Geopolymerization Potential of Gold Mill Tailings in Mindanao, Philippines

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Abstract: *This study focused on the characterization of gold mill tailings (GMT) from a mineral processing plant in Mindanao, Philippines and the evaluation of its potential for developing geopolymer products. The effect of alkali-activator concentration and curing temperature on the physical and mechanical properties of gold mill tailings-derived geopolymers were investigated. Scanning electron microscopy (SEM) imaging was performed to examine the microstructure of the produced specimens. Results showed that the most favorable alkali-activator concentration and curing temperature were 15M NaOH and 120°C, respectively. The geopolymers showed less than 10% water absorption and a maximum compressive strength of 9.3MPa. The GMT-derived geopolymer can be potentially used for eco-friendly materials like structural clays.*

Keywords: *Geopolymerization, gold mill tailings, alkali-activator, compressive strength*

1. INTRODUCTION

Tailings derived from hard rock mining accounts for a huge percentage of solid waste produced by large scale mining operations. A common method of disposing these tailings is by impounding them in a tailings pond. This impoundment structure, also termed as tailings service facility (TSF), covers large areas of land and requires maintenance and regular monitoring [1]. The waste rock in the form of slurry requires treatment to neutralize the effluents from the processing plant which entails additional costs. This may also cause environmental problems such as air contamination, water contamination and acid-mine drainage as well as safety problems related to dam failure. Tailings usually contain about 10-20 percent of the economic minerals that could not be recovered from the ore [2].

Various studies have been conducted for other mine waste disposal alternatives. Geopolymerization is a technology which can be used to recycle these mine waste/byproducts. The conversion or valorization of gold mill tailings to useable concrete can be done through this process. It is estimated that the majority of concrete demand in the US (75%) and Europe (100%) can be met by geopolymer concrete [3].

A geopolymer is a synthesized inorganic material that has a wide range of applications such as heat resistant ceramics, waste encapsulation and construction products. Aluminosilicates are key components of geopolymers [4]. The process makes use of an alkali activator like Sodium Hydroxide (NaOH) or Potassium Hydroxide (KOH) and water [5]. The alkali activator acts as supplementary binder similar to the properties of Ordinary Portland Cement (OPC) [6]. Compared to fiber-reinforced concrete which varies in tensile strength with the inclusion of fibers [7], the mechanical properties of geopolymers, including compressive strength, are influenced with varying silica and aluminum concentration. As a potential substitute to the use of OPC as binder, geopolymerization can minimize the associated negative environmental effects of OPC production including carbon dioxide emissions and high energy utilization [8]. Geopolymerization manufacturing produces less carbon dioxide and consumes less amount of energy making it more economically feasible and more environmentally sustainable.

Tailings are mostly used as aggregates in geopolymerization process. Mine tailings from gold mines generally contain significant amount of silica and alumina which makes it a suitable raw material. The total volume of mine tailings stored globally has reach billions of tons and increasing at a rate of up to 14 billion tons per year [9]. The application of this technology could help in reducing solid wastes from mines, lessen the related environmental impact and safety risk of tailings impoundment, and pave the way for producing environment friendly construction materials and hazardous waste encapsulation methods. Moreover, the disposal of mine tailings with its associated environmental effects including potential dam failure and landslide, dust generation, water contamination, and displacement of farming areas can be mitigated with the use of geopolymerization technology [10].

2. METHODOLOGY

2.1 Process Flow

The process flow of the study is shown in Figure 1. This describes the methodological framework used in geopolymerization and the various analysis methods utilized in this paper.

2.2 Collection of Gold Mill Tailings Sample

Gold mill tailings samples were obtained from Tailings Service Facility (TSF) 1, TSF 2 and TSF 3 of a gold mineral processing plant in Mindanao, Philippines. The mill utilizes Carbon-in-Pulp/Carbon-in-Leach (CIP/CIL) system for gold extraction. The ore for the mill feed comes from a vein-type gold deposit mined through sublevel stoping method. After processing, the tailings are pumped in the form of slurry and are impounded in the TSF. Over fifty percent of the initial ore mass is transformed into tailings during the mineral beneficiation process [11]. The collected samples taken from the TSF were mostly wet rock fines and were placed in a suitable container for transportation and subsequent laboratory analysis.

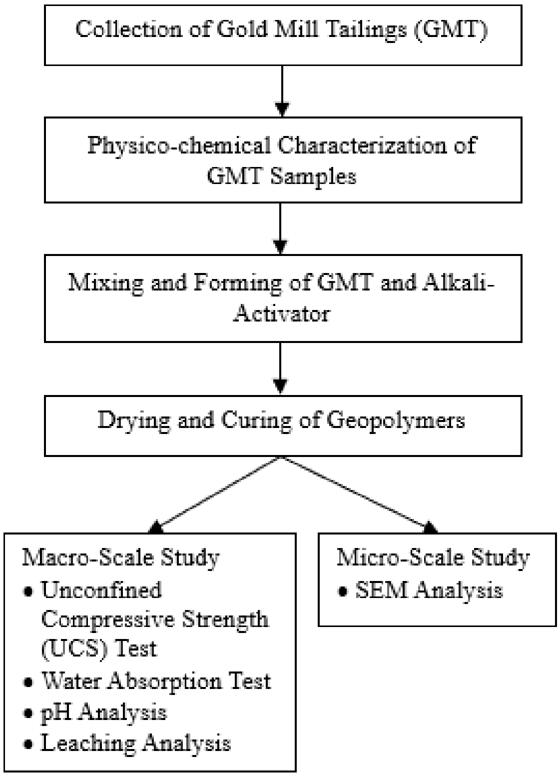

Fig. 1. Process Flow of the Study

2.3 Characterization of the Gold Mill Tailings Samples

Sample preparation for the collected gold mill tailings from TSF1, TSF2 and TSF3 was then performed including oven-drying prior to analysis. After drying, particle size analysis was done following the procedures set under the American Society for Testing and Materials (ASTM) D422-63 Standard Test Method for Particle-Size Analysis of Soil [12]. About 100 grams were collected from each TSF sample for elemental characterization using X-ray fluorescence (XRF) spectrometry.

2.4 Mixing and Forming of Gold Mill Tailings and Alkali Activator

Samples were subsequently prepared for the geopolymerization process. After drying, the samples were sieved passing 90 micrometers. Reagent-grade Sodium Hydroxide (NaOH) was used in the preparation of the alkali-activator. The NaOH was prepared in varying concentrations of 5M, 10M and 15M, respectively. Sodium Silicate (Na2SiO3) was also added in order to strengthen the binding of the Si and Al components. About 500 grams of GMT was weighed using the analytical balance and 100 mL of 5M NaOH solution was prepared. For the geopolymer samples, the ratio for NaOH and Na2SiO3 was 3:1, 100ml of 5M NaOH and 33mL of Na2SO4. The alkali activator and the mill tailings were mixed manually for 20 minutes until paste-like sample was obtained. After mixing, the sample was transferred to the 127 mm X 3166.92 mm^2 cylindrical molds. To eliminate bubbles, forming pressure was introduced and the sample was vibrated axially. Mixing and forming were also done for the other treatments with 10M NaOH and 15M NaOH, respectively. The different geopolymer samples prepared are presented in Table 1.

2.5 Drying and Curing

The cylindrical molds containing 5M NaOH, 10M NaOH, and 15M NaOH, were cured at different temperatures: 60°C, 90°C, 120°C, and 150°C, respectively, for a period of 24 hours. Generally, elevating the temperature accelerates the geopolymerization [12]. The samples were subsequently de-molded and the formed geopolymer specimens were cured additionally for seven (7) days at room temperature [13].

2.6 Macro-scale Study

The geopolymer specimens were sent to the materials testing laboratory for conducting compressional strength test.

Table 1. Treatments used for the geopolymer samples.

Sample Number	NaOH Conc. (M)	Curing Temp. (°C)	Curing Time (day)	No. of Samples
1A	5	60	7	3
1B	5	90	7	3
1C	5	120	7	3
1D	5	150	7	3
2A	10	60	7	3
2B	10	90	7	3
2C	10	120	7	3
2D	10	150	7	3
3A	15	60	7	3
3B	15	90	7	3
3C	15	120	7	3
3D	15	150	7	3

The UCS was determined using a Universal Testing Machine (UTM). A loading rate of 0.5mm/min was applied throughout the tests conducted for all samples. The porosity of the material was tested through water absorption. Leaching test was then performed by soaking the samples in distilled water. Other testing methods available were also considered like the effect of aggressive chemical reagents [14]. However, a focus was given to the physical properties of the geopolymer specimens and related environmental performance. After every end of the soaking time (24 hours, 96 hours and 168 hours), the leachate was collected and were tested for heavy metal concentration and pH level. Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) was used in the analysis of heavy metal concentration in the leachate. The difference in the initial and final weight of the specimen after soaking was also obtained for all samples to determine the percentage water absorption of the geopolymer specimens [15].

2.7 Micro-Scale Study

Selected geopolymer with optimum compressive strength were subjected for Scanning Electron Microscope (SEM) imaging. The specimens were powdered and 1 gram of the powdered sample was obtained and sent to an analytical laboratory for SEM analysis. This is done to evaluate the morphological characteristics of the geopolymer specimens.

3. RESULTS AND DISCUSSION

3.1 X-Ray Fluorescence Result

Based on the results of the XRF analysis, GMT sample taken from TSF2 exhibited the highest Si and Al concentration, containing 23.84 ppm and 55.95 ppm respectively. The elemental characterization from the XRF result showed a greater amount of aluminum than Silicon having a Si/Al ratio of 0.459. This necessitates the addition of Sodium Silicate given the high aluminum content. On the other hand, trace concentrations of heavy metals from TSF 2 showing presence of zinc, strontium and molybdenum were observed from the elemental characterization using XRF. These heavy metals were subsequently tested for leaching and the environmental performance of the geopolymer was evaluated through leaching test to check if the leachate satisfactorily meets water quality standards for heavy metals set by the Philippine Environmental Management Bureau (EMB) [16], United States Environmental Protection Agency (EPA) [17] and World Health Organization (WHO) [18]. Particle size analysis determined the classification of the material belonging to sand with fines.

3.2 Effect of Curing Temperature and NaOH Concentration

Thirty-six (36) geopolymer specimens obtained from three replicates of geopolymers formed from three (3) varying NaOH concentrations (5M, 10M and 15M) subjected to four (4) different curing temperatures (60°C, 90°C, 120˚C and 150°C) were tested for unconfined compressive strength test after 7 days of curing. Figure 2 shows the graph of the compressional strengths of different curing temperature and NaOH concentration.

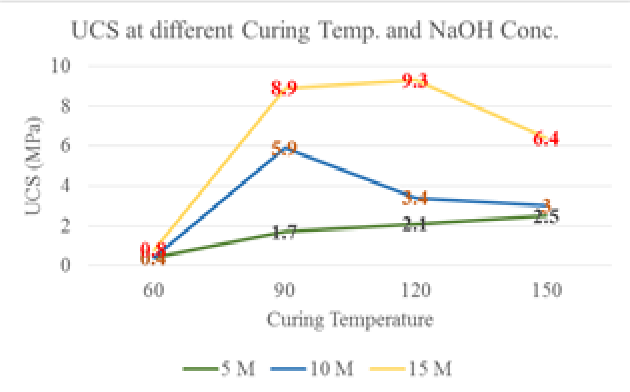

Fig. 2. UCS Result of the Geopolymer Specimens.

The mean UCS for the geopolymer samples with 5M NaOH concentration shows an increasing trend with increasing curing temperature from 60°C, 90°C, 120˚C and up to 150°C. For the samples derived from a higher NaOH concentration of 10M, an increase in UCS was observed between the products cured at 60°C and 90°C, but falls to a lower UCS for samples cured at 120˚C and 150°C, respectively. These observed results show a similar trend with the study of Ahmari, et. al. [19] describing that the effect of NaOH concentration on the UCS depends on the curing temperature. In general, the relationship between curing temperature and NaOH concentration is directly proportional – a higher alkaline concentration and lower source material reactivity will exhibit a higher optimum curing temperature. Conversely, a lower optimum curing temperature developed at lower NaOH concentration. At 15M NaOH, UCS increases with the curing temperature up to 120°C and then decreases at 150°C. At 60°C, the geopolymer specimens recorded low UCS even at higher NaOH concentration. Comparing UCS and NaOH concentration, there was an observed increase in UCS at 90°C from 1.7 MPa (at 5M) to 5.9 MPa (at 10M). This can be explained with the increase in dissolution of raw materials and acceleration of the geopolymer network formation at elevated pH levels [20]. Compared to 120°C, there is a slight increase from 5M to 10M with strengths at 2.1 MPa to 3.4 MPa respectively. The highest UCS was observed at 120°C with 15M NaOH concentration (9.3 MPa) relative to the other concentrations. There was a reduction in UCS at 150°C for samples with 10M and 15M NaOH concentration. An initial increase in UCS, followed by a decrease, with increasing curing temperature can be attributed to the NaOH content effect on the dissolution of Si and Al components including the subsequent polycondensation effects [21].

In this study, two-way ANOVA was used to determine if there were statistical differences for UCS in relation to NaOH concentration and different curing temperature levels. Two-way ANOVA is a statistical method used to compare the means of two independent variables to determine if there are statistically significant differences between them with respect to a dependent variable. The statistical tests were conducted at 95% confidence level. The test for equality of variances was done using SPSS software. The results showed that the p-value of 0.106 indicates that there was an equality of variances and also it indicates that the data are normally distributed as shown in Table 2.

Table 2. Result for the equality of variance test (Levene's Test)

F	df1	df2	p
1.823	11.000	24.000	0.106

In this case, two-way ANOVA was performed as a parametric statistical technique to test if there is a significant difference among the data as shown in Table 3.

Table 3. Two-way ANOVA analysis for UCS and Curing Temperature

Cases	Sum of Squares	df	Mean Square	F	p
NaOH Conc	132.394	2	66.197	4056.336	0.000
Curing Temp	134.076	3	44.692	2738.569	0.000
NaOH Conc* Curing Temp	54.357	6	9.060	555.139	0.000

Two-way ANOVA showed that there was a statistically significant difference ($p<0.05$) for UCS with varying NaOH concentrations. There was also a significant difference for UCS with respect to curing temperature. The difference of observed UCS with the interaction of both NaOH concentration and curing temperature was also deemed significant ($p<0.05$). The variation in UCS with respect to curing temperature can be explained by the underlying mechanism in geopolymerization. A study by Zhang, et.al. [21] showed that dissolution and polycondensation are the two main steps in geopolymerization. Increasing the curing temperature helps accelerate the dissolution of silica and alumina species until polycondensation. However, when the temperature is above a certain level, the fast polycondensation and rapid formation of geopolymer gel will hinder further dissolution of silica and alumina species which affects the strength adversely. A higher concentration of NaOH indicate that larger amounts of Na^+ cation was available to dissolve the silica and alumina and consequently thicker geopolymer was produced. The geopolymer binder serves as a link between the un-reacted or partially reacted particles and contributes directly to the strength of the geopolymer material. Summarizing the result, the derived geopolymer from the sample with 15M NaOH concentration cured at a temperature of 120˚C yielded the optimum unconfined compressive strength.

Based on the obtained UCS results, a comparison with ASTM-based requirements for possible applications were determined [19]. Based on ASTM C34-03, the geopolymer product can potentially be used as structural clay for the production of load bearing wall tile which meet UCS requirement ranging from 4.8 MPa (Grade LB/LBX) to 9.6 MPa (Grade LBX) [22]. Grade LB includes geopolymer material suitable for general use in masonry not exposed to frost action. On the other hand, Grade LBX meets suitability requirements for general use in masonry construction and adapted for use in masonry exposed to weathering.

3.3 Water Absorption Test

The water absorption test is related to the porosity of the geopolymer. This is an important factor to be considered since the pore structure influences the performance of geopolymers. Higher concentrations of activator solution can significantly impact the pore structure of the product. This is because of an increase in dissolution of the components including gold mill tailings as the concentration of alkali activator increases [23].

The results show that all specimens absorbed less than 7% water. The water absorption decreased with soaking time for all specimens as seen in Figure 3 below. After 24 hours of soaking time, the specimens have an average absorption of 6%. After 96 hours, the specimens have the average absorption of 5.29% and after 168 hours, the specimens have an average water absorption of 5.18%.

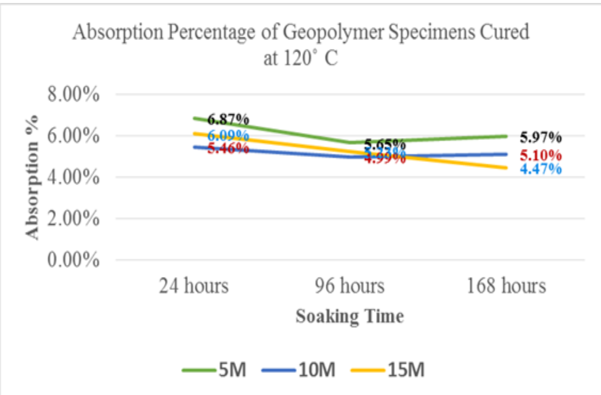

Fig. 3. Water Absorption Result of Samples Cured at 120°C

3.4 Leaching Analysis Result

Table 4 shows the level of concentration (mg/L) of the heavy metals (Sr, Mo, and Zn) present in the geopolymer sample leachate at different soaking time and the water quality standards for heavy metals. The heavy metals that were leached out from geopolymer samples soaked in distilled water increases minimally over time. The ICP-MS results were compared to the water quality standards such as EMB (Philippine Environmental Management Bureau), EPA (United States Environmental Protection Agency) and WHO (World Health Organization). The level of concentration for Sr of geopolymer specimen soaked at incremental time satisfactorily passed the EPA standard which is less than 4 mg/L. Also, the Mo concentrations from the geopolymer specimen's leachate at different soaking times have adequately passed the EPA and WHO standard (< 0.210 mg/L and 0.07 mg/L respectively. On the other hand, the Zn concentrations of geopolymer specimen soaked at different soaking time have passed except for geopolymer specimen soaked at 168 hours for EMB standard of Zn effluent (> 4 mg/L) but have tolerably passed the EPA and WHO standards which is less than 5 mg/L.

Table 4. Leachate concentrations vs water quality standards

Geopolymer Sample Soaking Time	Sr (mg/L)	Mn (mg/L)	Zn (mg/L)
24 hours	0.013	0.027	2.10
96 hours	0.031	0.027	3.00
168 hours	0.022	0.065	4.20
Water quality standards for heavy metals			
EMB	-	-	4.00
EPA	4.00	0.210	5.00
WHO	-	0.070	5.00

3.5 pH Analysis

The resulting pH were determined for the water samples obtained from the preceding tests. Based on the results, there was an observed increase in level of pH comparing initial and final levels. Since the gold mill tailings has a pH of 9.66 and there was an addition of basic solution of the experiment, an increase in pH was expected. In a concrete cement material, pH is an important parameter that was considered since lowering of the pH through carbonation can affect the compressional strength of the concrete material and high pH ranging from 11-13 indicates good quality of concrete material. The geopolymer specimens and cementitious material both exhibit a high pH level. The compressive strength of geopolymers exhibits a direct correlation with the activator solution's pH. Conversely, the setting time of the geopolymer mixture demonstrates an inverse relationship with increasing pH. At lower pH values, the geopolymer mix retains a viscous character, resembling traditional cement. Conversely, higher pH levels induce a transition towards a more fluid gel-like composition, characterized by reduced viscosity and enhanced workability. [24].

3.6 Microstructural Properties of Geopolymer Specimen

The results based on the UCS analysis reveals that the geopolymer specimen with the highest compressional strength resulted from the mixture of 15M NaOH for all curing temperatures (60°C, 90°C, 120°C and 150°C); Powdered samples from Sample No. 3A, 3B, 3C, and 3D were subjected for the SEM analysis. SEM images revealed morphological features of geopolymer specimens after subjected at different curing temperature (60°C, 90°C, 120°C and 150°C). Dense and aggregated images suggest the progress of geopolymerization reaction of samples activated with alkaline solution. The scanning electron images (SEM) of the gold mill tailings as raw material and geopolymer specimens produced before and after alkali activation are presented in Figure 4 and Figure 5. Geopolymers with a higher ratio of silicon to aluminum tend to have a more uniform binding material. This material might still contain some leftover particles from the original ingredients, along with a few tiny, scattered pores only a few micrometers wide [25]. Based on the results, samples cured at 90°C and 120°C were smooth and without much clusters as enough dissolution of Si and Al occurred as well as polycondensation. Samples from 60°C and 150°C, however, showed clusters due to very slow and very fast polycondensation, respectively. This creates a slow or rapid formation of geopolymer gel which hinders the dissolution of Si and Al. Additionally, at a curing temperature of 60°C, the slow rate of water evaporation resulted in slow formation of geopolymer structure. This can be related to the Energy Dispersive X-ray (EDX) analysis results which showed differences in atomic percentages for Ca, Si, Al, and Na as seen in Table 5. Results from the study of Wang, et. al. [20] showed that samples with lower Ca/Si and Na/Si ratios have weaker gel connectivity and aggregation. A higher pH concentration on the other hand promotes rapid dissolution that leads to encapsulation of unreacted material due to faster precipitation of silica-aluminate products which negatively affects the rate of dissolution of the remaining Si and Al components.

Table 5. EDX results for geopolymers with 15M NaOH subjected to different curing temperatures

Sample Curing Temp.	Elements (At%)			
	Ca	Si	Al	Na
60°C	0.66	7.55	7.38	1.54
90°C	0.34	7.41	7.53	1.15
120°C	0.36	7.7	6.99	1.60
150°C	0.50	6.62	8.57	0.20

4. CONCLUSION

This study was able to characterize the potential suitability of gold mill tailings from Mindanao, Philippines for the synthesis of geopolymers.

The geopolymer specimens prepared at 15M NaOH concentration showed significantly higher UCS than those with 10M and 5M NaOH concentrations. A higher NaOH concentration contributes to dissolution of more silica and alumina and formation of larger amount of geopolymer gels which consequently results in higher UCS.

Drying and curing temperature are important factors which affect the formation of geopolymers and thus the strength of geopolymer specimens. The compressive strength values obtained from the tests indicated that the curing conditions influenced the physical properties of geopolymer samples.

As the curing duration increased, the compressive strength increased. The UCS increased with increasing curing temperature up to a certain level and then decreases with extreme higher curing temperature (for 24 hours curing period). The geopolymer subjected to a curing temperature of 120°C exhibited the highest unconfined compressive strength.

The heavy metals (Sr, Mo and Zn) in the Gold MT-based geopolymer specimen's leachate have satisfactorily passed the water quality standards for heavy metals set by agencies such as EMB, EPA and WHO.

Geopolymer specimens cured at 120°C absorbed less amount of water compared to the other specimens formed at different curing temperatures. A higher curing temperature is necessary to accelerate the activation of the GMT in the geopolymerization process.

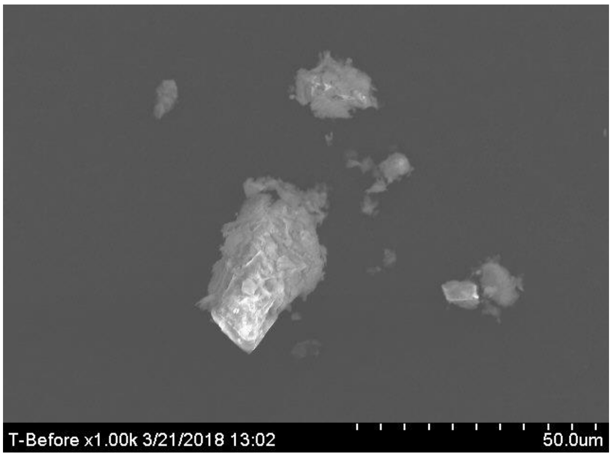

Fig. 4. SEM result of Pure Gold Mill Tailings.

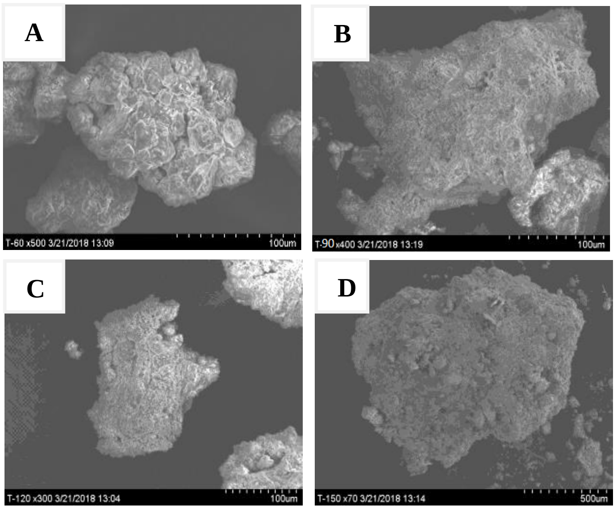

Fig. 5. Gold Mill Tailings after alkali activation at 15M for curing temperature of (A) 60°C, (B) 90°C, (C) 120˚C and (D) 150°C

Based on the result of experimentation, the researchers recommend the synthesized geopolymer as a possible material to be used as structural clay.

5. RECOMMENDATIONS

With a consideration of the results of this study, the following are recommended:

An actual assessment on the application of the geopolymer for construction applications including its use as load bearing wall tile and determine its long-term durability by comparing the dry and wet UCS of the geopolymer products before and after soaking;

For the curing time, the specimens could be cured in the oven to ensure a constant curing temperature (e.g. 30˚C - 40˚C) after inducing initial curing so that the heat will penetrate and more uniformly distributed in the specimen and come up a higher compressional strength;

In utilizing the GMT for geopolymerization, the presence of heavy metals in the GMT must be considered which may affect the properties of the GMT-derived geopolymers;

For future application of the study, further investigation on the application of the geopolymer material as refractory or heat insulating material in addition to its compressional strength and ability to immobilize heavy metals; and

Further study on the potential use of GMT-based geopolymer in the immobilization/encapsulation of the toxic wastes through geopolymerization may also be considered.

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