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Hydraulic Conductivity of Mine Waste Treated Using Enzyme-Induced Calcite Precipitation Method Under Various Curing Conditions

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Abstract: The hydraulic conductivity of mine waste soil is considered moderately high attributed to the high percentage of pore spaces. One of the risks associated with this poor property is possible intrusion of contaminated acid drainage into the groundwater or river. Biocementation using enzyme-induced calcite precipitation is a relatively new, more inventive, and environmentally sustainable method compared to the other techniques in improving soil properties. However, limited available data on how this method can be applied in improving heavymetal contaminated mining wastes. This paper summarizes the effect of this treatment, including different cementation concentrations, degree of compaction, curing temperatures and curing durations in reducing the hydraulic conductivity of mining waste. Results obtained indicate a greater effect of 1.0M compared to 0.5M concentration, a degree of compaction of 80% compared to 70%, a curing temperature of 25 °C compared to 15 °C and 5 °C, and immediate reaction effect after 1 then slowed down after 3 and 7-day curing. When compared to control samples, the reduction in hydraulic conductivity ranged from 75.66 to 97.14%. The positive result is attained due to the production of calcite, CaCO₃ that biocemented the soil particles together and reduced the pore spaces, indicated by their content obtained, ranging from 2.0-5.15 % in the treated soil. Visual images through SEM and spectra of x-ray diffraction confirmed the presence of CaCO₃ in soil particles. This work contributed significantly to the study of the properties of copper mine tailings in Ranau, Sabah, including the first study on biocementation of copper mine tailings. The method could be used to reduce the hydraulic conductivity of mining waste soils contaminated with heavy metals. Various scenarios such as curing temperature, duration, cementation concentration and degree of compaction have been proposed to optimize the effectiveness of the treatment.

Keywords: EICP, mine waste, soil contamination, biocementation, hydraulic conductivity

1. Introduction

Hydraulic conductivity, k is the measure of the ease of liquid or gasses to pass through a porous material. In some geotechnical cases, it is utmost important because pervious or semi pervious soils in nature, such as coarse-grained soil, can prevent the increase of high pore water pressure during loading. However, highly permeable soil may also indicate poor strength. The hydraulic conductivity of contaminated soil is dependent on many factors such as particle size

distribution, soil shape, texture and roughness, porosity and tortuosity and the degree of interconnection between the soil particles.

Biocementation treatment such as enzyme- and microbial-induced calcite precipitation (EICP and MICP) are known to improve the hydraulic conductivity and affected by the type of bacteria, treatment method, temperature, bacteria concentration, pH, degree of saturation and most importantly the geochemical properties of soil [1]. However, the hydraulic conductivity of heavy-metal contaminated soils using EICP are relatively new particularly under varying working parameters. In this study, the varying working parameters investigated were the concentration of reagent, the relative density or the degree of compaction, the curing temperature and the curing duration.

2. Research Significance

With increasing impact of heavy metal contaminations and the emerging needs for new land for development, studies on improving the properties of mine wastes are paramount. In the state of Sabah, Malaysia, it is even more critical because the biggest copper mine in Mamut, now with abandoned lakes, is located near several villagers and a deadly earthquake centered in Ranau on 5th June 2015, with 6.0 magnitude earthquake has renewed the cry for prompt action from the authority to provide safer solution in mitigation of mine waste area.

The hydraulic conductivity of mine waste soil is considered moderately high at the range of 3.67E-04 to 1.67E-06 cm/s [23]. The high hydraulic conductivity is attributed to the high percentage of pore space in mining waste soils. One of the risks associated with this poor property, is intrusion of contaminated acid drainage into the groundwater or river that could be utilized by communities living in the surrounding area, Hence the necessity of finding sustainable methods and techniques to reduce the hydraulic conductivity of the mine waste soils.

In general, methods and techniques for soil improvement, in particular to improve the technical properties of soils contaminated with heavy metals, can be divided into physical, chemical or biological techniques. Physical improvement methods are like soil replacement, thermal analysis or electrical repair; Chemical treatments are either by passivation or leaching, and bioremediation methods include phytoremediation (plant) and animal repair. However, these methods are considered expensive for small projects, are not fully effective, or may result in secondary pollution, questionable long-term durability, limited heavy metal accumulation, slow and long treatment cycles [10]. Moreover, different heavy metals may have different effects on the biochemical functions of animals and plants during bioremediation.

Therefore, the above limitations of these practices necessitate research on green and sustainable technologies and hence the use of biocementation processes such as EICP, a biological precipitation technology of calcium carbonate (calcite or CaCO₃).

Research in soil improvement methods is ever-growing includes uses of materials; natural and synthetic, agriculture and industrial-waste based and application techniques; such as chemical injection and application of columns. Most of the these requires substantial energy for implementation, costly and can cause environmental problems. Due to these limitations, alternative method such as EICP is an exciting opportunity as it is sustainable and environmentally friendly. It is possible through combination of geochemistry, microbiology and geotechnical engineering.

While EICP is gaining popularity to treat problematic soil, its potential in treating contaminated soil, particularly hydraulic conductivity of mine waste is still not explored fully. Thus, this paper reports the factors effecting the improvement of the hydraulic conductivity include the concentration of EICP cementation solution, degree of compaction, temperature and curing durations. In addition, the correlation between the changes in hydraulic conductivity to the amount of calcite produced were also discussed. The specific objectives are considered relatively new and the outcomes are significant and beneficial in civil and construction engineering.

3. Literature Review

There are particular actions involved in each stage of mining that have an impact on the environment. The general mining industry phases included the following: exploration, development, exploitation, processing, closure, and rehabilitation [2]. The end by-product of these stages are mine waste soils with various levels of contamination. Due to the movement of contaminants occurs along the hydrogeological parameters of soils and aquifers, proper study on using improvement method such as enzymically induced calcite precipitation (EICP) is beneficial. EICP utilizes the use of plant-based enzyme to form calcite (CaCO3) that locks the soil particles closer, causing an improvement to the soil strength and stiffness and reduce the hydraulic conductivity.

The mechanism of EICP processing is given by Eq. (1) to Eq. (6) [3]. The supplied urease enzyme hydrolyzes urea to produce ammonia and carbamate (Eq.1), and then the carbamate spontaneously decomposes into ammonia and carbonic acid (Eq. (2)). Ammonia is then hydrolyzed to ammonium and hydroxide ions, raising the pH of the sample and promoting carbonate precipitation (Eq. (3)).

$$CO(NH_2)_2 + H_2O \xrightarrow{\text{urease enzyme}} NH_3 + NH_2COOH$$
(1)

$$NH_2COOH + H_2O \rightarrow NH_3 + H_2CO_3 \tag{2}$$

$$2NH_3 + 2H_2O \rightarrow 2NH_4^+ + 2OH^- \tag{3}$$

$$2OH^{-} + H_2CO_3 \to CO_3^{2-} + 2H_2O \tag{4}$$

$$CO(NH_2)_2 + 2H_2O \to CO_3^{2-} + 2NH_4^{H+}$$
 (5)

The carbonic acid dissociates into inorganic carbonates (Eq. (4) and Eq. (5)) and supplying calcium chloride anhydrous (CaCl2) will provide calcium ions that combine with the carbonate ions to precipitate calcite, CaCO3 (Eq. (6)).

$$Ca^{2+} + CO_2^{2-} \to CaCO_3 \tag{6}$$

The calcite generated from the chemical reaction is acting as the bonding and cementation agent for soil particles. Due to these reactions, the properties of the treated soil altered in many forms include reduction in permeability, compressibility and volumetric responses and increase in stiffness and shear strength.

Studies showed that bioremediation treatment on residual soil reduced the hydraulic conductivity by 54-74% [4] on sand specimens' improvement by 0.09-0.15 ratios [5]. Both reductions were attributed to the natural calcite forming observed in the particle-particle contact per unit volume and pore spaces in soil specimens. In addition, it decreases the average coefficient of permeability of clean, cohesionless sand by 83%, from 1.15E-7 m/s to 1.92E-8 m/s [6]. The aggregation of mass associated with the addition of cementation solution and reagent were shown to be the significant factor.

Using the same treatment on porous media containing glass beads between 0.01 mm and 3 mm diameter, it was observed that the hydraulic conductivity reduced from between 8.38E-1 cm/s 3.27E-4 cm/s to between 3.7E-1 cm/s to 3.07E-5 cm/s. This was attributed to the precipitate aggregated between the glass bead that increase their diameter rather than formation of CaCO3 crystal [7]. Experimental investigation on desert aeolian sand found that increasing the biomediated concentration solution reduced the permeability significantly from1.08 x 10E-3 cm/s to range 2.17E-5 cm/s to 9.22E-6 cm/s [8].

For contaminated soil, investigation using spiked-up native Indian cohesive soil reduces the permeability of soil up to 47 folds and swelling pressure by 4-fold. The mark reduction was even more noticeable when casein protein is added with reduction from 5.3E-10 cm/s to 1.12E-10 cm/s red soil and from 7.83E-11 cm/s to 6.31E-11 cm/s in black soil [9].

Several factors attributed to the effectiveness of EICP treatment include particle size, shape and distribution patterns [10]. In addition, type of biocementation enzyme, treatment method, temperature, cementation concentration, pH level and degree of saturation are also critical [1]. As different soil types can produce different responses of the biocemented soil, research on factors affecting the treatment of heavy-metal contaminated soil, particularly mining waste is crucial. Research in soil improvement methods is ever-growing includes uses of materials; natural and synthetic, agriculture and industrial-waste based and application techniques; such as chemical injection and application of columns. Most of the these requires substantial energy for implementation, costly and can cause environmental problems. Due to these limitations, alternative method such as EICP is an exciting opportunity as it is sustainable and environmentally friendly. It is possible through combination of geochemistry, microbiology and geotechnical engineering.

4. Research Methodology

4.1 Materials

Soil was collected from Lohan Dam, a former storage facility for Mamut Copper Mine, located in the state of Sabah, Malaysia (Fig. 1(a)), which was abandoned in 1999. The sampling location was within the perimeter of abandoned dam facilities at approximate GPS of 6° 0' 45.936" N, 116° 44' 20.004" E.

The EICP solution used for biocementation treatment consists of urease enzyme (Fig. 1(b)), urea, and anhydrous calcium chloride. The urease enzyme of 3g/L is added just before adding to the soil sample to avoid early precipitation of CaCO3. Urease enzymes and chemical reagents were purchased from the Fischer Scientific Company in the United States and are 99% pure. The urease enzyme is of plant origin and has been extracted from *canavalia ensiformis* (jack bean) and has been reported to have an activity of 3500 U/g.





4.2 Characterization and Engineering Properties

Prior to the EICP treatment, soils were classified and characterized according to the Unified Soil Classification System (USCS). Further engineering property tests were conducted according to standard, and procedure listed in Table 1. The level of heavy metal contamination was determined by inductively coupled plasma optical (ICP-OES).

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Test	Reference	
All Soil Classification Tests	BS1377-2:1990	
Calcium Carbonate Content	Adopted Dejong et al. [1]	
Metal-Spiking	Adopted Moghal et al. [9]	
Leaching Test	Adopted Baba Musta et al. [23]	

Table 1 - Standard and procedure for engineering properties testing

4.3 EICP Treatment Series

The cementation solution was mixed with the sample, placed in a permeable cell and compacted into three layers to achieve a density (70, 80) compared to the optimum moisture content pre- determined in compaction test. Each layer of the mixture was carefully manipulated to achieve the final predetermined height. The cell is aged at various curing temperatures (5, 15, room) and days (1,3,7), and then subjected to a hydraulic conductivity test according to BS1377. Two different equimolar concentrations (1M and 0.5M) were used in the treatment process, according to table 2. Acid Washing Method were then used to determine the calcite or Calcium Carbonate Content (CCC) of treated samples.

Table 2 - EICP treatment series				
Series	Cementation Solution (Molar, M)	Relative Density (%)	Curing Temperature (°C)	
Raw 1	-	-	Room	
Raw 2	-	70	Room	
M05-L05	0.5	70	5	
M0.5-L15	0.5	70	15	
M0.5-L25	0.5	70	Room	
M0.5-M05	0.5	80	5	
M0.5-M15	0.5	80	15	
M0.5-M25	0.5	80	Room	
M1.0-L05	1.0	70	5	
M1.0-L15	1.0	70	15	
M1.0-L25	1.0	70	Room	
M1.0-M05	1.0	80	5	
M1.0-M15	1.0	80	15	
M1.0-M25	1.0	80	Room	

4.4 SEM Images

A scanning electron microscope (SEM) of Hitachi SU8020 model capable of magnification up to 10000x specification was used to produce high-resolution images and precisely measure very fine features on the surface of the soil sample.

4.5 X-Ray Diffraction

X-ray diffractometer of Rigoku SmartLab® model and specification was used for identification of materials based on their structural properties and atomic arrangement.

5. Results and Discussion

5.1 Soil Characterization and Chemical Properties

Fig. 2 shows the particle size distribution of the mine waste sample while Table 3 shows the mine waste properties. It is classified as SM by USCS due to presence of silt in predominantly sandy soil.

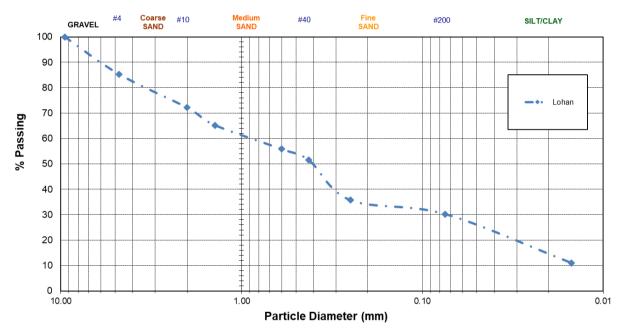


Fig 2 - Particle size distribution of Lohan Dam mine waste soil

Table 2 Mine waste properties

Table 3 - Mine waste properties		
Soil Properties	Value	
USCS Classification	SM	
LL, %	27.0	
PL, %	23.5	
Specific Gravity	2.66	
Organic Content, %	0.77	
pН	6.18	
MDD, kg/m^3	1640	
OWC, %	16	

PL, %	23.5
Specific Gravity	2.66

Meanwhile, chemical test result shown in Table 4 indicate presence of eight heavy metal elements, with the highest being iron (Fe) at 2916.9 mg/L. Also note that despite being a mine waste for copper extraction, the amount of copper (Cu) at 4.396 mg/L detected in the spoil properties is still high. By comparison with standard with Department of Environmental (DOE), Malaysia, presence of arsenic (As) at 0.323 mg/L is considered above the safety standard.

Table 4 - Heavy metals element		
Heavy metal (mg/L)	Level	
Arsenic, As	0.323	
Cadmium, Cd	-	
Cobalt, Co	-	
Chromium, Cr	0.591	
Copper, Cu	4.396	
Iron, Fe	2916.9	
Manganese, Mn	3.698	
Nickel, Ni	0.378	
Lead, Pb	0.541	
Zinc, Zn	0.560	

Table 4 - Heavy metals element

5.2 Hydraulic Conductivity of EICP Treated Lohan Mine Waste

Fig. 3 and Fig. 4 show the hydraulic conductivity, k of Lohan soil treated with EICP. Raw 1 has k of 3.77E-04 m/s on 1-day curing, and 3.50E-04 m/s on 3-day and 7-day curing. It can be observed that the general trend showing the reduction of hydraulic conductivity over time. The decrease on 3-day and 7-day could be due to rearrangement of soil particle in the sample during curing that reduces the volume of the void. Meanwhile, compacted Raw 2 showed k of 1.00E-04 m/s for all 1,3 and 7-day curing. Because the soil is compacted that reduces the void ratio, no significant changes in k in observed in all curing days.

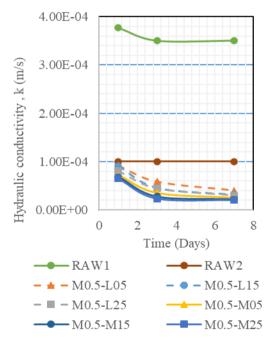
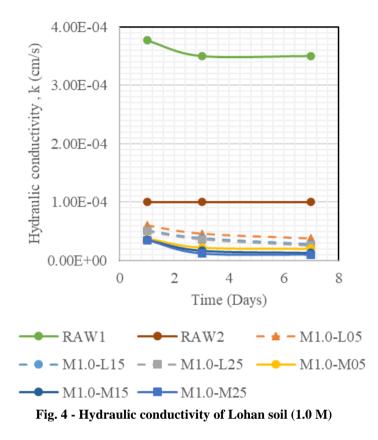


Fig. 3 - Hydraulic conductivity of Lohan soil (0.5 M)

5.2.1 Effects of Cementation Concentration

Fig. 3 shows the reduction in hydraulic conductivity for all series for 0.5M. The highest is observed in M0.5-M25 at 6.51E-05 (1-day), 2.31E-05 (3-day) and 2.02E-05 (7-day) m/s, equivalent to 82.73, 93.34 and 94.21% reduction compared to Raw 1 and 34.91, 76.71 and 79.75% reduction compared to Raw 2. Meanwhile, lowest reduction is observed in M0.5-L05 at 9.18E-05 (1-day), 5.85E-05 (3-day) and 4.03E-05 (7-day) m/s, equivalent to 75.65, 83.30, and 88.50 % reduction compared to Raw 1 and 8.22, 41.54 and 59.74 % reduction compared to Raw 2. Similar observation can be made soils treated with 1.0M (Fig. 4).

All treatment series have effectively changed or reduced the soil permeability. However, the effect of cementation concentration is greater in 1.0M series due to higher supply of materials. These outcomes reinforced the findings that supplement of greater sources or concentration of enzyme or bacteria can produce more and larger $CaCO_3$ crystals [11]. The greater amount of $CaCO_3$ is able to create more bonding between the particles and therefore close more voids that subsequently reduced the hydraulic conductivity.



5.2.2 Effects of Degree of Compaction

For soil treated with 0.5M concentration (Fig. 3), the highest reductions of hydraulic permeability for 1-day, 3-day and 7-day curing are observed in M0.5-M25, M0.5-M15 and M0.5-M05, all compacted at 80%, while the lowest reductions are observed at M0.5-L05, M0.5-L15 and M0.5-L25, all compacted at 70%. Compared to Raw 1, the reduction in all series compacted at 70% and 80% are ranging 75.65-92.71% and 80.94-94.21% respectively, while compared to Raw 2, the reductions are 8.22-70.12% and 28.15-79.76% respectively. Similar trend is observed in 1.0M (Fig. 4) concentration.

The significant changes in their hydraulic conductivity attributed to the decreases in porosity upon compaction. Previous study reported the differences in porosity of mine wastes compacted under different compactive energy [12]. Similarly, in a microstructure and porosity study using analytical permeability model, it was observed that various degrees of compaction (85, 95, 100, and 105%) affects the morphological parameters of the porous network [13].

5.2.3 Effect of Curing Temperature

Samples cured under 5 °C, i.e M0.5-L05, M0.5-M05, M1.0-L05, M1.0-M05 (both Fig. 3 and Fig. 4) show reduction of hydraulic conductivity after curing compared to Raw 1. The reductions are 75.66, 80.94, 84.04 and 89.87 % respectively on 1-day; 83.30, 89.86, 86.76 and 93.51 % respectively on 3-day and 88.50, 92.71, 89.12 and 94.12 % respectively on 7-day. Lower percentage of reduction is observed on their comparison to compacted Raw 2. Similar trend is observed for samples cured under 15 °C. However, the highest reduction in is observed for sample cured under room temperature (average 25 °C).

These findings supported previous notion that the rates and temperature dependence of biocementation treatment. The reaction was found to be more effective under higher temperature. The optimum temperature however varies depending on materials and treatments. The reaction is marginally higher in temperature closer to 30 °C on porous media [14] and the optimum urease activity increased with increasing temperature from 10 until reaching the peak at 60 °C on semi-arid tropical soil [15].

5.2.4 Effects of Curing Duration

The hydraulic conductivities of Lohan series (Fig. 3) M0.5-L05, M0.5-L15, M0.5-L25, M0.5-M05, M0.5-M15 and M0.5-M25 are 9.18E-05, 9.08E-05, 8.07E-05, 7.18E-05, 6.82E-05, and 6.51E-05 m/s respectively on 1-day. The values dropped to 5.85E-05, 4.63E-05, 4.46E-05, 3.55E-05, 2.80E-05 and 2.32E-05 m/s on 3-day and maintained at 4.03E-05, 3.21E-05, 2.99E-05, 2.55E-05, 2.23E-05 and 2.02E-05 m/s respectively on 7-day. Compared to Raw 1, the reduction of

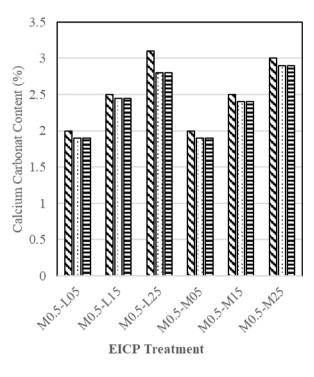
hydraulic permeability is at range 75.65-82.75%, 83.30-93.35% and 88.49-94.22% on 1-day, 3-day and 7-day respectively. Similar trend is observed in comparison with Raw 2.

The benefit of all treatment series occurred early because the cementation reaction is immediate. The effect of curing duration observed on first 24 hours then slowed down after 3 and 7-day curing. Compared to Raw 1, the 1-3 day and 3-7-day drop is at 9.46% and 3.13% average respectively for 0.5M, and 3.73% and 1.79% average respectively for 1.0M (Fig. 4). Similar pattern is observed for comparison with Raw 2. This indicate that the rate of CaCO₃ precipitation is highest during the first 24 hours and subsequently the rate dropped after 3-day and 7-day curing.

The findings are consistent with the previous biocementation study on residual soil, which indicate reduction of hydraulic conductivity on early stage (1-day) attributed to the urea hydrolysis but the reaction but slowed down after 3-day and was maintained after 7-day and beyond [16]. This probably indicate the lack of additional calcium carbonate crystals precipitated in soil [17].

5.3 Calcium Carbonate Content

The impact of the EICP treatment to the treatment series are associated with precipitation of $CaCO_3$, of which their effectiveness is link to the amount they are produced. Fig. 5 (0.5M) and Figure 6 (1.0M) show the amount of $CaCO_3$ produced by biocementation of Lohan soil.



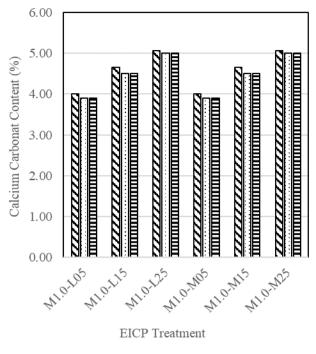
■Day1 ■Day3 ■Day7

Fig. 5 - Calcium carbonate content of treated Lohan soil (0.5M)

Higher CCC is observed in cementation concentration of 1.0M The is due to the higher supply of biocementation materials. The significant amount of calcite generated in both 0.5M and 1.0M is also previously observed in as study on permeability of porous media, however their efficiency is reportedly low compared to concentrations [18], [19].

Between the two compaction degrees, the CCC are the same for 5 °C and a slight edge for 70% for 15 °C and room temperatures attributed to the differences in void ratio of the two compaction energies. Despite the compaction efforts, formation of calcite is still possible because urease is small in size and highly soluble, therefor it allows the cementation solution to penetrate through the pore throat of soils, even for sample containing finer particles [18] such as Lohan. In addition, premixing the soil before compaction have proven that that the cementation solutions are homogenously distributed between the soil particles.

Based on curing durations, immediate reaction of EICP occurred after 1-day curing. The constant value of CCC thereafter (3 and 7-day) probably indicate the lack of additional calcite precipitation in soils. Amongst the working parameters investigated, temperature is the most influential factor to the amount of calcite precipitated. The CCC is highest in room temperature, indicate that the cementation reaction is optimum in temperature averaging 25 °C compared to 15 °C and 5 °C.



■Day1 □Day3 ■Day7



5.4 Relation Between Calcium Carbonate Content and Hydraulic Conductivity

Fig. 7 and Fig. 8 shows the effect of EICP treatment to the hydraulic conductivity and calcium carbonate content of Lohan soil treated with 0.5M cementation concentration under three curing durations. For all six series good correlations were observed between the value of hydraulic conductivity to the CCC. The amount of calcite generated and hydraulic conductivity are proportional, with values of $R^2 > 0.80$.

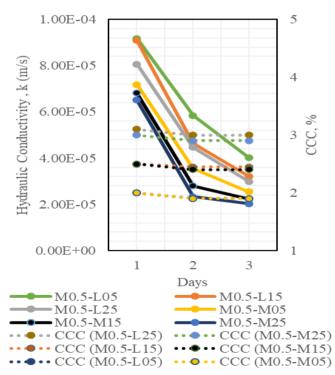


Fig. 7 - Hydraulic conductivity and calcium carbonate content (0.5M)

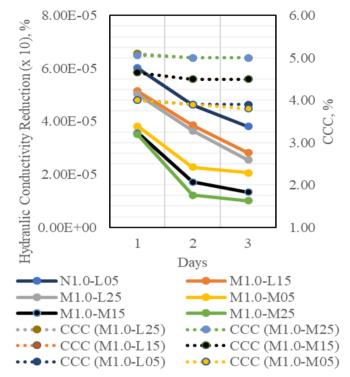


Fig. 8 - Hydraulic conductivity and calcium carbonate content (1.0M)

This is observed in all percentages of calcite and treatment series. However, for both cementation concentration, series treated with higher degree of compaction (80%) and higher temperature (room temperature) showed higher R^2 . The good correlation in this research mirrored that of studies on bio cementation of tropical residual soils. It was previously reported that good correlations ($R^2 = 0.65$) were observed between the calcite content and its reduction of hydraulic conductivity, particularly for calcite content beyond 1.0%. [5].

5.5 Scanning Electron Microscopy Images

Scanning electron microscopy (SEM) analyses were carried out on selected Lohan soil samples to visualize the formation of calcite bonds and their distribution are shown in Fig. 9(a) for original specimen and Fig. 9(b) for treated specimen.

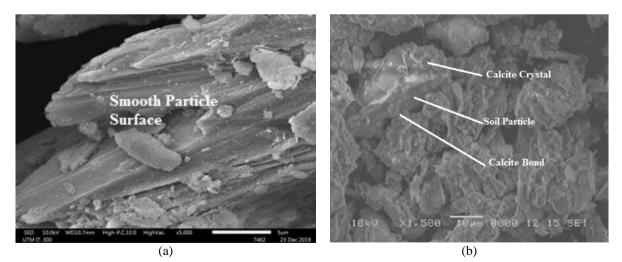


Fig. 9 - (a) SEM of original specimen and; (b) SEM of treated specimen

Treatment specimens show abundant calcite crystal on surfaces and forming bonds between the particles. The shape of precipitated calcites was observed to be in prismatic crystals bladelike form, with typical dimension estimated varied from 3 μ m to 10 μ m. The bladelike form was identical to the calcite form in biocementation of residual tropical soil [19], [20] and differ to the smooth rounded calcite formed in biocementation of sands [21].

5.6 X-Ray Diffraction Spectra

Fig. 10 shows the spectra of $CaCO_3$ crystals produced during biocementation process. The spectra confirmed that that calcite was produced when Lohan soil was treated with cementation solution. In addition, vaterite and aragonite (both $CaCO_3$) were the other predominant polymorphs detected.

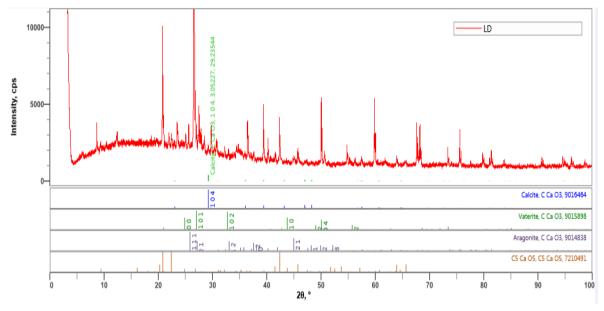


Fig. 10 - X-ray diffraction of treated specimen

The production of different polymorph during the precipitation of CaCO₃ are not well established, but previous research demonstrated that different parameters such as cell surface characteristics, soil composition, enzyme activities and extracellular polymeric substance have effect on the morphology of the precipitated particles [22].

6. Conclusion

This research contributes significantly to the study on the properties of copper mine wastes from Ranau, Sabah include the maiden study on biocementation of copper mine wastes. Biocementation by EICP method has been utilized to decrease the hydraulic conductivity of heavy-metal contaminated mine waste soil. Different scenarios have been proposed to optimize the effectiveness of EICP treatment i.e curing temperature, duration, cementation concentration and degree of compaction. From the results and discussion, following conclusion can be drawn:

- All treatment series show reduction in hydraulic conductivity. For 0.5M and 1.0M series, the reduction compared to control sample is ranging 75.66-94.22% and 84.04-97.14% respectively due to calcite precipitation.
- Results obtained indicate greater effect of 1.0M compared to 0.5M, compaction of 80% compared to 70%, curing temperature of 25 °C compared to 15 °C and 5 °C. Immediate reaction is observed after 1-day then slowed down after 3 and 7-day curing.
- The findings are supported by the amount of CaCO₃ content obtained ranging 2.0 3.1% and 3.9-5.15% for 0.5M and 1.0M series respectively, SEM images and x-ray diffraction.

Acknowledgement

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