Effect on Slump Flow of High-Strength Concrete caused by Flock Formation during Dry-Mixing

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Abstract: Construction using high-strength concrete is anticipated to increase, and the manufacturing technology of such concrete needs to adapt to this rise. High-strength concrete exhibits workability that can significantly vary depending on the mixing conditions even under the same mix proportion and environment. This research specifically focuses on the change in workability arising from dry-mixing (i.e., mixing of fine aggregate with cement in the early stage) in the mixing process of a high-strength concrete mix using a revolving-double paddle mixer. The slump flow values were confirmed to vary depending on dry-mixing. Furthermore, Cryo-SEM image analysis revealed that dry-mixing causes flocculation of cement particles, followed by analysis of the adsorption amount of admixtures. We concluded that the flocculation affects the adsorption amount and adsorption timing of admixtures in the later stages of the mixing process, leading to differences in concrete workability.

Keywords: High-strength concrete, Revolving-double paddle mixer, Dry-mixing, Flocculation, Cryo-SEM, Adsorption of admixtures

1. Introduction

Currently, we have social issues to address in Japan, such as "how to realize a low-carbon society as a countermeasure against global warming," "how to promote work efficiency against population decline," and "how to efficiently pass on our technologies to the next generation in this aging society."

In recent years, natural disasters have become more severe and more frequent. In response, a fundamental law was enacted for national resilience, specifically for disaster prevention, disaster mitigation, and countermeasures against aging infrastructure, such as constructing highly strengthened buildings. Accordingly, the use of high-strength concrete in construction is expected to increase in the future.

Additionally, we have another issue of overall population decline and lack of workers in the construction industry in Japan. Such an issue is likely to lead to reductions in construction time and greater usage of precast concrete products. This usage is anticipated to accelerate, as evidenced by the revision of the JIS A 5308 (2019) which was clearly aimed at promoting the use of high-strength concrete.

In light of this context, the construction of high-strength concrete is expected to further rise. Consequently, manufacturing at batcher plants, as well as construction technologies, need to adapt to the rise.

In concrete manufacturing, mixing conditions are known to affect the workability of concret[1).

Table 1 Mix Proportion of High-Intensity Concrete "80-60-20L"

Maximum Size of Coarse Aggregate	Slump Flow	Water Cement Ratio	Air Content	Fine Aggregate Content	Unit Volume (kg/m^3)				
					Water *W*	Cement *C*	Fine Aggregate *S*	Coarse Aggregate *G*	Admixtures *Pc*
(mm)	(cm)	(%)	(%)	(%)					
20	60.0	21.4	2.0	44.4	175	818	650	815	8.18

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Adsorption of admixtures to cement has been listed as one of the causes[2)]. The absorption influence is more pronounced in high-strength concrete which contains a larger amount of cement compared to standard concrete. However, there have been few attempts to explore the extent and mechanism of the influence on the mix proportion for high-strength concrete. Hence, there is an urgent need to investigate the differences in concrete workability depending on mixing conditions. We believe that this investigation will contribute to the stable supply of high-strength concrete in the future.

Therefore, in this research, we focused on the presence/absence of mixing of fine aggregate with cement (hereinafter referred to as dry-mixing) and the mixing time in the initial stage of mixing, so as to figure out the influence of dry-mixing on the workability of high-strength concrete. We have previously issued a technical paper[3)] about the influence and mechanism of dry-mixing on mortar in a Hobart mixer (5L). This time, however, a revolving-double paddle mixer was used to conduct multiple experiences in a manner closer to actual practice. Specifically, we concentrated on the mortar flow values, the slump flow values of concrete, the surface moisture on fine aggregate, and admixtures, under different dry-mixing conditions, where the surface moisture and admixtures are the important indicators in existing studies.

Based on the results, physical considerations using Cryo-SEM and chemical considerations on the adsorption of admixtures were made to investigate the causes that differentiate the workability of concrete.

2. Influence of Mixing on Flow Values

2.1 Experiment Overview

This section provides an overview common to all the experiments described in this paper. **Table 1** shows a mix proportion of high-strength concrete with a design strength of 80 N/㎟, which was used as the subject of the experiments. In this research, we calculated the required amount of each material for 36L concrete, the volume the mixer used is capable of handling, from the mix proportion and conducted the tests described below. **Table 2** shows the materials used.

Admixtures were added relative to the unit amount of cement, with the quantity obtained by adjusting the mix proportion based on the results of preliminary tests conducted on the concrete mix on the day of each test. Two types of revolving-double paddle mixers (60L) (hereinafter referred to as mixer A and mixer B) were used. They have different blade heights designed for different movements of materials. The tests were conducted indoors under a constant environment at a temperature of 20 ± 2℃ and at a humidity of 50% or higher.

2.2 Influence of Presence/Absence of mixing and its Time on Flow Values

(1) Overview

Figure 1 illustrates the mixing procedure. Based on the power data of the mixers obtained in the

Table 2 Materials Used

Materials	Type and Quality
Cement *C*	Low-heat Portland Cement Density: 3.23 g/㎤ Specific Surface Area: 3130 ㎠/g
Fine Aggregate *S*	Crushed Sand Rock Type: Andesite Density in Saturated Surface-dry Condition: 2.54 g/㎤
Coarse Aggregate *G*	Crushed Stone Rock Type: Andesite Density in Saturated Surface-dry Condition: 2.67 g/㎤
Admixtures *Pc*	High Range Water Reducing Admixture Main Component: Polycarboxylic Acid Compound

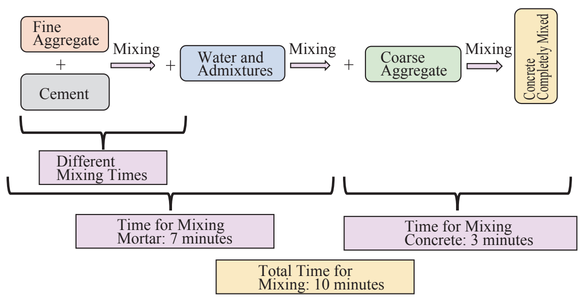

Figure 1 Mixing Procedure

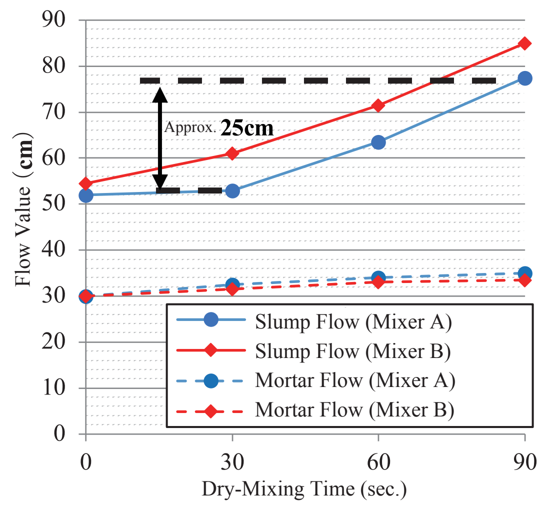

Figure 2 Flows with Dry-Mixing

preliminary experiments, mixing was carried out for a total of 10 minutes. This included 7 minutes of mortar mixing, which involved dry-mixing time, and 3 minutes of concrete mixing with coarse aggregate added.

To confirm the influence of the surface moisture on fine aggregate and cement onto the initial adsorption of admixtures, dry-mixing of the materials was performed. Subsequently, a certain amount of mixing water and admixtures were added to the materials, resulting in the preparation of mortar. The amount of the added mixing water was set without containing the surface moisture on the fine and coarse aggregate. Note that the surface moisture ratio of the fine aggregate in a wet state was regulated at 3%.

To confirm the influence of presence/absence of mixing and its time on the flow values, the mortar mixing time was fixed at 7 minutes in total, and the dry-mixing time with each mixer was set in four patterns: 0, 30, 60, and 90 seconds. The mixing time of the mortar after adding the mixing water was changed according to the dry-mixing times. To the resulting mortar, coarse aggregate was introduced to produce fresh concrete. The mortar flow and the slump flow were compared by sampling an amount of the mortar and fresh concrete at the set timings after mixing, the sample amount being sufficient for measuring.

(2) Experimental Results and Discussion

Figure 2 shows the mortar flow values and the slump flow values for each of the dry-mixing times. In this context, the slump flow value with a dry-mixing time of 30 seconds means the flow of the mortar made with a dry-mixing time of 30 seconds at which coarse aggregate was added.

In both cases with mixers A and B, as the dry-mixing time increased, the mortar flows and the slump flows increased.

Concerning the mortar flow, there was an approximate 3 cm difference in mortar flow values between 30 seconds and 90 seconds of dry-mixing in the mortar made using the revolving-double paddle test mixers. In contrast, the difference in mortar flow values reported previously[3] using Hobart mixer was approximately 10 cm. Accordingly, we confirmed the presence of a difference in the mortar flow values between the Hobart mixer and the revolving-double paddle test mixers.

In terms of slump flow, the flows increased with an increase in the dry-mixing time, and the difference in slump flow values in each mixer exceeded 20 cm even though the mix proportion was the same. The value difference is larger than the ±10cm specified in JIS A 5308 for a concrete slump flow of 60 cm.

The factor contributing to the difference in slump flow values is believed to exist in the dry-mixing rather than the mixing after the addition of coarse aggregate. We hold this belief because (1) as previously reported[3], dry-mixing significantly affects the mortar flow, and (2) in both cases with mixers A and B, the difference in slump flow values and the rate of increase in flow along with the increase in dry-mixing time

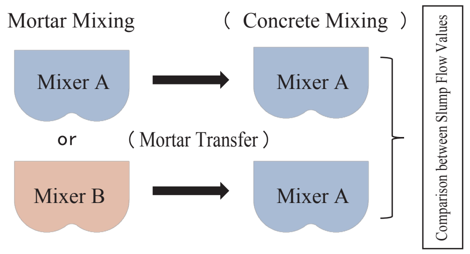

Figure 3　Procedure in Mortar Transfer Test

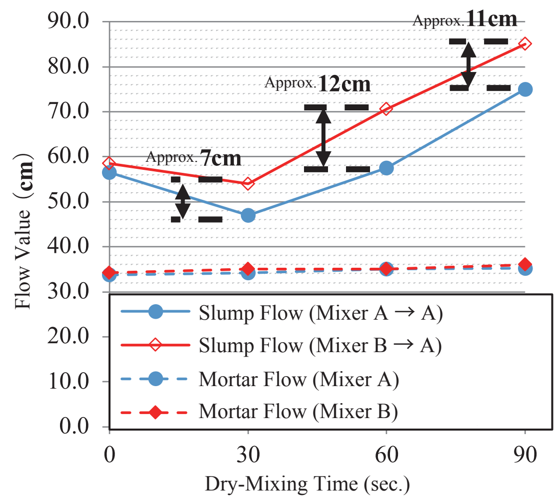

Figure 4　Flows in Mortar Transfer Test

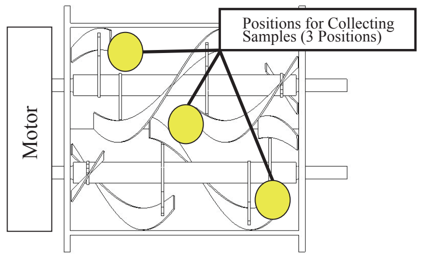

Figure 5　Positions for Sampling

were approximately the same.

However, the difference in mortar flow values in the case with the revolving-double paddle text mixers was 3 cm, which is smaller than that previously reported[3]. Hence, we hypothesized that the features of the mortar contributing to the workability of concrete exist in the properties that cannot be adequately represented by the flow values, and we decided to verify the hypothesis in the experiments which will be described below.

2.3 Influence of Variations in Mortar Properties on Slump Flow

(1) Overview

To clarify the existence of variations in mortar properties that cannot be adequately represented by differences in mortar flow values, as inferred from the research in the previous section, we performed the mixings as shown in **Figure 3**. Mortar was mixed in mixers A and B, and the mixed mortar in mixer B was transferred into mixer A, maintaining the mixing mechanism after the introduction of coarse aggregate. The slump flow values of the transferred and mixed concrete were then evaluated.

Four patterns of dry-mixing time were used: 0, 30, 60, and 90 seconds. Even in the case of mixing in the same mixier consistently, the mortar was discharged once and then reintroduced into the same mixer to maintain the same conditions as the case where the mortar was transferred. By controlling the time required for transferring the mortar to be constant, the experiment was conducted. Other test conditions remained the same as those in the previous section.

(2) Experimental Results and Considerations

Figure 4 shows the flow value results for each dry-mixing time in the mortar transfer test. As in the previous section, it was confirmed that the mortar flow does not vary depending on the mixer. However, when the mortar of the same flow value was mixed by a different mixer and transferred to mixer A to make concrete, the resulting slump flow values differed from those of the mortar consistently mixed by the same mixer.

The results indicate that variations in mortar properties, which cannot be adequately represented by mortar flow values, emerge during the dry-mixing. Furthermore, the variations in mortar properties are suggested to be the cause of the difference in slump flow values arising from the dry-mixing.

To confirm the variations in mortar properties, the following factors, believed to arise from dry-mixing, were investigated in the next chapter: the migration of surface moisture from fine aggregate to cement particles, the states of flocculation, and the differences

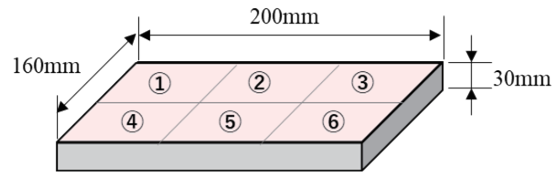

Figure 6　Positions for Color Absorbance Measurement

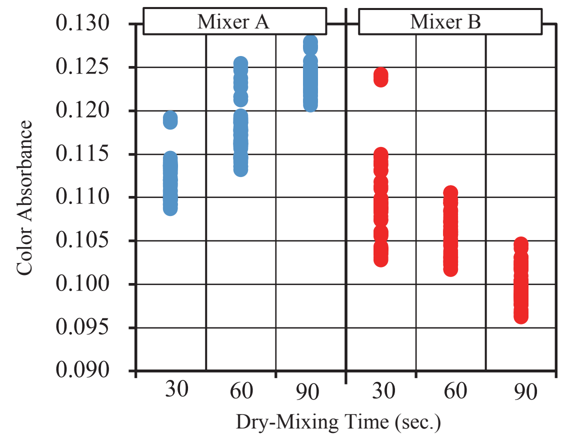

Figure 7　Color Absorbance according to Dry-Mixing Time

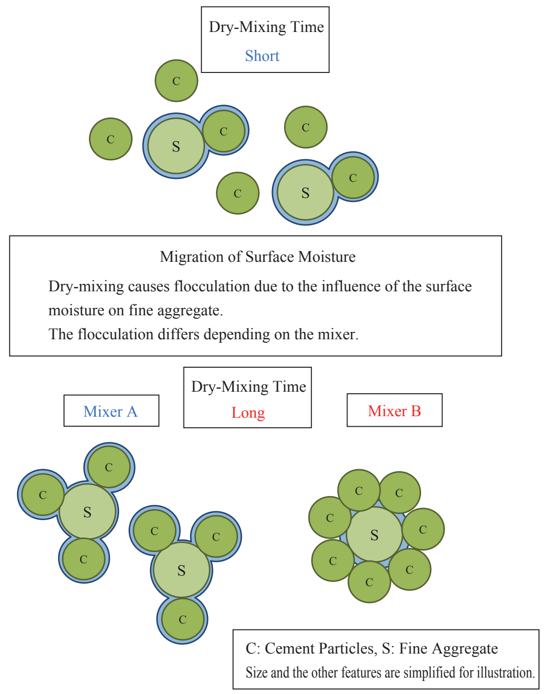

Figure 8　Mechanism of Flocculation with Dry-Mixing

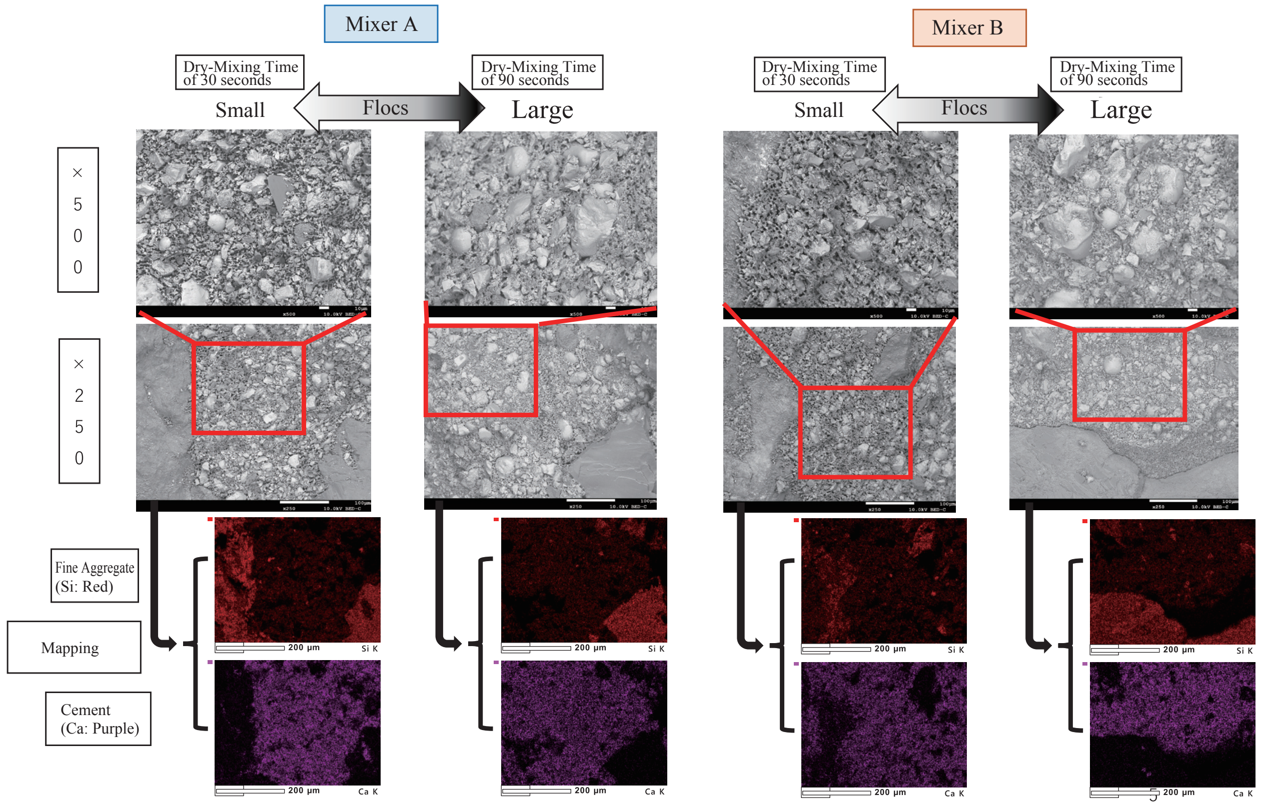

Figure 9　SEM Images and Mapping Images of Mortar at Different Dry-Mixing Times

in admixture adsorption.

3. Influence of Dry-mixing on Concrete Workability

In Chapter 2, we confirmed that the slump flow is influenced by the variations in mortar properties resulting from dry-mixing, which cannot be adequately represented by mortar flow. In this chapter, we experimented, as a physical test, to verify the influence of surface moisture on fine aggregate onto cement particles and the resulting state of flocculation induced by the surface moisture. In addition, as a chemical test, we conducted a measurement of the adsorption of admixtures that significantly affects the workability of concrete.

3.1 Experiments on Surface Moisture Migration on Fine Aggregate

(1) Overview

A test was conducted using an infrared multi analyzer to quantify the adsorption and migration of surface moisture on fine aggregate to cement particles according to dry-mixing time. The materials used in the experiment included calcium carbonate (density: 2.71 g/㎤) which is a white powder as a substitute for cement, and fine aggregate. Calcium carbonate does not react with water and the use of calcium carbonate was considered to allow the evaluation of pure moisture adsorption to the powder and fine aggregate.

Table 3　Specific Surface Area of Flocs

	Specific Surface Area (1/m)	
	Dry-Mixing 30 sec.	Dry-Mixing 90 sec.
Mixer A	24.66	19.44
Mixer B	21.78	18.67

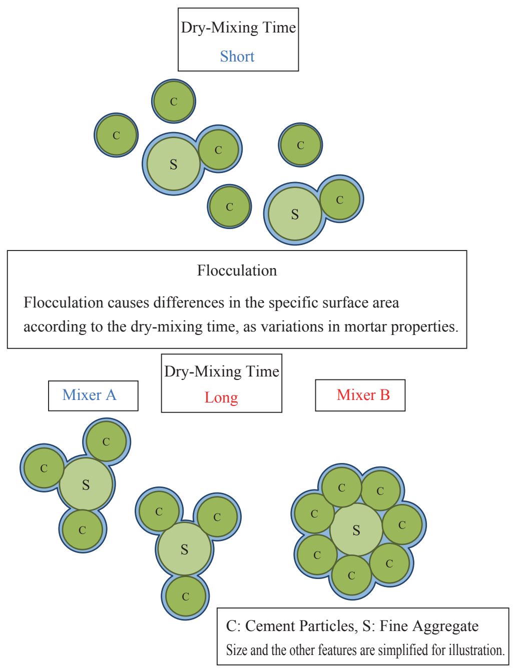

Figure 10　Mechanism of Differences in Specific Surface Area with Dry-Mixing

Moreover, calcium carbonate has a specific surface area of 3000 ㎠/g equivalent to that of cement. Hence, we determined that calcium carbonate could be used for evaluating the moisture migration without adverse effects.

The infrared multi analyzer was set to measure color wavelengths, and the adsorption was measured from the wavelengths at the material surfaces after dry-mixing. Note that any differences in wavelength are unlikely to occur between the surface moisture on fine aggregate and the original color of calcium carbonate. To clarify the difference in color wavelength between the fine aggregate and the calcium carbonate, we caused the fine aggregate in a dry state to absorb water colored with red food coloring, giving it a surface moisture of 3%.

The required amount of each material per 36 liters was calculated from the standard mix proportion shown in **Table 1**. The measurement was conducted for three patterns of dry-mixing time of 30, 60, and 90 seconds, focusing on the dry-mixing before main mixing. As shown in **Figure 5**, samples were collected from three locations: positions diagonal to the mixer plane and at the center of the mixer. The material immediately after dry-mixing was transferred to a stainless-steel vat and flattened. The material was then divided into six sections as shown in **Figure 6**. The surface color wavelength at the center of each section was measured three times, for a total of 54 measurements per batch.

(2) Experimental Results and Considerations

Figure 7 shows the color absorbance results measured for each dry-mixing time for each mixer.

For each absorbance, a larger value indicates more colored existing areas. In the case with mixer A, as the dry-mixing time increased, the absorbance increased, and more calcium carbonate was colored. On the other hand, in the case with mixer B, no increase in the number of colored areas of calcium carbonate was observed with an increase in the dry-mixing time; rather, the calcium carbonate covered the colored surface moisture. These different mixers exhibit distinct tendencies in changing color absorbance, suggesting differences in the flocculation process.

Figure 8 is a schematic diagram of the flocculation process we assumed based on the absorbance results.

In the case with mixer A, an increase in the dry-mixing time led to the migration of surface moisture from the fine aggregate to the cement, resulting in flocculation. In contrast, with mixer B, as the dry-mixing time increases, the cement particles adhered to the surface of the fine aggregate that retained the surface moisture, leading to flocculation. In both mixers with different flocculation processes, visual inspection confirmed that as the dry-mixing time increased, the number of flocs with excessively large lumps increased.

Furthermore, as the dry-mixing time increased, the variation in measurements at each of the measurement positions decreased, indicating that the flocs were uniformly formed.

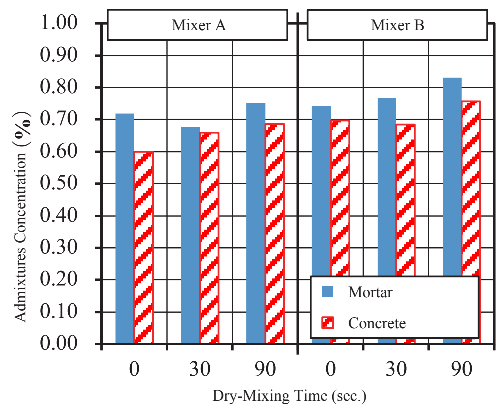

Figure 11　Residual Amount of Admixtures according to Dry-Mixing Time

3.2 Experiment on Formation of Cement Flocs

(1) Overview

When cement hydrates, a positive charge is generated on the surface of cement particles due to the hydration reaction, making the particles unstable and causing them to stick together, forming flocs. In this paper, Cryo-SEM was employed to quantify the amount of flocculation, aiming to confirm the influence of surface moisture migrating from fine aggregate to cement particles according to dry-mixing time.

In the measurement process, specimens of the mortar prepared by dry-mixing were flash-frozen with liquid nitrogen immediately after mixing. Subsequently, the specimens were cut, and the cut surfaces of the specimens were observed at 250x and 500x magnification by Cryo-SEM, which can observe the specimens in the frozen state. Concurrently, Si, the main component of fine aggregate, and Ca, the main component of cement, were mapped on the 250x SEM image to distinguish between the fine aggregate and the cement.

Dry-mixing by each mixer was conducted for two patterns of 30 and 90 seconds. The SEM images at 250x magnification for the two patterns were analyzed to calculate the specific surface areas, respectively. To ensure that the flocs were larger than the average grain size of cement, particles with a diameter of 15 μm or greater were observed as flocs.

(2)Experimental Results and Considerations

Figure 9 displays the SEM images and mapping images of the mortar relative to the dry-mixing times for each mixer. **Table 3** presents the results of the specific surface areas of the flocs calculated from these images.

As expected from the experimental results of surface moisture migration in the previous section, we confirmed that, with an increase in the dry-mixing time, the formed flocs become larger due to the influence of surface moisture on the fine aggregate, resulting in the differences in the specific surface areas of cement particles.

In addition, the sizes of the flocs were able to be visually confirmed in the SEM image at 500x magnification.

Figure 10 is a schematic diagram of the flocs in the mortar based on the results of specific surface area differences.

3.3 Experiments on Adsorption of Admixtures

(1) Overview

Sections 3.1 and 3.2 have revealed that cement particles form flocs due to the influence of surface moisture on fine aggregate. In this section, we measured the amount of admixtures adsorbed according to dry-mixing time, aiming to investigate how flocculation during the dry-mixing stage affects adsorption of admixtures. Mortar and fresh concrete were prepared with the required amount of each material per 36 liters calculated from the standard mix proportion shown in **Table 1**.

The remaining amount of admixtures in the liquid phase was measured by separating the liquid-phase components and the solid-phase components using a centrifuge and analyzing the extracted liquid-phase components thermally.

The measurements were conducted for three patterns of dry-mixing time: 0, 30, and 90 seconds for each mixer. The liquid-phase components separated from the prepared mortar and those of the mortar from the prepared fresh concrete were sampled and measured six times each.

(2) Experimental Results and Considerations

Figure 11 shows the measurement results of the residual amount of admixtures in the prepared mortar and fresh concrete for each dry-mixing time with each mixer. The results of each residual amount showed that, with both mixers, the amount of admixtures remaining in the liquid phase tended to increase as the dry-mixing time increased. This suggests that with a shorter dry-mixing time, more admixtures were adsorbed to the solid-phase component, and with a longer dry-mixing time, a smaller amount of admixtures were adsorbed. This surplus of admixtures can be considered to improve the workability of the concrete after the addition of admixtures, as observed practically with post-addition of admixtures.

Figure 12 is a schematical diagram showing the relationship between flocs and admixtures we assumed based on the measurement results of the amount of the admixtures adsorbed.

3.4 Investigation of Influence of Dry-Mixing on Workability of Concrete

The above test results indicate that dry-mixing causes the surface moisture on fine aggregate to come in contact with cement particles, resulting in flocculation of cement particles. The specific surface area of cement particles becomes smaller, leading to a decrease in the amount of admixtures adsorbed. We believe that, with more flocculation at the initial state of mixing, a lesser amount of water comes into contact with the cement particles during the main mixing where the water is introduced, resulting in less admixture adsorption in advance.

These results reveal that the presence/absence of dry-mixing and its time affect the mortar properties. Specifically, flocculation caused by the surface moisture of fine aggregate and cement particles influences the amount of admixture adsorption and the flow values immediately after mixing. Therefore, controlling flocculation can be assumed to allow control of the slump flow value immediately after mixing.

In our view, it is not important to lengthen the dry-mixing time; rather, it is crucial to prepare the cement particles in a state suitable for the adsorption of admixtures to be added in the main mixing process.

In addition, the results of the surface moisture migration in Section 3.1 have clarified that the flocculation varies depending on the mixer used. Based on these results, we believe that the performance of the mixer has an influence on the appropriate conditions for flocculation.

4. Summary

The findings obtained in this research are summarized below.

(1) In the revolving-double paddle mixer, variations in mortar properties, such as the amount of flocculation, which cannot be adequately represented by the flow value, significantly contribute to the subsequent slump flow value of concrete.

(2) Dry-mixing was confirmed to affect the properties of mortar even under the same mix proportion and environment. This is because, prior to the addition of admixtures, as the contact time between the surface moisture on fine aggregate and cement increases, flocculation of cement particles occurs. This process reduces the specific surface area of the cement particles and diminishes the amount of admixtures adsorbed to the cement particles. The surplus admixtures contribute to the increase in the slump flow value of fresh concrete prepared by the subsequent mixing.

(3) The flocculation process during dry-mixing varies depending on how the material moves in the mixing mechanism.

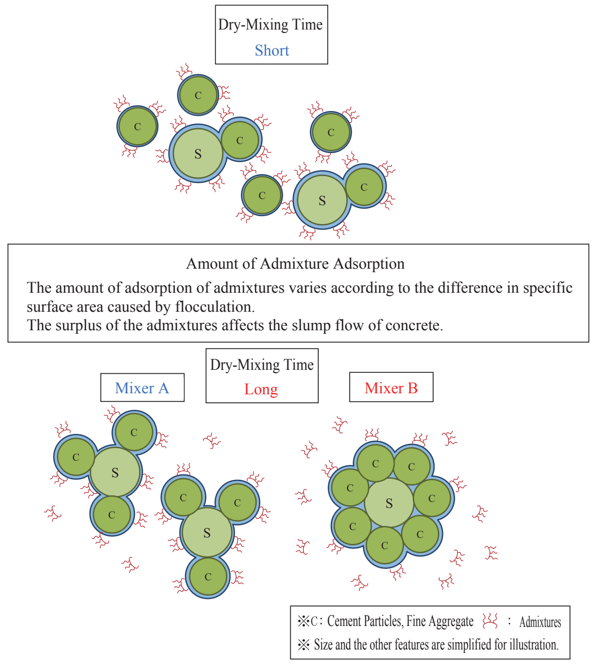

Figure 12 Mechanism of Difference in Amount of Admixture Adsorption with Dry-Mixing

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