Optimization design of basalt fiber reinforced stabilizer using response surface methodology

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Abstract. This paper addresses the strength development of a basalt fiber reinforced cement-based soil stabilizer. In order to develop eco-friendly high performance stabilizer, cementitious capillary crystalline waterproof material is used as alternative to partially replacesulphate aluminum cement. Driven by the purpose of achieving a high strength performance without impairing other desirable properties, calcium sulfate, basalt fiber and hydrophilicalumina suspension are selected as additives. By adopting the Response Surface Method, the combined effects of the three additives on the unconfined compressive strength of the stabilized soil are analyzed. The basalt fiber can constrain the growth of micro cracks and pores, and thus control their coalescence. Incorporating basalt fiber in combination with calcium sulfate and alumina suspension can not only increase the strength and hardening of stabilized soil, but also improve other properties, especially for enhancing the ability of cementing, and for raising the fracture roughness, and therefore improve the durability. The microstructure changes as a consequence of stabilization process are characterized by the scanning electron microscope to analyze the origination of the high strength and durability of stabilized soil. The difference of mineral components between the untreated and stabilized soil samples is identified by the X-ray diffraction analysis.

1. Introduction

A variety of stabilizers have been invented [1] and exploited in numerous geotechnical applications associated with soil engineering [2], since it was found that they have utility of solidification which helps to improve physical/mechanical properties of composites. The inorganic stabilizer has gained particular attention, due to the advantages over manufacturing cost and raw material availability. Cement and lime are the most commonly used materials for the design of soil stabilizers.

On account of the poor performance of most soil stabilizers in unconfined compressive strength and water stability, they were usually used on the foundation treatment of soils. To the authors' knowledge, there are few studies taking into account the high early strength combined with good durability in the research of soil curing materials. The soil stabilizer with high early strength and good durability has been extra expected as a consequence of the thirst for the rapid repair of roads after major natural disasters, for rapid paving of emergency exit, as well as for the rescue and plugging in flood season. Especially, the fast hardening and high strength soil stabilizer can not only be used as the temporary pavement material during rescue and relief to ensure the rapid transit of emergency rescue equipment...
and vehicles, but also can it be used on the stabilization/solidification soil contaminated with heavy metals or radioactive ions, or as the permanent stabilizer for the construction of rural roads with low strength grade in the context of eco-environmental protection.

2. Experimental program

In our study, the main raw materials used in the design of the soil stabilizer are sulphate aluminum cement (SAC), cementitious capillary crystalline waterproof material (CCCW), calcium sulfate (CS), basalt fiber (BF), hydrophilicalumina suspension (HAS) and red clay.

The specimen is prepared with reference to the Chinese National Standards: *Technical guidelines for application of soil stabilizer* (RISN-TG003-2007) and *Highway engineering inorganic binder stabilizing material testing procedures* (JTG E51-2009).

3. Response surface methodology design

Owing to the characteristic of high early strength, SAC is used as the base cementitious material of soil curing agent. CCCW is utilized to produce the capillary crystalline in the voids between soil particles. In the present study, for the purpose of developing a high performance stabilizer, the contents of SAC and CCCW used for the optimization design are 12% and 4%, respectively. In the context of developing high strength stabilizer and preserving the durability of stabilized soil, CS, HAS, and BF are selected to be the curing components of soil stabilizer according to the testing results obtained from the single-factor experiments. Additionally, the single-factor experiments prescreen and determine the optimum ranges of CS (0.32 g–0.96 g), HAS (0.32 g–0.64 g), and BF (2.5 g–7.5 g), which can be beneficial to the enhancement of mechanical properties of stabilized soil.

| Number | CS / g | HAS / g | BF / g | Basic materials / g | UCS / MPa |
|--------|--------|---------|--------|---------------------|-----------|
| 1      | 0.64   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.85     |
| 2      | 0.64   | 0.75    | 5.00   | 48.00 16.00 60.00 400 | 10.67     |
| 3      | 0.10   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.39     |
| 4      | 1.18   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 10.36     |
| 5      | 0.64   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.68     |
| 6      | 0.64   | 0.48    | 7.50   | 48.00 16.00 60.00 400 | 11.76     |
| 7      | 0.96   | 0.32    | 7.50   | 48.00 16.00 60.00 400 | 10.54     |
| 8      | 0.32   | 0.64    | 2.50   | 48.00 16.00 60.00 400 | 11.12     |
| 9      | 0.64   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.74     |
| 10     | 0.32   | 0.64    | 7.50   | 48.00 16.00 60.00 400 | 11.01     |
| 11     | 0.96   | 0.32    | 2.50   | 48.00 16.00 60.00 400 | 11.12     |
| 12     | 0.64   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.68     |
| 13     | 0.32   | 0.32    | 7.50   | 48.00 16.00 60.00 400 | 11.31     |
| 14     | 0.96   | 0.64    | 2.50   | 48.00 16.00 60.00 400 | 10.82     |
| 15     | 0.32   | 0.32    | 2.50   | 48.00 16.00 60.00 400 | 11.47     |
| 16     | 0.64   | 0.48    | 9.20   | 48.00 16.00 60.00 400 | 10.27     |
| 17     | 0.64   | 0.21    | 5.00   | 48.00 16.00 60.00 400 | 11.67     |
| 18     | 0.64   | 0.48    | 5.00   | 48.00 16.00 60.00 400 | 11.69     |
| 19     | 0.64   | 0.48    | 0.80   | 48.00 16.00 60.00 400 | 11.15     |
| 20     | 0.96   | 0.64    | 7.50   | 48.00 16.00 60.00 400 | 10.36     |

To authors’ knowledge, the design of soil stabilizer is mainly concentrated on orthogonal testing. However, orthogonal tests can only perform finite individual points in most cases. It has certain limitations and can not realize the optimization design in certain regional surface. Thus, in our study, we employ the response surface methodology (RSM) [3] for the optimization design of the developed stabilizer. The mixing ratios of factors and consequent UCSs are listed in Table 1.

3.1. Regression model and variance analysis

We use the software Design-Expert to conduct statistical analysis. The ternary quadratic mathematical regression equation for the 7-day UCS of solidified body is obtained:
\[ 7 - dUCS = 9 + 3.13A + 5.07B + 0.52C + 0.42AB - 0.12AC + 0.05BC - 2.81A^2 - 7.18B^2 - 0.06C^2 \] (1)

where A, B, and C represent the fractions of CS, HAS, and BF, respectively.

The analysis of variance is conducted on CCD scheme. \( R^2 \) value are important parameters for judging the degree of approximation of multivariate quadratic polynomial equation [4]. Statistically, the more close to 1 the correlation coefficient \( R^2 \) is, the better the approximation effect is; the closer \( R^2 \) and Adj\( R^2 \) are, the higher approximation reliability the model has. In our model, \( R^2 \) is 0.9649, Adj\( R^2 \) 0.9334. The deviation between \( R^2 \) and Adj\( R^2 \) is 0.0315.

3.2. Response surface analysis

Fig. 1 shows the impact of CS and HAS and their interaction on the 7-day UCS of solidified body. It can be seen that the strength gradually increases with the increase of the amount of CS and then gradually decreases with the further increase of the amount of CS. Increasing the amount of HAS can slowly raise the strength up to a threshold value, while a further increase has a gradual reduction effect. Fig. 2 describes the impact of CS and BF and their interaction on the 7-day UCS. The dramatically fluctuant changes of the strength with the variations of CS and BF indicates that the interaction between CS and BF has significant influence on the UCS. It is notable that with different amount of BF, the effect of CS exhibits different expression. When the amount of BF is small, the steep curve indicates that the change of CS can significantly affect the strength development; when the amount of BF is high, the change of CS only has gentle effect on the strength. CS and BF are mutual inhibition, which is in line with the approximation that the coefficient of term AC in the regression equation is negative. Fig. 3 illustrates the impact of HAS and BF on the strength of solidified body.
3.3. Optimizing formulation of soil curing materials

Based on the results calculated by the "Numerical" module of Design-Expert, an optimum mix proportion associated with the targeted highly unconfined compressive strength is attained. To verify the optimized mix proportion [5], we conduct three tests to estimate the compressive strength. It is notable that, the simultaneously repeated three measurements are used to increase the confidence in experimental data and decrease the uncertainty in our estimation. The mix proportion, 3-day UCS, and 7-day UCS are given in Table 2. The average 3-day UCS is 10.06 MPa, and the average 7-day UCS can achieve 11.78 MPa.

| Number | CS (g) | HAS (g) | BF (g) | SAC | CCCW | Water | Soil | 3-day UCS /MPa | 7-day UCS /MPa |
|--------|--------|---------|--------|-----|------|-------|------|----------------|----------------|
| 1      | 0.49   | 0.38    | 4.38   | 48.00 | 16.00 | 60.00 | 400  | 9.97           | 11.24          |
| 2      | 0.49   | 0.38    | 4.38   | 48.00 | 16.00 | 60.00 | 400  | 10.08          | 11.94          |
| 3      | 0.49   | 0.38    | 4.38   | 48.00 | 16.00 | 60.00 | 400  | 10.13          | 12.16          |

4. Microanalysis of solidified body

4.1. SEM

The scanning electron micrographs of stabilized soil with optimized mix proportion are depicted in Fig. 4. Comparing to the untreated soil, the soil particles after stabilization process are surrounded by hydration products. The addition of CS and HAS can intensify and accelerate the hydration reaction for gaining desirable properties, for instance fast hardening, self-stressing. CS reacts with anhydrous calcium sulphaaluminate (3Ca(OH)₂·3Al₂O₃·CaSO₄) of cement, producing ettringite and alumina hydrate gel.

\[
3\text{Ca(OH)}_2\cdot3\text{Al}_2\text{O}_3\cdot\text{CaSO}_4+2(\text{CaSO}_4\cdot2\text{H}_2\text{O})+34\text{H}_2\text{O} \\
=3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}+2(\text{Al}_2\text{O}_3\cdot3\text{H}_2\text{O}) \\
(2)
\]

The hydration of dicalcium silicate (β-C₂S) of cement generates calcium silicate hydrate and calcium hydroxide.

\[
2(2\text{CaO} \cdot \text{SiO}_2)+4\text{H}_2\text{O}=3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}+\text{Ca(OH)}_2 \\
(3)
\]

The produced calcium hydroxide and alumina hydrate gel can further react with CS and generate ettringite.

\[
3\text{Ca(OH)}_2+3\text{CaSO}_4\cdot2\text{H}_2\text{O}+\text{Al}_2\text{O}_3\cdot3\text{H}_2\text{O}+20\text{H}_2\text{O}=3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O} \\
(4)
\]

Eqs. (2) – (4) demonstrate the facilitation of CS to the hydration of cement. Especially, in cement, the linked anhydrous calcium sulphaaluminate crystals encircle the fine dicalcium silicate, blocking the hydration of dicalcium silicate. On the one hand, the addition of CS can facilitate the hydration of
anhydrous calcium sulphoaluminate, thus accelerating the exposure and hydration of dicalcium silicate, accordingly accelerating the development of the early strength of stabilized soil [6]. On the other hand, the addition of HAS can directly facilitate the hydration of CS (Eq. (3)), further promoting the hydration of cement. Additionally, on account of the infiltration and cementing effects of HAS, the fine particles in the system can be cemented, increasing the compactness of the system[7]. The ettringite generated during hydration process can have cementing effect between the hydraulic cement paste and soil particles, forming the skeleton of system; while the alumina hydrate gel and calcium silicate hydrate gel can fill the pores in the hydraulic cement paste and between soil particles, further densifying the system and giving strength to the system. However, excessive ettringite can result in significant volume expansion, thus impairing the mechanical integrity of stabilized soil system.

The hydration products have strong space frame structures formed by hydrated calcium silicate gel through binding the particles together by means of its cementing effect. Therefore, the microstructure of stabilized soil is transformed from the stacked structure of unconsolidated, low-strength aggregate of untreated soil to the dense, high-strength aggregate structure. There are many rod-shaped and acicular crystals crossly distributed in stabilized soil (Fig. 4 a). The crystals of hydration products such as ettringite and calcium hydroxide can be seen in Figs. 4 b and c, the columnar crystal is ettringite and the hexagon flake crystal is calcium hydroxide.

Fig. 5 displays the general view of BF dispersed in stabilized soil. The SEM image demonstrates that the BF provides a strong interfacial bonding, constraining the growth and coalescence of microcracks and pores. Consequently, the durability of stabilized soil under external stimulation can be improved[8].

![Figure 4. The microstructures of optimized solidified soil](image)

![Figure 5. (a) SEM image of the action of basalt fiber; (b) Schematic illustration of the effect of basalt fiber on the constraining of microcracks and pores.](image)

4.2 XRD

In order to explore the impact of the addition of the main stabilizer on the generation of chemical products in stabilized soil and the changes in phase characteristics, X-ray diffraction (XRD) analysis is conducted respectively on untreated soil sample (Fig. 6) and stabilized soil specimen with the designed optimum mix proportion (Fig. 7) [9].
It can be seen from Fig. 6 and Fig. 7 that there exists difference in composition between the stabilized soil and untreated soil. The mineral composition of naturally undisturbed soil sample mainly includes quartz, albite, banalsite and montmorillonite, etc. In comparison with the untreated soil, the stabilized soil has less albite and other minerals, like anorthite, gibbsite and tobermorite. Hydrated calcium silicate gel binds soil particles by using its cementing effects to form a strong connection structure and improve the strength of stabilized soil.

Figure 6. X-ray diffraction pattern of naturally undisturbed soil

Figure 7. X-ray diffraction of stabilized soil sample with optimized mix proportion

5. Conclusions

In this paper, a basalt fiber reinforced soil stabilizer incorporating cementsitious capillary crystalline waterproof materials is developed. Calcium sulfate, basalt fiber and hydrophilic alumina suspension are selected as additives for developing a high strength performance and eco-friendly stabilizer. The average 3-day and 7-day unconfined compressive strengths can achieve 10.06 MPa and 11.78 MPa, respectively. Both CS and HAS can significantly improve and accelerate the hydration of cement, forming the skeleton of the system. BF provides a strong interfacial bonding, which can constrain the growth and coalescence of micro cracks and pores. In the stabilized soil sample, anorthite and tobermorite are generated and form a strong connection structure between soil particles.

References
[1] Henghui F, Jianen G, Pute W, Prospect of researches on soil stabilizer, Jour. of Northwest Sci-Tech Univ. of Agri. and For. (Nat. Sci. Ed.) 2 (2006) 141-152.
[2] IlieşNM, CîrcuAP, NagyAC, CiubotaruVC, Kisfaludi-BakZ, Comparative study on soil stabilization with polyethylene waste materials and binders, Proc. Eng. 181(2017)444-451.
[3] Nassar, Ahmed I., N. Thom, and T. Parry, Optimizing the mix design of cold bitumen emulsion mixtures using response surface methodology, Constr. Build. Mater. 104(2016)216-229.
[4] MO Hamzah, SY Teh, B Golchin, J Voskuilen, Use of imaging technique and direct tensile test to evaluate moisture damage properties of warm mix asphalt using response surface method, Constr. Build. Mater. 132(2017)323-334.
[5] M. Yolmeh, M. Khomeiri, Z. Ahmadi, Application of mixture design to introduce an optimum cell-free supernatant of multiple-strain mixture (MSM) for Lactobacillus, against food-borne pathogens, LWT-Food Sci. and Technol.83 (2017) 298-304.
[6] Berger S, Coumes C C D, Bescop P L, Influence of a thermal cycle at early age on the hydration of calcium sulfoaluminate cements with variable gypsum contents, Cement & Concrete Research, 41(2011) 149-160.
[7] LatifiN, EissaZadehA, MartoA, MeehanCL, Tropical residual soil stabilization: a powder-form material for increasing soil strength, Constr. Build. Mater. 147(2017) 827-836.
[8] Mohammadhosseini, Hossein, J. M. Yatim, Microstructure and residual properties of green concrete composites incorporating waste carpet fibers and palm oil fuel ash at elevated temperatures, Journal of Cleaner Production 144(2017)8-21.
[9] MS Baghini, A Ismail, MR Karim, F Shokri, AA Firoozi Effect of styrene–butadiene copolymer
latex on properties and durability of road base stabilized with Portland cement additive, Constr. Build. Mater. 8 (2014) 740-749.