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Effect of oyster shell substituted for fine aggregate on concrete characteristics: Part I. Fundamental properties

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Abstract

An experimental study was carried out to investigate the recycling possibilities for fine aggregate of oyster shells (OS), which is an industrial waste, disposed of in open dumps at coastal oyster management areas. For this purpose, the chemical components of OS and reactivity of OS with cement paste were examined. More specifically, mechanical characteristics of fresh concrete and hardened concrete were quantitatively investigated in terms of fineness modulus (F.M.) and substitution rate (SR) of crushed OS. In addition, the Part II paper presents the performance of concrete up to 1 year with OS substituted for fine aggregate.

Test results show that the interaction between OS and cement paste did not occur and the workability of concrete decreased with F.M. decrease and SR of OS increase. In addition, it was found that mixing of OS did not cause reduction in the compressive strength of concrete at age 28 days and development of compressive strength was faster as SR of OS increased. Elastic modulus of concrete substituted with crushed OS decreases as SR increases. The decrease was approximately 10% at SR of 20%.

Keywords: Oyster shell; Substitution rate; Fresh concrete; Compressive strength; Elastic modulus

1. Introduction

Due to the shore ostreaculture, approximately 3.0×10^5 tons per year of oyster shell (OS) occurs in the southern sea and a portion of western sea of South Korea (especially, in Koje and Tongyong cities of South Korea). Currently, although a limited amount of OS is reused for OS seed collection breeding and fertilizer, etc., the extent of reuse is restricted due to problems of limits on reuse volumes, security of the container freight station at the time of handling, soil solidification from spreading of OS on farmland, and economic problems. Accordingly, most OS are not reused but are illegally discarded in the surrounding area.

When OS is reclaimed or accumulated, however, it becomes a source of environmental damage and pollution by leakage of water as well as difficulty of landfill security and maintenance and control of landfill. In addition, in the case of accumulating it in the surrounding area, OS is considered as undesirable material due to a serious odor and pollution and it causes a negative impact on the local health and living environment. Thus, objections from local residents occur continuously.

To solve this problem, programs under government control were carried out and many calcium and fertilizer factories were built to increase the amount of OS reused. Currently, however, treatment efficiency compared to investment cost is not satisfactory. Accordingly, new methods are necessary to solve the enormous treatment problem of OS. More specifically, when OS is reused as a substitution material, the effects must be carefully evaluated since OS contains some organic material and also

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contains a chloride. Methods must also be economically competitive compared to existing treatment expenses.

Some research [1,2] has been published on the possible use of OS as soft ground improvement materials or sand pile materials for civil construction in Japan. However, this has not been implemented and additional studies were not performed.

In the meantime, in South Korea, availability of construction materials has become a serious problem. More specifically, availability of good sand is a problem that must be solved when we consider the scale of the future construction market. Accordingly, much research on the usage of land sand, sea sand, etc. [3,4], has been performed. Research on utilization of crushed OS as substitution materials for fine aggregate mixed in concrete can contribute to disposal of OS and development of new concrete materials.

For this purpose, the chemical components of OS and reactivity with cement paste of OS were examined. The characteristic variances of fresh concrete and hardened concrete substituted with OS were quantitatively investigated in terms of fineness modulus (F.M.) and substitution rate (SR) of OS. For practical application, research was performed on durability and performance of concrete up to 1 year using OS as substitution materials of fine aggregate is described in an accompanying paper [5].

2. Experimental program

2.1. Material properties

Concrete for cylinder specimens was produced with Type I Portland cement, river sand (specific gravity: 2.61, absorption ratio: 0.81%, F.M. = 3.44), and crushed coarse aggregate (specific gravity: 2.74, absorption ratio: 0.60%, G_{max} = 25 mm). OS mixed in concrete is reduced to powder to pass 5 mm sieve using a jaw crusher after dehydration in a dryer at 110 ± 10 °C for 24 h. The dried OS, as it is, is used to simulate the practice expected in fields.

Table 1 shows the physical characteristics of OS. Air entraining water reducing admixture (AE WRA, naphthalene type) is used to compensate for reduction of workability with addition of OS and the dosage of admixture is fixed as 0.3% of unit cement amount. A vibrator was also used to improve consolidation of concrete. All specimens were removed from the mold after 24 h and were wet-cured in a water chamber at 23 ± 2 °C for 28 days before the testing date.

Table 1
Physical characteristics of OS

Type of OS	Specific gravity	Absorption ratio (%)	F.M.
Type A	2.39	4.40	2.7
Type B			2.1

Table 2
Concrete mixture proportions

Type of mixture	w/c (%)	s/a (%)	Unit weight (kg/m^3)			
			W	C	S	G
NN0	45	43.2	180	400	725 (0)	1002
OA05 OB05	45	43.4	180	400	689 (36)	998
OA10 OB10	45	43.6	180	400	653 (73)	995
OA20 OB20	45	44.0	180	400	580 (145)	988

Values in parentheses mean the substitution amount of OS dried.

2.2. Mixture proportions and test variables

Mixture proportions of concrete are selected to investigate the variance of concrete characteristics with substitution of OS. The target base slump of concrete is 8 ± 1 cm, the air content is 5 ± 1% when AE WRA was used. Mixture proportions excluding AE WRA are listed in Table 2. In this table, the values (0, 36, 73, and 145 kg/m^3) in parentheses are the substitution amounts of OS. To evaluate the contribution of AE WRA to reduction of workability with OS substitution, tests comparing the effect with usage of admixture (dosage rates: 0.0 and 0.3% of cement weight) in the same mixture proportions were also performed. The water–cement ratio (w/c) in Table 2 is a value required to ensure durability of marine concrete [6–8]. The expected average concrete compressive strength f'_c is 30 MPa.

Two types of OS were used and SR of OS was determined considering chloride ion amounts contained in OS to satisfy the requirements (0.3 kg/m^3) of Korea Concrete Institute (KCI) and Architectural Institute of Korea (AIK) Codes [6,9]. More specifically, SR was limited to 10% based on OS composition test results. However, to consider the case of 0.6 kg/m^3 substitution content as an exception to Code rules, SR of 20% was also selected for a comparison test. Therefore, SR of OS for fine aggregate increased to a maximum level of 20%. In Table 2, 0, 36, 73, and 145 represent SR of 0, 5%, 10%, and 20%, correspondingly. Also, to investigate the influence of curing time the mechanical tests were carried out at three ages.

2.3. Experimental method

Experiments in this study are divided into three groups:

(1) to evaluate the chemical components of OS and reactivity of OS with cement paste,
(2) to compare the rheological characteristics of fresh concrete with SR, and
(3) to investigate the mechanical characteristics of hardened concrete with SR.

ϕ 10 × 20 cm cylinder specimens are used to determine the development of concrete compressive

Table 3
Specimen nomenclature

Nomenclature of specimen	AE WRA (%)	Type of OS	SR of OS (%)
NN0	0.0	–	0
OA05	0.0	Type A	5
OA10			10
OA20			20
OB05		Type B	5
OB10			10
RA05	0.3	Type A	5
RA10			10
RA20			20
RB05		Type B	5
RB10			10

strength, splitting tensile strength, and elastic modulus. The measured values are determined from three identical specimens. Cylinder specimens are prepared according to ASTM C 469 [10] and a total of 150 specimens were prepared in this experiment. The specimen nomenclature is presented in Table 3. The modulus of elasticity [11], E_c, outlined in ASTM C 469 [10], was used and was calculated at 40% of the ultimate load.

3. Test results and evaluation

3.1. Chemical characteristics of OS

3.1.1. Component analysis of OS

To investigate the main components in OS, a sample of OS was collected in the oyster managing area of Tongyong city, and XRF test (X-ray fluorescence spectroscopic analysis) was performed on the sample. To perform XRF test, OS was pulverized after dehydration in a dryer at 100 °C for 24 h. The results of XRF test are shown in Table 4. From Table 4, the weight ratio of the components can be seen. To obtain the composition of the total loss weight, however, additional tests must be performed because a loss of the content of organic materials and CO_2 by heating. Though we cannot directly obtain the weight of calcium carbonate ($CaCO_3$) from Table 4, it can be obtained indirectly by Eq. (1) using the composition molecular formula of calcium carbonate. Also, the weight of calcium carbonate can be obtained by TG-DTA test (Thermo Gravimetry Differential Thermal Analyzer). The value obtained by TG-DTA test shows 97.8%. However, CO_2 weight may be affected by heating. The results using the two methods showed similar values.

Weight of calcium carbonate ($CaCO_3$)

$$= \frac{CaCO_3 \text{ molecular weight}}{CaO \text{ molecular weight}} \times CaO \text{ wieght ration } (\%)$$

$$= \frac{100}{56} \times 51.06 = 91.18(\%) \qquad (1)$$

3.1.2. Reactivity with OS and cement

As aforementioned in component analysis results of OS, OS is primarily composed of calcium carbonate, with a small quantity of mineral and organic materials. Therefore, when OS is substituted as fine aggregate it is necessary to evaluate whether or not calcium carbonate ingredients of OS react with cement. To clarify this, XRD (X-ray diffraction analysis) comparison test was performed. Also, the electron microscope photography (SEM) of the interface between OS and cement hydrate was done. XRD test results with specimen type are described in Fig. 1. In samples for XRD test, shell, opc-7, and s10-7, correspondingly represent materials made by only OS, materials (mortar) made by OPC and cured during 7 days, and materials made by substitution of 10% OS and cured during 7 days. For opc-7 and s10-7, *w/c* was 50%.

According to Fig. 1, disadvantageous reactions at the interface between cement and OS or formation of new materials were not found, and cement hydrate and OS are independently detected. That is, it is noted that, even though OS is mixed with cement paste, OS does not affect the cement hydrate and only performs a role as filler in concrete matrix. To visually investigate this phenomenon, SEM photography for cement paste ($w/c = 50\%$) at age 7 days substituted with 10% OS (of cement weight) was performed. The result is shown in Fig. 2. From Fig. 2, it is determined that there was no disadvantageous reactivity between OS and cement hydrate.

3.1.3. Investigation of organic impurities

OS used as substitution materials of fine aggregate might contain a small amount of organic impurities. Accordingly, an investigation on potential harm from organic impurities included in OS should be done. For this purpose, organic impurities test for sand substituted with OS was performed according to ASTM C 40 [12]. Six types of fine aggregate (NN0, OA05, OA10, OA20, OB05, and OB10) were selected, composed of one standard type (NN0) and 5 types with SR. The test results show that colors of all cases (5 types' samples substituted with OS) appeared lighter than the color of standard solutions. It was noted that there is no

Table 4
Chemical ingredient results of OS (unit; wt, %)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	Mn_2O_3	P_2O_5	SrO	Ig. loss*
2.00	0.50	0.20	51.06	0.51	0.60	0.06	0.58	0.02	0.02	0.18	0.09	44.16

* Including CO_2 and organic materials as the loss of the content by materials heating.

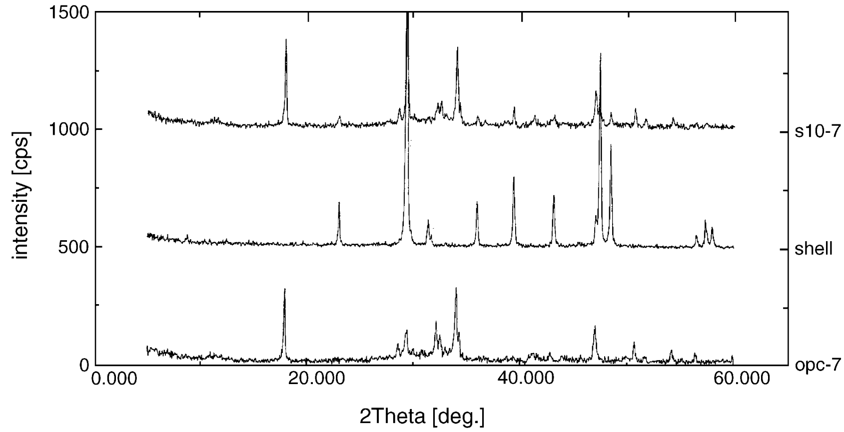

Fig. 1. XRD test results.

deep color changes even if OS is substituted up to 20% of sand weight. Accordingly, it can be concluded that a small amount of organic impurities included in OS do not represent noxious ingredients in the concrete.

3.2. Characteristics of fresh concrete substituted with OS

3.2.1. Slump

The crushed OS is expected to greatly influence workability of concrete because the particle is a dried, thin plate type. Therefore, to quantitatively evaluate the effect of OS substitution on workability of concrete, the slump changes with SR of OS are measured. SR of OS, for both F.M. values, was varied up to 20% of fine aggregate weight. The above procedure was also used for the concrete mixture with AE WRA. The test results are shown in Fig. 3.

As can be seen in Fig. 3, slump value is proportionally decreased with SR increase. In this test range, however, F.M. of OS has an insignificant effect on the workability of concrete substituted with OS. In addition, in the case of concrete mixture using AE WRA, the workability of concrete is improved. However, the slump value decreased for large SR. In any future study, the addition of proper admixture content should be selected to compensate for the reduction of workability due to high substitution of OS.

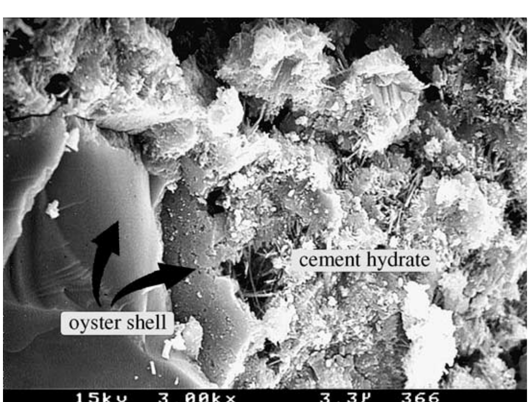

Fig. 2. SEM photography for interface of cement hydrate and OS particle.

3.2.2. Air content

To compare the effect of SR of OS on air content in concrete, air content for the same mixtures as used in the slump tests is measured by the pressure method. The results are shown in Fig. 4. According to Fig. 4, nearly constant values were measured regardless of types and SR of OS when AE WRA is not used. This is because air existing in concrete is entrapped air mixed at mixing and casting time. This air content does not contribute to durability improvement and is not suitable for marine concrete provisions [6–8].

AE WRA was added to minimize the above problem occurring in concrete substituted with OS. The entrained air variance of this case is clearly different from case without addition. That is, if AE WRA is used, the air content is significantly increased. However, the degree of increment with SR and types of crushed OS is quite variable. For example, the case of type A OS shows the considerable air content increase up to 10% SR, but air content did not increase at 20% SR. Meanwhile, the type B OS shows that there is no increment of air content even at 10% SR. This is because the dried OS adsorbs AE WRA including free water inside the concrete. In addition, it is noted that the absorption increases as F.M. of OS decreases. Therefore, it is necessary to establish the optimum amount of available admixture with SR of OS. However, this result is not conclusive because only a limited number of mixes were tested.

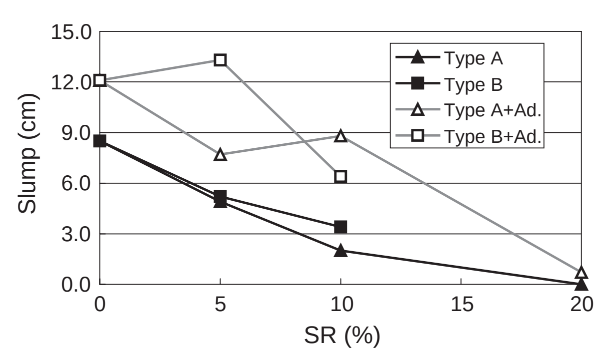

Fig. 3. Slump variance with SR of OS.

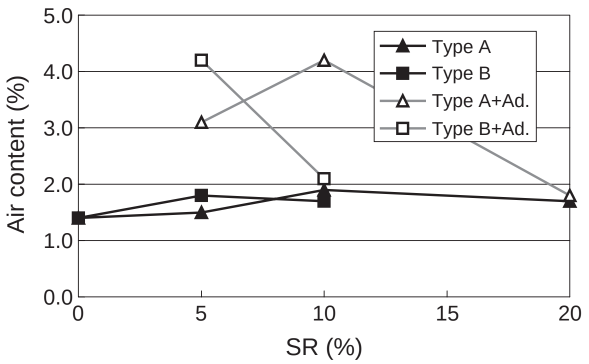

Fig. 4. Air content variance with SR of OS.

3.2.3. Setting time

The setting time test can be used to indirectly estimate the development of concrete strength at an early age. More specifically, when OS is used as concrete materials, it is very important to investigate the potential for disadvantageous reaction of concrete with OS. Therefore, the setting characteristics test considered the type of OS, SR of OS, and addition or not of AE WRA as test variables. The test results are shown in Table 5.

According to the test results, the setting time shows a nearly constant value regardless of type and SR of OS. However, the setting time is delayed approximately 1 h independent of type and SR of OS when AE WRA is added. This is a result of the effect of admixture, which delays the reaction between cement and mixing water. However, the effect was insignificant. The setting time test should be performed under a constant temperature condition because high temperatures shorten the setting time [11]. In this study, however, some deviation occurred in the temperature due to the laboratory internal conditions.

When we take into account the effect of temperature on the test results, it can be concluded that the final setting time is not greatly affected by substitution of OS and the setting time variance problem due to OS did not occur. There is no special concern about setting time by substitution OS up to a maximum of 20% of fine aggregate weight.

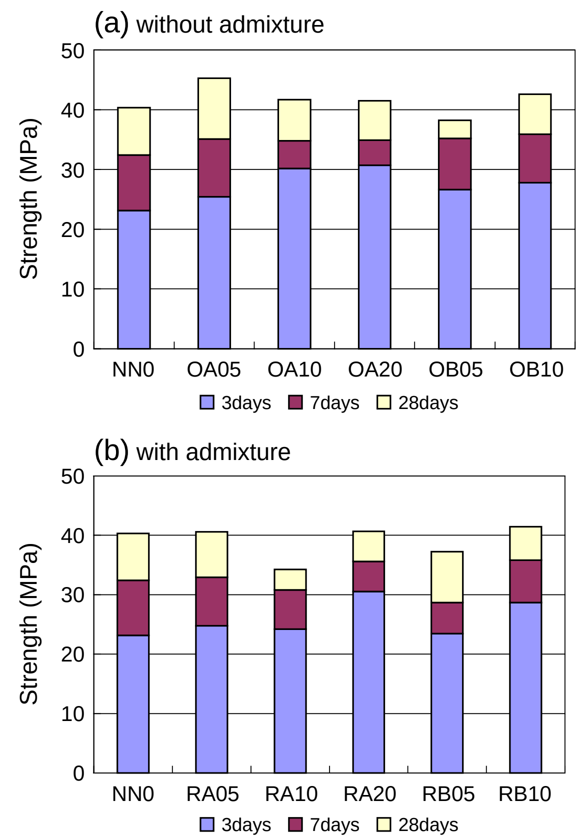

Fig. 5. Compressive strength variance with mixing of OS; (a) without admixture, (b) with admixture.

3.3. Characteristics of hardened concrete substituted with OS

In order to evaluate the mechanical properties of concrete substituted with OS, compressive strength and splitting tensile strength of concrete at various ages was compared. Also, the elastic modulus of concrete was examined in order to investigate the effect of substitution of OS on rigidity of concrete. The test results are illustrated in Figs. 5–10.

3.3.1. Characteristics of compressive strength

Fig. 5(a) shows the compressive strength of concrete developed with age and type and SR of OS. According to

Table 5
Setting time variance with SR of OS

SR (%)	AE WRA (%)	Setting time (h:min)					
		Type A			Type B		
		Initial setting	Final setting	Placing temperature (°C)	Initial setting	Final setting	Placing temperature (°C)
0	0.0	5:20	7:10	21.3	5:20	7:10	21.3
5		6:30	8:50	18.6	5:30	7:20	21.1
10		5:20	7:30	21.2	5:30	7:30	21.9
20		4:40	7:00	21.0	–	–	–
5	0.3	7:10	9:40	18.3	8:00	10:40	17.5
10		6:20	8:20	21.7	7:20	10:10	16.7
20		5:20	7:20	21.3	–	–	–

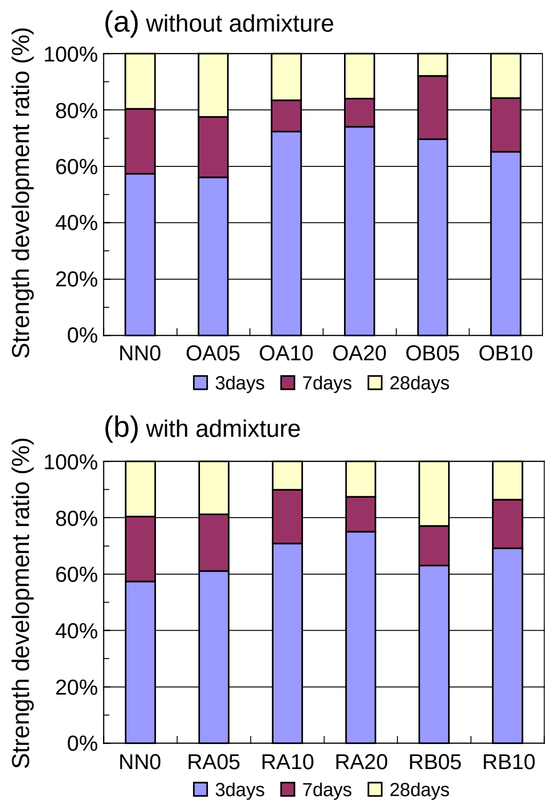

Fig. 6. Compressive strength development ratio variance with mixing of OS.

Fig. 5(a), compressive strength at an early age of concrete substituted with OS showed a higher value than NN0 mixture that does not include OS. Compressive strength at age 28 days was also higher except OB05 mixture blended with 5% OS of type B. More specifically, compressive strength at an early age demonstrates an increasing tendency as SR of OS increases. Such a tendency was gradually eliminated with age increase. However, the difference with type and SR of OS was minor. Finally, compressive strength gradually approached the strength of NN0 mixture. This is because OS mixed was fully dehydrated for pulverization. OS absorbs surrounding free water and, as a result, the relative w/c ratio decreases. Accordingly, compressive strength at an early age increases as SR of OS increases.

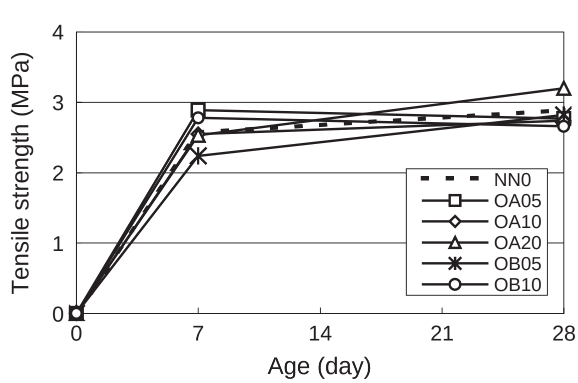

Fig. 7. Tensile strength variance with mixing of OS.

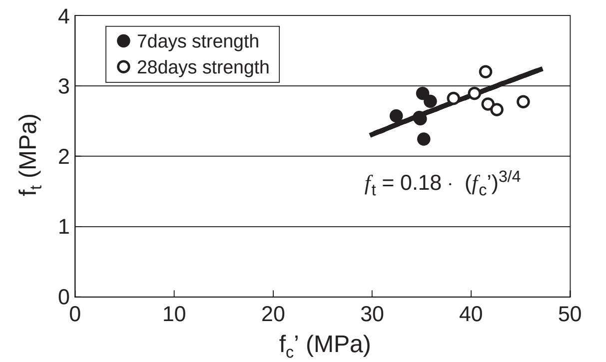

Fig. 8. Relationship between tensile strength and compressive strength of concrete mixed with OS.

When OS is blended, however, the compressive strength with increasing age comes close to that of normal mixture concrete. Despite of the relative w/c decreasing, this is because OS that exists within hardened concrete can result in a defect region as concrete strength increases. Among these test results, the influence of stress concentration can be seen after investigating characteristics of compressive strength up to 1 year in concrete substituted with OS. This is presented in detail in the Part II paper.

Fig. 5(b) presents compressive strength development of concrete with AE WRA added for consistency. This figure shows a mixture (RA10) with a reduced strength at an early age with SR of OS. In addition, it says that the strength with increasing age was also lower compared to that of a normal mixture. The reason is due to the air entraining effect of AE WRA including the effect of relative w/c ratio by OS. That is, in the cases of RA05, RA10, and RB05 mixtures which entraining voids increased by adding of AE WRA, the development of compressive strength is lower than OA05, OA10, and OB05 mixtures, respectively, by 10.4%, 17.9%, and 2.6%. The difference in OB05 mixture, compared to other mixtures (OA05 and OA10), is small since the strength obtained at age 28 days is unexpectedly small. If it is reevaluated based on the strengths at age 3 and 7 days, the reduction is 12.1% and 18.7%, respectively. These results are related to existing research results for normal concrete (i.e., approximately 5% compressive strength reduction with 1% air content increase) [11]. Therefore, in

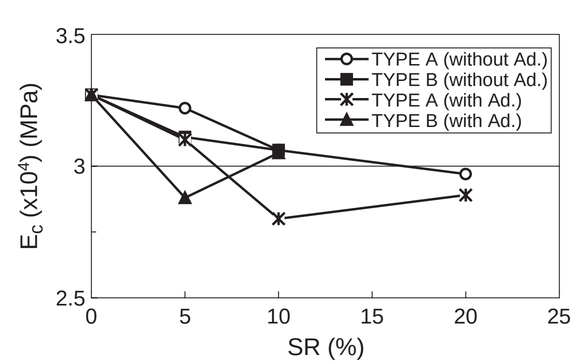

Fig. 9. Variance of concrete elastic modulus with mixing of OS.

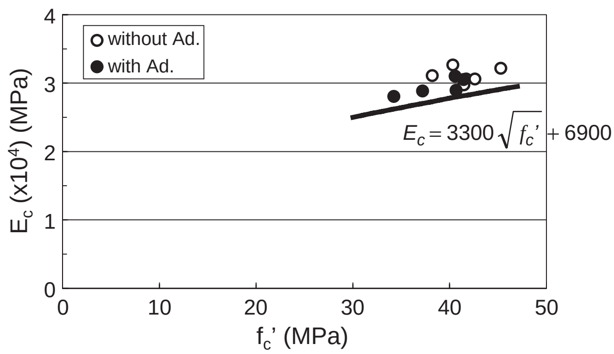

Fig. 10. Relationship between elastic modulus and compressive strength of concrete mixed with OS.

results until now, it is noted that concrete compressive strength at age 28 days, with blending of OS, is not decreased compared to the value of normal mixture.

In addition, to investigate the effect of SR of OS on the rate of development of compressive strength at an early age, compressive strength was normalized based on the strength at 28 days, and the results are shown in Fig. 6(a) and Fig. 6(b). In the case of NN0 mixture, high initial strength development, which is one of inherent characteristics of high-strength concrete, occurred [13,14]. That is, the strength at age 3 days exceeded 50% of the strength at 28 days and the strength at age 7 days was approximately 80% of the strength at 28 days.

Meanwhile, the rate of development of compressive strength at an early age in concrete substituted with OS varies with SR of OS. However, the strength at age 3 days is approximately 70% of the strength at age 28 days and the strength at age 7 days exceeds 80% of the strength at age 28 days. The strength development at an early age increases significantly as SR of OS. However, the increasing rate of the development did not change proportionally to SR. And, the difference with type (i.e., influence of F.M. of OS) of OS was not apparent within the scope of this study.

Therefore, if we want to utilize the relationship of strength at an early age and long-term age strength of concrete substituted with OS, it is necessary to study characteristics of strength development with SR of OS. When the strength development of normal concrete is assumed, we can secure a safety margin in determining the removal time of forms since the strength at an early age is underestimated. However, when we forecast long-term strength of concrete based on the strength at an early age, sufficient consideration should be done. From Fig. 6(b) it is noted that a difference in the absolute value of compressive strength is present, but, the results show a similar tendency to the cases without admixture.

3.3.2. Characteristics of splitting tensile strength

In order to examine the variance of splitting tensile strength with SR of OS, the development of tensile strength with mixture is shown in Fig. 7. Generally speaking, the tensile strength development of high-strength concrete at an early age is high and the development after age 7 days is insignificant. In this test, the change in strength after age 7 days is not distinct, but, the deviation with mixture was apparent. When comparing the splitting tensile strengths with mixture, the strong tendency found in the case of compressive strength was not present. Generally speaking, however, the tensile strength at an early age increased with SR increase. The tensile strength with SR at age 28 days decreased a little compared with the tensile strength of the standard mixture. This is because the influence of *w*/*c* ratio variance at early ages and the stress concentration occurring in the OS portion at old ages. More specifically, the stress concentration phenomenon happens more distinctly in the tensile stress state. Thus, the above phenomena should be studied more detail.

In general, after determining the relationship between compressive strength and tensile strength, it is possible to evaluate the tensile strength. It can be calculated by inputting measured compressive strength into the relationship [15,16]. Therefore, to compare relationship between compressive strength and tensile strength of concrete with substitution of OS, regression analyses on measured values of compressive strength and tensile strength at age 7 days and age 28 days were carried out and the results are graphed and shown in Fig. 8. As shown in Fig. 8, regression analyses were performed based on the form of Eq. (2) generally adopted in normal concrete.

$$f_t = A(f_c')^{3/4}. \qquad (2)$$

Since the number of data measured in this study is not large, verification of results of regression analyses should be performed. In addition, it is acknowledged that accumulation of additional experimental results and the relative comparison with normal concrete are also advisable.

3.3.3. Characteristics of elastic modulus

To evaluate the effect of substitution of OS on elastic modulus of concrete, compressive elastic modulus at age 28 days was measured. The results are shown in Fig. 9. Fig. 9 shows that elastic modulus is affected by type of OS, SR of OS, and usage of AE WRA. Concrete elastic modulus decreases with SR of OS increase since elastic modulus of the OS is smaller than the elastic modulus of fine aggregate. The elastic modulus was reduced approximately 10% when OS of 20% is substituted for fine aggregate. When AE WRA was added in concrete, the decrease phenomenon was significant not only for compressive strength but also for elastic modulus due to the increase of entrained air content.

Fig. 10 shows the relationship between compressive strength and elastic modulus of concrete substituted with OS. The formula proposed in concrete structure design standard [14] for compressive strength-elastic modulus is also presented. As shown in Fig. 10, the measured elastic modulus showed higher values than the proposed formula regardless of admixture usage or not. That is, the value (i.e., elastic modulus obtained using the compressive strength) suggested by ACI Code is more conservative than experimental results.

4. Conclusions

In this study, the effect of SR of OS on the mechanical properties of concrete was examined. This study was based on equal w/c and direct partial replacement of fine aggregate with OS. The conclusions obtained from this study can be summarized as follows:

(1) When the crushed OS is substituted in concrete mixture, workability of concrete decreases with F.M. decrease and with SR increase.
(2) The setting time is not affected by substitution of OS up to a maximum of 20% of fine aggregate weight.
(3) Compressive strength at age 28 days of concrete substituted with OS is equal to or exceeded compressive strength of normal concrete. It is shown that OS does not cause a decrease in early age strength of concrete.
(4) Elastic modulus of concrete substituted with OS decreased as SR of OS increases since elastic modulus of OS is smaller than that of fine aggregate. And, elastic modulus is reduced by approximately 10% when fine aggregate is substituted with 20% OS.

Notation

A_d	admixture
C	cement
d_a	maximum aggregate size
E_c	chord modulus of elasticity
f_c'	compressive strength of concrete
f_t	tensile strength of concrete
G	coarse aggregate
G_{max}	maximum aggregate size
S	fine aggregate
s/a	fine aggregate/(fine aggregate+coarse aggregate)
t	time
W	water
w/c	water–cement ratio of the concrete

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