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Improvement mechanism of sodium carbonate on traditional composite filler

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Certain issues may be encountered when traditional composite fillers are mixed with cement and bentonite (CF-CB) and applied to cavities in the ground. These issues include the bleeding and separating of water, cement and bentonite, and the long time needed for the strength development. In a series of studies, it has been shown that the suppression of bleeding, earlier gelling and earlier development of strength can be expected by mixing a suitable amount of sodium carbonate in the CF-CB, that is, creating a CF-CBS. The mechanisms of the various properties of CF-CBS and CF-CB were investigated by physical analyses. The following points were clarified from the results of this work. Earlier development of strength and gelling are brought about by the earlier formation of ettringite accompanying the promotion of the cement hydration reaction. The suppression of bleeding can be accomplished by earlier gelling due to the bentonite taking the Na^+ ionised from sodium carbonate as an interlayer cation. The increase in strength is attributed to the calcium carbonate produced by CO_3^{2-} ionising from the sodium carbonate bonding with the Ca^{2+} dissolved from the interlayer cations of the cement or bentonite.

1. Introduction

As the number of obsolete infrastructures being rebuilt has been increasing in recent years, it has become necessary to improve the quality of the fillers used as backfill in cavities in the ground formed during the removal of existing underground structures, such as piles. The role of the filler is to increase the recoverability of the target ground (Bauer *et al.*, 2016; Ghadimi *et al.*, 2017; Han, 2015). If proper treatment by filling is not carried out and/or the physical properties of the ground and the filling site differ greatly, then phenomena such as settlement may occur in the surrounding ground (Granata *et al.*, 2015; Inazumi *et al.*, 2017; Kuwahara and Inazumi, 2019).

A composite filler comprising cement and bentonite (CF-CB) has fluidity and self-hardness and does not require compaction; therefore, it is a typical filler widely used for filling in narrow spaces and spots that would be difficult to compact (Aqil *et al.*, 2005; Mikkelsen, 2005). The mechanical qualities of CF-CB are easy to produce and CF-CB can be mixed according to the conditions of the construction by performing mixing studies in most laboratories. However, some difficulties can be encountered, such as taking several hours for the CF-CB to develop in strength and not being able to control the CF-CB only by adjusting the blending (mixing). Due to these issues, an uneven development of strength is generated due to material segregation (bleeding).

The aim of this study was to overcome the above-mentioned issues and to develop a filler capable of ensuring a predetermined strength quickly and reliably. Hashimoto *et al.* (2017a, 2017b) and Inazumi *et al.* (2019) revealed that the effect of

suppressing bleeding and bringing about earlier gelling, earlier strength development and an increase in strength could be realised by mixing sodium carbonate (Na_2CO_3) with CF-CB.

An attempt was thus made to clarify the mechanism by which the above properties are exhibited by a composite filler comprising cement, bentonite and sodium carbonate (CF-CBS). For this purpose, typical physical analyses (X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) observations and energy-dispersive X-ray (EDS) analysis) were carried out.

2. Earlier studies on CF-CBS

The components of CF-CBS are water, bentonite, cement and sodium carbonate. By mixing 50–90% water, 1–10% bentonite, 10–40% cement and 0.5–3% sodium carbonate, based on the total weight of CF-CBS, the CF-CBS will be effective in suppressing bleeding and bringing about earlier gelling, earlier strength development and an increase in strength. Furthermore, bentonite, cement and sodium carbonate are introduced in this order into a mixer to which water is added in advance, and sufficient stirring is carried out every time each material is added. In this way, bleeding is mostly suppressed and gelling occurs early (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019).

The strength properties of CF-CBS have been evaluated by the uniaxial compressive strength according to the Japanese industrial standard (JIS A 1216) 'Uniaxial compression test method of soil' (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019). The initial strength of CF-CB is increased by

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Table 1. Components of ordinary Portland cement used

Silicon dioxide: %	Aluminium oxide: %	Ferric oxide: %	Calcium oxide: %	Magnesium oxide: %	Sulfur trioxide: %
21.4	4.6	2.5	63.0	0.8	1.7

mixing sodium carbonate. This indicates that the strength development can be accelerated. With regard to the hardening action at a material age of 1 d, CF-CB requires about 6 h to gel from the time the sample is mixed and stirred, but CF-CBS gels in about 1 h. Therefore, sodium carbonate promotes earlier gelling of CF-CB. Immediate gelling and earlier strength development have useful effects such as stabilising the target ground quickly and shortening the construction period. Moreover, even for the long-term strength (over 28 d), the increase in strength by mixing sodium carbonate has been confirmed. Thus, mixing sodium carbonate not only results in early gelling, but also has the effect of increasing the strength of CF-CB.

The problem of component material segregation has been evaluated based on the bleeding ratio according to the Japan Society of Civil Engineers standard (JSCE F 522) 'Bleeding ratio and expansion ratio test method for injection mortar of prepacked concrete (polyethylene bag method)' (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019). CF-CBS almost never causes bleeding and the component material segregation of CF-CB is suppressed by adding sodium carbonate. In addition, bleeding is suppressed even with a small amount of bentonite by using bentonite with a high swelling ability.

The flowability has been evaluated by the flowing time of the P-funnel according to the Japan Society of Civil Engineers standard (JSCE F 521) (JSCE F 521) 'Flowability test method for injection mortar of prepacked concrete (method by P-funnel)' (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019). There is no change in the flowability of CF-CB when mixing it with sodium carbonate; its high fluidity is maintained. Having high flowability without component material segregation shows that filling can be reliably performed even for irregularly shaped cavities and that it is possible to express a predetermined strength homogeneously at the filling point.

Earlier gelling and earlier strength development of CF-CBS is thought to be due to the promotion of the hydration of the cement reaction with sodium carbonate and the formation of calcium carbonate (CaCO_3). Calcium hydroxide (Ca(OH)_2) produced immediately after the hydration of cement is carbonated by carbonate ions (CO_3^{2-}) generated by the ionisation of sodium carbonate to produce calcium carbonate. The production of calcium carbonate promotes the hydration reaction by accelerating the consumption of calcium hydroxide, and it is expected to promote the gelling of cement and to enhance the initial strength.

It is considered that the mechanism for bleeding suppression is caused by an increase in the swelling property of bentonite. The nature of bentonite differs depending on the type of interlayer cation, and the sodium (Na) type with many sodium ions (Na^+) is more swellable than the calcium (Ca) type with a large amount of calcium ions (Ca^{2+}) (Onikita, 2007). When sodium carbonate and bentonite are mixed in water, the sodium carbonate dissolves in the water and ionises to generate Na^+ , which are ion exchanged with Ca^{2+} contained in the interlayer cations of the bentonite. It is thought that this increases the swelling property by strengthening the properties of the sodium type and contributes to the suppression of bleeding.

It is thought that the increase in strength is due to the formation of calcium carbonate. Calcium carbonate is also produced by the interlayer cations Ca^{2+} and CO_3^{2-} being bonded in the reaction between CO_3^{2-} and calcium hydroxide, as described above, and the cation-exchange reaction of bentonite. Calcium carbonate is a water-insoluble substance and its molecules are bonded to each other to form a hardened body. Therefore, it is thought that the generation of calcium carbonate may bring about an increase in the initial and long-term strength.

3. Experimental programme and devices

The components of the CF-CBS and CF-CB produced for this study were water, bentonite, ordinary Portland cement and sodium carbonate. Tables 1 and 2 respectively show the components of the cement and the bentonite used. For the bentonite, slightly swollen bentonite with a swelling degree of 4 was used, as recommended by the American Colloid Company (ACC) method (Kawatra and Ripke, 2001). For a comparison of different types of bentonite, highly swollen bentonite with a

Table 2. Components of slightly swollen and highly swollen bentonites

	Slightly swollen bentonite	Highly swollen bentonite
Silicon dioxide: %	67.1	68.4
Aluminium oxide: %	16.8	18.9
Ferric oxide: %	3.7	4.6
Magnesium oxide: %	3.3	2.2
Calcium oxide: %	2.7	1.7
Sodium oxide: %	2.2	2.1
Potassium oxide: %	1.3	0.6
Loss on ignition: %	2.9	1.7

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Table 3. Mix proportions

Specimen	Slightly swollen bentonite: kg/m ³	Highly swollen bentonite	Cement: kg/m ³	Sodium carbonate: kg/m ³	Water: kg/m ³	w/c
1	50	0	240	20	897	374
2	0	20	240	20	908	378
3	50	0	240	0	905	377
4	0	20	240	0	916	382

swelling degree of 20 was also used. Ordinary CF-CB is used in a weight ratio of cement to bentonite of approximately 4/6 and a weight ratio of water to cement (w/c) of approximately 300/500. In addition, from the results of preliminary tests, it was confirmed that adequate fluidity, strength and material separation resistance are exhibited by using 50–90% water, 1–10% bentonite, 10–40% cement and 0.5–3% sodium carbonate based on the total weight of CF-CB or CF-CBS (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019). Table 3 shows the mixing conditions of the specimens used in the physical analyses. The specimens used in the physical analyses were mixed so as to have the same fabrication conditions and show the same useful properties as the fillers described in Section 2 and used by Hashimoto *et al.* (2017a, 2017b) and Inazumi *et al.* (2019). In Table 3, specimen 1 is the CF-CBS specimen using slightly swollen bentonite, specimen 2 is the CF-CBS specimen using highly swollen bentonite, specimen 3 is the CF-CB specimen using slightly swollen bentonite and specimen 4 is the CF-CB specimen using highly swollen bentonite.

3.1 SEM

SEM was performed to observe the reactions and products occurring on the surfaces of the specimens. In the SEM observations, specimens 1, 2 and 3 were used to compare the difference depending on the presence or absence of sodium carbonate and the type of bentonite. The SEM equipment used was a JSM-6010LA instrument (Jeol Company Ltd.), and uncoated specimens were observed at an acceleration voltage of 15 kV.

3.2 XRD analysis

XRD analysis was performed to investigate the reaction of CF-CBS and CF-CB as well as the change in the hydration products generated in the specimen by adding sodium carbonate. In this analysis, a powder XRD method was carried out using Smart Lab manufactured by Rigaku Corporation. The X-rays used were CuK α ($\lambda = 1.5418 \text{ \AA}$).

3.3 EDS analysis

In the EDS analysis, specimens 2 and 4 of CF-CBS were used for comparing and examining the element distribution of the generated substances and the content ratio of the constituent elements. Also, because the type of bentonite depends on the interlayer cations and because there is no difference in the

constituent elements of the minerals, a comparison was made with fillers using highly swollen bentonite with outstanding physical properties. In this analysis, a JSM-7600F device (Jeol Company Ltd.) was used and uncoated specimens were observed at an acceleration voltage of 15 kV.

4. Results and discussion

4.1 SEM observation and XRD analysis

Figure 1 shows SEM images and Figure 2 shows the results of the XRD analysis.

In Figure 1, unreacted cement particles can only be confirmed at the age of 3 h for specimen 3, while hydration products can be confirmed around the cement particles for specimens 1 and 2. In particular, acicular crystals with a size of approximately 2 μm often observed, and ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) was considered in view of the shape of the crystals. Ettringite is a substance produced by the hydration reaction of the aluminate phase from the beginning of the test, and the hydration reaction of cement is accelerated by mixing sodium carbonate (JCA, 2012). Although the same reactions can be confirmed even at 1 d of age, in the case of specimens 1 and 2, the periphery of the cement particles was completely covered with a hydrate of low crystallinity, which is considered to be calcium silicate hydrate (CSH). It is supposed that the hydration reaction of alite ($3\text{CaO} \cdot \text{SiO}_2$) is promoted. Compared with the results of the XRD analysis shown in Figure 2, the peaks of erythrite are small in specimens 1 and 2 at 1 d of age, whereas sharp peaks of CSH appeared. This is consistent with the results of the SEM observations (see Figure 1). In specimens 1 and 2, calcium aluminate hydrate ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$), similar to the composition of ettringite caused by the hydration reaction of the aluminate phase, can be confirmed from 1 d of age. This agrees with the viewpoint of promoting the initial hydration reaction due to the hydration of the aluminate phase.

At 7 d of age, the hydration reaction was found in specimens 1, 2 and 3 and it was possible to observe the formation of a more dense and hardened body and the presence of some crystals (see Figure 1). In the XRD analysis, the peak of calcium hydroxide appeared strongly, and it is considered that calcium hydroxide was formed with a high degree of crystallinity (see Figure 2).

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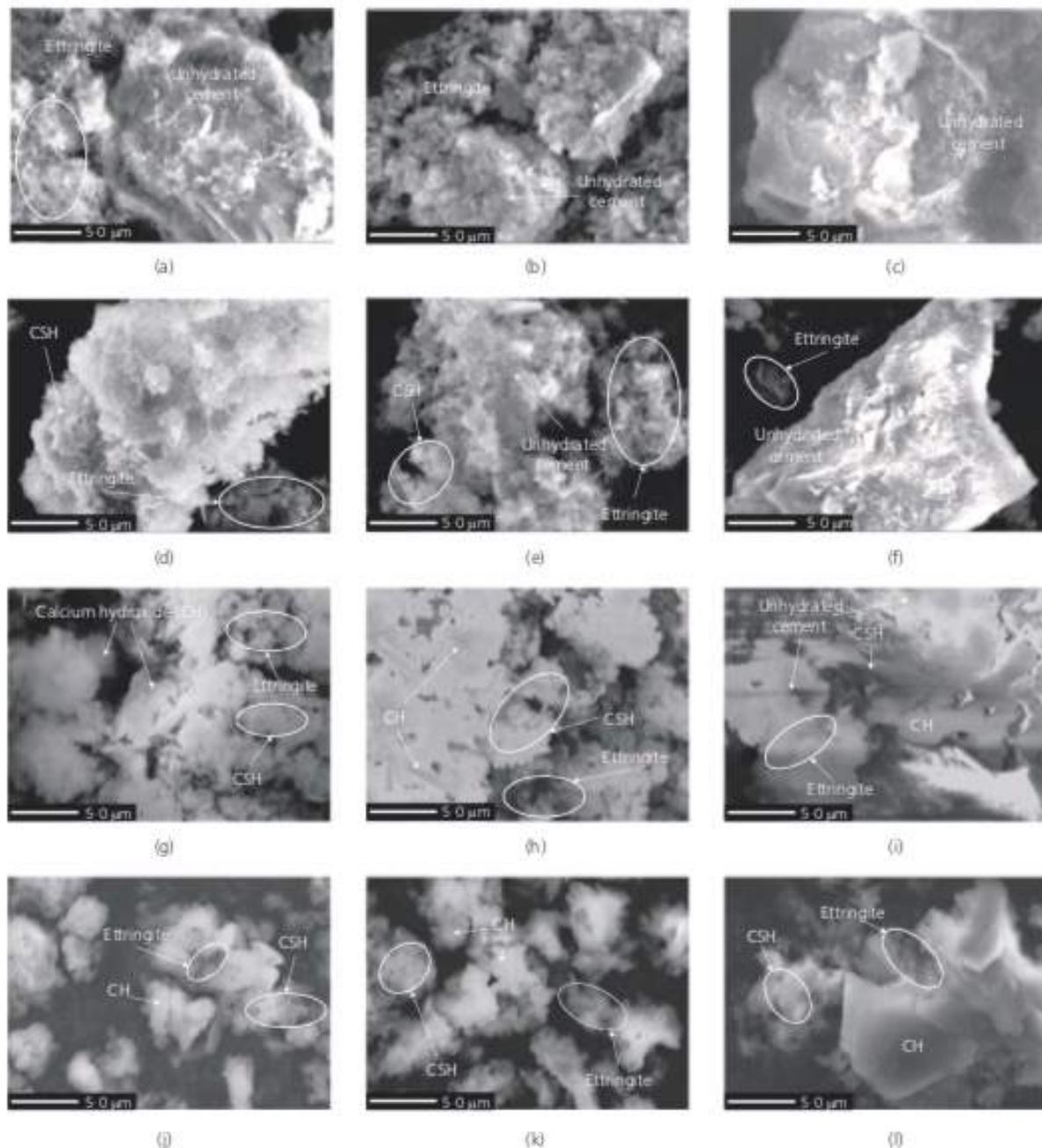


Figure 1. SEM images of CF-CBS. (a) Specimen 1 (sodium carbonate, low swelling bentonite), 3 h. (b) Specimen 2 (sodium carbonate, high swelling bentonite), 3 h. (c) Specimen 3 (no sodium carbonate, low swelling bentonite), 3 h. (d) Specimen 1 (sodium carbonate, low swelling bentonite), 1 d. (e) Specimen 2 (sodium carbonate, high swelling bentonite), 1 d. (f) Specimen 3 (no sodium carbonate, low swelling bentonite), 1 d. (g) Specimen 1 (sodium carbonate, low swelling bentonite), 7 d. (h) Specimen 2 (sodium carbonate, high swelling bentonite), 7 d. (i) Specimen 3 (no sodium carbonate, low swelling bentonite), 7 d. (j) Specimen 1 (sodium carbonate, low swelling bentonite), 28 d. (k) Specimen 2 (sodium carbonate, high swelling bentonite), 28 d. (l) Specimen 3 (no sodium carbonate, low swelling bentonite), 28 d.

A large amount of ettringite and CSHs are produced in specimens 1 and 2 at the age of 28 d, and several crystals were packed and bonded (see Figure 1). In specimen 3, it can be confirmed that a hydrate was formed around a large crystal.

but when compared with specimens 1 and 2, the amount of ettringite and CSH were both small (see Figure 2). That is, when sodium carbonate is added, a hydrate is densely formed. In the XRD analysis (see Figure 2), clear and sharp peaks can

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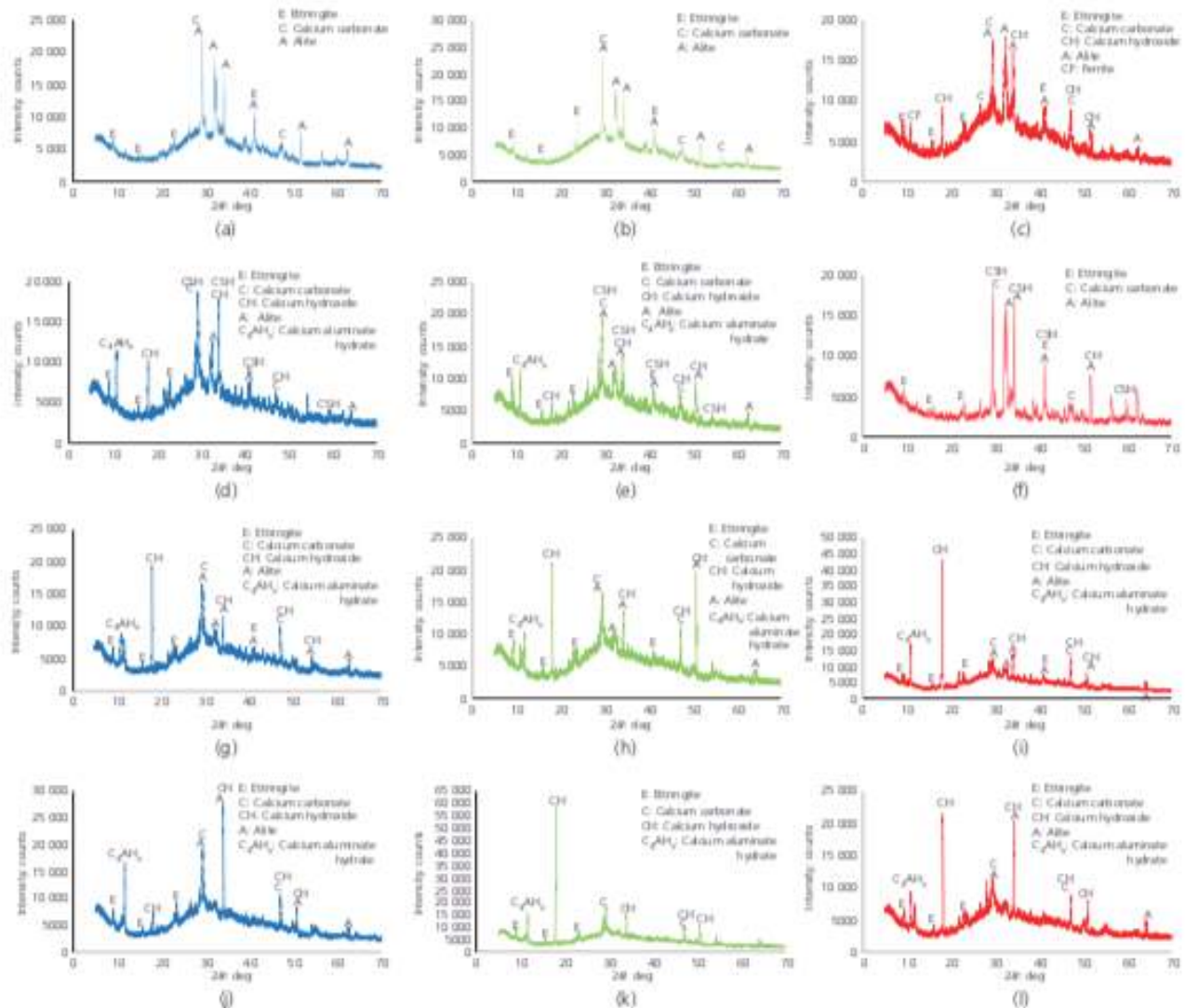


Figure 2. XRD patterns of CF-CBS. (a) Specimen 1 (sodium carbonate, low swelling bentonite), 3 h. (b) Specimen 2 (sodium carbonate, high swelling bentonite), 3 h. (c) Specimen 3 (no sodium carbonate, low swelling bentonite), 3 h. (d) Specimen 1 (sodium carbonate, low swelling bentonite), 1 d. (e) Specimen 2 (sodium carbonate, high swelling bentonite), 1 d. (f) Specimen 3 (no sodium carbonate, low swelling bentonite), 1 d. (g) Specimen 1 (sodium carbonate, low swelling bentonite), 7 d. (h) Specimen 2 (sodium carbonate, high swelling bentonite), 7 d. (i) Specimen 3 (no sodium carbonate, low swelling bentonite), 7 d. (j) Specimen 1 (sodium carbonate, low swelling bentonite), 28 d. (k) Specimen 2 (sodium carbonate, high swelling bentonite), 28 d. (l) Specimen 3 (no sodium carbonate, low swelling bentonite), 28 d.

be confirmed in specimens 1 and 2, indicating that substances with high crystallinity were generated. This point seems to contribute to the increase in strength of the CF-CB by adding sodium carbonate which has been clarified in earlier studies (Hashimoto *et al.*, 2017a, 2017b; Inazumi *et al.*, 2019).

4.2 EDS analysis

Figure 3 shows the element mapping obtained by the EDS analysis, while Figure 4 shows the results of the quantitative analysis in the EDS analysis.

A comparison based on the presence or absence of sodium carbonate at the age of 3 h shows that the ratio of aluminium (Al) and silicon (Si) to the content of all the constituent elements was relatively high and the proportion of calcium (Ca) was relatively low in specimen 2 mixed with sodium carbonate. In, as cement hydrates, aluminium is contained in the ettringite ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaO}_2\cdot32\text{H}_2\text{O}$) produced by the hydration reaction of the aluminate phase. It can be judged that aluminium becomes an index of ettringite, even though a large amount of aluminium is present on the crystal surface in the element mapping. Silicon is contained in CSH,

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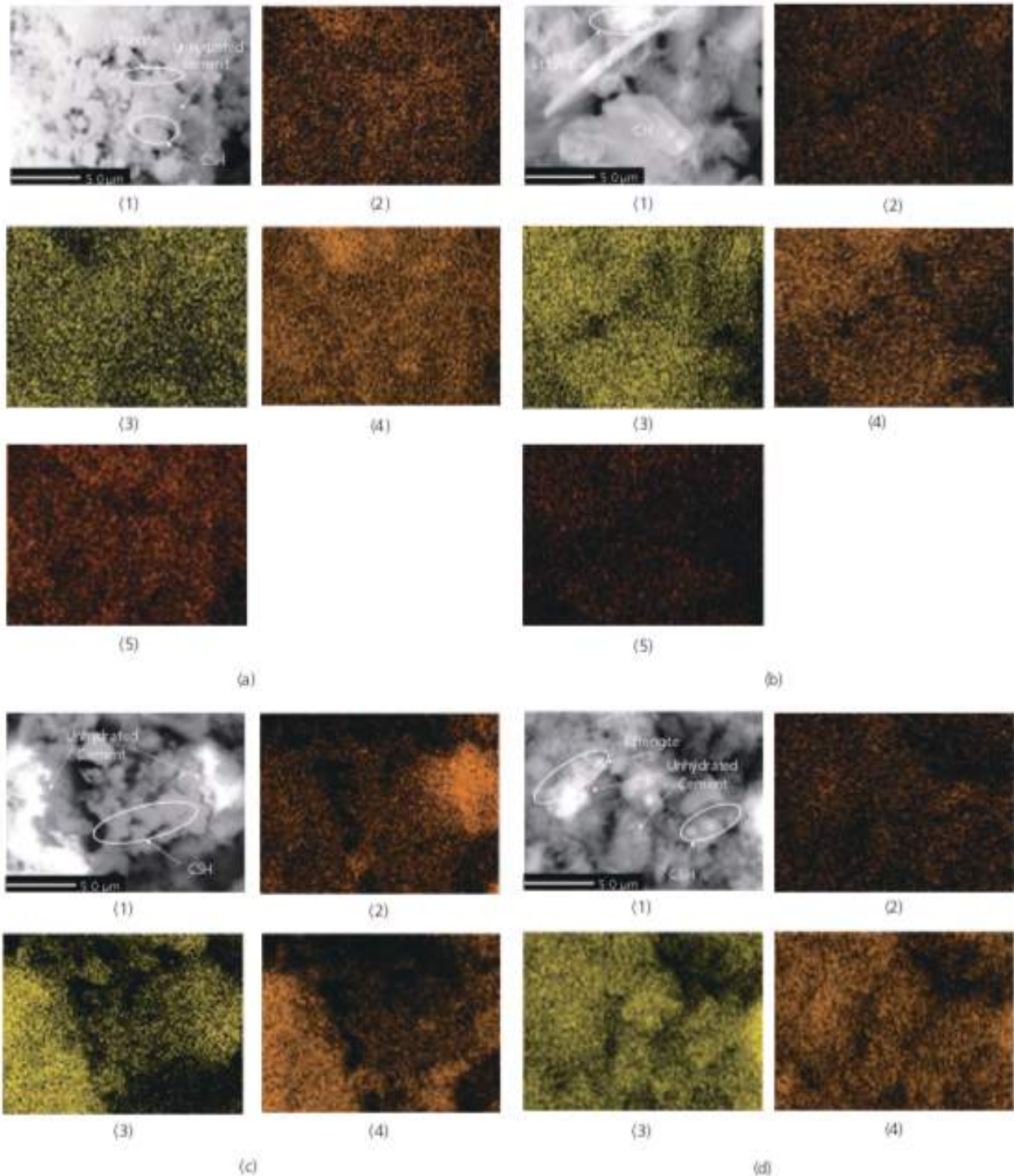


Figure 3. Digital mapping of each constituent element by EDS analysis. (a) Specimen 2 (sodium carbonate, high swelling bentonite), 3 h. (b) Specimen 2 (sodium carbonate, high swelling bentonite), 7 d: (1) SEM, (2) aluminium, (3) calcium, (4) silicon and (5) sodium. (c) Specimen 4 (no sodium carbonate, high swelling bentonite), 3 h: (1) SEM, (2) aluminium, (3) calcium and (4) silicon. (d) Specimen 4 (no sodium carbonate, high swelling bentonite), 7 d: (1) SEM, (2) aluminium, (3) calcium and (4) silicon

which is a hydrate of alite ($3CaO.SiO_2$); it was widely distributed among the crystals and therefore silicon can be judged to be an indicator of CSH. From this point, it is believed that the

amount of ettringite and CSH in CF-CB increased by adding sodium carbonate, which is consistent with the results obtained by SEM observations and XRD analysis. Moreover, because

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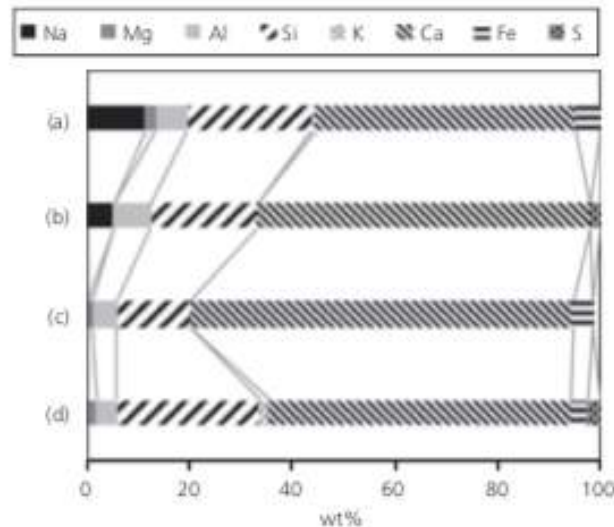


Figure 4. Results of quantitative analysis by EDS analysis. (a) Sodium carbonate; 3 h, (b) sodium carbonate; 7 d, (c) no sodium carbonate; 3 h, (d) no sodium carbonate; 7 d. Na, sodium; Mg, magnesium; Al, aluminium; Si, silicon; K, potassium; Ca, calcium; Fe, iron; S, sulfur

calcium is contained in almost all components of cement, calcium is not suitable for specifying ettringite or CSH. With regard to the cause of the difference in the proportion of calcium, calcium was not observed, as it was ionised in the middle of the hydration reaction in specimen 2, while specimen 4 contained a large amount of unhydrated cement and a great deal of calcium was observed. From the above it is clear that the hydration reaction of cement is seen to progress by adding sodium carbonate, and the formation of ettringite and CSH is promoted.

A comparison based on the presence or absence of sodium carbonate at the age of 7 h revealed that the ratios of aluminium and calcium were high and the proportion of silicon was low in specimen 2 mixed with sodium carbonate. Comparing the results obtained at 3 h and 7 d of age, the proportion of calcium increased in specimen 2, but decreased in specimen 4. It is considered that the calcium decreased because the selectivity of Ca^{2+} is as high as the interlayer cation of bentonite (Christidis and Koutsopoulou, 2013), and Ca^{2+} may not be observed due to the incorporation as ions in the bentonite during the process of the cation-exchange reaction. The increase in calcium during the addition of sodium carbonate is thought to be caused by the formation of calcium carbonate by Ca^{2+} contained as an interlayer cation of the bentonite and CO_3^{2-} from sodium carbonate in the process of the cation-exchange reaction. Also, sodium was observed only in specimen 2 mixed with sodium carbonate, and a comparison at 3 h and 7 d of age showed a decrease as time elapsed. It is thought that the sodium carbonate or sodium, which was crystallised, was taken in the bentonite as Na^+ by the cation-exchange

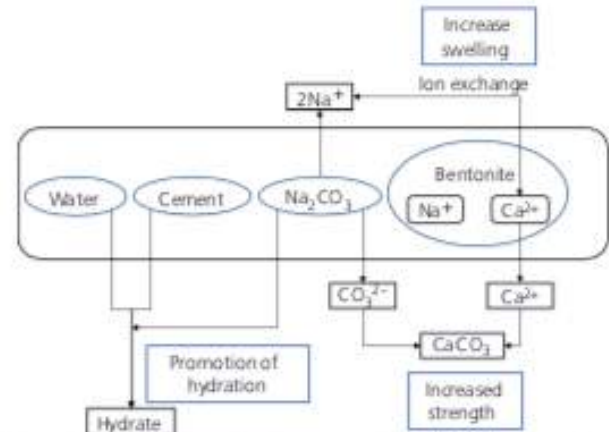


Figure 5. Mechanisms of various properties of CF-CBS based on SEM observations, XRD and EDS analyses. Na_2CO_3 , sodium carbonate; CaCO_3 , calcium carbonate

reaction. It is considered that the cation-exchange reaction of Na^+ from sodium carbonate and Ca^{2+} from bentonite occurs due to the above increase and decrease in calcium and sodium. Calcium carbonate was also present in the results of the XRD analysis, so calcium carbonate was considered to have been generated.

Figure 5 summarises the mechanisms by which various properties of CF-CBS develop based on the results of the SEM observations, XRD analysis and EDS analysis. Na^+ is generated by ionising sodium carbonate in water and Ca^{2+} from the interlayer cations of bentonite that undergo a cation-exchange reaction, so that Na^+ contributes to increasing the swellability as an interlayer cation of bentonite. Increasing the swellability improves the effect of suppressing bleeding. Ca^{2+} binds with the CO_3^{2-} generated by the ionisation of sodium carbonate to form calcium carbonate, which contributes to the increase in strength. Regarding the production of calcium carbonate, sodium carbonate is also produced by carbonating calcium hydroxide produced by the hydration of Ca^{2+} dissolved in cement at the beginning of the test. This leads to the expeditious consumption of calcium hydroxide and promotes the hydration reaction. As ettringite forms quickly, it causes earlier gelling and earlier strength development. In addition, rapid gelling suppresses sedimentation of the cement particles, which also leads to the suppression of bleeding.

5. Conclusions

The mechanisms by which various properties of CF-CBS develop was investigated using SEM, XRD and EDS analyses.

The findings are as follows.

- (a) Calcium hydroxide is carbonated by adding sodium carbonate to produce calcium carbonate. This contributes to the increase in strength of CF-CBS.

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- (b) As the consumption of calcium hydroxide is accelerated by the addition of sodium carbonate, the hydration reaction is promoted and ettringite is generated promptly. This contributes to earlier gelling and earlier strength development of CF-CBS.
- (c) As Ca^{2+} from the interlayer cations of bentonite and Na^+ from sodium carbonate generate an ion-exchange reaction, Na^+ as interlayer cations increase and the swelling property of the bentonite increases. This contributes to the suppression of bleeding of CF-CBS.
- (d) In the process of the cation-exchange reaction, calcium carbonate is formed by Ca^{2+} from the interlayer cations of the bentonite and CO_3^{2-} from sodium carbonate. This contributes to an increase in the strength of CF-CBS.

As a future prospect, it will be necessary to verify the applicability of CF-CBS as a cavity-filling material by implementing field tests and attempting a practical example.

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