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Effect of granules on the workability and the recovery of water tightness of crack self-healing concrete

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A fundamental study on whether improvements in self-healing performance can be achieved by mixing into concrete granules of self-healing agent whose main component is geo-materials instead of self-healing agent in powder form, for the purpose of preventing leakage of water through cracks was investigated. The aim of this research is rapid slump loss compensation based on the granulation process of self-healing agent whose main component is geo-materials that have high reactivity with water and includes a long-term retention of the self-healing capability of concrete incorporating self-healing agent. Comparison of concretes commercially prepared by blending in self-healing agent at ready mixed concrete plants established, through laboratory and field tests, that the 40 kg/m³ granule admixtures based on self-healing agent as a fine aggregate replacement improved the slump of ready mixed concrete and showed a high water leakage prevention effect through self-healing of cracks even after the lapse of 8 months.

Key words: Crack, Self-healing concrete, Water leakage, Granules, Field test.

Introduction

In recent years, the deterioration of concrete structures built during Japan's period of high economic growth (approx. 1954 to 1973) has become apparent. As the occurrence of cracks in concrete structures causes loss of functionality, aesthetic appearance, and durability, there is great social need to suppress it. Especially, East Japan Railway Company reported that maintenance and repair cost of railway bridges and tunnels are estimated at \$10 billion in case of Japan [1]. Furthermore, many potential markets of selfhealing cement-based materials have been reported by civil engineering from many different countries. Breugel reported that the cost for reconstruction of bridges in USA has been estimated between \$20 and \$200 billion. The average annual maintenance cost for bridges in that country is estimated at \$5.2 billion [2]. Li et al. [3] reported the self-healing in concrete materials and mechanical property recovery in selfhealed concrete materials. Henk [4] discussed the possibility of bacterial concrete for the self-healing approach. The authors have been working to develop selfhealing concrete incorporating self-healing agents based on the various mineral admixtures from 1997 [5, 6].

The self-healing mechanism using self-healing agents

that include geo-materials is considered to be a composite action consisting of diverse actions that include expansion and swelling through reaction with water, pozzolanic reaction, and carbonation reaction with CO_2 dissolved in water. Fig. 1 shows the basic design concept of cementitious composite materials with self-healing capability as reported in the previous research [6-8].

The geo-materials used by the authors are montmorillonite minerals such as sodium aluminum silicate hydroxide $[Na_{0.6}Al_{4.7}OSi_{7.32}O_{20}(OH)_4]$ [9]. The water leakage prevention performance is improved through the usage of self-healing agents that include geomaterials, but as self-healing agents that include geomaterials have high reactivity to water, considerable slump



Fig. 1. Design of cementitious composite materials with self-healing capability.

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loss of ready mixed concrete is an issue. Furthermore, stopping water leakage through reliable self-healing of cracks in concrete structures over long periods requires methods to preserve self-healing capability on a long term basis. Therefore, this study focused on three issues: (1) improvement of workability on self-healing concrete by granules, (2) performance evaluation of crack self-healing concrete incorporating various granules (3) The field test of self-healing concrete incorporating granules [10-12].

Experimental Methods

Materials

The self-healing agents were prepared based on selfhealing performance as reported in the previous research shown in Fig. 1[7]. The expansive agent, geomaterials and chemical agent used were commercial products produced in Japan. Fig. 2 shows self-healing behavior of cementitious composite materials incorporating self-healing agent. A crack with a width of 0.2 mm was induced in hardened self-healing cementitious paste with 45% W/C at the curing age of 120 days, and from the 3 days after water immersion, re-hydration products formed in the crack and crack healing was confirmed. Therefore, this agent was selected in order to fabricate granules in this research.

Preparation of granules based on self-healing agents

Fig. 3 shows the concept of crack self-healing in hardened concrete incorporating granules. When a crack occurs in hardened concrete, granules break up, crystals precipitate through the reaction, dissolution, diffusion, etc., of components contributed to the selfhealing agent through water supply in the cracked parts, and the crack closes. The two challenges of slump loss compensation and long-term preservation of the selfhealing capability of ready mixed concrete are thought to be solvable through the use of such granules. An additional issue is that self-healing agent is traditionally incorporated in concrete through partial substitution of cement, so that mixing in large quantities of self-healing agent in powder form in ready mixed concrete causes reduced compressive strength besides slump loss. By contrast, in the case of granules, the self-healing agent is done through fine aggregate substitution, which is thought to improve compressive strength also. Fig. 4(a) shows granules simply manufactured by stirring ordinary Portland cement (OPC) in a mortar mixer while dropping water with a dropper. In order to process powder into granules, water (liquid binder) is the minimum requirement for granulating the powder to produce granules. However, in the case of water only, the granules obtained by granulating the self-healing agent should be brittle and broken up when they are mixed into the concrete. Therefore, the addition of powder binder such as cement is necessary to maintain the shape of granules, but if the compressive strength of the granules becomes



Fig. 2. Self-healing behavior of cementitious composite materials incorporating self-healing agent (Water/Binder ratio = 0.45).



Fig. 3. Application concept of self-healing concrete incorporating granules for the water tightness.

excessive as the result of the hydration of the cement, the granules will become hard to break down when cracks occur in the concrete and the self-healing capability risks declining. To delay the hydration of the cement in the granules, it was decided to replace some of the water with ethanol, which is a hydrophilic volatile organic solvent.

Based on previous research [10] of the suitable water/cement ratio (W/C) for the manufacture of granules of self-healing agent that includes geomaterials, the fixed ratio of W/C = 20% was adopted. 200 grams of OPC per batch were mixed in a mortar mixer while adding 40 ml of a mixture of water and ethanol with a dropper to form granules. The substitution ratio of ethanol for water was experimentally varied at that time. The granules thus obtained were cured under sealed condition for 7 days.

Fig. 4(b) shows the result of XRD examination of the progress of hydration of the OPC contained in the granules. From the peak intensity of $Ca(OH)_2$, which is a hydration product of cement, it was confirmed that it is possible to delay initial hydration of cement by substituting ethanol for water at the volume ratio of 30% or higher. Therefore, the substitution ratio of ethanol was set to 30 vol % for the subsequent fabrication of granules.

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Fig. 4. Fabrication of OPC granules for the preliminary research (a) OPC granules (b) Effect of ethanol on the cement hydration.



Fig. 5. Outline of the granulator.

Table 1.	Mix-pro	portions	of granules	(Unit:	%)
				· · · · ·	,

		Self-healing agent		Bin	*PVA		
Sample	Expansive agent	Geo-materials (A, B type)	Chemical additives	Low-heat portland cement	Water + Ethanol	fiber (kg/m ³)	
EG	0	O 42	0	42	18	0.08	
NEG	_	O 33	0	49	18	0.08	

*PVA fiber: $\phi 27 \ \mu m \times \text{length } 6 \ \text{mm}$, density: 1.3 g/m³.

The diameter of most of the granules manufactured with the mortar mixer was on the order of $300 \,\mu\text{m}$ or approximately 5 mm, greatly differing from the particle size distribution of fine aggregate used for concrete. This is thought to be due to the shape of the blades of the mortar mixer and other factors not being well suited for granulation. It was thus decided to use a granulator of the type used to produce pharmaceutical

and food products (capacity: 25 l, processing amount: 12 kg/batch, processing time: 10 minutes/batch) to produce granules as shown in Fig. 5.

Table 1 shows the mix-compositions of the granules. Two types of granules, with and without expansive agent (EG and NEG) were fabricated. Low-heat portland cement, which has self-healing capability, was used as the cement for binding purposes. With regard to the



Fig. 6. Manufacturing process of granules with PVA fiber.

NEG mix, the reduction in expansive agent in the selfhealing agent was made up with low-heat portland cement. For the liquid binder, a mixture containing 70 vol% water and 30 vol% ethanol, based on the abovementioned previous results [10], was used. As fiber, PVA short fibers (27 μ m diameter × 6 mm length, 1.3 / cm³ density), which have been reported to contribute to self-healing of cracks in the ECC (Engineered Cementitious Composites), were added in the proportion of 0.08 kg/m³. [13, 14]

Fig. 6 shows the manufacturing process of granules. Following curing under sealed condition for 7 days, the manufactured granules were impregnated with a fatty acid saturated ethanol solution and then underwent air drying outdoors to evaporate the ethanol, leaving a thin impermeable membrane of fatty acid on the surface of the granules.

Fig. 7 (a) and (b) shows the powder and granules of self-healing agent and Fig. 7 (c) shows the sieve analysis results. The granules manufactured with the granulator shown in Fig. 5 were within the normal size range for fine aggregate of JIS A 5308 and confirmed to have a size distribution equivalent to that of fine aggregate.

Fabrication of new self-healing concrete incorporating granules

In order to fabricate the self-healing concrete (SHC) incorporating granules, various concrete tests were conducted at the actual ready-mixed plant of Japan. Table 2 shows the mixing proportions of concretes; a W/C ratio of 57.9% and an S/A ratio of 45.2% were applied to all concretes. (general strength = 24 N/mm², target slump = 12 cm, and target air content = 4.5%.)





Fig. 7. Fabrication of EG and NEG type granules (a) EG type (with expansive agent) (b) NEG type (without expansive agent) (c) Sieve analysis of granules.

	W/C S/A (%) (%	C/A	A Air 6) (%)	Unit (kg/m ³)					
Sample		5/A (%)		Water	Cement	Self-healing agent	Fine Aggregate	Coarse Aggregate	SP
Plain						_	826		2.3
P-SHC	57.9 45.	45.0	45.2 4.5	168	290	40	786	1019	8.7
EG-SHC		45.2							8.7
NEG-SHC									8.7

Table 2. Mix-proportions of concrete.

(P: powder type self-healing agent of EG, EG & NEG: granules type self-healing agent).



Fig. 8. Outline of artificial water-retaining structure.

The three self-healing agents such as one powder type and two granules types were substituted for fine aggregate in the amount of 40 kg/m³ for each type. The dosage of super plasticizer (SP) was $C \times 0.8\%$ for plain concrete and $C \times 3.0\%$ for the three types of SHC.

By measuring the slump and air content of concrete before and after discharge, the slump improvement effect obtained through the use/non-use of granules was investigated. Concrete $10\Phi \times 20$ cm cylinders were prepared after conducting the concrete slump test for the compressive strength test and the water tightness test of cracked concrete in the laboratory. The compressive strength was measured by JIS 1108 after 7, 28, and 270 days.

As specimens for field tests, one box shaped RC specimen such as shown in Fig. 8 (amount of concrete used = 0.48 m^3 , interior volume of water tub = 0.2 m^3) was fabricated for each mix proportion. The difference in thickness between the members on the south side and the north side of the specimen is to allow verification of the self-healing effect through differences in the flow path length by inducing cracks in the south side and north side.

Fig. 9 schematically shows the casting process of self-healing concrete and the crack-inducing process in the specimens. The formwork of the box shaped RC specimens (artificial water-retaining structures) for the field test was removed at 7 days curing and the

<image>

Fig. 9. Fabrication process of self-healing concrete incorporating granules and crack induction method.

specimens were then air cured outdoors with a cover placed over them to prevent the ingress of rainwater. After three months of curing, a load was applied with a hydraulic jack against the internal walls of the specimens to induce cracks (with crack width between 0.2 mm and 0.7 mm) in the south side and the north side, except for the box shaped RC specimen made of plain concrete, in which a crack was induced in the south side only because of the risk of breaking through the wall of the specimen during crack induction. Then tap water was poured into the box shaped RC specimens and the field test was commenced to verify the water leakage prevention effect like for the cylindrical specimens.

Verification methods for the recovery of water tightness on fabricated self-healing concrete

In case of the water tightness test, the specimens were split longitudinally using a compression tester.

The fractured surfaces of the split specimens were cleared of fine particles through the use of high pressure air to suppress the occurrence of clogging favorable to self- healing (in order to make severe condition). Then, using two steel hose clamps to restrain each specimen, the crack width was adjusted between 0.2 mm and 0.3 mm under digital microscope observation. Crack width measurement was done at a total of six points, three at the top end face and three at the bottom end face of the specimen. A vinyl chloride pipe measuring 100 mm in diameter and 100 mm in height was attached to the top end of the specimen to provide a water head of approximately 80 mm, and the space between the specimen and vinyl chloride tube as well as the cracked areas along the side of the specimen were closed up with silicone resin. The laboratory water permeability test was done by pouring tap water into the vinyl chloride tube in a 20 °C environment and comparing the degree of self-healing of the cracks yielded by the granules of self-healing material by recording water leakage over time.

The water tightness test in the laboratory was conducted by maintaining a constant pressure, continuous flow state in the vinyl chloride pipe as shown in Fig. 10(a), and measuring for five minutes the amount of water leaking from the bottom end face of the cracked specimens on days 0, 1, 3, 7, 14, 21, and 28, counting from the time the vinyl chloride pipe was filled with water. As shown in Fig. 10(b), measurement was done by attaching a funnel to the bottom end face of the specimens and attaching this assembly over a graduated cylinder to allow measurement of the amount of water leaked from the cracked bottom end face only. The water pressure applied to the specimens when the vinyl chloride pipe was filled with water was calculated to be around 0.8 kPa. The relationship between changes in the water leakage amount and changes in pH was determined through measurement of the pH of the leaked water with pH test paper. The water leakage prevention effect of the granules and powder of self-healing agent was also investigated on a comparative basis before and after the permeability test by checking for the presence of precipitates in the cracks and cross-sections of the specimen with a digital microscope.

The field test was also conducted by measuring the changes in water level drop from the level of 1 cm



Fig. 10. Permeability test setups (a) Water permeability test (b) Measurement of water leakage.

from the top of the box, at 60-minute and 10-minute intervals on days 0, 6, 8, 16, 26, 44, 93, 123, and 135, counting from the time the inside of the box shaped RC specimen was filled with water. All the box shaped RC specimens were filled with tap water following measurement and at days 2, 4, 7, 14, 18, 25, 33, 37, 41, 73, 78, and 112 following the start of permeation.

Results and Discussion

Effect of granules on the workability and compressive strength of self-healing concrete

Table 3 shows the effect of granules on the workability of self-healing concrete. Compared to P-SHC, which does not incorporate granules of self-healing agent, EG granules and NEG granules present improved slump values. In case of EG granules, material segregation was occurred partially, because of an excessive dosage of superplasticizer (SP). From these results, the addition of self-healing agent in the form of granules to concrete is considered to compensate slump loss. With regard to air content, while the incorporation of self-healing agent in powder form tends to cause excessive air content at the initial stage, the incorporation of granules keeps air content at a level comparable to that in plain concrete.

Fig. 11 shows the results of the compressive strength of the various concretes. No differences in compressive strength caused by the use or non-use of granules were observed at 7 days curing. Neither was a decline in compressive strength observed at 28 days and 270 days curing; on the contrary, a slight increase in strength compared to the plain concrete was seen. Based on these results, it was found that the substitution of self-healing

Table 3. The effect of granules on the workability of self-healing concrete.

	8	5	8			
Sample	Slum	p (cm)	Air con	itent (%)	Temperature (°C)	
	Initial (5 min)	Final (30 min)	Initial (5 min)	Final (30 min)	Initial (5 min)	Final (30 min)
Plain	14.0	12.0	5.0	3.5	14	15
P-SHC	16.5	14.5	10.2	5.2	14	15
EG-SHC	23.5	SF40	3.8	3.4	14	15
NEG-SHC	19.0	19.0	2.0	3.4	14	15

SF: Slump Flow.

agent for fine aggregate in the amount of 40 kg/m^3 does not have an adverse effect on compressive strength. In the case of EG granules, compressive strength was lower than that of plain concrete, which is thought to be due to an excessive slump value (material segregation). However, at 270 days curing, the compressive strength was somewhat higher than that of plain concrete, suggesting that the strength decrease is only in the initial stage.

Effect of granules on the recovery of water-tightness in cracked concrete

Fig. 12 shows the time-dependent changes in water leakage amount observed in the laboratory test, and Fig. 13 shows the time-dependent changes of the pH value of the leaked water immediately after leakage measurement. The results in these figures are the averages of three specimens for each mix proportion. Fig. 14 shows the changes in the cracks before and after water permeability test of specimens that incorporate granules. One can see in Fig. 12 that although the crack width of the specimens was adjusted between 0.2 mm and 0.3 mm, there is disparity among the initial leakage values. This is thought to be due to the differences in the topography of the crack surfaces (fracture surfaces resulting from splitting) among the specimens. However, compared to plain concrete, SHC incorporating selfhealing agent showed a tendency to have lower initial leakage values, whether or not granules of self-healing agent were included, and whether or not the granules contained expansive agent. This is considered to be due to the water cutoff effect obtained from the initial phase through the expansion and the swelling effects of the geo-materials included in the self-healing agent. Examination of the subsequent time-dependent changes in leakage reveals a large drop in leakage in the first day of permeation through the effect of water absorption, etc., even in the plain concrete, and the continuation of this effect until the fifth day, but no major change was seen after that and the leakage value never became nil. On the other hand, for SHC too, the leakage value greatly dropped in the first day of permeation, whether or not the concrete included granules, and the leakage value went on declining on the 14th and 21st day of permeation. The leakage value was finally observed to become almost nil on the 28th day after water permeability test.

Fig. 13 shows that the pH value of the leaked water dropped sharply from approximately 12 immediately after permeation to approximately 8 on the first day, and went on to decline to approximately 7. On the other hand, in the case of SHC, the changes in pH were less pronounced whether or not granules of self-healing agent and expansive agent were included. The pH value of the leaked water was approximately 11 on the first day of permeation, went on to decline to 8 on the 14th day, and remained level after that. From these



Fig. 11. Effect of granules on the compressive strength.



Fig. 12. Effect of granules on the recovery of water tightness under water leakage.



Fig. 13. Effect of granules on the pH of water after permeability test.

results, it is thought that in the case of SHC, the movement of water inside the cracks was suppressed by the expansion of the swelling materials, leaving the other self-healing components, cement hydrates, etc., in place rather than flushing them out and thus allowing them to heal the cracks, thus demonstrating a high water cutoff effect. Moreover, based on the fact that similar changes were observed for SHC up to the 14th and 21st day after water permeability test as shown in Fig. 12 and Fig. 13, it is considered possible to evaluate the retention of self-healing components and the water cutoff performance through measurement of



Fig. 14. Self-healing behavior of concrete incorporating various self-healing agent.



Fig. 15. Cross section of specimen after water permeability test.

the pH of the leaked water. In order to confer to SHC the full water cutoff effect, it is important to suppress leakage from the initial leakage phase through the use of swelling materials, etc. This allows the long-term retention of the self-healing components in order to obtain a strong water leakage prevention effect.

Based on the results of Fig. 14(c) and (d), the precipitation of crystals in the cracks of the EG-SHC and NEG-SHC following permeation was confirmed. In the case of P-SHC in Fig. 14(b) as well, the precipitation of crystals was observed, but not in the case of plain concrete in Fig. 14(a).

Fig. 15 shows a cross-section of samples after water permeability test. Surfaces of the cross section 10 cm, 15 cm, and 20 cm from the water inflow site (from top and bottom) were investigated by the digital microscopy. From these results, it was found that in the case of P- SHC, which has high water leakage prevention capacity, ample crystals precipitated so as to cover the surface of the fine aggregate exposed at the split surfaces. The amount of crystal precipitation tended to be particularly pronounced for SHC-granules, compared to SHCpowder. This suggests that the use of self-healing agent in the form of granules makes it possible to reduce the amount of self-healing agent for obtaining the requisite water leakage prevention performance, compared with the use of self-healing agent in powder form.

Based on the laboratory test results, specimens for 8 months demonstrated ample water leakage prevention effect whether or not they included granules of self-healing agent and expansive agent. Therefore, at the approximately 8 months curing, the long-term retention effect of the self-healing component in the form of granules could not be confirmed. However, the fact that



Fig. 16. Relationship between water level drop and various self-healing agents.



Fig. 17. Crack closing effect of various concrete after water leakage test in the field.

the concrete specimens that incorporated the selfhealing agent whose principal component was geomaterials used in this study processed in the form of granules rather than powder exhibited a larger amount of crystalline precipitate on their cross-sections even though the amount of self-healing agent was lesser, indicates that the performance of SHC can be improved by processing self-healing agent into granules.

Verification of granules effect from field test

Fig. 16 shows the time-dependent changes in the water level drop recorded during the field test of the box shaped RC specimens. Fig. 17 shows the cracks induced in the box shaped RC specimens and the appearance of the cracked areas immediately after the start of the water permeability test and at 123 days of permeation. In the case of the plain concrete specimen too, the water level drop appears to decrease as permeation test days pass. However, this is considered to be due to the water cutoff effect as the cracks healed naturally through CO₂ supplied from the atmosphere and also because in the case of the box shaped RC specimen made of plain concrete, cracking could not be induced in the north side of the specimen. As shown in Fig. 17(a), white-colored precipitates were actually found in the cracks of the plain concrete specimen too. Moreover, for the other box shaped RC specimens too, the water level drop appears to diminish as the permeation test days pass, and white-colored precipitates were found, like in the plain concrete specimen, in the cracks on the south and north sides as shown in Fig. 17. The difficulty of controlling the crack widths of the box shaped RC specimens prepared for the field test and the resulting differences in crack width made relative comparison difficult. Particularly in the case of NEG-SHC, the bottom wall of the specimen was broken through during crack induction, so that the timedependent initial water level drop was greatest, and crack healing was considered to be extremely difficult. However, as seen in Fig. 16(d), the water level drop of NEG-SHC greatly decreased on the sixth day of permeation. This is considered to be due to a strong water leakage prevention effect obtained by mixing in granules of self-healing agent, similar to the laboratory test results. The white-colored precipitates that appeared on the crack surfaces of the box shaped RC specimens in Fig. 17 were determined through XRD to consist principally of CaCO₃, which is thought to have been generated from inorganic carbonates in the self-healing agent. Further research will be continued on increasing of the self-sealing and the recovery of mechanical sense.

Conclusions

In this study, the new method of self-healing design to improve water tightness in cracked concrete was suggested, and the self-healing properties of cracked concrete using various self-healing agent (powder and granules) were investigated. The following conclusions can be drawn from the experimental study.

(1) Binder such as low-heat cement, water, and ethanol was added to powder of self-healing agent consisting principally of geo-materials that have high reactivity with water to achieve granulation, yielding granules with a size distribution comparable to that of fine aggregate used for concrete.

(2) The 40 kg/m³ admixture of self-healing agent in granule form as a fine aggregate replacement was found to improve the workability of ready mixed concrete without lowering compressive strength, compared with using self-healing agent in powder form.

(3) Investigation of performance in terms of preventing water leakage from cracks through laboratory tests and field tests through the incorporation of self-healing agent at a ready mixed concrete plant found that the use of granules of self-healing agent as a fine aggregate replacement improved the slump of ready mixed concrete and produced a high water leakage prevention effect through self-healing of cracks even at the 8 months curing.

(4) The findings suggest that processing self-healing agent into granules allows reduction of the amount of self-healing agent for obtaining the required water leakage prevention performance, compared with the use of self-healing agent in powder form, and can improve the water leakage prevention performance of self-healing concrete.

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