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TECHNICAL ARTICLE

Inorganic Precipitation of Calcite in Mine Tailings Using Trona

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Abstract

Mine tailings pose environmental risks such as dust emission and acid mine drainage (AMD) and also present geotechnical risks, i.e. tailings dam failures. In-situ cementation of tailings has the potential to reduce the risks associated with such failures as well as the related environmental problems. In this paper, a new inorganic method of inducing tailings cementation is introduced using a naturally occurring mineral called trona ($Na_2CO_3 \cdot NaHCO_3 \cdot 2H_2O$). Three methods of introducing dissolved trona, i.e. injection, slurry mixing, and infiltration, were tested and the results were compared. All methods proved successful on a laboratory scale but with different amounts of precipitated calcite at different sample depths. The precipitation profile distinctions could have applications in solving common problems associated with tailings including dust emission and liquefaction. Additionally, even distribution of precipitated calcite in the slurry mixing method shows potential for AMD prevention.

Keywords Mine waste · Cementation · Carbonate · Liquefaction · Acid mine drainage

Introduction

Precipitation of calcium carbonate in soils is becoming an increasingly common method of soil stabilization that has been shown to increase the strength and liquefaction resistance of sand and fine-grained soils (Pham et al. 2017). These applications could prove very beneficial to the mining industry, particularly in the design of tailings dams where precipitated carbonates could help mitigate geotechnical and environmental hazards by acting as a pH buffer, dust control agent, and/or a physical or chemical stabilizer (Catalan and Yin 2003). For example, tailings liquefaction is a major geotechnical concern in the mining industry as it can trigger tailings dam failure (Petkovšek et al. 2021; Power et al. 2021) by exerting larger horizontal stresses on the dam, i.e. stresses beyond the at-rest pressure that is applied on the dam by the tailings before liquefaction (Martí et al. 2021). Such failures result in larger volumes of debris flows with lengthier runouts affecting larger areas (Ghahramani et al. 2020). Cementation due to calcium carbonate precipitation can increase the liquefaction resistance of tailings before dam failure occurs. Additionally, tailings can present a significant environmental hazard as they often contain acid-generating minerals such as pyrite, pyrrhotite, or arsenopyrite. When exposed, these sulfide minerals react with oxygen and water to generate acid, which lowers the pH and increases the dissolved concentration of toxic metals in runoff to nearby bodies of water (Park et al. 2019). Calcite acts as a buffering agent, increasing the pH and precipitating some of the dissolved metals. It is worth mentioning that the reaction of calcite with sulfuric acid could negate the mechanical improvements of calcite precipitation and should be considered if increasing the liquefaction resistance of tailings is the main goal of precipitating calcite. The use of calcite and other calcium salts in treating tailings is currently practiced in industry and typically involves microbially-induced calcite precipitation (MICP), oxide cementing agents, or direct introduction of calcite.

MICP, also known as microbially-induced calcium carbonate precipitation (MICCP), has shown promising results as a method of soil stabilization (e.g. O'Donnell et al. 2017a, b; Riveros and Sadrekarimi 2020; Portugal et al. 2020). However, some of the major setbacks of MICP methods include the availability of nutrients needed for microbial

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growth, the relatively slow process when compared to other chemical methods, and decreased microbial growth due to the presence of toxins in the tailings (Dhami et al. 2013). Copper concentration in some tailings were shown to slow down the cell growth rate of *Sporosarcina pasteurii*, one of the more common microbes used for MICP via urea hydrolysis (de Oliveira et al. 2021). In addition, MICP methods that employ urea may create high concentrations of ammonium and certain trace elements in groundwater (Proudfoot et al. 2021).

Most inorganic methods of soil treatment using calcium oxide salts i.e. lime, cement, and fly ash, which functionally perform mechanical or chemical treatment but rarely both. Lime is commonly used in the chemical treatment of acid mine drainage (AMD) as it increases pH in the same way calcite does but without releasing CO_2 (Akcil and Koldas 2006). This however does not provide an environmental advantage since lime production releases CO_2. Cements are commonly used in the mechanical treatment of clays, especially in shallow soils (Mohammadinia et al. 2017). While chemically effective, the methods for introducing cement into soils are limited by viscosity, even with thinner grouts that present diminishing returns due to lower concentrations of cement.

In this paper, we introduce a new inorganic method of tailings cementation using trona ($Na_2CO_3{\cdot}NaHCO_3{\cdot}2H_2O$). Trona is a mixed carbonate/bicarbonate mineral that forms in evaporating lakes under a restricted set of chemical conditions (Eugster 1980). One of the largest trona deposits in the world is located in the Eocene Green River Basin of southwest Wyoming (Bradley and Eugster 1969). The major benefit of using trona to precipitate calcite is its relatively high solubility in water, i.e. 200 g/L at 25 °C (Ozdemir et al. 2010), compared to other inorganic compounds commonly used to stabilize tailings, such as lime with a solubility of 1.59 g/L at 25 °C (National Lime Association 2007). This means a higher concentration of carbonate ions required for calcite precipitation will be available in a trona solution than in other inorganic reactants. Furthermore, the significant difference in calcite solubility (0.013 g/L at 25 °C, Blackman and Gahan 2014) and trona results in almost immediate precipitation of calcite after the introduction of a calcium salt e.g. $CaCl_2$. This very high rate of calcite precipitation compared to the MICP and established inorganic methods could be important when moving from laboratory-scale to field-scale. Another possible advantage of using trona is that an additional mole of bicarbonate ion is available (Eq. 1) to increase the overall pH of the solution in the tailings (DiFonzo and Bordia 1998; Maziuk 2007).

$$\begin{aligned}&Na_2CO_3 \cdot NaHCO_3 \cdot 2H_2O + CaCl_2 \\ &\rightarrow CaCO_3 + 3Na^+ + 2Cl^- + HCO_3^- + 2H_2O\end{aligned} \tag{1}$$

Furthermore, one of the challenges associated with carbonate precipitation is the reduction in pore space caused by calcite formation. While the cementing effect of calcite and the reduction in pore space increases the strength of soil, it also decreases the permeability of the soil, creating a barrier for delivering nutrients or chemical constituents to the lower layers of a soil column (Buikema 2015). Therefore, when considering ground improvement methods for tailings, it is important to determine the best time and most suitable method to introduce the cementing agents to the tailings. A common method of transporting tailings from the mill to the tailings pond is to pump them as a slurry through large pipes. Tailings are discharged at the end of the pipes and deposited fluvially with fines traveling further from the point of discharge than the coarse particles (i.e. sand-size). This creates a particle size gradient in the tailings ponds that renders different permeabilities at different distances from the end of the pipe (Kossoff et al. 2014; Wielinga et al. 1999). Therefore, different methods of introducing the cementing agents might be required at different locations. Hence, this paper will also discuss different methods of introducing the cementing agents, i.e. trona and calcium salt solutions, into tailings samples and how it affects the precipitation process.

Materials and Methods

Mill tailings used for this experiment were taken from an open-pit copper mine. Samples were collected immediately after the milling process, i.e. before it was pumped to the tailings pond, to have a representative sample without particle segregation due to fluvial-like deposition at the pond. Sieve analyses and Atterberg limits tests were conducted on the samples according to ASTM D6913 (2017) and ASTM D4318 2005, respectively (Fig. 1). Using the results of these tests, the tailings were classified according to the Unified Soils Classification System (ASTM D2487 2013), to be SM-silty sand. The mineralogy of the tailings is the same as

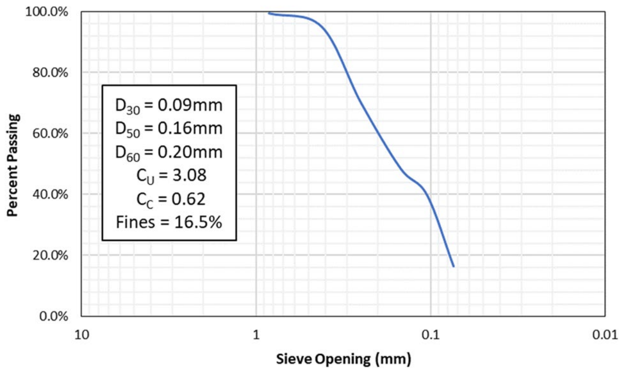

Fig. 1 Grain size distribution for mill tailings

the rock being mined, hydrothermally altered granite, and includes quartz, variably altered feldspars (both plagioclase and alkali feldspar), biotite, sericite, and clays. Further analysis using a shortwave-infrared (SWIR) handheld spectrometer (TerraSpec Halo) indicated that the tailings fine particles are best classified as illite. The tailings have a relatively low pyrite content ($< 2\%$) and contain small amounts of calcite from the hydrothermal alteration. All tailings used in the subsequent experiments were air dried. When deionized water was added back into the tailings, the pH was near neutral (7.07). The carbonate content of the tailings before and after treatment were measured using a rapid carbonate content chamber based on ASTM D4373 (2021). According to ASTM D4373 (2021), the results of this test should be reported to the nearest 1%, and any variations in the data are either due to variations of specimens or due to operator/equipment differences. As all the tests in this study were conducted by the same operator (the first author) using the same equipment, the variations in the results were assumed to be caused only by specimen variations. Also, for each sample, multiple tests were conducted, and the averages were used for comparison. Therefore, it was deemed appropriate to report the average results to two decimal places. All testing was conducted in a laboratory with an average ambient temperature of 23.7° C.

The trona used for the experiments was obtained from a trona mine in Green River, Wyoming. The ore was not treated to increase purity before being used. The carbonate content of the trona rock samples was determined using two methods, rapid carbonate content test as described above and X-ray diffraction (XRD). These combined methods confirmed the sample to be 85.8% ($\pm 0.2\%$) pure trona.

Concentration and Precipitate Crystal Size Experiment

According to O'Donnell et al. (2017a), even a 0.4% increase in calcium carbonate can significantly increase the cyclic shear resistance of treated soils. This resistance increase is dependent both on the concentration and crystal size of the precipitated calcite (Dhami et al. 2013). Therefore, the effects of the trona concentration and $CaCl_2$ on the size of the precipitated calcite crystals were first investigated. Second, the stoichiometric production of calcite had to be determined since trona contains both a carbonate and bicarbonate ion. The stoichiometry for this reaction (Eq. 1) shows a 1:2:2 molar ratio between trona, $CaCl_2$, and the precipitated $CaCO_3$, which assumes that the trona provides the carbonate and the bicarbonate to create an additional mole of calcite. At low pH, off gassing of CO_2 from the bicarbonate may present a concern. However, given the required discharge pH of most tailings, this will likely present no concern for the applications of these experiments. The molecular weight of trona and $CaCl_2$ are 226 and 111 g/mol, respectively. Therefore, the mass ratio of trona, $CaCl_2$, and $CaCO_3$ to produce one part calcite in a sample is approximately 1:1:1. It is worth noting that when this experiment is applied to tailings, a surplus of pore water calcium ions could eliminate the need for calcium salts. However, to ensure maximum efficiency, this ratio will be used for all experiments in this study. To determine the effects of concentration on crystal size in solution, three experiments were performed with logarithmically-related concentrations. Three beakers were filled with 100 mL of DI water and trona sample to create concentrations of 0.4, 4, and 40 g/L. To make sure that all the trona in each beaker dissolved completely, each solution was stirred for 15 min using a magnet spinning at 600 rpm in the solution. After all the trona dissolved, an equivalent mass of $CaCl_2$ was added to each beaker to precipitate calcite. Five minutes after adding the $CaCl_2$, each sample was filtered using a 0.2 µm filter paper. The retained material on each filter was then oven-dried before comparing the sizes of precipitated crystals under a microscope.

Methods of Introduction Experiments

Three methods of introducing the chemical constituents, i.e. trona and $CaCl_2$, into the tailings were devised to mimic potential field applications: (1) injection into the tailings at several different levels via a syringe; (2) infiltration of the solution from the surface; and (3) slurry mixing the tailings and then introducing the constituents. Four beakers were filled with 180 g of tailings placed loosely to a height of 4.5 cm from the inside base of the beaker. The first sample, tagged T5.C, was used as the control experiment where no chemicals were added to the tailings. For this control sample, 110 g of DI water was added to the tailings. Given the desired increase of roughly 1% calcite in each sample and the 55 g of DI water mass to reach saturation in the sample, a 40 g/L concentration was selected for the trona and $CaCl_2$ solutions used in these experiments.

Details of the chemicals used in the four samples are shown in Table 1. Each sample had an equivalent final mass and equal mass of chemical constituents, even though the chemicals and DI water were added in different portions of the procedure for each experiment. Each experiment was started by filling a beaker with 180 g of tailings and 55 g of trona solution. The trona solution was given 5 min to infiltrate the entire sample and settle to the saturation point before the $CaCl_2$ was introduced via the varied methods described below.

The injection method (T5.1) was performed to evaluate the use of a probe to inject treatment solution into the tailings. In the injection method (Fig. 2), a needle was lowered near the bottom of the sample, i.e. 0.75 cm from the base of the beaker, where 18.3 mL of $CaCl_2$ solution was

Table 1 Mass of the chemical constituents used in each experiment

Sample ID	Tailings (g)	Trona (40 g/L) Sol. (g)	$CaCl_2$ (40 g/L) Sol. (g)	$CaCl_2$ (g)	DI water (g)
T5.1	180	55	55	0	0
T5.2	180	55	0	2.2	52.8
T5.3	180	55	55	0	0
Control	180	0	0	0	110

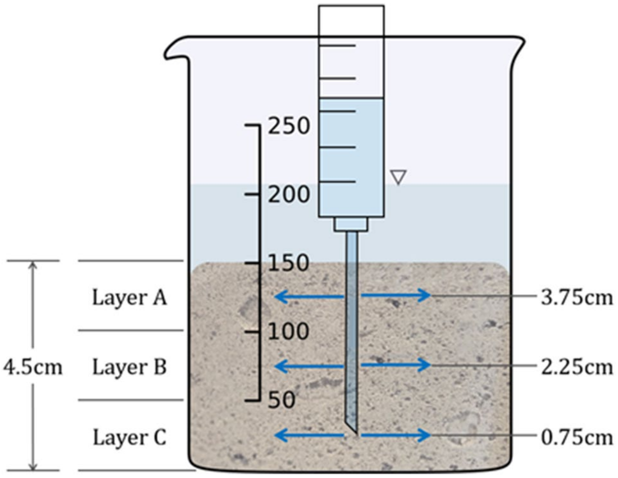

Fig. 2 Injection methods using a syringe at three variable depths

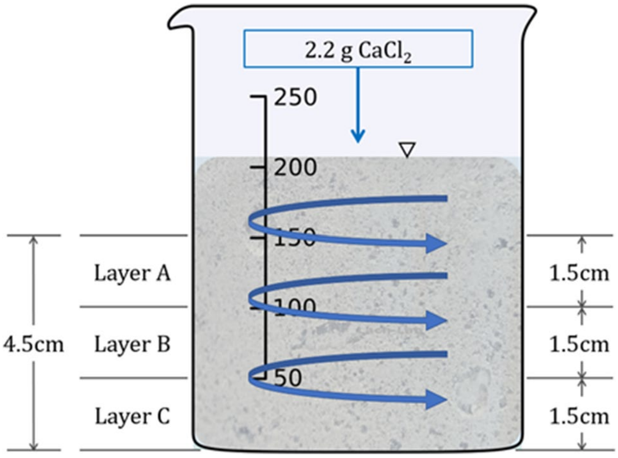

Fig. 3 Tailings layers for carbonate content measurements

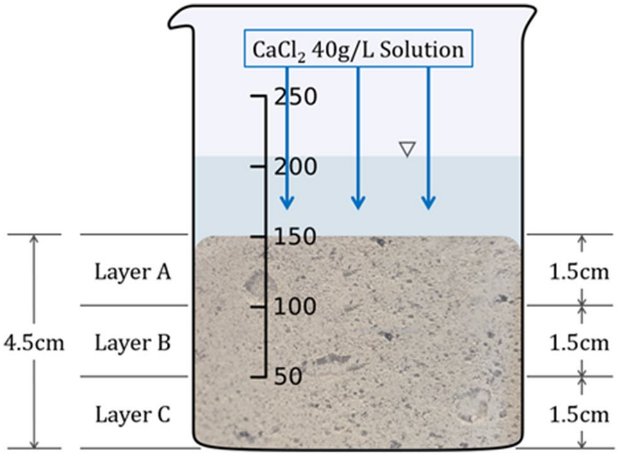

Fig. 4 Infiltration of $CaCl_2$ solution into tailings saturated with trona solution

injected. This process was then repeated at heights of 2.25 and 3.75 cm for a total injected volume of 55 mL across three intervals. This method was tested because if the chemical solution is introduced from one point, the precipitated calcite around the point of introduction reduces the porosity of the sample, which in turn inhibits the flow of the chemical solution to other areas of the tailings. Therefore, calcite precipitation would likely be limited to an area near the point of introduction. This method was an attempt to achieve a more evenly distributed precipitation.

The next introduction method was the slurry mixing method (T5.2). In this method, rather than using a $CaCl_2$ solution, all the DI water (107.8 g) for the experiment was added at the beginning so the tailings could be properly suspended in the beaker (Fig. 3). This mixture was stirred constantly using a magnet rotating at 600 rpm to keep the solids suspended. Once all the particles were in suspension, 2.2 g of $CaCl_2$ were added to the beaker over a period of one minute, after which the mixture was stirred for 5 min. The magnet was then removed, and the sample was allowed to settle. When the magnet was removed from the sample, approximately 0.5 g of magnetic sediments (mostly magnetite) were removed from the sample, leading to a small change in overall mass. This method was meant to simulate the introduction of chemicals into tailings at the end of the milling process or during pumping to the pond.

The third beaker consisted of an infiltration method (T5.3) meant to simulate the introduction of solution at the surface of existing mine tailings, i.e. fluvially deposited tailings. The tailings saturated with trona solution had 55 g of $CaCl_2$ solution added (poured) on top of the sample (Fig. 4).

After the procedures described above were completed, each sample was left to rest for 15 min at room temperature before oven drying. All four samples were oven dried at 57 °C for 48 h after which they were tested for strength using a vane shear device and then separated into layers A–C (see left side of Fig. 4) for carbonate content testing. Overall, 12 samples were tested using a carbonate content chamber providing a three-layered depth profile to determine the variation in precipitation over depth in each sample.

Stochiometric Efficiency and pH Experiment

To determine the rate of precipitation and the effects these experiments had on overall alkalinity, the slurry method was repeated a second time with a pH probe placed in the slurry. The pH was measured at 5-s intervals for the entire period that the $CaCl_2$ was added to the slurry. After the slurry was allowed to settle, the pore water was flushed multiple times to remove any dissolved carbonate or bicarbonate that may have contributed to the carbonate content of the oven-dried sample.

Results and Discussions

Concentration and Precipitate Crystal Size

The results of the microscopic imaging of the three samples used in the concentration and precipitate crystal size experiments are shown in Fig. 5. As can be seen, the variable concentrations used in this study had no significant effect on the size of the precipitated crystals. $CaCO_3$ can precipitate as a variety of polymorphs, i.e. vaterite, aragonite, and calcite, but given the rhombohedral structure of the precipitate seen in Fig. 5, it can be concluded that the $CaCO_3$ precipitate formed as calcite (this was also confirmed by XRD analyses). The average size of the calcite grains affected cementation within the tailings, which could have significantly affected the cyclic resistance increase. This experiment revealed that at these concentrations, one can expect to see calcite grains averaging 5.9 µm in diameter.

Method of Introduction Impacts on the Precipitated Calcite Concentration over Depth

The carbonate content of the control sample was measured, and the results are presented in Table 2. Given the solution concentrations, the mass of tailings, and the 1:1:1 ratio of trona, $CaCl_2$, and $CaCO_3$ used in our experiments, a 0.54% carbonate content increase was expected due to carbonate precipitation, with the potential for another 0.54% carbonate increase due to precipitation of $CaCO_3$ from bicarbonate (Cho et al. 2008). At a relatively high pH (> 9) under lab conditions, bicarbonate will deprotonate to release carbonate, which will likely bond to form additional calcite (Elamin 2011). Therefore, the expected total increase in samples assuming bicarbonate also contributes to the increase is 1.08%. To determine the carbonate content increase in each sample, the average in-situ equivalent calcite content (1.44%) was subtracted from the measured equivalent calcite content of each treated sample (Table 3).

The achieved increase in the carbonate content of the treated samples reveals the carbonate increase must have also included the bicarbonate as the average carbonate

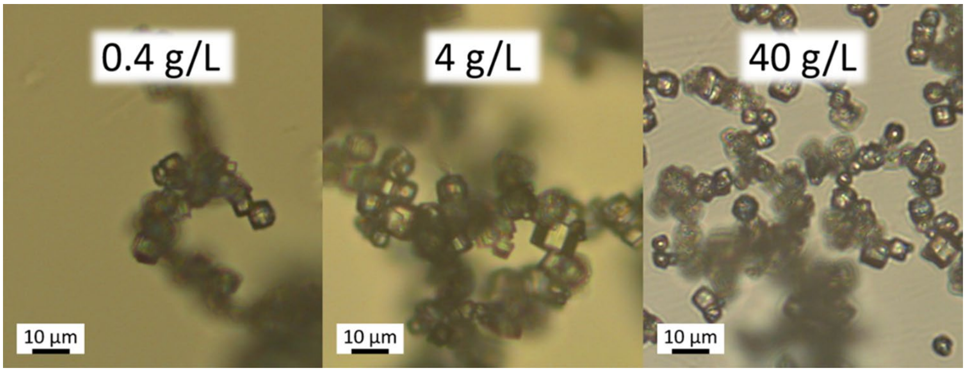

Fig. 5 Calcite precipitate at three different concentrations

Table 2 Average carbonate content for mine tailings sample

Sample ID	Mass (g)	CO_2 (kPa)	Time (min)	Equivalent calcite content (g)	Percent carbonate (%)
Control a	9.981	10.5	30	0.147	1.47
Control b	10.006	10.5	30	0.147	1.47
Control c	10.005	10.0	30	0.138	1.38
Control (avg)	–	–	–	0.144	1.44 (±0.05)

Table 3 Carbonate increase data across each sample layer

Method (sample ID)	Height (cm)[a]	Carbonate content (g)	Carbonate (%)	Carbonate increase (%)
Injection (T5.1a)	3.75	0.289	2.89	1.48
Injection (T5.1b)	2.25	0.244	2.44	1.04
Injection (T5.1c)	0.75	0.209	2.09	0.69
Slurry mix (T5.2a)	3.75	0.249	2.49	1.09
Slurry mix (T5.2b)	2.25	0.244	2.44	1.04
Slurry mix (T5.2c)	0.75	0.253	2.53	1.13
Infiltration (T5.3a)	3.75	0.271	2.71	1.31
Infiltration (T5.3b)	2.25	0.209	2.09	0.69
Infiltration (T5.3c)	0.75	0.200	2.00	0.60

[a] Average height measured from the base of the tailings in the beakers representing each 1.5 cm interval

increase exceeded the expected 0.54% due to calcite precipitation. The average increase in each sample was calculated as 1.07, 1.09, and 0.86% for the injection (T5.1), slurry mixing (T5.2), and infiltration (T5.3) methods, respectively. Apart from the last sample (T5.3), the precipitation appears to have formed at near 100% stoichiometric efficiency. The possible explanation for the discrepancy observed in the last sample is discussed later.

The calcite increase vs. depth in the specimens is plotted in Fig. 6. The results show that each method of introduction followed a different precipitation trend that appears consistent with the visual characteristics of each sample (Fig. 7). The sample treated with the injection method followed a linear path with carbonate decreasing with depth. This is likely the result of greater hydraulic conductivity at shallower depths, which might have been due to the immediate precipitation of calcite at lower depths, the small confining volume of the beaker, or the natural tendency for permeability to decrease with depth. It is also worth mentioning that a significant number of tension cracks were observed in the uppermost layer. This is consistent with a greater increase in the carbonate content at the surface layer. For all treated samples, the cementation caused by the carbonates led to apparent cohesion of the material, which in turn causes tension cracks during the drying process. The slurry mix method resulted in a consistent concentration of added carbonate over depth (Fig. 6), which was expected due to the homogeneity caused by the continuous stirring of the tailings and the chemical solutions.

Visual inspection of the slurry sample also revealed very few tension cracks from local shrinkage in the sample, which is consistent with the homogenous precipitation

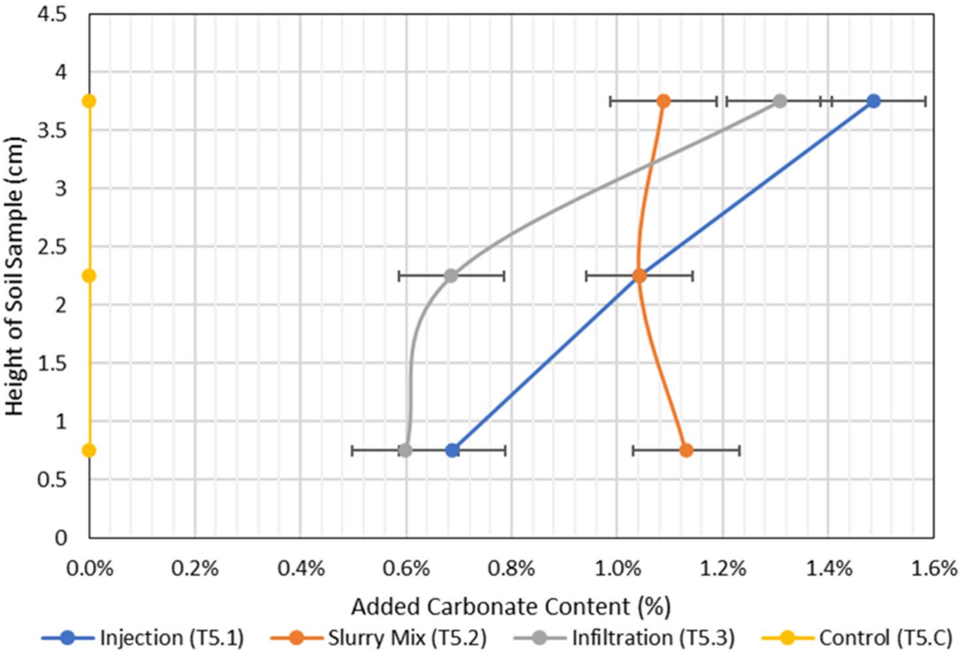

Fig. 6 Profiles for each method of introduction

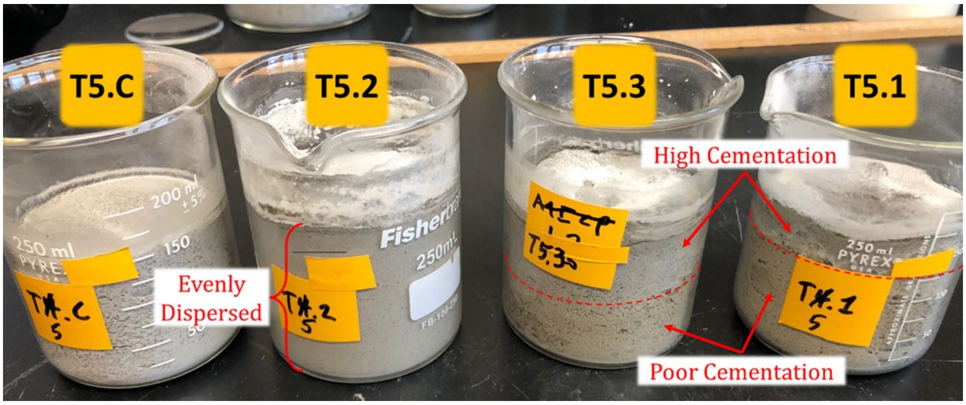

Fig. 7 Oven-dried samples after treatment before layer sampling

Table 4 Strength (Vane Shear) increase in the sample surface layers

Sample ID	Vane shear (kPa)	Strength increase	Added calcite (%)	Method of introduction
T5.1a	368	88%	1.48	Injection (T5.1)
T5.2a	662	238%	1.09	Slurry mix (T5.2)
T5.3a	260	33%	1.31	Infiltration (T5.3)
T5.Ca	196	0%	0	Control (T5.C)

of calcite in this method. In the sample treated by the infiltration method, the carbonate increased exponentially towards the surface. This was also apparent from the dense precipitate layer seen at the surface of the treated sample (Fig. 7). Unlike the samples treated with the other two methods, this specimen only had a measured calcite content increase of 0.86% (80% of the expected increase). This was most likely due to the sampling intervals used in this study. To be consistent among all the experiments, samples for calcite content measurements were taken from three layers of equal thickness, i.e. 1.5 cm, for each specimen. The measured calcite content increase for each interval is therefore an average increase over the whole 1.5 cm of the sampled layer. From the exponential calcite content increase seen in the T5.3 sample and visual inspection of the treated specimen, most of the calcite had precipitated in the upper 2 mm of the specimen. Therefore, the measured average calcite content increase for the top 1.5 cm layer underrepresents this top 2 mm of high concentration. Hence, adding the average calcite content increase measured for the three 1.5 cm layers does not equal the anticipated amount.

Table 5 Precipitation rates for MICP and Trona

Experiment	T5 experiments (avg)	Pham et al. (2017)	O'Donnell et al. (2017a)
Total increase (%)	1.29	0.93	1.94
Increase period (h)	0.033	1560	9600
Increase rate (%/h)	39.09	5.96E−04	2.02E−04

The vane shear test showed an increase in strength at the surface of each sample compared to the control sample (Table 4). Vane shear uses field equipment designed to estimate the strength of granular material on an undisturbed surface. As removing the top layers would disturb the surface of the bottom layers, the vane shear test results would be unreliable for the lower layers. Therefore, only the change in the strength of the top (surface) layers was compared. All samples showed some increase in strength compared to the control due to calcite precipitation. More investigations and more precise strength testing are required to potentially correlate the increase in strength and the amount of precipitated carbonate.

Comparative Methods of Calcite Increase

The most common alternative method for focused calcium carbonate increase found in the literature is MICP. When comparing the applications of this technology to trona, the most apparent distinction is the rate of cementation, distribution, and mechanical improvement. Table 5 shows that the trona methods precipitate calcite far more rapidly than MICP. For the MICP experiment, calcite precipitated closer to the point of introduction following a similar trend to the infiltration method. Further studies comparing these methods will likely focus on the relationship between the method of precipitation and strength increase.

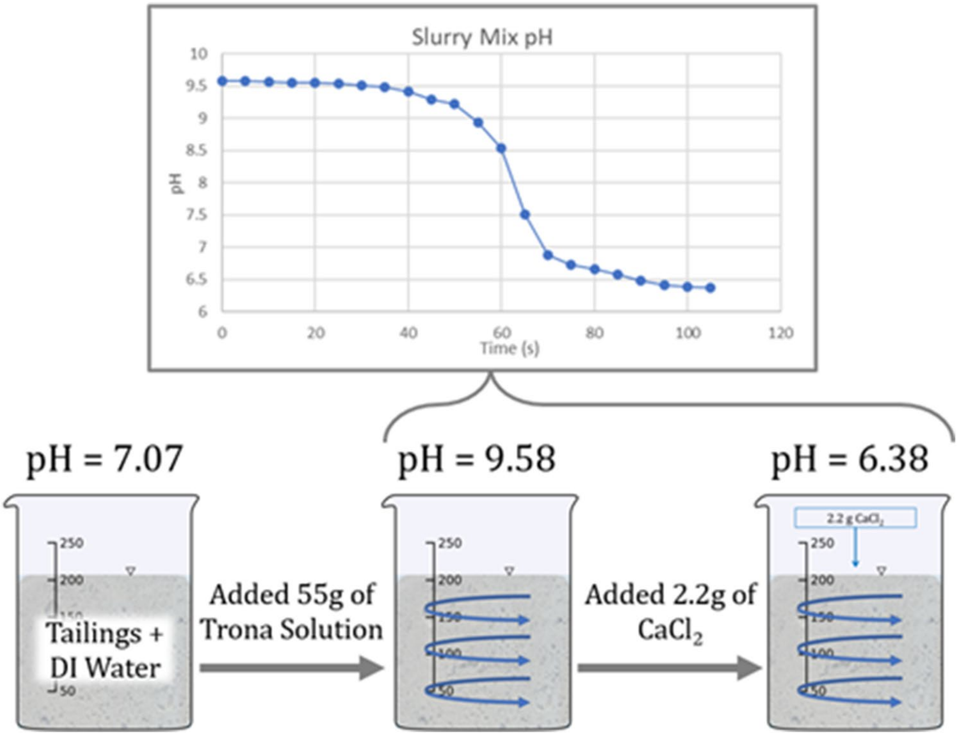

Fig. 8 Calcite forms rapidly after mixing trona and $CaCl_2$

Stochiometric Efficiency and pH Results

Flushing the pore water from the slurry sample did not reduce the added carbonate content (1.08%) compared to the initial slurry mix added content (1.09%), meaning the entire carbonate component in the starting trona was precipitated before the sample was oven dried. The high efficiency of precipitation in these experiments is likely due to a small confining space for experiments. When applied at a larger field scale, the efficiency may drop significantly due to common challenges associated with mixing solutions in a porous media (Piscopo et al. 2013). Figure 8 shows that the precipitate formed almost immediately given the rapid change in pH, which stabilized at 6.38 after all the $CaCl_2$ was added to the slurry mix. The drop in overall pH from 7.07 to 6.38 was likely due to the H^+ ions lost from the bicarbonate that precipitated calcite.

Conclusions

The results of this study show that trona can be used as an inorganic substitute for chemical agents typically used for ground improvement. Tailings treatment via trona and calcium salt is fast and efficient and shows potential to increase the strength of tailings. When considering the use of carbonate precipitation as a treatment method, the method of introduction could contribute significantly to leveraging resources to create efficient engineering solutions. For example, the infiltration method could be an effective dust control measure but appears less effective for stabilizing tailings at depth because the injected chemical solutions tend to flow upward and precipitate more calcite at shallower depths. This was speculated to be the result of decreasing the hydraulic conductivity of tailing with depth. This may be exacerbated in the field where larger differences in hydraulic conductivity exist. However, this method would have to be repeated with a larger lateral area to solidify this assertion. The slurry method can be very effective in improving the overall cementation of new tailings if the chemical solutions are added to the tailings at the end of the milling process or while it is being pumped to the pond. Current slurry methods often use added alkaline materials at this stage to balance pH but the use of trona may prove more effective due to its higher alkalinity and solubility when compared to calcite and lime. Regardless of the method of introduction, an increase of 1% calcite was observed across the entire media of tailings, which significantly improved the neutralizing potential of the tailings. This increase could help mitigate AMD by decreasing the net acid-generating capacity of the tailings.

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