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Improving CBR Parameter of Expansive Soil Using the Carbonate Precipitation Method with Tofu Waste as a Biocatalyst

Heriansyah Putra [1*], Alfaris B. Arrazzaq [1], Raihan M. I. Hidayatullah [1], Maulina Lamuse [1], Fauzan R. Ananda [1], Angga Prayoga [1]

[1] *Department of Civil and Environmental Engineering, IPB University, Dramaga, Bogor 16680, Indonesia.*

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Abstract

Expansive soil is problematic from an infrastructural perspective, including the subgrade of pavement construction. The high swelling and shrinkage of this soil promotes subgrade imbalance, resulting in severe pavement construction problems. One potential soil improvement method is the carbonate precipitation method; however, this method requires a catalyst. This research aims to evaluate the use of tofu waste as a biocatalyst in the carbonate precipitation method to improve expansive soil. Several variations of tofu waste reacted with reagents (urea and calcium chloride) as treatment solutions. Soil identification, hydrolysis, precipitation, Atterberg limits, California bearing ratio (CBR), scanning electron microscopy (SEM), and X-ray diffraction (XRD) tests were performed. The results showed that the optimum tofu waste concentration was 40 g/L. The swelling ratio in the soil treated with carbonate decreased by 12.5%. The CBR value of the treated soil also increased by 23.9%. The SEM and XRD analysis results showed the formation of aragonite, calcite, and vaterite. Moreover, this study confirmed that tofu waste is a promising biocatalyst for carbonate precipitation.

Keywords: CBR; Carbonate Precipitation; Expansive Soil; Swelling Ratio; Tofu Waste.

1. Introduction

Unstable soil structures cannot support traffic loads and frequently cause pavement problems such as settlement and cracking. The Ministry of Public Works and Public Housing of the Republic of Indonesia reported that in 2022, 62.35% of the total length of national roads for each province of 46964.77 km would still need to meet the required standards [1]. Poor subgrade conditions can result in pavement problems such as cracks and potholes [2]. A frequently problematic subgrade that causes damage is expansive soil with swelling characteristics [2, 3]. The Ministry of Energy and Mineral Resources (2019) reported the spread of expansive soil throughout Indonesia [4]. Expansive clay is strongly influenced by its shrinkage and expansion properties, which cause subgrade imbalances on roads, resulting in damage [5]. Montmorillonite minerals affect the expansion and shrinkage properties of expansive clays. Ali et al. [6] reported that the higher the number of these minerals in clay soil, the greater the potential for swelling. This condition changes the soil water content, influencing its structure [7]. Swelling of the soil occurs when the water content in the soil increases and shrinks when the soil loses its moisture content. These phenomena significantly impact the stability of road construction.

* Corresponding author: heriansyahptr@apps.ipb.ac.id

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The subgrade significantly influences the stability of the flexible pavement and 50–70% of the distribution load [8]. Flexible pavements on expansive soils are very prone to damage [9]. A solution is required to overcome road damage by improving the subgrade before construction [10]. One of the commonly used soil improvement methods is adding compacted lime granules [11]. Stabilization with lime effectively reduces the possibility of swelling and shrinkage in expansive soil [11]. Alternatively, various new green binders, such as fly ash, ground bottom ash, and blast furnace slag, can be used for stabilization [12, 13]. However, such an alternative requires activators, such as lime, to effectively mobilize pozzolanic activity [14]. Fly ash with an activator, such as lime, can improve the high and low swelling potentials [15]. However, this method has some drawbacks. For instance, limestone granules are difficult to mix, require heavy equipment, and are difficult to reach into the ground. It is not easy to mix soil with lime to achieve homogeneity or spread it evenly [16]. Using lime manually is also costly and energy-intensive. Therefore, an alternative method is required.

Recently, the carbonate precipitation method has been widely used to add a binder to problematic soils [17, 18]. A pure urease enzyme solution is injected to hydrolyze urea to produce calcium carbonate ($CaCO_3$) deposits that fill the pore space of the soil [17]. This method is applicable because it is easy to reach the soil pore spaces, making the soil mixture more homogeneous. However, high-purity urease enzymes are very expensive and uneconomical for large-scale construction [19]. Other potential substitutes for urease are plant materials. Some of them, such as jack beans, cabbage, watermelon seeds, and soybeans, have been developed. Soybeans are effective biocatalysts. With a single injection, it can improve the strength of treated sand by 2-fold [20, 21]. Pratama et al. [21] extracted soybean using the centrifugation method and then mixed it with the reagent to produce calcite precipitation. However, there is still a high demand for comestibles for soybeans. According to the Ministry of Agriculture (2020), Indonesia needs 2,819 tons of soybeans, 94% of which are used as food ingredients [22]. Therefore, alternative sources of biocatalysts for the calcite precipitation method need to be considered. Alternatively, tofu is one of the most common food ingredients processed from soybeans. Tofu production generates tofu waste that is not used [23]. According to the Ministry of Health (2018) [24], tofu waste contains 88% of the protein found in soybeans, making it a potential substitute for urease.

In terms of material potential, this study evaluates the possibility of tofu waste as an alternative material to replace urease in the carbonate precipitation method and its impact on road subgrade improvement. The applicability of tofu waste as a biocatalyst in carbonate precipitation instead of a purified urease enzyme or soybean powder is investigated. Various concentrations of tofu waste were mixed with a reagent of $CaCl_2$ and urea. The mass of carbonate and its impact on expansive soil parameters are evaluated. Urease activity and the carbonate form resulting from a solution composed of the reagent and tofu waste solution were also evaluated to confirm its applicability. The California bearing ratio (CBR) test was conducted to determine the soil-bearing capacity of the soil before and after treatment. The CBR test was conducted under soaked and unsoaked conditions to determine the worst condition when the soil completely swelled.

2. Materials

Merck Urea ($CO(NH_2)_2$) and laboratory-grade calcium chloride ($CaCl_2$) at a concentration of 1 mol/L were used as reagents. Tofu waste was obtained from a tofu factory in Bogor, Indonesia. Tofu waste was extracted by drying and grinding soybeans in the sun for 5 h. The tofu waste used was filtered using filter no: 50 sieves. The sample of soil material used was expansive soil collected from Jababeka, Bekasi, Indonesia 6o19′14.436″ S-107º10′31.05″ E). Figure 1 show the materials used in this study, including expansive soil (Figure 1-a), raw tofu waste (Figure 1-b), and powder tofu waste (Figure 1-c).

(a) expansive soil (b) raw tofu waste (c) powder tofu waste

Figure 1. Materials used in this study

Soil properties were tested to investigate the characteristics of expansive soil, as presented in Table 1. Soil properties were tested to investigate the characteristics of expansive soil. The test results show that the soil has a specific gravity (Gs) of 2.62, which classifies the soil as organic clay. The uniformity coefficient (Cu) and curvature coefficient (Cc) are 8.00 and 1.74, respectively. According to ASTM D2487-06 [25], the soil is classified as fine-grained with good grades.

The extent of soil expansivity was analyzed. From the analysis results, the liquid limit (LL) and plastic limit of the soil sample were 84.80% and 34.57%, respectively; thus, a plasticity index (IP) of 50.23% was obtained. This value indicates the high plasticity of the soil sample [26]. Because clay content correlates positively with soil, a high clay fraction increases the PI. CH soil types or inorganic clay soils with high plasticity were tested, indicating that the soil samples included clay with expansive characteristics [27]. Soil with an LL of more than 60% and an IP of more than 35% is classified as soil with a high level of swelling [28].

Table 1. Properties of the soil used in this study

Soil Properties	Value	Unit
Specific gravity, Gs	2.63	-
Uniformity coefficient, Cu	8.00	-
Curvature coefficient, Cc	1.74	-
Liquid limit, LL	84.80	%
Plastic limit	34.57	%
Plasticity index	50.23	%
Soil type	CH	-

3. Experimental Methods

Several experiments were conducted, including test tube experiments, CBR tests, mineralogy, and morphology analysis. A test tube experiment was conducted to determine the optimum concentration of tofu waste in the optimum composition of reagents of calcium chloride and urea using the hydrolysis rate and precipitation ratio of precipitated calcite. The CBR value is used as the main parameter evaluated through the CBR test in soaked and unsoaked conditions. XRD and SEM analyses are also conducted to evaluate the mineralogy composition and confirm the mineral shape of the precipitated mineral of calcite.

3.1. Determine the Optimum Concentration of Tofu Waste

The optimum concentration of tofu waste was determined by test tube experiments, including the precipitation test and hydrolysis rate. Carbonate precipitation uses reagents and tofu waste as ureases. Equations 1 and 2 illustrate the chemical reaction of the carbonate precipitation method.

$$(NH_2)_2 + 2H_2O \xrightarrow{\text{urease}} 2NH_4+ + CO_3^{2-} \quad (1)$$

$$Ca^{2+} + CO_3^{2}- \rightarrow CaCO_3 \text{ (precipitation)} \quad (2)$$

A precipitation test was used to evaluate the amount of calcite formation. Putra et al. [29] used a reagent concentration of 1 mol/L of urea and $CaCl_2$. Tests were conducted to determine the optimum concentration of tofu waste. The precipitation test was conducted in a transparent polypropylene tube with varying concentrations of tofu waste, which was used as the initial basis before the soil treatment test. The extraction of tofu waste for the precipitation test was performed in two ways: using filters no—400 with an opening of 0.037 mm and centrifugation for 20 min at 3,000 rpm. The hydrolysis rate was used as a biocatalyst to determine the speed at which tofu waste hydrolyzes urea. This method adopts research from Putra et al. [20]. Conductivity calculations with time were performed using the Hanna edge multiparameter 230. Since the tofu waste comprises of approximately 88% soybean protein, the concentration of tofu waste in this study is designed to be higher than the optimum soybean concentration of 20–30 g/L [20, 30, 31]. Variations in tofu waste concentration were 20, 40, 60, and 80 g/L. The urea concentration was 1 mol/L with a volume of 50 mL per sample.

3.2. California Bearing Ratio (CBR) Test

CBR tests were conducted to determine the CBR value of expansive soil and evaluate its capability as a subgrade. The procedures of the test were based on ASTM D1833-21 [32]. CBR tests were conducted under soaked and unsoaked conditions. The soaked CBR test was conducted to determine the level of swelling when the worst conditions occurred in the field. Calcite treatment was applied to the CBR test under soaked and unsoaked conditions. The optimum concentration was used in the calcite treatment in the previous test. The treatment procedures followed the previous study by Putra et al. [17]. The soil sample treated with the solution was mixed with soil and then cured for seven days. CBR values were used to determine the bearing capacity of the subgrade in road planning. The CBR value was calculated by comparing the strength of the soil in carrying traffic loads.

3.3. Mineralogy and Morphology Analysis

XRD and SEM analyzes were performed to evaluate the morphology of the montmorillonite mineral, which causes swelling and shrinkage in the soil. XRD analysis was performed to determine the minerals formed in the soil before and after treatment. The precipitation test results were also subjected to XRD analysis to ensure that the minerals formed were calcite. Furthermore, the morphology of the montmorillonite mineral was evaluated by SEM analysis. This test was also used to determine the formation of calcite and soil structure after treatment with calcite solutions.

4. Results and Discussion

4.1. Tofu Waste Optimum Concentration

The hydrolysis rate was tested to determine the rate of urease activity during urea breakdown. As shown in Figure 2, the highest hydrolysis rate was obtained at a tofu waste content of 80 g/L (4.8 U/g). There is a significant difference between tofu waste and other biocatalyst sources. The pure urease enzyme has a hydrolysis activity of 200 U/g in 1 g/L. Another comparison using soybeans yielded a value of 1600 U/g at 50 g/L [20, 30]. The lowest hydrolysis rate was obtained at a concentration of 20 g/L with a value of 0.9 U/g, compared with pure soybeans with the same concentration of 330 U/g [33]. A significant reduction in the hydrolysis rate occurred because the urease content of the tofu waste was denatured by heating soybeans during the manufacturing process. Akkerman et al. (2016) showed that proteins are denatured if heated at 60°C–90°C for 1 h or less [34].

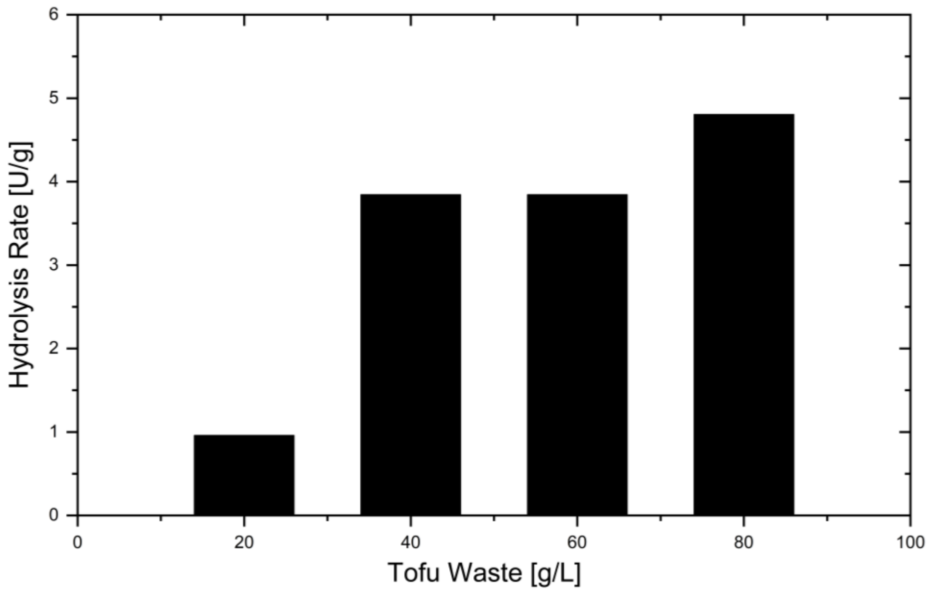


Figure 2. Hydrolysis rate reaction of urea by tofu waste

The hydrolysis rates at 40 and 60 g/L concentrations did not differ significantly; thus, the concentration of 40 g/L was selected as the optimum concentration by considering the technical ease of application with an activity of approximately 4 U/g. The lower concentration impacts the minimum requirement of the material source; thus, the processing for tofu waste preparation is also considerably reduced. Additionally, a higher concentration of tofu waste results in a higher organic material content. The organic content may affect enzymatic processes and the binding of calcite to the soil. Oktafiani et al. [35] reported that a high organic content can reduce soil strength. Furthermore, 80 g/L of tofu waste results in the highest hydrolysis rate, which is approximately 20% higher than the concentrations of 40 and 60 g/L. The twofold use of tofu waste does not appear to have a significant impact on the hydrolysis rate, and it has a negative impact on the ease of application and organic content.

Figure 3 shows the precipitation test results as a curve of the relationship between the tofu waste concentration and the precipitation mass ratio. The results show that the higher the tofu waste concentration, the higher the precipitation mass ratio and the actual mass of calcite produced. Filter treatment of tofu waste is superior to centrifugation. This result is indicated by the acquisition of a higher precipitation ratio using filters.

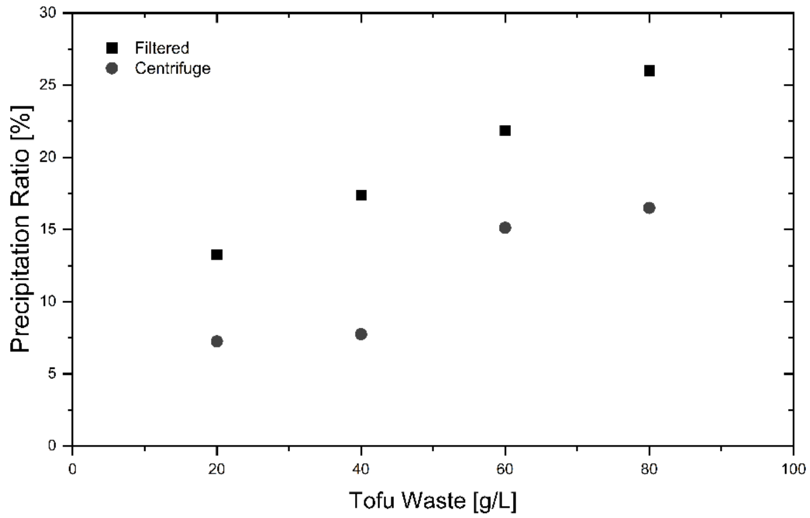


Figure 3. Precipitation test by variation in concentration

The filter method is preferable to centrifugation for separation of the solution. The filtrate treatment yielded the highest precipitation mass at a concentration of 80 g/L and a precipitation mass ratio of 26%, whereas the centrifugation treatment produced the lowest precipitation mass at 7.25%. Precipitation testing at a concentration of 40 g/L of soybeans yielded 90% [30] and 115.1% [36]. Meanwhile, in this study, tofu waste yielded 20% at the same concentration. As mentioned earlier, the ability of tofu waste to break down urea is low. This result indicates that the use of tofu waste should be re-optimized to increase the actual calcite precipitation mass. The precipitated content is the main parameter that determines the applicability of the carbonate precipitation method as a soil improvement method. The presence of carbonate content of more than 3% of the soil mass can produce sufficient soil strength of 300 kPa [19, 37, 38]. Therefore, increasing the precipitation ratio should be considered in future studies.

4.2. CBR and Swelling Ratio Parameters

The CBR test requires standard proctor soil compaction to obtain maximum dry density (MDD) and optimum moisture content (OMC) values. The test results showed that the MDD value was 1.38 g/cm^3 and the OMC value was 30.4% (Figure 4). To determine the effect of calcite solutions on the CBR value of expansive soil, subsequent soil samples were prepared under untreated and treated conditions according to the OMC results of the proctor standard test. The amount of soil and the required volume of solution were determined using the proctor standard compaction results.

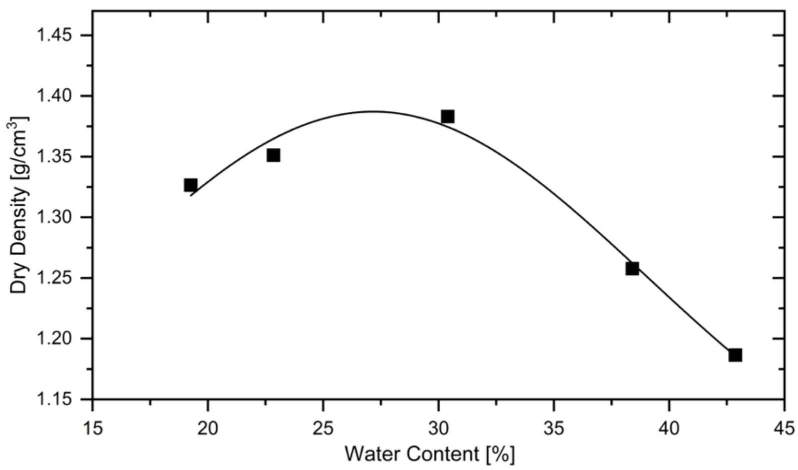


Figure 4. Compaction curve of the clay expansive soil used in this study

The soil-bearing capacity can be determined through CBR testing. The CBR value of the subgrade significantly influences the required pavement thickness when designing road pavements. The lower the CBR value of a subgrade, the higher the thickness required for the pavement structure [39]. Referring to SNI 1732-1989 [40], the requirements for CBR strength values for subgrade soils are greater than 3% and 6% for submerged and dry conditions, respectively. Figure 5 shows the CBR testing for expansive soil samples conducted under soaked and unsoaked conditions. Soaked CBR tests were conducted for treated and untreated soil samples. Soaked CBR test data were collected at OMC and 100% dry density according to ASTM D1833-21 [32].

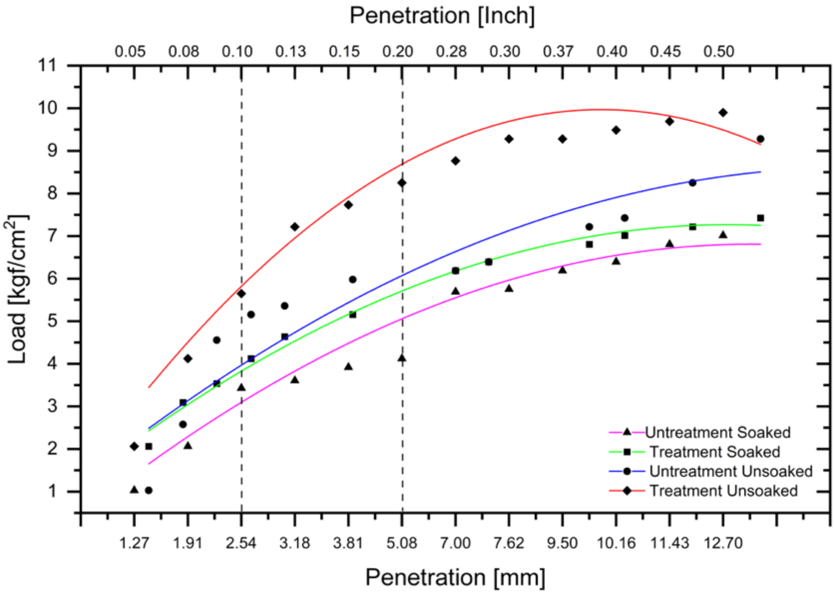


Figure 5. CBR curve of load and penetration

Figure 6 shows the potential for expansion (swelling) of the treated expansive clay samples, which is less than that of the untreated samples. A reduction in soil volume swelling from 3.68% in untreated soil to 3.22% after treatment was achieved. These results show the effect of treatment on soil samples in lowering the swelling ratio of 12.5%. However, soil treatment using carbonate precipitation does not significantly impact the CBR under soaked conditions. A relatively similar CBR of less than 5% was obtained. This could be attributed to high saturation hampering the carbonate precipitation process and its bridging to the soil particles.

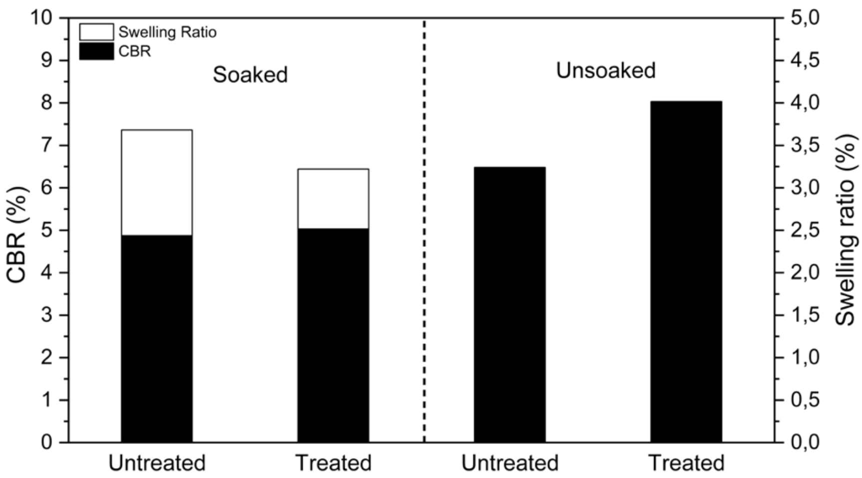
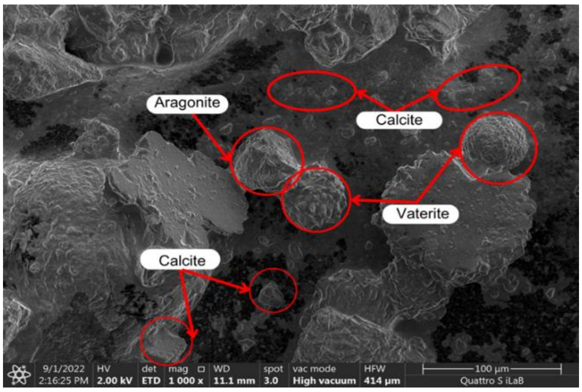


Figure 6. CBR and swelling ratio of treated and untreated samples under soaked and unsoaked conditions

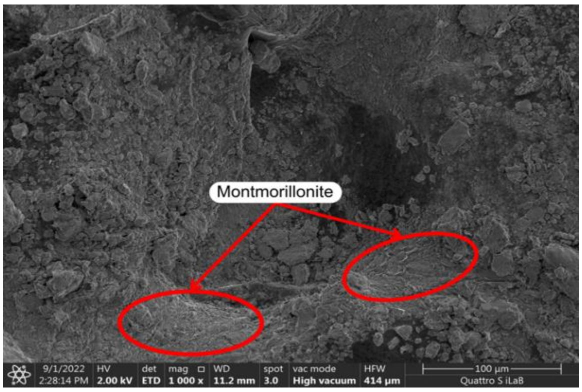
This result indicates that the precipitation process effectively reduces the swelling potential under soaked conditions. Data were obtained for laboratory CBR testing of unsoaked expansive clay using treated and untreated samples. The CBR value of the treated sample was higher than that of the untreated sample, with a 23.9% increase in the CBR value from 5.67% to 8.03%. The CBR value of the treated soil sample was 8.03%, indicating that soil stabilization with calcite solutions could produce better results on expansive soils. Furthermore, the CBR value meets the standard for Indonesian road subgrade pavement values [41]. Compared with other studies using Xhantan gum, reported by Hamza et al. [3], a similar increasing trend in the CBR values of 8% was also observed.

4.3. SEM and XRD Results

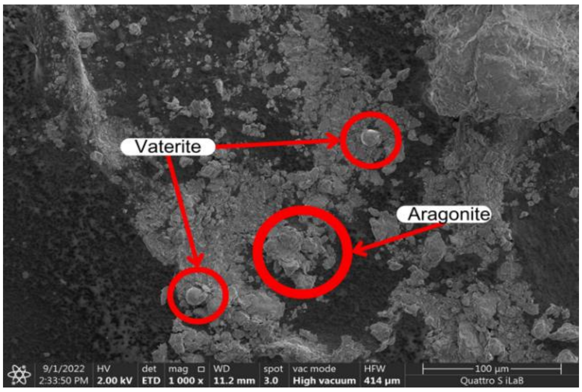
Figures 7 and 8 show the SEM and XRD analysis results, respectively. The tofu waste samples produced $CaCO_3$ crystals in three forms: aragonite, calcite, and vaterite. Calcite is the desired outcome because it is more stable than other polymorphs [42], and it has been proven that calcite can form. In untreated soil (T1), montmorillonite, kaolinite, illite, and quartz minerals are present. These minerals are generally found in expansive clay soils.



(a) Precipitated materials



(b) untreated soil sample



(c) treated soil sample

Figure 7. SEM results for precipitated minerals and untreated and treated soil

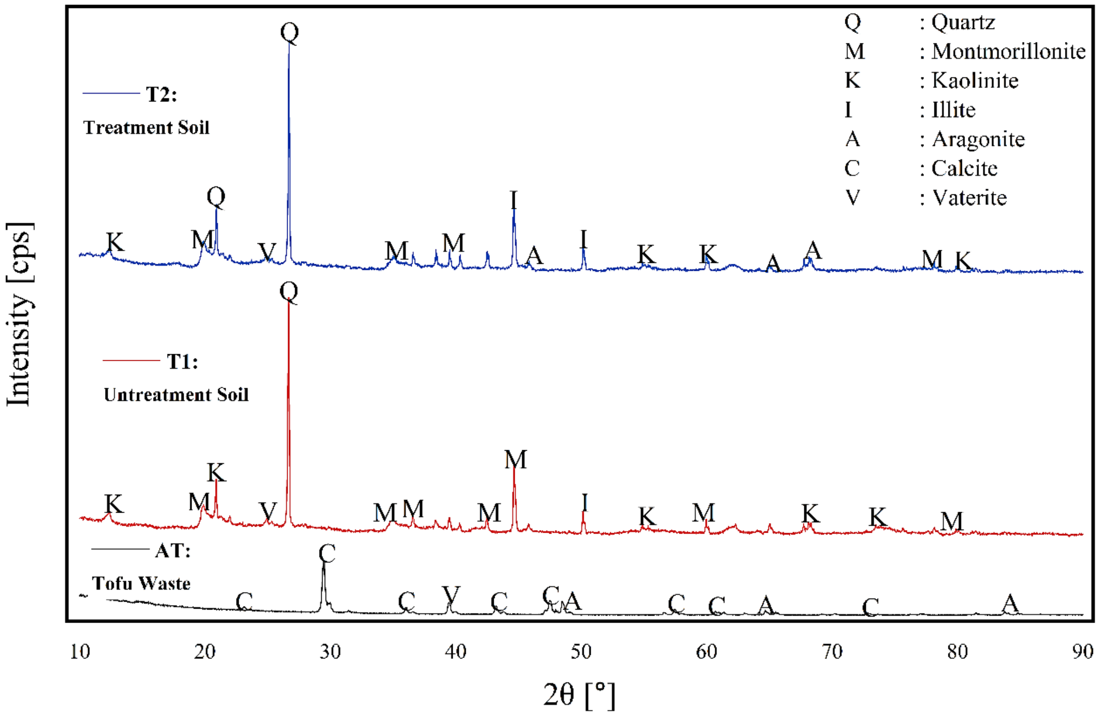


Figure 8. XRD results for precipitated minerals and untreated and treated soil

After treatment with calcite solutions (T2), aragonite and vaterite crystals were formed. Calcite did not form because it can bind with other compounds to form other minerals. According to the SEM analysis results, the treated soil sample showed the presence of polymorphs of calcite: aragonite and vaterite. The formed minerals may overlap; thus, the calcite may be covered by aragonite or vaterite [43]. According to the XRD and SEM data, the presence of many heterogeneous materials in the form of spherical minerals indicates that the calcite precipitation formation process needs to be optimized because of the low hydrolysis rate of the biocatalyst. These results indicate that tofu waste can act as a biocatalyst.

5. Conclusion

Expansive soils have a high swelling potential based on an LL value of 84.80% and a PI of 50.23%. The use of tofu waste as a biocatalyst was evaluated for optimum levels through precipitation and hydrolysis rate analyses. The optimum tofu waste concentration was 40 g/l with an activity and precipitation ratio of 4 U/g and 20%, respectively. The results showed that soil treatment with a tofu waste concentration of 40 g/l increased the carrying CBR value of the soil by 23.9% and met the Indonesian standard of 6% as a road subgrade. Additionally, the swelling ratio of the treated soil can be decreased by 12.5%. The SEM and XRD analyses show that tofu waste can form calcite, aragonite, and vaterite using the calcium carbonate precipitation method. According to the XRD spectral data, the peaks for vaterite, calcite, and aragonite appeared at 25.0°, 29.5°, and 45.9° 2Θ, respectively. This research has shown that tofu waste can be used as a biocatalyst in the calcium carbonate precipitation method to repair expansive clay soils. In conclusion, tofu waste has a high potential as a biocatalyst for decreasing swelling and increasing the CBR value using the carbonate precipitation method.

6. Declarations

6.1. Author Contributions

Conceptualization, H.P. and A.B.A.; methodology, H.P., A.B.A., R.M.I.H., F.R.A., M.L., and A.P.; validation, H.P., A.B.A., and R.M.I.H.; formal analysis, H.P. and A.B.A.; investigation, A.B.A., R.M.I.H., F.R.A., M.L., and A.P.; resources, A.B.A.; data curation, H.P.; writing—original draft preparation, A.B.A. and H.P.; writing—review and editing, H.P.; visualization, A.B.A. and H.P.; supervision, H.P.; project administration, H.P.; funding acquisition, H.P. All authors have read and agreed to the published version of the manuscript.

6.2. Data Availability Statement

The data presented in this study are available in the article.

6.3. Funding

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6.4. Conflicts of Interest

The authors declare no conflict of interest.

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