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Experimental study on geopolymer concrete prepared using high-silica RHA incorporating alccofine

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Abstract. This paper describes the experimental investigation carried out to develop geopolymer concrete using rice husk ash (RHA) along with alccofine. The study reports the fresh and hardened properties of the geopolymer concrete (GPC) activated using alkaline solution. GPC were prepared using different RHA content (350, 375 and 400 kg/m^3), the molarity of the NaOH (8, 12 and 16M). The specimens were cured at 27°C and 90°C. GPC was activated using NaOH, Na_2SiO_3, and alccofine. Prepared GPC samples were tested for compressive and splitting tensile strengths after 3, 7 and 28 days.

RHA was suitable to produce geopolymer concrete. Results indicate that behavior of GPC prepared with RHA is similar to fly ash based GPC. Workability and strength can be improved by incorporating the alccofine. Further, alccofine and heat curing improve the early age properties of the GPC. Heat curing is responsible for the initial polymerization of GPC which leads to high workability and improved mechanical properties of the GPC. High strength can be achieved by using the high concentration alkaline solution in terms of molarity and at elevated heat curing. Further, RHA based geopolymer concrete has tremendous potential as a substitute for ordinary concrete.

Keywords: high silica rice husk ash; geopolymer concrete; alccofine; workability; strength; alkaline solution

1. Introduction

Future is happening now as CO_2 is increasing at an unprecedented rate so does the global warming. Cement industries which contribute about 7% of overall greenhouse gases are responsible for it. The production of one ton of cement emits one ton of CO_2 in the atmosphere (Malhotra 1999). It is estimated that by the year 2020, the CO_2 emission will rise by 50% from the current levels. The energy intensive process (requires 4GJ energy) used in the manufacturing of the Portland cement annually releases about 13500 million ton of greenhouse gas (Malhotra 2002). Globally, concrete as a construction material is used and OPC is the main binder. Use of water and

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concrete are the primary and secondary as construction materials, with the cement usage being 4.0 billion tons and concrete usage being 10 billion cubic meters per annum with a growth rate of 4% per annum (Gartner 2004, Jewell *et al.* 2014). By-products from industries such as ground granulated blast.

Furnace slag (GGBS), mine waste (MW), fly ash (FA), red mud (RM), RHA etc, has been partially replaced with the concrete and found satisfactory. The production of rice being the world second important cereal crop has increased from about one fifty million tons in 1960 to above seven forty million tons in 2016 (Siddique *et al.* 2016). The one ton of rice husk when burnt at elevated heat produces approximately 0.185-0.20 tons of black ash (Bouzoubaa *et al.* 2001). Generally, RHA is used as a partial replacement to cement in ordinary concrete which reacts with the calcium hydroxide present in the matrix and produces an additional C-S-H gel. RHA serves as a viscosity modifier, microvoids filler (Mehta 1992) in ordinary concrete. This effect of the RHA in matrix refines the microstructure and it will be interesting to know the same when GPC is prepared using RHA. Geopolymer concrete is paving a path of better alternative construction material in terms of lowering the greenhouse gases, as it can reduce the CO_2 by 80% which is caused by cement industries itself only (Gartner 2004). GPC is hoped to be a sustainable material which may serve as a better alternative to OPC concrete by effectively utilizing industrial wastes like rice husk ash etc. (McLellan *et al.* 2011, Parveen *et al.* 2013, Slaty *et al.* 2015) and maintaining environmental standards.

Literature revealed that RHA can be partially replaced to fine aggregates, cement in ordinary concrete and shows great potential (Mehta 1992, Ganesan *et al.* 2008, Sanusi *et al.* 2014). However, less data is available on the strength properties of the RHA based GPC. Several studies have been done on fly ash based GPC but no such work have been reported which discuss the effects of different variables on the RHA based GPC. This study is focused to carry out the properties of the RHA based GPC and to promote its use as a precast member in the construction industry due to its economic and environmental benefits. GPC prepared in this study is made by mixing all the ingredients along with alccofine which acts as a micro-filler and as an activator to achieve better compressive strength as well as improved workability (Jindal *et al*. 2016, Jindal *et al.* 2017). The properties of this new class of material are studied in terms of workability, compaction factor, compressive and split tensile strength. Also, the relationship of the RHA content with the other parameters are derived. The relationship between compressive and split tensile strength of the RHA based GPC is discussed.

2. Research significance

Literature available on geopolymer concrete is based on fly ash or partially replacement of fly ash with the slag and rice husk ash. In this study efforts have been made to develop the geopolymer concrete using RHA with an aim to achieve the workability in fresh state and improved mechanical properties at room temperature as well as at elevated heat. Alccofine was used as an additive and to improve the strength at room temperature.

3. Experimental approach

3.1 Materials for geopolymer concrete mixture

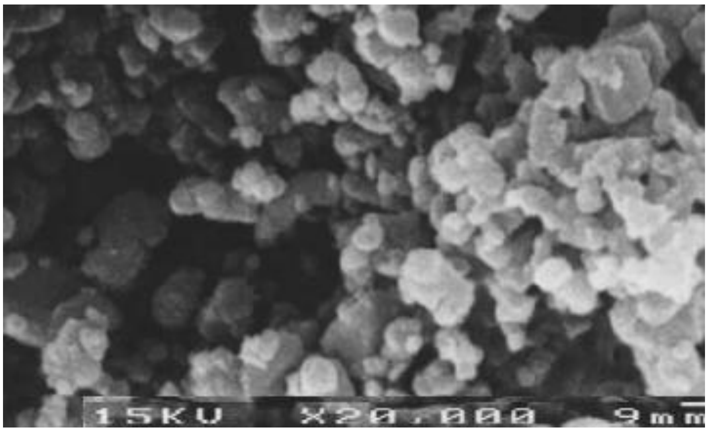
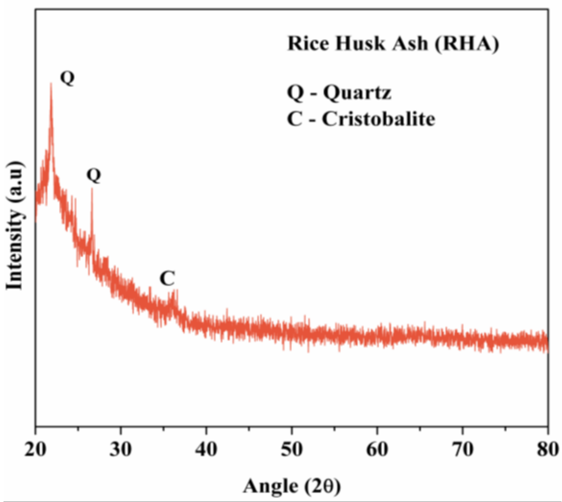


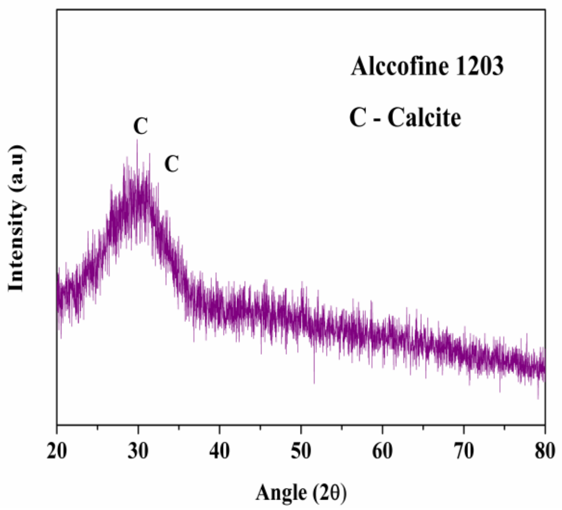
Fig. 1 SEM image of RHA particles

Table 1 Chemical composition and physical properties of processed rice husk ash

Sample	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3(%)	SO_3 (%)	CaO (%)	Na_2O (%)	LOI (%)	Specific surface area (m^2/kg)
RHA	92.96	0.14	0.05	1.32	0.45	0.29	3.24	1434.5
Alccofine	35.30	21.40	1.20	0.13	32.20	-	-	1200.0



(a) RHA



(b) Alccofine 1203

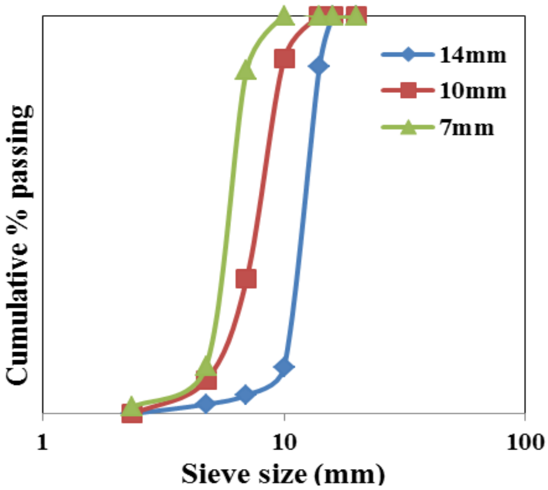
Fig. 2 XRD spectrum of RHA and Alccofine 1203

3.1.1 Rice husk ash

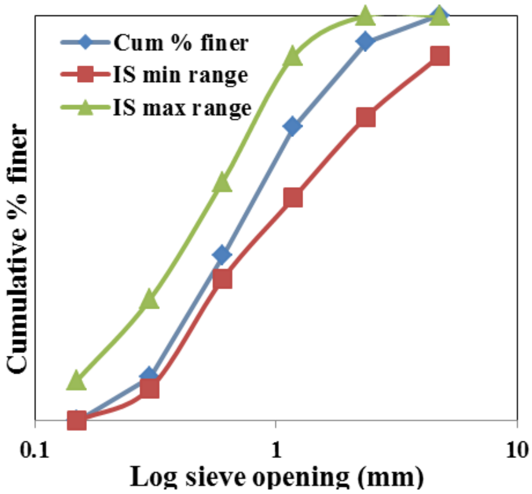
In this study, thermally treated high silica rice husk ash at temperature ranging from 675-700℃ in controlled conditions, with specific gravity 2.04 and average particle size 11.5 μm was procured from a commercial supplier N K Enterprises, Orissa. The SEM (scanning electron microscopy) image of the RHA is as shown in Fig. 1. The X-ray diffraction analysis showed presence of quartz and cristobalite to be major constituent of RHA and chemical compositions of RHA determined by

Table 2 Aggregate properties

Property	Fine Aggregates	Coarse Aggregates
Specific Gravity	2.32	2.60
Fineness Modulus	2.92	7.10
Water Absorption	1.50%	0.80%



(a) Coarse aggregate



(b) Fine aggregate

Fig. 3 Grading curve of (a) Coarse aggregate (b) Fine aggregate

X-ray Fluorescence (XRF) analysis are given in Fig. 2(a) and Table 1 respectively. Based on the XRF results RHA can be catagorised into pozolonic materials.

3.1.2 Alccofine

Alccofine 1203 (AF) is a microfine material. Alccofine controls high reactivity because of controlled granulation. It improves workability by reducing the water demand. Alccofine 1203 produces high performance concrete either as a cement replacement or as an additive. Chemical compositions of Alccofine 1203 used are depicted in Table 1. X-ray diffraction analysis showed presence of calcite to be major constituent of alccofine and is given in Fig. 2.

3.1.3 Aggregates

A good quality and well-graded aggregates in saturated surface dry (SSD) condition is used to prepare the GPC specimens. Natural available fine sand and coarse aggregates with maximum size 14, 10 and 7 mm in the proportion of 45%, 35%, and 20% respectively is used. Junaid *et al.* recommended differed size aggregates and these proportions to get the workable geopolymer mix. The properties of the aggregates are given in Table 2 and grading curve is as shown in Fig. 3. Both coarse and fine aggregates confirm to of IS 383-1970 (BIS 383-1970) while fine aggregate used are natural sand and graded as conforming to IS 2386 (Part I)-1963 (BIS 2386-1963).

3.1.4 Alkaline activators

Mixing of the alkaline activators plays an important role, and it affects the GPC properties

Table 3 Sodium silicate specifications

Item	Specification
Color	Color-less
Density, kg/m^3	1450-1550
Total solids content, by mass%	45:52

Table 4 Mix proportions used in this study

Mix Designation	F.A	C.A	RHA	M : NaOH	TAS	Extra water	Alccofine	SP	CT (^{o}C) / Rest Period (hr.)
M1RHAGC	528	1232	350	8	157.5	26	35.0	7.0	
M2RHAGC	528	1232	375	8	168.7	28	37.5	7.5	
M3RHAGC	528	1232	400	8	180.0	28	40.0	8.0	
M4RHAGC	528	1232	350	12	157.5	30	35.0	7.0	
M5RHAGC	528	1232	375	12	168.7	30	37.5	7.5	90 / 24
M6RHAGC	528	1232	400	12	180.0	32	40.0	8.0	
M7RHAGC	528	1232	350	16	157.5	32	35.0	7.0	
M8RHAGC	528	1232	375	16	168.7	34	37.5	7.5	
M9RHAGC	528	1232	400	16	180.0	36	40.0	8.0	
M10RHAGC	528	1232	350	8	157.5	26	35.0	7.0	
M11RHAGC	528	1232	375	8	168.7	28	37.5	7.5	
M12RHAGC	528	1232	400	8	180.0	28	40.0	8.0	
M13RHAGC	528	1232	350	12	157.5	30	35.0	7.0	
M14RHAGC	528	1232	375	12	168.7	30	37.5	7.5	27 / 24
M15RHAGC	528	1232	400	12	180.0	32	40.0	8.0	
M16RHAGC	528	1232	350	16	157.5	32	35.0	7.0	
M17RHAGC	528	1232	375	16	168.7	34	37.5	7.5	
M18RHAGC	528	1232	400	16	180.0	36	40.0	8.0	

*FA-fine aggregates, CA-coarse aggregates, M-molarity, TAS-total alkaline solution, SP- Superplasticizer, all quantities are taken in kg/m^3

(Kong *et al.* 2010). NaOH and Na_2SiO_3 are used in this study as an alkaline activator. When water is added to NaOH pellets, heat is generated. Heat plays a major role in the GPC manufacturing and in the geopolymerisation process. NaOH in the form of pellets with 98% purity and sodium silicate solution (Na_2SiO_3) with SiO_2/Na_2O between 1.90 and 2.01 were procured commercially. The specification of the sodium silicate is as shown in Table 3.

3.1.5 Superplasticizer

Sodium silicate (SS) and sodium hydroxide (SH) solutions are more viscous than water. Hence their use makes the GPC more cohesive and sticky than OPC concrete (Deb *et al.* 2014). So, to improve the workability of the fresh geopolymer mix, a Naphthalene Sulphonate based water

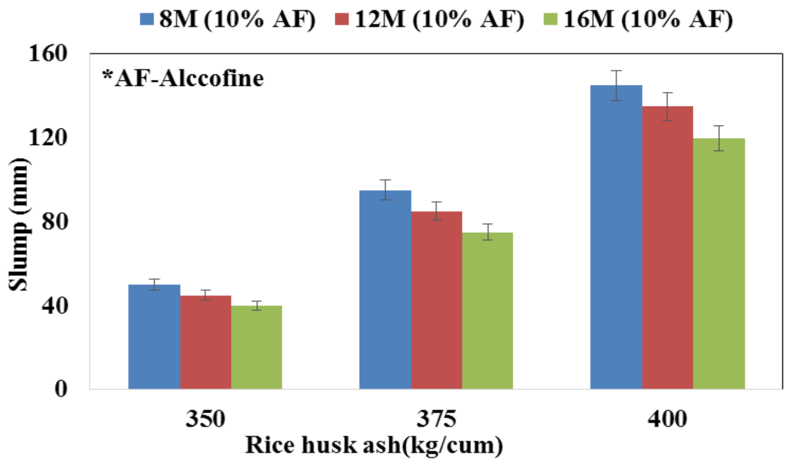


Fig. 4 Slump of GPC mixes with varying RHA content

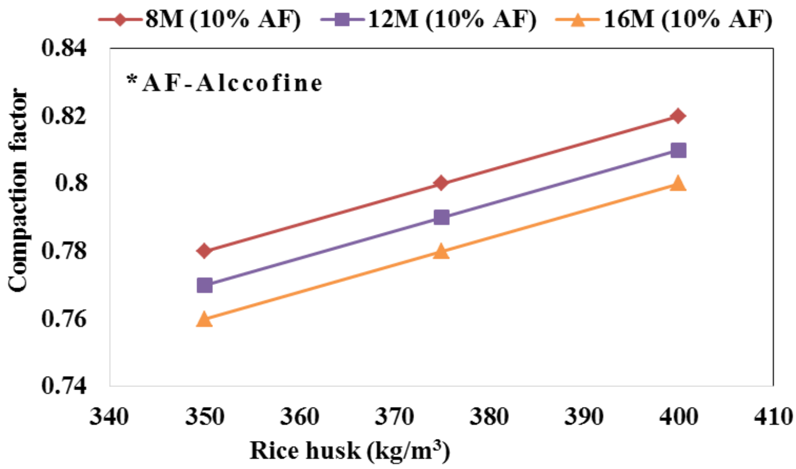


Fig. 5 Compaction factor of GPC mixes with different RHA content

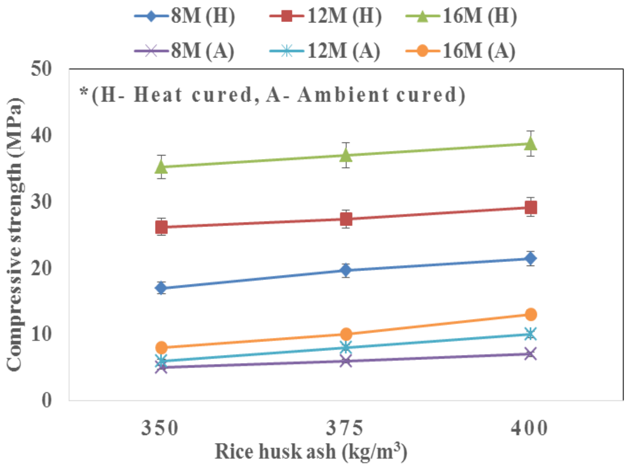
reducing superplasticizer confirming to IS 9103:1999 (1999) is used.

3.2 GPC mixtures

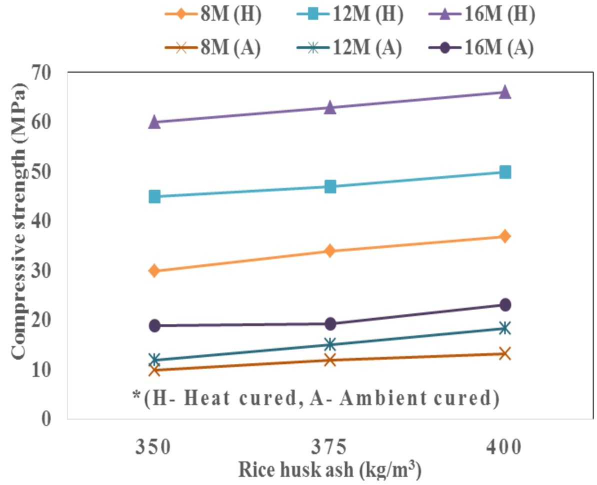
Eighteen GPCs mixture proportions are studied. The mixes were prepared based on the previous studies on the GPC (Lloyd *et al.* 2010, Deb *et al.* 2013, Junaid *et al.* 2015, Pavithra *et al.* 2016). All the eighteen mixes tabulated in Table 4 were prepared with 10% Alccofine, while superplasticizer amount was kept 2% of the RHA content. The different GPC mixtures considered are given in Table 4.

3.3 Preparation, casting, and curing of GPC specimens

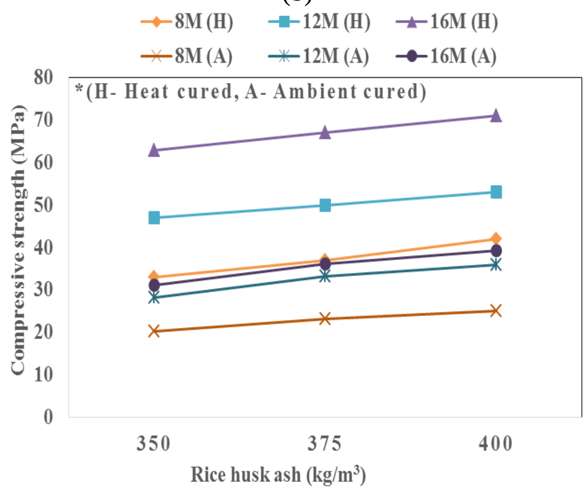
Before the mixing of the concrete ingredients, aggregates were prepared to the saturated surface dry condition. Sodium hydroxide was prepared 24 hours prior to final mixing. In this study, NaOH



(a) 3 days



(b)



(c) 28 days

Fig. 6 Compressive strength of RHA based GPC and Na_2SiO_3 were mixed about 1 hour before the mixing of dry components used in GPC. All the dry ingredients such as RHA, aggregates, and alccofine were dry mixed with the pan mixture. Then alkaline activator solution is added at a slow rate to the dry materials. The mixing is done for about 5 minutes to produced alccofine activated fresh GPC. However, superplasticizer and extra water were added during the mixing at a slow rate. After mixing all the ingredients in pan mixture, the mix is poured into cube moulds and put on the vibrating table for proper compaction. The compaction is done for 1 to 2 minutes. 150 mm cubes were prepared for compressive and split tensile strength tests respectively. A rest period of one day is given to all the specimens. The samples were then cured at the ambient and heat condition at 27℃ and 90℃. Indian Standard methods were used for sampling and testing of fresh and hardened GPC (BIS 1199-1959, BIS 516-1959, BIS 5816-1999).

4. Testing, results, and discussion

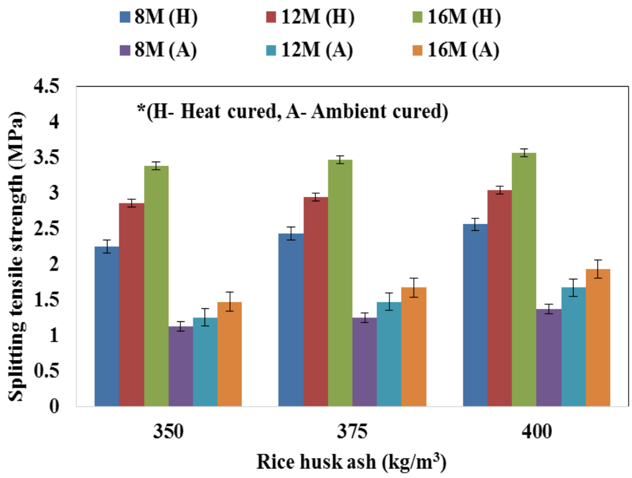
4.1 Workability

Workability is related to rheological properties of concrete in the fresh state including its mobility, stability, and compatibility. Slump and compaction factor test were carried out to study the properties of the fresh RHA based GPC. The size of the slump cone was 100×200×300 mm and testing was done as per IS 1199: 1959 (BIS 1199-1959) to study the effect of main raw materials. Compaction factor is the ratio of the weight of partially compacted concrete to the weight of the concrete when fully compacted in the same mould. The slump values and compacting factor values of GPC obtained at different RHA content and concentration are represented in the Figs. 4 and 5 respectively.

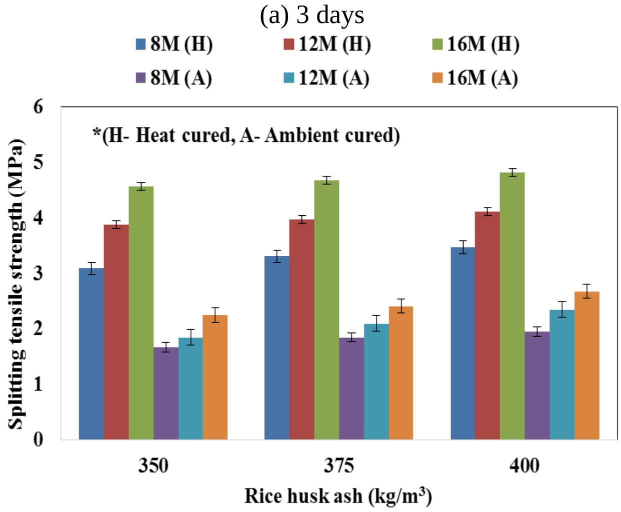
Alkaline activator solution has a lubricating effect and when combined with rice husk ash and AF gave flowability to the fresh GPC. The slump and compaction factor values obtained for all the GPC mixes prepared with 10% alccofine and 2% superplasticizer indicates less stickiness and good workability. Again, with the increase in rice husk ash content same behavior was obtained for the fresh GPC. The improvement in the workability of GPC with alccofine may be due to a decrease in the water requirement with the addition of superplasticizer (Jindal *et al*. 2017, Jindal *et al.* 2017). However, a higher concentration of the sodium hydroxide solution resulted in a lower slump and compaction factor values, and this was same for all the cases. The reason for lowered slump values could be the increased stickiness due to increased molarity of sodium hydroxide and higher water requirement of RHA which attribute to hardening process resulting in high compressive strength. The test results obtained in this study were in accordance with the previous studies on fly ash and RHA based GPC (Hardjito *et al.* 2005, Chindaprasirt *et al.* 2007, Yip *et al.* 2008, Nath *et al*. 2015).

4.2 Compressive strength

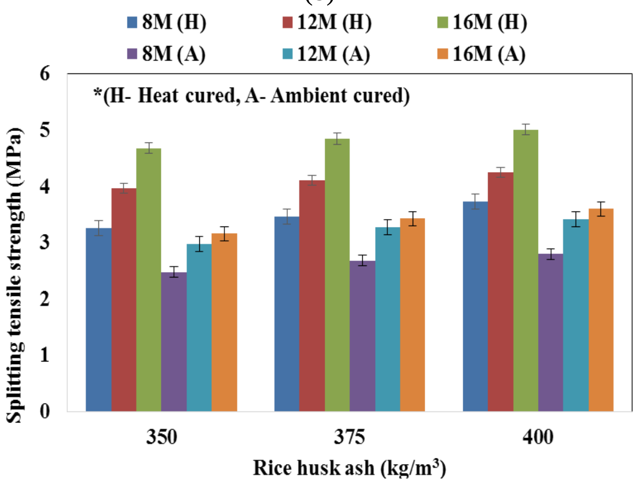
It is the most common and an indicator of the mechanical property of the concrete which is correlated with the other properties too. The bearing surface of the GPC samples of size 150×150×150 mm is wiped clean, and samples are placed in between the steel plates and packing is not used. The load on the samples is applied at a rate of 13.72 $N/mm^2/min$. Four identical samples were prepared for each case and tested (BIS 516-1959) after 3, 7 and 28 days of casting. The mean values of compressive strength obtained for the mixes depicted in Table 4 are



(a) 3 days

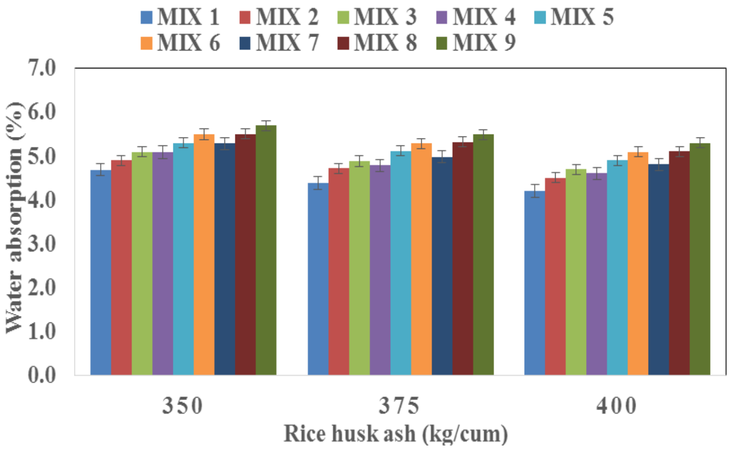


(b)

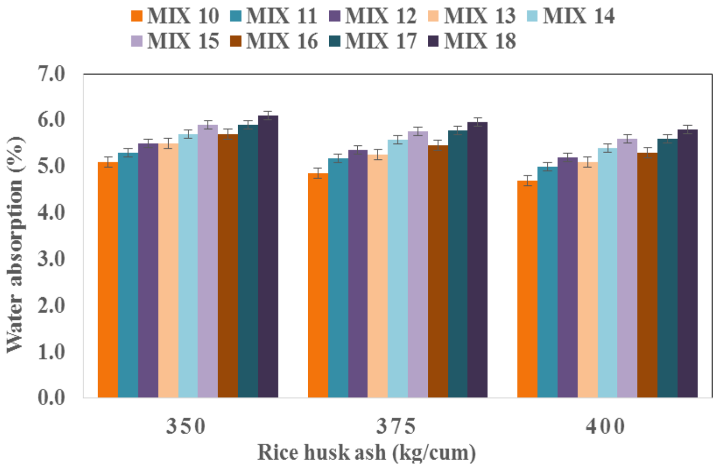


(c) 28 days

Fig. 7 Splitting tensile strength of RHA based GPC



(a) Heat cured



(b) Ambient cured (27℃)

Fig. 8 Water absorption of RHA based GPC after 28 days

represented in Fig. 6.

It is clear from the Fig. 6 that the compressive strength increases with the increase in RHA content and NaOH molarity. The rate of increasing is similar for all the mixes. The compressive strength of the GPC with different RHA content and the different molarity of NaOH, increased from 5 MPa to 39.4 MPa and from 17 MPa to 71 MPa at the ages of 3 days and 28 days for specimens cured at 27℃ and 90℃ respectively. The gain in compressive strength was higher up to 7 days when heat curing was adopted. The effect was reversed in the ambient cured GPC mixes. It can be noted from Fig. 6 to Fig. 8 that heat curing accelerates the rate of geopolymerization resulting in the improvement in the early age compressive strength. Further, the behavior of the RHA based GPC is similar to fly ash based GPC. The normal trend of increasing the strength for RHA based GPC observed was just like fly ash based GPC. However, the effect of alccofine at 400 kgs of RHA, temperature, and 16M sodium hydroxide solution is more pronounced (M9RHAGC) as the total specific surface area increased, a maximum of 71 MPa has been achieved. The increase in strength was due to the calcium silicate hydrate formed by the inclusion of alccofine in addition to the other polymeric products which formed due to RHA. Increased RHA content results into increased quantity of binder material as well as the development of denser concrete improving the compressive strength parameter. This increase in compressive strength at ambient and heat curing had been due to the properties of alccofine and RHA which when mixed with alkali solutions gives better results. Calcium present in the alccofine reacts with activators and heat was produced. This could have been the reason of enhancing the compressive strength products.

4.3 Splitting tensile strength

It comes in the mechanical property of the concrete. The splitting tensile strength of concrete is one of the prominent property used in various design aspects of structural members. The geopolymer concrete cubes of 150 mm size are used to investigate the split tensile strength a minimum bearing area of 12×150 mm is provided so that load can be applied over the entire length. The load is applied at a nominal rate within the range 1.2 N/(mm^2/minute) to 2.4N/(mm^2/minute) and without any shock. Splitting tensile strength test confirming to IS 5816-1999 (BIS 5816-1999) is conducted at the age of 3, 7 and 28 days with varying RHA as well as NaOH molarity. Influence of variation of RHA content using different molarity at ambient cured GPC specimens on splitting tensile strength at the age of 3, 7 and 28 days is shown in Fig. 7.

Fig. 7 shows a nominal increase in 28 days splitting tensile strength of 4% and 7% on increasing RHA content from 350 kg/m^3 to 375 kg/m^3 and 350 kg/m^3 to 400 kg/m^3 respectively when 12M NaOH solution is used along with alccofine. A significant increase in split tensile strength was observed when GPC cured at ambient and heat were compared. Therefore, it is concluded from the results that, split tensile strength increases significantly with increase in RHA content and molarity of the solution. The splitting tensile strength shows similar behavior as the compressive strength of the GPC. More significantly, splitting tensile strength increases with the increase in compressive strength of GPC and with the increase in curing temperature. The increase in strength of the RHA based geopolymer concrete was due to additional calcium in the system which reacted and formed additional CSH along with polymeric products. The combined network of polymerization and hydration was expected to have dense microstructure and therefore high strength values were observed due to alccofine.

4.4 Water absorption

The test was also conducted to study the effect of heat curing, NaOH concentration and RHA content on the water absorption property of the GPC. Further testing was done on all the mixes tabulated in Table 4. The results of the water absorption percentage for GPC mixes is depicted in the Fig. 8.

The effect of the curing, RHA content, and concentration of the sodium hydroxide solution on 28 days water absorption of the GPC prepared with alccofine and RHA is presented in Fig. 8. It can be noted from the Fig. 8 that the water absorption reduced by average 5% when heat curing is adopted instead of ambient curing. The increase in RHA content and molarity also affects the water absorption insignificantly by 4-5% and 5-6% respectively. The reduction of water absorption in GPC in heat curing may be due to the fineness of alccofine and RHA content together. However, with the increasing molarity of the NaOH, the water absorption reduces and it may be due to the high strength of the GPC at high molarity.

Table 5 Relationship between compressive and split tensile strength

	ACI 318-99	Gardner *et al.*	Rapheal *et al.*	Carino *et al.*	Current study	Ryu *et al.*	Lee *et al.*	Soffi *et al.*	Anuradha *et al.*
	Ordinary concrete				Geopolymer concrete				
	Split tensile strength, $f_{spt} = a * (\text{Compressive strength}, f_c)^{ß}$								
a	0.56	0.46	0.313	0.272	0.46	0.17	0.45	0.48	0.892
ß	0.50	0.60	0.66	0.71	0.56	0.75	0.50	0.50	0.422

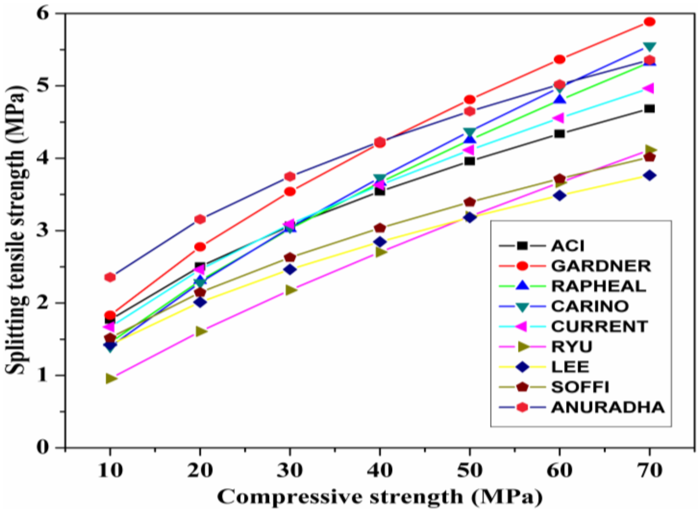


Fig. 9 Relationship between compressive and splitting tensile strength at 28 days

4.5 Relation between compressive and splitting tensile strength

The relation between compressive and splitting tensile strength for alccofine activated RHA based GPC specimens are shown in Fig. 9. Previous researchers depicted a close relation between splitting tensile and compressive strengths of concrete and the same has been shown in the Table 5. Nonlinear equations proposed were based on regression analysis between tensile and compressive strength of concrete. The relationship between the compressive and tensile strength for alccofine activated RHA based geopolymer concrete is proposed by Eq. (1). It can be seen from the Fig. 9 that the regression line for RHA based geopolymer concrete can be regarded as a realistic representation, which can be applied to heat and ambient cured specimens. It has a direct relationship with the compressive strength similar to ordinary concrete.

$$f_{spt} = 0.46 \times f_c^{0.56} \tag{1}$$

Where, f_{spt} = *Split tensile strength and* f_c = *Compressive strength*

5. Conclusions

1. Geopolymer concrete prepared with RHA has great potential to be used as a construction

material.

2. RHA can be used in the production of geopolymers which can produce similar compressive strength to those of fly ash (FA) based geopolymers.

3. Key properties of RHA based GPC shows similar behavior to FA based GPC.

4. Method and design provisions of the FA based GPC can be applied to produce RHA based GPC.

5. Maximum compressive and splitting tensile strength was obtained at heat curing (90℃) and using 16M NaOH.

6. The relationships between compressive and splitting tensile strength of the GPC possess similar trendline to ordinary concrete.

7. Alccofine is responsible for heat of hydration in concrete and it can be used to improve the strength at ambient temperature.

Acknowledgments

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