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UTILIZATION OF PHILIPPINE GOLD MINE TAILINGS AS A MATERIAL FOR GEOPOLYMERIZATION

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ABSTRACT: Mining minerals results in a waste material called mine tailings. In the Philippines, these waste materials are considered valueless and are just stored at tailing dams. The increasing demand for gold minerals in the country can cause an increase in the production of these waste materials. This can lead to a shortage of storage facilities. With this, this study used Philippine gold mine tailings as a material for geopolymerization. The process of geopolymerization produces a cementitious material with properties substantially comparable to those of conventional cement. It occurs after an aluminosilicate material reacts with an alkali hydroxide or silicate solution. In this study, gold mine tailings were mixed with the alkaline solution, or the combination of the 10-molar sodium hydroxide solution (10M NaOH) and the Water Glass Solution (WGS), to produce the geopolymer. A total of a four-mix proportion of WGS-to-10M NaOH and Alkaline Solution-to-Mine Tailings (AS-to-MT) was tested in this study. The compressive strength of each mix proportion was compared. Based on the results, the highest compressive strength has a mixed proportion of AS-to-MT ratio of 0.35 and the WGS-to-10M NaOH solution ratio of 2.5. X-Ray Diffraction was also performed to determine the chemical compound present in the gold mine tailings used. The results show that aluminum and silicon compounds are present in the material, hence, making the gold mine tailings viable for geopolymerization.

Keywords: Gold Mine Tailings, Geopolymerization, Geopolymer Binder, Waste Material

1. INTRODUCTION

The increase in mining of minerals such as gold and copper resulted in the growth of waste materials such as mine tailings. These materials are considered valueless and are always stored in tailing dams. Shortage in storage facilities can be a problem due to the increase in mine tailings. As a result, several studies proposed the application of mine tailings for embankment material [1-3]. Many studies have been done locally for sustainable materials [4-35] and another promising application is by utilizing mine tailings as a material for geopolymerization which can result in a geopolymer binder. Geopolymerization is a process that produces a cementitious material with properties substantially comparable to those of conventional cement. In a study done by Hardjito, Wallah, Sumajouw, and Rangan (2004), they used geopolymer paste instead of cement paste but applied the usual methods of creating concrete in manufacturing a type of geopolymer concrete [36]. Based on their results, the geopolymer paste works similarly to a normal cement paste as it binds all the other ingredients together, such as the coarse and fine aggregates, to form the geopolymer concrete. Several researchers already claimed that mine tailings can be used as a material geopolymerization. Rösner (1999) investigated the geotechnical, mineralogical, and geochemical parameters of gold mine tailings found in South Africa was investigated in the research [37]. A semi-quantitative X-Ray Diffraction (XRD) method was performed. This method is a common technique that identifies the phase and structural characteristics of crystalline solids [38]. Based on the result of the analyses of Rösner, gold mine tailings have high quartz content with an average of 78%. After thorough investigations, it was also revealed that the major chemical element of gold mine tailings found in the East Rand area was silica or SiO_2, with an average of 81.6%. Ahmari, J. Zhang, and L. Zhang (2011) also investigated the feasibility of fly ash-modified mine tailings-based geopolymer concrete as an alternative construction material [30]. They discovered that mine tailings have a significantly high Silicon to Aluminum ratio (Si-to-Al) and can be adjusted by using fly ash (ASTM Class F). In the study, the chemical composition of mine tailings and fly ash was compared. It was observed that mine tailings have more SiO_2 content and have less Al_2O_3 content than fly ash, thus, resulting in a greater Si-to-Al ratio. More of their test results revealed that the properties of the mine tailings-based geopolymers were intensively affected by the Si-to-Al ratio and alkalinity of the material, as well as its curing time, especially during the first week. Nevertheless, Ahmari et al. (2011) concluded that mine tailings may be used as the base material in creating geopolymer concrete [39]. Moreover, Ahmari and Zhang (2012) tested the feasibility of environment-friendly bricks made through the geopolymerization of copper mine tailings [40]. Unlike the conventional method of brick production, but similar to the production of geopolymer concrete, these environment-friendly bricks were produced by combining the tailings sample with an alkali solution. These bricks were tested for water absorption and unconfined compression tests. The results showed that the use of copper mine tailings in the production of environment-friendly bricks using geopolymerization technology met the ASTM requirements. It is therefore the objective of this study to investigate the potential of the gold mine tailings from the Philippines as a material for geopolymer. Specifically, establish the most appropriate ratio of the alkaline reagent and gold mine tailings to be able to create a geopolymer binder.

2. RESEARCH SIGNIFICANCE

One of the issues in the mining industry is the improper disposal of mine tailings. To address this concern, there is a need to investigate the possible uses of these waste materials. Specifically, its use as a sustainable alternative to binders similar to cement. In this study, the potential of gold mine tailings as material for geopolymerization was utilized which resulted in a geopolymer binder. The established geopolymer binder can be beneficial to reduce the carbon footprint from the production of cement which has a negative impact on the environment. Also, the developed material can lessen the construction of tailing dams or storage facilities in mining sites.

3. GEOPOLYMERIZATION

Geopolymerization occurs after an aluminosilicate material reacts with an alkali hydroxide or silicate solution [41-42]. Aluminosilicate materials are minerals composed of compounds with aluminum and silicon; examples of this type of material include fly ash and mine tailings [43]. Likewise, examples of an alkali hydroxide or silicate solution would be sodium hydroxide (NaOH), and sodium silicate (Na_2SiO_3) solutions, respectively. The mixture of the alkali hydroxide and silicate solutions is collectively known as the alkaline solution or alkaline reagent. The chemical reaction between the wet (alkaline solution) and dry (aluminosilicate material) constituents causes an amorphous gel to harden and condense into a geopolymer binder.

4. GOLD MINE TAILINGS

The gold mine tailings sample was from a mining site at the Northern Province of Luzon, Philippines. The index properties of the sample were first established based in ASTM standards. The results are tabulated in Table 1.

Table 1 Index Properties of Gold Mine Tailings

Material	Properties	Values
Gold Mine Tailings	Specific Gravity	2.63
	Liquid Limit	27
	Plastic Limit	22
	Plasticity Index	3
	Linear Shrinkage	3
	Uniformity Coefficient	2.47
	Coefficient of Curvature	1.93
	Soil Classification	Uniformly graded ML

X-Ray Diffraction (XRD) analysis was performed to identify the presence of crystalline compounds in the mine tailings sample. The result is shown in Fig. 1. The peaks on the XRD graph suggest that the material is crystalline, while if the lines are somehow flat, the material is amorphous. Based on Fig. 1, the gold mine tailings contain crystalline compounds. Additionally, illite, kaolinite, montmorillonite, and quartz were present in the mine tailings sample. Quartz has a chemical composition of SiO_2 which shows the presence of silicon compounds in the sample. However, no aluminum compound of Al_2O_3 was identified; thus, the aluminum may be amorphous which is desirable for geopolymerization. The XRD results of the mine tailings used in this study show that aluminum and silicon compounds are present in the material, hence, making the gold mine tailings viable for geopolymerization.

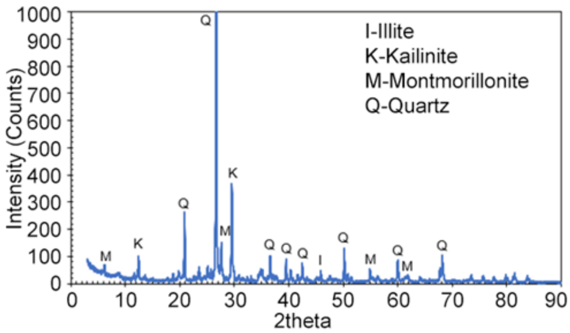

Fig. 1 XRD Results of Mine Tailings

To further investigate if the material is amorphous X-Ray Fluorescence (XRF) was also performed. It is a non-destructive procedure that performs the quantitative analysis of elemental composition in which the amount of aluminum (Al) and silicon (Si) present in the mine tailings sample can be found [44]. This was done by displacing the electrons from their atomic positions through a discharge of energy from the apparatus. This energy is identified by the detector to categorize the material by element [45]. The XRF analysis identified the major elemental components of the mine tailings. Based on the results as shown in Table 2, the top two major elemental components found in the mine tailings sample are silicon (Si) with 61.848% and aluminum (Al) with 12.867%. Since the XRF shows the presence of aluminum, it confirms with the XRD result that this aluminum oxide is amorphous.

Table 2 Chemical Composition from XRF analysis

Elements		Percentage Quantity (%)
Al_2O_3	Aluminum oxide	12.867
As_2O_3	Arsenic trioxide	0.064
CaO	Calcium oxide	5.731
Cr_2O_3	Chromium(III) oxide	0.008
CuO	Copper(II) oxide	0.009
Fe_2O_3	Iron(III) oxide	5.762
Ir_2O_3	Iridium sesquioxide	0.003
K_2O	Potassium oxide	4.546
MgO	Magnesium oxide	1.752
MnO	Manganese(II) oxide	0.357
PbO	Lead(II) oxide	0.011
RbO	Rubidium oxide	0.017
SO_3	Sulfur trioxide	6.335
SiO_2	Silicon dioxide	61.848
SrO	Strontium oxide	0.015
TiO_2	Titanium dioxide	0.587
V_2O_5	Vanadium(V) oxide	0.033
ZnO	Zinc oxide	0.047
ZrO_2	Zirconium(IV) oxide	0.011

5. GEOPOLYMERIZATION OF GOLD MINE TAILINGS

The gold mine tailings were first stored in a dry condition before the preliminary experiments and the production of the geopolymer binder. The sample was dried in an oven for 24 hours then placed in a plastic container to prevent the absorption of moisture into the atmosphere. Compacted lumps of mine tailings that resulted after the oven-drying were crushed using the mechanical crusher to ensure uniformity of the particle sizes. The texture of the sample after crushing was soft and fine, with physical properties like that of cement powder.

4.1 Gold Mine Tailings and Alkaline Reagent

An alkali solution was needed to initiate the geopolymerization process of gold mine tailings. A combination of the water glass solution (WGS) and the 10M NaOH was established as the alkaline reagent to create a geopolymer. For the geopolymerization of the gold mine tailings, an alkaline solution-to-mine tailings (AS-to-MT) ratio is also needed to ensure that the geopolymer can develop a capacity to hold compressive stress. The WGS used in this study was composed of 14.65% of Na_2O, 34.13% of SiO_2, and 51.22% of water. The 10M NaOH solution was made from 97%-98% pure sodium hydroxide pellets that were dissolved in distilled water. The ratio of the alkaline solution-to-aluminosilicate material affects the strength of the geopolymer. As the ratio increases, the strength also increases [46]. Based on previous researches, to form a geopolymer the WGS-to-10M NaOH ratio can be 1.0 or 2.5 [46-47]. Hence, the WGS-to-10M NaOH ratios that were used in this study were 1.00 and 2.50 to ensure that geopolymerization can occur. The alkaline reagent produced by combining the 10M NaOH and WGS is recommended to be prepared at least 24 hours before use [48-50]. For the alkaline solution-to-mine tailings (AS-to-MT) ratio, a value of 0.35 and 0.45 was implemented. These ratios were based on previous researches from Aleem and Arumairaj (2012), Patankar, Ghugal, and Jamkar (2014a), and Patankar, Ghugal, and Jamkar (2014b) where they used 0.35 as the AS-to-MT ratio; while Patankar, Ghugal, and Jamkar (2014b) used 0.35, 0.40 and 0.45 as their AS-to-MT ratio, and the results of their study showed that the ratio 0.45 resulted to the samples with the greatest compressive strengths [46, 47, 51].

4.2 Preliminary Experiment

A preliminary procedure was made to determine whether the gold mine tailings would react with the alkaline reagent to form a geopolymer and hence if it would harden as shown in Fig. 2. This is due to the fact that, although some studies have shown to use copper mine tailings to successfully produce geopolymer bricks, it cannot be guaranteed that the same reaction would happen to the gold mine tailings used in this study considering it was obtained from a different source and could possibly be of a different composition, especially the level of aluminum and silica content, both of which are vital to the geopolymerization of the material [40].

Five 50x50mm cubes of geopolymer binders were produced as seen in Fig. 2. The geopolymer binder was produced through the combination of the aluminosilicate material and the alkaline solution. This is analogous to cement paste or the product of the combination of water and cement powder. The geopolymer binder specimens were hand mixed to ensure a uniform consistency of the mixture since the use of a mechanical mixer did not effectively mix the ingredients well. The addition of water was limited to observe medium workability. As a result, four trial mixtures were successfully produced from the combination of the WGS-to-10M NaOH and AS/MT ratios. A minimum of ten 50-mm mortar cubes for each trial mix was made; therefore, an estimated total volume of $1.25x10^{-3}$ m^3 of geopolymer binder per trial was produced. The summary of the materials needed is tabulated in Table 3.

These specimens were heat cured for a temperature of 60°C for 24 hours after casting in cube molds for at least 5 days since the specimens were still moist after few days of production. The total number of created specimens for geopolymer binders was 42, wherein at least 10 cube samples were made for each ratio as shown in Table 4. The cubes were tested in an unconfined compression test to determine the compressive strength of the geopolymer binder. Five samples for each ratio were first tested on the 7th day, while the remaining samples were tested on the 28th day.

Fig. 2 Preliminary of Geopolymer Binder Samples

Table 3 Mix Proportions of Geopolymer Binder

Materials	Weight Proportions (kg)			
Trial	1a	1b	2a	2b
WGS-to-10M NaOH	1.00		2.50	
AS / MT	0.35	0.45	0.35	0.45

6. RESULTS AND DISCUSSION

The WGS-to-10M NaOH ratios used in this study were 1.00 and 2.50. The WGS, also known as sodium silicate solution, used in this study was composed of 14.65% of Na_2O, 34.13% of SiO_2, and 51.22% of water. Meanwhile, the 10-molar concentration of the NaOH was produced by mixing 400 grams of NaOH micro-pearls per 1L of the solution. Furthermore, the AS-to-MT ratios used were 0.35 and 0.45. The alkaline solution was mixed a day prior to the mixture of the wet and dry ingredients [49]. Consequently, four trial mixtures were produced based on the combination of the given ratios. Ten 50-mm mortar cubes of each trial were produced whose mix proportions may be found in Table 5.

Table 4 Design of Experiment

Materials	Weight Proportions (kg)			
WGS-to-10M NaOH Ratio	1		2.5	
AS-to-MT Ratio	0.35	0.45	0.35	0.45
Strength Test	Unconfined Compression Test			
No. of Samples	10	10	10	10

Fig. 3 Testing of Geopolymer Binder

The specimens were heat cured at a temperature of 60°C for about 24 hours after casting and setting in the cube molds for 5 days. Based on the results of the unconfined compression test, Binder 2a possessed the highest unconfined compression strength both on the 7th and 28th days, with a value of 1.541 MPa, and 1.636 MPa, respectively. These results may be seen in Table 6 and Fig. 4.

Table 5 Mine Tailings-Based Geopolymer Binder Mix

Sample ID	MT (g)	AS (g)	H_2O (g)	AS-to-MT	WGS-to-10M NaOH
Binder 1a	2834	992.5	114.8	0.35	1
Binder 1b	2224	1000	96.6	0.45	1
Binder 2a	2224	778.2	90	0.35	2.5
Binder 2b	2444	1100	106.3	0.45	2.5

Table 6 Summary of Unconfined Compression Test Results

Sample ID	Unconfined Compression Strength, MPa			
	Day 7	Std. Dev.	Day 28	Std. Dev.
Binder 1a	1.225	0.291	1.626	0.380
Binder 1b	0.831	0.413	1.362	0.137
Binder 2a	1.541	0.207	1.636	0.304
Binder 2b	1.155	0.478	1.237	0.201

Binder 2a has an AS-to-MT ratio of 0.35, and a WGS-to-10M NaOH ratio of 2.5. Comparing the two ratios for AS-to-MT of 0.35 and 0.45 shows that the amount of alkaline solution, that is 35% of the amount of mine tailings, is enough to produce a binder. However, increasing the amount of solution to 45% decreases the strength of the binder. WGS, which is used as a binder or adhesive, is required to have a greater amount than the 10M NaOH. This is to ensure the adhesivity of the binder and its ability to set. On the other hand, 10M NaOH is also used to increase the strength of the binder, but excessive 10M NaOH may result in the occurrence of efflorescence as observed in the preliminary investigation for the production of geopolymer binder (Fig. 4). This is the result of some of the sodium hydroxide solution not reacting completely with the mine tailings.

A study by Zhang et al. (2014) suggests that the presence of efflorescence in geopolymers could pose negative effects to its compressive strength development especially when exposed to humid environments [52]. The test results of their study show that the geopolymer binder samples subjected to the occurrence of efflorescence have lower strength values compared to the samples without efflorescence products. Moreover, Zhang et al. (2014) explained that the specimens produced with a higher amount of additional water might have a higher porosity which may lead to the formation of higher efflorescence products within the pores, which then translates to higher inner stress [52]. They proposed that this eventuality could account for the low strength of the geopolymers with efflorescence. The load-deformation curve of the mine tailings-based geopolymer binder may be seen in Fig. 5. This shows the relationship between the applied axial load and the corresponding deformation on the 7th day of binders 1a, 1b, 2a, and 2b. Binders 1a, 1b, and 2b show steeper slopes than binder 2a. This means that there is gradual deformation as the axial load is applied. However, binder 2a is more flexible in handling the axial load and can still resist greater magnitudes. Binder 2a also produced the greatest unconfined compressive strength at the age of 7 days as seen in Fig. 6. For the 28th-day unconfined compression test found in Fig. 7, the load-deformation curve has a steeper slope than that of the 7th day. Binders 1b and 2b show a lesser value of maximum applied axial load thus lesser unconfined compressive strengths. These two mixes, binders 1b and 2b, with the AS-to-MT ratio of 0.45 showed that too much alkaline solution may result in a more workable mixture, but binder specimens with lesser compressive strength. The amount of alkaline solution must be adequate to produce the geopolymerization. Binders 1a and 2a presented lesser steep graphs, but, binder 2a resisted a greater load. Thus, the ratio combination that will be used for the mine tailings-based geopolymer concrete is binder 2a, which consists of 0.35 as AS-to-MT ratio and 2.5 as WGS-to-10M NaOH. The summary of these compression strengths is found in Table 4 with its corresponding standard deviation.

Fig. 4 Occurrence of Efflorescence

7. CONCLUSION

Gold mine tailings were used to produce a geopolymer. Preliminary investigations on the mine tailings sample revealed that the gold mine tailings obtained was a fine-grained, poorly-graded soil having particles with roughly the same sizes or shapes, and may then be considered as uniformly graded inorganic silt with low plasticity (ML) with a specific gravity of 2.63.

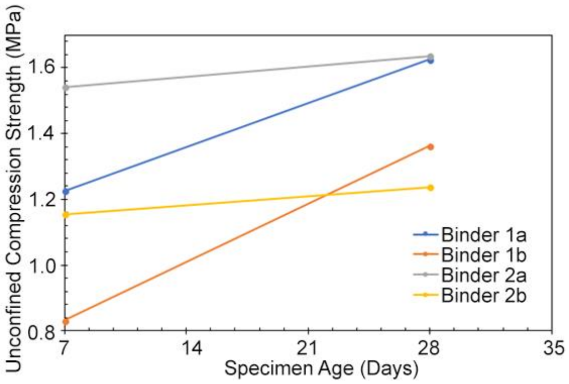

Fig. 5 Relationship between Unconfined Compression Strength and Specimen Age

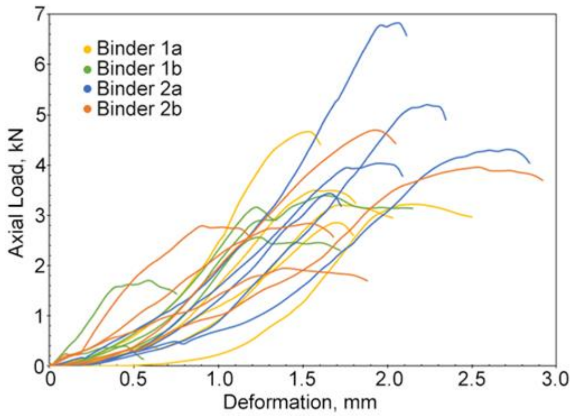

Fig. 6 7th Day Compressive Test

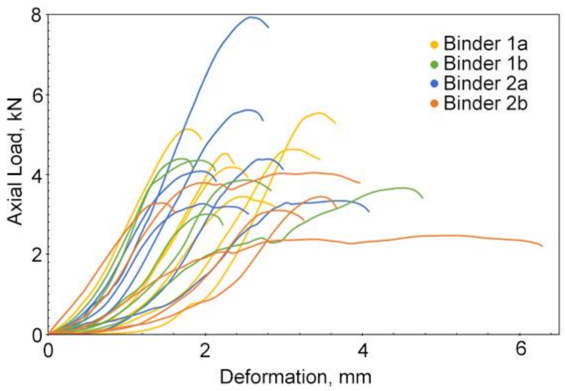

Fig. 7 28th Day Compressive Test

The average liquid limit of the gold mine tailings was found to be 27, while the average plasticity index was 3; on another hand, the plastic limit and linear shrinkage were found to be 22 and 3, respectively. Furthermore, the XRD analysis showed that the gold mine tailings sample is viable aluminosilicate material for geopolymerization since it has aluminum and silicon compounds in its crystalline forms. This was further verified when the XRF analysis was conducted. This also revealed that the two major elements present in the mine tailings specimen are silicon (Si) with a percentage quantity of 61.85% and aluminum (Al) with 12.87%. The results show that aluminum and silicon compounds are present in the material, hence, making the gold mine tailings viable for geopolymerization. For the production of the geopolymer binder, four proportion combinations of WGS-to-10M NaOH and AS-to-MT ratio were used to create ten 50mm x 50mm cubes for each ratio combination. After 5 days of setting in the molds, the cubes underwent heat curing for 24 hours using an oven temperature of 60^{o}C. An unconfined compression test was conducted to determine the compressive strength of the geopolymer cubes. The highest compressive strength obtained among the tested binders had a WGS-to-10M NaOH ratio of 2.5 and an AS/MT ratio of 0.35, with a value of 1.541 MPa on the 7th day, and 1.636 MPa on the 28th day. Based on the results, gold mine tailings have the potential to be used as a material for geopolymerization which can result in a geopolymer binder. For further studies, the binder can be mixed with concrete. The mixture is limited to non-load-bearing structures based on its compressive strength.

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