# ESTIMATING GYPSUM HEMIHYDRATE CONTENT USING DENSITY OF GYPSUM RECYCLED FROM WASTE GYPSUM BOARD

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**ABSTRACT:** Studies on recycled gypsum as a geotechnical improvement material have been extensively conducted, as the increase in gypsum board waste has become an urgent issue. In this paper, to investigate a method to measure gypsum hemihydrate content in a shorter period, the test results of the density method obtained in previous studies are reviewed to confirm whether sufficient accuracy could be secured even in a shortened measurement period. The results obtained showed that the way of the density test for cement is effective in measuring the density of gypsum, gypsum hemihydrate content could be estimated from the density with some accuracy, even with an elapsed period of 1 d, and a difference of only approximately 5% was observed. In addition, the density method was validated through a full-scale experiment using a rotary kiln furnace. The gypsum hemihydrate content obtained from the density method was consistent with that obtained from the X-ray diffraction analysis.

Keywords: Recycled Gypsum, Gypsum Dihydrate, Gypsum Hemihydrate, Gypsum Hemihydrate Content, Density

# 1. INTRODUCTION

As the amount of waste from gypsum boards has been increasing, the development of a preventive system has become an urgent issue, and studies on the applicability of waste gypsum boards as a geotechnical improvement material have been actively conducted [1-5]. Gypsum powder made by crushing, grinding, and separating waste gypsum boards needs to be heated to convert gypsum dihydrate (GD) to gypsum hemihydrate (GH), which is mostly used for geotechnical improvement.

In this study, the recycled gypsum obtained from waste gypsum boards is referred to as recycled gypsum dihydrate (RGD), and GH and gypsum anhydrate (GA) produced by heating RGD are referred to as recycled gypsum hemihydrate (RGH) and recycled gypsum anhydrate, respectively. The GH used in this study was type  $\beta$  GH, and the GA was type III anhydrate gypsum [6]. The gypsum types are abbreviated as GH or GA in the remainder of the paper.

Regarding the heating temperature for converting RGD to GH or GA, several studies [2,3,5] have reported that GD is converted to GH at 130–150 °C and then to GA at 200 °C. However, the authors experimentally investigated the thermal behavior of recycled gypsum and commercially available gypsum reagent in detail. A detailed

experimental investigation of the thermal behavior of recycled gypsum and commercially available reagent gypsum showed that GD (CaSO<sub>4</sub>·2H<sub>2</sub>O) is converted to GH (CaSO<sub>4</sub>·1/2H<sub>2</sub>O) by heating and dehydration at 90 °C for 24 h, and GH is converted to GA (CaSO<sub>4</sub>) by heating and dehydration at 120 °C for 24 h [7-9]. Thus, not only the temperature at the time of heating, but also the combination of temperature and time influences the morphological change of gypsum.

The authors believe that a quality control method should be established to promote the effective use of recycled gypsum in civil engineering and other fields. They have studied a method for estimating the gypsum hemihydrate content,  $C_{GH}$ , and gypsum dihydrate content,  $C_{GD}$  (=100- $C_{GH}$ ), using the "density method" [7,8] and "heating method" [9] to identify so-called "raw burnt gypsum" more efficiently. This is due to the fact that, at some sites where recycled gypsum from waste gypsum boards is used, we have received comments, such as "the quality of the delivered GH is not consistent" and even "it is doubtful whether it can be called GH," because of incompletely processed products.

Although the engineering effectiveness of recycled gypsum from waste gypsum boards has been reported, if low-quality products appear in the market, the credibility of research on the effective use of recycled gypsum from waste gypsum boards would be severely damaged. This would be a major obstacle in an era where the amount of waste gypsum boards is expected to increase, and the remaining capacity of controlled final disposal sites is expected to decrease. Therefore, to avoid such a crisis, we must actively focus on the development of quality control technology and a technology for the effective use of recycled gypsum.

As mentioned earlier, the authors investigated the basic properties of recycled gypsum in detail. In this process, they focused on the density of RGD and RGH, and proposed a "density method" that can estimate  $C_{GH}$  effectively [7,8]. However, the "density method" requires time to remove air bubbles and takes a relatively long time (4–7 d) to yield stable measurements for the density of commercially available reagent gypsum and recycled gypsum. This drawback led us to propose an alternative "heating method" [9]. The "heating method" can determine  $C_{GH}$  through furnace drying at 70 °C and 90 °C for 24 h each, for a total of 48 h, which is much shorter than the time taken by the "density method".

In this study, we reviewed the test results of the "density method" obtained in previous studies and examined the accuracy of  $C_{GH}$  for a short measurement period.

# 2. ESTIMATION EQUATION FOR GYPSUM HEMIHYDRATE CONTENT USING DENSITY

In the following paragraphs, we describe the construction of an equation to estimate the GD and GH contents from the density values. We assumed that only GD and GH existed in the gypsum for the following reasons. First, the produced gypsum may contain Type III gypsum anhydrite, but not Type I or Type II, considering the temperature at which the recycled gypsum is heated. Type III gypsum anhydrite absorbs water from the moisture in the air and instantaneously reverts to GH [6]. Thus, it was assumed that no GA existed in the target gypsum.

To construct the estimation equation, we first denote the density of GD as  $\rho_{GD}$  and the density of GH as  $\rho_{GH}$ . When GD and GH were mixed, the mass of the mixture was  $(m_{GD} + m_{GH})$  and the volume was  $(V_{GD} + V_{GH})$ . The density of gypsum  $\rho_{(GD+GH)}$  is expressed by Eq. (1).

$$\rho_{(GD+GH)} = \frac{m_{GD} + m_{GH}}{V_{GD} + V_{GH}} \tag{1}$$

where *m* is the mass of gypsum, *V* is the volume of gypsum, the subscripts indicate the form of gypsum,  $m_{GH}$  and  $V_{GH}$  are the mass and volume of GH, respectively, and  $m_{GD}$  and  $V_{GD}$  are the mass and volume of GD, respectively.

 $\rho_{GD}$  and  $\rho_{GH}$  can be expressed by Eqs. (2) and (3).

$$\rho_{GD} = \frac{m_{GD}}{V_{GD}} \tag{2}$$

$$\rho_{GH} = \frac{m_{GH}}{V_{GH}} \tag{3}$$

From Eqs. (2) and (3),  

$$V_{GD} = \frac{m_{GD}}{m_{GD}}$$
(4)

$$V_{GH} = \frac{\rho_{GD}}{\rho_{GH}} \tag{5}$$

Substituting Eqs. (4) and (5) into Eq. (1), we obtain

$$\rho_{(GD+GH)} = \frac{m_{GD} + m_{GH}}{\frac{m_{GD}}{\rho_{GD}} + \frac{m_{GH}}{\rho_{GH}}} = \frac{\frac{m_{GD}}{m_{GH}} + 1}{\frac{1}{\rho_{GD}} \times \frac{m_{GD}}{m_{GH}} + \frac{1}{\rho_{GH}}}$$
(6)

On the other hand,  $C_{GH}$  is defined by Eq. (7).

$$C_{GH} = \frac{m_{GH}}{m_{GD} + m_{GH}} \times 100 \tag{7}$$

By transforming Eq. (7), Eq. (8) can be obtained as

$$\frac{m_{GD}}{m_{GH}} = \frac{100 - C_{GH}}{C_{GH}} \tag{8}$$

Substituting Eq. (8) into Eq. (6), Eq. (9) can be obtained.

$$\rho_{(GD+GH)} = \frac{100}{\frac{100 - C_{GH}}{\rho_{GD}} + \frac{C_{GH}}{\rho_{GH}}}$$
(9)

Finally, by transforming Eq. (9), Eq. (10) can be obtained.

$$C_{GH} = \frac{\rho_{GH}(\rho_{(GD+GH)} - \rho_{GD})}{\rho_{(GD+GH)}(\rho_{GH} - \rho_{GD})} \times 100$$
(10)

In other words, in advance, we determined the  $\rho_{GD}$  and  $\rho_{GH}$  of the recycled gypsum (e.g., GD dried in a furnace at 90 °C for 24 h [7-9]), through which we intended to determine  $C_{GH}$ . Furthermore, we determined the  $\rho_{(GD+GH)}$  of recycled gypsum mixed with GD and GH, and then,  $C_{GH}$  and  $C_{GD}$  could be obtained through simple calculations using Eq. (10). In the following, we experimentally investigate the validity of Eq. (10) by measuring the density of the recycled gypsum and reagent gypsum.

In this study, we reviewed the test results of the "density method" obtained in previous studies and examined the accuracy of  $C_{GH}$  for a short measurement period.

Hereafter, we aim to discuss the equations (in 2.) used in the "density method", the details of the recycled gypsum (in 3.), and the details of the measurement method (in 4. and 5.).

### 3. MEASUREMENT OF DENSITY OF RECYCLED GYPSUM AND REAGENT GYPSUM

### 3.1 Recycled Gypsum and Reagent Gypsum

For the density test, we used RGD made by crushing, grinding, and separating the waste gypsum boards delivered to an intermediate industrial waste treatment facility and piled up in a warehouse. RGH was prepared by heating RGD in a furnace in Oita Prefecture. The recycled gypsum used for the test was in a granular state, with a maximum particle diameter of 2.5 mm. The chemical composition of RGD and RGH can be referred elsewhere [10].

Calcium sulfate dihydrate (Kanto Chemical Co., Inc. reagent, extra pure, with a content of  $\geq$  98.0 %) and calcined gypsum (Kanto Chemical Co., Inc. reagent, extra pure, with a content of  $\geq$  99.0 %) were used. Hereafter, "calcium sulfate dihydrate" is referred to as "reagent GD" and "calcined gypsum" is referred to as "reagent GH" to match the description of recycled gypsum.

### **3.2 Density Test**

The analysis method for gypsum is specified in the "Methods for chemical analysis of gypsum (JIS R 9101: 1995)" [11] standard, which differs from the analysis method for soil. To ascertain the basic properties of gypsum, both "Methods for chemical analysis of gypsum (JIS R 9101: 1995)" [11] and "Laboratory tests of geomaterials" need to be considered. To determine the density of soil particles as geomaterials, a pycnometer is usually used as a test instrument in accordance with the "Test method for density of soil particles (JIS A 1202: 2020)" standard [12]. However, when this method is applied to GH, the sample must be immersed in distilled water to determine the volume of soil particles. Therefore, GH cannot be tested because it reacts with water and becomes a solid. In addition, when oven-drying is performed at 110 °C for 24 h after the test to obtain the mass of soil particles, GD may convert to GH or GA depending on the oven-drying temperature. Therefore, in this study, we conducted a density test using mineral oil instead of water to measure the volume, as specified in the "Physical testing methods for cement (JIS R 5201: 1997)" standard [13]. To investigate the effect of different drying conditions on the density, the test was conducted on three types of samples: samples in the "natural state", "oven-dried state at 45 °C" [11], and "oven-dried state at 110 °C", where the latter two samples were oven-dried at the corresponding temperatures for 24 h.

Mineral oil was stored in a thermostatic chamber at a temperature of 20 °C in advance, and the temperature of the mineral oil was stabilized in a Le Chatelier flask. Subsequently, gypsum in three different states was placed in the Le Chatelier flask to remove air, but in the case of gypsum, it was difficult to remove air. After removing the air, the flask was placed in a water tank, and the operation was repeated until the liquid level of the mineral oil hardly changed. However, as this method requires a



Fig. 1 Relationship between the elapsed period and the density of recycled gypsum from Oita Prefecture

longer test time and does not allow for efficient testing, the following operation was performed.

In other words, on the day the test was started (elapsed period 0 d), after removing the air, the flask was placed in a water tank and allowed to stand for approximately 1 d under the same conditions. Then, the liquid level of the mineral oil was read, and with sufficient removal of air by applying vibration again, the flask was allowed to stand for 1 d in the water tank. When no bubbles were observed and the level of mineral oil was almost unchanged, the measured value was used. Furthermore, based on the density test of cement [13], approximately 100 g of cement was used for the test. However, the density of gypsum was lower than that of ordinary Portland cement (3.15 g/cm<sup>3</sup>); therefore, the amount of sample was adjusted to 85–90 g.

# **3.3 Effect of Elapsed Period on the Density of Recycled Gypsum and Reagent Gypsum**

Fig. 1 shows the relationship between the elapsed period and the density of recycled gypsum from Oita Prefecture. The density of RGD in the natural state (symbol o) and oven-dried state at 45 °C ( $\Delta$ ) increased at the elapsed period of 1 d compared with that at an elapsed period of 0 d, and then remained almost constant at 2.35 g/cm<sup>3</sup> at an elapsed period of 3 d. In contrast, the density of oven-dried gypsum at 110 °C (□) was higher than that of natural gypsum  $(\circ)$  and oven-dried gypsum at 45 °C ( $\triangle$ ), and was almost the same as that of RGH  $(\bullet \blacktriangle \blacksquare)$ . On the other hand, regarding the RGH, the values for the natural state  $(\bullet)$ , oven-dried state at 45 °C ( $\blacktriangle$ ), and oven-dried state at 110 °C  $(\blacksquare)$  varied when the elapsed period was 0 d; however, the density increased when the elapsed period was 1 d, and then remained almost constant, reaching a stable value of 2.65 to 2.66 g/cm<sup>3</sup> when the elapsed period was 1 d to 5 d.

This indicates that the form of gypsum changed from GD to GH when RGD was dried in a furnace

at 110 °C for 24 h. We could confirm through these density results that GD is converted to GH by heating and dehydration at 90 °C for 24 h and GD is converted to GA by heating and dehydration at 120 °C for 24 h [7-9]. As mentioned earlier, even if some of the GD is converted to GA, GA is quickly converted to GH when left in the air [6]. Therefore, GA was considered GH at least at the beginning of the density test.

Fig. 2 shows the relationship between the elapsed period and the density of reagents GD and GH. The density of reagent GD was constant at an elapsed period of 7 d, the density of reagent GH was stable at an elapsed period of 6 d, and the densities of reagents GD and GH were 2.326 and 2.681 g/cm<sup>3</sup>, respectively.

The above results of the density tests of recycled gypsum and reagent gypsum indicate that the density varied owing to air bubbles that remained in the gypsum on the day of the test, that is, an elapsed period of 0 d. However, the air bubbles in gypsum were generally removed after an elapsed period of 1 d; subsequently, the density increased and the variation in density with the increase in elapsed period was slight.

Table 1 summarizes the densities of the recycled gypsum and reagent gypsum. For reference, the density of calcium sulfate dihydrate (content: 98.0% or more) is 2.32 g/cm<sup>3</sup> (20 °C) according to the safety data sheet [14] for the reagent (extra pure) from Kanto Chemical Co., Inc.. From the table, the density of RGD was 2.347 g/cm<sup>3</sup> under the natural state and oven-dried state at 45 °C, and the corresponding values for RGH were 2.660 and 2.662 g/cm<sup>3</sup>, respectively, which are almost the same. The density of RGD in the oven-dried state at 110 °C was 2.634 g/cm<sup>3</sup>, which is almost the same as that of RGH (2.651 g/cm<sup>3</sup>). As mentioned earlier, this indicates that the recycled gypsum oven-dried at 110 °C is converted to RGH, as predicted from the density of RGD oven-dried at 110 °C. Subsequently, regardless of the state of gypsum, the density of RGH ranged from 2.651 to 2.662 g/cm<sup>3</sup>, and the values were similar.

In contrast, for the reagent gypsum, the density was 2.326 g/cm<sup>3</sup> for GD and 2.681 g/cm<sup>3</sup> for GH. This indicates that the densities of recycled gypsum and reagent gypsum differ significantly depending on the form of gypsum. Based on the above results, the following values were adopted in this study for recycled gypsum from Oita Prefecture and commercially available reagent gypsum.

(1) RGD: 2.347 g/cm<sup>3</sup>; RGH: 2.662 g/cm<sup>3</sup>;

(2) Reagent GD: 2.326 g/cm<sup>3</sup>; reagent GH:  $2.681 \text{ g/cm}^3$ .



Fig. 2 Relationship between the elapsed period and the density of reagent gypsum

Table 1 Densities of recycled gypsum and reagent gypsum

(a)	Recycled	gypsum
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Condition of	Density (g/cm <sup>3</sup> )						
gynsum	Dihydrate	Hemihydrate					
Sypsum	gypsum	gypsum					
Natural state	2.347	2.660					
Oven-dried	2 347	2.662					
state at 45 °C	2.347						
Oven-dried	2 634	2.651					
state at 110 °C	2.034						
(b) Reagent gypsum							
Condition of	Density (g/cm <sup>3</sup> )						
Condition of	Dihardusta	Hamibudanta					

gypsiim	Dihydrate	Hemihydrate	
8) poun	gypsum	gypsum	
Natural state	2.326	2.681	

## 4. EFFECT OF ACCURACY OF DENSITY TEST ON GYPSUM HEMIHYDRATE CONTENT

In this section, we examine the accuracy of  $C_{GH}$ measured by substituting the density of gypsum with a different elapsed period determined in Section 3 into the equation for estimating  $C_{GH}$  using the density determined in Section 2. Furthermore, in advance, if the density of recycled gypsum in the dihydrate state ( $\rho_{GD}$ ), the density of recycled gypsum in the hemihydrate state ( $\rho_{GH}$ ), and the density of recycled gypsum ( $\rho_{(GD+GH)}$ ) in the target state are determined, we can calculate  $C_{GH}$  and  $C_{GD}$ using Eq. (10). However, if it is difficult to determine the density of recycled gypsum in the dihydrate state ( $\rho_{GD}$ ) and the density of recycled gypsum in the hemihydrate state ( $\rho_{GH}$ ), the density of reagent GD ( $\rho_{GD} = 2.326 \text{ g/cm}^3$ ) and the density of reagent GH ( $\rho_{GH} = 2.681 \text{ g/cm}^3$ ) can be used for convenience (see Eq. (12)). In the following, the densities of GD and GH used in the calculations are



Fig. 3 Relationship between the elapsed period and the density of reagent gypsum  $C_{GH}$  set in the specified ratio

referred to as reference values.

1) When using the density of RGD from Oita Prefecture,  $\rho_{GD} = 2.347$  g/cm<sup>3</sup>, and the density of RGH,  $\rho_{GH} = 2.660$  g/cm<sup>3</sup>, as reference values,

$$C_{GH} = \frac{2.660 \times (\rho_{(GD+GH)} - 2.347)}{(2.660 - 2.347) \times \rho_{(GD+GH)}} \times 100$$
(11)

2) When using the density of reagent GD,  $\rho_{GD} = 2.326 \text{ g/cm}^3$ , and the density of reagent GH,  $\rho_{GH} = 2.681 \text{ g/cm}^3$ , as reference values,

$$C_{GH} = \frac{2.681 \times (\rho_{(GD+GH)} - 2.326)}{(2.681 - 2.326) \times \rho_{(GD+GH)}} \times 100$$
(12)

Fig. 3 shows the relationship between the elapsed period and the density of a mixture of reagents GD and GH in a given ratio. The density tended to be lower at an elapsed period of 0 d, as in the case of recycled gypsum and reagent gypsum (see Figs. 1 and 2), but increased at an elapsed period of 1 d and remained almost constant thereafter.

Fig. 4 shows the relationship between the  $C_{GH}$  of reagent gypsum mixed with reagents GD and GH in the specified ratio and the density adopted in Fig. 3. Furthermore, the results obtained using Eqs. (11) and (12) (solid and dashed lines) and the density of recycled gypsum (•) as reference values are shown in Fig. 4. From the figure, the density values ( $\odot$ ) for reagent gypsum with a  $C_{GH}$  of 20%–80% are almost identical to the results obtained using Eqs. (11) and (12), indicating that  $C_{GH}$  can be estimated using these equations. For reference, Eq. (13) was obtained using the least-squares method to approximate the experimental results.

$$\rho_{(GD+GH)} = 2.323 + 0.003591C_{GH}(r = 0.999)$$
(13)

Fig. 5 shows the relationship between the elapsed period and  $C_{GH}$  (Eq. (12)), calculated from the density of a mixture of reagent dihydrate and reagent hemihydrate in a given ratio.  $C_{GH}$  increased



Fig. 4 Relationship between set  $C_{GH}$  and the density of mixture with reagent GD and reagent GH in the specified ratio



Fig. 5 Relationship between the elapsed period and  $C_{\text{GH}}$  (Eq. (12))



Fig. 6 Relationship between the elapsed period and the difference between  $C_{\text{GH}}$  (Eq. (12)) and set  $C_{\text{GH}}$ 

at an elapsed period of 1 d and remained almost constant thereafter.

Fig. 6 shows the relationship between the elapsed period and the difference between  $C_{GH}$  (Eq. (12)) calculated from the density and set  $C_{GH}$  when reagent GD and reagent GH are mixed in a specified ratio. The difference was between -7% and 5% when the elapsed period was 0 d, whereas it was

between 0% and 5% when the elapsed period was 1 d. This indicates that there is a large difference in  $C_{GH}$  calculated from the density for the elapsed period of 0 d. Nevertheless, after an elapsed period of 1 d, the difference was approximately 5%, indicating that  $C_{GH}$  can be estimated from the density with some accuracy even for the elapsed period of 1 d.

# 5. EVALUATION OF GYPSUM DIHYDRATE CONTENT WHEN GYPSUM RECYCLED FROM WASTE GYPSUM BOARDS IS HEATED USING ROTARY KILN FURNACE

The recycled gypsum used in Section 4 was made from waste gypsum board powder (RGD) delivered and crushed at an intermediate treatment facility in Oita Prefecture. It was then hemihydrated using a heating system available in Nagasaki Prefecture, but there were no intermediate treatment facilities in Oita Prefecture. As most intermediate treatment facilities are equipped with incinerators, the possibility of producing GH using existing furnaces needs to be investigated to develop an effective utilization technology for increasing the amount of waste gypsum boards. Therefore, in this study, we conducted a demonstration experiment to recycle waste gypsum board powder using an existing heating system (rotary kiln furnace, 1 m in diameter, 6 m in length, 1.7% gradient, and maximum heating temperature 900 °C). In this intermediate treatment facility, a rotary kiln furnace is typically used as a carbonizing furnace for wood chips, and the kiln burns at a high temperature of approximately 630 °C. The temperature inside the kiln was regulated using a temperature-control system. The temperature in the kiln was measured by controlling the temperature of the burner at the inlet side of the kiln. It was maintained at a predetermined level by readjusting the burner based on the measured temperature.

As mentioned earlier, the temperature control during heating is an important factor because the amount of crystal water in gypsum varies depending on the heating temperature, and the form of gypsum varies accordingly. However, the temperature distribution in a rotary kiln furnace is not usually measured. Therefore, a device needs to be installed to measure the temperature distribution in the furnace. Therefore, we modified the existing rotary kiln furnace to enable temperature control. First, thermocouples were fixed at four locations to measure the temperature inside the furnace. The thermocouples used were "coated thermocouples of type DP" (Sanko Electric Co., Ltd., + leg: Chromel, - leg: Almer), and the cables were passed through the pipes fixed to the furnace wall and taken out of the furnace through the rotary connectors. The temperatures measured by the four thermocouples



Fig. 7 Relationship between the elapsed period and the density of recycled gypsum (RGD from Oita Prefecture heated in rotary kiln furnace)

were continuously inputted into a recorder (TR-V500, Keyence Co., Ltd.). Using the modified rotary kiln furnace, we repeated the preliminary experiments under different temperature conditions to determine the optimum regeneration conditions for gypsum.

The amount of RGD fed into the rotary kiln furnace at one time was set to approximately 5 kg, and the heating temperature and rotation speed of the rotary kiln furnace were set to 140-170 °C and 30-50 Hz, respectively. The heating temperature and rotation speed were set based on the results of preliminary tests conducted in advance. The heating temperature was set high as RGD would be in contact with the high-temperature surface for a short duration owing to rolling in the furnace. The time required to discharge 5 kg of recycled gypsum from the furnace outlet was 5–9 min and the time required to complete the discharge was 12-29 min, for a total of 17-38 min.

The purpose of this full-scale demonstration using a rotary kiln furnace is to convert GD to GH by combining the heating temperature and rotation speed, and to minimize the ratio of GD and GA in the heated gypsum. Eqs. (11) and (12) were used to calculate  $C_{GH}$  and  $C_{GD}$  from the density of recycled gypsum, as proposed earlier. The density of gypsum was determined according to a density test of the cement. Eq. (13) obtained from Fig. 4 was used for the comparison.

Fig. 7 shows the relationship between the elapsed period and the density of RGD from Oita Prefecture heated in a rotary kiln furnace under various conditions. The density was 2.55 to 2.61 g/cm<sup>3</sup> at an elapsed period of 0 d under all conditions; these values lie between the density of 2.347 g/cm<sup>3</sup> for RGD and 2.651 to 2.662 g/cm<sup>3</sup> for RGH. However, after an elapsed period of 1 d, the density increased to 2.60–2.65 g/cm<sup>3</sup>, which is close to the density of RGH. It was evident from the density values that heating the RGD in the rotary kiln furnace under the specified heating temperature



Fig. 8 Relationship between the elapsed period and  $C_{\text{GH}}$  (Eq. (11)) (RGD from Oita Prefecture heated in rotary kiln furnace)



Fig. 9 Relationship between the elapsed period and  $C_{\text{GH}}$  (Eq. (12)) (RGD from Oita Prefecture heated in rotary kiln furnace)

and rotation speed caused a morphological change from GD to GH.

Fig. 8 shows the relationship between the elapsed period and  $C_{GH}$  when the RGD from Oita Prefecture was heated using a rotary kiln furnace. Eq. (11), which uses the densities of RGD and RGH as reference values, was used to calculate  $C_{GH}$ . As shown in the figure,  $C_{GH}$  ranged from 68% to 86% at an elapsed period of 0 d in the rotary kiln, whereas  $C_{GH}$  ranged from 85% to 95% at an elapsed period of 1 d in the rotary kiln; the variation in  $C_{GH}$  with the increase in elapsed period was slight.

Fig. 9 shows the relationships between the elapsed period and  $C_{GH}$  when the RGD from Oita Prefecture was heated using a rotary kiln furnace. For calculating  $C_{GH}$ , the results of Eq. (12) were used for convenience, assuming that the densities of RGD and RGH could not be obtained as reference values. The figure shows that  $C_{GH}$  ranged from 68% to 82% at an elapsed period of 0 d. In comparison, it ranged from 80% to 90% at an elapsed period of 1 d, which is smaller than that obtained using the values of RGD and RGH (Eq. (11)) in Fig. 8.

Fig. 10 shows the relationships between the elapsed period and the difference in  $C_{GH}$  obtained



Fig. 10 Relationship between the elapsed period and the difference between  $C_{\text{GH}}$  in Eq. (11) and  $C_{\text{GH}}$  in Eq. (12) (RGD from Oita Prefecture heated in rotary kiln furnace)



Fig. 11 Relationship between the elapsed period and  $C_{\text{GH}}$  (Eq. (13)) (RGD from Oita Prefecture heated in rotary kiln furnace)

from Figs. 8 and 9 when the RGD from Oita Prefecture was heated using a rotary kiln furnace. There was a difference of 3% to 4% after an elapsed period of 1 d. This indicates that, if the values of RGD and RGH could not be obtained, the error in calculating  $C_{GH}$  using the values of reagent GD and reagent GH as reference values would be 3% to 4%. This further indicates that, even if the values of RGD and RGH could not be obtained, the accuracy would be sufficient to obtain an approximate value.

Fig. 11 shows the relationship between the elapsed period and  $C_{GH}$  when the RGD from Oita Prefecture was heated using a rotary kiln furnace. For the calculation of  $C_{GH}$ , the result of Eq. (13), which is the experimental equation for the case of using reagent gypsum shown in Fig.8, is used.  $C_{GH}$  ranged from 64% to 81% at an elapsed period of 0 d, whereas it ranged from 79% to 89% at an elapsed period of 1 d, which is smaller than that obtained using the values of RGD and RGH in Fig. 8.

Fig. 12 shows the relationship between the elapsed period and the difference in  $C_{GH}$  (using Eqs. (11) and (13)) obtained from Figs. 8 and 11 when the RGD from Oita Prefecture was heated using a rotary kiln furnace. There was a difference of 5%

between the two after an elapsed period of 1 d. This indicates that the experimental value obtained from Eq. (13) is 5% lower than the reference values of RGD and RGH, but it is sufficiently accurate to obtain a rough estimate.

Table 2 summarizes the densities of the recycled gypsum heated in a rotary kiln furnace and  $C_{GH}$ estimated using the density (using Eqs. (11)-(13)). Here, the heating temperature was the set temperature of the rotary kiln furnace. From the table, the measured densities are in the range of 2.60 to  $2.65 \text{ g/cm}^3$ , which is close to the density of GH, i.e., 2.68 g/cm<sup>3</sup>. Based on the density results, the gypsum content was calculated as follows: the GD content of 8.2%–19.4% and the  $C_{GH}$  of 80.6%– 91.8%. For comparison, the gypsum obtained under these heating conditions was subjected to X-ray diffraction analysis and compared with the calculated  $C_{GH}$ . We obtained the areas of the dihydrate peak ( $2\theta = 11.6^{\circ}$ ) and hemihydrate peak  $(2\theta = 14.7^{\circ})$  as 1211 and 80817 deg·cps, respectively, indicating that 87.0% of the gypsum was hemihydrate. In contrast, the calculated gypsum contents were 96.1% from Eq. (11), 91.8% from Eq. (12), and 90.5% from Eq. (13), all of which were higher than those obtained via X-ray diffraction. However, as there are peaks of GD, GH, and GA in the scanning range of  $2\theta = 5^{\circ}-25^{\circ}$ , a result higher than 87% is likely to be obtained when the scanning range of  $2\theta$  is further extended. Therefore, the value of gypsum content obtained using density is considered reasonable.

In any case, although there was a difference of approximately 3% between the results obtained using the adopted density and those obtained using the density with an elapsed period of 1 d, the gypsum content obtained using density is generally consistent with the results obtained via X-ray diffraction analysis in the demonstration experiment at the full-scale level using a rotary kiln furnace. Thus, we believe that the method proposed in this study for determining  $C_{GH}$  using density at an elapsed period of 1 d is useful for determining  $C_{GH}$ in a simple and rapid manner. Furthermore, these results indicate that the optimum heating conditions for producing GH (a heating temperature of 140 °C and a rotation speed of 50 Hz) were set for the existing rotary kiln furnace used in this study.

On the other hand, when applying the quality control method using density proposed in this study, recycled gypsum of a certain quality can be stably supplied by using the density as an index in normal quality control and periodically controlling quality using X-ray diffraction analysis.

#### 6. CONCLUSION

In this study, to use recycled gypsum mainly as a geotechnical improvement material, a method to



Fig. 12 Relationship between the elapsed period and the difference between  $C_{\text{GH}}$  in Eq. (11) and  $C_{\text{GH}}$  in Eq. (13) (RGD from Oita Prefecture heated in rotary kiln furnace)

Table 2 Densities of recycled gypsum and reagent gypsum

T (°C)	RS (Hz)	Density (g/cm <sup>3</sup> )	Gypsum hemihydrate		
			content obtained using		
			equations (%)		
			Eq.	Eq.	Eq.
			(11)	(12)	(13)
	30	2.610	85.2	82.2	79.9
		2.619	87.8	84.5	82.4
140	40	2.646	95.5	91.3	89.9
140		2.644	94.9	90.8	89.4
	<u>50</u>	2.648	<u>96.1</u>	<u>91.8</u>	<u>90.5</u>
		2.636	92.7	88.8	87.2
	30	2.628	90.4	86.8	84.9
150		2.624	89.2	85.8	83.8
150	40	2.604	83.4	80.6	78.3
		2.614	86.3	83.2	81.0
	30	2.630	90.9	87.3	85.5
		2.619	87.8	84.5	82.4
160	40	2.610	85.2	82.2	79.9
160		2.607	84.3	81.4	79.1
	50	2.630	90.9	87.3	85.5
		2.617	87.2	84.0	81.9
170	30	2.629	90.6	87.0	85.2
		2.622	88.6	85.3	83.3
	40	2.624	89.2	85.8	83.8
		2.616	86.9	83.7	81.6

*T:* Heating temperature, RS: Rotation speed Upper row: Using adopted density Lower row: Using the density for the elapsed period of 1 d <u>Value:</u> Heating conditions resulting in the largest  $C_{GH}$ 

estimate  $C_{GH}$  focusing on density was investigated as a simple approach to determine the quality of recycled gypsum.

The main results obtained in this study are listed below.

1. The way of the density test for cement is effective in measuring the density of gypsum, and the results differ depending on the drying condition of the sample.

2. If the densities of recycled gypsum in the dihydrate and hemihydrate states are determined in advance, and if the density of recycled gypsum in which dihydrate and hemihydrate are mixed is determined,  $C_{GH}$  and  $C_{GD}$  can be determined by using such as Eqs. (11)–(13) examined here.

3. Although there was a difference of approximately 5% in  $C_{GH}$  calculated from the density after an elapsed period of 1 d,  $C_{GH}$  can be estimated from the density with some accuracy even after an elapsed period of 1 d.

4. In the results of a full-scale experiment using a rotary kiln furnace, despite a difference of approximately 3% in  $C_{GH}$  between the cases using the adopted density and the density with an elapsed period of 1 d, the validity of the method for estimating  $C_{GH}$  using density was experimentally confirmed from the general agreement with X-ray diffraction tests.

To prevent gypsum recycled from waste gypsum boards from becoming waste, it is necessary to focus on the development of quality control technology and a technology for the effective utilization of recycled gypsum. In this study, we focused on  $C_{GH}$ , but there are concerns about fluorine leaching and hydrogen sulfide generation when using recycled gypsum; therefore, we will need to consider these issues in the future. By using the method proposed in this study to determine  $C_{GH}$  simply, "recycled gypsum with consistent quality" and "recycled gypsum that has been sufficiently heated and dehydrated" can be delivered to the site. In the future, a system that can immediately determine the  $C_{GH}$  of recycled gypsum delivered to the site on-site needs to be established, and it is believed that the further development of quality control technology will be necessary for the future.

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