J. Myanmar Acad. Arts Sci. 2019 **Vol. XVII. No.1B**

MECHANICAL PROPERTIES AND CHARACTERIZATION OF PREPARED GEOPOLYMER, BLENDED CEMENT AND COMMERCIAL CEMENT

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Abstract

The focus of this research mainly concerned with mechanical properties and characterization of prepared geopolymer(GP),blended cement (BC) and commercial cement (CC). Geopolymer is a new family of synthetic aluminosilicate materials formed by alkali activation of solid aluminosilicate raw materials. Rice husk ash and kaolinite based geopolymer had been prepared from rice husk ash which was obtained from Taungoo Township, Bago Region and kaolinite sample was obtained from Kyaut Taga, Kyauk Padaung Township, Mandalay Region. In this research work, prepared geopolymer, blended cement (GP+commercial cement) and commercial cement samples were characterized by EDXRF, XRD, FT IR and SEM techniques. Physical properties such as normal consistency and setting time and mechanical properties such as compressive and tensile strength of prepared geopolymer and commercial cement were studied. The normal consistency values were found to be 30 % for prepared geopolymer at setting time between 125 and 448 min, 28% for blended cement at 90 and 350 min and 28% for commercial cement at 95 and 240 min. The maximum compressive strength values of prepared geopolymer (GP), blended cement and commercial cement (elephant brand) were 38.36 N/mm^2, 60.99 N/mm^2 and 69.08 N/mm^2, respectively, at 56 days. The highest tensile strength values for prepared geopolymer, blended cement and commercial cement were 198 psi, 243 psi and 264 psi, respectively. To determine the acid resistance, prepared GP, blended cement and commercial cement (Elephant Brand) were immersed in various dilute acid (10 % HCl, 10 % H_2SO_4, 10 % CH_3COOH). After three months immersion in acid period, the mechanical strength and tensile strength were determined. Compressive strength of prepared, blended cement and commercial cement after treating with 10 % HCl were found to be highest. Tensile strength values of prepared (GP), blended cement and commercial cement after treating with 10 % HCl were also found to be the highest. Prepared geopolymer was found to be more resistant to acid than that of the commercial cement.

Keywords: geopolymer, normal consistency, setting time, compressive strength, tensile strength, acid resistance

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Introduction

Carbon dioxide produced during Portland cement manufacture. Which result from calcination of limestone and silico-aluminate material. The basic need for cement in developing countries is capable for producing the necessary alimentary goods, concrete and building houses (Gowthami, 2016). Economic development is reflected by the growth rate of infrastructure and highlighted by the growth rate of cement production. Minor reduction of carbon dioxide emission with blended cements is (a) reabsorption of atmospheric carbon dioxide during the carbonation of concrete and (b) blending of Portland cement with industrial by-products.

Geopolymer is an amorphous alumino-silicate material. Framework structure produced by condensation of aluminosilicate units with alkali metal ions balancing the charge associated with tetrahedral aluminium (Szmal and Zainuddin, 2015). Tetrahedral complex consisting of Si or Al coordinated through covalent bonds to four oxygen(Brock,2011). Source materials corresponds to industerial waste like fly ash, rice husk ash, granulated blast furnance slag and red mud, etc (Rohit and Mayerghan, 2017).

Rice husk ash is also industrial waste produced by burning rice husk primarily for the generation of electricity, a kind of sustainable biomass energy (kartini, 2011). Rice husk, a hard protective shell of rice grains, is an agricultural by- product of rice mills. The main component of a rice husk ash is silica (>90-95 wt. %), existing predominantly in amorphous and partly in crystalline phase of silica. Rice husk ash is alternative source high specific area silica (Zeynab and Nitafar, 2015).

Kaolinite is the principal mineral of the kaolinite group clay minerals. Soil dominated by oxide and kaolinite clays are characterized by very stable soil aggregates and they exhibit a low degree of plasticity. Large amount of oxide and kaolinite clays in a soil contribute to the formation of extremely stable soil aggregrates because the clays tend to neutralize each other. Kaolinite has a net negative charge. Kaolinite directly formed from primary minerals in soils of the humid tropics. Kaolinite provides a structure-forming species to the overall geopolymerization process (Liew, 2011). Addition of metakaolin as partial replacement of cement causes increase in mechanical property and decrease in workability of concrete (Parthiban and

Vaithianathan, 2015). Although kaolinite is very stable, it can weather to form gibbsite, $Al(OH)_3$. Kaolinite and rice husk ash are abundantly available to replace totally manufactured cement and make a concrete-like materials.

Geopolymer cement is a binding system that hardens at room temperature, like regular Portland cement. It has ability to form chemical bond with silicate rock based aggregates. Kaolinite and rice husk ash are abundantly available to replace totally manufactured of cement and make a concrete-like materials. Geopolymeric materials can have better chemical and mechanical properties than Ordinary Portland cement products (Rajesh *et al*., 2014).

Mechanical properties are normal or standard consistency, setting time, compressive strength and tensile strength, etc. Normal consistency is the percentage water requirement of cement paste at which viscosity of the paste becomes such a plunger in a specially designed apparatus (Vicat's apparatus) penetrates a depth 5 mm to 7 mm, measured from bottom of the mould (Suresh *et al*., 2009). Setting time is the time elapsed between the moment the water is added to the cement, and the time when the paste has completely lost its plasticity and has completely sufficient firmness to resist certain definite procedure. The capacity of a material or structure to withstand loads tending to reduce size.

This paper presents an experimental study on the mechanical properties and characterization of a geopolymer composites derived from silica and alumina rich raw materials. Therefore, the main objective is to investigate the mechanical properties of rice husk ash and kaolinite based geopolymer and to characterize the composition and microstructure of the resulting products.

Materials and Methods

Sample Collection

Local rice husk samples collected from Taungoo Township, Bago Region and Kaolinite from Kyaukpadaung Township, Mandalay Region (Figures 1 and 2) were used in the present work.

Figure 1: Rice husk sample

Figure 2: Kaolinite sample

Sample preparation

Rice husk sample (100 g) was placed in a clean dry porcelain basin and its weight was determined. The sample was placed inside the Electric Muffle Furnace and the temperature was raised gradually from 200 °C to 700 °C until it was burnt completely. After three hours, the samples were cooled and kept in a dessicator and then weighed again. The percent ash of the sample was calculated.

Kaolinite sample was piled up into a cone and dividing it into quarters. Opposite quarters were rejected and the remaining half–portion again treated as before, rolling the sample back and forth on a paper. After quartering, the sample was ground in a motar and pestle and then sieved with 200 mesh sieves.

Preparation of rice husk ash and kaolinite based geopolymer, blended cement and commercial cement (Elephant Brand)

For preparation of geopolymer, rice husk ash and kaolinite (1:1, 2:1, 1:2 weight ratios) was mixed with 3mL of alkali solution in 2:3 (v/v) of 8 M NaOH : 0.1 M Na_2SiO_3 and 28 mL of water. Immediately the mixture was placed in the plastic mould. After 7, 14, 28, 56, 70 days, the specimens were removed from the mould. The blended cements (BC) were prepared by various mixing weight ratios of prepared GP3 : commercial cement (1:2). Cement (Elephant Brand) was used for commercial cement (CC) which was also placed in mould and after 7, 14, 28, 50 and 70 days, the speciemens were removed from the mould.

Component of sample number for prepared geopolymers

Source materials (Rice husk ash : kaolinite)		Prepare geopolymer samples
(1)	1:1	: GP1
(2)	2:1	: GP2
(3)	1:2	: GP3

Determination of Acid Resistance

Prepared geopolymer GP3, BC and CC were placed in (10 % HCl, 10 % H_2SO_4, 10 % CH_3COOH) for 6 months. After limiting time, the mechanical strength(compressive and tensile strength) of samples were be measured.

Methods

Geopolymer, blended cement and commercial cement (Elephant Brand) were characterized by EDXRF, XRD, FT-IR and SEM. Relative abundance of elemental oxide in rice husk ash and kaolinite samples was quantitatively determined by EDXRF analysis using -8000, Shimadzu. Co. Ltd, Japan). X-Ray differction pattern of the sample was recorded on X-ray diffractometer (Cat-No.9240 J101, Japan). SEM micrograpg of the samples was recorded on (Model Jeol-JSM- 5610LV, JEOL Ltd). FT-IR (Perkin Elmer 1600 Fourier Transform Infrared Spectrometer, Japan).Normal consistency and setting time were determined by Vicat apparatus. Compressive strength and tensile strength were determined by using compressive testing machine and tensile testing machine.

Results and Discussion

In this study, the first part is concerned with the mechanical properties and characterization of prepared geopolymer. The second part is the comparison of the mechanical properties of prepared geopolymer, blended cement and commercial cement.

Physical and Mechanical Properties of Prepared Geopolymer

For physical properties, ASTM standard cement types having specified certain physical requirement for each type of cement. These properties include normal consistency and setting time. Each one of these properties has an influence on the performance of the cement. Vicant needle was used to determine the amount of mixing water to make cement paste of a given consistency. The setting time depends on the composition of cement, temperature and quality of water used in gauging for ordinary cement. The initial and final setting time of geopolymer is important in practice because it established the time available for transport placing and compaction of geopolymer. According to results (Table 1) the initial and final setting times were within the limits of ASTM (American Society for Testing and Materials) standard setting times. A change from fluid to rigid state in GP2 was faster than those in GP1 and GP3 because high content of alkaline activator, NaOH, increased the setting and delayed polymer formation.

Table 1: Normal Consistency and Setting Time of Prepared Rice Husk Ash (RHA) and Kaolinite Based Geopolymer in Various Ratios

Prepared GP	Normal consistency (%)	Setting time (min) Initial	Final
GP1	32	100	430
GP2	33	108	415
GP3	30	125	448
*ASTM type(General Use)	-	Not less than 45 min	Not more than 480 min

For compressive strength, GP3 was found to have highest compressive strengths at different storage times. However, GP3 delays in polymer formation than GP2 and GP1 (Table 2).

Tensile strengths are also good in GP3 (Table 3). Among the prepared geopolymers, GP3 has highest compressive strength and tensile strength

Table 2: Compressive Strength of Prepared Geopolymer (GP) at Different Storage Time

Prepared GP	Compressive strength (N/mm^2) at different storage time (days)					
	7	14	28	42	56	70
GP1	10.62	15.17	19.03	23.82	30.28	28.22
GP2	9.11	13.13	18.46	21.90	22.21	19.98
GP3	12.53	16.17	20.48	28.47	38.36	36.34

Table 3: Tensile Strength of Prepared Geopolymer (GP) at Different Storage Times

Prepared GP	Tensile strength (psi) at different storage times (days)					
	7	14	28	42	56	70
GP1	50	82	132	169	184	165
GP2	40	46	72	119	139	128
GP3	70	103	175	193	198	185

Physical and Mechanical properties of prepared geopolymer, blended cement and commercial cement

In rice husk ash and kaolinite based geoplymer, GP3 sample with RHA: kaolinite wt. ratio of 1:2 was selected for geopolymer based cement. The physical and mechanical properties of prepared geopolymer GP3, blended cement BC and commercial cement CC were determined.

For physical properties of prepared geopolymer, initial setting value is not less than 45 min and final setting time value is not less than 480 min. The initial and final setting times of GP3, BC and CC values were found to be within ASTM standard cement ranges (Table 4). GP3 needs more time for setting than BC and CC.

For compressive strength, BC strength is nearly to CC strength. The compressive strength of samples are found to be high in 56 days (Table 5).

Table 6 shows the tensile strength of GP3, BC and CC. The strength of GP3 value was found to be high in 7, 14 and 28 days.

Table 4: Normal Consistency and Setting Time of Prepared Geopolymer (GP), Blended Cement (BC) and Commercial Cement (CC) Samples

Cement Samples	**Normal consistency (%)**	**Setting time (min)**	
		Initial	**Final**
GP3	30	125	448
BC	28	90	350
CC	28	95	240
*ASTM type(General Use)	-	Not less than 45 min	Not more than 480 min

Table 5: Compressive Strength of Prepared Geopolymer (GP) , Blended Cement (BC) and Commercial Cement (CC) at Different Storage Time

Cement Samples	Compressive strength (N/mm^2) at different storage times (days)					
	7	**14**	**28**	**42**	**56**	**70**
GP3	12.53	16.17	20.48	28.47	38.36	36.34
BC	25.65	25.81	38.33	46.01	60.99	54.28
CC	31.41	36.14	40.77	58.60	69.08	63.25

Table 6: Tensile Strength of Geopolymer (GP), Blended Cement (BC) and Commercial Cement (CC) at Different Storage Times

Cement Samples	Tensile strength (psi) at different storage times (days)					
	7	**14**	**28**	**42**	**56**	**70**
GP3	70	103	175	193	198	185
BC	50	98	172	202	243	238
CC	192	197	200	231	264	241

Characterization of the prepared geopolymer, blended cement and commercial cement

The composition and microstructure of the products were also characterized and compared by EDXRF, XRD, FT IR and SEM .

Semiquantitative analysis of prepared GP3, blended cement and commercial cement were characterized by EDXRF (Figures 3, 4, 5 and Table 7). Prepared GP3, contained higher content of Si than other elements. But for blended cement and commercial cement, Ca content was higher than the other elements. The rest of the other elements showed nearly the same composition. Therefore, the prepared geopolymer GP3 was used for the replacement for Portland cement.

According to XRD, semicrystalline state are formed in all type of cement samples (Figures 6, 7and 8).

In the FT IR, spectra of prepared GP, blended cement BC and commercial cement CC stretching vibration of Si-O are found in all cement samples (Figures 9, 10, 11 and Table 8).

The microstructure of aluminosilicate inorganic polymer was analysed using scanning electron microscope (SEM). Figures 12,13 and 14 show the three dimensional network of polysodium aluminosilicate in the prepared GP3, blended cement (BC) and commercial cement (CC). Moreover, prepared GP3 also exhibits the high density of pore or air bubbles. Micro cracking are also formed in GP3. CC has less void than the other two, GP3 and BC. Large voids are formed in BC because it has unreacted particles in the structure. All the three sample have nearly similar morphology

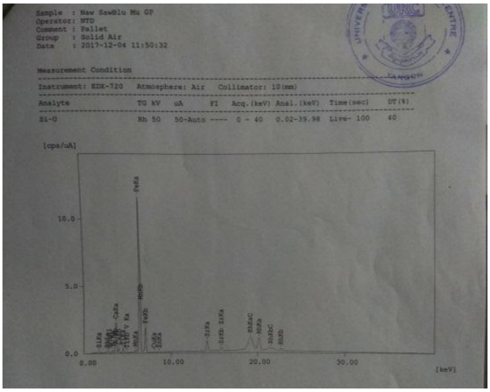

Figure 3: EDXRF spectrum of prepared geopolymer

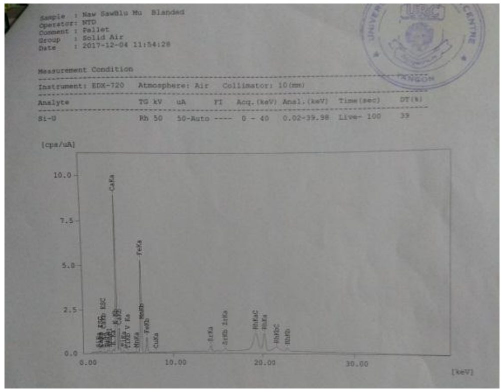

Figure 4: EDXRF spectrum of blended cement

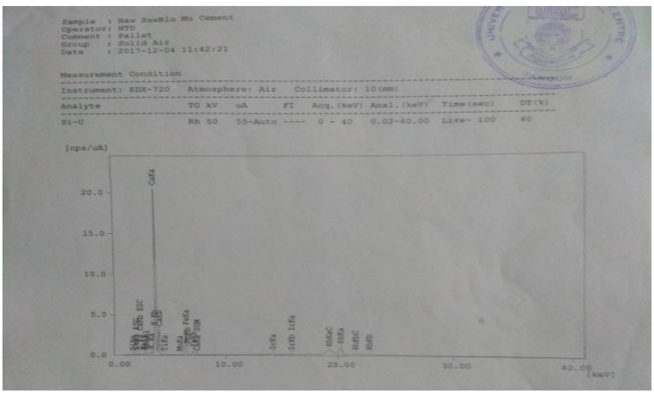

Figure 5: EDXRF spectrum of commercial cement

Table 7: Relative Abundance of Some Elements in Prepared Geopolymer, Blended Cement and Commercial Cement Samples by EDXRF Method

Cement Samples	Relative abundance of some elements in different samples (%)						
	Si	**Fe**	**Ca**	**K**	**Ti**	**Al**	**Mn**
GP3	32.21	31.18	25.09	3.35	2.14	1.03	0.67
BC	13.12	15.82	66.58	1.91	1.16	ND	0.30
CC	7.10	6.29	83.34	1.67	0.37	ND	0.06

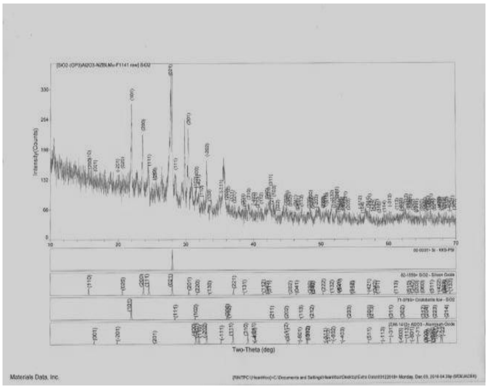

Figure 6: XRD diffactogram of prepared geopolymer (GP3)

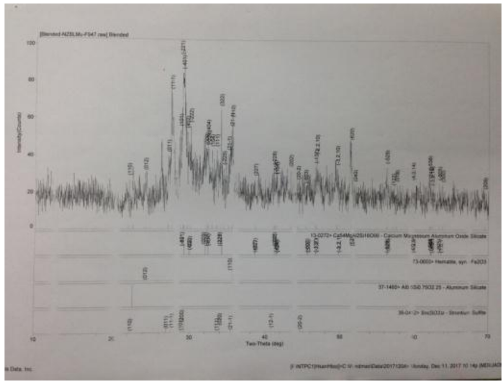

Figure 7: XRD diffactogram of blended cement (BC)

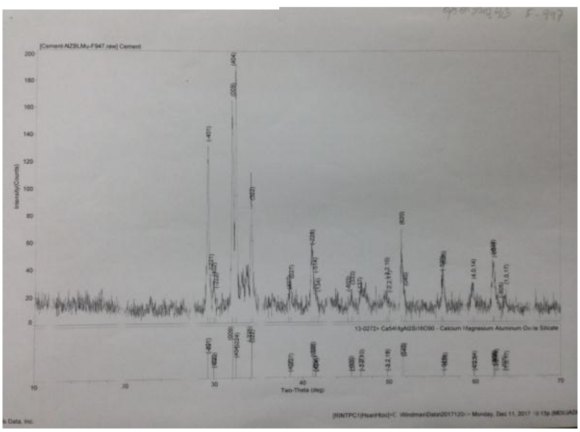

Figure 8: XRD diffractogram of commercial cement (CC)

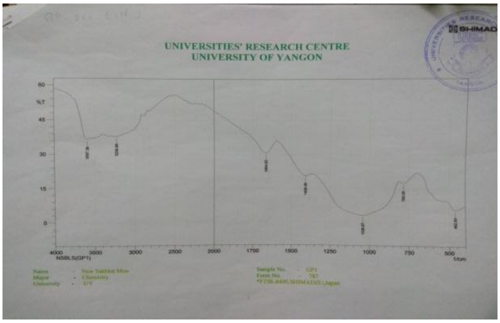

Figure 9: FT-IR spectrum of prepared geopolymer (GP3)

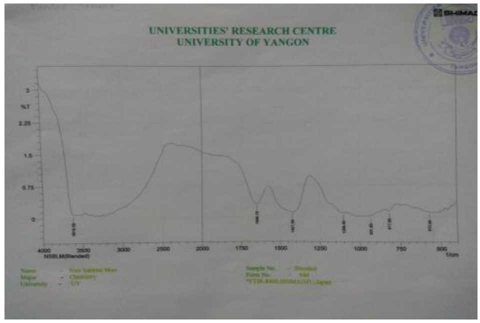

Figure 10: FT-IR spectrum of blended cement (BC)

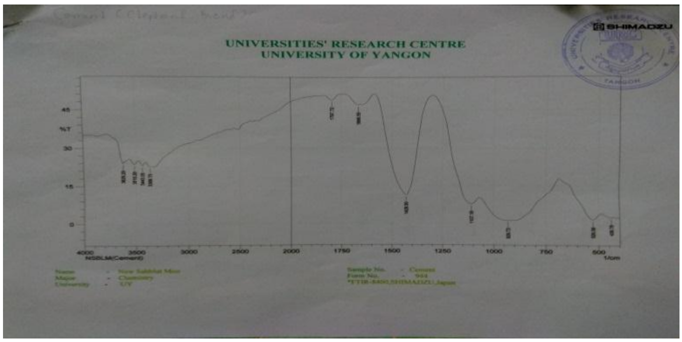

Figure 11: FTIR spectrum of commercial cement (CC)

Table 8:FT-IR Spectral Assignment of the Prepared Geopolymer (GP), Blended Cement (BC) and Commercial Cement (CC) Samples

Frequency (cm-^{1})			*Literature frequency (cm^{-1})	Band assignment (cm^{-1})
GP3	**BC**	**CC**		
3597	3515	3443,3518,3369	3000-3600	$\nu_{\text{(O-H)}}$
1022	1099	1107	1200-800	$\nu_{\text{(Si-O-Al)}}$
462	458	457	460	$\delta_{\text{(Si-O)}}$
1039	817,931	929	1080-790	$\nu_{\text{(Si-O)}}$

*Kuma & Kamar (2011)

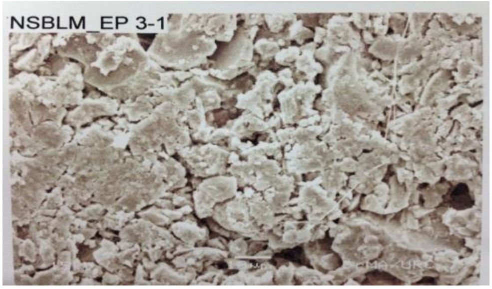

Figure 12: SEM micrograph of the prepared geopolymer (GP3) sample at 1000 × magnification

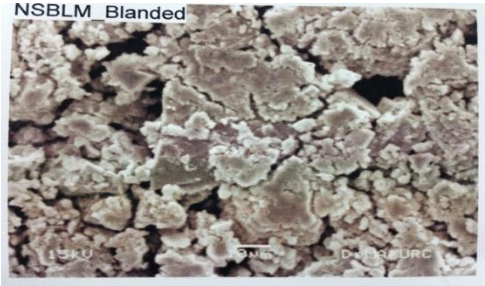

Figure 13: SEM micrograph of the blended cement (BC) sample at 1000 × magnification

Figure 14: SEM micrograph of the commercial cement (CC) sample at 1000 × magnification

Mechanical properties of prepared geopolymer, blended cement and commercial cement after treating with different acids at 28 days

After treating with different acids, the compressive strength values of GP3, BC and CC are shown in (Table 9). Acetic acid is more detrimental than mineral acid. So, samples in acetic acid condition is less strength than sulphuric acid and hydrochloric acid condition. In acid condition, GP3 and BC strength reduce 10% and 20 % than normal condition (immersed in water). And CC also reduce 25 percentage. So GP3 has high resistance in acid condition.

High alkalinity possesses higher degree of reaction and maintains a density to prohibit permeation of corrosion element. The tensile strength of CC, after treating with different acids decreased more than that of only treating with water (Table 10).

Table 9: Compressive Strength of Prepared Geopolymer (GP) Blended Cement (BC) and Commercial Cement (CC) After Treating With Different Acids

Cement samples	Compressive strength of prepared (GP),(BC) and (CC) after treating with different acids (N/mm^2)		
	10 % H_2SO_4	10 % HCl	10 % CH_3COOH
GP	23.72	24.41	22.21
BC	38.81	39.05	23.19
CC	40.32	45.25	32.96

Table 10: Tensile Strength of Prepared Geopolymer (GP) Blended Cement (BC) and Commercial Cement (CC) After Treating With Different Acid

Cement samples	Tensile strength of the prepared (GP),(BC) and (CC) after treating with different acids (psi)		
	10 % H_2SO_4	10 % HCl	10 % CH_3COOH
GP	179	187	166
BC	201	212	168
CC	219	223	172

Conclusion

This paper presents an experimental study that aim to convert two raw materials, RHA and kaolinite, to potentially useful construction material via geopolymerization, resulting in RHA-kaolinite geopolymer composites. A wide variety of synthesis parameters, including the curing duration, weight ratio of samples, particles size and alkalinity were examined to understand the extent and degree of geopolymerization and their influence on mechanical properties of final products. The microstructure and composition of the final products were also characterized by X-ray diffraction, SEM and chemical analysis. The mechanical properties of GP3 was found to be high strength value in prepared geopolymer. Blended cement (BC) strength value is nearly to commercial cement (CC) value. But, compressive and tensile strength of GP3, after treating with different acids were less than BC and CC. From this study, rice husk ash and kaolinite based geopolymer have found to be potential material for replacing the use of Ordinary Portland Cement in infrastructure development. However the different samples may give different ractivity due to their varying chemical compositions. The use of rice husk ash and kaolinite based geopolymer is more ecofriendly due to the redution of carbon dioxide emission and costs compared to Ordinary Portland Cement. Moreover, the durability of the prepared geopolymer is better than Ordinary Portland Cement when exposed to an agresssive environment.

Acknowledgements

The authors would like to express their profound gratitude to the Department of Higher Education (Yangon Office), Ministry of Education, Yangon, Myanmar, for provision of opportunity to do this research and Myanmar Academy of Arts and Science for allowing to present this paper.

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