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Pavel Akimov · Nikolai Vatin
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Proceedings of FORM 2021

Construction The Formation of Living
Environment

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Effect of Liquid-to-Alumino-Silicate Material Ratio and Rice Husk Ash Content on the Properties of Geopolymer Concrete



Tang Van Lam, Pham Van Ngan, and Nguyen Dac Binh Minh

Abstract This paper combines various contents of Vietnamese rice husk ash (RHA) and fly ash (FA) with an alkali-activator solution to produce geopolymer concrete (GPC). In which FA and RHA are used as alumino-silicate material in GPC mixtures. The effects of the liquid-to-alumino-silicate material (L/ASM) ratio (0.35–0.50) and the RHA content (0–60%) on the properties of the GPC were then investigated. The theoretical calculation combined with the experiment was used to determine the compositions of these GPC. The workability of fresh GPC was tested by the slump flow test. Further, the strength of specimens was performed in accordance with Russian standards. Results found that both the L/ASM ratio and RHA content greatly affected the workability of mixtures and compressive strength of the specimens. The compressive strength of GPC-specimens prepared with a L/ASM ratio of 0.45 and an RHA content of 40% exhibited higher than the control GPC-specimens. Moreover, the relationship between 28-day compressive strength and the L/ASM ratio of GPC was also determined. These results refer to the use of RHA and FA in mixes GPC is not only environmental but also cost-effective for concrete producers, as well as improved properties of the green concrete in the future.

Keywords Liquid-to-alumino-silicate material ratio · Rice-husk ash · Fly ash · Geopolymer concrete · Compressive strength

1 Introduction

Portland cement (PC) clinker is made by heating a mixture of raw materials, including limestone and clay, to a calcining temperature of above 600 °C and then a fusion temperature, which is about 1450 °C to sinter the materials into clinker. Ordinary

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Portland/pozzolanic cements manufacture are not only an energy-intensive process but also releases significant quantities of toxic fumes, and carbon dioxide into the atmosphere [1, 2]. In recent years, many investigations were focused on increasing attention on the potential for natural materials, as fly ash, bottom ash, slag, rice husk ash... to be used as a replacement for PC not only to reduce the cement content in the mixture concrete but also on the development of modern cementitious materials in construction materials manufacturing. In 1978, Davidovits [3, 4] proposed that a new binder could be produced by a polymerisation process involving a reaction between alkali-liquid solutions and compounds containing aluminium and silicon. These binders created were termed “geopolymers”. The geopolymer, including alumino-silicate material with alkaline liquids, is currently one of the most promising of these environmentally-friendly binders for green concretes in building constructions [5].

Material types containing silicon (Si) and aluminium (Al) atoms in amorphous form, which come from natural mineral additions or by-product materials, and industrial wastes, could be used as source materials for geopolymer concretes and mortar. On the other hand, some pozzolanic materials are by-product materials such as fly ash (FA), bottom ash, rice husk ash (RHA), and palm oil fuel ash etc. [6, 7]. For the manufacture of geopolymer products, the choice of raw materials depends mainly on their availability and cost, the type of application, and the specific demand of the producers [8, 9].

In the synthesis of geopolymer materials, fly ash and Vietnamese rice husk ash-based GPC specimens are provided excellent properties in both the fresh and hardened state these made them suitable materials for structural applications in green buildings [10–12].

For the geopolymer concrete and mortar, the type of alkali-liquid used plays an important role in the polymerisation process as a binder of concrete. The alkali-activator solutions (AAS) of NaOH and Na_2SiO_3 or KOH and K_2SiO_3 with different concentrations are the most common alkali-liquid solutions used in geopolymerisation [13, 14]. The previous research [9–11] showed that the engineering properties of concrete/mortar increases when waterglass (Na_2SiO_3) is added to the sodium hydroxide, compared with using only NaOH. Further, the addition of waterglass increases the ratios of Si/Al and Na/Al, resulting in increased formation of sodium aluminosilicate gel ($\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$) which indicates higher strength behavior of tested samples.

Industrial waste material, in particular FA obtained from thermal power plants (TPP), was used widely as mineral additive materials for concrete because of its pozzolanic characteristics. At present, already over a million tons of FA TPP are generated each year globally [15–17]. The class-F FA TPP, containing a high content of amorphous SiO_2 and Al_2O_3 , was used as a raw material to produce geopolymer concrete and mortar.

Moreover, rice husk ash-RHA is an agricultural waste that contains a high silica content in the form of non-crystalline or amorphous silica— SiO_2 . In Vietnam, a source of serious environmental pollution is agricultural waste, as the rice husks and straws, which are most often dumped into ponds, lakes, and rivers [18, 19]. Hence,

the reduction of agricultural waste, especially rice husks, is very necessary today. The Vietnamese rice husk ash, which has been obtained in burning rice husks and used as pozzolanic material for concrete and mortar. While rice husks or hulls are generated during the first stage of rice milling. Depending on the burning conditions of rice husks, the SiO_2 content in the RHA varies from 85 to 95 wt%, which exists predominantly in an amorphous phase and to the very large surface area. This material is reactive with the alkali-activator solution to produce the aluminosilicate gel that binds the aggregate types and provides the mechanical properties of GPC [20, 21].

Because both Vietnamese RHA and FA TPP “Vung Ang” are contained high levels of Si and Al, these alumino-silicate materials are effective for producing GPC and mortar. Furthermore, the AAS plays an important role in the dissolving process of Si–Al from amorphous silica-alumina to form gel geopolymer precursors and alumino-silicate materials. In addition, the liquid-to-alumino-silicate material ratio significantly affects the workability of mixtures and compressive strength of the tested GPC-specimens. Therefore, the overarching purpose of the current research is to investigate the effect of liquid-to-alumino-silicate material ratio and RHA content on the properties of fly ash and rice husk ash-based geopolymer concrete.

2 Materials and Experimental Methods

2.1 Material Properties

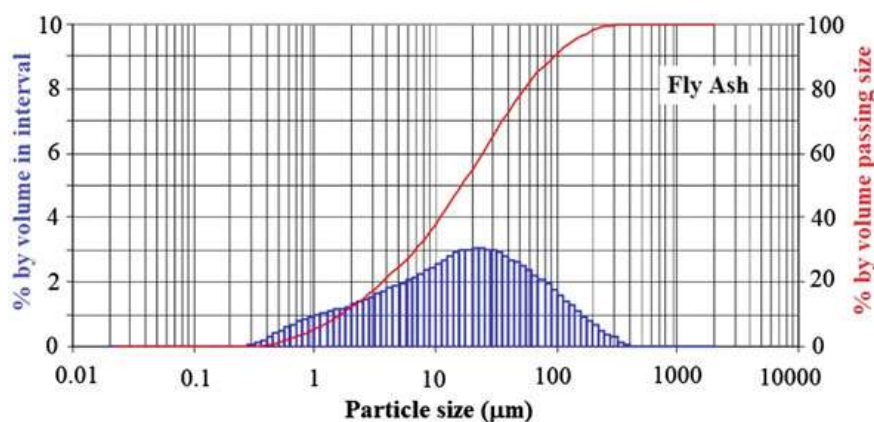
- (a) A coarse aggregate wasn’t used for making geopolymer concrete in this test. The fine aggregate in this geopolymer concrete of the mixtures was used Red River quartz sand (QS) (Vietnam) with fineness modulus $M_k = 3.0$ and a specific gravity of 2.65 g/cm^3 .
- (b) The alumino-silicate materials (ASM) used include rice husk ash -RHA and fly ash -FA from TPP “Vung Ang” (Vietnam) class-F in this study. The experimental results of chemical compositions and physical properties of RHA and FA TPP “Vung Ang”, determined by X-ray fluorescence analysis of raw materials, are given in Table 1. The class-F FA TPP “Vung Ang” was composed mainly of SiO_2 (54.62%) and Al_2O_3 (25.17%) and that the main constituent of RHA was SiO_2 (88.2%) (Fig. 1).

Particle size distributions of FA and RHA, determined by XRD analysis, are given in Figs. 2 and 3, respectively. Moreover, these results showed that the particles of RHA were significantly smaller than the particles of FA.

- (c) Alkaline liquid: In the current experimental research, a combination of Na_2SiO_3 and NaOH solutions was used as the alkali-activator solution. The molarity of this NaOH solution was 14.
 - 1. The sodium hydroxide solution was obtained by dissolving the NaOH flakes in the water. In this case of the study, taking 14 Molar of the NaOH

Table 1 Physical properties and average chemical compositions of Fly ash TPP “Vung Ang” and Rice husk ash

Materials		Fly ash TPP “Vung Ang”	Rice husk ash
Physical properties	Specific weight (g/cm^3)	2.35	2.25
	The volume of natural porous state (kg/m^3)	765	572
	Specific surface area (m^2/g)	0.755	0.850
	Mean particle size (mm)	17.6	14.8
Average chemical composition (%)	SiO_2	54.62	88.2
	Al_2O_3	25.17	1.25
	Fe_2O_3	7.11	1.75
	SO_3	0.25	0.5
	K_2O	1.28	1.14
	Na_2O	0.2	2.67
	MgO	1.57	0.8
	CaO	1.45	0.52
	TiO_2	2.35	0.15
	P_2O_5	765	0.25
	Loss on ignition	0.755	2.77

**Fig. 1** Particle size distribution of Fly ash TPP “Vung Ang”

solution, this can be obtained by mixing 42.4% of a NaOH solid with 57.6% of water. This solution’s specific gravity is $1.45 \text{ g}/\text{cm}^3$.

2. The Na_2SiO_3 liquid used was procured from Viet-Tri Co., Ltd., with a ratio $\text{SiO}_2/\text{Na}_2\text{O} = 2.5$, containing 29.5% SiO_2 , 11.8% Na_2O , 58.7% H_2O , and its specific gravity of $1.55 \text{ g}/\text{cm}^3$.

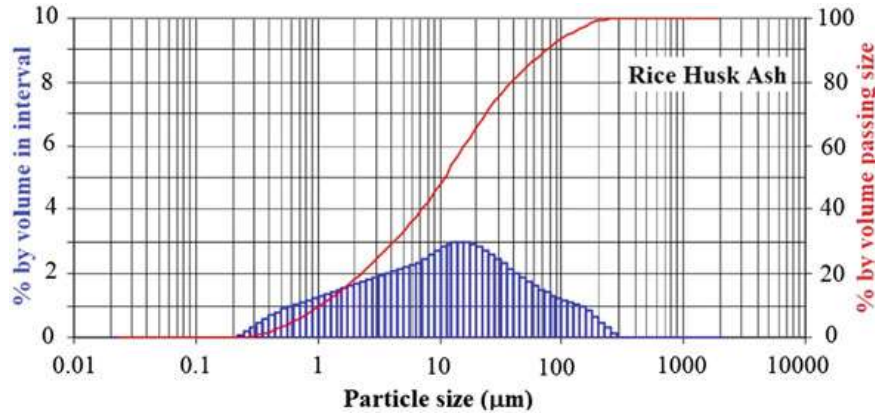


Fig. 2 Particle size distribution of Vietnamese RHA

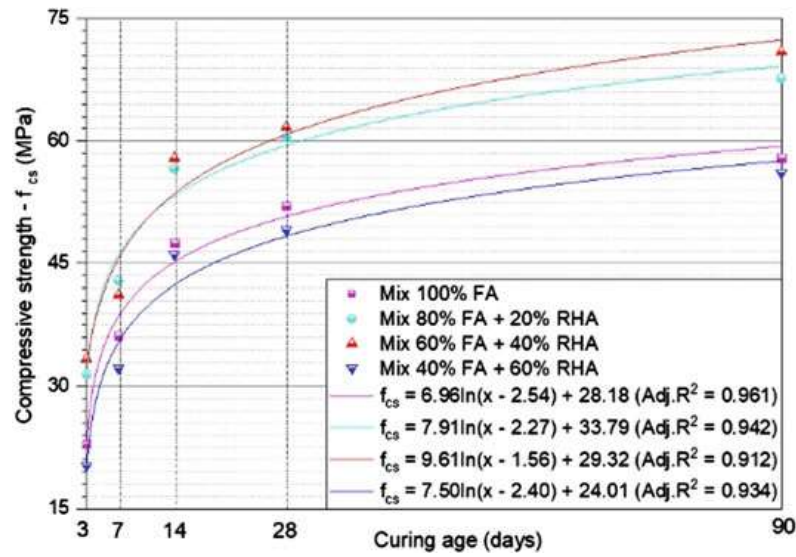


Fig. 3 The development of GPC compressive strength at $\frac{L}{ASM} = 0.35$

- (d) The super-plasticizer admixture SR-5000F “SilkRoad” (SP) was procured from Hanoi-Korea Co., Ltd., with a specific gravity of 1.12 g/cm³. This admixture was used to increase the workability of the fresh concrete and to reduce the ratios of water-cement, while increasing the strength of GPC-samples.

2.2 Methods of Test

To date, there have been very limited studies on the mixture design of geopolymer concrete raw materials, let alone the combined effects of alumino-silicate raw materials on the GPC properties. In 2008, the previous research [22–24] proposed the

method for a composition of fly ash-based GPC but this method did not discuss how to deal with the effects of a super-plasticiser content or the air content in the concrete mixture. In this study, the calculation method of the compositions of GPC mixture was applied in accordance with the absolute volume method combined with the experimental results.

- The workability of concrete mixture is determined by the standard slump cone with dimensions of $40 \times 70 \times 80$ mm by Vietnam standard TCVN 3106:2007.
- The compressive strength of GPC was conducted to evaluate the compressive strength development of the tested GPC-specimens for different time periods.

This test was performed on $100 \times 100 \times 100$ mm cubic these specimens at 3, 7, 14, 28, and 90 days of curing age using a 500 T computer-controlled compression tester machine “Controls Advantest 9” with a constant loading rate of 1000 N/s in order to keep the loading rate to a minimum rate in the processing test of concrete patterns. The compressive strength test was performed in accordance with GOST 10180-2012 (Russian standard).

2.3 Sample Preparation

The sodium hydroxide solution was initially prepared by dissolving the NaOH flakes in water in concentrations of 14 Molarity. This solution was then mixed with the sodium silicate solution and allowed to cool to room temperature, while, the alkali-activator solutions had $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios of 2.5 and liquid-to-alumino-silicate materials (L/ASM) ratios of 0.33, 0.40, 0.45, and 0.50, respectively. Data of Table 2 details shows the mixture composition used in 1 m^3 of the concrete by weight. Furthermore, a quartz sand-to-alumino-silicate materials ratio of 1.30 was maintained for all of the mixtures in this study. The super-plasticizer “SR-5000F SilkRoad” is equally to 1.0% by mass of ASM ($\text{ASM} = \text{RHA} + \text{FA}$) [5]. In addition, using the RHA to replace from 0, 20, 40, to 60% of mass the FA TPP “Vung Ang” in the geopolymer concrete mixtures. Besides, relative volume of entrapped air is 3.0% in 1 m^3 of the tested concretes.

In this work, all the tested patterns of geopolymer concrete series were demoded 24 h after casting. Next, the cubic geopolymer samples were heated at 100°C for 6 h in the oven and subjected to standard maintenance Treatment (temperature $20\text{--}25^\circ\text{C}$ and 90–95% relative humidity) until the required testing ages.

3 Results and Discussion

The all compositions of GPC-mixture used in this investigation are calculated in Table 2. Therefore, density, and slump of fresh geopolymer concrete, as shown in Table 2.

Table 2 Mix compositions, density, and slump of GPC-mixture

No.	Compositions of GPC-mixture	$\frac{L}{ASM}$	Mix proportions of ingredients (kg/m ³)						Density (kg/m ³)	Slump (cm)
			FA	RHA	NaOH	Na ₂ SiO ₃	QS	SP		
Mix-01	100% FA	0.35	840	0	84	210	1092	8.4	2233	15.5
Mix-02	100% FA	0.40	816	0	93	233	1061	8.2	2212	15.5
Mix-03	100% FA	0.45	794	0	102	255	1033	7.9	2193	16.5
Mix-04	100% FA	0.50	774	0	111	276	1006	7.7	2174	17.5
Mix-05	80% FA + 20% RHA	0.35	670	167	84	209	1088	8.4	2226	14.5
Mix-06	80% FA + 20% RHA	0.40	651	163	93	233	1058	8.1	2205	15
Mix-07	80% FA + 20% RHA	0.45	634	158	102	255	1030	7.9	2186	16.5
Mix-08	80% FA + 20% RHA	0.50	617	154	110	275	1003	7.7	2167	17.5
Mix-09	60% FA + 40% RHA	0.35	500	334	83	209	1084	8.3	2219	14.5
Mix-10	60% FA + 40% RHA	0.40	487	324	93	232	1055	8.1	2198	14.5
Mix-11	60% FA + 40% RHA	0.45	474	316	102	254	1026	7.9	2179	16
Mix-12	60% FA + 40% RHA	0.50	461	308	110	275	1000	7.7	2161	17
Mix-13	40% FA + 60% RHA	0.35	333	499	83	208	1081	8.3	2212	13
Mix-14	40% FA + 60% RHA	0.40	323	485	92	231	1051	8.1	2191	13
Mix-15	40% FA + 60% RHA	0.45	315	472	101	253	1023	7.9	2172	15
Mix-16	40% FA + 60% RHA	0.50	307	460	110	274	997	7.7	2154	15.5

Data in Table 2 was shown that the addition of RHA with different levels only slightly decreased the workability of GPC-mixtures, their slump was in the range of 13–17.5 cm when the molarity of the NaOH solution and the ratio of the sodium silicate-to-sodium hydroxide solutions remain the same in all mixes. This could be explained by (1)—the specific surface area of RHA was larger, and (2)—the RHA particles were significantly smaller than the FA TPP “Vung Ang” particles, which have tended to increase water requirement in the mixing for these concrete mixtures.

These results also are shown that with effects combined of liquid-to-alumino-silicate material ratio and SR-5000F super-plasticizer on the microstructure of mixtures concrete, different values for slump mixes concrete were obtained from different L/ASM. In the present study, the workability increase in the slump was obtained that corresponded to the increase in liquid-to-alumino-silicate material ratio from 0.35 to 0.50. The results of this investigation, similar to the results found in previous studies [5, 13].

Effect of liquid-to-alumino-silicate material ratio and rice husk ash content on compressive strength of fly-ash and rice-husk ash based geopolymer concrete samples used in this study are presented in Table 3.

Data in Table 3 presented that the compressive strength values at 3, 7, 14, 28, and 90 days curing time of the tested geopolymer concrete samples were, respectively, in the range of $20.3 \div 36.0$, $32.2 \div 44.1$, $46.1 \div 59.1$, $48.7 \div 62.9$, and $55.4 \div 73.1$ MPa. The experimental results also were shown the compressive strength development of the GPC-specimens prepared with not only different Liquid-to-alumino-silicate material ratio, but also different levels of RHA content.

Figures 3, 4, 5, and 6 are presented in detail in the development of the compressive strength of GPC-samples at the different of RHA contents.

The relations between the 28-day compressive strength— f_{cs} and the liquid-to-alumino-silicate material ratio of the GPC-samples are shown in Fig. 7.

Figure 7 shows the relationship between the liquid-to-alumino-silicate material ratio and 28-day compressive strength of GPC-samples. Similar findings were reported by Ferdous et al. [5]. The data required to plot these relationships for GPC-samples were obtained from laboratory conditions in which the cubic samples were heated at 100 °C for 6 h in the oven. The relationship is useful at the start of the calculation of the compositions in different geopolymer concrete types when the liquid-to-alumino-silicate material ratio has still not been clearly determined by the researchers.

(a) *Effect of liquid-to-alumino-silicate material ratio on strength development of fly ash and rice husk ash based geopolymer concrete*

The compressive strength of the GPC-specimens was expected to increase with increased liquid-to-alumino-silicate material ratio due to the fact that more Si and Al atoms in an amorphous phase of FA and RAH are dissolved in the alkaline solution. It can be seen in Table 4, an increase in the liquid-to-alumino-silicate material ratio from 0.35 to 0.45 clearly increased the compressive strength of the GPC. However, when the $\frac{L}{ASM}$ was 0.50, the compressive strength started to slightly decline. Based on the final results of this study, it

Table 3 Average value of compressive strength of GPC-samples at different liquid to alumino-silicate material ratio and different levels of RHA content

No.	Compositions of GPC-mixture	$\frac{L}{ASM}$	Compressive strength f_{cs} at different curing ages (MPa)				
			3-day	7-day	14-day	28-day	90-day
Mix-01	100% FA	0.35	24.9	36.6	46.5	49.9	56.7
Mix-02	100% FA	0.40	25	37.9	49.2	53	57.9
Mix-03	100% FA	0.45	25.8	38.7	51.1	55.7	61.7
Mix-04	100% FA	0.50	22.9	36.2	47.5	51.8	57.9
Mix-05	80% FA + 20% RHA	0.35	34	43.4	55.1	58.8	63
Mix-06	80% FA + 20% RHA	0.40	33.2	44.1	56.7	60	65.9
Mix-07	80% FA + 20% RHA	0.45	32.3	43.8	58.3	62.0	68.4
Mix-08	80% FA + 20% RHA	0.50	31.5	42.9	56.6	60.2	67.7
Mix-09	60% FA + 40% RHA	0.35	35.2	42.6	57.6	60.8	70.8
Mix-10	60% FA + 40% RHA	0.40	36.0	43.4	58.8	62.4	72.8
Mix-11	60% FA + 40% RHA	0.45	34.7	42.9	59.1	62.9	73.1
Mix-12	60% FA + 40% RHA	0.50	33.4	41.2	57.4	61.6	70.9
Mix-13	40% FA + 60% RHA	0.35	23.6	33.9	47.5	48.8	55.4
Mix-14	40% FA + 60% RHA	0.40	24.1	34.7	48.3	50.8	58.1
Mix-15	40% FA + 60% RHA	0.45	22.9	35.5	48.9	51.6	60
Mix-16	40% FA + 60% RHA	0.50	20.3	32.2	46.1	49.1	56.1

was observed that the GPC samples activated with $\frac{L}{ASM} = 0.45$ had the most ideal alkali-activator solution and had the highest strength average values of all tested GPC. The tested GPC-samples compressive strength determined in the current work confirms the trends observed in similar studies [5, 10, 11, 13].

(b) *Effect of rice-husk-ash content on strength development of fly ash and rice husk ash—based geopolymer concrete*

Next, data presented in Table 3 indicates that these GPC compressive strengths were increased not only with curing periods (from 3 to 90 days) but also with the increase in content of Vietnamese rice husk ash for all of tested patterns. According to the study by Hwang et al. [6], it has reported that silica atoms increase with the increase in levels of RHA content. Besides, it was believed

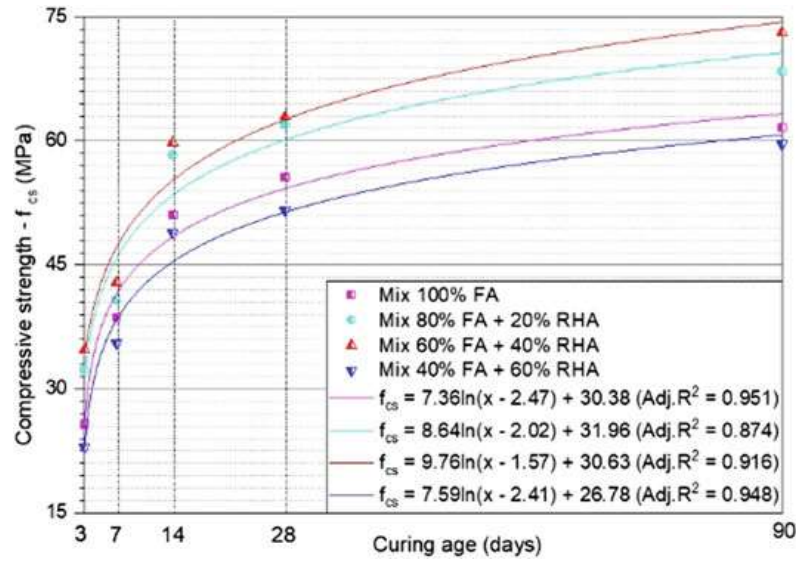


Fig. 4 The development of GPC compressive strength at $\frac{L}{ASM} = 0.40$

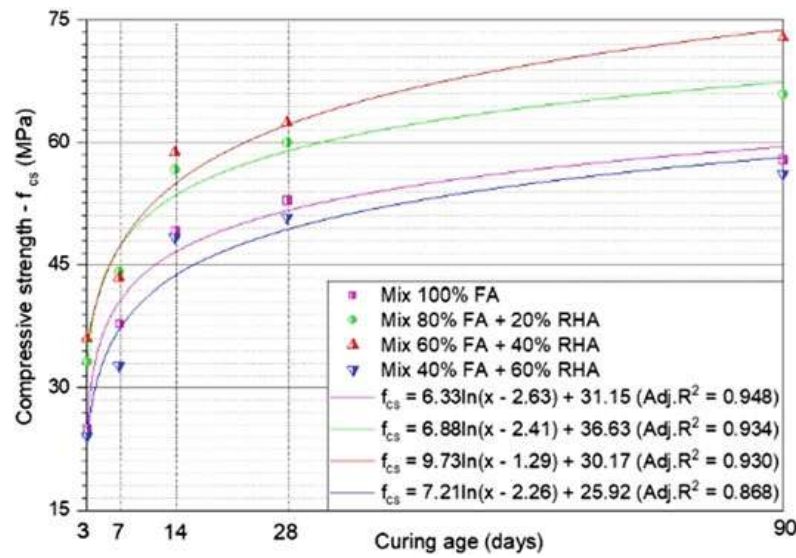


Fig. 5 The development of GPC compressive strength at $\frac{L}{ASM} = 0.45$

that Si–O–Si bonds can be stronger than either Si–O–Al bonds or Al–O–Al bonds [6, 7, 21]. And finally, this strength of aluminosilicate network in GPC structures should increase significantly with the increase in amount of rice husk ash. It is evident, in the range of this investigation, the GPC compressive strength increased with the RHA content from 0 to 40% by mass of FA TPP “Vung Ang”, then descends. The results of the current research, average value of compressive strength at the 28-day curing age of GPC-specimens these

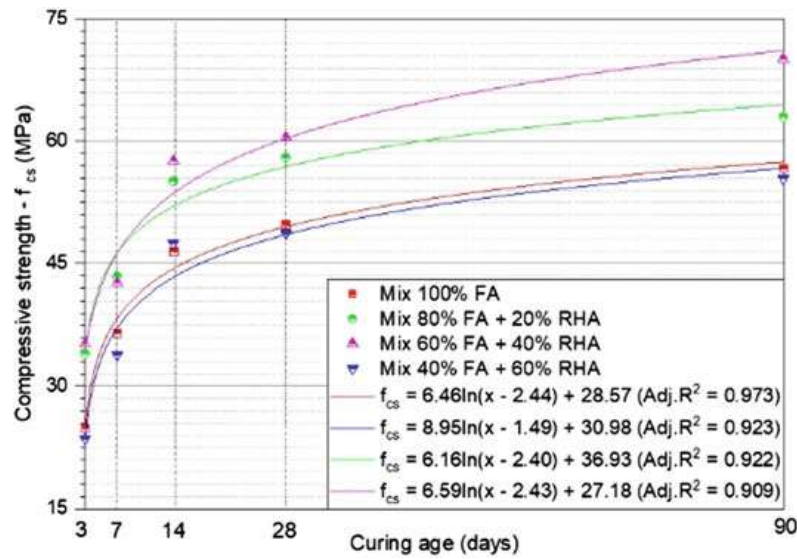


Fig. 6 The development of GPC compressive strength at $\frac{L}{ASM} = 0.50$

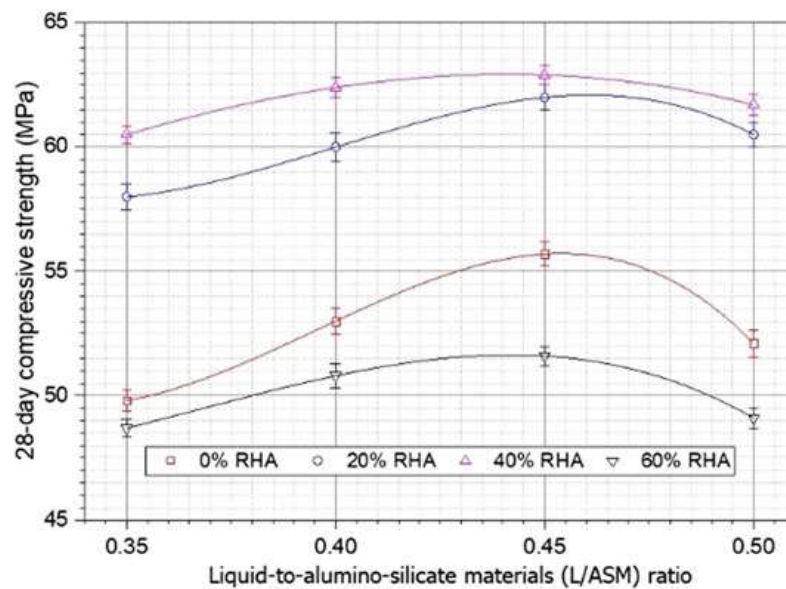


Fig. 7 Correlation between 28-day compressive strength of GPC-samples and the liquid-to-alumino-silicate material ratio

contained 0%, 20%, 40%, and 60% RHA were, respectively, in the range of $49.9 \div 55.7$ MPa; $58.8 \div 62.0$ MPa; $60.8 \div 62.9$ MPa; and $48.8 \div 51.6$ MPa.

In particular, in Figs. 3, 4, 5, and 6 were shown that the addition of Vietnamese RHA in the concrete mixes modifies significantly the GPC properties, especially with the RHA contents of 20 and 40%. These are caused by: on the one hand—the increased levels of reactive silica (Si in SiO_2) from the Vietnamese RHA resulted

in a higher density of Si–O–Si bonds, leading to higher compressive strength of the GPC-samples, on the other hand—with a higher levels of rice husk ash content containing a high specific surface area for modifying and improving the compressive strength of the GPC-specimens. Similar to the results presented in published studies by Hwang et al. [6] and He et al. [21].

Moreover, contrary to these principles, while the current work found that the strength behavior increased as Vietnamese rice husk ash increased to 40%, that strength levels start declined after the content of RHA content is 60% (Table 3 and Figs. 3, 4, 5 and 6). This could be explained by the effect of other parameters in the geopolymerization process on the engineering properties of tested GPC-samples [6, 26]. Factors possibly responsible for these phenomenon include: (1) relatively larger Vietnamese rice husk ash solid particles negatively affect the rate and extent of the geopolymerization process when this rice husk ash comprise a higher content of the GPC mixture [21], leading in weaker GPC-samples; and (2) a higher concentration of soluble silica, but lower soluble alumina, which hinders the reorganization of Si and Al atoms in the material structures and weakens tested GPC-samples [6, 11, 24]. According to experimental results of the strength behavior, the current research found a level of 40% rice husk ash amount, delivering the highest compressive strength value for the GPC-samples.

4 Conclusions and Future Work

1. Both the liquid-to-alumino-silicate material ratio and Vietnamese rice husk ash content greatly affected the workability mixtures concrete and strength behavior of GPC-specimens.
2. The compressive strength of all of GPC-specimens increased with curing times of the test from 3 to 90 days. These results of this study are supported that curing periods is promoted the development of compressive strength of GPC-specimens and was similar to the basic principles of PC concrete.
3. With regard to the FA and RHA-based geopolymer concrete, the optimum of both liquid-to-alumino-silicate material ratio and rice husk ash content was consistently associated with a higher average value of strength. The compressive strength of GPC-specimens maximized at the optimum value and then decreased gradually as liquid-to-alumino-silicate material ratio and Vietnamese rice husk ash content increased. Based on the results in the current research showed that the geopolymer samples prepared with a liquid-to-alumino-silicate material ratio of 0.45 and an RHA content of 40% exhibited high strength behaviors that were comparable with or even much higher than the control specimens of GPC.
4. The development of compressive strength in the GPC- specimens was dependent on not only the curing age of this test but also the liquid-to-alumino-silicate material ratio (0.35–0.50) and RHA content (0–60%).

5. Producing geopolymer concrete using FA TPP “Vung Ang” and RHA of Vietnam is feasible. The use of fly ash and Vietnamese rice husk ash as alumino-silicate material in mixes GPC is not only environmental but also cost-effective for concrete producers, as well as improved properties of the green concrete in the future.

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