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Mechanical and Thermal Properties of Geopolymers from Mixtures of Coal Ash and Rice Hull Ash using Water Glass Solution as Activator

***Martin Ernesto L. Kalaw** *,1*
***Alvin B. Culaba** 1*
***Hoc Thang Nguyen** 2*
***Khoi Nguyen** 2*
***Hirofumi Hinode** 3*
***Winarto Kurniawan** 3*
***Susan M. Gallardo** 4*
***Michael Angelo B. Promentilla** 4*
[1] Mechanical Engineering Department, De La Salle University, Philippines
[2] Faculty of Materials Engineering, Ho Chi Minh City University of Technology, Vietnam
[3] Department of International Development Engineering, Tokyo Institute of Technology, Japan
[4] Chemical Engineering Department, De La Salle University, Philippines
**e-mail:* martin.kalaw@dlsu.edu.ph

Geopolymers, from industrial wastes such as blast furnace slag, red mud, and coal ash, among others, have emerged as technically viable, economically competitive, and environmentally attractive supplements and even alternatives to ordinary Portland cement (OPC). Furthermore, while the most impact shall be achieved with large-scale use in the general building and structural sector, as replacement or supplement to OPC, the properties of these geopolymers may be optimized for special niche applications. One of these applications is for light weight, low thermal conductivity, heat resistant, and moderate strength cement binder for low rise residential buildings. In this study, compressive strength, heat resistance, volumetric weight, mass loss, water absorption and thermal conductivity of geopolymers formed from mixtures of coal bottom ash and rice hull ash (CBA-RHA) and coal fly ash and rice hull ash (CFA-RHA) with sodium silicate solution (modulus 2.5) as activator were evaluated. Using mixture design and the JMP statistical software, the CBA-RHA combination at a mass ratio of 46% CBA, 32% RHA with 22% WGS gave properties at maximum desirability of 17.6 MPa compressive strength, 1640 kg/m^3 volumetric weight, 273 kg/m^3 water absorption, 28 MPa compressive strength after high temperature exposure (1000°C for 2 hours) with 4.4% mass loss, and 0.578 W/m-K thermal conductivity. On a performance basis, even as the geopolymers are formed as paste, these properties fall within the standards for lightweight OPC based-concrete with strength requirements for residential buildings. The low thermal conductivity and higher strength after high temperature exposure vis-à-vis OPC are additional advantages for consideration.

Keywords: bottom ash, fly ash, geopolymers, rice hull ash, waste utilization, water glass activation

INTRODUCTION

Geopolymerization (alkali activation, in general) has become a significant opportunity for the alleviation of the environmental impact, health risks, issues on lack of landfill areas, etc. due to the increasing generation of industrial solid wastes and by-products particularly alumina- and silica- rich materials such as blast furnace slag, red mud, and coal ash (Khale and Chaudhary 2007, Juenger et al. 2011, Suhendro 2014, Mellado et al. 2014, Part et al. 2015). This process of geopolymerization mainly involves the dissolution of the amorphous alumina and silica content of these materials in highly alkali solutions and the subsequent polycondensation resulting in the formation of a three-dimensional amorphous alumina-silicate network. Davidovits coined the term geopolymer in 1978 for these products and presented its many potential and tested applications, among which are as binders (alternative to OPC), as a matrix for radioactive and toxic waste immobilization and containment, as fire and heat resistant materials, as thermal insulation materials, etc. (Davidovits 1994, 2002, 2011).

It has been shown in many geopolymer formulations that geopolymers, as alternative binders, can have comparable if not better properties than OPC but with significantly lower CO_2 emissions and potentially more economical for both users and raw materials sources/ providers (Khale and Chaudhary 2007, Juenger et al. 2011, Suhendro 2014, Mellado et al. 2014, Part et al. 2015, Davidovits 1994, 2011). However, other sources of alumina and silica, as newer precursor materials or additives may produce a different set of geopolymer properties that may be adapted for specific applications (Duxson et al. 2007, Davidovits 2011, Provis and van Deventer 2014).

In this study, the precursor materials were coal ashes, coal bottom ash (CBA) and coal fly ash (CFA) as alumina and silica sources. Rice hull ash (RHA) was added for its rich amorphous silica content and lower thermal conductivity.

These materials were used in consideration of the large generation rates of coal ash and rice hulls of the Philippines and Vietnam in particular, and the Asian region in general. Table 1 below shows the 2014 rice production and 2012 coal consumption for some Asian countries. From these data, the amount of rice hulls generated (about 20% by volume), and coal ash (between 10 to 30% by mass depending on type and source) can be estimated. Annual generation rates have an increasing trend as can be seen from the yearly data from sources cited in the Table 1.

Table 1. Rice production and coal consumption

Country	2014 Paddy rice production[1], MMT	2012 Coal consumption[2], MMT
Philippines	19.4	16.5
Vietnam	44.9	24.4
Thailand	28.4	35.3
Japan	10.8	183.6
China	206.4	3773.3

[1] World Rice Statistics, International Rice Research Institute,

[2] World Crude Oil Consumption, Index Mundi

Table 2. Particle sizes* and bulk density of raw materials

Raw Material	Mean, µm	Median, µm	S.P. Area, cm^2/cm^3	Density, kg/m^3
RHA	20.39	11.34	7647.2	1160
CBA	32.11	19.66	6541.4	2560
CFA	52.91	35.71	4412.3	1970

* obtained using Horiba LA-920 laser diffractometer

Table 3. Composition* of raw materials

Components	RHA	CBA	CBA$^+$	CFA	CFA$^+$	WGS
Al_2O_3	0.37	20.85	22.81	16.72	23.45	-
SiO_2	83.20	52.63	57.59	32.97	46.25	32
Fe_2O_3	1.70	9.08	9.94	13.32	18.69	-
K_2O	6.60	4.75	5.20	3.60	5.05	-
CaO	1.50	0.82	0.90	0.97	1.36	-
TiO_2	-	1.39	1.52	1.19	1.67	-
Na_2O	-	0.22	0.24	-	-	12.5
H_2O	-	-	-	-	-	55.5
Others	2.03	1.65	1.80	2.51	3.53	-
LOI	4.60	8.61		28.72		-

* obtained using XRF, except for WGS

$^+$ composition after LOI was deducted

In this study, the effect of rice hulls ash (RHA) as a precursor material and the amount of waterglass solution (WGS) were evaluated in the development of light weight, low thermal conductivity, heat resistant, and moderate strength geopolymer binder.

MATERIALS AND METHODS

Dry Raw Materials

The rice hulls ash (RHA) was produced by burning rice hulls obtained from Dong Thap province in the Mekong Delta, Vietnam. The coal fly ash (CFA) and coal bottom ash (CBA) were obtained from the thermo-electric plant of Tan Rai Bauxite Plant (Lam Dong, Viet Nam).

Prior to use in the making of geopolymer specimens, all the raw materials, CFA, CBA and RHA were ground, dried and sieved. The particle sizes, surface area and bulk density of the raw materials are shown in Table 2. Practical and economic considerations indicate as little pre-processing of materials as possible however studies have shown that smaller particle sizes and larger surface areas of the raw materials result in higher reactivity and better strength properties of the geopolymers formed (Khale and Chaudhary 2007, Alvarez-Ayuso et al. 2008, Davidovits 2011).

The major mineral components of the raw materials are tabulated in Table 3. It is seen that CFA and CBA are mainly composed of alumina (Al_2O_3), silica (SiO_2) and iron oxide (Fe_2O_3) with CBA having more alumina, per unit mass of mixture, at

Table 4. Range of mass proportions of components in ternary mixtures considered

Mix	CFA	CBA	RHA	WGS
TM1	0,0.43, 0.86 0,0.41, 0.82 0,0.39, 0.78	-	0,0.43, 0.86 0,0.41, 0.82 0,0.39, 0.78	0.14 0.18 0.22
TM2	-	0,0.43, 0.86 0,0.41, 0.82 0,0.39, 0.78	0,0.43, 0.86 0,0.41, 0.82 0,0.39, 0.78	0.14 0.18 0.22

20.85%. The RHA used in this study has more than 83% silica and only very small amounts of alumina and iron oxide. One notable characteristic of the CFA in this study is its high loss on ignition (LOI) 28.72% which indicates the presence of high unburned carbon content (Davidovits 2011). The composition of CBA and CFA after LOI was deducted are also shown in Table 3, here it is seen that, on the basis of burned components, CBA has more silica and less iron oxide than CFA.

Alkali Activator

Alkali activators used for geopolymerization processes can be alkali silicate, alkali hydroxide, alkali sulfates or alkali carbonates. But alkali silicates, specifically sodium silicate (waterglass), are the most commonly used for geopolymerization due to lower efflorescence, lower enthalpy of dissolution, higher reaction with solid alumina-silicates, and lower CO2 emissions compared to other alkali activators (Davidovits 2011, Petermann et al. 2012, Provis and van Deventer 2014) .

In this study, the alkali activator used is sodium silicate (waterglass solution, WGS) with a modulus of 2.5 obtained from the Bien Hoa Chemical Factory also in Vietnam. The composition of the WGS is also shown in Table 3. The amount of WGS used in the geopolymer samples are 14%, 18% and 22% of the mixture mass.

Mixture Design

With WGS considered as a mixture component, two ternary mixtures, TM1 AND TM2, as shown in Table 4, were formed with each component at three levels.

Geopolymer specimens were formed using the above proportions for each ternary mixture of TM1 and TM2. The components were mixed for 15 minutes using a portable, electric laboratory mixer with water slowly added to achieve satisfactory workability and then poured into 50mm x 50mm x 50mm molds. The specimens were de-molded after 24 hours at room conditions and thereafter cured for 28 days also at room conditions.

After curing for 28 days, the volumetric weight and thermal conductivity (via the hot-wire method using the QTM-500 thermal conductivity meter) were measured. On separate samples, compressive strength (via Universal Testing Machine), water absorption, and heat resistance tests were conducted. The heat resistance was obtained by exposing the geopolymer samples to 1000^{o}C for 2 hours in a muffle furnace afterwhich the mass

Table 5. Response variables and desirability conditions

Response Variable	Units	Condition	Importance
Compressive strength	MPa	> 11.7	5
Volumetric weight	kg/m^3	< 1680	3
Water absorption	kg/m^3	< 320	3
Strength at 1000°C	MPa	maximum	2
Mass loss	%	minimum	2
Thermal conductivity	W/mK	< 0.59	4

loss was recorded and the compressive strength compared with the as-cured compressive strength.

Analysis using response surface methodology (RSM) and desirability conditions via the JMP software

Using the JMP software, and mixture design-RSM, the input variables are the mass proportions of the three (3) mixture components:

TM1	CFA, RHA and WGS
TM2	CBA, RHA and WGS

The response variables and the desirability conditions set for each to meet the target application selected are summarized in Table 5.

The above compressive strength, volumetric weight and water absorption requirements are based on ASTM C90-14 for light weight, load bearing concrete masonry units. The condition for thermal conductivity is based on equivalent thermal conductivity (American Concrete Institute, 2002) using d = 1680 kg/m^3.

$$k_c = 0.072\, e^{0.00125d}$$

where d = density of concrete, kg/m^3

k_c = thermal conductivity, W/m-K

Please note that, even as the samples developed are for geopolymer paste and not geopolymer concrete, the properties of OPC-based concrete are used as reference for performance properties only.

RESULTS AND DISCUSSION

As summarized in Table 6, it is seen that only in ternary mixture, TM2, was an optimum mixture obtained that met the requirements set for the specific application considered.

The corresponding ternary plots for each mixture are shown in Figures 1 and 2 below. The narrow band in the ternary plot indicates the range of mass proportion of the WGS (0.14 to 0.22) while the colored curved lines each represent a response variable. The point of intersection of the three solid lines represents the optimum condition at highest desirability. The values are tabulated in Table 6.

Table 6. Geopolymer properties at maximum desirability

Geopolymer Property	TM1	TM2
CFA (mass fraction)	0.41	-
CBA (mass fraction)	-	0.46
RHA (mass fraction)	0.41	0.32
WGS (mass fraction)	0.18	0.22
Compressive strength	4.23MPa	17.6 MPa
Volumetric weight	1199 kg/m^3	1641 kg/m^3
Water absorption	430 kg/m^3	273 kg/m^3
Strength at 1000°C	7.58MPa	28.1 MPa
Mass loss	15.6%	4.45%
Thermal conductivity	0.60 W/mK	0.58 W/mK
Desirability	0	0.416

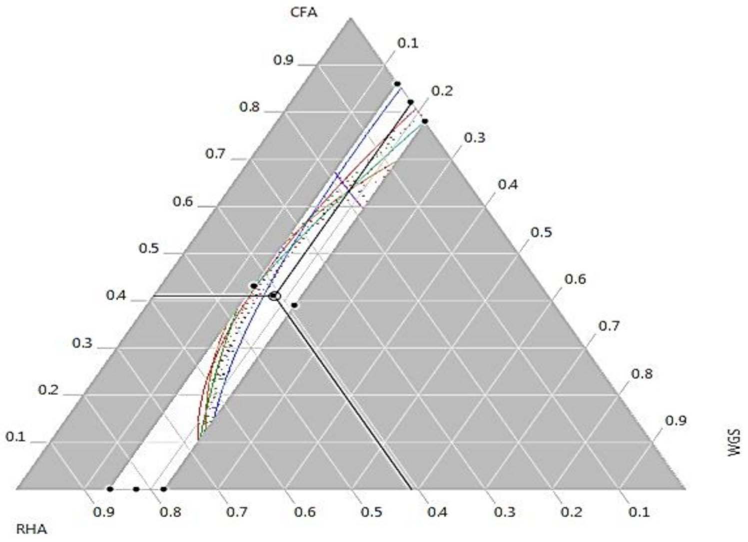


Fig. 1: Ternary plot for TM1

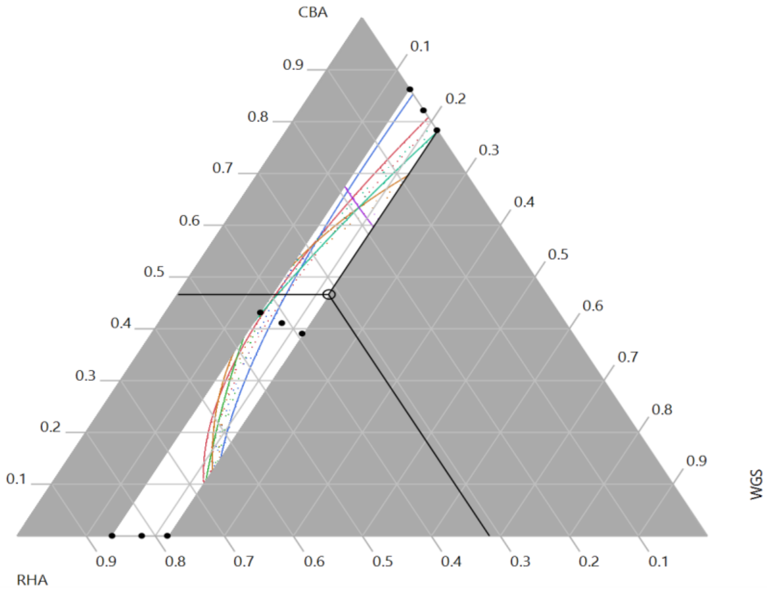


Fig. 2: Ternary plot for TM2

A summary of the test results for the two ternary mixtures and the corresponding Si/Al atomic ratios for each mix ratio are shown in Table 7 and Table 8. Table 9 shows the properties of binary mixtures and Table 10 shows the properties of the ternary mixtures. Figures 3 and 4 show the heat resistance for the binary and ternary mixtures, respectively.

From these results, the following are observed:

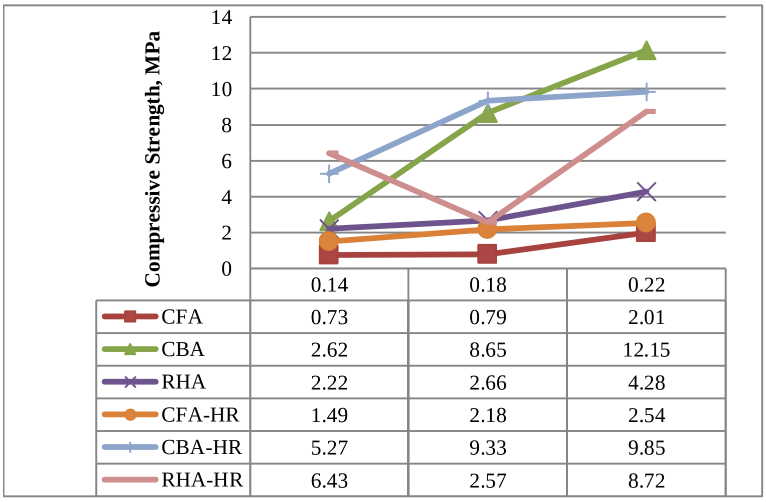
1. Effectively, binary mixtures of RHA and WGS for both TM1 AND TM2, from Tables 7 and 8, led to Si/Al atomic ratios > 200. For these mixtures, as seen in Figure 3, strengths are low but were still increasing as the amount of activator increased from 0.14 to 0.22.
2. Binary mixtures of CFA and WGS in the TM1 set, from Table 7, presented separately in Table 9 (a) and Figure 3, have increasing strength as amount of

Table 7. Summary of test results for TM1

CFA	RHA	WGS	Com Str	WatAbs	Str@ 1000	Vol Wt	Massloss	Th con	Na/Al	Si/Al
0.86	0.00	0.14	0.73	493.4	1.49	1097.8	32.0	0.33	0.20	1.94
0.00	0.86	0.14	2.22	418.8	6.43	1275.7	7.9	0.54	9.05	203
0.43	0.43	0.14	2.64	462.4	5.52	1111.6	17.4	0.58	0.39	6.29
0.82	0.00	0.18	0.79	471.5	2.18	1104.7	15.2	0.34	0.27	2.03
0.00	0.82	0.18	2.66	410.6	2.57	1320.7	5.5	0.55	12.2	207
0.41	0.41	0.18	5.01	430.5	8.61	1195.3	16.8	0.60	0.53	6.48
0.78	0.00	0.22	2.01	460.6	2.54	1096.5	15.2	0.40	0.35	2.13
0.00	0.78	0.22	4.28	386.4	8.72	1387.5	5.0	0.56	15.7	212
0.39	0.39	0.22	5.12	395.5	8.79	1299.7	13.1	0.64	0.68	6.68

Table 8. Summary of test results for TM2

CBA	RHA	WGS	Com Str	WatAbs	Str@ 1000	Vol Wt	Massloss	Th con	Na/Al	Si/Al
0.86	0.00	0.14	2.62	344.6	5.27	1571.5	13.5	0.96	0.18	2.36
0.00	0.86	0.14	2.22	418.8	6.43	1275.7	7.9	0.54	9.05	203
0.43	0.43	0.14	6.23	367.6	13.39	1472.3	8.4	0.50	0.33	5.86
0.82	0.00	0.18	8.65	288.2	9.33	1626.5	9.8	0.97	0.23	2.43
0.00	0.82	0.18	2.66	410.6	2.57	1320.7	5.5	0.55	12.2	207
0.41	0.41	0.18	15.22	334.8	26.24	1546.5	6.2	0.59	0.44	6.00
0.78	0.00	0.22	12.15	253.8	9.85	1667.3	8.7	0.97	0.30	2.51
0.00	0.78	0.22	4.28	386.4	8.72	1387.5	5.0	0.56	15.7	212
0.39	0.39	0.22	19.46	259.9	35.6	1655.3	3.4	0.47	0.56	6.16



	0.14	0.18	0.22
CFA	0.73	0.79	2.01
CBA	2.62	8.65	12.15
RHA	2.22	2.66	4.28
CFA-HR	1.49	2.18	2.54
CBA-HR	5.27	9.33	9.85
RHA-HR	6.43	2.57	8.72

Legend:
x-axis: WGS 0.14, 0.18, 0.22
y-axis: Compressive strength, MPa
HR : heat resistance (exposure to 1000°C)

Fig. 3: Heat resistance vs WGS for binary mixtures of specified components with WGS (i.e. CFA-WGS, etc.)

activator increased but the amount of increase is very small. This is also true for ternary mixtures in this set as seen in Table 10 (a) and Figure 4. This low rate of increase in strength can be attributed to the high unburned carbon content of CFA inhibiting geopolymerization (Davidovits 2011).

Table 9. Results of tests for (a) binary mixtures of CFA and WGS, (b) binary mixtures of CBA and WGS and (c) binary mixtures of RHA and WGS

(a)

CFA	WGS	Com Str	Vol Wt	Wat Abs	Str@ 1000	Mass loss	Th con
0.86	0.14	0.73	1097.8	493.4	1.49	32.0	0.33
0.82	0.18	0.79	1104.7	471.5	2.18	15.2	0.34
0.78	0.22	2.01	1096.5	460.6	2.54	15.2	0.40

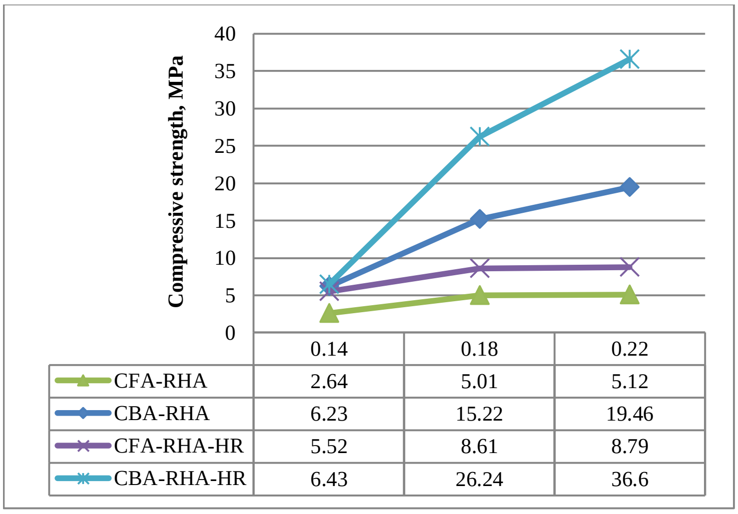
(b)

CBA	WGS	Com Str	Vol Wt	Wat Abs	Str@ 1000	Mass loss	Th con
0.86	0.14	2.62	1571.5	344.6	5.27	13.5	0.96
0.82	0.18	8.65	1626.5	288.2	9.33	9.8	0.97
0.78	0.22	12.2	1667.3	253.8	9.85	8.7	0.97

(c)

RHA	WGS	Com Str	Vol Wt	Wat Abs	Str@ 1000	Mass loss	Th con
0.86	0.14	2.22	1275.7	418.8	6.43	7.9	0.54
0.82	0.18	2.66	1320.7	410.6	2.57	5.5	0.55
0.78	0.22	4.28	1387.5	386.4	8.72	5.0	0.56

3. Binary mixtures of CBA and WGS in the TM2 set from Table 8, presented separately in Table 9 (b) and Figure 3, have significant increase in strength as amount of activator increased. The rate of increase was even higher for the ternary mixtures in this set as seen in Table 10 (b) and Figure 4. Thus increasing the amount of activator beyond 0.22 may result in further increase of strength. However this will increase the cost input to the process while decreasing the rate of waste utilization.
4. As the highest strength was obtained from the ternary mixture of CBA, RHA and WGS corresponding to the highest amounts of RHA and WGS, it may be attributed to the high reactivity (i.e. amorphousness) of the RHA used and to the effect of higher amounts of activator used (Duxson et al. 2007, Davidovits 2011, Provis and van Deventer 2014).
5. In Tables 9 and 10, the following were observed: (a) lower thermal conductivity corresponded to lower volumetric weight, (b) lower thermal conductivity corresponded to higher mass loss (and thus higher initial moisture content), and (c) lower thermal conductivity corresponded to higher RHA content.
6. From Tables 7 and 8 and Figures 3 and 4, the strength of the geopolymer increased after exposure to 1000°C.



	0.14	0.18	0.22
CFA-RHA	2.64	5.01	5.12
CBA-RHA	6.23	15.22	19.46
CFA-RHA-HR	5.52	8.61	8.79
CBA-RHA-HR	6.43	26.24	36.6

Legend:
x-axis: WGS 0.14, 0.18, 0.22
y-axis: Compressive strength, MPa
HR : heat resistance (exposure to 1000°C)

Fig. 4: Compressive strength vs WGS for ternary mixes of specified components with WGS (i.e. CFA-RHA-WGS)

Table 10. Results of tests for ternary mixtures (a) CFA, RHA and WGS and (b) CBA, RHA and WGS

(a)

CFA	RHA	WGS	Com Str	Vol Wt	Wat Abs	Str@ 1000	Mass loss	Th con
0.43	0.43	0.14	2.6	1111	462	5.5	17.4	0.58
0.41	0.41	0.18	5.0	1195	430	8.6	16.8	0.60
0.39	0.39	0.22	5.1	1300	396	8.8	13.1	0.64

(b)

CBA	RHA	WGS	Com Str	Vol Wt	Wat Abs	Str@ 1000	Mass loss	Th con
0.43	0.43	0.14	6.2	1472	368	13.4	8.4	0.50
0.41	0.41	0.18	15.2	1547	335	26.2	6.2	0.59
0.39	0.39	0.22	19.5	1655	260	35.6	3.4	0.46

This is an advantage of geopolymer materials over OPC (Duxson et al. 2007, Davidovits 2011, Provis and van Deventer 2014).

7. For low Ca alkali activated materials (to which fly ash-based geopolymer belongs), the typical range of 1<Si/Al<5 for construction applications (Provis and van Deventer 2014). As Si/Al available from raw materials is 2<Si/Al<7, the final geopolymer may be expected to have a Si/Al ratio within the above range. For Si/Al >> 5, geopolymers formed will have low strength, low thermal stability, and generally low chemical resistance.

CONCLUSIONS

Based on the results of this study, the following conclusions are made:

1. Geopolymers from mixtures of CBA, RHA and with WGS as activator can be developed with properties suitable for lightweight, low thermal conductivity, heat resistant and moderate strength cement binder that can be used for low rise residential buildings.
2. The thermal characteristics of the geopolymers formed, low thermal conductivity for building heat load reduction and high heat resistance at elevated temperatures, enhance the consideration for its application as an alternative to OPC in low rise residential buildings.
3. Under suitable conditions of RHA reactivity, silica and alumina content and amorphousness, a certain ternary mixture of CBA, RHA and WGS may be formulated to produce a geopolymer with a set of properties that is fitted for a particular niche application such as the one considered in this study.

From the specific results of this study corresponding to the CBA-RHA-WGS mixture at a mass ratio of 46% CBA, 32% RHA and 22% WGS, the RHA content at 32% is significantly high. This may serve as impetus for the use of rice hulls for power and heat generation at combustion temperatures that will produce the highest amorphous silica content for potential use in this application thus maximizing all potential benefits as stated in the Introduction of this paper.

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