One-part geopolymers from mining residues – Effect of thermal treatment on three different tailings

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ABSTRACT

Use of mine tailings as an aluminosilicate precursor in alkali activation is becoming an interesting alternative to manage the high-volume of waste generated from mining industries. However, very few tailings have so far been studied for their mineralogical properties and alkali activation potential. This study aims at understanding the ability of mine tailings from phosphate, kaolinite and lithium mines for their efficient participation in alkali activation. Biotite, muscovite, kaolinite, albite, and quartz were found to be the major minerals present in them. The impact of variation in mineralogy on silica and alumina solubility of these tailings was analyzed. The solubility was found to be high in impure kaolinite compared to the other two. Effectiveness of thermal treatment (750 °C and 900 °C) on improving the reactivity of these tailings in alkaline condition was also investigated. It was observed from the results that the effect of thermal treatment on the crystalline structure and solubility of an aluminosilicate material mainly depends on the mineral structure of the material, as well as the treatment temperature. Interestingly, thermal treatment reduced the solubility of lithium tailings with albite and quartz mineral. Effort has been made to relate the strength attained by alkali activation of mine tailings to their solubility values. However, despite of the higher solubility offered by impure kaolinite, phosphate tailings gives the maximum strength improvement by 62%. This can be due to the presence of calcium compounds in phosphate tailings that resulted in additional hydration products.

1. Introduction

Geopolymers are materials obtained through alkali-activation of aluminosilicate rich precursors. The aluminosilicate precursor contains the matrix-forming components that polymerize to form the geopolymer structure, and it can be any aluminosilicate material that contains soluble silicate and aluminate (Panagiotopoulou et al., 2007; Van Jaarsveld et al., 1997). For applications, where good mechanical and chemical properties are needed, the suitable molar Si/Al ratio is 1–5 (Provis and Van Deventer, 2013, p. 95). A lower ratio results in a more zeolitic structure, while with a higher ratio silicate derivative dominates the matrix (Wan et al., 2017). The most common aluminosilicate sources are various industrial byproducts and residues (i.e. fly ash, slag, mine tailings i.e. from copper, iron and gold ores), natural sources (i.e. sedimentary diatomaceous earth, volcanic glass) and activated aluminosilicates (calcined clays like metakaolin) (Provis and Van Deventer, 2013, pp. 103–112). Sometimes reactive fillers or setting additives are needed to improve the properties of the product material (Khale and Chaudhary, 2007). For example, too low solubility or availability of aluminum or silicon ions from the precursor can be compensated by adding metakaolin (Provis and Van Deventer, 2013, p. 107). Especially when using waste materials as aluminosilicate sources, thorough monitoring of the raw material properties is imperative. For example, the elemental compositions, alkalinity and granulometry can be used to evaluate the suitability of a material for geopolymerization (Davidovits, 2015, pp. 417–422).

Various natural minerals, such as calcined kaolinite, albite or volcanic ashes have been used in geopolymer synthesis (Provis et al., 2015). Experiments have revealed that some aluminosilicate minerals can form geopolymers on their own (i.e. stiblite, sodalite), while other, less reactive minerals require the supplementary sources (such as metakaolinite) in order to form proper geopolymer structures during alkali-activation (Xu and Van Deventer, 2000). The group of minerals that appears as the most ideal for geopolymerization is the 1:1 layer lattice aluminosilicates (MacKenzie et al., 2007). Due to the complexity of the dependence between solubility, mineral chemistry and structure, it is not possible to determine the suitability of a given mineral for geopolomerization theoretically, but experimental validation is always...
needed (Xu and Van Deventer, 2000).

Mine tailings are the mineral residues gained from the refining process at the mining sites. It usually has a fine particle size (> 100 μm) and is stored in dammed ponds. Mine tailings properties (like chemical composition) depend entirely on the mineralogy of the mining site and the used refining method. The tailings compose of a mixture of different minerals, which makes the characterization and utilization of a potential material even more difficult (Kinnunen et al., 2018). Due to their varying chemical compositions and commonly high Si/Al ratios, mine tailings are challenging and unpredictable raw materials (Wan et al., 2017). The parameters controlling the material properties are difficult to determine and understanding in this direction is given importance in recent years (Provis et al., 2015). The particle size and chemical distributions of the tailings are non-evenly distributed in the pond due to sedimentation, making the utilization further difficult. Pumping the waste slurry into the pond through a pipeline leads to the larger sized particles settling close to the discharge pipe as the finer material flows further. Industrial processes usually demand homogenous raw material supply to maintain a uniform quality of the product. In general, low alkali-reactivity of the tailings results in the primary problem and raises a need for various pretreatments (Kinnunen et al., 2018).

The heterogeneous nature of mine tailings is a challenge regarding their use as raw materials. Mine tailings, as well as other reused wastes, require more characterization than virgin raw materials (Duxson et al., 2007). Furthermore, due to the varying chemical compositions between different batches, robustness of the industrial process becomes imperative. Geopolymerization process is relatively robust and insensitive to variation of the amounts of the non-reactive components. The most studied mine tailings are so far tungsten mining side streams, copper mine tailings and oil sand tailings (Kinnunen et al., 2018). The main aim of this study is to screen the alkali activation potential for a set of mine tailings samples from three different sources viz., phosphate, kaolinite and lithium mining locations. Solubility tests are used to measure the Si and Al solubility of the samples, and alkali activation of the tailings are evaluated by preparing the paste samples. Thermal treatment is employed as a method to improve mineral solubility in alkaline condition. It is observed from the results that the effect of thermal treatment on the crystalline structure and solubility of an aluminosilicate material depends on the mineral structure of the material, as well as the treatment temperature.

2. Materials and methodology

2.1. Materials

The origin and main mineral components of the mine tailings studied in this work are listed in Table 1. The elemental compositions of the mineral components (which constitute the mine tailings) are listed in Table 2. The samples contained varying moisture contents and particle size distributions. The overall elemental compositions of mine tailings based on XRF analysis are listed in Table 3. According to Table 3, samples impure kaolinite (K) and Lithium tailings (L) possessed high silica and alumina contents when compared to Phosphate tailings (P). This observation is in agreement with their mineralogical composition as in Table 1. On the other hand, sample P contained relatively high amounts of MgO, and Fe₂O₃ (originating from the mica minerals) and CaO (originating from CaCO₃). All the samples contained significant portions of potassium oxide (K₂O), and L had a high concentration of sodium oxide (Na₂O) originating from albite.

2.2. Methods

2.2.1. Pre-treatment

2.2.1.1. Drying and grinding. The crude samples were placed in aluminum trays and were dried in oven at 100 °C for 24 h. Fig. 1 shows the images of samples after drying. Sample K had initially a very


**Table 2**
Elemental composition of various mineral components of the samples.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Composition</th>
<th>Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcite</td>
<td>CaCO₃</td>
<td></td>
</tr>
<tr>
<td>Phlogopite</td>
<td>KMg₃(AlSi₃O₁₀)(OH)₂</td>
<td>Mica mineral</td>
</tr>
<tr>
<td>Muscovite</td>
<td>KAl₄(AlSi₃O₁₀)(OH)₂</td>
<td>Mica mineral</td>
</tr>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td></td>
</tr>
<tr>
<td>Kaolinite</td>
<td>Al₄Si₄O₁₀(OH)₄</td>
<td></td>
</tr>
<tr>
<td>Albite</td>
<td>Na(AlSi₃O₈)</td>
<td>Feldspar mineral</td>
</tr>
<tr>
<td>Microcline</td>
<td>K(AlSi₃O₈)</td>
<td>Feldspar mineral</td>
</tr>
</tbody>
</table>

**Table 3**
Composition of mine tailings samples expressed as oxides according to XRF (%).

<table>
<thead>
<tr>
<th>Sample</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>MgO</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>Na₂O</th>
<th>Other*</th>
<th>LOI m (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>32.99</td>
<td>7.09</td>
<td>17.27</td>
<td>12.92</td>
<td>7.99</td>
<td>5.53</td>
<td>0.71</td>
<td>1.49</td>
<td>14.01</td>
</tr>
<tr>
<td>K</td>
<td>72.52</td>
<td>16.43</td>
<td>0.82</td>
<td>0.05</td>
<td>1.90</td>
<td>3.05</td>
<td>0.08</td>
<td>0.73</td>
<td>4.42</td>
</tr>
<tr>
<td>L</td>
<td>78.44</td>
<td>12.57</td>
<td>0.14</td>
<td>0.30</td>
<td>0.51</td>
<td>2.80</td>
<td>4.44</td>
<td>0.20</td>
<td>0.59</td>
</tr>
</tbody>
</table>

*Includes other minor oxides present in the tailings (MnO, P₂O₅, and SO₃).

High moisture content and behaved like clay, but upon drying, it transformed into one solid, with agglomerated particles. After drying, the samples were ground in a vibratory disk mill in order to standardize the particle size distributions. The grinding was performed at 1500 rpm for 3 min, in batches of 20 g. The vibratory disk mill Retsch RS 200 was used with a hardened steel grinding set.

### 2.2.1.2. Calcination

Batches consisting of 30 g of each sample were placed in corundum crucibles, calcined at 750 °C and 900 °C for 2 h and left to cool down in a Controller P320 furnace (Naberrherrn, Germany). These samples were used for XRD analysis and solubility tests. Later calcination of new batches of 100 g of each sample for alkali activation were carried out with the same method except for a holding time of 3 h. Several hypotheses were involved while considering the samples for calcination. Sample K was presupposed to be the most promising sample of the series, since kaolinite is known to form highly reactive metakaolin (Al₄Si₄O₁₀) during calcination (Sabir et al., 2001). Also sample P was assumed to give a strong response to the thermal treatment, since CaCO₃ is known to form lime (CaO) when heated, releasing CO₂ in the process (Rodriguez-Navarro et al., 2009). Sample L was supposed to be the most inert of the series. Quartz and the feldspar components usually appear thermally stable under the calcination conditions (Deer et al., 1963, pp. 12, 180; Feng et al., 2012; Heaney et al., 1994, p. 314). Due to its highly crystalline nature, quartz is known to have a relatively high chemical stability compared to other silicate minerals (Loughnan, 1969, p. 53).

### 2.2.2. Characterization of treated materials

#### 2.2.2.1. XRF and TGA

X-ray fluorescence analysis (XRF) was done to determine the elemental compositions. The analysis was made using fused bead test, where the sample was melted to form glass at 1150 °C. Hence, the results represented partly calcined samples, and the possible reactions during the bead preparation posed a risk of error in the calculations. Thermogravimetric analysis (TGA) was carried out to observe changes in the material during the heating process, and hence exposed the possible reactions. The equipment used was PrepASH 129 (Precisa, Switzerland), and the temperature ranged between room temperature (23 °C) and 950 °C. The weight of the analyzed specimens were maintained below 7.7 g. XRF results were corrected by adjusting the results for a changed total mass. It was assumed that no measured components, especially Si or Al, was lost during the heating. The equation for calculating corrected values is presented as Eq. (1).

\[
m(\%) = \left(1 - \frac{\text{LOI}}{100}\right) \times m_m(\%)
\]

where m (%) is the corrected weight percent of a component (%), LOI (loss on ignition) is the percent of sample mass lost in TGA and mₘ is the measured weight percent of a component.

#### 2.2.2.2. XRD

XRD analysis was performed to observe changes in the crystalline structure of samples during thermal treatment. Therefore, the analysis was carried out for each sample prepared in different calcination temperatures. Rigaku SmartLab (9 kW) with 40 kV and 135 mA radiations was used, and the crystalline structures were identified from the data by comparing the diffraction patterns on the ICDD database (the International Center for Diffraction Data). Cu-Kβ radiation was used with step interval of 0.02° per step and scan rate of 4°/min for a scan range of 5–110° (2θ).

#### 2.2.2.3. Alkaline solubility test

The solubility tests were carried out with 6 M NaOH solution. For each test 0.5 g of sample was mixed with 20 g of alkali solvent. The mixture was then shaken in a shaker mixer (KS 260 basic, IKA) at 150 rpm for 24 h. The solution was filtered through a polypropylene membrane with 0.45 µm pore size. The pH of the collected solution was set below 2 with 6 M nitric acid solution to prevent precipitation. The dissolved Si and Al amounts were determined from the collected solution by inductively coupled plasma optical emission spectrometry (ICP-OES) according to standard SFS-EN ISO 11885:2009.

#### 2.2.3. Geopolymerization

Geopolymers involving the alumino-silicate precursor and activator solution can be referred to as two-part geopolymers. One-part geopolymers are prepared by mixing the solid activators with the alumino-silicate precursor and can be activated with the addition of water. Hence, one-part geopolymers are ‘just-add-water’ products and suitable for situations, where the handling of liquid alkali activators would be problematic. In this study one-part geopolymerization process was adopted. Mine tailings and alkali activators (Na₂SiO₃ + NaOH or Na₂SiO₃) were co-grinded in the ratio as mentioned in Table 4. The paste samples were prepared by adding measured quantity of water. Thinky ARE-250 planetary mixer was employed for 90 s at 1000 rpm.
speed to obtain a homogeneous mix. Water content was kept constant throughout the mixtures except for non-calcined (NC) P tailings which needed 5.5 g extra water to maintain the minimum workability for making the specimens. Isothermal calorimetry to understand the reactivity of different mine tailings in alkali activation was performed with an 8-channel Tam Air isothermal calorimeter (TA Instruments, USA).

The samples for each mix were molded in to six cylindrical (25 mm diameter and 25 mm height) plastic molds and vibrated with the Vortex-Genie 2 vortex mixer (Scientific Industries, Inc., USA) for 10 s to remove the air captured inside them. The molds with the fresh alkali activated paste were placed in sealed plastic bags and cured at 40 °C for 24 h. After demolding, the specimens were stored at room temperature until testing. Three specimens of each mix were sulphur capped and used to measure the compressive strength. Zwick 100 testing machine (Zwick Roell Group, Germany) with the maximum load capacity of 100 kN and a cross head speed of 3 mm/min was employed. Water absorption was measured by immersing the specimens in water bath for 24 h in room temperature (23 ± 2 °C). After wiping with wet cloth, saturated mass of the specimen was measured (Wsat). The specimens were then placed in oven at 105 ± 2 °C for 24 h and the oven dried mass was measured (Wdry). Water absorption was then calculated in percentage by,

\[
\text{Water absorption(\%)} = \frac{W_{\text{sat}} - W_{\text{dry}}}{W_{\text{dry}}} \times 100
\]

3. Results and discussion

3.1. Effect of pre-treatment on mineralogy

The XRD results for sample P are presented in Fig. 2. According to the analysis, the material initially contains phlogopite, calcite, tremolite and dolomite. These results are in line with the initial mineralogical data. Phlogopite is a magnesium rich biotite mineral (Deer et al., 1962). As supposed, calcite (CaCO\(_3\)) decomposes forming CaO when the sample is calcined at 750 °C. It can be noted that dolomite was also decomposed. Furthermore, the data shows an amorphous hump that does not appear before the calcination. When the sample is calcined at 900 °C, CaO disappears and phases like quartz, cordierite, crystalline silicates, are formed. The formation of CaO and decrease of crystallinity during the first calcination stage indicates an increase in the reactivity of the material. On the other hand, the new crystalline quartz and silicate phases formed during the second calcination stage are assumed to be stable (Loughnan, 1969, p. 53), indicating a decrease in reactivity. Considering the phase changes during the calcination, sample P is the most reactive of the three studied samples.

XRD measurements reveal that sample K consists of quartz, muscovite and kaolinite, as can be seen in Fig. 3. According to the analysis, none of the different calcinations resulted in the formation of any new crystalline phases. The only observed change is decomposition of kaolinite at 750 °C. It is assumed that metakaolin is formed from kaolinite during its decomposition. However, metakaolin has amorphous structure and hence is not detected in XRD analysis (Wan et al., 2017). Metakaolin is known to react further into crystalline, insoluble mullite, which begins to appear in temperatures over 800 °C (Provis and Van Deventer, 2013, p. 106), but the XRD analysis does not show traces of mullite in the sample calcined at 900 °C. Large contents of mullite were not expected, since the mineral mainly forms in temperatures higher than 1000 °C (Meinhold et al., 1985). On the other hand, some amorphous intermediate phases are known to form during calcination of kaolinite at higher temperatures (Meinhold et al., 1985).

The XRD graphs for sample L are presented in Fig. 4. The non-calcined sample contains quartz, albite and microcline, which corresponds to the initial mineralogical data. The XRD does not show any signs of major phase changes during calcination at 750 °C, but as the treatment temperature is raised to 900 °C, albite decomposes partially. There is a reduction in the intensity of Albite peak. The transformation of albite can be assumed to have occurred due to chemical reaction with other components, since pure albite is known to be thermally stable in temperatures below 1000 °C (Feng et al., 2012). No new crystalline phases emerge during the calcination, which indicates that the new components formed during the albite transformation are amorphous.

3.2. Effect of pre-treatment on solubility of Si and Al

Relative solubility was calculated based on the ICP-OES results and XRF analysis, using formula (2).

\[
s(\%) = \frac{m_d}{m_s} = \frac{c_d \times V_d}{w_s \times m_s}
\]

where \(s\) – relative solubility (%), \(m_d\) – dissolved mass of the component (mg), \(m_s\) – component mass in the solid sample before dissolution (mg), \(c_d\) – component concentration in solvent after dissolution (mg/l), \(V_d\) – volume of the solution (l), \(w_s\) – mass percentage of the component in the solid sample and \(m_s\) – total sample mass before dissolution.

Fig. 5(a) shows the relative solubility of Si, and Fig. 5(b) shows the relative solubility of Al of non-calcined and calcined mine tailings (result is expressed as % of element released with respect to its initial content in the sample before solubility test). During the tests, the solubility of Al from sample P increases drastically with both treatment temperatures (Fig. 5b). The Si solubility improves during calcination at 750 °C but reduces as the temperature increased further to 900 °C (Fig. 5a). Sample K has the highest solubility among the studied samples. An optimal calcination temperature considering both Si and Al solubility exists at 750 °C by a large margin compared to the other calcination stages. Sample L has a low initial solubility, which further decreases as the calcination temperature was raised.

For all samples, the relative solubility of Al exceeds Si, but due to the large content of Si compared to Al in all the samples, the total dissolved amount of Si exceeds Al in almost all cases. For non-calcined sample P, elemental solubility of both Si and Al are very low, and probably not sufficient for a successful geopolymerization. The increasing solubility of sample P after the first calcination at 750 °C can be explained with the reduction of crystalline phases as detected with XRD (Fig. 2). The decrease in solubility of Si after calcination at 900 °C can be explained by the formation of inert Quartz and crystalline silicates that stabilize silicon ions. However, unlike Si solubility, Al solubility of sample P increases slightly after calcination at 900 °C. This could mean that during the thermal treatment, some aluminum containing amorphous phases were formed leading to its increased solubility.

Sample K had high silicate and aluminate contents (73% and 16% respectively) and with the exceptionally high Al solubility of the sample calcined at 750 °C, the total dissolved Al is highest among all the samples. On the other hand, the solubility of Si in the sample calcined at 900 °C is lower than the initial solubility. The increase in solubility during the first calcination stage supports Kaolinite transformation into metakaolinite. Also, the increase of total dissolved Al and Si appears roughly equal in ratio supports the hypothesis, since the Si/Al ratio of metakaolinite is approximately one. The decline in solubility after calcination at 900 °C may indicate the formation of amorphous inert components from metakaolinite. Based on solubility, sample K appears to be the most promising sample for geopolymerization.

The experiments with sample L support the hypothesis of the sample

<table>
<thead>
<tr>
<th>Series</th>
<th>Mine tailings (g)</th>
<th>Na(_2)SiO(_3) (g)</th>
<th>NaOH (g)</th>
<th>Solid/liquid ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>SN</td>
<td>63.41</td>
<td>14.69</td>
<td>1.91</td>
<td>0.25</td>
</tr>
<tr>
<td>S</td>
<td>63.41</td>
<td>14.69</td>
<td>–</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Table 4 Mix proportion of geopolymer paste.
Fig. 2. XRD results for sample P at different calcination stages.

Fig. 3. XRD results for sample K at different calcination stages.
being inert. The partial decomposition of albite observed with XRD implies that some chemical reactions took place during the thermal treatment, and the declining solubility supports the interpretation. Therefore, the new phases that formed during the calcination appear to be amorphous but insoluble. The solubility decreases continuously as the calcination temperature rises, which implies the reaction occurs gradually as the temperature is increased.

3.3. Geopolymer properties

3.3.1. Setting characteristics

The setting of alkali activated mine tailings before and after calcination was observed visually and represented in Figs. 6–8. Setting properties were similar with both SN and S series of mixtures. Hence, S series are represented in the figures for discussions. The setting of alkali activated mixtures with P tailings does not show any abnormality (Fig. 6a). Setting of geopolymer happens with crystal reorganization and growth of nuclei as it reaches a critical size (Duxson et al., 2007) and hardening of the matrix takes place as excess water escapes the solidifying mixture (Davidovits, 2015, p. 424; Khale and Chaudhary, 2007). However, this is not the case with other tailings. The K tailings calcined at 900 °C does not set after 24 h curing regime (Fig. 7c). This can be related to the mineralogical changes of kaolinite mineral which becomes amorphous at 750 °C and recrystallizes at 900 °C. This is also supported by the drastic reduction in solubility of S-tailings when calcined at 900 °C (Fig. 5a and b). Whereas, any of the alkali activated L tailings samples hardened only partially. Extending the curing period does not induce setting and the plastic nature of cured samples can be observed in Fig. 8. This can be related to the lower Si and Al solubility of L-tailings compared to other tailings. Slow polymerization process and relatively rapid precipitation of geopolymers, results in incomplete setting. As a result the gained geopolymer matrix has an amorphous and

![Fig. 4. XRD results for sample L at different calcination stages.](image-url)

![Fig. 5. Effect of calcination on relative solubility of (a) Si and (b) Al.](image-url)
3.3.2. Water resistance

The alkali activated SN (Na$_2$SiO$_3$ + NaOH) and S (Na$_2$SiO$_3$) series of mine tailings pastes were examined for their resistance in water and the results are shown in Fig. 9. Specimens from series SN with NaOH were not stable in water. Initially, white crystals were formed on the surface of the specimens showing the possible formation of sodium carbonate due to the exposure to atmospheric carbon-dioxide (Slaty et al., 2015). This is also explained by Barbosa et al. (2000) that the excess Na$^+$ ions comes to the surface of the specimen and undergoes carbonation. This can be further justified by the appearance of S series (without NaOH) specimens which does not show any carbonation on the surface. Hence, the water resistance of S series specimens was studied and represented in Fig. 10.

3.3.3. Compressive strength

Compressive strength of SN and S series of the alkali activated mine tailings specimens are shown in Fig. 11. As specimens of alkali activated non-calcined and calcined L tailings had setting problem (Fig. 8), testing its strength was not possible for those mixtures. Due to imperfect dissolution, some of the precursor material might have left inside the final material as unreacted residues and worked as an aggregate. This behavior of the material may either improve or weaken the material properties (Provis et al., 2005; Xu and Van Deventer, 2000). Very low heat evolved during the geopolymerization of L tailings measured at initial 70 h (Fig. 12c) and lower solubility (Fig. 5) can be an indication of the weak product formation leading to setting problems.

Calcination of P tailings at 750 °C improved the strength by 62% and 75% in SN and S series, respectively. The high magnesium content of P tailing from Phlogopite mineral could be a reason for this behaviour. MgO is already known to improve the mechanical strength of alkali activated materials by the formation of hydrotalcite as the reaction product (Ben Haha et al., 2011; Sreenivasan et al., 2017). Also, P tailings is rich in carbonate content from calcite and dolomite which increased the pH with the addition of water. This would help in the dissolution of Al and Si containing minerals and further geopolymer formation. However, there is a reduction in strength when the calcination temperature is increased to 900 °C (Fig. 11). Considering the release of Si and Al ions in solubility test, the reduction in solubility of Si and increase in solubility of Al at 900 °C affects the Si/Al ratio. The Si/Al ratio in the final geopolymer matrix affects the structure and application of the material and has an optimal value which varies based on the precursors used. For example, Wan et al. (2017) observed in their study that the optimum Si/Al ratio for metakaolin-based geopolymers is 2. A smaller ratio leads to a porous, zeolitic structure, while a larger ratio resulted in formation of a well porous silicate derivatives forming structures. The heat evolution curves corroborate this argument as the heat energy increased at 750 °C and reduced at 900 °C (Fig. 12a).

K tailings shows the highest strength among the three tailings that are studied here. The Muscovite and kaolinite mineral by itself participate in alkali activation even before any treatment. And calcination at 750 °C improved the strength by 25%. This can be related to the formation of metakaolin with the heat treatment of clay mineral present in this tailings (Fig. 3). However, due to recrystallization that could occur with further calcination of metakaolin, the decline in strength was noticed at 900 °C (Provis and Van Deventer, 2013). This is also matching with the behavior of this tailings during geopolymerization and the heat evolution as shown in Fig. 12b.

4. Conclusion

In an effort to improve the reactivity of mine tailings from three different sources, calcination was applied as a treatment method and the effect on reactivity in alkali activation was studied. The following conclusions are drawn based on the material properties of the tailings acquired from this study:

- Mineralogical characterization of the mine tailings can be
considered as an important identifier to categorize the material from its response to any treatment or its reactivity in alkali activation. The mineralogical composition of different mine tailings affected the Si and Al solubility. P and K tailings responded positively to calcination until 700 °C, beyond which it declined. Whereas, L tailings showed continuous reduction in the solubility values.

- Alkali activation was successful in tailings with carbonates (P) and kaolinite (K) even without any heat treatment. However, heat treatment resulted in the formation of CaO, MgO and metakaolin which helped in the dissolution of Si and Al at high pH and further strength improvement.
- Compressive strength improved with calcination of mine tailings with a maximum of 62% for P tailings and 25% for K tailings at 750 °C. Increase in solubility of Al ions at 900 °C is likely to affect...
the Si/Al ratio that lead to reduction in strength compared to calcination at 750°C. However, it is important to fix the optimum treatment temperature of treatment to attain maximum strength. This is due to the possibility of recrystallization of amorphous content in the tailings at higher treatment temperatures.

It can be concluded that in order to adopt a treatment technique and to improve the reactivity in alkali activation, mine tailings need location specific characterization. Detailed investigation on different treatment methods based on mineralogical composition would help in effective utilization of this material to a larger extend.

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References


Fig. 12. Heat evolved during alkali activation of (a) P (b) K and (c) L tailings (S-series).

