

Solidification Technique for Fluorine Contaminated-Bassanite Using Composite Recycling Materials for Ground Improvement Applications

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Abstract

Waste management is a serious problem over the world due to the huge amount of waste materials produced and increased annually. This paper investigates the development of solidification technology, based on the formation of ettringite, for fluorine contaminated bassanite using waste and recycled materials. For this purpose, recycled bassanite was mixed with coal ash, blast furnace slag and furnace cement type-B in different proportions to obtain the optimal production for ettringite based on their chemical responses. Leaching tests were performed on these different admixtures to measure the solubility of fluorine. Scan electron microscope (SEM) and X-ray diffraction (XRD) tests were conducted on the investigated samples to explore the formation and intensity of ettringite. Results show that the suggested admixture has a significant effect on the production of ettringite and increasing cement content in admixture increases the formation of ettringite. The increase of the ratio of cement-admixture has a clear effect on the production of ettringite and the reduction of fluorine solubility compared to the case of cement-bassanite mixture. Both size and length of ettringite crystals are increasing with the increase of the ratio of cement-admixture/bassanite and intensity of ettringite in material matrix. The solubility concentration of fluorine decreases with the increase of admixture content due to concomitant increase in the production of ettringite. Dimensions of ettringite crystals have a significant effect on the reduction of the solubility of fluorine, the increase of ettringite prisms the decrease of fluorine solubility. The investigated limits of the suggested admixture did not show any adverse effect on the geo-environment properties in term of the release of fluorine. The suggested admixture is recommended to be used as stabilizer material for soft clay soil since it is friendly with environment, economic and meet the standards of environment in Japan.

Keywords: Gypsum wastes, Recycled bassanite, Ettringite, Solidification agent; Ground improvement

1. INTRODUCTION

The huge growth of waste materials in Japan is one of the largest challenges nowadays. Therefore, most attention has been directed to maintain a sound environment by using wastes as alternative materials in earthwork projects to reduce these quantities and to reduce the cost of ground improvement used in construction, keep sound environment and create a balance for the limitation of natural resources. Gypsum waste plasterboards, coal ash and blast furnace slag are three examples of wastes that have a negative effect on the environment and society in terms of soil contamination and the cost of their disposal. Approximately 1.6 to 1.7 million tons of gypsum waste plasterboards produced annually in Japan during the three stages of production, construction and demolition¹⁾⁻³⁾.

Gypsum waste plasterboards are a serious problem due to the environmental problems associated with their disposal in landfill sites and then it is burning issue in Japan nowadays. This is due to the restricted environmental regulations in Japan require

that gypsum wastes must sent to the controlled landfill sites and then the cost of their disposal increased¹⁾. The quantity of coal ashes production in Japan is reached to 7.1 million tons and approximately 1.5 million tons of coal ashes sent to landfill sites⁴⁾. These quantities of coal ashes may be expected to increase in future because most of energy resources in Japan are based on power plant which running by fuel, especially after the nuclear accident occurred in 2011. Steel and iron industries in Japan produce a large amount of blast furnace slag annually and this amount reached to 14,160 Metric Kilo ton in 2011 according to Nippon Slag Association in Japan⁵⁾. Several studies have been investigated using coal ash and blast furnace slag in cooperated with other materials as a stabilizing agents to enhance the mechanical properties and strength of soft clay soil⁶⁾.

While limited studies have been investigated the incorporation of recycled bassanite produced from gypsum wastes as a stabilizing agent for soft clay soil compared to coal ash and blast furnace slag^{1)-3),7),8)}. This is related to the fact that the use of recycled bassanite in soil stabilization has many challenges especially in a wet environment since bassanite is a soluble material. These challenges are related to durability of bassanite-treated soil and increasing the solubility concentration of fluorine more than the permitted standard, which may results in soil

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contamination⁸⁾⁻¹⁰⁾. Increasing the concentration of fluorine solubility in water, more than the standard, has a toxic effect on all human, animals and plants¹¹⁾. World health organization reported that increasing fluorine in drinking water more than the guideline value of 1.5 mg/l may increase the risk of causing health hazards^{12),13)}. As the environmental regulations in Japan are restricted, the permitted value for fluorine was determined 0.8 mg/l in accordance soil and environmental standards in Japan¹⁴⁾.

Consequently, it is essential to develop solidification technique for bassanite-treated soil to avoid the occurrence of soil contamination and meet the environment standards in Japan when recycled bassanite produced from gypsum wastes used in earthwork projects. The solidification technology used to remediate the contaminated soil involves mixing the contaminated soil with binder to constrain the release of harmful substances. Portland cement and lime have a long history to be used as binders to develop solidification technology for the contaminated soil¹⁵⁾. The solidification technology for the contaminated soil using cement or lime is based on the development of the production of cementation compounds such as ettringite in soil matrix. This is related to ettringite has a potential to capture the fluorine on its surface and then the concentration of fluorine solubility will be reduced¹⁵⁾.

As result increasing the cost of Portland cement and lime in Japan and environmental problems raised when cement incorporates in earthworks such as the release of chromium and boron more than the standards. The concept of the development of new solidification technology for fluorine contaminated bassanite using waste materials such as coal ash and blast furnace slag in combination with recycled materials such as furnace cement type-B is proposed. This application is introduced to reduce the cost of solidification technology for the contaminated fluorine bassanite and it is considered as a way to reduce the huge quantities of wastes in Japan. Also, the suggested admixture is proposed to be used as a stabilizer material for soft clay soil since all of its ingredients used successfully before as a stabilizing agents for soft clay soil. This concept is based on the shortage in the performance of some wastes will be improved by using the advantages from other wastes or recycled material.

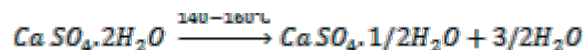
As mentioned above solidification technique is mainly based on the production of ettringite in soil matrix which is capable to capture fluorine from the contaminated soil. The formation of ettringite is mainly produced due the induced chemical reactions for calcium oxide, aluminate, sulfate, and water¹⁶⁾. Blast furnace slag is rich with aluminate and calcium oxide, coal ash is rich with silicate and aluminate, furnace cement is rich with calcium silicate and aluminate while recycled bassanite is rich with

calcium and sulfate. Therefore, the development of solidification technique for fluorine contaminated bassanite using the suggested wastes and recycled material is proposed.

For this purpose, recycled bassanite was mixed in different proportions with the admixture of furnace cement type-B, coal ash and blast furnace slag as well as it was mixed with different proportions with furnace cement type-B alone. This is to investigate the use of the suggested admixture and furnace cement on the remediation of fluorine contaminated bassanite as well as on the production of ettringite.

2. Materials and Testing

Four different types of materials were used in this research include recycled bassanite, coal ash, blast furnace slag and furnace cement type-B as binders for the formation of ettringite. Recycled bassanite was produced from gypsum waste plasterboards and production process for recycled bassanite was discussed in details in previous works. Dried-air gypsum wastes were pulverized and then screened prior to subject for heating under specified temperature for a certain time to produce recycled bassanite/gypsum as presented in the equation below.



The chemical compositions of the produced bassanite in this research were found calcium sulfate hemi-hydrate ($\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$) with a content of 100% based on the result of X-ray diffraction (XRD) test. Mechanical properties for the produced recycled bassanite are presented in Table 1.

Coal ash used in this research was produced from Kanden Power-Tech Co. Ltd., in Japan. Chemical compositions for the used coal ash are presented in Table 2. It is well-known that the chemical compositions of coal ash are different from site to another depending on the type of fuel used as well as type, content and quality of additives used in power plant. Based on the data presented in Table 2 the type of coal ash used in this research is classified as F-class type in accordance ASTM C 618-78. Because of the sum percentages of SiO_2 , Al_2O_3 , and Fe_2O_3 is found 90.10, LOI is less than 6%, and the retained percentage in sieve# 325 is less than 34%. As the used coal ash is classified as F-class, which has no self-cementing property, and then it is not recommend to be used alone for the development of solidification for the contaminated fluorine bassanite.

While, coal ash in this research plays two important roles, the first it is considered the main source for alumina and the second to promote the chemical reaction for producing ettringite since coal ash has a pozzolanic activity.

Blast furnace slag used in this research was brought from Shinko Slag Co., Ltd. in Japan. Chemical compositions for the used furnace slag are presented in Table 3. Blast furnace slag plays an important role in this study by providing calcium and alumina for the suggested admixture, which are essential for the formation of ettringite.

The type of cement used in this research is furnace cement type-B and this type is produced mainly by-product of Portland cement and other wastes in Japan. The used cement was brought from company for cement production in Tokyo, Japan. Chemical compositions for the used cement are presented in Table 4. The cement (C) used in this research as a main solidification agent for recycled bassanite (B) when it is used alone as well as it is used as a solidification agent also in the case of the suggested admixture (A) which comprises recycled bassanite, coal ash, and blast furnace slag. Five different ratios of cement-bassanite (C/B) and cement-admixture (C/A) were investigated. The investigated ratios are 0, 2, 4, 8, 16 and 32 for the case of bassanite (B) as well as for the case of admixture (A). The content of admixture/bassanite and furnace cement was mixed with water at a ratio of 1:1 prior to subject the samples for 28 days curing and then tested for the solubility of fluorine as well as tested for SEM and XRD.

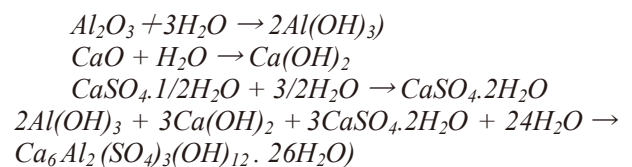
The solubility of fluorine is determined in accordance with the standard leaching method for measuring the solubility of fluorine reported in soil and environmental countermeasures, Ministry of environment, Japan. All details for the process of measuring the concentration of fluorine are provided in the guidelines testing for fluorine, Ministry of Environment, Japan as well as in previous work. In a brief, the tested material was mixed with water and then cured for 28 days in the controlled room. The 28 days cured sample was crushed and then mixed with distilled water with a ratio of 1:10. The mixture was mshacked for 4 hours and kept standing 30 minutes for precipitation prior to filtering process. The concentration solubility of fluorine is measured with Ion chromatograph Z-8200 type on the filtered mixture. Scanning electron microscope (SEM) test was done on the tested samples to investigate the

production of ettringite using JEOL JSM 6580 scanning electron microscope type. The cured sample was pulverized and then small pieces form these samples were considered to represent the whole sample. The small pieces of the tested soil were mounted on a copper specimen holder prior to coat with special material which needed to provide surface conductivity. More attentions were considered for sample preparation using special technique for this test and then the tested samples were moved to SEM machine which operating at 15kV. While X-ray diffraction test was done for the same samples used in SEM to identify their intensity for the production of ettringite.

3. Results and Analysis

The main chemical elements contribute in the formation of ettringite are calcium, alumina and sulfate. The substance of calcium, alumina and sulfate are available in the form of calcium oxide (CaO), aluminate (Al_2O_3) and hemi-hydrate calcium sulfate ($CaSO_4 \cdot 1/2H_2O$), respectively. As mentioned early, these substances are available in coal ash, furnace slag and recycled bassanite. Both calcium oxide and aluminate are available in blast furnace slag and coal ash while hemi-hydrate calcium sulfate is available in recycled bassanite as presented in Table 5.

Synthesizes of ettringate ($Ca_6 Al_2 (SO_4)_3 (OH)_{12} \cdot 26H_2O$) is presented in the following equations.



The mole ratio of the main chemical materials (binders) contributes in the production of ettringite of Al_2O_3 -to-CaO-to- $CaSO_4$ was found 1-to-3-to-3. While the atomic weight for these materials used in the formation of ettringite of Al_2O_3 , CaO, and $CaSO_4 \cdot 1/2H_2O$ are 102, 56, and 145, respectively. Consequently, the required mass for each material can be obtained by multiplying atomic weight and mole ratio. The mass ratio for the materials of (Al_2O_3 -to-

Table 1. Mechanical properties of recycled bassanite.

Property	Density, ρ_s g/cm ³	D ₁₀ , mm	D ₃₀ , mm	D ₅₀ , mm	D ₆₀ , mm	Coefficient of uniformity, U_c	Coefficient of curvature, C_c
Value	2.64	0.22	0.37	0.50	0.60	2.73	1.04

Table 2. Chemical compositions for the used coal ash.

Pattern	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Ret.#325	LOI
Content, %	58.30	27.60	4.20	2.80	1.10	20	2.4

Table 3. Chemical compositions for the used blast furnace slag.

Pattern	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	TiO	MnO	LOI
Content, %	33.60	14.30	0.20	42.50	7.30	0.90	1.20	0.20	0.90

Table 4: Chemical compositions for the used furnace cement type-B.

Pattern	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	R ₂ O	TiO	P ₂ O	MnO	Cl	Ig. loss	Insol.
Content, %	26.30	8.70	1.90	54.10	3.70	2.00	0.26	0.42	0.54	0.69	0.08	0.28	0.01	0.80	0.20

Table 5. The percentages of aluminate, calcium oxide and hemi-hydrate calcium sulfate inhabit in the used materials.

Substance	Al ₂ O ₃	CaO	CaSO ₄ .1/2H ₂ O
Coal ash (X)	27.60%	2.80%	0.00%
Blast furnace slag (Y)	14.30%	42.50%	0.00%
Recycled bassanite (Z)	0.00%	0.00%	100%

CaO-to-CaSO₄.1/2H₂O) used in the formation of ettringite was found 1-to-1.65-to-4.26. Based on the data presented in Table 5, the optimal ratio for the materials of (Al₂O₃-to-CaO-to-CaSO₄.1/2H₂O) require for producing the optimal ettringite intensity can be determined according to the equations presented below. These equations are developed based on the mass ratio of the materials contribute in the formation of ettringite as presented before as well as based on the percentages of these materials inhabited in binders (waste and recycled materials) used in the ingredients of admixture (A) as presented in Table 5.

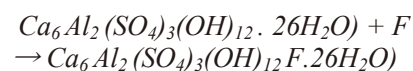
$$Al_2O_3 = 0.276X + 0.143Y = 1.00 \quad (1)$$

$$CaO = 0.028X + 0.425 Y = 1.65 \quad (2)$$

$$CaSO_4.1/2H_2O \quad Z = 4.26 \quad (3)$$

The values of X, Y and Z referee to coal ash, furnace slag, and recycled bassanite and by solving equations 1 and 2, these values are found 1.67, 3.77 and 4.26, respectively. Consequently, the mass ratio for coal ash-to-furnace slag-to-recycled bassanite used to form the suggested admixture (A) for producing the optimal ettringite intensity was found 1-to-2.26-to-2.55. For example, to obtain 1000 gm of the suggested admixture (A), the contents of coal ash, furnace slag and recycled bassanite are found approximately 170, 390, and 440 gm, respectively. Consequently, the pattern of the suggested admixture (A) was built on the chemical basis presented above to obtain the optimal production for ettringite.

SEM images for the suggested admixture (A) treated with furnace cement proved that the formation of ettringite for all the investigated cases as presented in Figure 1. The increase of cement-admixture (C/A) ratio has a significant effect on the increase of the formation of ettringite. Both size and length of ettringite prisms (crystals) increase with the increase of the ratio of cement-admixture (C/A) as presented in Figure 2. This result is probably attributed to the increase of the contents of binders contribute in the formation of ettringite. The increase of the ratio of cement-admixture (C/A) is associated with the decrease of fluorine solubility as presented in Figure 3. This result is most likely related to the increase of the formation of ettringite in material matrix and then the possibility of capturing fluorine on the surface of ettringite prisms increased to form F-ettringite as presented in the following equation.



Besides, the decrease of the solubility of fluorine with the increase of the intensity of ettringite is attributed to the increase of ettringite intensity increasing both length and size of ettringite prisms as presented in SEM images (Figure 1) and Figure 2. Consequently, the possibility of attracting fluorine on the surface area for the prisms of ettringite increased. The increase of the surface area of ettringite prisms promotes the capture of fluorine on their surfaces and

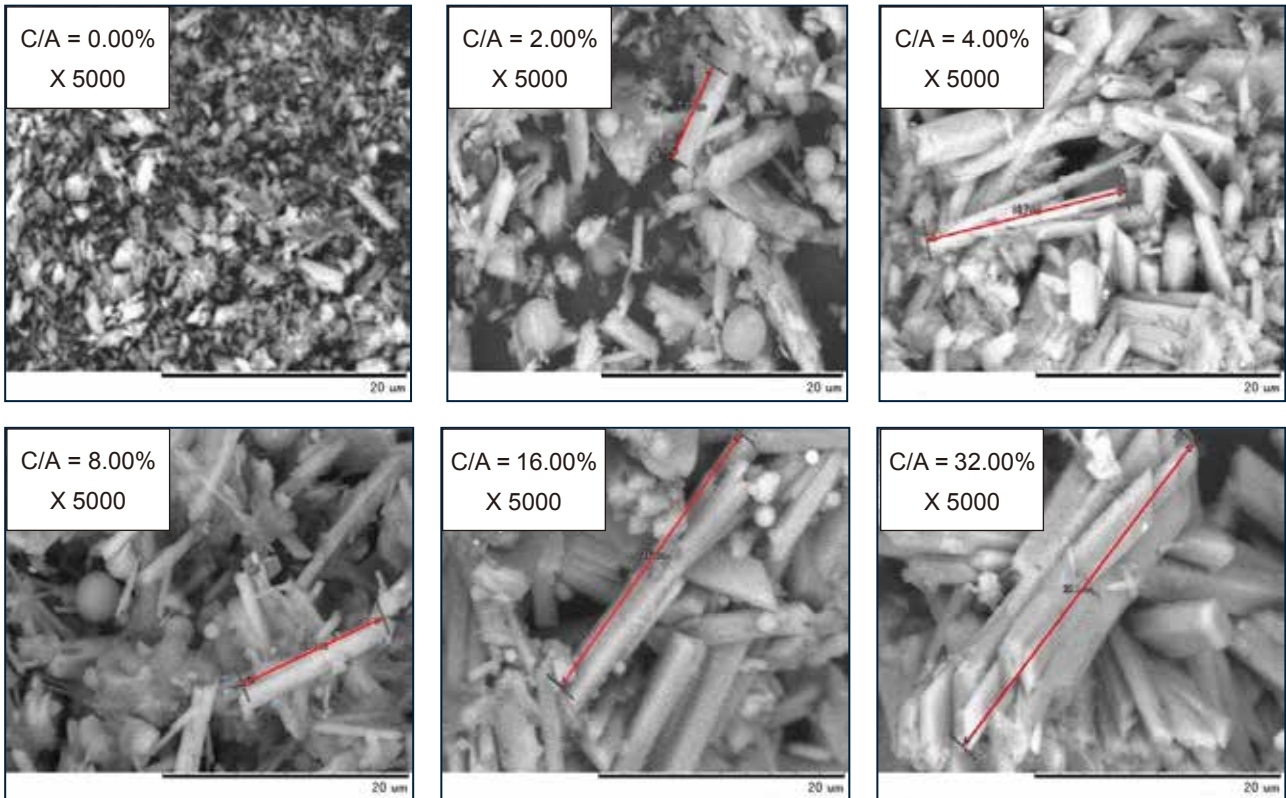


Fig. 1. SEM images for samples treated with different percentages cement-admixture ratios (C/A).

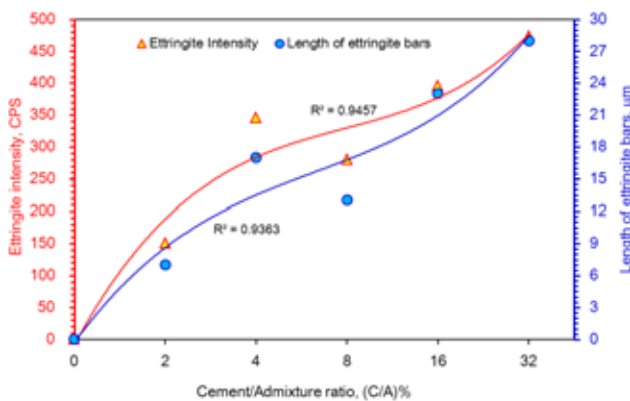


Fig. 2. The influence of cement-admixture ratio on the properties of the formed ettringite.

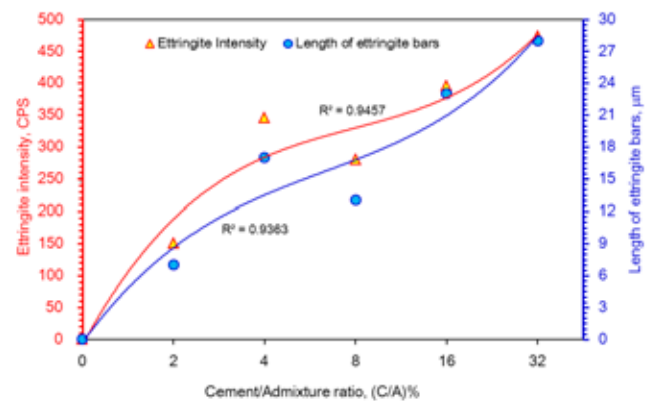


Fig. 3. Relationships between the ratio of cement-admixture (C/A) against ettringite intensity and fluorine solubility.

then the release of fluorine is solidified. Ultimately, the solubility of fluorine decreased with the increase of the ratio of cement-admixture (C/A) due to the increase of ettringite intensity. The production of ettringite when cement added to admixture (A) is not only related to the presence of cement in mixture while the ingredients of the admixture play the major role for the formation of ettringite as mentioned before. It is important to evident that the solubility of fluorine in the case of $C/A = 0$ was found 1.09 mg/l while the solubility of fluorine in the case of pure bassanite was found 6.60mg/l. Table 6 shows the concentration of fluorine inhabited in cement and other materials used in the ingredient of the admixture. The use of

admixture alone without the addition of cement decreased the solubility of fluorine inhabited in bassanite with 84%, approximately. This confirms the merit of the suggested admixture for the reduction of fluorine solubility of the contaminated fluorine bassanite.

The availability of chemicals contribute in the production of ettringite has a significant effect on the formed shape and size crystals of ettringite. Results presented in Figures 1 and 2, which indicated that prisms (crystals) sizes of ettringite increase with the increase of the ratio of cement-admixture, supported the data presented in previous work. Besides, these results are in agreement with the results presented by

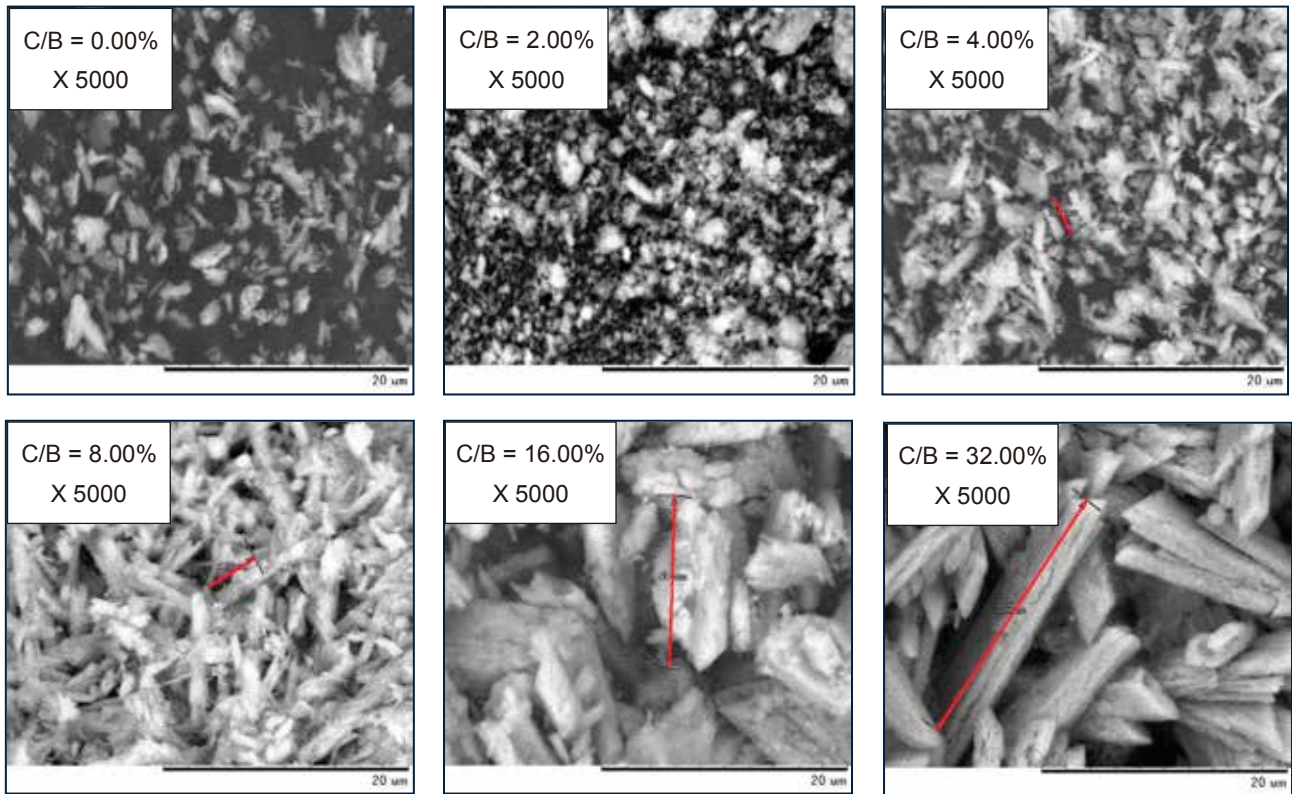


Figure 4. SEM images for samples treated with different percentages of cement-admixture ratios (C/B).

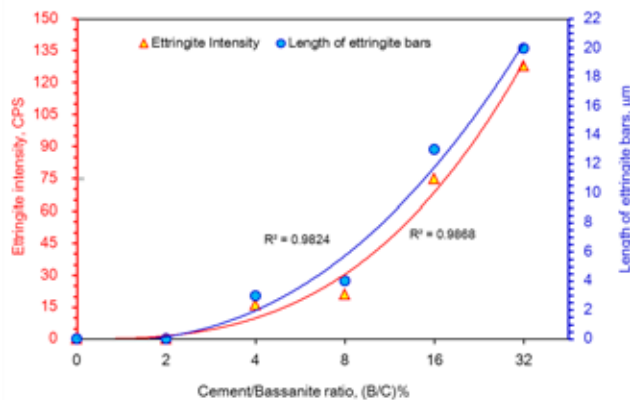


Fig. 5. The influence of cement-bassanite ratio on the properties of the formed ettringite.

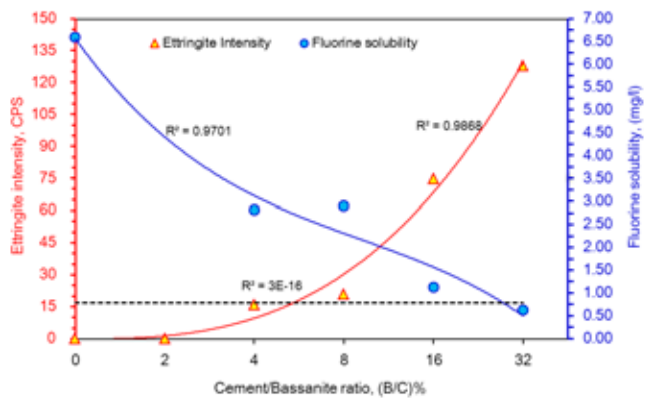


Fig. 6. Relationships between the ratio of cement-bassanite (B/C) against ettringite intensity and fluorine solubility.

another previous work which proved that binders used for the production of ettringite have a clear influence on the size and growth of ettringite crystals. The size of crystals of ettringite is not only important for the capture of fluorine but also it plays an important role for the stability of ettringite, the increase the size of crystals the increase the stability of ettringite. The morphology of the developed ettringite crystals has a significant effect on geotechnical properties such as strength, the increase of ettringite crystals the increase of strength. This issue is important when admixture is introduced in ground improvement projects. Generally, the formation of ettringite is not only influenced by the chemical compounds (binders) used to produce

ettringite while there are some parameters control the formation, growth and size of ettringite crystals. These parameters are pH, temperature, degree of oxyanion, curing temperature and carbonation. The influence of these parameters on the production of ettringite using the suggested admixture (A) is not investigated in this study. In this context, the required pH to get acceptable stability for ettringite should be more than 10.5. The suggested admixture treated with furnace cement has pH more than 10.5 since the alkalinity of cement, furnace slag and coal ash used as binders to form ettringite are ranging between high to mild. The favored temperature needed for the formation of ettringite was found below 50 °C. In this study the

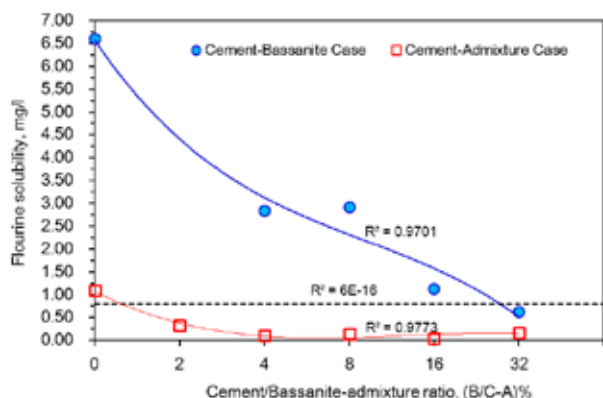


Fig. 7. Effect of the percentage ratios of cement-admixture (C/A) and cement-bassanite (C/B) on the solubility of fluorine.

average temperature of the mixture of cement-admixture-water was found below the recommended temperature for the formation of sulfate ettringite. Damidot and Glasser [1992] reported that the high concentration of sulfate ettringite the more stable phase of ettringite. Bassanite used in admixture as a source for sulfate and it is well known bassanite is rich with sulfate which helps to increase the stability of ettringite. This appeared clearly in the results presented in Figures 1 and 3 which indicated that the increase of admixture content the increase the growth of ettringite. Carbonation plays a negative role for the dissolution of ettringite and then the stability of ettringite decreased with the presence of carbonation. While the increase of excess water in the mix of binders used in the formation of ettringite helps to slow down the carbonation reaction. For that the mixture of cement-admixture was mixed with water at a ratio of 1-to-1 to slow the carbonation reaction. It is evident to report that the increase of water more than specified limit has a negative effect on the stability of ettringite. This is related to the presence of more excess water helps the dissolution of ettringite to gypsum, alumina gel and calcium carbonate. The preferred curing temperature for binders (chemical compounds) used for the formation of ettringite should be ranged in between 20 to 50 °C. In this study curing temperature was selected 21±1 to meet site condition as well as to meet the favored curing temperature needed for the growth of ettringite.

SEM images for recycled bassanite treated with cement only are presented in Figure 4. These images show that the addition of cement produces the formation of ettringite and increasing the percentage

of cement-bassanite ratio (C/B) is associated with the increase of ettringite intensity as presented in Figure 5. The formation of ettringite when cement added is attributed to the fact that cement has aluminate and calcium hydroxide which reacts with sulfate in bassanite to produce ettringite. The increase of ettringite intensity decrease the solubility of fluorine as presented in Figure 6. Both size and length of ettringite prisms increase with the increase of cement-bassanite ratio (C/B) due to the increase of ettringite intensity. These results are in agreement with the results presented in the case of admixture (A) treated with cement.

The use of admixture (A) in conjunction with cement has a significant effect on the reduction the solubility of fluorine compared to the case of bassanite-cement mixture as presented in Figure 7. This is most likely related to admixture-cement has more potential to produce ettringite compared to the mixture of bassanite-cement due to the availability of binders used for the formation of ettringite. The minimum ratio of cement-admixture (C/A) needed for the reduction of fluorine up to the standard limit in Japan (0.8 mg/l) was found more than 2. While in the case of cement-bassanite, the needed ratio (C/B) for the reduction of fluorine solubility up to the standard limit of fluorine in Japan was found 32. Consequently, the use of the suggested admixture treated with furnace cement saved approximately 94 % from the actual cost of the treatment process compared to the case of cement-bassanite (C/B). This confirms that the suggested admixture is more economic and friendly with the environment in the term of the release of fluorine. Consequently, it is recommended using the suggested admixture for reduction the solubility of fluorine when recycled bassanite used in ground improvement projects as a stabilizer.

Besides, the expected results for the enhancement of strength and durability of soft clay soil when the suggested admixture (A) in conjunction with furnace cement used as a stabilizer will be achieved effectively compared to the use of bassanite-cement mixture. This result is related to the fact that the suggested admixture (A) in conjunction with furnace cement has a potential to produce adequate amounts of ettringite, which helps for the improvement of strength. This is due to ettringite plays an important role for the improvement of the strength of stabilized soil due to its needle structure that creates crystal interlocking. The creation of interlocking helps for the improvement of

Table 6. Fluorine solubility for the used virgin materials.

Material	Coal ash	Blast furnace slag	Recycled bassanite	Furnace cement
Fluorine solubility (mg/l)	0.57	0.62	6.60	0.58

geotechnical properties of stabilized soil. Further, the production of ettringite when admixture (A) treated with furnace cement used as a stabilizing agent for soft clay soil is expected to be increased more because clay soil has also alumina and calcium that promote the formation of ettringite. Also, admixture treated cement is expected to be achieved acceptable durability compared to the case of bassanite treated cement. This is related to the fact that the increase of solidification property for stabilizer is associated with the enhancement of durability. Consequently, clay soil stabilized admixture has a potential to resist the actions of weathering conditions such as freeze-thaw and wet-dry cycles due to the developed interlocking and hardening between soil particles.

4. Conclusions

This study investigated the feasibility of the development of solidification technology based on the formation of ettringite using waste and recycled materials for the reduction of fluorine-contaminated soil when recycled bassanite used as a stabilizer material for ground improvement projects. Based on the results of SEM, XRD and leaching tests, conclusions can be drawn as follows:

1. The suggested admixture has a potential to produce ettringite and reduce the solubility of fluorine compared to the use of traditional solidification agents alone. The optimal mass ratio for the ingredients (binders) of the suggested admixture to obtain the optimal production of ettringite was found 1: 2.26: 2.55 which corresponding to coal ash: furnace slag: bassanite, respectively.
2. The formation of ettringite and the reduction of fluorine solubility increases with the increase of the ratio of cement-admixture (C/A) and the same behaviors were obtained in the case of cement-bassanite (C/B).
3. The use of the suggested admixture treated with furnace cement has a clear effect on the production of ettringite and the reduction of fluorine solubility concentration compared to the case of bassanite treated with the same furnace cement alone.
4. Both length and size of ettringite prisms increased with the increase of the intensity of ettringite and the mixing ratio for both cement-admixture (C/A) and cement-bassanite (C/B).
5. The use of the suggested admixture treated with furnace cement type-B is recommended to be used as a stabilizer for soft clay soil because it has low cost and it is friendly with the environment.

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