EVALUATING THERMAL PROPERTIES OF GEOPOLYMER PRODUCED FROM RED MUD, RICE HUSK ASH AND DIATOMACEOUS EARTH

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Abstract

In this paper, thermal properties of geopolymer from the mixture of red mud, rice husk ash, and diatomaceous earth were investigated. Red mud is an industrial waste from bauxite plants which has a negative impact to the environment if not properly managed, especially for countries mining this bauxite ore. On the other hand, rice husk ash is an agricultural waste abundant in Asian countries whereas diatomaceous earth is a natural mineral locally abundant in some parts of Vietnam. In this study, red mud was mixed with rice husk ash and diatomaceous earth at high alkaline condition to synthesize geopolymer, an inorganic polymer materials produced from geopolymerization reactions forming the alumino-silicate network. For thermal specifications of the geopolymer products, thermal conductivity, the thermal expansion and thermal gravimetric (TG) values from room temperature to 950^oC are measured. For example, the coefficient of thermal conductivity at room temperature was measured around 0.5 W/m-K, the coefficient of thermal expansion around 950^oC is in range of $5.71x10^{-6}$ K^{-1} to $12.42x10^{-6}$ K^{-1} and TG at 950^oC is below 10%. Response surface method was also used to determine the thermal properties of geopolymer as a function of mix proportions of raw materials. The study also investigated the changes of microstructure when the geopolymer is subjected to high temperature via X-ray diffraction (XRD) and scanning electron microscopy (SEM).

Keywords: Coefficient of thermal expansion, Diatomaceous earth, Geopolymer, Red mud, Rice husk ask, Thermal conductivity

Introduction

Geopolymer, originally named as "soil cement", is a kind of synthetic alumino-silicate material that is found to have several applications such as an alternative material for high-performance composites, ceramics, as well as, as a replacement for Portland cement [1-5]. Geopolymerization is based on a chemical reaction between different alumino-silicate oxides with silicates under highly alkaline conditions, yielding polymeric Si – O – Al – O bonds [1]. The thermal and chemical stability of these bonds are hypothesized to be determined by the nanostructure and molecular structure within the gel phase [1, 6]. In the raw materials, these oxides are believed to be in amorphous phases that participate in geopolymerization process whereas the crystalline phases do not take part in forming the geopolymers [1-2, 6]. The geopolymerization is initiated first with the dissolution of SiO_2 and Al_2O_3 from solid aluminosilicate materials in high alkaline condition [1-2, 5]. The basic oligomer chains are then formed that include silicate, alumina, and alumino-silicate and other oligomer chains.

Raw materials to produce geopolymers can be derived from waste materials such as red mud waste and rice husk ash. In this study, red mud (RM) came from industrial waste of Lam Dong bauxite plant in Viet Nam that can be dangerous to local ecological system as well as to the life of local residents if not properly managed. On the other hand, rice husk is an agricultural waste that is abundant in Asian countries such as China, India, Indonesia, Thailand, Philippines, and Viet Nam. Burning of such rice husk produces rice husk ash (RHA) containing high silica content [7]. For example, the total estimated RHA reserves are over 28 million metric tons every year [8]. Rice husk ash used in this study is from Mekong delta (South of Viet Nam) which accounts for 90% total rice production of this country. Thus, there has been a large amount of this agricultural waste discharged that need to be treated or reused. On the other hand, diatomaceous earth (DE) which is a natural mineral locally abundant in some parts of Vietnam, especially in Lam Dong province (near Lam Dong bauxite plant) contains high amorphous silica and alumina that can also be used in geopolymerization reaction. DE is typically used as a raw material for production of insulating or refractory materials because of its advantages for thermal specifications at high temperature [9-10]. Hence, DE is considered in the mix to improve the thermal properties of geopolymer-based materials at high temperature.

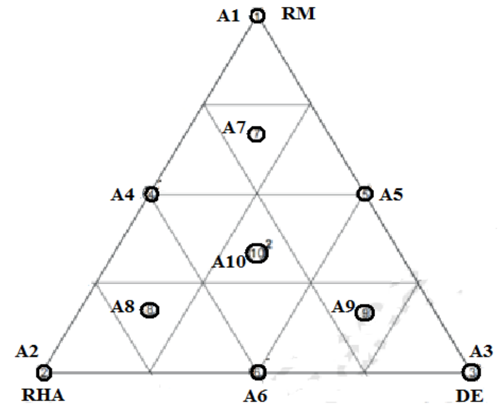
Since RM is rich in alumina while RHA contains high silica and DE has both alumina and silica in its composition, these raw materials are viable raw materials to form geopolymer-based material. The product of this geopolymerization process is an inorganic material with potentially high heat resistance and thermal properties comparably better than cement-based materials as reported in the literature [5]. In this study, the thermal properties of geopolymers obtained from the mixture of red mud, rice husk ash, and diatomaceous earth are investigated and compared it with the reference cement paste.

Materials and Method

All raw materials namely RM, DE and RHA were pre-treated and subjected to drying, grinding, and sieving processes. These were then tested for characterization using X-ray fluorescence (XRF), X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR). Water glass or sodium silicate with a silica modulus parameter of 2.4 (32% SiO_2, 13% Na_2O and 55% H_2O) was used to increase the pH value and form cohesiveness in the mixtures. The raw materials were mixed in different mix proportion as shown in Table 1 and added with 15% water glass solution (by weight of solids), and produced a paste mixture with the pH value of around 12 by adding water. The mixtures were then placed in 50x50x100 (mm^3) molds to form geopolymer samples. Reference cement pastes were also prepared by mixing Portland cement powder and water with a water-to-cement ratio of 0.4. After molding, the geopolymer and cement paste samples were cured at room condition (28^oC, 80% humidity) for 28 days and then were tested for thermal properties (see Figure 1) such as thermal conductivity and coefficient of thermal expansion.

Table 1. Mix Proportions of Raw Materials Used to Produce Geopolymer Product

Position	Proportion (%)		
	RM	RHA	DE
A1	100	0	0
A2	0	100	0
A3	0	0	100
A4	50	50	0
A5	50	0	50
A6	0	50	50
A7	66	17	17
A8	17	66	17
A9	17	17	66
A10	33.3	33.3	33.3



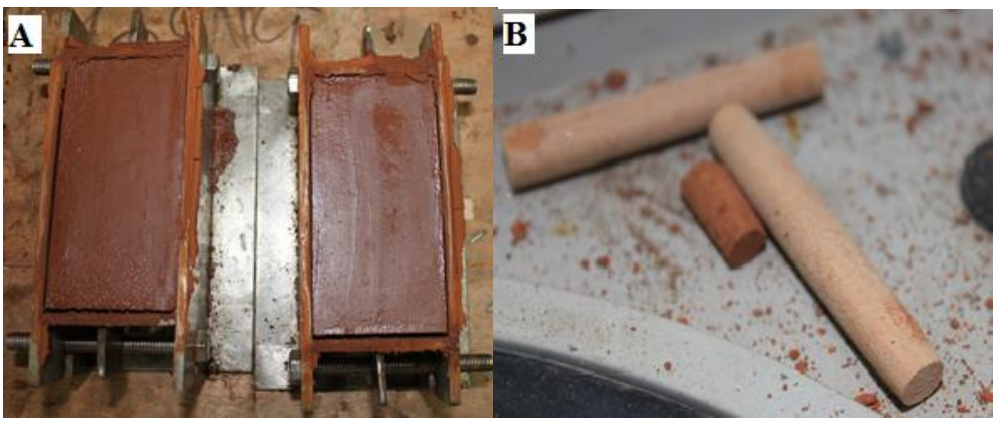


Figure 1. Geopolymer samples tested for thermal conductivity (A) and thermal expansion (B)

The thermal conductivity of the samples was measured using QTM – 500 Instrument that uses hot wire method to determine the coefficient of thermal conductivity (λ, W/m.K)**.** The coefficient of thermal expansion (α, K^{-1}) of geopolymer samples was measured using a dilatometer (Netzsch DIL 402 PC Instrument). In the experiments, the temperature was set up from 25^{o}C (room temperature) to 950^{o}C. The weight changes were also measured after subjecting the specimen to 950^{o}C inside a furnace using thermogravimetric analyser (Rigaku thermo plus TG8120). X-ray diffraction (Rigaku X-ray diffractometer, $\lambda_{Cu\ K\alpha}$ = 1.54Å) measurements were done to selected geopolymer sample before and after subjecting to high temperature.

Results and Discussions

Characterization of Raw Materials

Table 2 and Figure 2 summarize the results from the XRF and XRD analysis of the raw materials. As shown in Table 2, red mud (RM) has high iron and aluminum content. There are 2 outstanding peaks of gibbsite ($Al(OH)_3$) and iron oxide (Fe_2O_3) crystals associated with red mud as shown in Figure 2. As expected, rice husk ash (RHA) contains high silica which is 90.90% SiO_2 (wherein crystalline phase is in cristobalite structure). The broad hump in the XRD spectrum also suggests of amorphous silica phase in this waste material. On the other hand, diatomaceous earth (DE) contains 49.61% SiO_2, 16.81% Fe_2O_3, 16.63% Al_2O_3 in quartz crystal and clay minerals such as nontronite ($Na_{0.3}Si_2Fe_4O_{10}(OH)_2.4H_2O$), halloysite ($Al_2Si_2O_5(OH)_4.2H_2O$), and kaolinite ($Al_2Si_2O_5(OH)_4$). Note that clay minerals are more advantageous for forming or molding the geopolymer samples because of their high plasticity but it could also be a drawback for thermal properties of geopolymer product. When clay minerals are exposed to high temperature, they will have thermal reactions and dehydration that could change into other crystalline microstructures [11]. Furthermore, both RM and DE have high loss on ignition values (LOI) at 16.22% and 9.64% respectively whereas RHA has relatively small LOI.

Table 2. Chemical Composition (in weight) of RM, RHA, and DE

Oxides	Al_2O_3	SiO_2	Fe_2O_3	K_2O	CaO	TiO_2	Others	L.O.I	Water Content (%)
RM	18.98	4.52	49.90	0.05	0.87	5.62	0.94	16.52	2.66
RHA	1.12	90.90	0.54	4.66	1.41	-	0.60	0.77	0.23
DE	16.63	49.61	16.81	2.01	1.00	1.51	2.73	9.64	7.03

As shown in Figure 3, the structural bonds in raw materials were detected by Fourier Transform Infrared (FTIR). Standard spectrum of inorganic ions [12] was used to find the bonds existing in the raw materials. RM and DE contain clear structure particularly to that of unbounded water (H_2O), $O\text{-}H^-$, Si-O, Al-O, Fe-O and possibly tetra-silicate $[SiO_4]^{4-}$, tetra-aluminate $[AlO_2]^-$. On the other hand, FTIR results suggest that only Si-O bonding is present in RHA microstructure and there is very little information for water or $O\text{-}H^-$ since this material was obtained from burning rice husk.

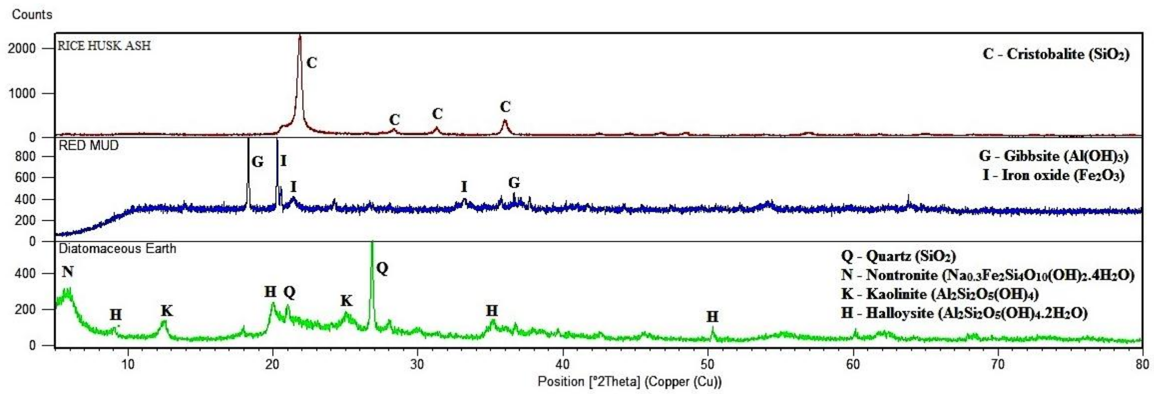


Figure 2. XRD patterns ($\lambda_{Cu\ K\alpha}$ = 1.54Å) of RHA, RM, and DE

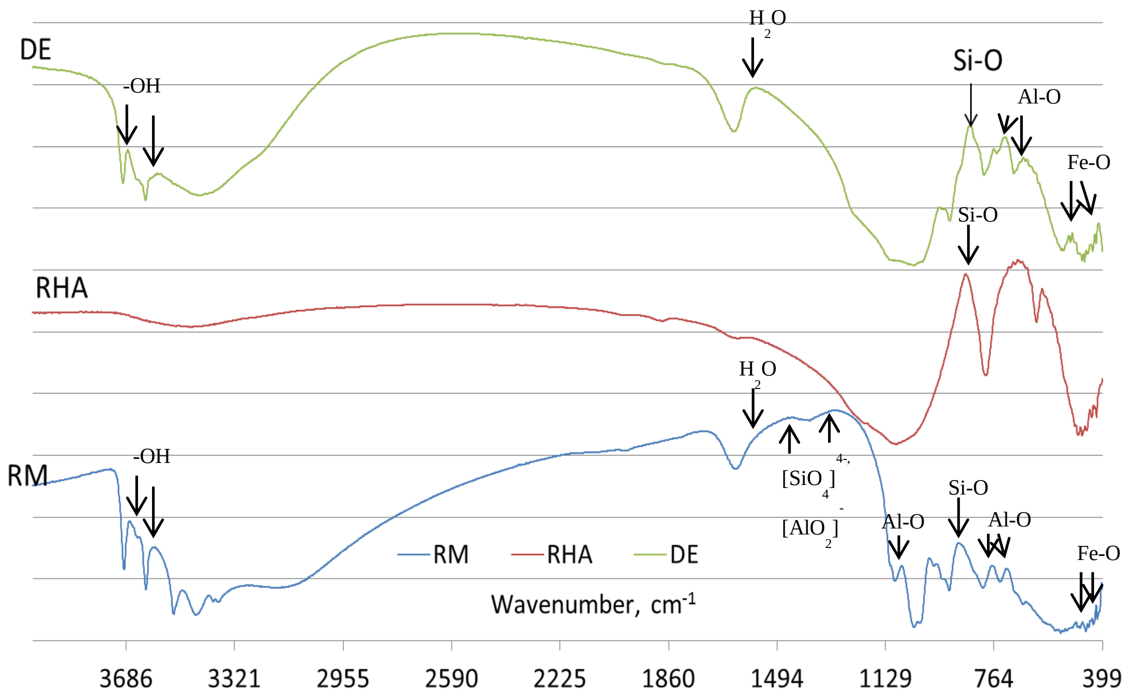


Figure 3. FTIR (FT/IR-6100 FT-IR Spectrometer) of raw materials (RM, RHA, and DE)

Thermal Properties of Geopolymer-Based Material

The coefficients of thermal conductivity of geopolymer-based materials are summarized in Table 3. Using statistical analysis (Design-Expert software program), the regression model and optimal region for thermal conductivity in the three-component diagram (RM, RHA, DE) are shown in Table 4 and Figure 4, respectively.

Table 3. Thermal Conductivity λ (W/m-K) of Geopolymer Samples

Samples	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
λ (W/mK)	1.2035	0.4713	0.9072	0.4842	0.5332	0.4994	0.6209	0.5234	0.5155	0.5298

Table 4. Analysis of Variance for the Significance of Regression Model for Themal Conductivity (W/m.K) As Response Variable

Response 1	Themal Conductivity (W/m.K)				
ANOVA for Reduced Quadratic Mixture Model					
Source	Sum of Squares	df	Mean Square	F-Value	p-value (Prob > F)
Model	0.47	4	0.12	14.27	(0.0061) Significant
Linear Mixture	0.21	2	0.11	12.93	(0.0106) Significant
AB	0.069	1	0.069	8.39	(0.0339) Significant
AC	0.19	1	0.19	23.02	(0.0049) Significant
Residual	0.041	5	8.265E-003		
Regression model	Themal conductivity (W/mK) = 11.87E-003*RM + 4.40E-003*RHA + 8.30E-003*DE – 1 .17E-004*RM*RHA – 1.94E-004*RM*DE				

Values of “Prob > F” less than 0.0500 indicate model terms are significant.
A = proportion of red mud; B = proportion of rice husk ash; C = proportion of diatomaceous earth;

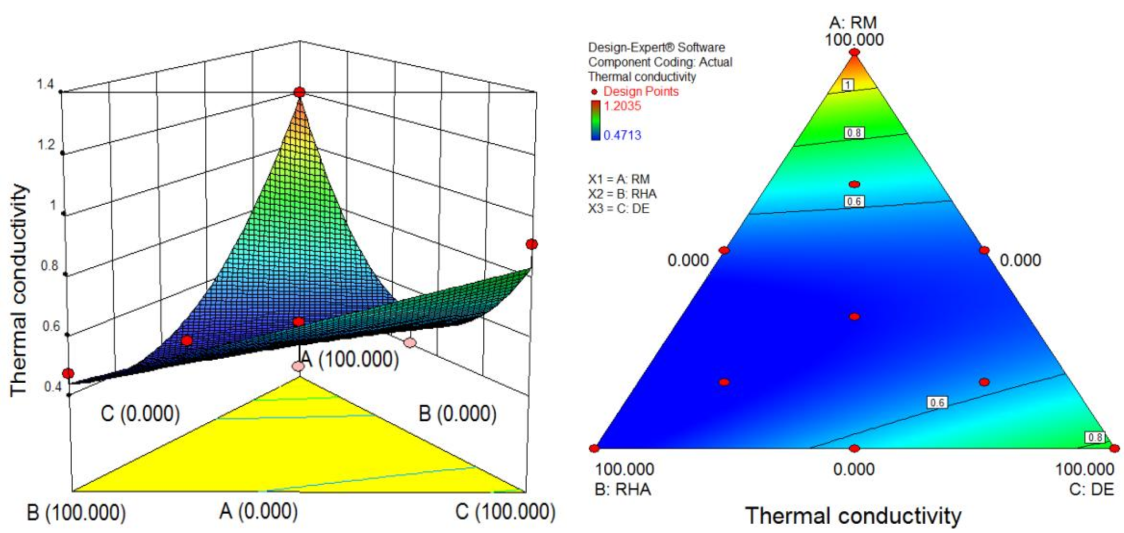


Figure 4. Response surface plots and their projections onto the ternary diagram for themal conductivity (W/m.K) of geopolymer specimens after 28 days of curing

Among the ten samples, A1 (100% RM) and A3 (100% DE), have higher thermal conductivity values at 1.2035 (W/m.K) and 0.9072 (W/m.K), respectively. This is expected as these samples contain high clay with high water content [13] and the degree of geopolymerization is the lowest in comparison with the other samples [14–15]. In addition, both DE and RM have high iron content that would result to higher thermal conductivity. On the other hand, the other samples have thermal conductivity that range from 0.47 to 0.62 W/m.K which are lower than that of reference cement paste with a value of 1.206 W/m.K. Indication suggests from the response surface plot that ternary-blended geopolymer with high RHA mix proportion relative to the other two raw materials would yield better thermal properties in terms of thermal conductivity than that of conventional Portland cement-based materials.

Results from dilatometer experiments of geopolymer-based materials are summarized in Table 5. Statistical analysis results are summarized in Table 6-7 and Figure 5-6. The change in linear dimension (dL/L, %) has the highest value of 1.19% at 950^{o}C. The lowest values belong to samples A6, A9, A3 containing high DE with dL/L values of 0.55%, 0.60%, 0.64%, respectively. The linear expansion coefficients (α, K^{-1}) are in the range of 5.71x10^6 K^{-1} to 12.42 x10^6 K^{-1}, and these values are similar to that of normal ceramics [16]. On the other hand, thermal properties such as coefficient of thermal expansion and linear dimension were not measured for cement paste samples since they were broken at such high temperature. This demonstrates the advantage of the ternary-blended geopolymer over cement paste in terms of thermal stability at high temperature. Indication suggests from the response surface plots that ternary-blended geopolymer with high DE mix proportion relative to the other two raw materials would yield better thermal properties in terms of thermal expansion. Note that DE is a raw material for refractory production because of its high fire resistance as well as high structural stability at high temperature [9-10, 17].

Table 5. Coefficient of Thermal Expansion of Geopolymer – Based Materials

Samples	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
dL/L (%)	1.02	1.19	0.64	0.83	0.72	0.55	0.82	0.90	0.60	0.73
αx10^6 (K^{-1})	10.54	12.42	6.69	8.70	7.48	5.71	9.46	9.78	6.49	7.89

Table 6. Analysis of Variance for Significance of Regression Model for Change of Linear Dimension, dL/L (%) As Response Variable

Response 2	**Change of Linear Dimension, dL/L (%)**				
ANOVA for Reduced Quadratic Mixture Model					
Source	Sum of Squares	df	Mean Square	F-Value	p-value (Prob > F)
Model	0.33	4	0.083	23.08	(0.0020) Significant
Linear Mixture	0.22	2	0.11	30.26	(0.0016) Significant
AB	0.039	1	0.039	10.69	(0.0222) Significant
BC	0.077	1	0.077	21.32	(0.0058) Significant
Residual	0.018	5	3.608E-003		
Regression model	Change of linear dimension, dL/L (%) = 9.82E-003*RM + 12.02E-003*RHA + 6.02E-003*DE				

	– 8 .73E-005*RM*RHA – 1.23E-004*RM*DE

Values of “Prob > F” less than 0.0500 indicate model terms are significant.
A = proportion of red mud; B = proportion of rice husk ash; C = proportion of diatomaceous earth;

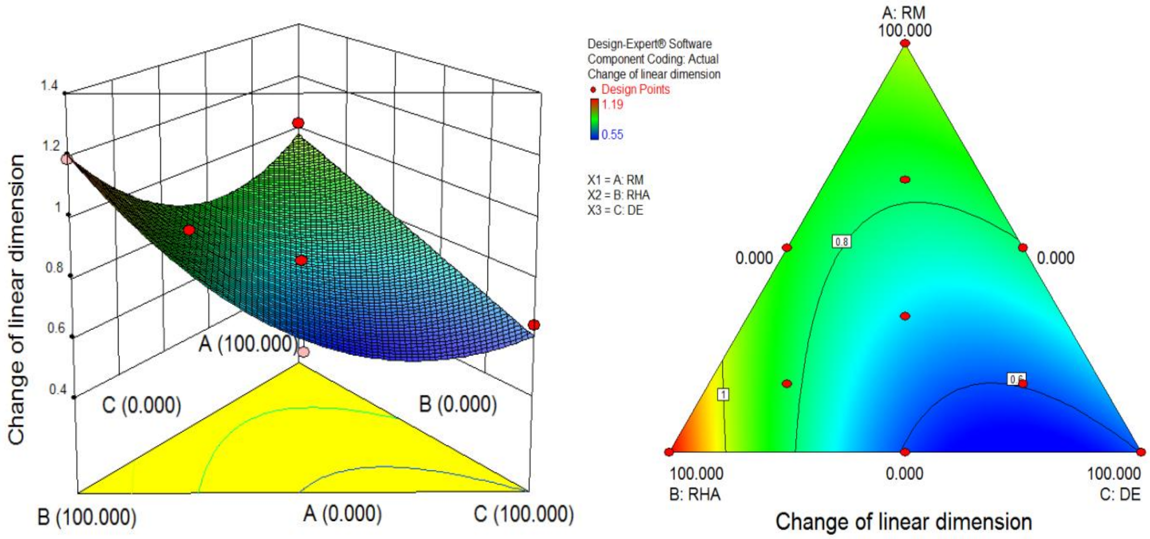


Figure 5. Response surface plots and their projections onto the ternary diagram for change of linear dimension (%) of geopolymer specimens after 28 days of curing

Table 7. Analysis of Variance for Significance of Regression Model for Coefficient of Thermal Expansion, α (1/K) As Response Variable

Response 3	**Coefficient of thermal expansion, α (1/K)**				
ANOVA for Reduced Quadratic Mixture Model					
Source	Sum of Squares	df	Mean Square	F-Value	p-value (Prob > F)
Model	3.241E-011	3	1.080E-011	10.42	(0.0086) Significant
Linear Mixture	2.483E-011	2	1.242E-011	11.96	(0.0081) Significant
BC	7.577E-012	1	7.577E-012	7.30	(0.0355) Significant
Residual	6.229E-012	6	1.038E-012		
Regression model	Coefficient of thermal expansion, α (1/K**)** = 9.62E-008*RM + 1.17E-007*RHA + 6.42E-008* – 1.22E-009*RHA*DE				

Values of “Prob > F” less than 0.0500 indicate model terms are significant.
A = proportion of red mud; B = proportion of rice husk ash; C = proportion of diatomaceous earth;

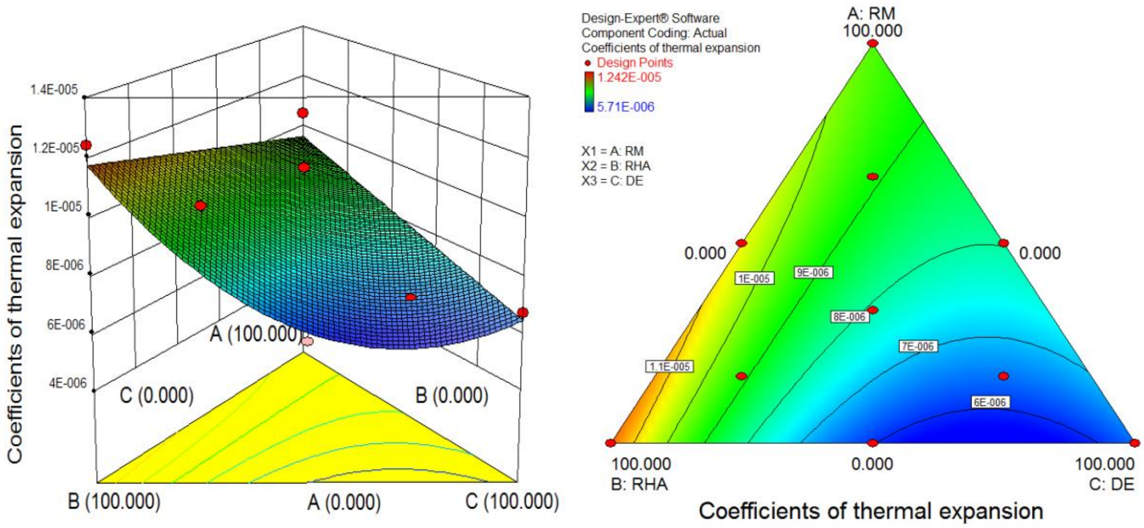


Figure 6. Response surface plots and their projections onto the ternary diagram for coefficient of thermal expansion (1/K) of geopolymer specimens after 28 days of curing

Results from themogravimetric analysis are shown in Table 8. It describes the weight loss values of the geopolymer-based materials at 950^{o}C. Statistical analyses of these results are summarized in Table 9 and Figure 8. Weight loss values increased significantly when the geopolymer mixtures were mixed more of DE and RM. It increased from 3.04% (sample A2, 100%RHA) to 21.22% (sample A1, 100%RM) and 19.96% (sample A5, 50%DE and 50%RM). Such behavior could be explained by the higher LOI values of RM and DE. As lower weight loss is desired for structural stability of geopolymer at high temperature, indication from the response surface plot (see Figure 8) suggests that the ternary-blended geopolymer with high RHA mix proportion relative to the other two raw materials would yield better thermal properties in terms of weight loss. Geopolymer samples A2, A4, and A8 have the lowest values of mass loss at 3.04%, 9.75%, and 10.31% because they contain high rice husk ash (or high silica). On the other hand, the reference cement paste was observed to have a weight loss of 28 %. Note that the weight loss of cement paste at high temperature is attributed to the dehydration and decomposition of hydrated cement minerals which result to structural degradation [18-19].

Table 8. Weight Loss Values of Geopolymer – Based Materials at 950^{o}C

Samples	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Weight Loss (%)	21.22	3.04	17.12	9.75	19.96	14.24	17.52	10.22	14.53	15.58

Table 9. Analysis of Variance for Significance of Regression Model for Weight Loss (%) As Response Variable

Response 4			**Weight Loss (%)**		
ANOVA for Reduced Quadratic Mixture Model					
Source	Sum of Squares	Df	Mean Square	F-Value	p-value (Prob > F)
Model	251.50	3	83.83	42.47	(0.0002) Significant
Linear Mixture	238.24	2	119.12	60.35	(0.0001) Significant
BC	13.26	1	13.26	6.72	(0.0411) Significant
Residual	11.84	6	1.97		
Regression model	Weight loss = +207.58E-003*RM + 25.296E-003*RHA + 167.26E-003*DE +1.62E-003*RHA*DE				

Values of "Prob > F" less than 0.0500 indicate model terms are significant.
A = proportion of red mud; B = proportion of rice husk ash; C = proportion of diatomaceous earth;

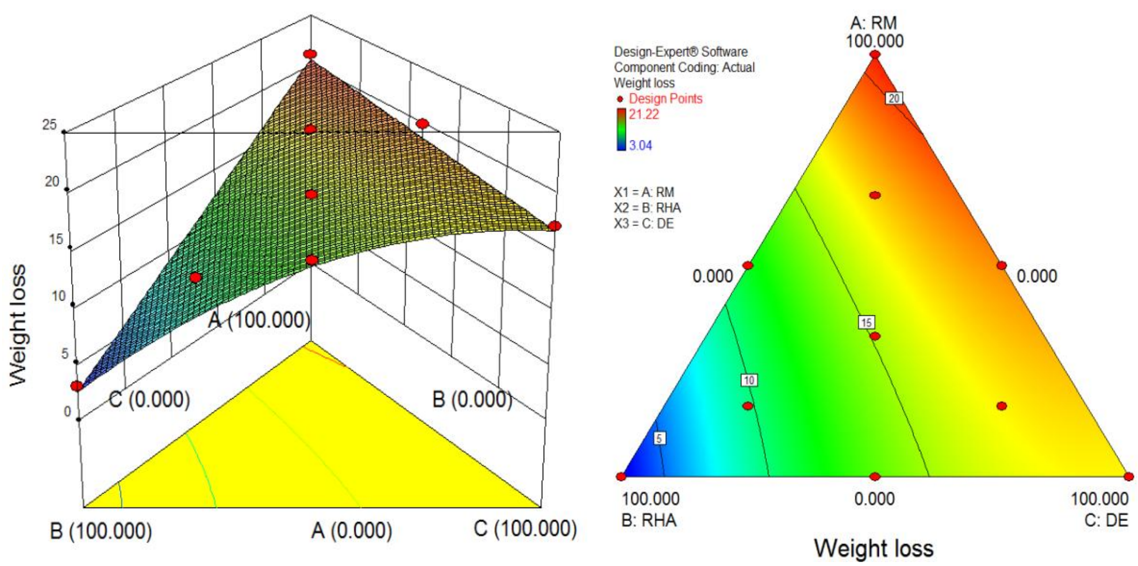


Figure 8. Response surface plots and their projections onto the ternary diagram for mass loss (%) of geopolymer specimens after 28 days of curing

As reported in Nguyen et al. (2013), A8 sample has the highest compressive strength among the samples [14]. Geopolymer A8 represents the ternary blended mixture with most thermal specifications comparable or better than the other samples. Thus, A8 sample was also further characterized before and after heating at 950 ^{o}C.

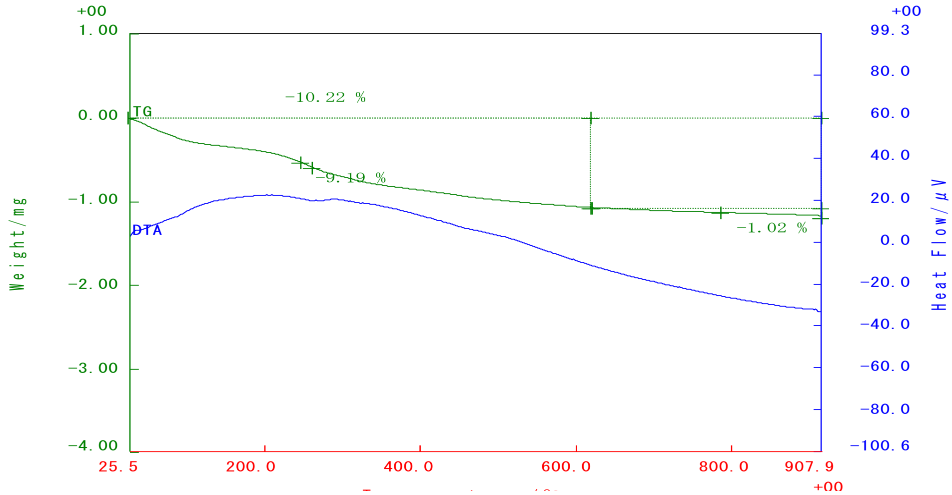


Figure 8. TG/DTA of geopolymer -based material, sample A8

Ternary-blended geopolymer A8 (17%RM, 66%RHA, 17% DE in weight) was further analyzed to determine the physical and chemical properties as the material during the heating process from room temperature to 950^oC. Figure 8-10 illustrate the DTA/TG curve and XRD pattern of A8, respectively. Indications suggest from the DTA curve that there were no significant thermal reactions in the geopolymer matrix. Only loss of weight of 10.22% associated with water content and L.O.I is indicated in the TG curve. Weight loss around 9% is observed below 600^oC due to the evaporation of water trapped in clay minerals of the raw materials (RM, DE) [11], as well as, burning of organic or cellulose impurities.

The XRD patterns also indicate high microstructural stability of geopolymer-based materials even for samples exposed at 950^oC as shown in Figure 9 and 10. Comparing the XRD pattern of geopolymer before and after heating to 950^oC, no significant difference was observed except that of the peak associated from gibbsite. After heating, the presence of gibbsite is no longer detected from the XRD pattern. This could be explained by the thermal reaction of gibbsite at around 280^oC to produce aluminum oxide in the form of χ-Al_2O_3, γ-Al_2O_3, η- Al_2O_3, and even amorphous Al_2O_3 [20-21]. In addition, the results also indicate higher intensity of peak associated with cristobalite than that of quartz and gibbsite in both XRD patterns suggesting the large presence of silica associated with rice husk ash.

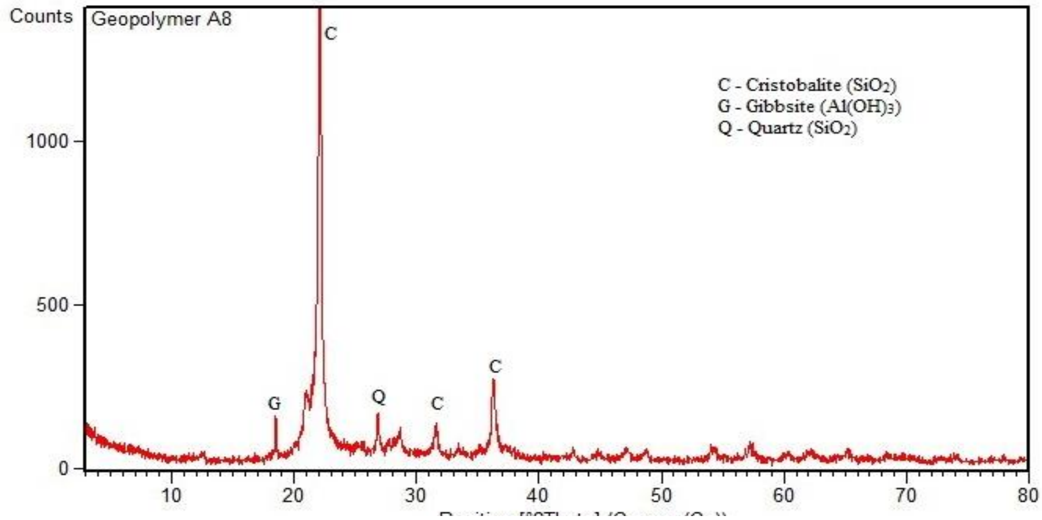


Figure 9. XRD pattern ($\lambda_{Cu\ K\alpha}$ = 1.54Å) of geopolymer sample A8 before heating

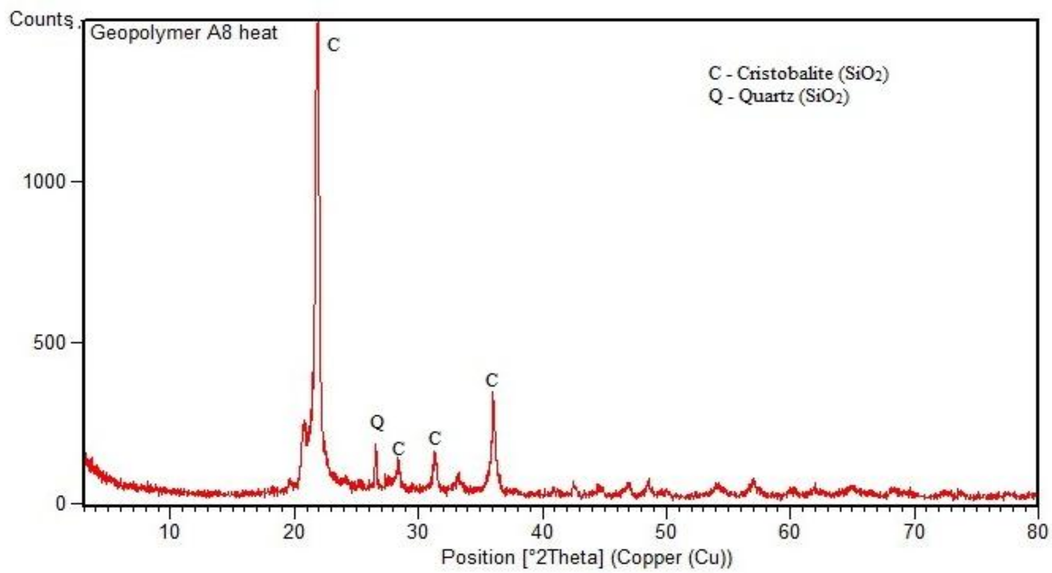


Figure 10. XRD pattern ($\lambda_{Cu\ K\alpha}$ = 1.54Å) of geopolymer sample A8 after heating at 950^{o}C

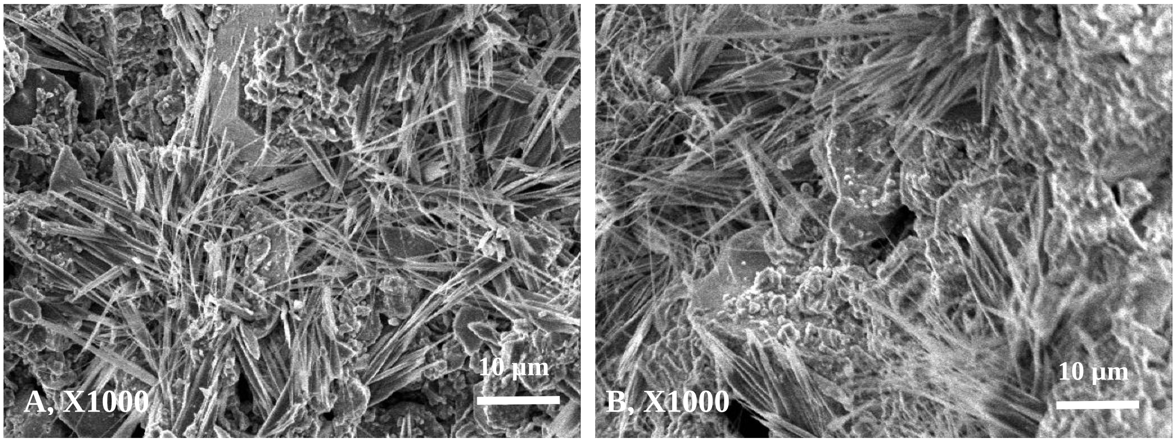


Figure 11. Micrograph of geopolymer sample A8 before (A) and after heating to 950^0C (B)

As indicated in Figure 11, long rods suggesting cross-linked alumino-silicate polymeric network in geopolymer microstructure were observed in the SEM micrograph and no significant changes were observed even if the sample was subjected to high temperature. Thus, the geopolymer product demonstrates high thermal stability even at high temperature.

Conclusions

This study investigates the thermal properties of geopolymer produced from the mixture of red mud (RM), rice husk ash (RHA), and diatomaceous earth (DE). This geopolymer-based material shows promising property that can be used as an alternative material and a solution to manage the red mud and rice husk ash waste. Indication suggests that the ternary-blended cement with high mix proportion of RHA relative to RM and DE, for example, sample A8 (17% RM, 66% RHA, and 17% DE) would yield thermally stable insulating material that would be comparable or even perform better than Portland cement-based materials even when subjected to high temperature. Future studies will be done to determine the optimal mix formulation based on desired engineering and thermal specification using multiple objective optimization technique.

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