MECHANICALPROPERTIES OF RICE HUSK ASH-GROUND GRANULATED BLAST FURNACE SLAG TERNARY BLENDED CEMENT MORTAR

Cheah Chee Ban
School of Housing, Building & Planning
Universiti Sains Malaysia,
11800 USM Pulau Pinang, Malaysia
Email: cheahcheeban@usm.my, Tel: 604- 653 2827

Bob Harris Norbert
School of Housing, Building & Planning
Universiti Sains Malaysia,
11800 USM Pulau Pinang, Malaysia
Email: bobharrisnorbert@gmail.com, Tel: 601-9537 0139

ABSTRACT

Rice husk ash (RHA) and ground granulated blast-furnace slag (GGBS) are two materials that have a high potential in improving the properties of cement mortars. Intensive investigations have been performed to study the usage of supplementary cementitious materials (SCM) aiming to find the suitable materials that can someday replace cement, but very few were done on the ternary blend consisting of RHA, GGBS and cement. As the production of cement has posed many environmental issues regarding the production of carbon dioxide, these two materials, RHA and GGBS emerged as good pozzolans to partially replace cement for concrete or mortar production. In the present study, it has been reported that both materials has enhanced the properties of cement mortars in terms of mechanical performances. Optimization process of RHA and GGBS as cement replacement was performed to get the optimum performance of this ternary mix. RHA and GGBS were characterized in terms of minerals content to investigate the major elements that are present in them. The X-Ray Fluorescence (XRF) results show that RHA is high in amorphous silica content, which is similar to Silica Fume, while GGBS is high in calcium and also silica. RHA was further grinded for 30 mins to increase its reactivity. The combination of these two materials (RHA and GGBS) in cement mortars was investigated and resulted in a higher performance in mechanical strength compared to the equivalent control OPC mortar specimen. The synergic reaction between amorphous silica and one of the hydration products of cement, calcium hydroxide ($Ca(OH)_2$) contributed to the development of strength. The fine and mesoporous particles of RHA were also established to play an important role in strength development of the ternary blended cement mortar.

Keywords: Rice husk ash, pozzolans, ternary blend, mechanical properties, synergic.

Introduction

RHA and GGBS are good pozzolans, both materials has a good reputations in partially replacing cement as a SCM. GGBS was known for its sulfate resisting properties and its effect on enhancing the mechanical properties of cement mortar or concrete. GGBS is a byproduct of the reduction of iron ore into iron in a blast furnace, before it is ground to a powder form; it is initially in a larger particle size which is in granules state. According to Regourd (Regourd, 1986), slag granulation process are now used in which air or water under pressure breaks down the liquid slag into small droplets which are solidified into grains size of up to 4 mm, mostly in a glassy state, this is called granulated slag. The raw slag is glassy and coarse; after it is grinded it can be added as an enhancer with Portland cement. In addition to high durability and aesthetic value, GGBS handles much like Portland cement. GGBS was well-known for its significant uses in the construction industry; it will react with water to produce cementitious properties. Cement has been the major binder materials that are being used in the construction field for as long as since it was first invented. The production of 1 ton of cement will yield 1 ton of carbon dioxide (CO_2), in which 6% of the world's CO_2 emissions is accounted by the cement and aggregates production industries. By establishing a new phase of binder, in a way of replacing cement partially, it can reduce the usage of cement, thus reducing the production of cement which hopefully contributes to the reduction of CO_2 emissions. The GGBS are high in calcium, it contain 35-40% of CaO. The aim of this research was to investigate the effect of the reaction between GGBS and RHA as a partial cement replacement on the RHA-GGBS ternary blended cement mortars in terms of mechanical properties. RHA was known for its high content of silica which attributed its high reactivity. In rice cultivating countries such as Malaysia, Vietnam, China and India, Rice husk is abundant. Rice husk is the outer layer of rice kernel; it is also an agricultural waste from the process of paddy milling. For every 1 ton of milled paddy, about 20% (200kg) of husk is produced. These husks will then be burnt to generate steam, the burnt rice husk is what produced the RHA. Proper combustion process of rice husk and at controlled temperature will yield RHA with high content of silica (SiO_2) in amorphous state. Analysis of present studies on the influence of combustion conditions on the nature of silica suggests that temperatures below 750° will be sufficiently safe to yield rice husk ash with high reactivity and amorphousness (Boateng & Skeete, 1990; Bui, 2001). RHA has shown a promising results as an established SCM, it is considered as one of the highly reactive pozzolanic materials due to its high content of amorphous silica (Jamil, Kaish, Raman, & Zain, 2013). So far, there are not much investigations were conducted on the mechanical performances of the RHA-GGBS ternary blended cement mortars. GGBS is high in CaO, which when in the presence of water (H_2O), will liberate Ca^{2+} , OH^- ions, this gives rise to the idea where the reaction of amorphous silica and the Ca^{2+} , OH^- ions, and also the hydration products, calcium hydroxide ($Ca(OH)_2$) will form a secondary C-S-H gel that mainly contributes to the strength development. This is evidently portrayed from past literature by several author, they reported that the chemical reaction of amorphous silica with Ca^{2+}, OH^- ions and calcium hydroxide during the cement hydration forms more calcium silicate hydrate gel (C–S–H) that is known to contribute to the improvement in the strength and durability properties of concrete (Feng, Yamamichi, Shoya, & Sugita, 2004; Givi, Rashid, Aziz, & Salleh, 2010).

Materials And Method

This section presents the properties of materials used in the study, experimental programme and methodology of investigation.

Experimental Program

The aim of this research is to study and investigate the potential of RHA-GGBS ternary blended cement mortar performances in terms of mechanical properties in comparison with that of control concrete. Characterization of materials was also done in this research. The mechanical tests includes compressive and flexural test on 7 and 28 days of moisture curing. The materials used (RHA and GGBS) were characterized in terms of their oxides or chemical composition through X-Ray Diffraction and X-Ray Fluorescence test. Blaine fineness test was also performed to determine their respective surface area. The RHA used in this research was ground for 30 mins to achieve the fineness comparable to cement’s fineness. Before proceeding to the fabrication of the specimens for testing, the hybridization ratio of RHA-GGBS was performed by using the Slag Activity Index (SAI) test in accordance with ASTM Standard : C989 – 05 (ASTM, 2005). Table 1 shows the designation of the hybridization ratio for RHA-GGBS, the compressive strength of the specimens was taken after 7 days of curing age, and the highest recorded strength will be taken as the optimum hybridization ratio for RHA-GGBS. All the SAI test specimens were tested in accordance with the ASTM Standard C109/C109M (ASTM, 2006). Based on the result, the optimized value achieved from the SAI test was used as a fixed ratio as a cement replacement in mortars specimen. All specimens were fabricated in triplet to get the average values. The flow of the mixes was maintained at 140 ± 15 mm throughout the study, and the curing regime used was normal moisture curing. Table 2 shows the mix proportion of the ternary blend cement mortar, for this particular proportion, the optimum ratio of RHA-GGBS which was obtained from Table 1 was used as fixed ratio, and gradually replaces cement from 0% to 100% level of replacement with gradual increment of 10%.

Table 1: Slag Activity Index (SAI) mix proportion

Level of RHA in GGBS%	RHA	GGBS	CEMENT	SAND	7D STR (Mpa)	w/b	flow	SAI (%)	Grade
(Reference)	0	0	750	2063	8.2868	0.73	210	100	120
0	0	375	375	2063	8.9498	0.76	220	108	120
5	18.75	356.25	375	2063	9.77692	0.73	210	117	120
10	37.5	337.5	375	2063	9.28028	0.76	210	112	120
15	56.25	318.75	375	2063	10.8688	0.77	210	131	120
20	75	300	375	2063	8.98908	0.81	215	108	120
25	93.75	281.25	375	2063	9.21456	0.75	215	111	120
30	112.5	262.5	375	2063	7.97548	0.76	210	96	120
35	131.25	243.75	375	2063	10.7284	0.74	220	129	120
40	150	225	375	2063	10.58	0.73	210	127	120

Table 2: RHA-GGBS ternary blended mortars mix proportion.

Level of Cement replacement by RHA-GGBS (%)	RHA(g)	GGBS(g)	CEMENT(g)	SAND(g)
100	135	765	0	2700
90	121.5	688.5	90	2700
80	108	612	180	2700
70	94.5	535.5	270	2700
60	81	459	360	2700
50	67.5	382.5	450	2700
40	54	306	540	2700
30	40.5	229.5	630	2700
20	27	153	720	2700
10	13.5	76.5	810	2700
0	0	0	900	2700

Materials

PORTLAND LIMESTONE CEMENT
The cement used in this research was a CEM II lime-composite cement, it was obtained from a local cement production plant, and was packed in 50kg bag. It has a specific gravity of 3.02 g/cm^3 and a fineness of 3500 cm^2/g.

RICE HUSK ASH (RHA)
The RHA used in this research was obtained from a local rice miling industry Serba, Wangi Sdn Bhd, Alor Setar, Kedah. The as-received RHA has a specific gravity of 1.977 g/cm^3, and fineness of 1503 cm^2/g. The as-received RHA was then further ground to a degree of fineness which it can be comparable to that of cement. It was ground for 30 minutes and has a specific surface area of 6131 cm^2/g, with specific gravity of 2.214 g/cm^3.

GROUND GRANULATED BLAST-FURNACE SLAG (GGBS)
The GGBS used in this research was obtained from a local cement production company in Malaysia. It has a specific gravity of 2.86 g/cm^3 and fineness value of 2140.75 cm^2/g.

FINE AGGREGATE
The fine aggregate used in this investigation were sourced from a local sand supplier which harvest from quarzitic natural river sand. The maximum aggregate particle size of the sand was 5mm and has a specific gravity of 2.65. The fine aggregate were graded in conformation with the BS EN196: Part 1 (EN, 2005).

WATER
The water used for the fabrications of specimens throughout the research was normal tap water which is easily available. The specifications of the tap water used is complying with the requirements of BS EN 1008 (EN, 2002).

Materials Characterization

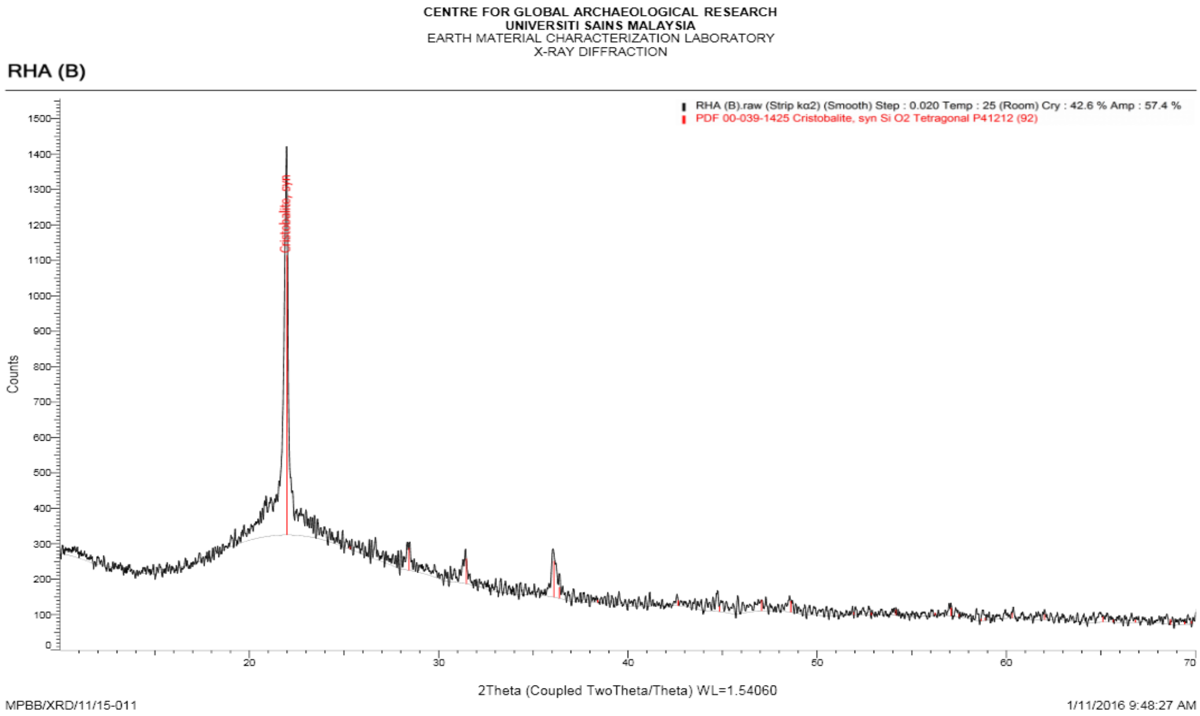
XRF
From the XRF analysis results, it was observed that RHA is mainly silica in content with 90.18% by mass which conform to the past literature review stating that RHA are reactive pozzolan due to its high content of silica in amorphous state. GGBS is mainly of Calcium and Silica and Alumina with value of 40.80%, 32.84% and 13.27% respectively. The presence of high content of CaO in GGBS is essential in strength development by the formation of C-A-S-H and C-S-H during the hydration reaction. The presence of SiO_2 in GGBS also indicates that the material possesses pozzolanic properties.

Table 3: Chemical composition of the binder materials

Chemical Compound	Value (% by total mass)	
	RHA	GGBS
Na_2O	0.046	0.2
MgO	0.714	6.08
Al_2O_3	0.255	13.27
SiO_2	90.180	32.84
P_2O_5	1.478	0.01
SO_3	0.353	-
Cl	0.544	-
K_2O	3.969	0.36
CaO	1.507	40.80
TiO_2	0.019	0.47
Cr_2O_3	0.016	-
MnO	0.115	0.1
Fe_2O_3	0.748	0.3
CuO	0.018	-
ZnO	0.025	-
SeO_2	0.002	-
Br	0.002	-
Rb_2O	0.007	-
SrO	0.003	-

XRD
Based on the XRD analysis of RHA, the graph generated shows a broad hump at the in between 20°-30° of 2ϴ scale, which indicates that this material is mainly in amorphous state. From the XRF we observed that the value of silica content is high (90%), silica in amorphous state is highly reactive, it can reacts with calcium hydroxide to form C-S-H gel.

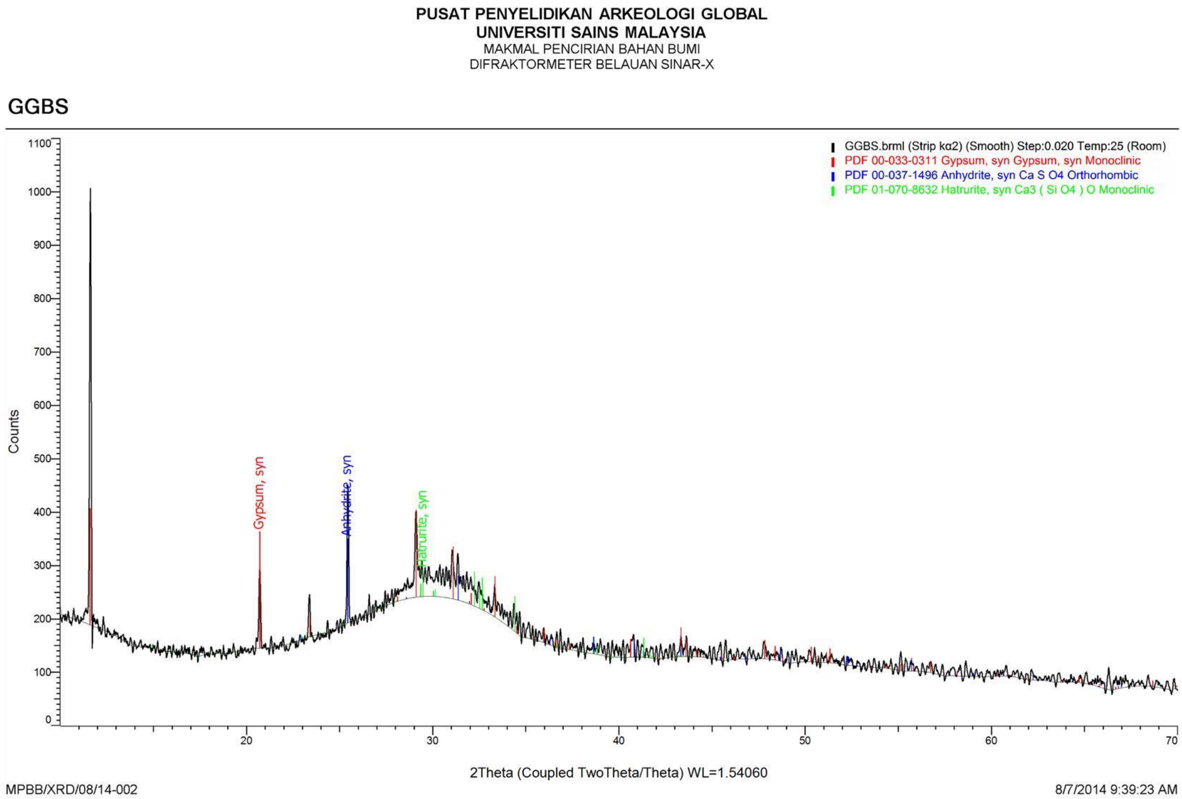
Figure 1: XRD Pattern of RHA



GGBS

Based on the XRD results in Figure 2, there is a broad hump between 25° to 35° on the 2Ө axis which indicates that GGBS is also an amorphous component. The XRD pattern of GGBS also shows the presence of gypsum, anhydrite, and hatrutrite with significant peak at approximately 21°, 25°, and 29° respectively on the 2Ө axis.

Figure 2: XRD Pattern of GGBS



Methodology

X-RAY FLUORESCENCE (XRF) ANALYSIS

The XRF analysis was performed to determine the major and trace elements of the materials (RHA and GGBS) in terms of their mineral oxides. The spectrometer used for both tasks (major and trace element) was fully automated AXIOS Pan Analytical. The samples were further ground into approximately 20µm grain size in order for it to be suitable for XRF technique. The specimens for the major element analysis was made by igniting 0.5g of sample and 0.5g of spectro-flux at 1050°C for 20 minutes, before casted into glass discs with 32mm diameter. Calibration technique was employed before performing the analysis. The 10 element curves were constructed using 20 high quality international standard reference materials, comparable in composition to the unknown sample. The specimen for trace element analysis is in the form of pressed-powder pallet. The analysis was done by scanning the presence of elemental peaks from the lowest to the highest angle of incidents and the X-ray intensity at characteristic peak of particular element is compared to the intensity of the same element in the series of standards stored in the software OMNIAN.

X-Ray DIFFRACTION (XRD) ANALYSIS

XRD analysis is a non-destructive test which an X-ray beam was diffracted on a crystal and generates scattered beams and diffraction pattern of spots would form. The data generated was in the form of diffractogram which provides information like mineral phases, chemical compositions, and crystal structure of a material. This test was performed on the materials, RHA and GGBS to characterize the respective materials. The machine used in this test is a fully Automated Bruker's D8 Advance X-Ray Diffractometer. The analysis was performed at one of the sophisticated Earth Material-Characterization laboratory in the Center of Global Archeological Research, Universiti Sains Malaysia. The Specimen for XRD analysis was made by hydraulic-pressing the powdered sample into 25mm diameter circular disc on the sample holder. The sample surface was made flat and parallel to the surface of the sample holder by pressing gently on a glass slide. The experimental parameters are as follows: Source: Kα, λ = 1.54060Å, Scanning range 10 - 70° 2Θ, scanning speed: 0.02° 2Θ/sec.

CURING REGIME: MOISTURE CURING

The curing regime employed throughout the research was normal moisture curing. The specimens were wrapped with plastic cling wrap to prevent moistures from leaking out from the specimens to enable further hydration process by the moisture itself. The curing period was designed at 7 days and 28 days before subjected to tests.

MECHANICAL ACTIVATION BY GRINDING

The grinding of RHA was done manually in the School of Housing, Building & Planning Concrete Laboratory. The ball mill was used to ground the RHA. Two types of steel balls are used, small and big steel ball, with a ratio of 1:1. The maximum capacity of the ball mill is 60kg, and the ratio of materials to steel ball used is 1:10. The grounding method was in circular motion with a predefined angular velocity below the critical value for optimal grind. The grinding of the materials occurs by the collision between the steel balls with materials, which will provide both the pressing and abrassion action. By grinding the materials, it also helped in increasing the reactivity by altering its physical properties in terms of increased specific surface area. The main factor that is affecting the physical properties of materials in grinding is the duration of grinding, abrasion hardness of the media and grinding medium to media ratio. The table below shows the grinding durations of RHA and the changes towards its respective fineness and specific gravity after grinding for a given duration.

Table 4: The Grinding duration of RHA, its fineness and specific gravity after grinding for a specific duration.

Duration (Hour)	Specific Gravity (g/cm^3)	Blaine Fineness (cm^2/g)
0H	1.977	1503
0.5H	2.062	6131
1H	2.062	8443
2H	2.105	9116
3H	2.116	12588

Based on the grinding time, the author chooses 0.5 hour (30 minutes) of grinding time as it is comparable to cement. If the RHA was grind for more than 3 hours, the particles became too fine and are not suitable to be used as an admixture as it will possess detrimental effect towards the mix.

COMPRESSIVE STRENGTH TEST

The compressive strength of the specimens were determined by subjecting load to a portions of prisms broken in flexure conforming to ASTM standards: C349-97 (ASTM, 2002b) standard testing method. This test method covers the determination of the compressive strength of the specimens using for the specimens portions of prisms made and broken in flexure in accordance with ASTM Standards: C348(ASTM, 2002a). The instrument used for this test is the GOTECH compression test machine. The average value of compressive strength is obtained from 3 specimens in the unit of N/mm^2. Any specimens exceeded the allowable deviation value of 8.7% from average was discarded from the calculation.

FLEXURAL STRENGTH TEST

The flexural strength of the specimens was determined from a prism with dimension of 40mm x 40mm x 160mm subjected to 3 point load by using the GOTECH testing machine. The procedure of this test was performed in accordance with the ASTM Standards : C348 (ASTM, 2002a). The specimens were fabricated in triplet and the average value was obtained from the 3 specimens. Any results which exceeded the standard deviation of 10% from the average were discarded from the calculation.

Results And Discussion

MECHANICAL PROPERTIES

COMPRESSIVE STRENGTH

In terms of mechanical properties, at the early age (7 days) the optimum strength was achieved at the replacement level of 70%, which yielded a compressive strength of 24.83 MPa with not much difference with the 30% level of replacement, which yielded a value of 24.72 MPa. Both 70% and 30% level of replacement succeeded the compressive strength of the control by 16%. On the age of 28 days, the highest compressive strength was achieved at the replacement level of 30% with compressive strength of 30.72 MPa which is comparable to that of control with a compressive strength value of 30.66 MPa. From the overall compression performance, it can be concluded that the optimum replacement level of RHA-GGBS in cement was 30%. All the specimens showed increment in compressive strength at the later age which compliments the hypothesis the increment of compressive strength is directly proportional to the age of the specimens, except one mix, where at the 80% level of replacement, the compressive strength shows no increment from 7 days age to 28 days age. At the replacement level of 100%, the mortar specimen shows insignificant compressive strength which yielded a compressive strength of 0.51 Mpa at the 7 days age, and 0.56 MPa at 28 days age. This is mainly due to the absence of cement in mix proportion. Cement in this case plays an important role to stabilize the ternary mix as it will act as an alkaline activator during the dissolution process of geopolymerisation. The early strength development can be related to the early hydration process of Alite (C_3S) and Belite (C_2S) in cement that forms the C-S-H gel in the matrix system of the ternary blend, the strength development was also contributed by the pozzolanic reaction of CaO and SiO_2 in the presence of water that also forms C-S-H gel. The pozzolanic reaction occurs at the later age of the specimens, where the hydration process of cement will liberate Calcium Hydroxide ($Ca(OH)_2$),that will then react with silica in RHA and forms a kind of C-S-H gel, these reactions occurs in a synergic manners towards the age of specimens. Another factor that contributed to the strength increment in the RHA-GGBS ternary blended cement mortar was the filler effect of fine RHA particles. This effect was achieved by further grinding the RHA for 30 minutes to increase its fineness, the increase in fineness contributed to the higher surface area, which then lead to the higher reactivity of the RHA particles. But Mehta et al. suggested that over grinding of RHA will cause the particles to exhibits detrimental effects (Malhotra & Mehta, 1996). By introducing the ternary system in the matrix system, it actually alters the packing density to a more packed and compact system, as the particles should be selected to fill up the voids between large particles with smaller particles and so on, leading to a smaller volume of gaps within the aggregate skeleton (Nguyen, 2011; Stovall, De Larrard, & Buil, 1986). The concept of packing density, i.e. the ratio of the volume of solids to a given volume, is introduced to evaluate the arrangement of granular mixture. Figure illustrates how the concept of packing density can be applied with three granular systems, i.e. single-, binary-, and ternary- systems (Stovall et al., 1986).

Figure 3: Typical packing arrangements of binary and ternary mixtures (Stovall et al., 1986).

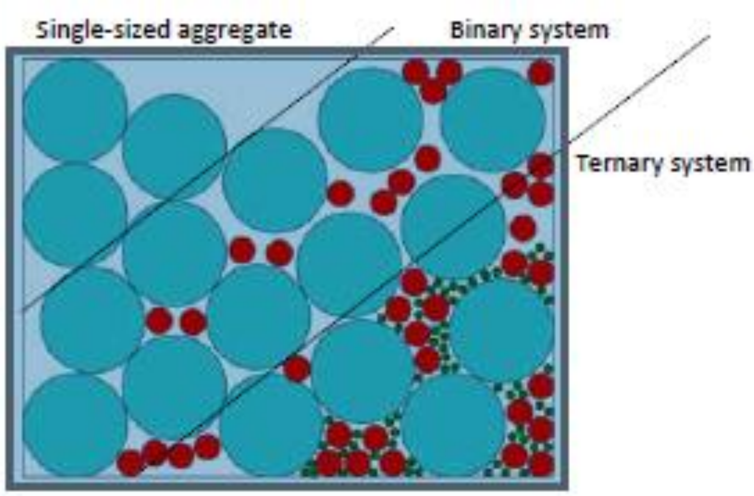
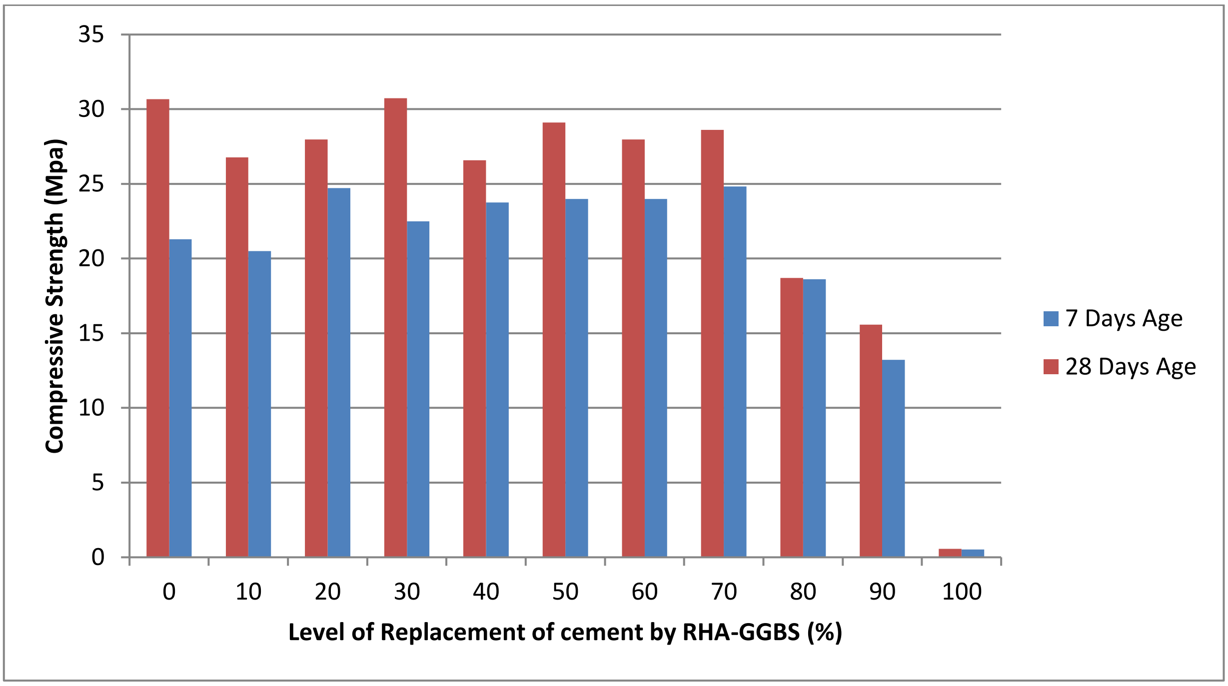


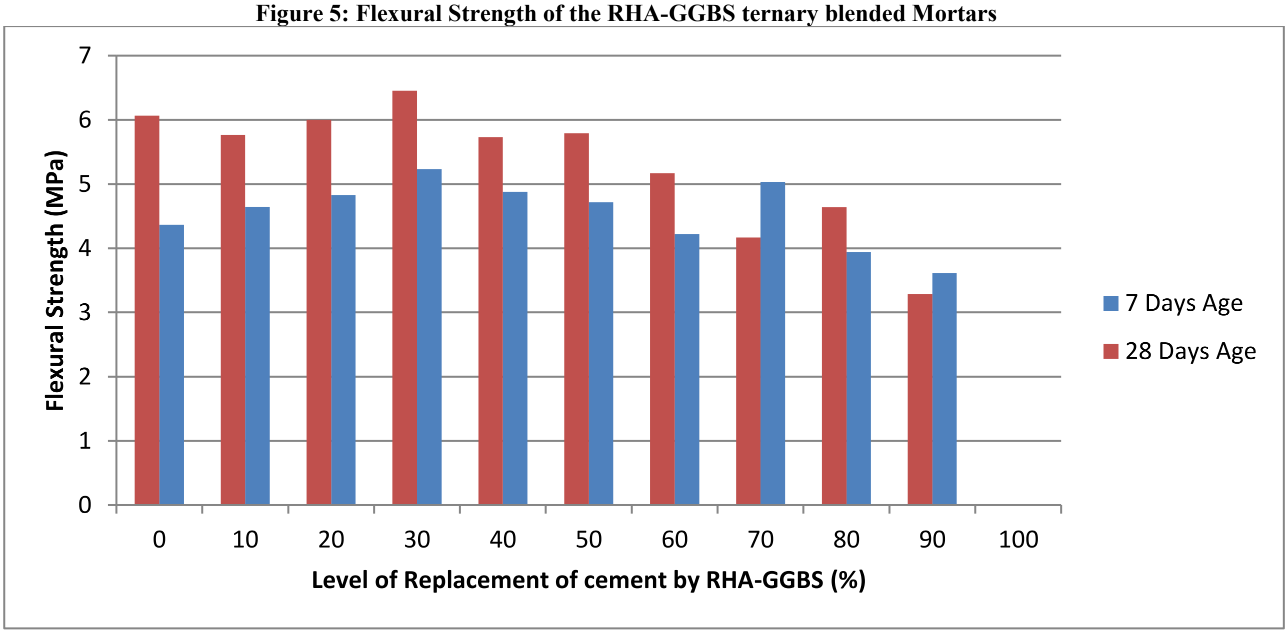
Figure 4: Compressive Strength of RHA-GGBS Ternary Blended Mortars



FLEXURAL STRENGTH

In terms of flexural strength, the RHA-GGBS ternary blended cement mortars shows an optimum value at the 30% replacement level for both 7 and 28 days of age, with flexural strength of 5.23 MPa and 6.45 MPa respectively. At 100% level of replacement, the mortar specimen exhibits no strength at all due to unstable condition of the mortars specimen. The zero strength from 100% level of replacement was attributed from the absent of cement which acts as a stabilizer or activator for the specimens, this can be clearly portrayed by the specimens of 10% to 90% level of replacement where the presence of cement stabilize the ternary blend mortar specimens.

Figure 5: Flexural Strength of the RHA-GGBS ternary blended Mortars



Conclusion

RHA and GGBS both have shown a great potential in enhancing the properties of cement mortars. Based on the results obtained from this investigation, the optimum level of cement replacement by RHA-GGBS is at the 70% replacement at the early age of 7 days and 30% at the extended curing duration of 28 days. However, there is a limitation to the research, rice husk ash is known for its high carbon residual content, it can be portrayed by the black color of the material, high energy consumption is needed to reduce the carbon content as it requires the rice husk ash to be further incinerated. The author also suggested that the method of inter-grinding of RHA and GGBS shall be commenced to increase it reactivity. In order to summarize the overall mechanical performances, it can be concluded that 30% replacement of cement using RHA-GGBS is the optimum for such purpose. Few other conclusions of the study are summarized as follows:

1. The addition of RHA improved the strength of the cement mortar. The RHA particles act as filler in between the cement particles due to the fine particles of RHA.
2. GGBS provides supplementary Ca^{2+} ions when in contact with water (H_2O), which also liberate OH^- ions that will then react with the amorphous silica in RHA to form C-S-H gel.
3. The amorphous silica in RHA reacted with the hydration products of cement, calcium hydroxide ($Ca(OH)_2$) to form secondary C-S-H gels at the early age of hydration which contributed to the high rate of early mechanical strength development.
4. The ternary system (RHA-GGBS-CEMENT) increased the packing densities of specimens by filling up of voids between large particles with smaller particles and contributed to the strength development.
5. The replacement of CEM II with the use of RHA-GGBS binary supplementary cementitious material up to 70% by binder weight is possible in order to achieve the equivalent 28 days compressive strength performance as compared to the use of CEM II only in cement mortar fabrication.
6. In conclusion, the ternary blended mortar could yields a higher mechanical strength compared to that of control mortar, provided that the replacement level of RHA-GGBS is at the range of 10% - 30% optimum. This shows that RHA-GGBS ternary blended concrete has a high potential in producing a concrete/mortar with similar or higher strength value compared to conventional concrete/mortar.

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