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Correlation between Compressive Strength and Fire Resistant Performance of Rice Husk Ash-Based Geopolymer Binder for Panel Applications

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Abstract. Panel structures which are mainly used as insulation materials should possess high fire resistance characteristic. In addition, their mechanical requisites for walls and doors such as compressive strength must not be unduly compromised. Rice husk ash (RHA) was used as an aluminosilicate source and two factors namely RHA/AA ratio and NaOH concentration were analyzed using statistical tool to study the effect of both factors on the compressive strength. Surface morphology and fire resistant behavior of four selected samples based on their compressive strength (brittle, semi-brittle, ductile, and semi-ductile samples) were studied to determine the correlation between compressive strength and fire resistant performance of those selected samples. Results showed that RHA-based geopolymer sample recorded high compressive strength above 28 MPa when its RHA/AA ratio and NaOH concentration were high ranging from 0.7 to 0.8 and 12M to 14M, respectively. Brittle geopolymer sample (GS) with low Si/Al ratio shows high compressive strength together with high degree of geopolymerization. Ductile GS in comparison, shows low compressive strength irrespective of its degree of geopolymerization. Semi-ductile GS showed the best fire resistant properties with a maximum non-exposed surface temperature of only 50°C after 50 minutes (after it was exposed to a direct fire with temperature of 900°C) followed by semi-brittle and brittle GS.

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1 Introduction

Rice husks (RHs) is a byproduct of paddy rice processing. It is a good source of renewable energy and weight approximately 20% of the harvested paddy. In 2008, about 661 million tons of rice were produced worldwide yieldingly 132 million tons of RHs [1]. In Malaysia, approximately 3.6 million tons of RH was produced annually [2]. RHs is one of the largest available biomass resources and the low cost of RH [3, 4] may potentially reduce the costs of geopolymer through reducing cost of production compared by using other resin systems available in the market such as polyimides, cyanide esters, silicone, epoxy, polyurethane and acrylic resins.

Geopolymer is known for its good mechanical properties and high early-aged strength. The potential advantages of RHA-based geopolymer material, in enhancing its mechanical and thermal properties, are mainly associated with RHA which having high silica content. Compared to fly ash or other aluminosilicate sources, RHA contained the highest amount of silica which is approximately 85 to 95% and relatively low alumina content which is approximately 0.5 to 2.0%. Higher loading of RHA will result in higher silica content which consequently leads to high Si/Al ratio. Therefore, high compressive strength can be achieved as proved by Komnitsas et al. [5] and Silva et al. [6]. Although high Si/Al ratio proved to provide higher compressive strength, further increase in Si/Al ratio has a tendency to reduce the compressive strength as studied by Songpirijakij et al. [7].

Some existing materials for wall panels in the market have poor fire resistant properties which made from plastic based. Promoting the use of environmental-friendly and energy consumption materials as an alternative for the plastic materials can be considered. Applying geopolymer binder as part of the panel materials tends to improve the fire resistance properties of the panel. Even though the main criteria of some panel structures should possess high fire resistance characteristic, their mechanical requisites for walls and doors must not be sacrificed too. Moreover, intensive research study on the relationship between compressive strength and fire resistant properties of geopolymer for fire resistant panel materials are not well established yet.

Therefore, the objective of this study were to elucidate the correlation between compressive strength and fire resistant performance of different geopolymer material behavior, namely, brittle and ductile, and to study the effect of two factors namely RHA/AA ratio and NaOH concentration on the compressive strength.

2 Materials and experimental details

2.1 Design of Experiment.

Experiment was statistically designed and analyzed using Minitab software. Three replications were considered with a total number of 30 experimental runs. RHA/AA ratio and NaOH concentration were chosen as factors. Other factors, such as the ratio of AA solution, curing temperature and curing time were kept constant at 5.5, 50°C, and 7 days, respectively.

2.2 Raw materials.

Rice husk ash (RHA) provided by Maerotech Sdn. Bhd and was use as received. The RHA was then ground using planetary mill Pulverisette 4. Then, follow by sieve and the produced particle size were tested under particle size distribution test.

2.3 Sample preparation.

Geopolymer was first prepared by mixing the sodium hydroxide and sodium silicate solutions at designated ratios. The AA solution was mixed with RHA and the mixture stirred gently for 30 seconds before using mechanical stirrer for another 30 minutes until the solution become homogenous. For compressive strength test, the mixture was poured in a mould with a dimension of (12.7 x 12.7 x 25.4) mm. For fire resistant test, the mixture was casted in mould with dimension of (100 x 100 x 10) mm. The samples were then cured in an oven for 24 hours followed by 6 days at room temperature for complete curing.

2.4 Compressive strength test.

The compressive test was conducted by using the Instron 3382 Floor Model Universal Testing System in accordance with ASTM D695 standard. The specimen was placed between the compressive plates and load was applied at constant speed of 1.30 ± 0.13 mm/min.

2.5 Fire resistant test.

The fire retardant tests were conducted by using the ThermaCam S500 (infrared camera). Distance between infrared camera and sample, (60 cm) and between sample and blow, (7 cm) were kept constant throughout the test. Geopolymer samples were exposed to direct fire with temperature around 900°C for at least 20 minutes or until equilibrium temperature was reached. Temperature on the back of the samples was plotted as a function of time.

2.6 Morphology analysis.

Experimental data were analyzed using MINITAB 7. Scanning electron microscopy (SEM) was performed using JEOL JSM-6400 to analyze the microstructure of selected geopolymer coating.

3 Result and discussion

3.1 Raw materials.

Size of RHA particles were in the range of 3.4 to 59.5 µm, with an average size aggregate of approximately 22.8 µm. The chemical composition of RHA is shown in Table 1. SiO2 was found to be the major constituent in RHA.

Table 1. Chemical composition of RHA

Component	SiO_2	PdO	Al_2O_3	Fe_2O_3	CaO	K_2O	Cr_2O_3	MnO	NiO	CuO	ZnO
Mass %	87.4	6.0	3.0	1.49	1.40	0.49	0.27	0.19	0.068	0.045	0.035

Sodium silicate solution used comprised of H2O = 30.3%, Na2O = 11.9% and SiO2 = 57.8%. Sodium hydroxide (NaOH) pellets with 97% purity was used and five different concentrations were prepared using NaOH pellets dissolved in de-ionized water.

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3.2 Effect of factor on compressive strength.

Figure 1 illustrates the contour plot on effect of RHA/AA ratio and NaOH concentration on compressive strength of geopolymer binder. In geopolymerization process, use of high concentration of NaOH promotes greater dissolution of RHA and improves bonding between particles. As can be seen from Figure 1, it is clear that compressive strength increased with increasing the value of both factors which are NaOH concentration and RHA/AA ratio. Higher compressive strength of more than 28.00 MPa was observed in the range of 0.7 to 0.8 for RHA/AA ratio and 12M to 14M of NaOH concentration. Geopolymer binder contained NaOH solution with concentration of 10M and S/L of 0.85, is sufficient to achieve remarkable compressive strength up to 34.60MPa. It was reported that high concentration of 12M alkaline activator solution in FA-based geopolymer will promote rapid dissolution and hydrolysis, thus increasing compressive strength [8]. However, the compressive strength value starts to decrease as the S/L ratio reduces from 0.85 to 0.25, even though the NaOH concentration is maintained at 10M. This indicates that high volume of water presence during preparation of alkaline solution which affects the geopolymerization process. In addition, the S/L ratio for geopolymer based on RHA was found to be nearly similar to the S/L geopolymer based metakaolin which is 0.8 [8]. The low S/L ratio indicated that the source materials require higher liquid demand due probably to its finer particle size and difference in particle shape. However, increased further the NaOH concentration, lead to the reduction of the compressive strength at any S/L ratio. This is due to the formation of aluminosilicate gel precipitation at the early stages of the mixing process and hence decrease the compressive strength property of the geopolymer binder [9].

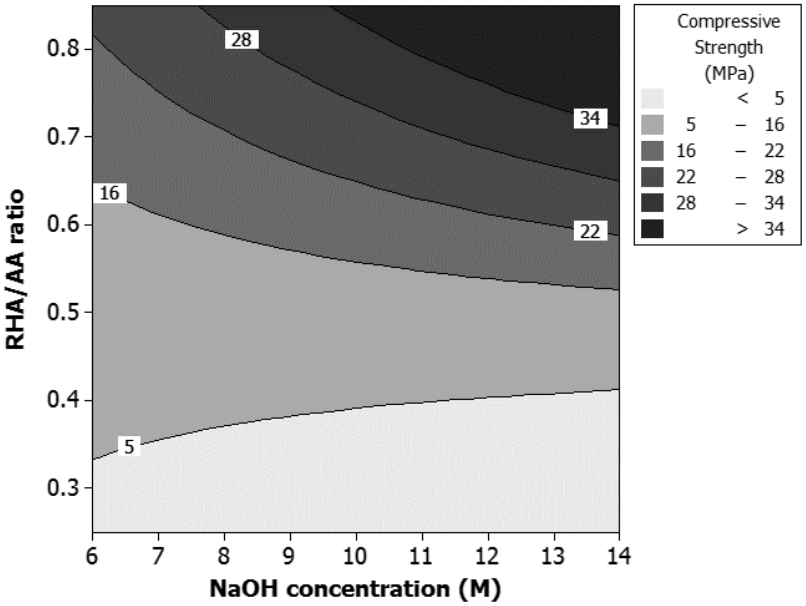

Fig. 1. Contour plot of the effect of RHA/AA ratio and NaOH concentration on compressive strength.

Table 2 also indicates the design matrix for the factors and all responses for 30 experimental runs. Experiment were conducted based on five factors which are the ratio of AA solution, RHA/AA ratio, NaOH concentration, curing temperature, and curing time.

However, only RHA/AA ratio and NaOH concentration were showed in Table 2 since other factors namely the ratio of AA solution, curing temperature, and curing time were kept constant for each sample at 5.5, 50°C, and 7 days, respectively. In order to easily determine the relationship between RHA/AA ratio, NaOH concentration, and compressive strength, contour plot in Figure 1 was generated based on the data shown in Table 2.

Table 2. Experimental results of compressive strength test

Sample	RHA/AA ratio	NaOH Concentration (M)	Compressive strength (MPa)	Sample	RHA/AA ratio	NaOH Concentration (M)	Compressive strength (MPa)
S01	0.40	8	0.80	S16	0.40	12	0.64
S02	0.70	12	31.07	S17	0.70	8	22.52
S03	0.70	12	33.55	S18	0.55	10	15.34
S04	0.70	8	23.20	S19	0.55	6	16.12
S05	0.40	8	0.89	S20	0.85	10	30.97
S06	0.40	12	0.48	S21	0.25	10	0.05
S07	0.55	10	14.39	S22	0.55	14	20.29
S08	0.70	12	33.32	S23	0.85	10	34.60
S09	0.55	10	13.82	S24	0.55	6	15.53
S10	0.40	12	0.65	S25	0.25	10	0.04
S11	0.55	10	15.10	S26	0.55	14	17.78
S12	0.70	8	25.49	S27	0.85	10	30.19
S13	0.40	8	1.03	S28	0.25	10	0.04
S14	0.55	10	13.31	S29	0.55	14	21.90
S15	0.55	10	14.88	S30	0.55	6	15.64

3.3 Surface morphology and geopolymer properties.

Study on the surface morphology was performed on four selected samples of geopolymer binder based on their compressive strength properties. The samples were S23 and S7 which exhibited brittle and semi-brittle behavior, whereas S28 and S5 which were exhibited ductile and semi-ductile behavior. Figure 2 shows SEM images of four geopolymer samples (S28, S5, S7 and S23) with different material behavior subsequent to compressive test. Sample S28 showed that its amorphous matrix was homogenous with no obvious crystalline precipitates present. The material initiated no cracks and had large plastic deformation range (which enabled it to stretch a few times its original length). Sample S5, S7, and S23 initiated minor to major cracks due probably to the presence of voids that initiate formation of cracks in the structure of the geopolymer binder. During plastic deformation, presence of voids will increase in number and grow bigger. Finally, the sample will fracture and form cracks due to the growth and coalescence of the voids [10]. Sample S5 had a homogenous amorphous matrix with very minimal unreacted RHA particles which were bonded in the matrix. Although no void can be seen on the surface of sample S5, there were microvoids present which initiated the cracks. Cracks formed in sample S5 were fewer and narrower compared to those in sample S7.

Sample S7 and S23, as shown in Figure 2 (c) and (d), had a non-homogeneous amorphous matrix with some of the RHA particles remaining unreacted but were bonded in the matrix. However, the unreacted particles had only minor effect on compressive strength since they were not acting as filler [11]. Compared to sample S7, the higher number and larger size of voids in S23 led to wider and higher number of cracks formed. Although S23 had a higher number and larger size of voids compared to the other three samples, it still exhibited the highest compressive strength. However, this sample failed under compression load at a rate of four times faster compared to that in S7. The wide range of behavior shown in geopolymer binder suggests wide option potentials in industrial applications.

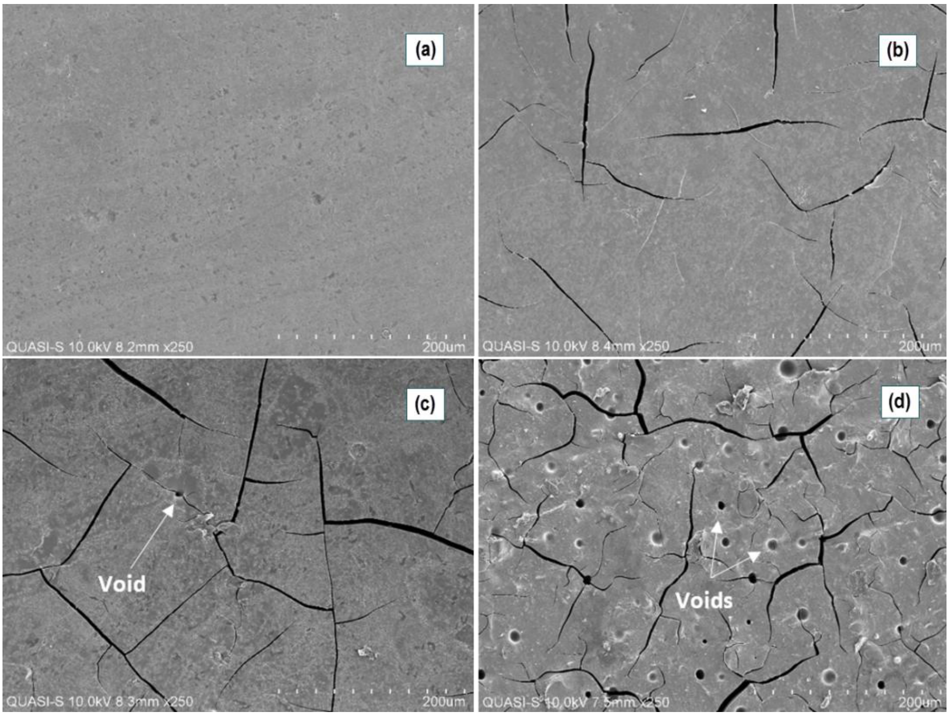

Fig. 2. The SEM micrographs of RHA-based geopolymer after compressive test; (a) S28 – ductile, (b) S5 - semi-ductile, (c) S7 - semi-brittle, and (d) S23 – brittle.

Semi-brittle material such as S7 possessed lower compressive strength compared to the brittle sample S23. The behavior of the former material was due to its lower viscosity caused by a higher water content and Si/Al ratio as shown in Figure 3. A semi-ductile material, such as S5, possessed higher compressive strength due to lower water content and Si/Al ratio compared to fully ductile material S28.

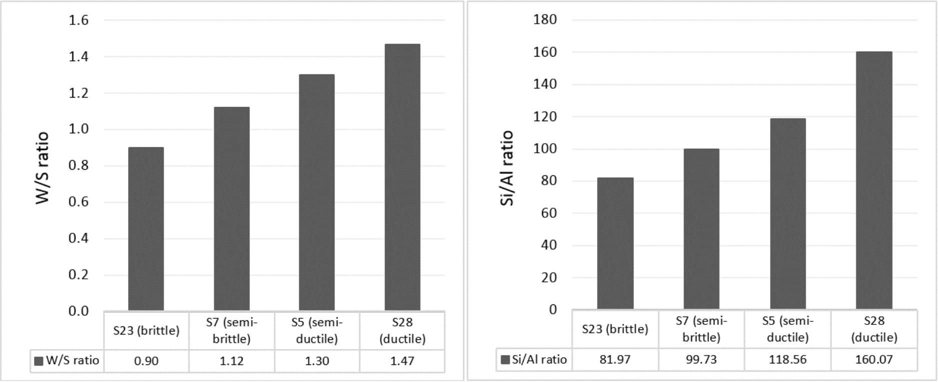

Fig. 3. W/S ratio and Si/Al ratio in selected brittle and ductile samples.

3.4 Fire resistant performance.

Sample S28 which exhibited ductile behavior has poor compressive strength and fire resistant behavior as shown in Figure 4 (a). One of the major reasons for this is the high water content. When the sample was heated rapidly to a high temperature, part of its water content, in the form of bound or absorbed water, evaporated. Pressure developed simultaneously in the pores of the material vacated by the evaporation. According to Luna-Galiano et al. [12], evaporated water molecules supposedly travel from the hot fire-exposed surface to the inner part of the material which was cooler. Due to rapid evaporation rate, however, the material pores expanded faster resulting in a weak matrix structure and the release of the evaporated water from the material. Simultaneously, the intumescent process was not able to take place and the geopolymer surface could not crystallize in time. With the failure of the geopolymer system, heat and fire were able to travel through the binder matrix and created a hole.

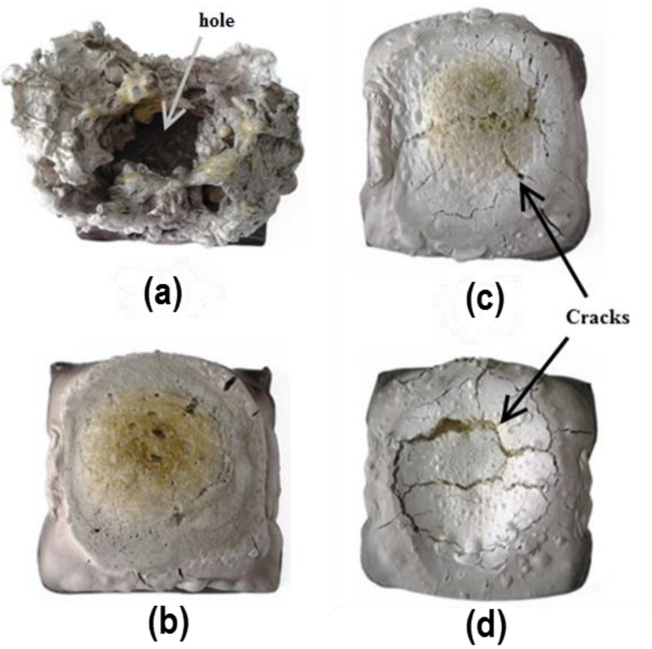

Fig. 4. Images of RHA-based geopolymer samples (a) S28, (b) S5, (c) S7, and (d) S23 after fire resistant test.

Sample S5 (see Figure 4 (b)) which shows semi-ductile behavior produced the best fire resistant properties with a maximum non-exposed surface temperature of only 50°C after 50 minutes (see Figure 5). There were no cracks or holes formed after the fire test which indicated that the sample exhibited a proper intumescent process. A portion of the water content evaporated when exposed to the fire, and generated pressure in the pores of the material and probably in the microvoids. Since crystallization of the geopolymer surface and intumescent process successfully took place in the sample, the evaporated water molecules were transported to cooler area of the material resulting in very low temperature at the non-exposed area which has been identified previously by Luna-Galiano et al. [12].

Sample S7 (see Figure 4 (c)) which showed semi-brittle behavior also possessed good fire resistant properties. This sample had similar process as that in the semi-ductile S5. The only difference was the presence of small voids in S7 which initiated cracks when the voids and pores expanded under pressure created by evaporated water. The surface cracks directly in contact with the fire allowed heat to enter and penetrate to cooler area in the material thus raising the non-exposed surface temperature.

Sample S23 as shown in Figure 4 (d) which exhibited brittle behavior also displayed similar process as S7 and S5. However, due to more voids present in the geopolymer material and wider crack openings generated during the fire test, a higher amount of heat can penetrate and dissipate in the material resulting in higher non-exposed surface temperature of around 85°C after 50 minutes. With further propagation of surface cracks, the exposed geopolymer structure began to collapse especially at the center of the material.

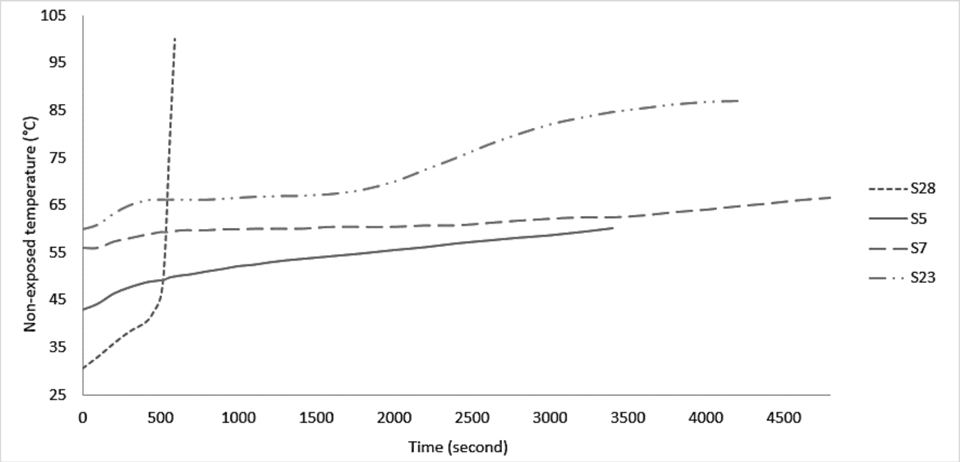

Fig. 5. Time-temperature curves of selected geopolymer coating samples.

4 Conclusion

RHA-based geopolymer sample recorded high compressive strength above 28 MPa when its S/L ratio was within range of 0.7 to 0.8, and its NaOH concentration within 12M to 14M. Brittle geopolymer sample (GS) with low Si/Al ratio (88.95) was high in compressive strength (33.55 MPa) and also showed high degree of geopolymerization. Ductile GS in comparison registered low compressive strength (0.04 MPa) irrespective of its degree of geopolymerization. Ductile GS with very low compressive strength (0.04 MPa) did not show any cracks while non-ductile GS (semi-ductile, semi-brittle, and brittle) initiated minor to major cracks. Brittle GS had higher amount of unreacted RHA particles compared to ductile ones. Water content within the GS had a major effect on its fire resistant properties. Semi-ductile GS showed the best fire resistant properties with a maximum non-exposed surface temperature of only 50°C after 50 minutes (when exposed to a direct fire with temperature of 900°C) followed by semi-brittle and brittle GS. Ductile GS however failed as fire resistant panel material due to very high amount of water which not allowed intumescent process and surface crystallization of the sample to take place.

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