Mechanical and microstructural study of rice husk ash geopolymer paste with ultrafine slag

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Abstract. This paper presents the mechanical and microstructural properties of the geopolymer paste which was developed by utilizing the industrial by-products, rice husk ash (RHA) and ultra-fine slag. Ultra-fine slag particles with average particle size in the range of 4 to 5 microns. RHA is partially replaced with ultra-fine slag at different levels of 0 to 50%. Sodium silicate to sodium hydroxide ratio of 1.0 and alkaline liquid to binder (AL/B) ratio of 0.60 is taken. Setting time, compressive, flexural strengths were studied up to the age of 90 days with different concentrations of NaOH. The microstructure of the hybrid geopolymer paste was studied by performing the SEM, EDS, and XRD on the broken samples. RHA based geopolymer paste blended with ultrafine slag resulted in high compressive and flexural strengths and increased setting times of the paste. Strength increased with the increase in NaOH concentration at all ages. The ultra-small particles of the slag acted as a micro-filler into the paste and enhanced the properties by improving the CASH, NASH, and CSH. The maximum compressive strength of 70MPa was achieved at 30% slag content with 16M NaOH. The results of XRD, SEM, and EDS at 30% replacement of RHA with ultra-fine slag densified the paste microstructure.

Keywords: geopolymer; ultra-fine slag; rice husk ash; strength; setting time

1. Introduction

The manufacture of conventional Portland cement (PC) demands high energy (requires 4GJ), cost and greenhouse gases emissions (annually 13500 million) (Malhotra 1999, Malhotra 2002). It is estimated that cement industries itself contribute about 5-7% of total greenhouse emissions and CO₂ emission will rise by 50% in the future from current levels (Malhotra, 1999). The construction industry demand of Portland cement may rise up to 5.2 billion metric tons in 2020 (Green 2015) which is perceived to be unsustainable. Further, growing industrialization and urbanization led to the release of industrial waste by-products such as fly ash, rice husk ash, and slag. The environmentally-compatible disposal of industrial, agricultural and construction wastes is also another serious concern. Its disposal causes consequential environmental impacts like global warming and many other serious issues. Reports indicate the constructional waste production in terms of percentage in different countries such as Spain, England, Australia, Japan, Italy, Poland, Finland, and India as 73%, 51%, 45%, 32%, 29%, 22%, 14%, 13% in average (Zareei et al. 2017) Thus, the need of the hour to develop sustainable construction and building materials with reduced environmental footprint

through both manufacturing and operational phases is currently a key focus in the global housing and construction industry.

Globally, the engineering community and researchers have shown their serious concern towards environmental sustainability by partially utilizing industrial, agricultural and construction wastes into the construction materials.

Geopolymeric materials do not emit any greenhouse gasses during production as they utilize by-products such as fly ash, slag and rice husk ash as the precursor material. Such material, when reacted with metallic alkalis, can form binders with equal or superior engineering properties than conventional cement-based concrete. These types of Geopolymeric binders can also help in achieving sustainability of cementitious materials in the construction industry by not only reducing the energy requirements during production but also tackling the issue of industrial waste to a large extent.

Rice husk ash (RHA) is a waste by-product obtained from burning of rice husk. It is estimated that nearly 647.7 million tons of rice husk are produced per year worldwide which on burning generates approximately 22% rice husk ash (Chindaprasirt *et al.* 2010). RHA possesses good pozzolanic properties and fine particle size and is generally used as an admixture to enhance concrete properties in high-performance concrete (Safiuddin *et al.* 2010, Kartini 2011, Zareei *et al.* 2017). RHA can be activated with sodium hydroxide and sodium silicate for dissolution of Al and Si ions, to produce geopolymers. Fly ash-based geopolymers are proposed by a number of researchers who showed that mechanical and durability properties of the

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geopolymers can be enhanced by utilizing products with high calcium content (Buchwald *et al.* 2005, Dombrowski *et al.* 2007, Mehta and Siddique 2017, Nath and Sarker 2015, Jindal *et al.* 2017a, Jindal *et al.* 2017b, Jindal *et al.* 2017c, Parveen *et al.* 2018). The high calcium content led to the formation of calcium silicate hydrate (CSH) and aluminum calcium silicate hydrate (CASH) (Yang *et al.* 2008, Timakul *et al.* 2016, Punurai *et al.* 2018, Chareerat *et al.* 2006, Chindaprasirt *et al.* 2010).

Ultra-fine slag (US) is a microfine material obtained from slag through controlled granulation which is a byproduct of steel plant with non-toxic and environmentfriendly nature (Pawar and Saoji 2013). To produce geopolymers it can be used as an additive as it is rich in SiO₂ and CaO. Ultra-fine slag improves the characteristics of fly ash based geopolymer concrete (Parveen *et al.* 2018). It has also been shown that the improvement in the strength characteristics of concrete with ultra-fine slag was due to co-existence of sodium aluminosilicate hydrate (NASH), calcium silicate hydrate (CSH) and aluminum calcium silicate hydrate (CASH) gels (Jindal *et al.* 2017c, Aziz *et al.* 2019).

Several researchers have reported the studies on the mechanical and microstructural properties of concrete incorporating ultrafine materials as additives (Jindal 2019), but the prevalence of rice husk ash-ultra fine slag (RHA-US) geopolymers have not been reported vet. The current research is aimed to address the issue of environmental sustainability by utilizing rice husk as a primary aluminosilicate source in the development of geopolymer paste. Ultrafine slag is used as a source of calcium as a partial replacement of RHA in geopolymer paste. Properties like compressive strength, flexural strength, initial and final setting times are evaluated through an extensive experimental program. In addition, porosity and microstructure of the paste have been studied to promote the use of RHA and ultrafine slag for making geopolymer based binary system.

2. Experimental programme

2.1 Materials used

The chemical compositions of the RHA and ultra-fine slag are given in Table 1. RHA contains a high amount of silica content. Ultra-fine slag which is considered as a pozzolan contains a high content of CaO and Al₂O₃ which can show good reactive properties in the matrix to form the additional calcium-based hydration products.

Table 1 Chemical composition (wt%) of rice husk ash and ultrafine slag.

Raw Material	SiO ₂	Al ₂ O ₃	SO ₃	K ₂ O	P ₂ O ₅	CaO	MgO	Fe ₂ O ₃	MnO
Rice Husk Ash	90.0	0.46		4.60	2.43	1.10	0.77	0.43	0.11
Ultra-Fine Slag	33.9	22.6	0.23			32.8	7.8	1.4	



Fig. 1(a) SEM image of RHA particles, (b) XRD spectrum of RHA particles



Fig. 2 XRD spectrum of ultra-fine slag particles

The Scanning Electron Microscopy (SEM) and X-ray diffraction pattern of the RHA particles are shown in Fig. 1(a) and 1(b). It can be seen from Fig. 1 that RHA particles are not smooth and have an irregular shape. Crystalline phases in the X-ray diffraction analysis of RHA was obtained which are quartz and cristobalite. It is clear from the XRD spectra of ultra-fine slag (Fig. 2) that the crystalline phase is of calcite. A broad hump was observed at spectrum between 25°-40°, 2-theta angle and suggests the presence of glassy phase (Guo *et al.*, 2017, Timakul *et al.*, 2016), the same can be confirmed from the SEM image of ultra-fine slag.

Further, ultra-fine slag has higher amorphous content than RHA. RHA and ultra-fine slag were tested for specific gravity and reported to be 2.35 and 2.19, respectively. RHA and ultrafine slag are having the specific surface area of RHA and ultrafine slag is 14340 cm²/gm and more than 12000 cm²/gm, respectively. Sodium hydroxide and sodium silicate mixture were used as an alkaline activator. Sodium hydroxide pellets were used to prepare the NaOH solution with required molarity and sodium silicate solution with SiO₂/Na₂O between 1.90 and 2.01 was used.

2.2 Sample preparation and testing

Rice husk ash based geopolymer paste was developed using NaOH and Na₂SiO₃ (Na₂O=12.60%, SiO₂=29.25% and H₂O=58.15%) as an alkaline activator. For setting time testing, geopolymer paste was developed with different concentrations of NaOH (6, 8, 10, 12, 14 and 16M). Since 12 M solution showed optimum results for setting times, therefore for the rest of the properties, only 12M

Mix Designation	RHA (gm)	US (gm)	Na ₂ SiO ₃	NaOH
100RHA	100	-	30	30
90RHA10US	90	10	30	30
80RHA20US	80	20	30	30
70RHA30US	70	30	30	30
60RHA40US	60	40	30	30
50RHA50US	50	50	30	30

Table 2 Mix proportions of geopolymer pastes

RHA: Rice Husk Ash; US: Ultrafine slag

NaOH was used. The ratio of NaOH to Na₂SiO₃ was 1.0, to produce an economical mix and alkaline activator to binder ratio (AL/B) of 0.6 was used for the mixes tabulated in Table 2. Rice husk ash was partially replaced with the ultrafine slag particles in the experimental program to achieve the desired strength at ambient temperature. Replacement levels of 0, 10, 20, 30, 40 and 50% by weight were considered. All the fresh geopolymer mixes were mixed for about 5 min and setting time test was performed as per procedure explained in ASTM C191-13 (Punurai *et al.* 2018). Average of three samples was taken to report the setting time.

In this study, five samples of rice husk ash-based hybrid geopolymer paste for each mix were cast and tested. Size of prisms for compressive strength, flexural strength, and drying shrinkage tests were taken $50 \times 50 \times 50$ mm, $40 \times 40 \times 160$ and $25 \times 25 \times 285$ mm, respectively. All specimens were then stored in a room at ambient temperature (27°C) and demolded after 24 h. They were then kept at a dry room until the testing was done. Tests on the geopolymer paste specimens were performed at the ages of 7, 28 and 90 days and densities, compressive and flexural strengths were recorded.

Bulk density of the geopolymer paste were also determined at the age of 28 days using the prism of size 50×50×50 mm and as per the procedure described in ASTM C138/C138M-17a (ASTM 2001) The compressive, flexural strengths of geopolymer pastes was performed according to ASTM C109/C109M-16a (ASTM 2002) and ASTM C348-14 (ASTM 2014), respectively. To study the microstructure of the geopolymer mortar, samples were obtained from the broken pieces of the cubes at the age of 28 days.

The microstructure of the geopolymer mortar was studied by performing the XRD, SEM and EDS analysis. XRD spectrum was analyzed in the range of 10° to 80° two-theta angle. Peak positions in the spectrum were marked and then compared with JCPDS files. The gold coated samples were used to perform scanning electron microscopy. Mercury intrusion porosimetry (MIP) was used to measure the porosity of each specimen at the age of 28 days and calculated using the Washburn equation (Washburn 1921).

3. Results and discussion

The results of the tests which were conducted for the study are discussed here.



Fig. 3 Variation in initial setting time with the different percentage of ultra-fine slag



Fig. 4 Variation in final setting time with the different percentage of ultra-fine slag

3.1 Setting time

In this section, the effect of incorporating the ultra-fine slag in rice husk ash based geopolymer paste on the setting time properties has been discussed. The initial and final setting times of the RHA based geopolymer paste with different percentage of ultra-fine slag and NaOH concentration are presented in Fig. 3 and Fig. 4.

Fig. 3 and Fig. 4 documents the relation between variations in setting time of the geopolymer paste as a function of ultra-fine slag contents. It can be seen from the experimental observations that setting times of the geopolymer paste with ultra-fine slag were considerably lower than the geopolymer paste with 0% ultra-fine slag. 10% addition of ultra-fine slag in the mix reduced initial setting time (IST) from 111 min to 86 min and final setting time (FST) from 168 min to 135 min at a concentration of 6 M NaOH. Initial and final setting times of geopolymer paste with 0% ultra-fine slag, 6M NaOH was 111 min and 168 min, respectively, which further reduced to 95 min and 135 min when 16M NaOH was used. Although, the decreasing trend of setting time continues with the increase in NaOH concentration and ultra-fine slag content but with a marginal difference in percentage reduction. Maximum reduction of about 79-80% in setting time (IST & FST both) can be done by using 50% ultra-fine slag and 16M NaOH.

On average, setting time reduced with the increment of the ultrafine slag and NaOH concentration in all the geopolymer mixes. This can also be related to lower



Fig. 5 Effect of ultra-fine slag on compressive strength at different ages

leaching of calcium, alumina and silica ions (Guo *et al.* 2017, Hoy *et al.* 2017, Punurai *et al.* 2018), higher water demand of RHA (Givi *et al.* 2010) and finer particle size of the ultra-fine slag (Parveen *et al.* 2018).

In ordinary Portland cement (OPC) based paste and geopolymer paste, setting time is directly based on the development of calcium silicate hydrate gel (C-S-H) and sodium aluminate silicate hydrate gel (N-A-S-H) (Parveen *et al.* 2017, Glasser and Zhang 2001), respectively. However, due to the presence of CaO in the ultra-fine slag additional C-S-H may have formed in the matrix which might be responsible for the reduction in the setting time. Similar results were reported by the Saha *et al.* (Saha and Rajasekaran 2017), they reported that CaO present in the slag extended the setting time. Thus, RHA based geopolymer paste have substantial initial and final setting time as observed above (Lee and Lee 2015) and may appreciably be used for construction works.

3.2 Compressive strength

The average values of compressive strength for rice husk ash based geopolymer paste incorporating ultra-fine slag are reported in Fig. 5.

Fig. 5 documents the results of compressive strength at various ultra-fine percentages for all the studied geopolymer paste. The compressive strength of 100RHA, 90RHA10US, 80RHA20US, 70RHA30US, 60RHA40US and 50RHA50US pastes were 16.7, 27.3, 36.1, 49.0, 47.2 and 43.2 MPa, respectively, at the age of 7 days. This shows that the new geopolymer paste achieved 63%, 116%, 193%, 182%, and 158% higher strength compared to 100RHA paste, respectively. The maximum compressive strength of 49.0 MPa was obtained with 30% ultra-fine slag at 7days. Similarly, at 28 days, the compressive strength of 100RHA, 90RHA10US, 80RHA20US, 70RHA30US, 60RHA40US and 50RHA50US pastes were 28.1, 38.5, 43.3, 51.3, 49.2 and 45.2 MPa, respectively. This corresponds to the increase in compressive strength of about 37%, 54%, 82%, 75%, and 60%, respectively, when compared to 100RHA paste. Strength increment at 28 days was low compared to 7 days strength. The compressive strength of 100RHA and 70RHA30US pastes at 7, 28, 56 and 90 days were 16.7,



Fig. 6 Effect of ultra-fine slag on flexural strength at different ages

28.1, 28.2, 29.8 MPa and 49.0, 51.3, 58.9, 59.1 MPa, respectively.

This reveals that compressive strength increases with age, however, to obtain the best results for compressive strength, the optimal percentage of ultra-fine slag in replacement to RHA in the paste would be 30% at all the ages. The increase in compressive strength after 28 days was not much high, this corresponds to 1-9% and 5-15% increment in compressive strength at 56 and 90 days when compared to 28 days compressive strength. Al, Si and Ca ions leached out in the presence of NaOH from the starting materials. Also, calcium present in the matrix reacts and develop CSH, CASH bonds along with NASH in the matrix which are more pronounced for setting and hardening (Phoo-ngernkham et al. 2016). CSH, CASH and NASH gels combined together and form a hybrid product C(N)ASH gel which increases with the percentage of ultrafine slag and responsible for the development of strength and filled the pores in the matrix (Ismail et al. 2014).

Ultra-fine size of the slag could act as the microaggregate and useful in filling the micropores, also its high content increases the amorphous phase which contributes in the formation of CASH gel and in turn, resulted in high strength. Further, when ultra-fine slag content increased above 30% in the matrix, few particles do not react in the matrix and reduced the ionic strength was low due to improper hydrolysis (Pacheco-Torgal 2015). Overall, the results of this study indicated that the use of ultra-fine slag up to 30% replacement level in the matrix is beneficial to improve the strength parameters.

3.3 Flexural strength

Fig. 6 document the results of compressive strength at various ultra-fine percentages for all the studied geopolymer paste. It is clear from Fig. 6 that flexural strength increases with the increase in ultra-fine slag content moreover, with the increase in CNASH hybrid gel. Ultra-fine slag content (up to 30%) is the main factor which influences the flexural strength of the paste.

Results of this study indicated that flexural strength increases in the same way as observed in compressive strength results, for example, the flexural strength of

Table 3 Effect of ultra-fine slag on porosity at 28 days

Mix Designation	Total Porosity
100RHA	39.47
90RHA10US	53.26
80RHA20US	30.49
70RHA30US	22.48
60RHA40US	26.32
50RHA50US	28.49

100RHA, 90RHA10US, 80RHA20US, 70RHA30US, 60RHA40US and 50RHA50US pastes were 2.9, 3.7, 4.2, 4.9, 4.8 and 4.6 MPa, respectively, at the age of 7 days. This shows the increase of 27%, 47%, 71%, 68%, and 60% higher strength compared to 100RHA paste, respectively. This improvement in the flexural strength was related to the formation of CSH, NASH, CASH gels, in addition, ultra-fine slag filled the micropores in the matrix. This was confirmed through SEM images which were taken from the broken samples. Overall, flexural strength increased with ages and replacement of RHA to ultra-fine slag up to 30%.

3.4 Porosity

Table 3 shows the effect of ultra-fine slag on the porosity of the geopolymer paste at 28 days. The 28-day porosity percentages of all mixes are shown in Table 3. It can be observed from the results that the percentage porosity decreased when ultra-fine slag was added into the matrix.

However, best results were obtained at a 30% replacement level. Geopolymer paste indicates decreased porosity which may be accredited to the formation of CSH, CASH and NASH gels due to sufficient NaOH and Na2SiO3 contents. These products modified the internal pore structure of the geopolymer paste and resulted in higher strength. This has also been confirmed by Punurai *et al.* (Punurai *et al.* 2018, Ye *et al.* 2006).

3.5 XRD analysis

Fig. 7 exhibited the X-ray diffraction (XRD) patterns of the 100RHA and 70RHA30US geopolymer pastes. As shown in Fig. 7, XRD analysis confirms the presence of polymeric (Nepheline which is associated with sodium aluminate silicate hydrate (NASH)) and calcium-based products (calcium silicate hydrate (CSH) and calcium aluminate silicate hydrate (CASH)). The presence of ultrafine slag increased the peak intensities of the crystalline quartz, Nepheline, bevenite.

It is clear from Fig. 7 that higher intensities of the peaks were obtained in the ultra-fine slag based geopolymer paste and therefore, dense & homogeneous microstructure was obtained as confirmed from the SEM analysis. On the other hand, the possible reasons for the poor and loose microstructure above 30% ultra-fine slag was may be due to additional calcium-based products that hindered the polymerization process. Similar findings were also reported by Ankur *et al.* (Mehta and Siddique 2018) who reported that calcium-based products CSH and CASH improved the



Fig. 7 XRD of geopolymer paste after 28 days

strength up to a level and after that, they create hindrance in the polymerization process.

3.6 SEM and EDS analysis

Figs. 8 (a)-(f) shows the microstructure of the produced solid geopolymer pastes particles. RHA particles exhibited gaps in the 100RHA samples (Fig. 8(a)). However, the denser microstructure was obtained for 90RHA10US, 80RHA20US. 70RHA30US. 60RHA40US and 50RHA50US samples at the age of 28 days. Increasing ultra-fine slag content resulted in the development of CASH, CSH and NASH which can also be termed as C(N)ASH gel, causing a solid microstructure with almost all the reacted particles on the surface (Fig. 8(b)-8(f)). Moreover, ultra-fine slag particles could act as a micro-filler which could then enhance the mechanical properties. This phenomenon confirms the development of mechanical properties of the geopolymer pastes. Similarly, minor cracks were observed in the microstructure of the RHA based geopolymer paste with 30% ultra-fine slag (Fig. 8(d)) when compared with the 100RHA sample. This effect was consistent with ultra-fine slag content. Fig. 8(e)-8(f) shows the microcracks, voids and un-reacted RHA particles, these were due to the incomplete chemical reaction of the materials. Thus, the resulting microstructure of the 60RHA40US (Fig. 8(e)) and 50RHA50US (Fig. 8(f)) was poor than 70RHA30US (Fig. 8(d)) and porosity increased which further reduces the mechanical properties. Cracks observed in the microstructure of the paste were may be shrinkage or load-induced cracks which are generally caused due to water evaporation during curing and formed during testing, respectively. These voids and cracks reduce the strength of the geopolymer paste above 30% ultra-fine slag content.

He *et al.* (2013) and Hwang and Huyuh (2015) previously reported similar findings. Interactions between the matrix, RHA and ultra-fine slag were understood by further performing the EDS.

Figs. Fig. 9(a) and Fig. 9(b) shows SEM/EDS of the geopolymer paste at 28 days which confirmed the presence of CASH, CSH, and NASH as major chemical compositions were Ca, Si, Na, and Al. Results indicate that mechanical properties improved with the increase in Ca, Si, Na, and Al ions moreover, with the addition of ultra-fine



Fig. 9(a) SEM/EDS of geopolymer paste (100RHA) after 28 days

slag. Thus, the strength and porosity of the geopolymer paste were regulated by ultra-fine slag.

4. Conclusions

To investigate the effects of partial replacement of RHA with ultrafine slag on the fresh and hardened properties of geopolymers, experimental works were conducted. Based on the results of the experimental work, the following conclusions could be arrived upon:

• The initial and final setting times of all the geopolymer samples decreased with all the replacement levels. This finding supports the formation of extra bonds between the geopolymer paste components which led to improved hardening.

• Maximum compressive strength was achieved at 30% replacement level for all the samples and at all the ages. Further, the curing age promoted compressive and flexural strengths values.

• Development of compressive and flexural strengths was dependent on ultra-fine slag content. It can be concluded from the results of this study that an ultra-fine slag content of 30% was the optimum values for the RHA and ultra-fine slag -based geopolymers with 12M NaOH solution.

• Chemical analysis showed the presence of aluminosilicate and calcium silica hydrates in RHA and ultra-fine slag based geopolymers. Quartz, mullite, cristobalite, NASH, and CSH were the major crystalline phases observed in the resulted geopolymers.

• Denser structure of the geopolymers derived from RHA and ultra-fine slag indicated in SEM observation which supports the strength development of the geopolymers in presence of ultra-fine slag.

• Overall, ultra-fine slag along with RHA can be utilized to develop the geopolymer paste which not only facilitates the safe disposal of agricultural and industrial wastes but also improved the compressive, flexural strengths and porosity.

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