The Influence of Thermal and Thermochemical Pre-treatment of Mine Tailings on their Solubility and Phase Composition for Geopolymerization Application

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ABSTRACT: The largest hurdle for the reuse of mine tailings as a source material for geopolymerization and the production of construction materials is the tailings' low reactivity. The highly crystalline tailings require a strongly basic alkali activator, which is user-hazardous and unfavorable for industrial use. In order to increase the reactivity of the mine tailings, pretreatment methods can be used to reduce the crystallinity and increase the solubility of aluminosilicates. Thermal pretreatment was able to increase solubility of silicon and aluminum, but this did not seem to be directly related to the increase in amorphous content. Thermochemical pretreatment was even more effective than purely thermal pretreatment for increasing the quantity of amorphous phase and producing soluble aluminosilicates that can be available for geopolymerization reactions. The aluminum solubility of pretreated tailings is closely related to the quantity of muscovite present after the treatment. Further research is needed to assess the effectiveness of pretreatment for geopolymerization of mine tailings and to link the amorphous content to the solubility of aluminosilicates. Mechanical and durability evaluation of geopolymers made from pretreated mine tailings is ongoing.

1 INTRODUCTION

Mine tailings are the largest category of waste produced during mining activities. Mine tailings are produced as a byproduct during processing and extraction of precious metals. This process begins with the excavation of ore that is subsequently crushed into a fine powder. The extraction process liberates and separates the valuable metals, leaving behind a slurry of rock and clay-like minerals, often imbued with heavy metals from the parent rock or chemical processing. These mine tailings are usually kept in slurry ponds and treated as hazardous waste and during storage, the toxins from the tailings can be spread to the surrounding soil (Zhang et al. 2020), microbial organisms (Fashola et al. 2016; Passos et al. 2020), and the environment (Sheoran and Sheoran 2006). The slurry ponds are contained by earthen dams, which have a failure rate of approximately one disaster every eight months (ICOLD Committee on Tailings Dams and Waste Lagoons 2001). Therefore, it is critical to address the challenge of identifying reuse options as an effective method for tailings management.

One alternative way to manage tailings is through solidification and stabilization (S/S). Solidification and stabilization is a twofold method: solidifying the waste into a monolithic specimen reduces the mobility of contaminants, while stabilization involves physical encapsulation or chemical sequestration of the heavy metals. S/S can be achieved through cement stabiliza-tion, but cement production releases a large quantity of carbon dioxide and therefore is not very environmentally friendly. An alternative method of S/S is geopolymerization. This process involves adding a strongly alkaline activator to an aluminosilicate source material, which then reforms into a solid specimen. The alkali activator has a very high pH, which depolymerizes the source material into its constituent silica and alumina phases. The free silica and alumina then form an amorphous gel, which undergoes polycondensation reactions at room temperature to form a 3D polymer network. For S/S of mine tailings, the tailings are used as the aluminosilicate source material and form a solid monolithic specimen. Geopolymers have been shown to immobilize the heavy metals in mine tailings (Kiventerä et al. 2018a,b; Zhang et al. 2016) and can be used as a concrete-like material in applications such as mineshaft backfill and masonry construction bricks (Ahmari and Zhang 2013). This solution not only reduces the environmental and human health risk of keeping tailings in storage ponds, but it also reduces the cost of tailings management and the land area required for hazardous waste storage.

When using mine tailings as a source material for geopolymerization, the largest hurdle is the low reactivity of the tailings. Because tailings are highly crystalline and usually have a high quartz content, they do not react readily with the alkali activator. Therefore, a strongly basic activator must be used, which can be user-hazardous and is hard to store in an industrial setting. It is important to find ways to increase the reactivity of mine tailings through pre-treatment meth-ods so that the aluminosilicates can be made available for reaction. The three main methods of pre-treatment are mechanical, thermal, and thermochemical. Mechanical pre-treatment involves wet or dry grinding to increase the reactivity by reducing the particle size (i.e., increasing sur-face area) and inducing crystal defects (Krishna et al. 2021). Thermal pre-treatment involves roasting the tailings at high temperatures (600-1200 °C), increasing the amorphous content by melting the aluminosilicates and reforming them into a glassy phase (Dabbebi et al. 2020; Kiventerä et al. 2018b; Luo et al. 2020; Perumal et al. 2019). The glassy phase is more reactive because it is less crystalline and therefore less stable (Rao and Liu 2015). Thermochemical pre-treatment is similar to thermal pre-treatment but involves adding the solid alkali activator to the mine tailings before roasting. This reduces the temperature needed to melt the mixture and therefore the energy demand of the pretreatment. The alkali activator in this study, sodium hydroxide, melts at 320 °C (US National Bureau of Standards 1954). Thermochemical activation also has the advantage of producing one-part geopolymers. Thermochemically activated mine tailings already contain the solid alkali activator, so the production of geopolymers only requires adding water. This makes the process less user hazardous, and more similar to traditional ready-mix concrete. The one-part geopolymer can be more attractive for industry use and also more feasible for larger-scale production.

This paper compares thermal and thermochemical pre-treatment for mine tailings from an artisanal gold mine in the Arequipa region of Peru. It investigates the effect of pre-treatment on the solubility of aluminosilicates as measured by ICP and static leaching, and the change in phase composition (crystalline and amorphous) via quantitative X-ray diffraction (qXRD). The research question addressed in this paper is as follows: how do thermal and thermochemical pre-treatment affect the mineralogical and chemical composition of mine tailings, and how does this affect the solubility of aluminosilicates in alkaline environments

2 EXPERIMENTAL METHODS

2.1 *Material Characterization Methods*

2.1.1 *Thermogravimetric Analysis (TGA)*

Thermogravimetric analysis was performed to determine the thermal decomposition temperatures of the minerals in the mine tailings. The mine tailings were loaded into a platinum crucible and heated from 40 to 1000 °C at 10°C/minute. The DTG curve was calculated by taking the derivative of the TGA curve. The peaks in the DTG curve indicate the temperature values at which the rate of change of the sample mass is highest.

2.1.2 *Differential Scanning Calorimetry (DSC)*

DSC shows the exothermic and endothermic reactions of the mine tailings as they are heated. It was performed using Perkin Elmer DSC8500. The sample was sealed inside of a platinum crucible and heated from 40 to 600 °C at 5°C/minute under a nitrogen flow of 20 mL/minute. The heat flow was measured in milliwatts per milligram and then reported as a function of temperature.

2.1.3 *Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS)*

In order to semi-quantitatively determine the chemical composition of the mine tailings, SEM-EDS was used. Five images of the tailings were recorded at 1000x magnification, and the quantity of characteristic x-rays was measured for each image. These values were averaged over all five images to increase the accuracy.

2.2 *Analysis of Reactivity*

2.2.1 *Static Leaching and Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES)*

In order to determine the solubility of silicon and aluminum, static leaching tests were performed on the thermally and thermochemically activated materials. Five grams of the pretreated mine tailings were added to 500 mL of ultrapure water (resistance > 18 MΩ) to give a sample to solution ratio of 1:100, as suggested by Wu et al. (2020). The solution was mechanically stirred for 30 minutes, followed by 30 minutes of resting time to allow the particles to settle to the bottom. A small aliquot was collected using a syringe and a 0.45 µm filter. Two milliliters of the solution were added to a Falcon tube, then diluted with deionized water to prevent the high concentration of sodium ions from clogging the ICP nebulizer. Seven drops of nitric acid were added to the diluted sample to achieve a pH of less than two. The filtered, diluted, and acidified samples were sent for ICP analysis with NIST standard reference 1643f and continuing check verification (CCV) for metals and nonmetals.

2.2.2 *Quantitative X-ray Diffraction (qXRD) with Internal Standard*

To determine the identity and quantity of crystalline phases in the activated tailings, quantitative x-ray diffraction was performed using a PANalytical PW3040 X-ray diffractometer and an internal standard of synthetic diamond powder (particle size 4-8 µm). The tailings samples were first sieved using a Hosokawa Micron Air Jet Sieve MAJSx2 and a US #450 sieve (32 µm mesh). The synthetic diamond powder was added at a dosage of 20% by weight and the sample was thoroughly mixed. The mixed sample was loaded into the sample holder and the surface was leveled off using a microscope slide. The smooth surface minimizes unwanted diffraction of the x-ray beam. The pattern was collected by using Cu Kα radiation at a voltage of 45 kV and a current of 40 mA. The scan was performed from 7° to 90° 2θ at a speed of 0.0425 °/second and 0.0334° per step. To determine the phase composition of the sample, PANalytical High Score software was used with the PDF 4+ database from the International Centre for Diffraction Data. The phases were identified using known chemical composition data and the search and match function of the High Score software. Rietveld refinement was used to determine the quantity of each phase present (McCusker et al. 1999; Santini 2015). The effectiveness of the refinement can be evaluated via the summary statistics "weighted profile R-factor" and "good-ness of fit". The weighted profile R-factor is calculated from the square root of the residuals and weighted by the signal intensities. The goodness of fit compares the weighted profile R-factor with the expected R-factor, which measures the quality of the collected x-ray diffraction pattern. The Rietveld refinement was considered suitable if the weighted profile R-factor was less than 10 and the goodness of fit was less than 4 (Toby 2006). The quantification of the in-ternal standard was used to infer the amorphous content of the sample. By normalizing the quantity of the crystalline phases to match the 20% diamond by weight, the quantity of amor-phous phases can be determined.

2.3 *Thermal Activation*

Small scale furnace tests were performed in order to determine the effects of thermal pretreatment. To prepare the mine tailings for roasting, the raw mine tailings were first sieved using a US #30 sieve (0.600 mm) for five minutes. The sieved tailings were then oven dried at approximately 100° C overnight to remove any moisture from the material. Thermal activation was performed using a benchtop furnace (ThermoScientific) at either 600, 800, or 1000° C for 2, 4, or 6 hours. Fifteen grams of the sieved and dried mine tailings were placed in a crucible with a second crucible as a lid. The furnace was heated up to the specified temperature and held at that temperature for the specified amount of time. After the time period had elapsed, the furnace was turned off and the samples was left in the furnace to cool. Once the crucible was cool enough to handle, the mine tailings were removed from the furnace and kept in a sealed container at room temperature until further analysis.

2.4 *Thermochemical Activation*

To prepare the thermochemically activated samples, the same sieved and dried tailings were used as for the thermal treatment. In addition, sodium hydroxide pellets were ground using a mortar and pestle until the particles passed a US #30 sieve (0.600 mm) and mixed with the tailings at a dosage of 10% by weight (citation). The sodium hydroxide/mine tailings mixture was loaded into a crucible with another crucible acting as a lid. The crucible was placed into the furnace for 2, 4, or 6 hours at 400, 600, or 800 °C. The same procedure was followed for roast-ing: the sample was placed in the furnace, heated to the specified temperature, and the furnace was turned off after the specified amount of time. Once the sample had cooled to room tempera-ture, the activated tailings were removed from the crucible and kept in a sealed container until further analysis.

3 MATERIALS CHARACTERIZATION

Gold mine tailings were collected from an artisanal gold mine in the Arequipa region of Peru. The mine tailings were characterized through a series of physical, mineralogical, and chemical tests. Physical testing included Harvard miniature compaction based on US Bureau of Reclamation standard GR-84-14, sieve analysis and hydrometer testing based on ASTM C136 and D7928 standards, respectively, and Atterberg tests based on ASTM D4318. The particle size distribution and results of the physical testing are shown in Figure 1.

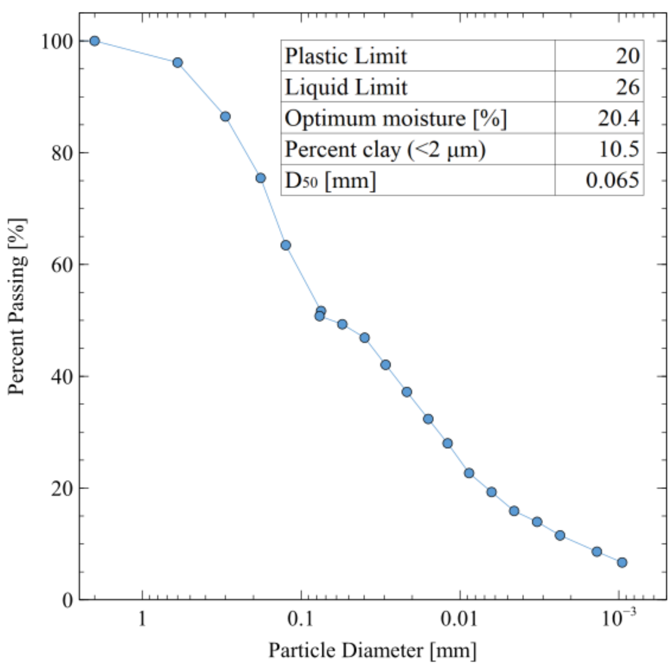

Figure 1: Particle size distribution and results of physical characterization

The mineralogical composition of the mine tailings was determined by qXRD, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). The qXRD analysis provided the phase composition of the crystalline component of the mine tailings.The major miner-al phases of the mine tailings were muscovite (31%) and quartz (20%). Muscovite is a hydrated phyllosilicate mineral with a monoclinic crystal system which also contains potassium, aluminum, and fluorine. Quartz is composed of silicon dioxide and has a tetrahedral crystal framework. The raw mine tailings also contained 30% amorphous phase and 9% albite. Albite is a plagioclase feldspar mineral and contains sodium, aluminum, and silicon in a triclinic crystal system. The mine tailings' minor phases were composed of sodium aluminosilicate hydrate, portlandite, calcium aluminum hydroxide hydrate, and calcite.

TGA was performed to further refine the phase composition based on the thermal decomposition of minerals and byproducts in the mine tailings. DSC was used to determine the thermodynamic behavior of the mine tailings at elevated temperatures, including specific phase transitions and thermal shifts. The results of the TGA and DSC measurements are shown in Figure 2. The largest peaks in the DSC curve occur at approximately 600 °C and 700 °C. These peaks can be attributed to the dehydroxylation of the minerals that make up the mine tailings. While muscovite, albite, and quartz do not decompose below 1000 °C, calcination of these minerals can strip the -OH groups away and cause mass loss during heating.

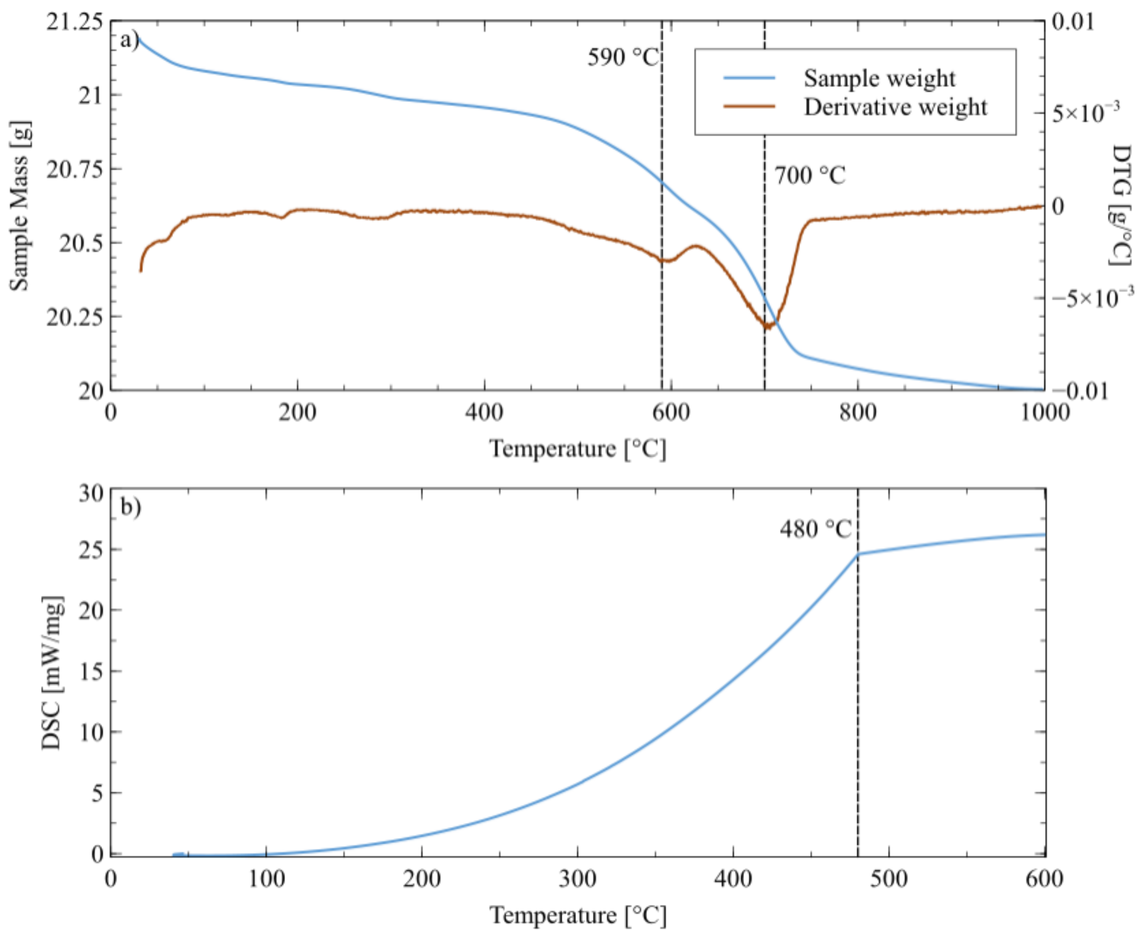

Figure 2: (a) TGA weight percent curve and differential weight curve; (b) DSC curve

The chemical composition of the mine tailings was determined through SEM-EDS and a static leaching test. The SEM-EDS scans were used to semi-quantitatively determine the elemental characterization of the tailings, while the static leaching test determined the proportion of each element that was soluble in water and/or alkaline solutions. The solution from the static leaching test was drawn off, filtered, diluted, acidified, and analyzed using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The results of both the SEM-EDS of the raw tailings and the ICP-AES from the static leaching are shown in Figure 3. The most mobile elements were the alkalis: calcium and sodium, plus potassium to a lesser extent. Silicon and aluminum were more soluble in sodium hydroxide than water and dissolved with an Si:Al ratio of 5.2 in sodium hydroxide and 11.1 in water.

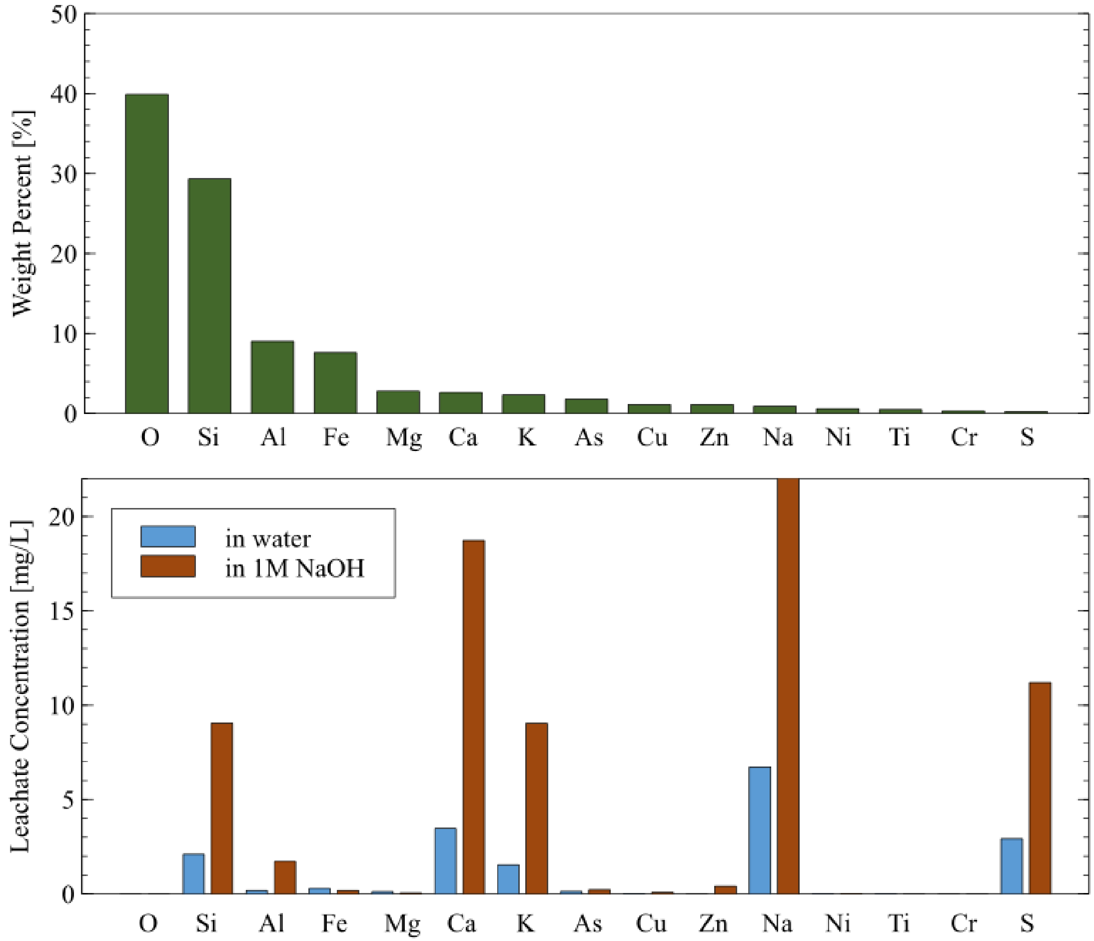

Figure 3: Comparison of chemical composition of mine tailings vs. proportion that is soluble and available for reaction (determined by ICP-AES and static leaching)

4 RESULTS AND DISCUSSION

As a proxy for reactivity testing, ICP-AES and qXRD were used to determine the effect of pre-treatment on the mine tailings samples. qXRD provides the phase composition of the crystalline phases and the use of an internal standard allows for calculation of the amorphous phase. ICP and static leaching were used to determine the solubility of silicon and aluminum, which are the main elements required for geopolymerization. The hypothesis is that a greater solubility of silicon and aluminum will allow the pre-treated tailings to produce greater quantity of aluminosilicate gel during geopolymerization, therefore producing a stronger and more durable geopolymer. A greater quantity of amorphous content is generally associated with higher solubility because crystalline phases are usually more stable and thus less reactive (Rao and Liu 2015).

The ICP results from the thermally activated mine tailings are shown in Figure 4. The effect of thermal pre-treatment on silicon and aluminum is not the same, so it is important to specifically analyze solubility of aluminum and silicon separately in order to judge the effectiveness of the thermal pre-treatment. There is not a clear trend of solubility based on temperature and/or time. The greatest solubility of aluminum was achieved through treatment at 800°C for two hours, while greatest quantity of silicon was achieved by treatment at 600°C for six hours. However, the difference between the 800° treatment and the 600° treatment for silicon availability is insignificant and therefore the best condition for pre-treatment appears to be calcination at 800°C for two hours. The 1000 °C pre-treatment was not effective, especially for solubility of aluminum.

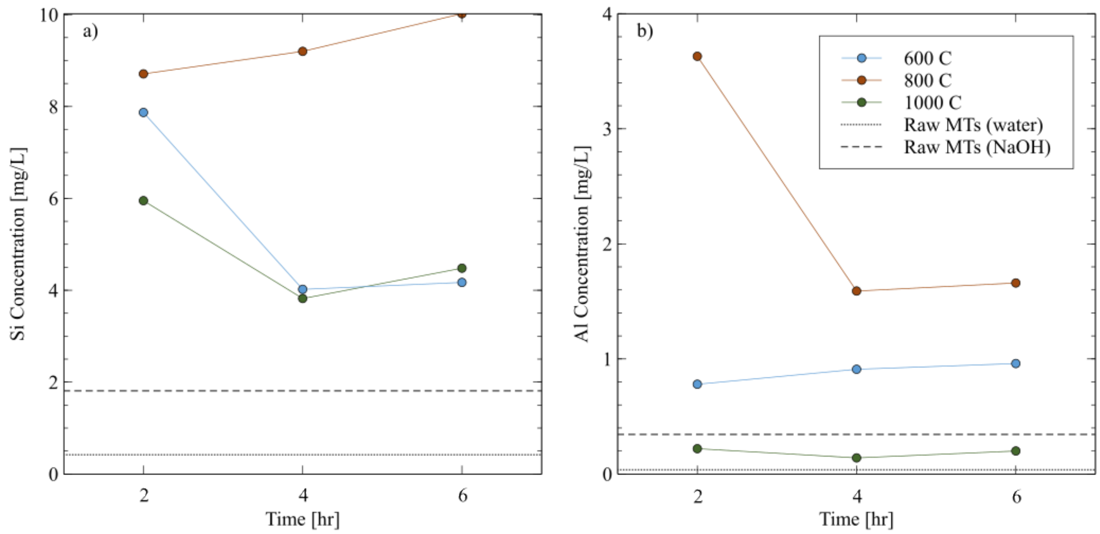

Figure 4: Results of ICP-AES from thermally activated tailings for a) silicon and b) aluminum

The phase quantification determined via qXRD for the thermally activated mine tailings is shown in Figure 5. It appears that the thermal treatment increased the amorphous content in nearly all cases, but the quantity of quartz increased in most cases as well. For the 600 °C treatment, the maximum percentage change in any phase was only 7%. It does not appear that purely thermal treatment at this temperature is severe enough to cause changes in the phase composition of the mine tailings. However, the ICP results (Figure 4) demonstrated that this treatment increased the availability of silicon. This could be from the change in minor phases, where the decomposition of sodium aluminosilicate hydrate increases the solubility of silicate ions. Because albite and muscovite are the Al-bearing minerals, it is justifiable that the 600 °C pretreatment was not effective in increasing the solubility of aluminum in the static leaching test.

The 800 °C treatment had the most time-dependent results: the 2-hour treatment decreased the quantity of amorphous phase and increased the amount of quartz, while the 4- and 6-hour treatment increased the amorphous content of the mine tailings. The solubility of silicon was increased compared to the raw mine tailings and was greatest for the 800 °C with the 2-hour treatment. The quantity of albite was barely affected by the thermal treatment, but the quantity of muscovite increased for 2-hour treatment, stayed mostly the same for the 4-hour treatment, and decreased for the 6-hour treatment. The aluminum solubility was most affected by treat-ment at 800 °C and was greatest when the duration was 2 hours (see figure 4b). The increased quantity of muscovite seemed to contribute the most to this aluminum release.

The treatment at 1000 °C was the only case that affected the albite or muscovite: the amount of albite increased from 9% to 17-20%, and the amount of muscovite decreased from 31% to 13-19%. The solubility of aluminum after this treatment was not improved, which matches with the decrease in quantity of muscovite. The amount of quartz increased in each of the 1000 °C tests as well, and the amorphous content was not strongly affected either way. The solubility of silicon was slightly increased, but not as much as for the 600 °C or 800 °C treatments.

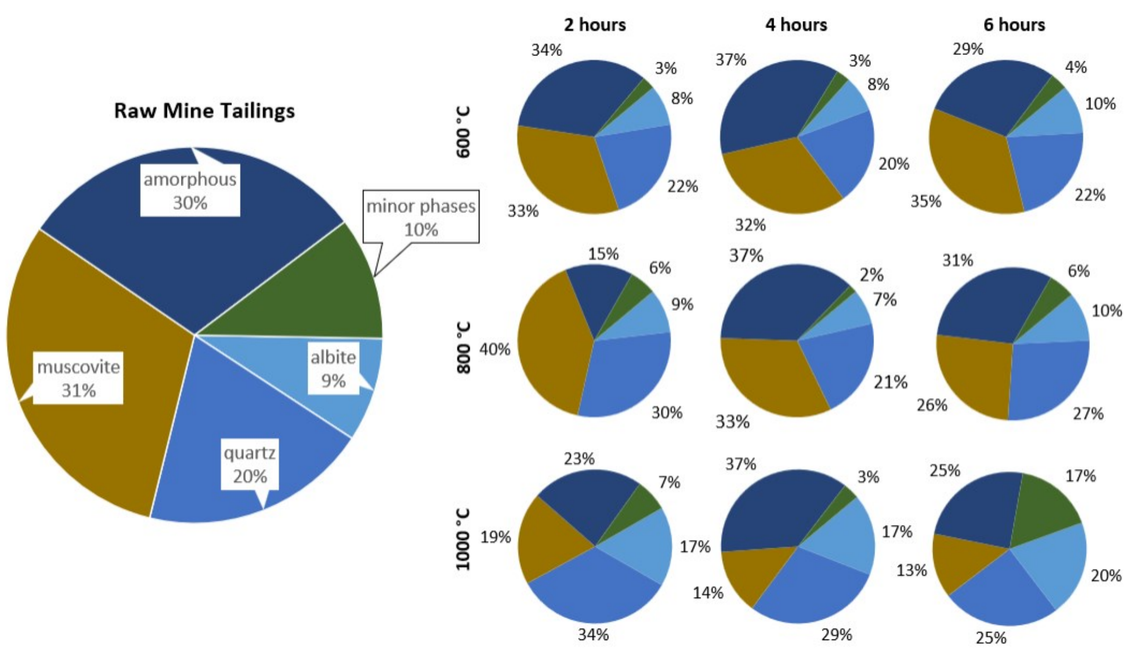

Figure 5: Results of phase determination for thermally activated samples

The ICP results for the thermochemically activated samples are shown in Figure 6. The best combination of parameters appears to be roasting the tailings/sodium hydroxide mixture at 400°C for two hours. This results in a silicon availability of 81.4 mg/L, which is an improvement of about eight times from the raw mine tailings in 1M sodium hydroxide (9.1 mg/L). The aluminum availability increased from 1.72 mg/L to 11.7 mg/L, an increase of nearly seven times. The general trend is that higher temperatures are less effective for solubilizing aluminosilicates, as the 600° treatment seemed to be only half as effective as the 400 °C treatment, and the 800 °C treatment is less effective still. The general trend is the same for silicon and alumi-num, where 400 °C > 600 °C > 800 °C.

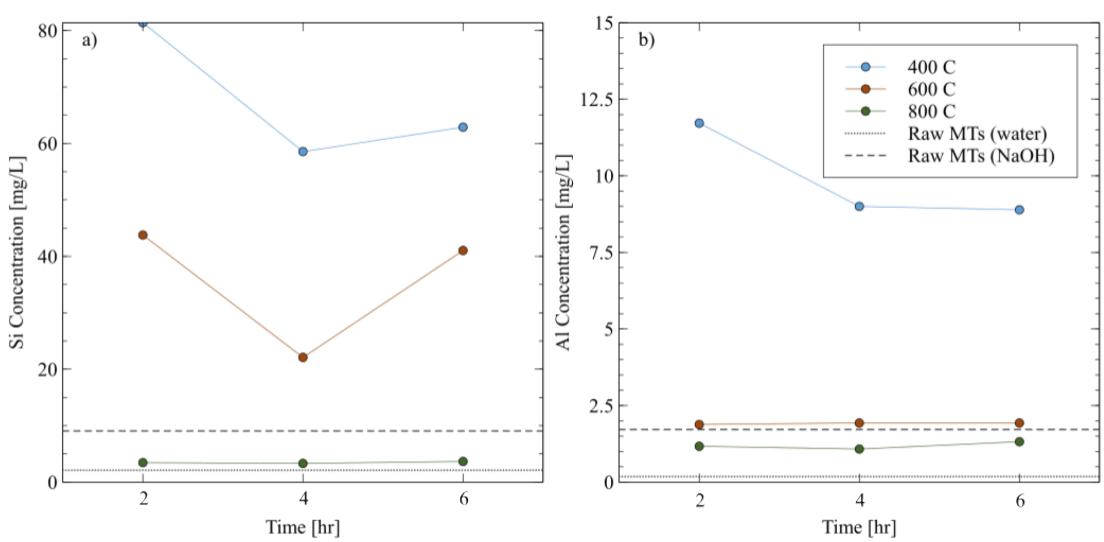

Figure 6: Results of ICP-AES from thermochemically activated tailings for a) silicon and b) aluminum

The phase composition of the thermochemically activated samples is shown in Figure 7. The raw mine tailings have an amorphous content of 30%, with major phases of muscovite (31%), albite (9%), quartz (20%), and calcium aluminum hydroxide-hydrate (CAH-H) (7%). The minor phases are composed of primarily sodium aluminosilicate hydrate, portlandite, calcium aluminum hydroxide hydrate, and calcite.

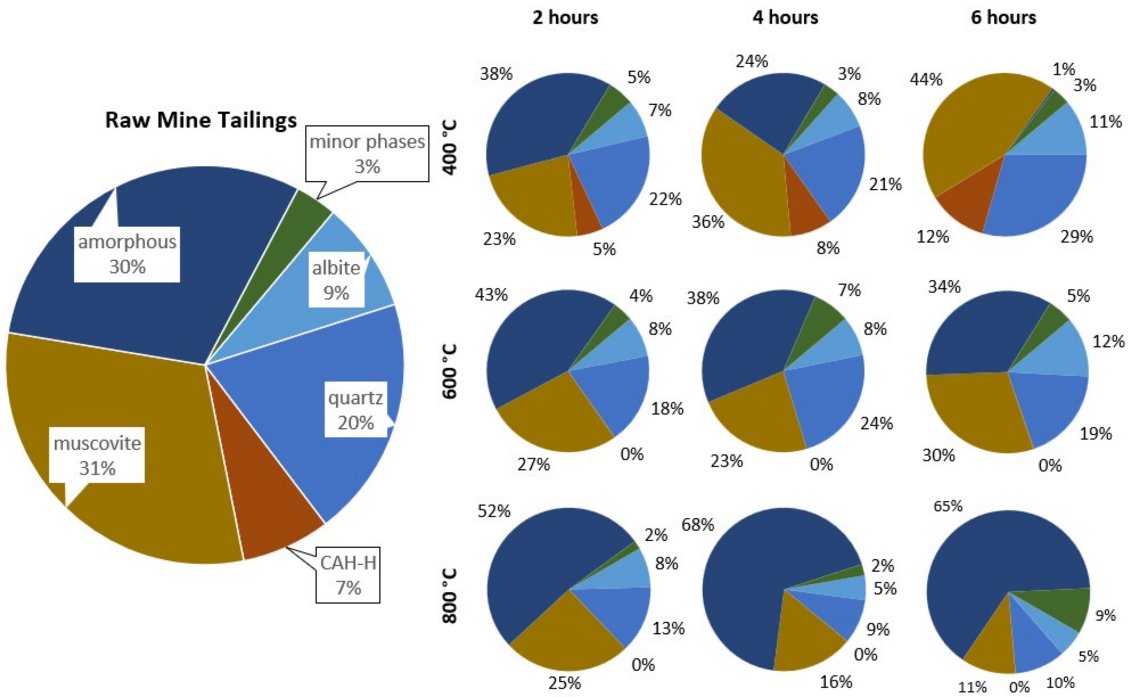

Figure 7: Results of phase quantification for thermochemically activated tailings

The thermochemical treatment increased the amorphous phase in all cases except for 400 °C at 6 hr. There is not a clear directional trend for the time variable, but it appears that a higher temperature produces more of the amorphous phase. The 800 °C treatment increased the amorphous phase by two times or more, while the 400° and 600° treatment increased the amorphous phase by about 1.5 times. The muscovite content seems to be inversely related to the amorphous content, and the amount of quartz was reduced in nearly all cases as well. It seems that the amorphous content is not directly related to the solubility of aluminosilicates, despite the commonly accepted belief that increasing amorphicity always increases a material's reactivity. The testing shows that this may not always be the case, and that more thorough analysis is needed to draw conclusions about the reactivity of mine tailings after pre-treatments.

5 CONCLUSIONS

1. Thermochemical treatment is more effective than thermal treatment for increasing the quantity of amorphous phase and produces more soluble aluminosilicates that can be available for geopolymerization reactions.
2. Aluminum solubility in pre-treated mine tailings is closely related to the quantity of muscovite present.
3. The quantity of amorphous phase is not necessarily related to the availability or solubility of aluminosilicates in pretreated mine tailings, and specific testing is needed to assess the effectiveness of the various pretreatment parameters and methods.
4. Further research is needed to link the results of the qXRD and ICP testing to the geopolymerization potential via production of geopolymer samples from the pretreated tailings. Mechanical and durability testing of those geopolymer samples is ongoing.

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