to obtain specific quantities (Shi, 2015). As there are multiple published design methods final mix design requires comparison with existing proportions, and analysis utilising the specifications and guidelines provided in the European Guidelines for SCC (EFNARC, 2005).

Inclusion of RHA as a pozzolanic material in SCC has the potential to provide significant advances in the construction industry, and in addition provides an alternate source of pozzolanic material. Inclusion of RHA allows for a reduction in OPC and opportunity to reduce construction costs and utilise sustainable waste material. Further advances in the utilisation of SCC in construction provide significant opportunities for more complex structural elements and potential for further advances in concrete technology and construction capabilities.

## Chapter 3

## Materials & Mix Design

#### 3.1 Chapter Overview

The following section identifies all materials required for the successful development of SCC samples. It provides details regarding the specific properties of each material, including test results for coarse and fine aggregates, cement and FA which were obtained from the supplier. Accurate knowledge of properties such as bulk density, water absorption percentage, moisture content and specific gravity are required to formulate an accurate SCC mix design. Characteristics of RHA, superplasticizer (HWRW) and water are also discussed. Information provided in section 3.2 assists in defining the mix design proportions and quantities adopted for each of the 6 trial mixes. Further details regarding the design process are provided in section 3.3.

#### 3.2 Materials

The fundamental constituents of an SCC mix do not vary significantly from a general purpose concrete mix, which includes cement, aggregates and water. In order to meet various demands relating to material properties required for construction numerous additives can be included. This includes pozzolanic materials such as FA, RHA and blast furnace slag, and admixtures such as high range water reducers (i.e. superplasticiser) and viscosity modifying agents (VMA's). Alterations to the composition of a mix design through addition or partial replacement of these constituents has the potential to significantly alter the final quality of a SCC mix. All major and minor constituents required for this research are discussed below.

#### 3.2.1 Rice Husk Ash

The RHA material attained for this research project was procured at the University of Southern Queensland's Ceramics department using a Kiln under controlled burning conditions. In order to produce the final ash product, rice husk was first obtained from Cardiff Produce in Newcastle NSW, which was originally sourced from the supplier CopRice at their Leeton rice mill in regional NSW. The Leeton mill produces rice and rice products for an extensive range of purposes, their current marketing strategy for managing rice husk is to sell as rice husk bedding intended for application as animal bedding. Figure 3.1 below shows the overall quantity of rice husk that was used for this project. The rice husk was re-bagged for more manageable transportation to the University of Southern Queensland. 125 kg of rice husk was reduced to approximately 50 kg of RHA following the firing process.



Figure 3.1: Rice husk bagged for transport

The firing and RHA preparation process has been discussed further in Chapter four, however to summarise this process the following procedures occurred:

- Rice husk was burnt at temperatures not exceeding 650°C; the firing method required a ramp rate of 200°C per hour until it reached 600°C after three hours. The temperature was then held just above 600°C for one hour.
- The resulting RHA remained as a silica skeleton following this process, and required grinding before application as a pozzolanic material would be possible

- The RHA was ground using a Raymond Mill (Hammer Mill) to a particle size less than 75 micron
- Experimental analysis was conducted on the RHA to confirm adequate quality

Figure 3.2 illustrates the primary stages the rice husk went through from its original raw form to the final homogenous ground husk.



Figure 3.2: Stages of RHA procurement

A comprehensive understanding of the physical properties and chemical composition of RHA is essential when conducting experimental research. The experimental analysis procedure and testing results are discussed in detail within chapters 4 and 5 respectively. Findings from the applied testing methods provide significant indication as to whether the procured material is an effective SCM. The colour of RHA resulting from the firing process was dark grey. This was likely due to conditions within the kiln being less than optimal. Due to the placement of husk at the bottom of the kiln, heat dispersion was not even resulting in temperature variations within the material. This burning process resulted residual unburnt carbon, thus the subsequent dark colour was developed. Through refinement of the burning method, development of unburnt carbon can be minimised resulting in a lighter SCM (Krishnarao, 2001).

The EFNARC guideline (2005) have identified that the use of pozzolanic materials in SCC has shown promising results in reducing the quantity of cement required and improving certain physical characteristics of SCC. However these effects must be cautiously and independently evaluated for both short and long term negative impacts on the concrete. Results from the materials experimental analysis have been included in Chapters 4 as a detailed discussion of RHA's impact as a pozzolanic material.
#### 3.2.2 Cement

Cement is the essential binding component of concrete, the appropriate selection of quantities and application is important in order to obtain a balance of properties necessary for a particular concrete mix design. As such, an understanding of each chemical and physical characteristic of cement and how each component influences the properties of concrete is necessary to ensure an adequate result is achieved (*Guide to Concrete Construction, 2002*). General purpose Portland cement has been used for this research project. Although there are blended cements available containing percentages of FA, to ensure accuracy and reliability in the experimental process each component will be combined separately. Figure 3.1 identifies the cement type and supplier used for the experimental research.



Figure 3.3: GP Cement bag for cement used in research

The primary constituents of Portland cement include calcium carbonate primarily in the form of hydrated limestone, alumina, silica and iron oxide. These materials are combined and kiln dried at temperatures ranging from 1300 to 1500°C where they fuse to form a clinker, which are hard balls of ceramic like material. During the firing process a heat induced reaction occurs that produces new chemical compounds capable of reacting with water in the previously discussed hydration reaction. Once removed from the kiln and cooled, the clinker is combined with gypsum and ground to a fine powder. Gypsum is essential in controlling the rate of hydration when cement and water are combined to create the paste component of concrete (*Guide to Concrete Construction, 2002*). Test results for the specified cement used in this research have been provided in Appendix B.

#### 3.2.3 Fly Ash

Fly Ash has been identified as an effective addition for application in SCC as it provides an increased cohesion and reduced sensitivity to changes in water content. Due to the fresh property requirements of SCC, pozzolanic additions are commonly incorporated in order to improve and maintain elements such as cohesion and segregation resistance. The addition of FA commonly leads to improved fluidity of fresh SCC for improved placing ability. This compound also assists in reducing the quantity of superplasticiser required whilst maintaining an adequate flow. However, it has been identified that high levels of FA may result in a paste fraction that is so cohesive that it becomes resistant to flow (EFNARC, 2005). This can significantly impact on a mix design's ability to meet the acceptance criteria required for fresh SCC. Figure 3.4 provides a visual representation of the FA used in this research project.



Figure 3.4: Fly Ash used for this project

In general, the replacement of cement with FA results in a reduction of the initial strength of concrete. This effect can be reduced with the adequate use of certain admixtures. Benefits of FA are observed at later stages with an accelerated increase in strength development, increased durability and sulfate resistance. Test results for the FA used in this research project have been provided in Appendix 2. Overall this material consists predominantly of 57.8% amorphous silica (SiO<sub>2</sub>) and 28% Aluminium Oxide (Al<sub>2</sub>O<sub>3</sub>), which accounts for the pozzolanic contribution of this material.

### 3.2.4 High-Range Water-Reducing Admixture

High-range water-reducing admixtures (HRWR) such as superplasticiser are an essential component in achieving a high quality SCC mix. HRWR admixtures are a formulation of chemicals that disperse cement particles and increase the fluidity of concrete. This essentially means that for a given workability, these admixtures permit a reduction in the amount of water required in the concrete mix (*Guide to Concrete Construction, 2002*). Although there are other water reducing admixtures, HRWR produce a very highly workable concrete, or a concrete with a very low water-cement ratio. The effects of this admixture have a limited time period, and often do not last for much longer than an hour.

Viscosity Modifying Admixtures (VMA) may also be used to assist in reducing the segregation and sensitivity of the mix resulting from minor variations in moisture content and excess fines. This makes the mix more robust and less sensitive to variations in proportions and variations in other materials (EFNARC, 2005). As this research project has been designed to closely monitor the condition of all material constituents, VMA's were not used. For a much larger application of SCC it would be appropriate to utilise the effects provided by VMA's.

Superplasticisers are used for SCC in order to acquire two key benefits:

- To significantly increase the workability of concrete of a predetermined watercement ratio
- To increase the strength of concrete whilst permitting significant reductions in water content

For application in this research project the purpose of using a HRWR such as superplasticiser was to achieve both of the above outcomes. With proposed target strength of 40MPa the water content was required to be low, whilst it was still vital to maintain a high workability of each mix. The HRWR admixture used for this research was MasterGlenium 1466, this product was selected due to its effectiveness in producing mixtures with reduced portland cement contents without compromising the concretes 28 day strength requirements. A technical data sheet has been provided in Appendix B. The overall outcome resulting from the application of SCC has been discussed in Chapter 5.

#### 3.2.5 Course Aggregate

Coarse aggregate specified for this project were classified as 10/7 mm concrete Blue Metal Aggregate in SSD condition. This aggregate was to conform to AS2758.1 Australian Standard for concrete aggregate. The aggregate was described in its associated testing report as being very robust, unweathered, hard, angular fragments. A grading profile and testing report is provided in Appendix B for reference. Other details provided in the material testing report include its bulk density, moisture content and water absorption percentage. This data has been included in the SCC mix design developed for this research.

The aggregate used in this research was provided by a nearby on site concrete batching plant. This allowed for flexibility in trialling mixes without risk of exhausting materials. This aggregate was maintained at SSD conditions throughout the duration of the experimental process with no alternate materials being added. Although these conditions were maintained the coarse aggregate was described as being non-porous in its technical report, and as such additional moisture would not significantly affect the water / cement ratio of the SCC trial mixes.



Figure 3.5: 10/7 mm Coarse Aggregate

Figure 3.2 shows the coarse aggregate used for this research project. The EFNARC guidelines (2005) indicate that the moisture content, water absorption, grading and variations in fines content of all aggregates should be closely monitored and taken into account when producing a trial SCC mix. This document also noted that reinforcement spacing is the primary factor in determining the maximum aggregate size, as passing

ability will become reduced with larger aggregates. As such the advised maximum aggregate size was stipulated as being between 12 and 20 mm, the 10/7 aggregate size selected was based on its suitability for application in dense reinforced members.

#### 3.2.6 Fine Aggregate

Fine aggregate specified for this project were classified as uncrushed washed fine dune sand. The purpose of washing aggregates is to remove silt and fine particles less than 0.125mm. Washed sand requires a lower water cement ratio in order to achieve high workability, primarily due to the absence of finer particles that would otherwise absorb significant portions of the water. Particles size fractions less than 0.125m are to be included in a SCC mix design's overall fines content and subsequently affects the water paste ratio (EFNARC, 2005). As such the fine sand used in this research project was graded to a minimum of 0.150mm, material test results have been provided in Appendix B which includes a particle size distribution curve. Other details provided in the material testing report include its bulk density, moisture content and water absorption percentage. An example of the sand used for this research project is in Figure 3.3 below. A sieve analysis of the sand confirmed that it contained approximately 65% medium particles and 35% fine particles.



Figure 3.6: Fine dune sand used in this project

#### 3.2.7 Water

The minimum requirement advised for water used in the production of concrete is that it is potable, or that it be clean and free from impurities harmful to concrete. Impurities may be a result of suspended solids, organic matter and/or salts (*Guide to Concrete Construction, 2002*). In instances where potable water is not available a guide provided for solids in suspension has been provided in AS2758.1 which permits up to 2% of material finer than 2mm in suspension.

High quantities of organic matter within a mix proportions water content is likely to affect its strength, and in some cases can prevent concrete from setting. Common effects of organic matter contamination in water include retardation of the rate of strength gain, this can be compensated through allowing a longer duration for concrete to gain strength or by increasing cement proportions. Neither of these solutions are considered practical in the construction industry of for testing of trial mixes, therefore water quality is to me monitored and acceptance confirmed. For the purpose of this research potable water was used, this water was incorporated at a concrete batching plant and therefore was deemed adequate.

# 3.3 Self-consolidating Concrete Mix Design

There is no standard mix design method for SCC, as such many academic institutions and concrete manufacture companies have developed their own specific mix proportioning methods which have been adapted through extensive trial mixing. The EFNARC guidelines (2005) include mix composition recommendations, previously discussed in section 2.4.2 (Table 2.4), which provide an overall indication of the typical range of constituents in SCC by both weight and volume. The provision of two proportioning parameters, weight and volume, enables the application of various mix design methods. For ease of application, this research project has based its mix design on weight. More accurately, the compressive strength mix design method as described by Ghazi (2010) was applied.

In order to achieve the properties required for SCC, the primary criteria that was focused on was achieving an acceptable fluidity and viscosity. This characteristic is adjusted and balanced through careful proportioning of cement and additives, limiting the water/paste ratio, and then by adding admixtures to achieve sufficient workability (EFNARC, 2005). In order to attain a suitable SCC mix all proportions included must be carefully balanced and each material continuously monitored for changes in quality, i.e. water content, existence of fines in aggregates, as this will likely impact on the final product. In the event that the required performance is not achieved, attention should be given to a fundamental redesign of the mix. Likely methods of modifying a mix design have been included in the EFNARC guidelines (2005), and predominantly focus on adjustment of the cement/powder ratio, water/powder ratio and types/quantities of admixtures that are added. Annexure C from EFNARC guidelines (2005) provides an in-depth guide regarding the types of defects that may be encountered and methods of mitigating and resolving these issues. Any visible deformity observed in the final sample specimens have been reported in Chapter 5.

The EFNARC guidelines (2005) also provide an overview of the various classes of SCC and their intended application. Table 3.1 identifies the classes for which fresh concrete tests can be observed against. The properties of SCC differ depending on the intended application. The application of each trial mix has been discussed further in Chapter 5.



Table 3.1: Properties of SCC for various applications (EFNARC, 2005)

#### 3.3.1 Mix Design Proportions

The mix design method used for development of a control SCC was calculated using the methods described by Ghazi (2010). This academic report provided a defined, specific method of calculating the required mix proportions and allowed for the inclusion of pozzolanic materials. The calculated mix proportions, illustrated below in Tables 3.1 and 3.2, were first determined in kg/m3 and then converted to allow for a quantity of 0.0286 m3 of concrete. This final quantity was developed using the proportions provided in Australian Standard AS1012.2 Methods of testing concrete – Preparing concrete mixes in the laboratory (AS1012.2, 2014).

Table C1 of this standard identifies that each compressive cylinder sample using a 100mm x 200mm mould requires 0.002 m<sup>3</sup> of concrete, whilst each flexural beam sample using a 100mm x 100mm x 350mm beam requires 0.005 m<sup>3</sup> of concrete. For a total of three compressive and three flexural samples this equates to 0.021 m<sup>3</sup> per trial mix, with the additional amount being included to allow for quantities potentially lost during the mixing and fresh concrete testing process. The water powder ratio was maintained at 0.32 and superplasticizer, aggregate, water and FA contents were maintained at a constant amount. In order to develop each of the six trial mixes, the cement was reduced by the same percentage increments that RHA was added.

Mix proportions were compared against the requirements identified in the EFNARC guidelines (2005). Although the coarse aggregate content was slightly less than the advised typical range, it was determined that this was likely due to the use of 10/7mm aggregate as this resulted in a lower bulk density (1400 kg/m3). Fine aggregate reached the advised criteria with a content equal to approximately 52% of the total aggregate weight. The original mix design calculated for this project included superplasticiser at a quantity of 1.3%, however upon conducting the first mix design, intended as a control sample, it was determined that the SCC mix failed. This was due to retardation in setting time beyond 24 hours. As this is certainly not satisfactory for application in construction the mix design proportions were re-evaluated to allow for 1% superplasticiser content. Results have been provided in Chapter 5.

Compressive Strength Mix Design (m <sup>3</sup> )										
Mixture	Cem kg	FA kg	RHA kg	Tot. Powder	Fine Sand	10/7 Agg	Water (L)	SP (L)	SP%	w/p
CM1FA20	435.9	114.0	-	570	753.0	702.1	183.5	5.7	1%	0.32
M1RHA10	399.0	114.0	57.0	570	753.0	702.1	183.5	5.7	1%	0.32
M2RHA15	370.5	114.0	85.5	570	753.0	702.1	183.5	5.7	1%	0.32
M3RHA20	342.0	114.0	114.0	570	753.0	702.1	183.5	5.7	1%	0.32
M4RHA25	313.5	114.0	142.5	570	753.0	702.1	183.5	5.7	1%	0.32
M5RHA30	285.0	114.0	171.0	570	753.0	702.1	183.5	5.7	1%	0.32

Table 3.2: SCC mix design proportions calculated in kg/m<sup>3</sup> for research project

Table 3.3: SCC mix design proportions representative of trial mix

Compressive Strength Method (For 0.0286 m <sup>3</sup> Mix)										
Mixture	Cem kg	FA kg	RHA kg	Tot. Pdr	Fine Agg	Coarse Ag	Water (L)	SP (L)	SP%	w/p
CM1FA20	12.47	3.26	-	16.3	21.54	20.08	5.25	0.163	1%	0.32
M1RHA10	11.41	3.26	1.63	16.3	21.54	20.08	5.25	0.163	1%	0.32
M2RHA15	10.59	3.26	2.44	16.3	21.54	20.08	5.25	0.163	1%	0.32
M3RHA20	9.78	3.26	3.26	16.3	21.54	20.08	5.25	0.163	1%	0.32
M4RHA25	8.96	3.26	4.07	16.3	21.54	20.08	5.25	0.163	1%	0.32
M5RHA30	8.15	3.26	4.89	16.3	21.54	20.08	5.25	0.163	1%	0.32

## 3.3.2 Mix Design Methodology

The compressive strength method for proportioning of a SCC mix design has been reviewed and applied in similar research conducted by Ghazi (2010) as discussed previously in section 2.4.1. This method provides a procedure capable of achieving a specified strength, unlike many other documented methods. As this research project aims to assess each mix design based on its strength characteristics it was determined this method was the most appropriate. The following procedure has been specified by Ghazi (2010) as the correct compressive strength method. The proportions listed in Tables 3.1 and 3.2 provide a summary of the calculations performed to attain the required proportions. Detailed mix design calculations have been provided in Appendix C.

The following steps have been applied for proportioning of the trial SCC mix, as detailed in the experimental research conducted by Ghazi (2010) and using the tables published in the aforementioned literature (tables mentioned below refer to Ghazi, 2010):

- Using Table 2b, the maximum weight of water and air content in the mixture is identified according to maximum coarse aggregate size. Bulk density of 10/7mm aggregate was sourced from the test results provided in Appendix B
- 2. The required water weight (Ww) was determined from Figure 1, and the water/cement ratio (w/c) was determined based on the required compressive strength as listed in Table 3
- 3. The above result was used to determine the cement content (*Wc*) and volume of cement (*Vc*) were  $Wc = \frac{Ww}{w/c}$  and  $Vc = \frac{Wc}{SGc} \times 1000$  (*SGc* = specific gravity of cement)
- 4. The dry rodded volume of gravel (Vg) is obtained from Table 4b
- 5. The dry weight of gravel (Wgd) was then determined by multiplying Vg b the compacted bulk density of gravel (Appendix B)
- 6. The saturated surface dry (SSD) weight of gravel is then calculated where  $Wg = Wgd \times (1 + \frac{A}{100})$  where A is the absorption percentage of aggregate (Appendix B)
- 7. Table 5 was then used to determine the volumetric ratio of water, and using this result the volume of FA ( $V_L$ ) was determined
- 8. The weight of FA was then calculated by multiplying  $V_L$  by the materials specific gravity (Appendix B)
- 9. These results were then used to calculate the total weight and volume of powder, and the overall figure was compared with the EFNARC guidelines (2005) to determine acceptance
- 10. The final step calculated the fine aggregate content using the absolute volume method

## 3.4 Chapter Summary

The mix proportions utilised for this project have been identified in the above section. Further details regarding material test data and mix design calculations have been provided in Appendix B and C respectively. Control materials were acquired from the Daracon Williamtown batching plant, with the rice husk being the only independently sourced product. All control materials received regular quality testing through Nata accredited laboratories. The cement utilised for this research project was OPC, with FA being the control pozzolanic material, aggregates used were 10/7 mm concrete Blue Metal Aggregate and washed fine dune sand, the water was acquired from a potable water source and the HRWR superplasticiser was MasterGlenium 1466.

The mix design procedure was applied in accordance with the methodology described by Ghazi (2010), and proportion results were compared with similar research and the EFNARC guidelines (2005) in order to determine its acceptance in achieving the properties required for SCC. This comparison determined that the overall mix design was acceptable. Although the coarse aggregate quantity was lower than the minimum requirement, this was attributed to the bulk density of 10/7 mm aggregate being lower than that of higher grade aggregates. The water content and superplasticiser percentage were kept constant throughout all six trial mixes.

Once an acceptable RHA material was acquired, this compound was applied to the existing concrete mix with replacement percentages starting at 10% replacement and increasing in 5% increments to a total replacement percentage of 30%. The purpose of initiating the RHA replacement at 10% inclusion was to initially develop an observable change in the materials effects in comparison with the control. This procedure was determined as optimum in attaining an overall representation of the impact of RHA. One control sample and five trial mix samples were developed as shown in Table 3.2, with the final mix design achieved an overall SCM replacement of 50%. Observations developed from the fresh and hardened concrete testing has been discussed in Chapter 5, however it is expected that an increase in RHA will result in a reduced SCC flow, increase in water demand and a decrease in strength. The extent of these effects will determine to what extent RHA can be applied as a pozzolanic material.

# Chapter 4

# Experimental Methodology & Preparation

## 4.1 Chapter Overview

The following chapter provides an overview of the experimental methods and specimen preparation techniques applied in this research. A diverse range of technical equipment was utilised for the project, all have been discussed thoroughly within the following chapter. The methodology involved in preparation of the RHA and both compressive and flexural strength sample specimen have also been discussed to provide an overall understanding of the depth of practical experimentation required in this project. The methods of conducting both fresh and hardened concrete testing have also been explained.

# 4.2 Project Resources & Equipment

## 4.2.1 Material Preparation Equipment

The preparation of constituent materials required for this project was primarily based around the effective preparation of RHA to achieve a high quality product. Due to the accessibility of constituent materials such as cement, FA, coarse and fine aggregates located at the Daracon Williamtown batching plant, I was able to obtain each material at optimal quality. All coarse and fine aggregates were maintained at SSD condition, although oven drying aggregates is the preferred method, this was not possible due to unavailability of this resource. The process of adequately preparing RHA first required the husk to be burnt within a kiln at temperatures not exceeding 700°C. As mentioned the firing procedure resulted in the husk being burnt to a temperature not exceeding 640°C. Figure 4.1 identifies the kiln used for this process. Some minor limitations identified regarding the use of the kiln include a firing ramp rate of 200°C per hour, and the thermostat's position at the top of the kiln.



Figure 4.1: Visual representation of the Kiln used for firing

The firing ramp rate was not a significant issue, with the resulting impact being a longer duration for each firing sequence than originally anticipated. The position of the thermostat at the top of the kiln resulted in potential temperature variations between the top and bottom of the kiln that could not be measured. In order to remediate this issue it was decided to increase the firing temperature to 640°C, with the anticipated result being that the temperature at the bottom of the kiln would still be maintained at or above 600°C.

Following the firing process it was observed that the RHA was not a fine homogenous material. As this is a preferred characteristic for a suitable pozzolanic material, the RHA was ground using a Hammer Mill at Bureau Veritas Cardiff, a material testing laboratory located in Newcastle. Figure 4.2 provides a visual representation of the Hammer Mill used for this project.



Figure 4.2: Example of the Hammer Mill used for grinding of RHA

The final plant item required for preparation of the samples was a 65L tilting drum petrol operated cement mixer. This mixer was in reasonable condition and functioned adequately in producing a homogenous trial mix. Figure 4.3 provides an example of the mixer used in this project.



Figure 4.3: Tilt-drum petrol concrete mixer used for project

#### 4.2.2 Sample Testing Equipment

The development of an accurate research result depended significantly on the application of effective sample testing equipment. The details of each testing procedure have been outlined in section 4.4, however the following test equipment was utilised in order to achieve successful results.

#### **Experimental Analysis Equipment**

The equipment used for experimental analysis of the RHA was previously discussed in Section 2.3. XRD, XRF and SEM analysis were conducted on a representative sample of the product in order to determine its primary constituents and particle characteristics. The XRD and SEM analysis were conducted at the University of Newcastle with the assistance of academic staff. The SEM analysis also incorporates the application of an EDS analysis tool to further identify compounds. Following the provision of results, an XRF analysis was conducted at Bureau Veritas in Newcastle to provide an overall confirmation of the total elemental constituents of the material. Application of this testing method was essential in providing quantitative evidence supporting the materials quality.

#### Fresh Concrete Testing Equipment

The requirements for SCC fresh concrete testing vary from the standard methods of testing concrete. Whereas some of the same equipment is used for both methods, the SCC fresh concrete testing focuses predominantly on four key characteristics, as discussed in the EFNARC guidelines (2005):

- Flowability using the Slump-flow test
- Viscosity assessed by rate of flow using the  $T_{500}$  slump-flow test
- Passing ability In this project the J-ring passing ability test was used
- Segregation Segregation resistance (sieve) test

The slump flow and  $T_{500}$  flow test can be conducted concurrently in order to reduce the duration of testing. In order to conduct this test a regular slump cone (Abrams cone) is required, the other apparatus include a sturdy board with circles measured at 200mm and 500mm, along with a marker at the centre, a shovel for filling the slump cone and a stopwatch. Details regarding the testing procedure are provided in section 4.4.2. This is depicted in Figure 4.6 below.



Figure 4.6: Base plate dimensions for fresh concrete testing

The J-ring test utilises the equipment listed above however it also incorporates a J-ring, depicted in Figure 4.7. The J-ring simulates the concrete sample's ability to flow through dense reinforcement. Results from this test are compared with the previous slump-flow test to determine whether sample mix is acceptable under this parameter. The J-ring used in this research project was designed to conform with the requirements set out in the EFNARC guidelines (2002).



Figure 4.7: J-ring apparatus used for testing passing ability

The final fresh concrete test was the sieve segregation test, conducted to determine the resistance of a SCC sample to segregation. The apparatus required for this test method include a standard 5mm aperture size plate sieve with frame diameter 300mm and height 40mm, a sample container and scales. The test method requires a segregation percentage less than 20%, or preferably less than 15%.

## Compressive Strength Testing Equipment

The compressive strength test undertaken for this project utilised testing equipment located at the Coffey testing & analysis laboratory in Newcastle. This is a NATA accredited testing laboratory, with compressive strength testing equipment and procedures implemented to comply with Australian Standard *AS1012.9 - Methods of testing Concrete: Method 9 – Compressive strength tests* (Standards Australia, 2014). The cylinder specimens used for this component of the project were sized approximately 100mm x 200mm in accordance with AS112.9 (2014).

The machine uses a hydraulic ram to apply a continuous pressure by lowering the top plate. Once the load builds to a point beyond what the concrete sample can withstand, a failure occurs in the specimen. This maximum load at failure is used to determine the overall compressive strength of the concrete sample. Refer to Figure 4.4 for a photo of the compressive strength testing machine used for this testing requirement.



Figure 4.4: Compressive Strength Testing Equipment

## Flexural Strength Testing Equipment

The flexural strength test undertaken for this project utilised testing equipment located at the Coffey testing & analysis laboratory in Newcastle. This is a NATA accredited testing laboratory, with flexural strength testing equipment and procedures implemented to comply with *RMS (NSW) Method T328 – Modulus of Rupture test*. The test utilised to determine the samples flexural strength was a four-point load test. This method required a 100mm x 100mm x 350mm rectangular concrete beam, aligned centrally within the testing machine with an overhang at both ends.

The machine used for this testing used a hydraulic ram that was capable of applying a maximum load of 100 kN. Although there is also a three-point bending test, the inclusion of two points of stress results in a much larger area of the beam that is subject to the maximum stress, and therefore a more accurate depiction of the samples flexural strength. Refer to Figure 4.5 for a photo of the flexural strength testing equipment used for this component of the project.



Figure 4.5: Flexural strength testing equipment

# 4.3 Sample Preparation Method

Creating all components of the test specimen required multiple stages of development, this ranged from the preparation of RHA to the mixing of each SCC sample and creation of sample specimen for testing. The following section identifies each stage of development and the associated procedure applied.

The first step in preparing the materials required preparation of the RHA, which was developed using the methods previously discussed. This process required the RHA to first be burnt to a minimum of 600°C, which was achieved through firing a kiln at a ramp rate of 200°C per hour until it reached 640°c, and then maintaining this temperature for one hour. The reason for applying this methodology has been discussed in section 4.2.1. Following the firing process the resulting material appeared as a silica skeleton, with a particle size much greater than was required. Figure 4.6 provides a visual representation of the RHA following this process.



Figure 4.6: RHA material following firing process

The light colour represents the silica skeleton remaining after all other constituent materials were removed, whilst the black particles have been identified as the unburnt residual carbon that remained. As mentioned previously, the RHA was then ground in a Hammer mill resulting in a fine homogenous powder. The ash was ground until it passed through a 75 micron screen located within the machine in order to achieve the required maximum particle size.

All other constituent materials were regularly monitored for quality by the Daracon batch plant, with test results provided in Appendix B. The test results provided indicate all relevant technical data required for each material, including the materials specific gravity, particle size distribution, bulk density, water absorption %, moisture content and the overall material composition of both the FA and cement used in this research project. Specific details regarding the materials used in this project have also been provided in Section 3.2.

During the sample preparation process, it was confirmed that the washed dune sand did not contain any fines, particles less than 0.125mm. This was of particular interest due to the requirements outlined in the EFNARC guidelines (2005) that advised any particles less than this size would impact on the paste volume of an SCC mix. All materials were prepared on site and weighed using scales with an accuracy measured to 1 gram which was deemed sufficient for this research. Grading curves have been provided in Appendix B for both coarse and fine aggregates, detailing the specific parameters for each constituent.

Prior to the mixing of each sample batch, the required constituent materials were accurately weighed and measured so they could be combined in the concrete mixer in a timely manner. The moulds required for each sample also required preparation. This involved applying oil to the inside of three cylinder and three rectangular moulds for each trial mix, dimensions complying with the parameters stated in section 4.2.2.

Following this preparation process, each trial mix was developed through the addition of materials in compliance with Australian Standard *AS1012.2 preparing concrete mixes in the Laboratory*. Minor modifications were made to this procedure based on advice provided in the EFNARC guidelines (2005). The primary modification that was required involved an increase in SCC mixing time to allow the superplasticiser time to fully activate. As such an initial trial mix was conducted to better understand this process. The trial mix was then discarded.

The mixing process method was repeated for each trial mix. The first step was to combine both the coarse and fine aggregates in a mixer with roughly 90% of the total water whilst the mixer was operating. All cementitious materials were then added, and once a somewhat homogenous mix developed the admixture and remaining water was added. The mixer was then left operating for no less than five (5) minutes. This allowed time for the superplasticiser to react with the water and paste. Following this time period the mixer was turned off and allowed to rest for two (2) minutes. During this time the mix was visually assessed to determine whether the superplasticiser had activated. On all occasions the SCC was mixed for a further two (2) minutes, followed by fresh concrete testing to confirm acceptance. Photos of the moulds have been provided in Figure 4.7. Variations in colour are due to increasing percentages of RHA.



Figure 4.7: Flexural & Compressive specimen moulds

The SCC mixture was then removed from the mixer and placed in the associated moulds. The samples were then left to set for a period of two hours, and then placed in a storage container under wet hessian for twenty-four hours. Each mould number was recorded in order to correspond with its associated trial mix. Following this time period the moulds were cracked and transported to the Coffey test laboratory in Newcastle to be placed in a curing tank for a maximum period of twenty-eight days. The curing tank provided at the testing facilities was closely monitored to ensure a constant temperature and conditions were maintained.

Following the curing process it was possible to conduct flexural and compressive strength tests to determine both seven and twenty-eight day strength. The associated testing methodology has been discussed in section 4.4, whilst results have been discussed in Chapter 5.

# 4.4 Sample Testing Method

Multiple testing components were required throughout various stages of this research project. Each procedure has been documented in the following section. All parameters involved in testing for Elemental Analysis, fresh SCC acceptance, compressive strength and flexural strength of the samples have been discussed. Elemental Analysis was conducted at the University of Newcastle and Bureau Veritas Cardiff, Newcastle. Methodologies for these tests have been provided as a general overview of the procedure required. Fresh SCC testing was conducted in accordance with the guidelines set out by the EFNARC (2005). Compressive and flexural strength tests were carried out in accordance with the standards and guidelines mentioned in section 4.2.2.

### 4.4.1 RHA Elemental Analysis

Elemental analysis was determined as an essential component of the research project. Through identifying the quality of the produced RHA it was possible to determine the compounds effectiveness as a pozzolanic material, and whether the applied firing process was suitable. As stated in section 2.3 there were three methods of experimental analysis applied in this research project. These were X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), and X-ray Fluorescence (XRF). A collaboration of these methods provides both an initial representation of the samples composition, and also further analyses the quantities of each element.

The procedure required for XRD analysis involved a representative sample of the RHA being ground to a fine powder to allow adequate quantities of diffraction to occur once the scanning process is initiated. The sample was packed onto a sample holder to assume different orientations and ensure reflections from varying planes. In order to attain a suitable result care must be taken to create a flat upper surface, and also the prepared sample must achieve a random distribution of particle orientations. Following effective sample preparation, the specimen is then subject to x-rays and the corresponding diffraction pattern is recorded. A printout of the resulting constructive interference rays is then provided, with the resulting diffraction pattern identifying the existence of various particles in crystalline form. Figure 4.8 illustrates the samples used for XRD analysis, the left hand sample is unground while the right hand sample has been ground to a fine powder. Details regarding results of this process have been provided in Chapter 5.



Figure 4.8: Samples prepared for XRD analysis

In order to conduct the SEM analysis a focused beam of electrons is applied to a similarly prepared sample of RHA. The resulting output of this test produces a high resolution, high depth of field surface image that is able to identify the characteristics of individual particles. Preparation of the specimen was conducted at the University of Newcastle, with the resulting SEM output being provided in Chapter 5. Whilst conducting the SEM analysis an Energy-Dispersive Spectroscopy (EDS) analysis was also applied. This method involves the application of an x-ray spectrum to an existing SEM analysis. This allows the production of an image that identifies certain elements within a sample based on their primary constituents. When applied to RHA the EDS output is intended to identify the existence of silica and oxygen.

In order to confirm the overall composition of the RHA material produced the final method applied was an XRF analysis. As discussed in section 2.4.2, this method measures the intensity of x-rays fluoresced by individual major and minor elements within a representative sample. This test was conducted at the NATA accredited Bureau Veritas Cardiff laboratory in Newcastle. The output of this test confirmed the overall percentage of  $SiO_2$  in the sample along with the existence of any other minor constituents or trace elements. Results of this test were provided by the testing laboratory for inclusion in this research report.

#### 4.4.2 Fresh Concrete Testing

As mentioned previously, the testing method for fresh SCC focused on four key parameters. These were flowability, viscosity, passing ability and segregation resistance. The attainment of these parameters is essential as SCC is specifically designed to have a very high flow whilst maintaining cohesive characteristics high enough so that aggregate us uniformly suspended and does not segregate. Four tests have been listed in section 4.2.2 as methods of determining each trial mix has met these parameters. It is important to note that no global standardised testing methods have been established for SCC, the testing methods described in both the 2002 and 2005 editions of the EFNARC guidelines provide only advised ranges that the testing results should fall within. As this is the primary resource utilised for this research, all identified testing methods have been adhered to and acceptance criteria attained. All results and observations have been provided in Chapter 5.

### Slump-Flow Test

In order to determine the flowability of a sample the slump-flow test was used. The slump flow value identifies the flowability of a fresh mix in an unconfined state. This test method has been determined as the primary test for SCC acceptance, the reason being that it can be readily applied in both laboratory and site conditions in similar fashion to the slump cone test used for OPC. Apparatus required for the slump-flow test have been identified in section 4.2.2. Through application of the following methodology typical slump-flow ranges that an SCC trial mix should achieve are between 660mm and 750mm for standard SCC (class SF2), however there are both upper and lower limits based on the intended concrete application.

In order to conduct a slump-flow test the apparatus is first arranged so that the slump cone is in position on a board or similar hard surface. The cone is then held in position whilst it is filled, ensuring no concrete can leak from under the cone. The cone is to be filled without any agitation or rodding, and any surplus struck off from the top of the cone. The filled cone is to stand for no more than 30 seconds. The cone is then lifted vertically in one movement without interference to the flow. Without disturbing the baseplate or concrete, the largest flow spread diameter is measured and recorded, followed by a measurement taken at right angle to the previous measurement. The average of these measurements will be the final slump-flow.

Aside from the acceptance criteria identified in Figure 4.8, it is possible to assess segregation within the mix. If there is a cement paste ring extending greater than several millimetres beyond the coarse aggregate segregation is likely to have occurred.

Table 4.1: Slump-flow passing criteria

Class	Slump-flow in mm
SF1	550 to 650
SF2	660 to 750
SF3	760 to 850

#### T<sub>500</sub> Slump-flow Test

The  $T_{500}$  slump-flow test is applied in order to determine the viscosity of a trial mix. Although there are alternative methods of testing viscosity, the  $T_{500}$  test can be applied concurrently with the slump-flow test which minimises the time taken before a trial mix can be placed in moulds. The test follows identical parameters to the slump-flow test, however uses the 500mm circle shown in Figure 4.6 to measure the time taken to flow to an approximate diameter of 500mm. The  $T_{500}$  also measures the speed of flow and hence viscosity of the SCC trial mixes.

As described previously for the Slump-flow test, the slump cone is filled and then struck off to remove any excess concrete. Once the cone is lifted off its surface, a stopwatch is initiated. The time is measured until any portion of the concrete exceeds the 500mm diameter line, at which point the stopwatch is stopped. As detailed in section 6.4.2 of the EFNARC guidelines (2005), the  $T_{500}$  measurement is only provided to identify the rate of flow, however if a mix were to fall significantly below or greater than two seconds it could still be acceptable. A mix below 2 seconds is more likely to suffer from bleeding and segregation, whilst a mix significantly greater than 2 seconds may result in a poor surface finish or difficulty flowing through dense reinforcement.

Table 4.2: T<sub>500</sub> slump-flow test parameters

Class	T <sub>500</sub> , s	V-funnel time in s
VS1/ VF1	≤ 2	≤ 8
VS2/ VF2	> 2	9 to 25

Aside from providing an analytical tool for determining the viscosity of a mix design, the  $T_{500}$  slump-flow test can also be used as a way of confirming uniformity between concrete batches and potentially identify the effects of various modifications to an existing SCC mix design.

## J-ring Test

The J-ring test is utilised to determine the passing ability of SCC, essentially its ability to flow through obstructions and reinforcement. The equipment required for this test has been identified in section 4.2.2 of this report. Through analysis of the testing method it was determined that there are two key parameters that are assessed. The first being a comparison between the slump-flow result and an identical measurement of the flow subject to the inclusion of a J-ring, of which the values should not differ by more than 50mm. The second test measures the internal and external height at four points. The difference between these two heights must be less than 10mm. Figure 4.8 provides a visual representation of the J-ring testing procedure.



Figure 4.8: Testing procedure using J-ring apparatus

#### Sieve Segregation Resistance Test

Segregation resistance is a fundamental component in developing a high quality, homogenous SCC mix. The appearance of segregation often occurs during or after placing but before stiffening of the concrete. The EFNARC guidelines (2005) discuss the various segregation classes in order to identify the various parameters required for different applications. The apparatus required for this testing method have been listed in section 4.2.2.

In order to conduct a sieve segregation resistance test the first step is to collect a 10L sample of the trial mix in a sample container. This mix is then allowed to stand in a level position without disturbance for fifteen (15) minutes. The surface is then inspected for any sign of bleed water and note. Following this time period a sieve with 5mm aperture size is placed on a bucket with a predetermined weight, and the placed on a scale. Pour the top two litres of SCC sample into a pouring container. Ensure weights of all filling and measuring apparatus are recorded. Finally pour all concrete from the container onto

the sieve from a height of 500mm in one continuous pour. Weigh the empty pouring container to determine the mass poured into the sieve. Allow the mortar to flow through the sieve into the bucket below for a period of two (2) minutes. Remove the pan and weigh the contents remaining in the bucket to determine the overall segregation. Finally divide the quantity in the bucket by the amount originally poured into the sieve and multiply by 100 to attain the percentage passing through the sieve, record results. This result was recorded after the SCC had been transferred to moulds, with all trial mixes meeting the requirements. This was due to the inability to allow the mix 15 minutes rest time before pouring. Results are to comply with the criteria shown in Table 4.3 below.

Table 4.3: Sieve segregation resistance test parameters

Class	Segregation resistance in %
SR1	≤ 20
SR2	≤ 15

## 4.4.3 Compressive Strength Test

Compressive strength testing samples were prepared in accordance with the dimensions outlined in section 4.2.2. The EFNARC guidelines (2005) identified that SCC with similar water cement ratio to that of traditionally vibrated OPC will usually have a slightly higher compressive strength. This can be attributed to an improved interface between the aggregate and paste constituents due to a lack of vibration. In addition it was identified that the general strength development would be similar to that of SCC.

A total of three samples were prepared for each trial mix. The moulds were filled with the respective SCC mix and excess concrete struck from the top resulting in a level surface. Once cured the samples were tested via static uni-axial compressive load in the previously discussed compressive strength testing machine. Tests were conducted after seven and twenty eight days, with the result from the 28 day strength test using the average of two tested samples. Images of each cylinder corresponding to a trial mix have been provided in Figure 4.9 below. In this figure it is evident that the inclusion of RHA resulted in a significant colour change in the samples.

A restrained natural rubber capping mechanism was used for the specimen to assist in even load distribution; the rubber lining was positioned inside a steel cap and placed on top of the cylinder. Capping system conformed to the standards set out in Clause 6.3.1 of AS1012.9 (2014). The cylinder was then centred within the machine and the top platform

lowered to be positioned flush with the steel cap. Once secure a guard was placed in-front of the specimen as a safety measure. Finally the machine was programmed so that a static load was applied at a rate of 20 MPa per minute, as specified in AS1012.9 Clause 8(i). An example of the specimen positioned within the testing machine has been provided in Figure 4.4.



Figure 4.9: Cylinders specimen for each mix design

Following this setup procedure the test was initiated, resulting in the hydraulic ram applying the specified load onto the sample. The load was continually applied to the cylinder until failure occurred. The machines electronic analysis system was able to identify the point at which the sample failure occurred and provide data indicating this result. The test was repeated for each trial mix and results reported in Chapter 5, including the resulting compressive strength in MPa and images of the sample specimen following the testing procedure.

#### 4.4.4 Flexural Strength Test

In order to determine the flexural strength of each trial mix, a total of three SCC flexure test beams were developed in accordance with the dimensions outlined in section 4.2.2. The moulds were filled with the respective SCC mix and excess concrete struck from the top resulting in a level surface. Following the specimen curing procedure both seven and twenty eight day flexural strength testing was conducted through the application of a four point bending test. This test involves the application of a load at two points along the beam, resulting in a larger area of the specimen being exposed to the applied stresses. This provides a comprehensive representation of the factors contributing to the samples resulting failure. Figure 4.9 provides a representation of the apparatus set out for a fourpoint bending test. An image of each flexural beam following failure has been provided in section 5.5.

The initial equipment methodology required both bars supporting the flexure test beam to be located at a span of 308mm apart (AS1012.11, 2000). The two point load was also positioned above the beam at a distance of span/3 ( $\approx$  103mm) apart and the mechanism lowered onto the beam ensuring both the load and support bars were positioned flush against sample to allow for uniform load distribution across the beams surface. Finally a load ramp rate of 1 MPa per minute was programmed to be applied by the ram. These parameters were applied in accordance with AS1012.11 (2000). An example of the specimen positioned within the testing machine has been provided in Figure 4.5.



Figure 4.10: Visual representation of four point bending test (AS1012.11, 2000)

Following the specified preparation procedure the test was initiated, with the machine applying the indicated load at a constant rate until failure. The machines electronic analysis system was able to identify the point at which the sample failure occurred and provide data indicating this result which was then used to identify the samples flexural strength. The test method was repeated for each trial mix and results reported in Chapter 5, including the resulting flexural strength in MPa and images of each trial mix sample specimen following the testing procedure.

## 4.5 Chapter Summary

The resources provided for preparation of each sample have been listed, along with the specimen preparation and testing methodology for each stage of the research project. Preparation equipment required included a kiln, hammer mill and 65L tilt-drum mixer. The testing equipment used for experimental analysis includes an XRD, XRF and SEM analysis machine. The SEM analysis machine also utilised an EDS analysis tool, as discussed in section 4.2.2. Testing equipment for SCC fresh concrete testing included a slump cone, solid board with the required markings, a J-ring, sieve with 5mm aperture size, scales and a stopwatch. For hardened concrete testing an automatic hydraulic operated compression testing machine was used to identify compressive strength, and for testing of flexural strength a 4 point load bending test was applied using the equipment identified in section 4.2.2.

Preparation of samples involved attaining three cylinder and three rectangular beam moulds, dimensions identified I section 4.2.2. A total of six SCC mixes were developed, resulting in the creation of eighteen cylinder and flexural beam specimen. The procedure used for each mix design firstly required all materials to be collected and weighed to the nearest 1 gram. The mixing procedure required 90% of the water to be first added, along with the coarse and fine aggregates whilst the mixer was operating, following this the cementitious materials were added, and finally the superplasticiser and remaining water. The mixer then operated for a minimum of five (5) minutes before being stopped to assess the activation of superplasticiser and allow the mix to rest. Following an additional mixing time, fresh concrete testing was conducted followed by the mix being transferred into its respective moulds.

After 24 hours the moulds were cracked, and the samples were transferred to a curing tank to undertake the curing process. Following this process hardened concrete testing was conducted at seven and twenty-eight day time periods to attain compressive and flexural strength. Results for these tests have been provided in Chapter 5, along with results for the experimental analysis testing.

# Chapter 5

# Data Analysis & Results

## 5.1 Chapter Overview

This chapter provides an overview of the results obtained from elemental analysis, fresh concrete testing, and compressive and flexural strength tests. Results obtained by these testing methods identify both the quality of RHA used in this project and its application as a pozzolanic material, and also provides evidence identifying any resulting effects the increased replacement of RHA has on a SCC mix design. Additionally the findings assessed in this project provide further identification regarding the suitability of SCC as a construction material in replacing the application of OPC concrete. Summaries are provided from analysis of each specimen in order to identify the mix design properties of all samples and provide recorded data to quantify the projects research methodology.

# 5.2 Data Analysis

The data analysis requirements for this project required the application of varying investigative methods, largely depending on the type of test required. For experimental analysis procedures the predominant method of analysis was comparing the experimental results with that of prior research to identify whether the quality of RHA was acceptable. When analysing the fresh concrete, application of simple formula and existing SCC parameters was an acceptable technique in determining acceptance of the data. The compressive and flexural strength testing required application of multiple equations to analyse the results and provide an overall definitive strength. Aspects of each method have been clarified in the following section.

#### 5.2.1 Experimental Analysis

The experimental analysis component of this project required the application of XRD, XRF, SEM and EDS testing equipment. Each of these testing methods utilise a machine to conduct advanced material analysis in order to determine the quality of the produced Rice Husk Ash. The following technical information has been included to provide an overall understanding of the data analysis results.

### **XRD** Analysis

Application of the XRD analysis test results in the machine applying single wavelength x-rays to the sample and measuring the diffraction pattern resulting from the planes of atoms within the material. As such it can be used to measure the crystalline content of materials, and identify the crystalline phases present. Overall the primary requirement for analysis of these results is to compare with similar research results in order to determine the existence of amorphous silica. It has been identified that the amorphous silica will be evident as a broad peak located approximately at position two Theta  $(2\Theta) = 22^{\circ}$ . It was expected that broad nature of the peak suggests although this compound was recognised, the XRD analysis was unable to identify a potentially large amount of crystalline particles. The results provided in section 5.3 illustrate the output provided by this test.

In order to determine the percentage of amorphous silica and other identified elements within a sample based on the XRD output a manual weighted integration of the areas under all peaks may be applied. This can be through application of the Trapezoidal rule. However as an XRF analysis was later conducted this component was not required.

#### **XRF** Analysis

Due to the XRF analysis being conducted by the Bureau Veritas laboratory in Newcastle, very little technical understanding was required. Based on the results provided in section 5.3 it could be determined the exact quantity of each major and minor element within the sample. This test provided fundamental certainty regarding the chemical nature of the material.

#### SEM & EDS Analysis

These analysis techniques are used concurrently to identify the particle size and characteristics within a sample, and also provide further indication of the major constituents through x-ray analysis. This would provide a strong indication as to which phases were present within the RHA. Once again this experimental analysis method provides an output of results, on this occasion on the form of a high magnification, high resolution image of the material. The EDS also provides a visual representation of the major elemental constituents of the sample.

Observations of these results have been provided in section 5.3, along with identified material characteristics and resulting compound morphology. This data was essential in identifying the quality of the RHA product.

#### 5.2.2 Fresh Concrete Testing Analysis

Testing the fresh concrete parameters of SCC is a relatively simple process. The criteria that must be met have been provided in section 4.4.2, with the following equations being used to identify results. Testing results have been recorded in section 5.3.

#### Slump flow test –

Following the execution of methodology discussed in section 4.4.2, the average is to be taken of both perpendicular measurements to identify the final slump flow, whereby:

$$S_{flow} = \frac{D_1 + D_2}{2}$$

Where,

 $S_{flow}$  = the calculated slump flow of the trial mix (mm)

 $D_1 \& D_2$  = the distance both flow diameters as specified (mm)

#### T<sub>500</sub> slump-flow test –

The  $T_{500}$  test is conducted concurrently with the above slump flow test, the components of this testing method have been discussed in section 4.4.2. The time is initiated once the cone is lifted from the boards surface, and stopped once the 500mm line is broken. No calculations are required for this test.

#### J-ring passing ability test –

The j-ring test incorporates similar testing methodology to the slump flow test, with the addition of a j-ring, methodology discussed in section 4.4.2. Flow dimension calculations are to be conducted as shown in the slump-flow test explained above. In order to determine the height variation the following should be used:

$$H_{avg} = \frac{h_1 + h_2 + h_3 + h_4}{4}$$

Where,

 $H_{avg}$  = Average variation in height (mm)

 $h_n$  = Difference in height at each location (four total) (mm)

#### Sieve segregation test –

The sieve segregation test uses variations in weight as described in section 4.4.2 to identify the SCC trial mix's ability to resist segregation. The following equation has been applied to achieve this result:

$$SEG_{\%} = \frac{M_b}{M_a} \times 100$$

Where,

 $SEG_{\%}$  = total segregation of mix (%)  $M_a$  = Mass of concrete poured into sieve (g)

 $M_b$  = Mass of concrete sample passing sieve (g)

## 5.2.3 Uniaxial Compressive Strength Analysis

In order to determine the compressive strength of samples, each specimen is subject to a point load acting downward. Through completion of the test a result was provided indicating the maximum load applied at failure for each cylinder. This final load is then used to identify the concretes compressive strength in MPa.

The following equation was applied in order to identify the seven and twenty eight day compressive strength ( $\sigma_c$ ) of trial mix:

$$\sigma_c = \frac{F}{A_0}$$
 (AS1012.9, 2014)

Where,

 $\sigma_c$  = Compressive strength of specimen (MPa) F = Maximum load applied (N)

 $A_0 =$ Cross-sectional area of the cylinder (mm<sup>2</sup>)

$$A_0 = \frac{\pi \times d_c^2}{4}$$

Where,

 $d_c$  = Average cylinder diameter based on two measurements

Following the application of the above formula each cylinder will have a corresponding compressive strength measured in MPa. This result provides the overall seven day strength however for 28 day strength the resulting value will be the average of both 28 day test cylinders. The above methodology is to be applied for all six trial mixes, with a total of eighteen samples tested. Test results have been provided in section 5.3

#### 5.2.4 Flexural Strength Analysis

The four-point bending test was applied in order to accurately determine the flexural strength of each concrete sample. The test method requires the sample to be placed on two supporting bars at equal distance to the centre, with two loading bars placed above the sample equal distances from the centre. The methodology for conducting this procedure has been described in section 4.4.4. Through completion of the test a result was provided indicating the maximum load applied at failure for each flexural beam.

In order to calculate the resulting seven and twenty eight day flexural strength ( $\sigma_f$ ) of each trial mix the following procedure was applied:

$$\sigma_f = \frac{F * L * 1000}{b \times d^2}$$
(AS1012.11, 2000)

• For a four point bending test where the loading span is one third that of the support span, positioned in the middle third of the specimen

Where,

- $\sigma_f$  = Flexural strength of beam (Modulus of Rupture) (MPa)
- F = Load applied at failure (kN)
- L = Length of support span (mm)
- b = Width of test beam (mm)
- d = Depth of test beam (mm)

Following the application of the above formula each beam will have a corresponding flexural strength measured in MPa. This result provides the overall seven day strength however for 28 day strength the resulting value will be the average of both 28 day test beams pertaining to the respective mix. The above methodology is to be applied for all six trial mixes, with a total of eighteen samples tested. Test results have been provided in section 5.3
# 5.3 Experimental Analysis Results

The following section provides a summary of the experimental results attained from this research project. Along with the quantitative results, an in-depth analysis has also been provided that identifies any challenges encountered or issues with the experimental method throughout the testing process. Finally the chapter summarises all key findings from each section in order to provide a comprehensive overview of the research outcomes.

### 5.3.1 XRD Analysis Results

The XRD analysis, conducted at the University of Newcastle, provided an analysis of the RHA sample that was essential in determining whether there was silica present in amorphous form. Results from the XRD analysis have been provided in Figure 5.1 below. Analysis was conducted for both ground and unground RHA, in this instance the ash was manually ground using a mortar and pestle to achieve a fine homogenous powder.



(a) XRD analysis result for uncrushed sample



(b) XRD analysis result for ground sample



(c) Overlay comparing the two methods of sample preparation

Figure 5.1: XRD analysis results, (a) analysis from testing of unground sample, (b) analysis for ground sample (c) overlay of results comparing the two methods

Table 5.1: Identified phases determined from XRD analysis, (a) unground sample (b) ground sample

Table (a): Matched elemental phases from unground sample

Ref. Code	Compound Name	Chemical Formula	Chemical Name	Mineral Name
00-041-1476	Potassium Chloride	K Cl		Sylvite, syn
04-015-2405	Carbon	С		
04-008-7642	Silicon Oxide	Si O2		Cristobalite, low

Table (b): Matched elemental phases from ground sample

Ref. Code	Compound Name	Chemical Formula	Chemical Name	Mineral Name
04-013-0293	Carbon	С		
00-041-1476	Potassium Chloride	K Cl		Sylvite, syn

Results from this procedure have identified that the proposed firing method was successful in procuring amorphous silica. Initially it was determined that the results appeared semi-quantitative at best due to a diffraction count less than anticipated. Upon further investigation it was determined the lower count quantity was likely due to the existence of high percentages of amorphous silica, as this compound would not cause x-rays to diffract as effectively.

The main phases illustrated in Table 5.1 (a) and (b) have identified the existence of Carbon and Potassium Chloride in crystalline form, with (a) also identifying the existence of crystalline Silicone Oxide. The existence of Potassium Chloride is a result of the rice production process, as this compound is primarily used as fertiliser. Through a brief literature review it was determined that Potassium chloride does not attack cement, and aside from some minor retardation in setting time there was minimal negative impact on the short or long term characteristics of concrete. Furthermore, Venkateswara (2011) identified that with increasing concentration of the compound, there was a subsequent increase in the concretes compressive and tensile strength. Further research regarding this compound was not included in the scope of this project.

The XRD outputs provided in Figure 5.1 (a) and (b) identify the existence of a broad peak at the approximate position of  $2\Theta = 22^{\circ}$ . This value is consistent with the figures identified in section 2.3.1 for the location of amorphous silica, and reiterated in research conducted by Abu Bakar (2016) and Music (2011) regarding the identification of amorphous silica using XRD analysis. Figure 5.1 (c) provides an overlay of the two resulting XRD patterns. As identified in this figure there is not a significant difference in result between the two methods, with the ground result providing a slightly higher quantity of diffraction counts than the unground sample. This result suggests that the unground sample was packed successfully in order to avoid preferential orientation. The existence of minimal sharp peaks throughout both samples indicates promising results in confirming a very high percentage of silica. If there were to be large proportions of other elements there would be additional sharp peaks visible.

It was also determined that Silicon Oxide was found in the unground sample, whilst there were not sufficient crystalline quantities of the compound for it to be identified in the ground sample. It was assumed that this outcome was a result of the elements being found in very low quantities, and the grinding process resulted in the reduction in any preferential orientation of these particles. Overall, this result indicates that RHA manufactured with the previously discussed firing method produced a suitable end product with sufficient quantities of amorphous silica. As stated in section 4.4.1 the percentage of amorphous silica and other identified elements within a sample can be determined based on the XRD output through application of the Trapezoidal rule. However, as an XRF analysis was later conducted this component of the XRD analysis was not required.

### 5.3.2 SEM / EDS Analysis

The purpose of an SEM / EDS analysis is to identify the main elements present in a sample, the results from this test provide a strong indication of which phases are present within the procured RHA. Through conducting an area scan of the sample vital information can be attained in order to identify both the particle characteristics of the material and the quantity of Silicon and Oxygen elements present. In this instance the EDS scan will provide an overlay image of the SEM analysis identifying the existence of Silicon in red, and Oxygen in cyan. This further clarifies the elemental constituents of the broad peak identified in the XRD analysis located at  $2\Theta = 22^{\circ}$ . The SEM analysis was conducted with a RHA sample that had not been ground; as such the particle size identified was greater than the advised maximum of 75 micron. Results from the SEM analysis have been provided in Figures 5.2 (a, b & c), whilst results from the associated EDS scan have been provided in Figure 5.3.



(a) SEM analysis at 103x magnification – Carbon coating not applied scale in LH corner 100 micron



(b) SEM analysis at 100x magnification – Carbon coating applied scale in LH corner 100 micron



(c) SEM analysis at 2000x magnification, without Carbon coating (LHS) and with Carbon coating (RHS), scale in LH corner 10 micron

Figure 5.2: SEM analysis result, (a) 103x magnification without C coating, (b) 100x magnification with C coating, (c) 2000x magnification with and without C coating as detailed

Results from the above figures provide valuable insight into the particle characteristics of the RHA sample. The Carbon coating applied to the SEM samples was recommended as it improves the quality of EDS analysis, however there is a likelihood of bias in the sample due to the Carbon's presence. As such both coated and uncoated samples were provided.

The particle shape observed in the above results detailed in Figure 5.2 illustrate that the size and shape of the RHA particles is consistent with the results provided by Sua-Iam (2014) and Tahn Le (2016). Due to the porous shape of the RHA sample it can be proposed that trial mixes with a higher percentage of the material will result in an increased water demand. This statement will be reviewed further at a later stage. Due to the samples identifying components with a particle size greater than 100 micron, it was determined that grinding of the manufactured RHA was essential in attaining a suitable pozzolanic material.

Figure 5.2 (a) & (b) illustrate a sample analysis of RHA both with and without the Carbon coating. Analysis of these two figures identifies that the current unground RHA has a significant variance in particle size, with larger particles measuring as much as 300 micron. This result is not acceptable for use as a pozzolanic material, as documented by Meryman (2009) pozzolanic materials with a high particle size are unable to react as effectively and function as a hydration enhancer. This was also determined by Antiohos (2014) who identified that the particle size and surface area of RHA displays a direct correlation to the final performance of concrete's strength. In this instance trial mixes

conducted with RHA displaying a greater percentage of fine particles achieved higher compressive and flexural strength results.

The particle size and physical characteristics are provided for observation in Figure 5.2 (c). In this instance it is apparent each particle is characterised by a rough angular shape, and presents a porous structure. It should also be noted that there was no identification of any crystalline particles within the sample. Tahn Le (2016) identified that the angular shape of RHA particles in SCC would result in a significant increase in plastic viscosity and segregation resistance. This statement was supported by Sua-Iam (2014), who identified that when RHA is added to a SCC mix the materials angular shape results in greater friction between particles. Thus the angular shape and higher porosity of RHA results in not only increased water retention, but also less free water and decreased flowing ability of the SCC. Contradicting Tahn Le (2016), Sua-Iam (2014) concluded that this may result in the mixture displaying levels of segregation and decreased workability. Further SCC testing will provide sufficient results to identify which statement is most applicable to this research project.

The EDS provides an x-ray spectrometry analysis of a sample through application of a concentrated beam of electrons as utilised in the SEM analysis. The secondary electron dispersion resulting from a SEM analysis provides a compositional contrast identifying each element and its dispersion. The identification of each particles elemental composition can then be identified similarly to an XRD analysis, in which the phase and proportionate counts are recognised. However this analysis method is not limited by the existence of crystalline particles. Results from an EDS analysis of the RHA sample have been provided in Figure 5.3.



(a) EDS Analysis without Carbon Coating (1000x magnification)



(b) Identification of phases by element (without Carbon coating)



(c) EDS Analysis with application of Carbon Coating (1000x magnification)



(d) Identification of phases by element (with application of Carbon coating)

Figure 5.3: Results from EDS Analysis, (a) 1000x magnification without Carbon coating, (b) identification of phases by element, (c) 1000x magnification with application of Carbon coating, (d) Identification of phases by element

Results displayed in Figure 5.3 provide an EDS analysis of the SEM results provided in Figure 5.2, represented at a magnification of 1000x the original resulting image. When the EDS is utilised in conjunction with an SEM analysis, the representative elements of an image are identified as individual colours. In this case Silicon, illustrated in red, and Oxygen, illustrated in cyan. As discussed previously, the application of a Carbon coating is recommended for improved EDS analysis results, however with potential bias due to the existence of Carbon atoms.

Figure 5.3 (a) & (b) illustrate the results provided for the sample prepared without Carbon coating. In this analysis the EDS images shown in Figure 5.3 (a) illustrate a dense array of both Silicon and Oxygen atoms. This result suggests that the primary constituent elements of this material were both Silica and Oxygen. Figure 5.3 (b) provides the associated phase identification of the sample which confirmed the existence of a comparatively high quantity of Silica phases. The second dominant peak in this figure has been identified as Oxygen, which is also represented quite significantly. Other smaller peaks represented in this sample include Potassium (K), Chloride (Cl) and also Magnesium (Mg). The K and Cl particles had been identified previously in the XRD analysis, with the appearance of Mg likely due to its potential application in fertiliser during the rice production process.

Figure 5.3 (c) & (d) identify similar results to that of (a) and (b), however this sample was treated with a Carbon coating prior to the testing process. The EDS analysis identified in Figure 5.3 (c) confirms the presence of Silica and Oxygen in exceptionally high proportions. Through comparison of this result with the EDS image shown in Figure (a) it is possible to identify the existence of fewer dark areas and an increased intensity of atoms identified throughout the image. Therefore it can be confirmed that the application of a Carbon coating assists in producing a better quality EDS output. Figure 5.3 (d) provides the associated output of this test identifying the presence of various phases. In this particular instance the presence of intense, sharp peaks of Silica and Oxygen replicate the previously identified results. However in this instance other minor constituents identified include only Potassium (K) and Carbon (C). Although the presence of K was expected, C identified within the sample was likely a result of the Carbon coating applied prior to testing. Chloride atoms were likely not identified as the elements phase count was not significant enough to be identified.

Results provided by the EDS analysis confirm the existence of Silica and Oxygen as the primary constituents of the RHA material. Through identification of particle characteristics represented by the SEM analysis it was also confirmed that there did not appear to be any identifiable crystalline particles within the sample. This result serves to validate the findings provided in the previously conducted XRD analysis. Furthermore, it is possible at this stage to comment with relative certainty that amorphous silica forms the main constituent of the RHA material that was developed.

### 5.3.3 XRF Analysis

The XRF analysis was not originally intended for application in this research project, however as the laboratory utilised for grinding of the RHA also provided an XRF machine, it was decided that this procedure would be a valuable addition to the research. The result for XRF analysis has been provided below in Figure 5.3.4. As illustrated in this figure the primary constituent of the RHA material has been identified as Silicon in the form of SiO<sub>2</sub>. This result confirms the findings determined by Ramezanianpour (2009) and Sua-Iam (2014), in which amorphous silica was deemed the primary constituent material within their respective RHA samples. This result provides overall confirmation that the RHA used in this research project is of sufficient quality, with a high proportion of reactive silica, low particle size and minimal quantities of other elements that may affect the strength development of SCC. Refer to Figure 5.4 for details regarding the materials overall elemental composition. Minor constituent materials identified in the XRF include Potassium, Phosphorous, Calcium and Sulphur, along with some other elements in very minor or negligible quantities. This result also provides further justification regarding its acceptability as a sustainable pozzolanic material, and additionally highlights the argument that the components of RHA pose significantly less contamination threat through concrete leaching to that of FA or Blast Furnace Slag which both contain hazardous compounds.

NQ070 - Loss on Ignition Ignition Loss (800 Deg.C) % -100 2.40NQ797 - Analysis of Materials-fused bead XRF Aluminium as Al2O3\* % 0.02 0.09 Barium as BaO\* % 0.02 nd Calcium as CaO \* 0.02 0.39% Copper as CuO \* % 0.02 0.02 Total Iron as Fe2O3 \* 0.02 0.06 % Potassium as K2O \* 0.02 3.02 % Magnesium as MgO \* 0.05 0.21 % Sodium as Na2O \* 0.05 0.07 % Phosphorus as P2O5 \* % 0.02 0.32 Silicon as SiO2 \* 0.02 93.2 % Sulphur as SO3 \* 0.21 % 0.02 Zinc as ZnO \* % 0.02 nd

Figure 5.4: Result from XRF Analysis

As illustrated above, the XRF analysis identified Silicon in the form of  $SiO_2$  or in reference to the previously reviewed results, amorphous silica. This value indicates that the silica produced through the combustion process was of a high quality and therefore suitable for use in this project. Due to the results provided by the above experimental analysis methods it was determined that the RHA compound developed in this research project could be utilised as a pozzolanic material. Therefore it was determined that application in SCC could be conducted.

# 5.4 Fresh Concrete Testing Results

Fresh concrete testing was conducted in accordance with the parameters set out in section 4.4.2. In order to confirm whether the quality of SCC produced was suitable it was essential that all previously identified parameters had been satisfied. The following section provides the results of each testing component and also specifies any observations regarding the quality of SCC produced during these stages. Methodology used for testing of fresh SCC follows the EFNARC guidelines (2002 & 2005), all identified testing methods have been adhered to and acceptance criteria attained. Table 3.1 identifies the suitable applications for each SCC class as specified in the EFNARC guidelines (2005). An initial control was developed and also applied for this test, however as the sample was not set after a 24 hour period this mix was discarded and a new control was developed with a HRWR content reduced from 1.3% to 1%. This modification was applied as a result of industry advice from the Technical officer at the batching plant where trial mixing was conducted. Results for the initial trial mix have been included for reference.

### 5.4.1 Slump-flow Test

The slump-flow test has been applied in order to determine the flowability of each mix design sample. The slump-flow class intended to be achieved was SF2, which required a flow between 660mm and 750mm. With reference to Table 3.1, it was determined that class SF3, with a flow between 760mm and 850mm, was also applicable as an upper range. Table 5.2 below provides results from the slump-flow test.

Slump-flow Test (660 <sf>750)</sf>						
	D1	D2	Slump Elow (mm)			
Trial Mix No.	(mm)	(mm)	Siump Flow (mm)			
Trial 1	750	745	747.5			
Mix 1 - Control	735	735	735			
Mix 2 - RHA10	710	710	710			
Mix 3 - RHA15	705	700	702.5			
Mix 4 - RHA 20	700	700	700			
Mix 5 - RHA 25	695	690	692.5			
Mix 6 - RHA30	690	690	690			

Table 5.2:	Slump-flow	Test Results
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Results from the slump-flow test indicate that all mix designs passed the required parameters for SCC class SF2. This included the initial Trial mix which was later discarded. All measurements were rounded to the nearest 5mm for ease of measurement in site conditions. Overall it can be determined that the addition of RHA resulted in a decreased slump-flow. This was an expected result cited in prior research by Sua-Iam (2014) which indicated the addition of RHA would result in greater water demand and friction between particles and therefore a decrease in flowability. From initial observations it does not appear that the increase in water demand is significant enough to impact on the overall mix design. Some excess free water was evident in the control mix however it was observed that this factor reduced with the increased amount of RHA that was added. This supports claims that the porous nature of RHA reduces the quantity of free water and may subsequently result in an increase in water demand.

### 5.4.2 T<sub>500</sub> Slump-flow Test

The  $T_{500}$  slump-flow test was applied in order to determine the viscosity of each trial mix. For this research project the  $T_{500}$  test was applied concurrently with the slump-flow test, with a circle of 500mm diameter being placed central to the base plate. In this instance a lower time indicates greater viscosity. Although parameters illustrated in Table 3.1 indicate that the desired flow for SCC used in slabs is less than or equal to two (2) seconds, the EFNARC guidelines (2002) indicate that a range between two and five seconds is suitable for civil engineering purposes. Therefore the target range for this component of the test will be less than five (5) seconds. Results from this testing have been provided in Table 5.3 below.

T500 Slump-flow Test					
Trial Mix No.	Time (sec)				
Trial 1	1.63				
Mix 1 - Control	1.85				
Mix 2 - RHA10	2.46				
Mix 3 - RHA15	2.52				
Mix 4 - RHA 20	2.86				
Mix 5 - RHA 25	3.48				
Mix 6 - RHA30	3.67				

Results from the  $T_{500}$  test indicate that an increase in RHA replacement results in a subsequent decrease in viscosity of the concrete, indicated by an increased flow time. This was most likely due to both an increase in angular particles in the mix resulting from the RHA and also an increase in water demand and a subsequent reduction in free water content. The findings illustrated in Table 5.3 indicate that the control sample falls in SSC class VF1, while due to an increase in plastic viscosity, the samples with RHA fell in SCC class VF2. Applications of this class have been provided in section 5.4.

### 5.4.3 J-ring Test Results

The application of a J-ring test provides vital information regarding the trial mix's ability to pass through reinforcement and around obstruction without potentially resulting in blockages or voids. The requirements for passing ability, identified in section 4.4.2, indicate the maximum difference in height between the internal and external area of the j-ring is to be no greater than 10mm. The second requirement involves a comparison of flow between this test and the previously determined slump flow, this requires a difference in flow less than 50mm between the two results. Results from this test have been provided in Table 5.4 below.

J-ring Passing Ability Test						
	D1	D2	Flow	H1	H2	Difference
Trial Mix No.	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)
Trial 1	750	745	747.5	1.39	0.78	0.61
Mix 1 - Control	735	730	732.5	1.4	0.82	0.58
Mix 2 - RHA10	705	705	705	1.42	0.8	0.62
Mix 3 - RHA15	700	700	700	1.4	0.77	0.63
Mix 4 - RHA 20	695	695	695	1.42	0.78	0.64
Mix 5 - RHA 25	695	690	692.5	1.4	0.74	0.66
Mix 6 - RHA30	690	685	687.5	1.43	0.78	0.65

Table 5.4: J-ring Test Results

Results provided in Table 5.4 indicate that the slump-flow was reduced by a small quantity when the j-ring was included. Due to the relatively small variations in this result it can be suggested that human error in interpreting the tape measure could have contributed to this result. The other likely cause was that some of the SCC material was built up within the j-ring due to the existence of this barrier. When analysing the average

heights both internally and externally it appears that there is no significant variations resulting from the increased quantity of RHA. Additionally it was determined that there was no instance in which the height difference was greater than 10mm. This result identifies that each of the mix designs met the requirements for passing ability and therefore were suitable for further testing.

### 5.4.5 Sieve Segregation Test Results

The sieve segregation resistance test is applied in order to assess the resistance of SCC to segregation. This is essential in determining whether the trial mix design proportions are sufficient in providing a material suitable for application in both civil and structural development. The sieve segregation test required the concrete mix to sit for 15 minutes before the test could be conducted. As such, following the acceptance of each SCC mix based on the previously identified criteria, 5L of the concrete was set aside in a bucket to rest while the moulds were prepared. The segregation resistance test provided the final justification as to whether the produced SCC was acceptable, however due to the extended rest time it was not possible to conduct it concurrently with the other previously conducted tests. Results from this test were to conform to the parameters listed in Table 4.3, which required a maximum segregation resistance less than 20% for class SR1 and 15% for class SR2. Results provided in Table 5.5 below.

Sieve Segregation Resistance Test							
Trial Mix No.	Ma (kg)	Mb(kg)	Segregation (%)				
Trial 1	4.91	0.52	10.59				
Mix 1 - Control	4.78	0.88	18.41				
Mix 2 - RHA10	5.12	0.75	14.65				
Mix 3 - RHA15	5.15	0.72	13.98				
Mix 4 - RHA 20	5.19	0.71	13.68				
Mix 5 - RHA 25	5.25	0.74	14.10				
Mix 6 - RHA30	5.31	0.67	12.62				

Table	5 5.	Sieve	segregation	test results
Iaure	5.5.	SIEVE	segregation	lest results

This result indicates that each of the trial mixes was in a stable enough condition to resist segregation. Trial mix 1 provided the lowest result; however through observation of this mix design it was determined that this is likely attributed to the increased quantity of HRWR in the mix. The higher percentage of this admixture resulted in a significant

reduction in the mix's workability after the tests fifteen (15) minute standing time. Therefore it can be identified that although this result for Trial mix 1 appeared suitable, the longer term effects of superplasticiser resulting in an extended setting time coincided with the mix not being practical for use. The remaining trial mixes resulted in higher, but suitable results. It was observed that the control mix resulted in a higher segregation percentage which again comes back to previous comments indicating slightly higher instances of observable free water in the control mix than that of the RHA samples.

Overall it appears the segregation resistance with inclusion of RHA did not differ significantly after the initial reduction. Repetition of this test may provide further indication regarding the overall effects RHA has on segregation resistance. It does not appear this method is particularly suitable for application in field testing, as such any future development toward the application of SCC should focus on identifying a suitable alternative that can be readily applied with a high degree of accuracy and reduced testing duration.

# 5.5 Hardened Concrete Testing Results

## 5.5.1 Compressive Strength Results

Compressive strength analysis was conducted on each specimen in accordance with AS1012.9 – Methods of testing concrete: Method 9 – Compressive Strength Tests (Standards Australia, 2014). The machine used for this testing process was programmed to apply a continuous load downward on the cylinders until failure. This was conducted at a ramp rate of 20 MPa per minute as previously stated. Through application of pressure by the machines hydraulic ram it is possible to identify the point at which the concrete sample fails. The machine is able to provide data in the form of MPa per minute, however as this data does not provide any analysis methodology beyond this, it was omitted from the report. The maximum load identified at failure is used to determine the samples compressive strength ( $\sigma_c$ ). Figure 5.5 identifies the compressive strength for each mix design as determined through conducting one test for 7 day strength and two tests for 28 day strength. The average was taken of the latter to allow for abnormalities in a particular samples result or for premature failure such as cap failure in the specimen.



Figure 5.5: Results from Compressive Strength Testing

The above figure identifies that the control specimen achieved a compressive strength of 40.4 MPa. This result was consistent with the target strength identified in the previously calculated mix design. The strength development between 7 day and 28day strength observed in the control was relatively similar to the strength development apparent in each of the trial mix designs, regardless of their RHA replacement percentages. However it is evident through review of the above data that inclusion of RHA in replacement of OPC results in decreased strength development.

The most likely reason for an overall decline in strength is due to the reduction in Calcium Hydroxide  $(Ca(OH)_2)$ , the reactive ingredient in cement. As discussed in section 2.2.1, when a pozzolanic material such as RHA is added to a mix design it reacts with the excess  $Ca(OH)_2$  following the cements hydration reaction. This forms additional quantities of C-S-H gel, the compound that contributes significantly to the strength properties of concrete. When the cement content is reduced and quantity of pozzolan is increased there is a reduced amount of leftover  $Ca(OH)_2$  available as a reactive ingredient. As such the remaining amorphous silica is only able to serve as a filler in the mix, and therefore does not successfully contribute to strength development. One potential mitigation method regarding this component is the use of  $Ca(OH)_2$  (hydrated lime) as an additive. This would provide additional components for RHA to react with.

	Mix Design 1 - Control							
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.7	100.4	100.55	7940.61	199	276.2	34.8	
Cylinder 28	100.3	100.3	100.3	7901.18	197	332.2	42.0	
Cylinder 28	100.7	100.7	100.7	7964.32	198	308.4	38.7	
	Mix Design 2 - RHA10							
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.6	100.6	100.6	7948.51	200	169.6	21.3	
Cylinder 28	100.1	100.3	100.2	7885.43	199	205.1	26.0	
Cylinder 28	100	100.4	100.2	7885.43	198	221.8	28.1	
	-		Mix Des	ign 3 - RHA15	-		•	
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.7	100.6	100.65	7956.42	200	129.7	16.3	
Cylinder 28	100.5	100.1	100.3	7901.18	199	164.1	20.8	
Cylinder 28	100.6	100.7	100.65	7956.42	198	173.4	21.8	
	-		Mix Des	ign 4 - RHA20			1	
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.9	100.7	100.8	7980.15	200	83.3	10.4	
Cylinder 28	101	100.6	100.8	7980.15	197	108.2	13.6	
Cylinder 28	100.7	100.8	100.75	7972.23	198	128.5	16.1	
			Mix Des	ign 5 - RHA25				
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.5	100.6	100.55	7940.61	200	89.1	11.2	
Cylinder 28	100.6	100.3	100.45	7924.83	201	142.5	18.0	
Cylinder 28	100.4	100.6	100.5	7932.72	200	127.5	16.1	
	-		Mix Des	ign 6 - RHA30			1	
	D1 (mm)	D2 (mm)	Avg D (mm)	Area (mm^2)	H (mm)	Load (kN)	Strength (Mpa)	
Cylinder 7	100.9	100.7	100.8	7980.15	198	73.2	9.2	
Cylinder 28	101	100.6	100.8	7980.15	201	106.8	13.4	
Cylinder 28	100.7	100.8	100.75	7972.23	198	114.8	14.4	

Table 5.6: Tabulated results from compressive strength testing

Table 5.6 provides results from each mix designs compressive testing process. Utilising the formula provided in section 5.2.3, in accordance with AS1012.9, the average diameter was used to calculate the area of each specimen. The final calculated load was divided by the cross-sectional area of the cylinder in order to identify the final compressive strength of each mix design. Each concrete sample was required to meet the specified dimension parameters identified in AS1012.9 (2014). This identified that the diameter at any cross-section was not to deviate from either end diameter by more than 2mm, and also that the height was to be no less than 1.95 times the diameter. These criteria were satisfied.

As identified, an overall analysis of the above results indicates that an increase in RHA replacement results in a subsequent decrease in compressive strength. This is an expected result and reflects the findings identified by other researchers, such as the research papers presented by Tahn Le (2016) and Chopra (2015). In previous studies conducted by these two researchers it was observed that following an initial lower strength than the control sample, later age compressive strength testing (at 56 days) identified that samples with replacement percentages of RHA actually increased in strength development and in some instances resulted in strengths that outperformed that of an OPC mix. This result can be attributed to the late age strength development resulting from pozzolanic materials. As discussed in section 2.2.1, due to the porous nature of RHA resulting in water absorption it is able to continue reacting using the pozzolanic effect to provide later age strength development.

Also observed in Figure 5.5, it appears mixes 4 and 5 do not follow the expected trend in strength reduction. In this instance the strength development of mix 4 is less than anticipated while mix 5 presents a higher strength. This result indicates there may have been an error in either the sample procurement process, or in regards to sample 4, potentially a cap failure in testing that resulted in premature specimen failure. Errors in procurement may include factors such as incorrect measurement of constituent materials, however due to the procedures applied throughout the procurement stage it is more likely that there were inconsistencies in filling the samples. Due to inexperience in filling moulds, there may potentially have been errors in placement. Errors such as this can result in impacts such as voids or segregation within the sample mould.



Figure 5.6: Typical failure modes of cylinders

Figure 5.6 provides an example of typical failure modes identified in each cylinder for reference. Through analysis of the above images it is evident that although SCC is intended to provide a material capable of consolidating under its own weight with no voids, there appear to still be voids evident within all samples. This factor can be identified as a significant contributor to premature specimen failure. Any voids present within a sample provide a weaker path of travel for failure to occur. Also identified above is the shear failure that resulted from sample segregation. This failure was found in a 28 day sample of Mix 4. As mentioned previously, it is evident that a large portion of aggregate has been placed at the bottom of the specimen mould. This was likely a result of unintended error in placement when making this particular sample. The cone failure observed for Mix 1 was the preferred failure method, however upon further investigation of the failed specimen, there was evidence to suggest the presence of voids. Other methods of failure identified include shear and cap failure. Throughout the extent of this testing process there were instances of all three failure methods. The only observed instance of segregation appeared in the sample for Mix 4.

Overall it appears many of the samples suffered premature or poor methods of failure. In addition to the possible causes listed above, other factors contributing to sample failure include the impact of fines coating coarse aggregate, further assessed in section 5.5.2, and insufficient quantities of coarse aggregates in the SCC mix. This may have resulted in weaker concrete samples due to the existence of fewer obstructions in the samples failure path. The results provided through compressive strength analysis serve as an example of the many factors contributing to the strength and failure mechanisms of concrete.

### 5.5.2 Flexural Strength Results

The flexural strength analysis conducted on each sample utilised a four point bending test to identify the maximum flexural load that could be applied before specimen failure occurred. Testing parameters were prepared as detailed in sections 4.2.2 and 4.4.4 in order to comply with AS1012.11 (2000). Through application of the equation specified in section 5.2.4 it was then possible to apply the identified maximum load, span length and beam dimensions to determine the overall flexural strength ( $\sigma_f$ ) of each sample. This process was conducted to identify both 7 and 28 day flexural strength of each trial mix design, in which the average was taken of the latter. Figure 5.7 below identifies the results from this process.



Figure 5.7: Results from Flexural strength results

Results illustrated in Figure 5.7 indicate that the control sample achieved a 7 day flexural strength of 5.2 MPa, and a 28 day flexural strength of 6.45 MPa (actual results attained from Table 5.7 below). This result is consistent with the expected flexural strength resulting from 40 MPa concrete. As such it can be ascertained that the mix design developed for this research project was sufficient in providing an adequate strength SCC, or more accurately a concrete that met the strength parameters identified through the mix design methodology process.

Through analysis of the above figure (5.7) it can be ascertained that a similar trend has occurred to the previously discussed compressive strength results. In this case there is a decrease in strength resulting from the partial replacement of RHA, however the initial strength reduction is not as significant. Also similar to the previous result it appears mix 4 did not achieve a flexural strength as high as what was expected, and concurrently mix 5 attained a relatively high result by comparison. Additionally it appears that the strength development for mix's 2 and 3 are quite significant between the 7 and 28 day samples. The most likely reason behind this result is the presence of voids within the 7 day samples, which would negatively affect the final strength of the beam. Initial comparison of the results provided in Figure 5.7 with that of Figure 5.6 indicate that Flexural strength is not affected as significantly as compressive strength with the addition of RHA. Tabulated results for each of the sample tests have been provided in Table 5.7.

Mix 1 - Control								
	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	99.85	100.3	308	17	5.2			
Beam 28	100.05	100.2	308	21.8	6.7			
Beam 28	99.85	100.15	308	20.05	6.2			
Mix 2 - RHA10								
	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	98.1	100.45	308	10.4	3.2			
Beam 28	100.1	99.9	308	17.83	5.5			
Beam 28	100.2	99.45	308	19.2	6.0			
		Mix	3 - RHA15					
	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	101.65	98.25	308	7.3	2.3			
Beam 28	100.35	99.35	308	12.1	3.8			
Beam 28	100.15	99.9	308	14.4	4.4			
		Mix	4 - RHA20					
	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	99.85	100.3	308	7.9	2.4			
Beam 28	100.3	99.6	308	9.062	2.8			
Beam 28	100.2	99.9	308	10.309	3.2			
		Mix	5 - RHA25					
	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	100.25	100.3	308	9.2	2.8			
Beam 28	101.35	99.65	308	11.485	3.5			
Beam 28	100.7	100.85	308	12.5	3.8			
		Mix	6 - RHA30					
Mix 6 - RHA30	b (mm)	d (mm)	Span (mm)	Load (kN)	Strength (Mpa)			
Beam 7	100.55	100.6	308	7.33	2.2			
Beam 28	100.55	99.65	308	8.16	2.5			
Beam 28	100.45	100	308	8.91	2.7			

Table 5.7: Tabulated results from Flexural strength testing

The above test results provide the data required in order to then calculate the required strength of each SCC mix. The testing machine utilised for this section of the report was capable of providing data in the form of MPa per minute, however as this data does not provide any analysis results of value it was omitted from the report. Further observations regarding the cause of each samples failure are possible through analysis of each test

specimen following its failure. The information obtained through this process was essential in providing further research development regarding how RHA affected each SCC sample. Figure 5.8 provides a sample of each mix's flexural beam sample after failure.



(a) Mix 1







(d) Mix 4

(e) Mix 5

(f) Mix 6

Figure 5.8 Sample flex beams following testing

Analysis of the above test samples (Figure 5.8) provide key observations in regards to the possible failure methods of each sample. When interpreting each sample it is first apparent that a line of segregation appears, this is most evident in Mix 1 (a) where the top 15mm of the sample does not contain coarse aggregate. There also appears to be a fine line at the top of the sample, this is a result of bleed water that has set on the surface. As indicated previously, the incorporation of RHA in SCC results in a decreased quantity of free surface water. This is clearly evident through comparing the results provided in mixes 1 through 6. As illustrated above, the segregated section apparent in Mix 1 (a) appears to decrease in direct correlation with increasing percentages of RHA. It can also be observed that the line of bleed water previously evident in Mix 1 (a) subsides and eventually disappears. Interpretation of this result indicates that RHA provides an added benefit within a SCC mix by reducing the existence of additional free water present.

Also evident in Figure 5.7 is the existence of voids in the samples. It is possible to identify the voids in each image, apparent as dark spots within each sample. The sample with the largest quantity of voids is Mix 4 (d). This can likely be attributed as the cause of premature failure in the specimen. Additionally the concept of aggregate debonding was also suggested. Due to the storage of aggregates in an open environment (batch plant), there was an increased potential for fines to contaminate the coarse aggregate mix. The addition of fine particles coating the aggregate may have resulted in the produced C-S-H gel being unable to effectively bond the aggregates together, and subsequently causing premature failure of the specimen.

As identified in section 5.5.1, although SCC is designed to consolidate under its own weight it appears there is an underlying issue regarding the development of small air voids in the concrete mix. This is a result that would traditionally be avoided through vibration of ordinary concrete however in this instance further research should be conducted in determining suitable methods of reducing this negative factor.

Overall through conducting flexural strength testing on each specimen it was possible to identify the strength development of each mix and interpret the impact RHA has on the properties of SCC. The primary key point of interest identified in this portion of the project was the existence of voids, it is believed this component is a potentially avoidable contributing factor relating to the premature failure of SCC specimen.

# 5.6 Cost Analysis of SCC with RHA

In order to determine the appropriateness of RHA as a pozzolanic material it is essential to identify whether it is economically feasible. Despite many of the advantageous properties of SCC, one primary factor limiting its use in construction is the costs associated with increased quantities of admixture. As compounds such as HRWR and VMA's are essential in obtaining the properties required for SCC, it is necessary to reduce costs where possible through modification of other components of the mix (Sua-Iam, 2014). Therefore it is essential to reduce the quantity of cement, as this is a primary cost of concrete, and replace this compound with a cost-effective alternative incorporating more economically beneficial materials. Khan (2012) identified that increasing demand for cheap pozzolanic materials has resulted in push toward the utilisation of agricultural waste.

In order to determine the feasibility of RHA, it is first necessary to identify the cost of each supplementary constituent. In this case due to the necessity of including admixtures, coarse and fine aggregates and water, these parameters have not been assessed. Therefore the primary constituents for which cost was evaluated are cement, FA and RHA. Table 5.8 provides a summary of these costs in terms of Australian dollars (\$) per tonne.

Table 5.8: Cost of Cementitious Materials

Unit Cost of Cementitious Materials						
MaterialsOPC (per tonne)FA (per tonne)RHA (per tonne)						
Unit Cost (\$)	\$140	\$100	\$60			

The above Table has been provided in order to determine an approximate price comparison between each material. The cost identified for both OPC and FA has been determined through identification of current industry trends to provide an accurate estimate. The cost of RHA was calculated through identifying that RHA is currently a waste product with no substantial value and no current market demand. Therefore it was essentially determined that the primary costs associated with the application of RHA would be from transport of the product to various locations. If an environmentally method of burning the husk was applied at a rice mill in order to burn husk for energy, the waste product would be extremely cheap to acquire at the source.

By comparison, incorporation of FA reduces the proportion of cementitious material used by almost 30%, additionally through the application of RHA in similar circumstances, the cost savings associated with the replacement percentage used would reach 57%. Therefore the savings associated with using RHA provide a potential source of economic saving that may outweigh the costs associated with the use of admixtures. Additionally, through incorporating agricultural waste materials there is a flow on effect associated with the reduction of greenhouse gases in the atmosphere caused by the production of OPC. Improved methods of combusting rice husk will also assist in mitigating this issue.

# 5.6 Chapter Summary

The inclusion of RHA in a SCC mix design provided both advantages and disadvantages regarding the compounds properties and characteristic strength. Application of experimental analysis testing methods identified that the RHA produced through the proposed firing method was suitable for application as a pozzolanic material. In this instance it was determined through XRF analysis that the ash had an amorphous silica content of 93.2%. This result provided the final portion of evidence to confirm the quality of the product. Initial testing through XRD and SEM / EDS analysis confirmed the existence of this compound and the level of amorphous particles which served to provide further validity of the projects research methodology.

Application of the fresh concrete testing methods identified in the EFNARC guidelines (2002 & 2005) were responsible for confirming the acceptance of each SCC mix. Given the experimental nature of the project it was essential that existing analytical parameters were applied to assist in developing a suitable SCC material. Results from fresh concrete testing identified potential applications include walls and piles, tall and slender members, and floors and slabs for mixes with higher viscosity.

Through conducting both compressive and flexural strength tests on the hardened SCC samples it was determined that the application of RHA resulted in decreased strength performance. When referring to compressive strength, it was identified that the likely cause of this strength reduction was due to the reduction in quantities of cement in the SCC mix. The consequences of this factor resulted in a decrease amount of Ca(OH)<sub>2</sub>, which meant there was less secondary quantities of the compound to produce additional C-S-H gel. One primary advantage identified from the application of RHA was the reduction in free surface water. Through analysis of the flex beams shown in Figure 5.8, it was suggested that inclusion of RHA resulted in decreased segregation in the mix and also an apparent reduction in bleed water.

A cost benefit analysis of RHA in comparison with FA and OPC confirmed that application of this material would certainly provide a cost effective SCM for partial replacement of OPC. Further development of combustion methods and available sources are required to allow for commercial applications. Although there were some issues identified pertaining to the application of SCC in substitution of ordinary concrete in construction, further research and technological development in this area will be able to mitigate these issues.

# Chapter 6

# **Conclusion & Recommendations**

## 6.1 Research Conclusion

RHA has been identified as a suitable material for application as a pozzolan in the Australian construction industry. The primary factor influencing this is the presence of high quantities of amorphous silica, capable of reacting with cement in the hydration reaction. Concurrently, the application of SCC has the potential to overcome many limitations currently evident in the construction industry. Through utilisation of this material it is possible to construct larger structures with dense reinforcement without affecting its strength characteristics. Overall the research conducted in this report has served to provide an analysis of the advantages and disadvantages of supplementation of OPC with RHA. Furthermore, incorporation of SCC has allowed for development of this materials strengths and potential limitations.

In development of a RHA material it was identified that combustion over 700<sup>o</sup>C would result in the occurrence of silica in crystalline form. As this is a non-reactive silica production of this compound was to be minimised. When the husk was burnt at temperatures not exceeding 640<sup>o</sup>C, experimental analysis confirmed that an end product was developed that contained an optimum level of amorphous silica. Experimental analysis methods applied in this research project included XRD, SEM/EDS and XRF analysis. Each of these methods was invaluable in ascertaining the quality of RHA produced. This included identification of crystalline compounds, particle size, shape and porosity, and also the percentage of major and minor elements present within the sample. It was also determined that grinding of the ash was essential in obtaining a RHA material with suitable particle size for application as a SCM.

Hardened concrete testing in the form of compressive and flexural strength tests provided necessary data confirming the impact RHA has on the strength parameters of SCC. Both test results confirmed that the inclusion of RHA resulted in subsequent reductions in both compressive and flexural strength. Although this result was expected, the strength reduction was larger than originally anticipated. As such there was no specific application of RHA considered optimal in this instance. There is however still potential for application of each mix design. In instances requiring lower strength concretes significant replacement of OPC with a cheaper pozzolan creates an economically beneficial product.

Overall it was identified that the application of RHA in SCC in this instance provided an end material that was not overly suitable for structural application. However, through interpretation of the results a comprehensive understanding has been attained regarding the interaction between RHA and OPC, and the limitations currently impacting on the application of SCC. The most significant limitation identified in this research was the existence of voids in all trial mixes. As discussed in section 2.4, due to the properties of SCC vibration is not possible, therefore further development of an appropriate standardised mix design is essential in resolving issues regarding air voids. Although the final strength properties achieved by RHA did not meet the design compressive strength, the data collated from experimental analysis provides sufficient evidence to support the application of RHA as a SCM. Through attaining further understanding regarding the characteristics of RHA and its interaction with cement there is potential to develop a product suitable for application as a sustainable building material.

# 6.2 Recommendations for Future Research

With reference to the research outcomes identified in this report, there is certainly potential for development of RHA as a pozzolanic material, and also the application of SCC in both civil and structural development. The following recommendations are provided as suggestions for future research regarding the development of these materials:

- Development of a standardised SCC mix design incorporating fresh concrete testing parameters than can be readily applied on a construction site
- Identification of alternative rice husk combustion methods that do not require significant outputs of energy to produce, such as gasification.
- Undertake a detailed analysis that examines the cost of RHA production in Australia, as oppose to an estimate based on it being supplied as a waste material

- Further development of SCC as a construction material, currently there are many uncertainties relating to the development of this product. Through further research and development many of these issues could be mitigated.
- Undertake further research into the application of RHA in high proportions as a pozzolanic material. This particular material would be suitable for application as a grout to fill large voids where undermining has occurred. Current methods utilise high volume FA mix designs, it would be possible to further reduce costs by replacing FA with RHA whilst still attaining the low strength required (i.e. 5MPA)

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Appendix A

**Project Specification** 

### ENG4111/4112 Research Project

## **Project Specification**

For:	Cameron Smith
Title:	Utilisation of Rice Husk Ash and Fly Ash as a Partial Replacement for Cement in Self Consolidating Concrete
Major:	Civil Engineering
Supervisors:	Yan Zhuge
Enrolment:	ENG4111 – EXT S1, 2016
	ENG4112 – EXT S2, 2016

Project Aim: To determine the optimum quantity of Rice Husk Ash and Fly Ash that can be added in replacement of Portland cement in self-consolidating concrete

#### Programme: Issue A, 16th March 2016

- 1. Research background information relating to the properties of Rice Husk Ash when added to self-consolidating concrete as a Pozzolanic Material
- 2. Research similar projects that use a Fly Ash or Rice Husk Ash mix design to determine what the current literature advises.
- 3. Review Australian Standards to determine required testing parameters, which will include a j-ring flow test, compressive strength and flexural strength test
- 4. Analyse multiple mix designs that contain varying percentages of Rice Husk Ash, Fly Ash, additives and Cement
- 5. Determine the optimum ratio of materials through analysis of the compressive and flexural strength of all mix designs
- 6. Research the overall cost variance for each mix design to determine whether the optimum ratio is also the most cost effective

If time and resources permit:

7. Using the results provided conduct a test on the optimum concrete mix design to determine the durability of the sample.

Appendix B

Material Test Data Sheets
#### 10/7 mm Concrete Blue Metal Aggregate Test Results **B**.1

Sample Description	on:	an en	10/7mm Concrete Blue Metal S/P 123
Location:		and the second second	Seaham Quarry
Client Sample No			WSE-15/00012-Q04
Date Sampled:		and the second	9.03.15
Laboratory Samp	le No:		163688
Test Method	Test	Specifications	Results
AS1141.11.1*	% Passing A.S. Sieve 13.2mm 9.5mm 6.7mm 4.75mm 2.36mm 1.18mm	100 80-100 - 5-22 0-10 0-3	100 95 51 16 1
AS1141.12	Material finer than 75 micron (%)	0-1	1
AS1141.14	Mis-shapen particles (%) Ralio 2:1 Ratio 3:1	Max. 25 Max. 10	N/A N/A
AS1141.15	Flakiness Index (%)	Max. 25	29
RMS T235	Average Least Dimension (mm)		3.4
RMS T235*1	Average Least Dimension (mm) Fraction tested: -9.5+6.7mm -6.7+4.75mm		5.4 4.1
RMS T278*2	Average shape by the ratio of greatest to least dimension: The Ratio of AGD to nominal least dimension of each group: 0-2mm 2-4mm 4-6mm 6-8mm The Ratio of AGD to ALD of the sample	Max. 2.25	7.90 2.63 1.98 1.50 2.50
AS1141.18*3	Crushed Particles in Coarse Aggregate Derived from Gravel % of Crushed Particles % of Uncrushed Particles	Min. 80	100 Nil

Page 1 of 2

Note: \* Sample washed over 75 micron sieve as per AS1141.11.1 Clause 5.6. \*1 Sample tested to RMS T235 with individual fractions tested and reported - Test is not NATA Accredited. \*2 Test RMS T278 is not NATA accredited \*3 Test AS1141.18 is not NATA accredited

Sample Descriptio	in:		10/7mm Concrete Blue Metal S/P 123
- sellen:			Seaham Quarry
Location:			WSE-15/00012-Q04
Client Sample No.			9.03.15
Date Sampled:			163688
Laboratory Sampl	e No:	Specifications	Results
Test Method	Test	The second s	1.28
AS1141.4	Compacted Bulk Density Vm <sup>3</sup>	Min. 1.2	1.40
DMS T262	Moisture condition of the aggregate (%)		1./
AS1141.6.1	Particle Density (Dry) 1/m <sup>3</sup> Particle Density (SSD) 1/m <sup>3</sup> Apparent Particle Density 1/m <sup>3</sup> Water Absorption (%)	Min. 2.1 Max. 2.5	2.55 2.58 2.64 1.4
AS1141.22 (Duplicate)	Average Aggregate Dry Strength (kN) Average Aggregate Wet Strength (kN) Wet/Dry Strength Var. (%) Fraction Tested (mm) The amount of significant breakdown (%) Size of cellider used: 150mm diam	Min. 70 Max. 35	-9.5+6.7 <0.2
AS11/1 23	Los Angeles Value Grd. 'K' (% Loss)	Max. 30	14
AS1141.24	Sodium Sulphate Soundness (Total weighted % Loss)	Max. 6	0.5
	Fraction tested: -9.5mm+4.75mm (% Loss) -4.75mm+2.36mm (% Loss)		0.5 0.4
AS1141.32	Weak Particles (%) The % of original sample passing 2.36mm sieve = 1.4	Max. 0.3	111
AS1141.25.2	Degradation Factor - Coarse Aggregate The wash water was Clear after using Permitted 500ml	Min. 50	Yes

Material sampled by laboratory staff as per AS1141.3.1. Clause 9.4 under NATA accreditation 547

## B.2 Fine Dune Sand Aggregate Test Results

Sample Details	A DATE OF STREET	C. S. P. C. S. S.	ALC: THE REAL	Particle S	ize Distribution	
Sample ID: NEW15 Sampling Method: Sampler Date Sampled: 08/07/20 Source: Salt Ash Material: Sand Specification: Uncrush Project Location: Janet P Sample Location: Quarry S	N-1400S01 I by Client I15 Quarry ed Fine Aggregate arade, Salt Ash, NS Stockpile	w		Method: Drying by: Note: Sieve Size 9.5mm 6.7mm 2.36mm 1.18mm 1.18mm	AS 1141.11.1 Oven Sample Not Washed % Passing 100 100 100 100 100	Limits 100 90 - 100 60 - 100 30 - 100
Mitter Tault Resultis	Marchart		S. Det	425µm	97	13-100
Description	Method	Result	Limits	300µm	55	5 - 50
Finer 75µm (%)	AS 1141.12	0	0-5	150µm	0	0 - 20
Drying Method		Oven		75µm	0	0-5
Loss 0.600 to 0.300 mm (%)	AS 1141.24	0.4				
Total Weighted Loss (%)		0.4	≤12	-		
Clay and Fine Silt (%)	AS 1141.33	2		_		
Uncompacted Bulk Density (t/m³)	AS 1141.4	1.18				
Compacted Bulk Density (t/ms)		1.30 Dried				
Aggregate Moisture Condition		Uned				
Nominal Size Of Sample (mm)	AC 11115	2.67				
Apparent Particle Density (1/m°)	A0 1141.0	2.65				
Particle Density Dry (Vm*)		2.66				
Mater Absorption (%)		0.4		and a state	the second second second second	and the second
- 1995 - 1945 -				55 /herrig 10 10 10 10 10 10 10 10 10 10 10 10 10	A.	1



Port Adelaide 5A 5015



Despatched from Norgan Camert Internationa: - P.O Box 230, Port Kembla, NSW, 2505 Grab sample taken during despetch from 03 Will on the 9th of October 2015 MORGAN TYPE S., CEMENT ABN 96 007 870 199

TEST CERTIFICATE NUMBER 5520

Telephone (08) #300 0300 International +613 3300 0300 Facsimile (06) 8342 1591 www.adbri.com.au

Drying Shrinkage 28d Jastrain	540	NAX 750	
20d 20d MPa	63.6	ABN 45.0	
0.11 Strot 7d MPa	46.9	35.0	
AS 235 3d MPa	32.8	N/H	
Final Set hamin	2:45	MAX 6:00	turk.
Set frittal	1:15	MIIN 0:45	except in t
Normal Cons	27.8	NIR	produced
Soundness	-	s S	shall not be re
Fineness Inclose incluse	305	NIN	boratory and
ö 🕫	0.024	98.AX 0.1	alhead La
N P	1.2	N/R	he Birther
SO3	3,0	MAX 3.5	ced by ti
Equivalent Altatis	0,44	NN	st was produ
0 <sup>BM</sup>	1.4	N/R	document
Ca0	63.6	NIR	This
Fe <sub>2</sub> O <sub>3</sub>	3.0	NUR	
A2,03	5.8	N/R	
SiO <sub>2</sub>	21.3	NUR	
Sample Date	09-Oct-15	772-2010 and Limit	
Sample Number	B04dbof	AS 39 Standa	

Grab sample of cement with sample number DDG091015-03 Mill delivered into the Birkenhead Works for the date shown.

Besults applicable only to sample tested. Oxide analysis by X-ray Fluorespance (tused boad) Testing performed on samples as received at the Birkenhead Division Laboratory (Accredited number: 352) in accordance with AS 2350.2.2006, AS 2350.4.2006 and in-house method BH-TM-0544 Physical testing performed at the Angaston Division Laboratory (Accredited number: 352) in accordance with AS 2350.4.2006, AS 2350.4.2006 and in-house method BH-TM-0544 and AS 2350.13:2006 This cament was produced from Portland Cement Clinker, natural Gypsum, Mineral and Processing auditions only as per AS 3872:2010 N/F = No Requirement



Accredited Number: 252 Accredited Number: 73 NATA Laboratories

Accredited Number: 73 16-November-2015

Accredited Number: 252

Approved Signatory V.Bhagwat

Issue date:

Approved Signatory adod."

#### **Cement Test Results B.3**

Part Adelaide SA 5025 Birkenhead SA 5015 PO Box 77 62 Elder Road



CLASSIFIED FLYAGH (ATHC)

Despatched from Morgan Cement Informational - PC Box 230, Port Kembla, NSW, 2505 TEST CERTIFICATE NUMBER 8519 For the month of September 2015

Relative Strength 28 day	18	10	NR
Relative Water	2	102	NIR
Chloride	y.	<0.001	NR
Relative Donsity		2,12	NIR
Moisture Content	2	<0.1	MAX 1.D
Fineness % 45 um	Residue	98	75
P	2	3.0	MAX 4.0
55	-	0.1	a.c
IC <sub>0</sub>	20	1.07	NIR
Na <sub>2</sub> O	3%	0.79	NIR
ofigi	N.	0.6	NR
CaO	st.	4.1	MR
Feg03	*	2.8	MIR
ALO3	12	28.0	NUB
SIO	18°	57.8	N/H
Sample Date		01-Oct-15	52.1-1998 and Limit
Sample Number		Bo4daSt	AS 35 Standt

This document was produced by the Birkenhead Laboratory and shall not be reproduced except in full.

Fly ash produced by Vales Point Power Station. Sample Method: Grab sample of ash with sample number MA-011015 delivered into the Birkenhead Works for the date shown.

Results applicable only to samples tested.

Oxole analysis by X-ray Fluorescence (tused bead) Testing performed on samples as received at the Birkenhead Division Laboratory (Accredited number 25) in accordance with AS 2350.2:2006, AS3583.1:1998 and AS3583.3:1991 Relative Water and Relative Strength tested at the Angaston Division Laboratory (Accredited number 31) in accordance with AS 2350.2:2006, AS3583.1:1998 and AS3583.3:1991 Relative Water and Relative Strength tested at the Angaston Division Laboratory (Accredited number 33) in accordance with ASS583.6:1995 using Internal Reference Cement A061316 as the

control N/R – No Requirement

NATA

Accredited for compliance with ISO/IEC 17025. The results of the tests, and/or measurements included in this document are increable to Australian/mutorial stanctance.

Accredited Number: 252 Accredited Number: 73 NATA Laboratories ACCREDITATION

**DOWING** 

considered Numbers 73 Approved Signatory

acredited Number: 252 Approved Signatory

12-November-2015

Issue date:

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### B.5 MasterGlenium 1466 HRWR Safety Data Sheet

## MasterGlenium<sup>®</sup> 1466

High-Range Water-Reducing Admixture

Formerly PS1466\*

#### Description

MasterGlenium 1466 readyto-use high-range waterreducing admixture is a new generation, patent pending admixture based on polycarboxylate chemistry. MasterGlenium 1466 admixture is very effective in producing concretes with different levels of workability.

MasterGlenium 1466 admixture is particularly effective in improving concrete mixtures with reduced portland cement contents without compromising 28-day strength requirements. MasterGlenium 1466 admixture meets ASTM C 494/C 494M requirements for Type A, wate-reducing, and Type F, high-range wate-reducing, admixtures.

#### Applications

- Recommended for use in: Concrete with varying
- water reduction requirements (5-40%)
- Concrete where high flowability, increased stability and durability are needed
- Producing selfconsolidating concrete (SCC)
- Strength-on-demand concrete, such as 4x4<sup>™</sup> Concrete
- Pervious concrete

#### Features

- Maximum dosage effectiveness for a given water reduction
- Controlled rheology
- Robust air-entraining admixture compatibility
- Improved strength development

#### Benefits

- Can be used in a wide variety of concrete mixtures as a Type A or Type F admixture
- Improved finishability and surface appearance
- Mixture development flexibility for cement reductions and/or increased use of supplementary cementitious materials

#### **Performance Characteristics**

Compressive Strength: Concrete produced with MasterGlenium 1466 admixture achieves significantly higher 28-day compressive strength compared to plain concrete and concrete mixtures containing naphthalene, melamine, and early generation polycarboxylate high-range water-reducing admixtures.

#### Mixture Data: Type I portland cement; Ambient Temperature, 70 \*F (21 \*C)

- Mix 1: 620 lb/yd² (367 kg/m²); w/c = 0.43; Conventional PC HRWR
- Mix 2: 620 lb/yd² (367 kg/m²); w/c = 0.43; MasterGlenium 1466
- Mix 3: 600 lb/yd<sup>a</sup> (356 kg/m<sup>a</sup>); w/c = 0.44; MasterGlenium 1466

Mix 4: 580 lb/yd² (344 kg/m²); w/c = 0.46; MasterGlenium 1466





### MasterGlenium 1466

#### Guidelines for Use

Dosage: MasterGienium 1466 admixture has a recommended dosage range of 2-10 fl oz/cwt (130-650 mL/100 kg) of cementitious materials. For most applications, dosages in the range of 2-6 fl oz/cwt (130-390 mL/100 kg) will provide excellent performance. Because of variations in concrete materials, job site conditions and/or applications, dosages outside of the recommended range may be required. In such cases, contact your local sales representative.

Mixing: MasterGlenium 1466 admixture can be added with the initial batch water or as a delayed addition. However, optimum water reduction is generally obtained with a delayed addition.

#### **Product Notes**

Corrosivity – Non-Chloride, Non-Corrosive: MasterGlenium 1466 admixture will neither initiate nor promote corrosion of reinforcing steel embedded in concrete, prestressing steel or of galvanized steel floor and roof systems. Neither calcium chloride nor other chloride-based ingredients are used in the manufacture of MasterGlenium 1466 admixture.

Compatibility: MasterGlenium 1466 admixture is compatible with most admixtures used in the production of quality concrete, including normal, mid-range and high-range water-reducing admixtures, air-entrainers, accelerators, retarders, extended set control admixtures, corrosion inhibitors, and shrinkage reducers.

Do not use MasterGlenium 1466 admixture with admixtures containing naphthalene sulfonate. Erratic behaviors in slump, workability retention and pumpability may be experienced.

#### Storage and Handling

Storage Temperature: MasterGlenium 1466 admixture must be stored at temperatures above 40 °F (5 °C). If MasterGlenium 1466 admixture freezes, thaw and reconstitute by mechanical agitation. Do not use pressurized air for agitation.

Shelf Life: MasterGlenium 1466 admixture has a minimum shelf life of 6 months. Depending on storage conditions, shelf life may be greater than standard. Please contact your local sales representative regarding suitability for use and dosage recommendations if the shelf life of MasterGlenium 1466 admixture has been exceeded.

#### Packaging

MasterGlenium 1466 admixture is supplied in 55 gal (208 L) drums, 275 gal (1040 L) totes and by bulk delivery.

#### **Related Documents**

Safety Data Sheets: MasterGlenium 1466 admixture

#### Additional Information

For additional information on MasterGlenium 1466 admixture or its use in developing concrete mixtures with special performance characteristics, contact your local sales representative.

The Admixture Systems business of BASF's Construction Chemicals division is the leading provider of solutions that improve placement, pumping, finishing, appearance and performance characteristics of specialty concrete used in the ready-mixed, precast, manufactured concrete products, underground construction and paving markets. For over 100 years we have offered reliable products and innovative technologies, and through the Master Builders Solutions brand, we are connected globally with experts from many fields to provide sustainable solutions for the construction industry. Appendix C

Mix Design Calculations

### Mix Design Calculations – 40 MPa SCC mix design

1. With reference to Table C1, determine the maximum weight of water and air content (*a*) in the mixture according to maximum aggregate size:

	Maximum aggregate size (MAS), mm			
	9.5	12.5	19.0	
Water content, kg/m <sup>3</sup>	200	190	180	
Air content, %	3.0	2.5	2.0	

Table C1: Modified maximum water requirements for SCC, (Ghazi, 2014)

Note: 1 kg/m3 = 1.68 lb/yd3; 1 mm = 0.0393 in.

Through linear interpolation of the above table for a maximum aggregate size of 10mm:

$$y_2 = \frac{(x_2 - x_1) * (y_3 - y_1)}{(x_3 - x_1)} + y_1$$
 (Equation 1)

Which determined;

water cont. = 
$$\frac{(10 - 9.5) * (190 - 200)}{(12.5 - 9.5)} + 200$$
  
=  $198 kg/m^3$   
Air content (a) =  $\frac{(10 - 9.5) * (2.5 - 3.0)}{(12.5 - 9.5)} + 3.0$   
=  $2.92 \%$ 

2. Obtain the weight of water ( $W_w$ ) from Figure C1, and obtain the water /cement ratio (w/c) from Table C2 based on strength requirements

Figure C.1: Proposed water quantities based on strength



1kg/m3=1.68 lb/yd3, 1 mm=0.0393in, 1 MPa= 145 psi.

Table C.2: W/c ratio versus compressive strength

 Table 3—w/c versus SCC compressive strength relationship

 f\_c', MPa
 15
 20
 25
 30
 35
 40
 45
 50
 55
 60
 65
 70
 75

 w/c
 0.8
 0.7
 0.62
 0.55
 0.48
 0.43
 0.38
 0.35
 0.34
 0.33
 0.32
 0.31
 0.29

 Note:<1<MPa = 145 psi.</td>
 MPa
 145 psi.
 MPa
 145 psi.
 MPa
 145 psi.

The weight of water is determined through linear interpolation of Figure 1, to allow for 10mm maximum aggregate. Using Equation (1):

$$Ww = \frac{(40 - 35) * (180 - 187)}{(45 - 35)} + 187$$
$$Ww = 183.5 \ kg/m^3$$

The w/c ratio is determined through selection of the value provided in Table C2:

w/c = 0.43, For this design the ratio 0.421 was selected

3. Calculate the cement content ( $W_c$ ) and volume of cement ( $V_c$ ) where:

$$W_C = \frac{W_w}{W/c}$$
$$V_C = W_c/SGC$$

(SGC = specific gravity cement)

$$W_C = 435.944$$
  
 $V_C = 138.835 L/m^3$ 

4. The dry-rodded volume of gravel  $(V_q)$  is obtained from Table C.3

Table C.3: Modified bulk volume of coarse aggregate

	FM				
MAS, mm	2.40	2.60	2.80	3.00	
9.5	0.50	0.48	0.46	0.44	
12.5	0.53	0.51	0.49	0.46	
19.0	0.56	0.54	0.52	0.50	

Note: 1 mm = 0.0393 in.

5. Calculate the dry weight of gravel ( $W_{GD}$ ) by multiplying ( $V_G$ ) by the dry-rodded unit weight of gravel (from test results)

$$W_{GD} = 0.5 * 1400$$
  
= 700 kg/m<sup>3</sup>

6. Calculated the Saturated Surface Dry weight of gravel

$$W_G = W_{GD} * (1 + \frac{\Delta}{100})$$
$$W_G = 700 * (1 + \frac{0.3}{100})$$
$$W_G = 702.1 kg/m^3$$

7. From Table C.4, determine:

$$\frac{V_W}{W_C} + V_L$$

Ratio for a known compressive strength and mix aggregate size, and then calculate volume of powder ( $V_L$ )

$f'_c$ , MPa	$V_W/V_C + V_L$
15	1.10
25	1.05
35	1.00
45	0.95
55	0.90
65	0.85
75	0.80

Table C.4: Proposed water volume to powder ratio

Note: 1 MPa = 145 psi.

$$\frac{V_W}{W_C} + V_L = 0.975$$
$$V_L = 53.767$$

$$V_G = 0.5$$

8. Calculate the powder weight

$$W_L = V_L * SGL$$

Where SGL = specific gravity of powder (FA)

$$W_L = 113.986 \ kg/m^3$$

9. Calculate the weight of total powder content  $(W_c + W_L)$  and total powder volume  $(V_c + V_L)$  and check with the limitations of the EFNARC method

$$W_C + W_L = 569.93 \ kg/m^3$$
  
 $V_C + V_L = 192.60$ 

10. Calculate the fine aggregate content by absolute volume method:

$$1m^{3} = \frac{W_{w}}{1} * 1000 + \frac{W_{c}}{3.15} * 1000 \frac{W_{L}}{SGL} * 1000 \frac{W_{S}}{SGS} * 1000 \frac{W_{G}}{SGG} * 1000 + a\%$$

$$= 753 kg/m^3$$

Superplasticiser content = 1%

Water/paste ratio = 0.3219694 = 0.32

Or with reference to the EFNARC guildelines (2005):

w/p = 0.9527387 = 0.95>0.85 therefore ok

Appendix D

Project Risk Assessment

### D.1 Risk Matrix

Likelyhood	Consequences					
	Insignificant Risk is easily mitigated by narmal day to day process	Minor Delays up to 10% of Schedule Additional cost up to 10% of Budget	Moderate Delays up to 30% of Schedule Additional cast up to 30% of Budget	Major Delays up to 50% of Schedule Additional cost up to 50% of Budget	Catastrophic Project abandoned	
Certain >90% chance	High	High	Extreme	Extreme	Extreme	
Likely 50% - 90% chance	Moderate	High	High	Extreme	Extreme	
Moderate 10% - 50% chance	Low	Moderate	High	Extreme	Extreme	
Unlikely 3% 10% chance	Low	Low	Moderate	High	Extreme	
Rare <3% chance	Low	Lew	Moderate	High	High	

### D.2 Workplace Safety Risk Assessment

Task	Hazard	Risk	Control
3A	Risk of injury due to	High	Attend workplace safety induction
	unfamiliar site		
3A	Cuts and abrasions from	Moderate	Operate equipment using correct
	operating mixer		procedure, wear gloves and a long sleeve
			shirt
3A	Dust inhalation from	Moderate	Wear a dusk suppression mask
	materials		
3A	Eye injury due to splash	High	Wear safety glasses
	back when mixing		
3A	Strains and back injury from	Moderate	Practice safe manual handling techniques
	manual handling		
3A	Chemical Burns from	High	Wear PPE appropriate to the task. i.e.
	exposure to cement		long sleeve shirt and long pants, gloves,
			eye protection

3B	Crush, strain injury from	High	Practice safe manual handling
	handling heavy samples		techniques, wear appropriate PPE, i.e.
			steel cap boots
3B	Injury from operating	High	Ensure training is received on correct
	testing equipment		operation of equipment before work
			begins

## D.3 Project Completion Specific Risks

Task	Hazard	Risk	Control
1A	Incorrect mix design	High	Confirm mix design with supervisor
	calculation		
1C	Sample Size	High	Confirm quantities required with
			supervisor
2A	Incorrect burning method	High	Ensure multiple suppliers have been
	for RHA		contacted in case issue arises
2B-	Unable to produce samples	High	Ensure regular communication with
3A	as not able to access lab		testing lab to stay informed of possible
			issues
2C	Some materials not	Low	Procure materials at least three weeks
	available		prior to date required to account for
			locating other sources
3B	Unable to collect samples as	High	Identify other means of testing, possible
	test equipment is		option within the Newcastle area is
	unavailable or not		University of Newcastle. Only
	operational		considered as last resort
4B	Unable to access USQ	Low	Perform extensive literature review prior
	database for information		to start of semester 2

# Appendix E

# **Project Timeline**

### E.1 Project Specific Timeline

