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Thermal treatment and utilization of Al-rich waste in high calcium fly ash geopolymeric materials

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Abstract: The Al-rich waste with aluminium and hydrocarbon as the major contaminant is generated at the wastewater treatment unit of a polymer processing plant. In this research, the heat treatment of this Al-rich waste and its use to adjust the silica/alumina ratio of the high calcium fly ash geopolymer were studied. To recycle the raw Al-rich waste, the waste was dried at 110°C and calcined at 400 to 1000°C. Mineralogical analyses were conducted using X-ray diffraction (XRD) to study the phase change. The increase in calcination temperature to 600, 800, and 1000°C resulted in the phase transformation. The more active alumina phase of active θ-Al_2O_3 was obtained with the increase in calcination temperature. The calcined Al-rich waste was then used as an additive to the fly ash geopolymer by mixing with high calcium fly ash, water glass, 10 M sodium hydroxide (NaOH), and sand. Test results indicated that the calcined Al-rich waste could be used as an aluminium source to adjust the silica/alumina ratio and the strength of geopolymeric materials. The fly ash geopolymer mortar with 2.5wt% of the Al-rich waste calcined at 1000°C possessed the 7-d compressive strength of 34.2 MPa.

Keywords: waste utilization; calcination; geopolymers; fly ash

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1. Introduction

The Al-rich waste is generated at the wastewater treatment unit of a polymer processing plant. In this process, aluminium chloride ($AlCl_3$) is used as a catalyst for the polymerization of hydrocarbon resin [1]. Neutralization with sodium hydroxide (NaOH) solution is performed on the polymer product. The resulting sewage contains aluminium hydroxide ($Al(OH)_3$) gel and is discharged into the wastewater treatment unit. The sewage is then treated with sulphuric acid (H_2SO_4) solution in order to adjust the pH value and to coagulate the Al-rich waste. The solid waste is finally separated from the wastewater and disposed off in landfill. The leaching of aluminium ions and other contaminants into groundwater is the main concern. The flowchart of the waste generation and treatment process is shown in Fig. 1.

The aluminium waste contains many forms of aluminium compounds. $Al(OH)_3$ is the major compound, and aluminium and sodium sulphate are the minor ones. The high annual output of Al-rich sludge leads to the problems of waste management and increases the treatment costs. A number of researches [2-4] have been conducted to utilize this Al-rich industrial sludge as a filler in cement and ceramic products. Due to the high water content up to 85wt% and the impurity of this sludge, treatment is needed to improve its properties before it can be effectively used. After the filter pressing

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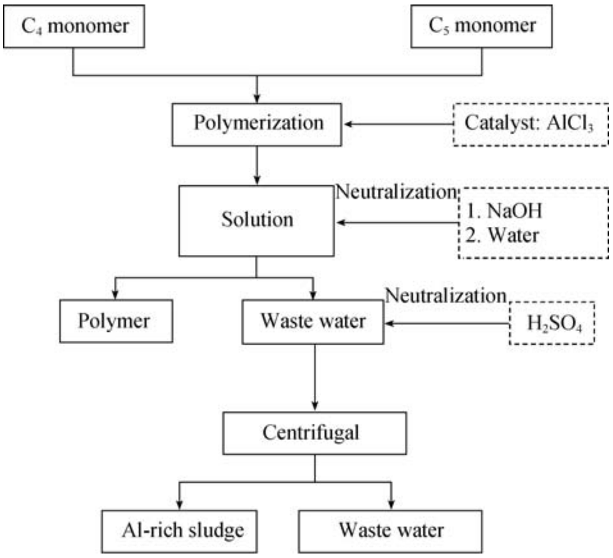

Fig. 1. Flow diagram of waste generation.

process, this sludge is basically the colloidal aluminium hydroxides with some soluble salts such as aluminium sulphate [2].

The high water content and the gel-like consistency of the sludge make it difficult to form the homogeneous mixture with other raw materials. This is the main reason for an increase in the drying shrinkage of ceramic bodies obtained from this sludge [3]. The pre-drying of Al-rich sludge at 100°C gives a strong agglomerated structure and also makes it difficult to obtain the proper mixing with other materials. Due to its high alumina content, calcination provides the opportunity for the application of this waste in the alumina-based materials such as mullite or mullite-alumina products.

In Thailand, the transportation cost for disposal is rather high at US$ 25-30/t and keeps rising. The amount of this waste increases every year and thus prompts the industry to find ways to utilize it. Therefore, this work aims to reuse the Al-rich waste by improving its properties with calcination. The calcined product should then be ready as an aluminium source material to produce the aluminosilicate binder or the geopolymeric material using low temperature curing [5-7]. Geopolymeric material is the aluminosilicate compound prepared at 40-90°C [6-7]. This material possesses high early compressive strength compared to the normal concrete at the same curing age.

Fly ash is widely used as an aluminosilicate source material for making geopolymer. It has been shown that high calcium fly ash is a suitable source material for making geopolymer [7-8]. The properties of the fly ash geopolymer can be further adjusted with the addition of chemical additives and mineral compounds such as rice husk ash [9-10]. In many instances, the source material contains low aluminium content, and the strength and other properties of the geopolymeric materials are impaired [11]. The utilization of the Al waste as an additional source material to increase the aluminium content of the geopolymer was therefore proposed.

2. Materials and methods

2.1. Materials

The Al-rich waste was obtained from a local polymer plant in Thailand. This waste contained high water content (ca. 80wt%); therefore, it was oven dried at 110±5°C for 24 h and the whitish yellow solid was obtained. The dry sample was then ground for 1 h using Rapid Mill 1105 (Compound Clay Ltd., Thailand) at 500 r/min. The median particle size of 15 μm measured by the particle size analyzer (Malvern Mastersizer S) was obtained. Grinding apparently reduced the grain size and promoted an amorphization, resulting in the enhancement of reactivity and structural changes of materials [12-13]. The chemical composition of this waste is presented in Table 1. The loss on ignition (LOI) was determined according to ASTM D7348. Differential scanning calorimetry (DSC) analysis was performed on the ground sample and on the commercial $Al(OH)_3$ sample using Pyris Diamond DSC (Perkin-Elmer) at a heating rate of 10°C/min, nitrogen purging at 20.0 mL/min, and the operation range from 50 to 400°C.

For the preparation of geopolymeric materials, the high calcium fly ash from the pulverized coal combustion process (PCC), sodium silicate solution (9wt% Na_2O, 30wt% SiO_2,

Table 1. Chemical composition of as-received Al-rich waste and fly ash **wt%**

Composition	Al-rich waste	Fly ash
$Al(OH)_3$	82.5	—
Na_2SO_4	4.7	—
NaCl	4.6	—
SiO_2	—	39.4
Al_2O_3	—	21.6
CaO	—	12.0
Fe_3O_4	—	11.4
SO_3	—	2.8
MgO	—	10.9
K_2O	—	2.1
LOI	8.2	0.8

and 61wt% H_2O), 10 M NaOH, and graded river sand with the fineness modulus of 2.8 were used [8].

2.2. Calcination of the ground sample

Since the Al-rich waste could not be used directly because of the high organic contents as indicated by the high LOI of 8.2%, the calcination was therefore employed. The LOI indicated the amount of organic compounds decomposed at 400-500°C since this waste was from a polymer plant. The ground samples were subjected to the thermal treatment at 400, 600, 800, and 1000°C for 1 h. The preliminary test indicated that 1-h calcination was sufficient. Although the cost of preparing this waste could be slightly higher than the normally used raw material, the method provides a new recycling opportunity for this kind of sludge. Mineralogical analysis was conducted using X-ray diffraction (XRD, PANalytical/Expert). Functional characterization of the calcined wastes was also performed by Fourier transform-infrared spectroscopy (FT-IR, Perkin-Elmer System 2000).

2.3. Preparation of geopolymeric materials

The Al-fly ash geopolymeric material was prepared by mixing the aluminosilicate source materials with sodium silicate solution (water glass), 10 M NaOH, and sand. The source materials were the blend of the calcined wastes (at 800 and 1000°C) and fly ash. The fly ash was replaced with 2.5wt% and 5.0wt% of the calcined wastes. The liquid was the mixture of water glass and 10 M NaOH with the mass ratio of 2:1. Powdery blend was then mixed with the liquid at the mass ratio of 60:40. Sand was used as a filler with a sand-to-powdery blend ratio of 2.0 [7-8]. The original pre-dried waste was also used to prepare the geopolymeric materials for comparison. Mix proportions are tabulated in Table 2.

Table 2. Mix proportion of geopolymeric materials g

Sample	Fly ash	Al waste	Water glass	10 M NaOH	Sand	Remark
Control	60	—	27	13	120	Control sample
2.5Al (PD)	58.5	1.5	27	13	120	Pre-dried waste
5Al (PD)	57	3	27	13	120	
2.5Al (800)	58.5	1.5	27	13	120	Calcined waste (800°C)
5Al (800)	57	3	27	13	120	
2.5Al (1000)	58.5	1.5	27	13	120	Calcined waste (1000°C)
5Al (1000)	57	3	27	13	120	

The pastes were cast into 5 cm cube acrylic plastic molds and cured at 60°C for 24 h to activate the reaction [7-8]. Specimens were then demolded and left in the controlled room of 23±2°C and with the relative humidity (RH) of 50%. The compressive strengths of specimens were measured at the age of 7 d using the compression testing machine (CB-10M, CBN Co.). The specimen was placed between the two steel plates and compressed at a uniform loading rate in accordance with the ASTM C109. Compressive strength was calculated by dividing the maximum load by the cross-sectional area of the specimen. The reported results are the averages of three samples.

The microstructural study of the hardened pastes was performed using scanning electron microscopy (SEM, Leo 1455VP SEM, Zeiss). Nonconductive specimens were coated with gold before the examination with the microscope.

3. Results and discussion

3.1. Characterization of pre-dried waste

DSC was used to study the thermal transition of the commercial pure $Al(OH)_3$ and the ground pre-dried waste. This technique observes the exothermic and endothermic events such as glass transition temperature (T_g), crystallization temperature (T_c), and melting temperature (T_m). The temperature range of the investigation was from 50 to 400°C. The results of the DSC-thermogravimetric (TG) analysis (endothermic up) are presented in Fig. 2. The commercial $Al(OH)_3$ exhibits T_c of 240°C. At this temperature, the crystalline arrangement occurred as indicated by the drop in the heat flow. The material gave off heat when it crystallized in the exothermic transition. Endothermic transition (T_g) was also found at 345°C as heat was absorbed for phase transformation and the broad peak appeared. For the ground pre-dried waste, only the crystallization peak T_c was found at 165°C. These endothermic peaks indicated the transition of gibbsite ($Al(OH)_3$) to boehmite (AlO·OH) at ~250°C and the transition of boehmite to γ-alumina (γ-Al_2O_3) at ~350°C [14-15]. The change in location of T_c was due to contaminants in the waste. The contaminants were the unreacted hydrocarbons, polymer residue, and sodium sulphate from the polymerization and neutralization processes. These impurities of

Al-rich sludge hindered the homogeneous formation; therefore, the removal was needed to increase its efficiency. Calcination was consequently applied to this waste.

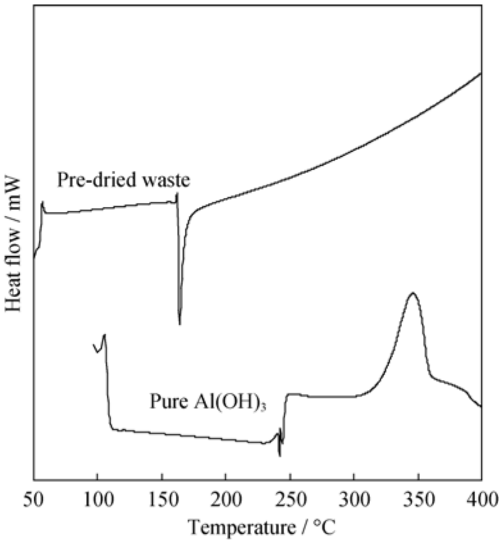

Fig. 2. DSC-TG curves of commercial pure Al(OH)$_3$ and pre-dried waste.

3.2. Characterization of the calcined waste

3.2.1. XRD analysis

The XRD results of the waste after calcination are shown in Fig. 3. The calcination duration and the temperature are significant factors for the phase change and removal of impurities. The calcination resulted in the amorphous phase with some crystalline phases [16]. The compounds detected in the pre-dried sample were gibbsite ($Al(OH)_3$), sodium sulphate ($NaSO_4$), and sodium chloride (NaCl) as shown in Table 1. At the calcination temperature of 400°C, γ-Al_2O_3 was formed. For the calcination temperatures of 600, 800, and 1000°C, two distinct peaks at 37° and 46° indicated the incipient phase of active θ-Al_2O_3. The glassy phase was increased due to the increase in calcination temperature as indicated by the appearance of broad peaks. The θ-Al_2O_3 was formed in large quantity with some impurities of NaCl and Na_2SO_4 at 600 and 800°C. The decomposition temperatures of NaCl and Na_2SO_4 were 780 and 880°C, respectively. At 1000°C, NaCl disappeared and the sample contained a higher amount of the amorphous phase. The calcination of gibbsite produced a series of transitions of alumina as shown in the following equation [14, 17]:

$$\text{Gibbsite} \xrightarrow{200^\circ C} \gamma\text{-boehmite} \xrightarrow{400^\circ C}$$

$$\gamma\text{-}Al_2O_3 \xrightarrow{750^\circ C} \delta \xrightarrow{900^\circ C} \theta \xrightarrow{1200^\circ C} \alpha\text{-}Al_2O_3.$$

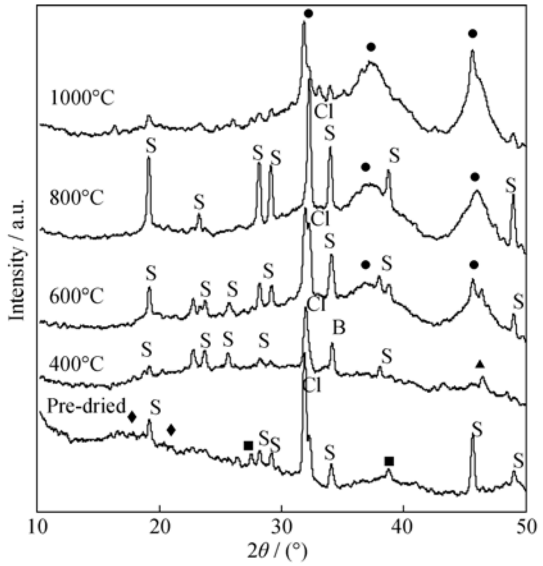

Fig. 3. XRD patterns of the Al-rich waste. Cl—NaCl; S—Na_2SO_4; ♦—gibbsite; ■—boehmite; ▲—γ-Al_2O_3; ●—θ-Al_2O_3.

All transition phases are stable at room temperature. It has been pointed out that the transformation temperatures are somewhat variable depending on the impurities of the starting materials [3, 17]. Practically, various substances can be produced with mixed crystalline forms of $Al(OH)_3$, AlO·OH, and aluminium oxide (Al_2O_3) with water molecules. These are known as hydrated alumina. Calcination of the hydrated hydroxide resulted in the loss of water and produces various kinds of active alumina. They differ in porosity, number of remaining –OH groups, and particle size. They can be used as catalyst supports and in chromatography.

3.2.2. IR spectra

Fig. 4 shows the FT-IR spectra of the pre-dried and the calcined samples at 800 and 1000°C. Spectrum characterization of the products is based on the analysis of the absorption bands, which are due to the vibrations of –OH groups in the ranges of 800 to 1300 cm^{-1} (bending vibrations) and 3000 to 3800 cm^{-1} (stretching vibrations). Despite the significant similarity of the structures, the arrangements of the layers in some hydroxides were different in the O–H····O–H distances (predetermining the length of the hydrogen bond), which led to a change in the arrangement of the IR bands [18]. The vibration stretching of S=O of the sulfonate group of Na_2SO_4 was located at ~1100 cm^{-1}. The absorption peaks in the region of 700 to 400 cm^{-1} corresponded to the existence of $Al(OH)_3$ and Al_2O_3. The IR spectra of the calcined samples were slightly different from that of the pre-dried sample. In the region of 1000 to 400 cm^{-1}, the calcined samples showed a noticeable change with peak shift compared to that of the pre-dried sample and a broader peak at 1000 to 800 cm^{-1} resulting from the phase transformation of gibbsite to active θ-Al_2O_3. At high calcination temperature, $Al(OH)_3$ dehydrated to Al_2O_3, resulting in the absorption band shift. The change of the crystal structure could also be responsible for the differences. In addition, the band of gibbsite could partially overlap with that of boehmite at 1070 cm^{-1} as in the pre-dried sample. From the results of XRD and IR spectra, the waste samples with the calcination temperatures of 800 and 1000°C were selected to blend with fly ash for making the geopolymeric materials.

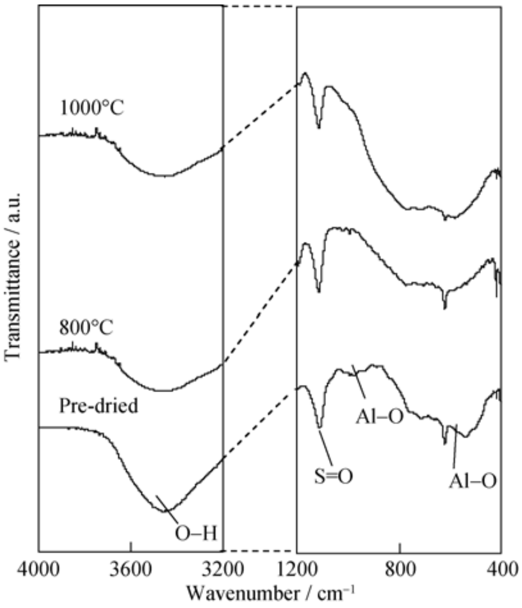

Fig. 4. IR spectra of the pre-dried and calcined samples.

3.2.3. Compressive strength

The pre-dried sample and calcined samples (if applicable) were mixed with fly ash, water glass, 10 M NaOH, and sand to form geopolymeric materials. The compressive strengths of the specimens are presented in Fig. 5. The control geopolymeric specimen had the compressive strength of 27.6 MPa. Blending of the fly ash with the pre-dried waste gave slightly lower strengths of 27.4 and 24.8 MPa for 2.5wt% and 5.0wt% replacements, respectively. The pre-died waste contained impurities and the low strength geopolymers were thus obtained. The low replacement level of 2.5wt% pre-dried waste resulted in a small decrease in strength. However, at the 5wt% replacement level, the strength was significantly reduced. In contrast, the calcined waste at 800 and 1000°C gave higher strengths than that of the control. For the calcined waste at 800°C, the 7-d compressive strengths of the mixes with 2.5wt% and 5wt% replacements were 31.2 and 29.9 MPa, respectively. At a high calcination temperature of 1000°C, the compressive strengths of the 2.5wt% and 5wt% replacements were significantly increased to 34.2 and 31.6 MPa, respectively. The higher strength was obtained as a result of the increase in amorphous phase of the calcined waste at high temperature. The amorphous phase was relatively active and enhanced a chemical bonding between the Al-compound and sodium silicate. The Al dissolved in sodium silicate solution and replaced the four-coordinate Si in the silicate network leading to a strong chemical bonding [13, 19]. The presence of gibbsite ($Al(OH)_3$) increased the strength of geopolymer compared with that containing both boehmite and gibbsite. The gibbsite readily dissolves at moderate hydroxide concentrations and temperatures. The dissolution of θ-Al_2O_3 and other Al forms, however, requires more rigorous conditions and generally is kinetically slow [20]. As a result, NaOH in the mixture helped the dissolution of gibbsite, resulting in the strength enhancement of geopolymeric materials. In addition, calcination removed impurities and promoted the phase transformation.

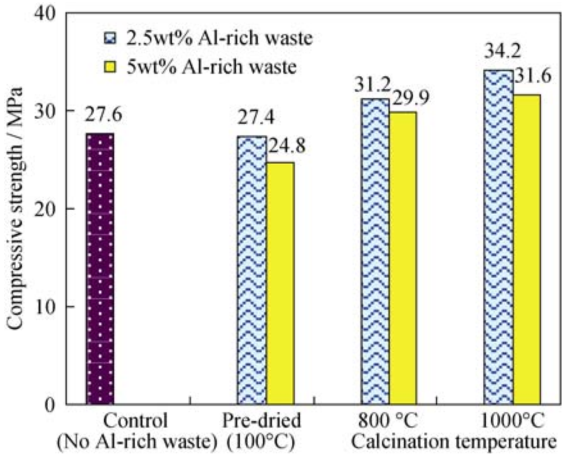

Fig. 5. Compressive strength of Al-fly ash geopolymeric materials at the 7-d curing.

However, the increase in Al content could adversely affect the strength. The surplus aluminium acted as the filler because of the insufficient amount of Si for geopolymerization reaction. However, for the source material with high silica content such as rice husk ash, the high Al dosage is beneficial for the strength development of the geopolymer [21]. The mechanical strength of the material is known to be enhanced by a high initial silica-to-alumina (SiO_2/Al_2O_3) ratio, which increases the number of strong Si–O–Si bonds in the final product [22]. The Al_2O_3 content in the system also plays an important role in the controlling process kinetics

[23-24]. The SiO_2/Al_2O_3 ratios of the specimens are shown in Table 3.

Table 3. SiO_2-to-Al_2O_3 ratio in geopolymer mixtures

Geopolymer sample	SiO_2-to-Al_2O_3 molar ratio
Control	4.32
2.5wt% Al-rich waste	3.88
5wt% Al-rich waste	3.51

It was reported that the optimum SiO_2/Al_2O_3 ratio was between 3.5 and 4.0 for good strength geopolymer. The use of low SiO_2/Al_2O_3 ratio resulted in an Al-rich aluminosilicate gel and a low strength geopolymer. The mechanical strength rose during the transformation of the Al-rich into a Si-rich aluminosilicate gel [23]. In addition, some calcium silicate hydrate (C–S–H) was also formed and coexisted with the class C fly ash geopolymeric products [24]. The formation of C–S–H contributed partly to the strength development and resulted in less silica available for the geopolymerization reaction. The aluminosilicate phase with a low Si/Al ratio was therefore obtained [25]. A decrease in Si/Al ratio could lead to the aluminosilicate compound of lower strength accompanied by the increase in crystalline phase [26]. Therefore, the increase in the fly ash replacement level of the Al-rich waste from 2.5wt% to 5wt% resulted in the geopolymer with a lower strength.

3.2.4. Microstructural study

The microstructures of Al-fly ash geopolymer pastes prepared by the pre-dried and the calcined samples (1000°C) with 2.5wt% replacement were investigated using SEM. Fig. 6 shows the geopolymer gel formation on the fly ash particles. The hardened gel prepared using the non-calcined Al-rich waste was not homogenous and agglomerations of the gel were easily detected. Partially reacted fly ash particles were easily detected as a result of the low reactivity of the waste. For the calcined Al-rich waste, the formation of the gel fully covered the fly ash particles resulting in the more homogenous matrix compared with the pre-dried Al waste sample due primarily to the increased reaction of active alumina (θ-Al_2O_3). The gel bound the fly ash particles and formed a dense geopolymer matrix, resulting in an increase in compressive strength. Therefore, calcination enhanced the reactivity of Al-rich waste, making it suitable for use as an alternative aluminium source material.

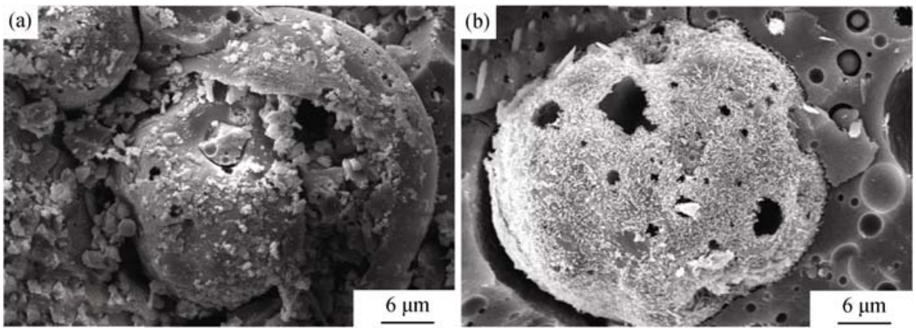

Fig. 6. SEM images of Al-fly ash geopolymer: (a) 2.5Al (PD) sample; (b) 2.5Al (1000) sample.

4. Conclusions

The improvement of the Al-rich waste from a polymer plant can be made by grinding and calcination. The calcination transformed the inactive boehmite to active alumina (θ-Al_2O_3). According to the XRD pattern, the calcination temperature of the waste needs to be higher than 800°C to obtain the phase transformation. The ground calcined waste with a median particle size of 15.0 μm can be used as an additional source material for adjusting the silica-to-alumina ratio of the fly ash geopolymeric materials. A reasonable compressive strength of 34.2 MPa was obtained at the age of 7 d for fly ash geopolymer mortar with 2.5wt% replacement of the Al-rich waste (calcined at 1000°C) compared with the strength of 27.6 MPa of the control.

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