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The property of inorganic insulation material depending on CSA contents and atmospheric steam curing condition

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In this study, we have made a cement based inorganic insulation material and added CSA (Hauyne Clinker) to reduce the demolding time and enhance the handling workability. CSA contents were varied by 0%, 1%, 3%, 5% and the atmospheric steam curing was tried for enhancing the compressive strength. As the CSA contents are increased to 5%, a rapid reaction of hydration caused the sinking of the slurry. So, the setting-retarder was added to control the reaction of hydration. By this, the sinking of the slurry was controlled but the height of the green body after expansions was a little bit lowered. In the CSA-added slurry, it was possible to demold within 24 hours and in case of CSA 5%-added, the sufficient workability was secured. Atmospheric steam curing (temperatures −40 ~ 80 °C, for 6~10 hrs.) was attempted to improve the compressive strength and found that an excellent strength of 0.25 MPa was achieved at 80 °C for 8 hrs. Specific gravity was about 0.12 ~ 0.13 g/cm^3 and heat conductivity was about 0.045 W/mK in all specimens. This strategy significantly improves the compressive strength of CSA 5%-added specimen up to 25% compared to without CSA added specimen.

Key words: Inorganic Insulation, CSA, Atmospheric Steam Curing, Compressive Strength.

Introduction

Recently, the importance of greenhouse gas and energy consumption reduction has dramatically emerged. However, Korea's energy intensity (energy consumed per GDP 1,000 dollars) is 0.339, which is 3 times higher than that of Japan and is in the highest level among Organization for Economic Co-operations and Development (OECD) member countries [1]. The domestic construction sector's greenhouse gas emission accounts for around 25% of total greenhouse gas emission. It is expected that the energy consumption of the structure rises by 40% in the future [2-3]. Accordingly, the various studies on a structure's greenhouse gas emission reduction and energy consumption reduction are being conducted. The advanced countries have been developing a lot of high-performance insulation materials [4-5].

Most of the insulation materials are categorized into the organic and inorganic material. The organic insulation materials include styrofoam, urethane foam, phenolic foam, etc. To date, about 70% of the structures in Korea uses organic insulation materials. So in case of fire, the problem of loss of life caused by toxic gas emissions has seriously emerged [6]. On the other hand, inorganic insulation materials such as glass wool and rock wool are noninflammable materials and not harmful to the human body in case of fire. However, the vulnerability to water, tangling and sagging of the fiber hindered them from commercial application [7-8].

Recently, the study on the noninflammable insulation materials to use cement as a main raw material being carried out and found one of the candidate insulating material because of its superior nonflammability, water-resistance, etc. property considered superior to other materials [9]. In this work, we used cement and CSA to prepare inorganic insulation materials and applied atmospheric steam curing conditions to get the effect of early strength improvement. In addition, the study was examined insulation performance by measuring specific gravity and heat conductivity [10].

Research Method

The study was conducted with cement, blast furnace slag, anhydrite, quicklime, and CSA as the main raw materials of the inorganic insulation material and aluminum (Al) powder as the foaming agent. CSA was utilized to enhance hardening and improve initial strength. Blast furnace slag fine powder was used to improve long-time strength. The fine powder of blast furnace slag was made by grinding blast furnace slag(4,500 cm^2/g) with a vibration mill to a fineness of 6,000 cm^2/g. Ordinary portland cement (OPC) was ground to a fineness of 7,500 cm^2/g and CSA contents were controlled with 1%, 3% and 5% to improve the strength and reduce hardening time of the cement-used inorganic insulation material. In the condition of CSA 5%, the rapid reaction of hydration caused the sinking

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Table 1. Mixing ratios of thermal insulation used CSA (unit: %).

No.	OPC	Blast furnace slag	Anhydrite	Lime	CSA	Retarder	Al powder	Mixed water
1	70	10	10	10	0	0	0.6	130
2	69	10	10	10	1	0	0.6	130
3	67	10	10	10	3	0	0.6	130
4	65	10	10	10	5	0.55	0.6	130

of the slurry. The setting retarder was added to control the reaction of hydration.

Table 1. shows the mixing ratios of inorganic insulation material used CSA. Cement content was 70% slag, anhydrite and quicklime contents were 10% respectively, CSA replacing cement was controlled at 1%, 3%, and 5%. Al powders were used to create an expanded green body of 0.6%, Silicon repellent used to prevent water penetration fixed at 1%. When CSA was added at 5%, the setting-retarder was used at 0.55%.

To make the inorganic insulation material, it was mixed OPC, slag, anhydrite, quicklime, and CSA without water at the first mix stage and mixed them after adding the water by 130% at the second mix stage. Al power and silicon repellent were mixed at the third mix stage. After all mixed, the slurry was poured into the styrofoam mold (230 × 230 × 230 mm). Hardening speed of the slurry was measured from 1 hour after pouring (after maximum expansion) by a Vicat apparatus. After 24 hours, the green body was removed the styrofoam mold and conducted atmospheric steam curing. The temperature for atmospheric steam curing was set at 40 °C, 60 °C, and 80 °C. The time for atmospheric steam curing was set at 6 hrs., 8 hrs. and 10 hrs. The specimens finished atmospheric steam curing stayed for 28 days in the condition that the indoor temperature was 25 ± 2 °C and relative humidity was 60%. After 28 days, inorganic insulation material was measured the basic properties such as density, compressive strength, and heat conductivity and analyzed microstructure, etc.

Result & Discussion

Characteristics of Slurry Hardening

The slurry for making inorganic insulation material has a disadvantage that the hardening time is long because of the high mixed water content. Thus the hardening time has to shorten for increase productivity and workability. In this study, to quantify the hardening shown above, we measure the depth of penetration with the Vicat apparatus as indicated in Fig. 1.

The Vicat apparatus is used mainly to measure the cement setting time. Thus the use of the apparatus is thought to be valid in measuring the hardening speed of the cement-used inorganic insulation material as well. We measure the depth of penetration every 2 hours by slowly lowering the Vicat needle from the

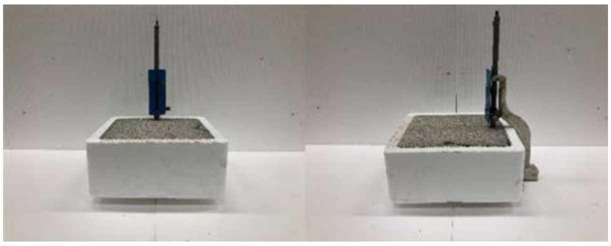


Fig. 1. Measurement of hardening time used Vicat apparatus.

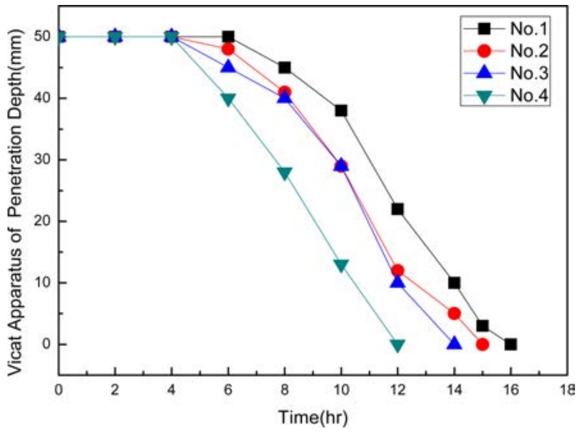


Fig. 2. Depths of penetration on No.1~No.4 by curing time.

surface.

Fig. 2 shows the penetration depth of the Vicat needle in No.1~4 depending on the curing time. In all the specimens, as the curing time increases, the penetration depth decreases. By increasing the CSA contents, the penetration depth decreases rapidly. Furthermore, specimen No.4 with CSA 5% takes 12 hrs. and No.1 with CSA 0% takes 16hrs. until the Vicat needle cannot penetrate. It means that as the CSA contents increases, the hardening speed increases very rapidly. Thus, it is expected that under the condition of CSA 5% the removal of the mold is possible at the earliest time [11].

The optimal demold time was verified after making No. 4 slurry of the fastest hardening time. We remove the mold in curing for 6 hrs., 12 hrs. and 24 hrs. in order to check the demold time after charging the slurry. Fig. 3 indicates the extent of damage of the molded object depending on curing time. The molded objects cured for 6 hrs. and 12 hrs. were partially destroyed in removing the mold and damaged during the process which suggests that hardening is not

(a) 6hrs. (b) 12hrs. (c) 24hrs.

Fig. 3. Appearance condition of No.4 specimen as curing time.

sufficiently. On the other hand, the molded object cured for 24 hrs. was neither destroyed nor damaged while removing the mold during the process. Thus, it is judged that the optimal curing time for removing the mold is 24 hrs.

Properties of insulation materials

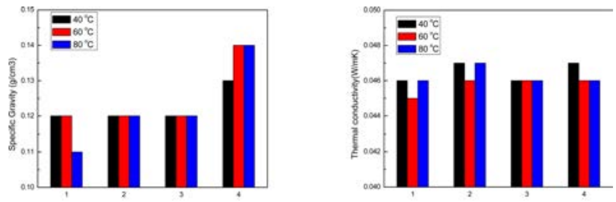
The molded object made after completing atmospheric steam curing was stayed for 28 days in the condition that the indoor at temperature 25 ± 2 °C and 60 RH%. The molded object (insulation material) was dried for 24 hours at 100 °C and then its compressive strength and heat conductivity were measured.

Fig. 4(a) represents specific gravity depending on CSA contents and curing temperature (curing time was fixed for 8hrs.) In the case of specimen No.1, No.2 and No.3, their specific gravities are within a narrow range of 0.11 ~ 0.12 g/cm^3 while No. 4 stands at 0.14 g/cm^3 which is a bit higher than specimens. This could be due to the early hardening resulted from rapid hydration prompted by the increase of CSA contents makes the slurry foam in a little bit low height.

Fig. 4(b) shows the heat conductivity in the same condition. All the insulation materials made after curing for 28 days are within the narrow range of heat conductivity (0.045 ~ 0.047 W/mK). We infer that this effect could be due to the amount of pore and the kind of pore in the material rather than CSA contents.

Fig. 5 indicates compressive strength after 28 days with different CSA contents and atmospheric steam curing time. As the CSA contents and atmospheric steam curing time increases, the compressive strength is improved. But in case of curing for 10 hours, the compressive strength values of all the specimens were sharply dropped due to a lot of the cracks occur inside the insulation specimen exposed to heat for a long time. Furthermore, the specimen No. 4 has the best compressive strength of 0.25 Mpa which is 25% higher than specimen No. 1 (0.20 Mpa). Based on these results, it is revealed that CSA contents and atmospheric steam curing have a great effect on the compressive strength. Hence, it is suggested that the optimal condition for the inorganic insulation material is 5% CSA contents and 8hrs. atmospheric steam curing time.

Fig. 6 shows the compressive strength of the sample after 28 days for different atmospheric steam curing temperature and time. It shows that that as atmospheric



(a) specific gravity (b) heat conductivity

Fig. 4. Specific gravity and heat conductivity by CSA content and curing temperature.

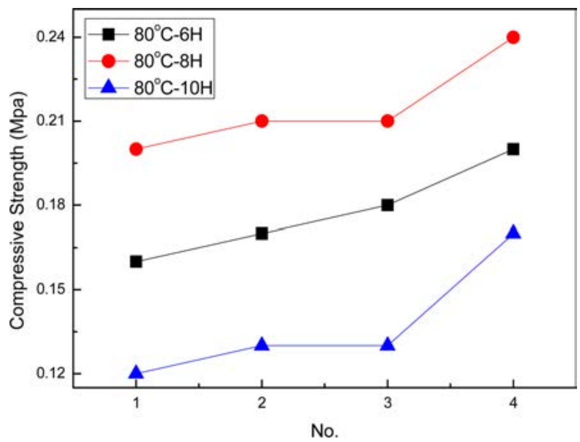


Fig. 5. Compressive strength as CSA content and curing temp. at 28 days.

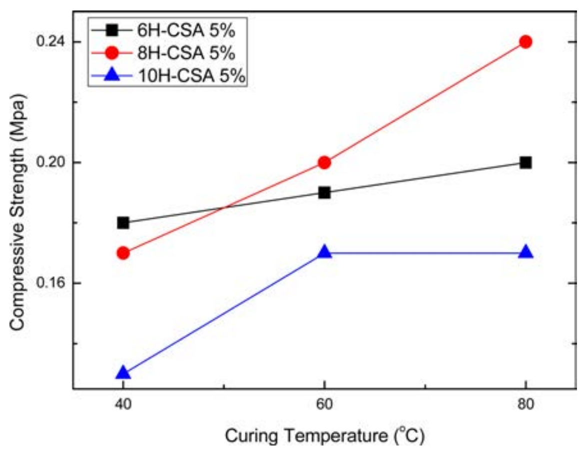


Fig. 6. Compressive strength as curing temp. and time at 28 days.

steam curing temperature is increased, compressive strength is also grown up. It can be seen that specimen with 80 °C-8 hr. had an excellent compressive strength (0.25 Mpa) which is about 32% higher than 40 °C-

8hr.(0.17Mpa). We believe that this improvement could be due to active hydration which can be provoked by the increase of curing temperature.

Crystal Characteristics and Microstructure of the Insulation Material

Fig. 7 indicates the XRD patterns to analyze the crystal characteristics of the molded objects made after curing for 24 hrs. $CaCO_3$, Ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$) and $Ca(OH)_2$ were detected as the major crystal in all the specimens. Ettringite and $Ca(OH)_2$ are the main hydrate of the cement while $CaCO_3$ appears due to the presence of limestone in the cement. Furthermore, as shown in Table 2 that as the CSA contents increase the ettringite peak intensity also increases. Overall, the ettringite phase helps to reduce the hardening time and the reduction of mold removal time and finally securing workability. In addition, the XRD patterns are similar to the result acquired in the

Table 2. Ettringite peak intensity at 24 hrs. (unit: Count per Sec.)

No.	1	2	3	4
Intensity	1800	2200	2400	2800

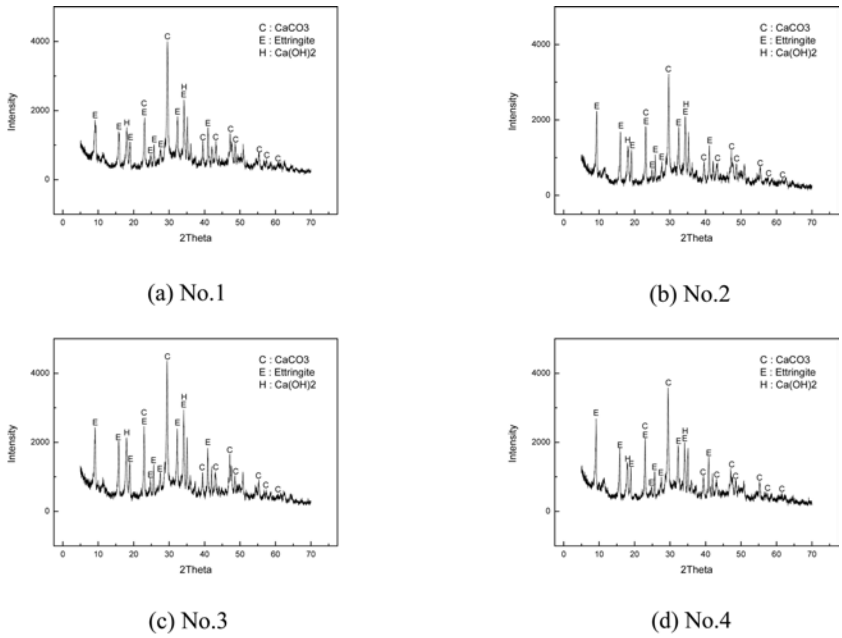


Fig. 7. XRD patterns as CSA content at 24 hrs.

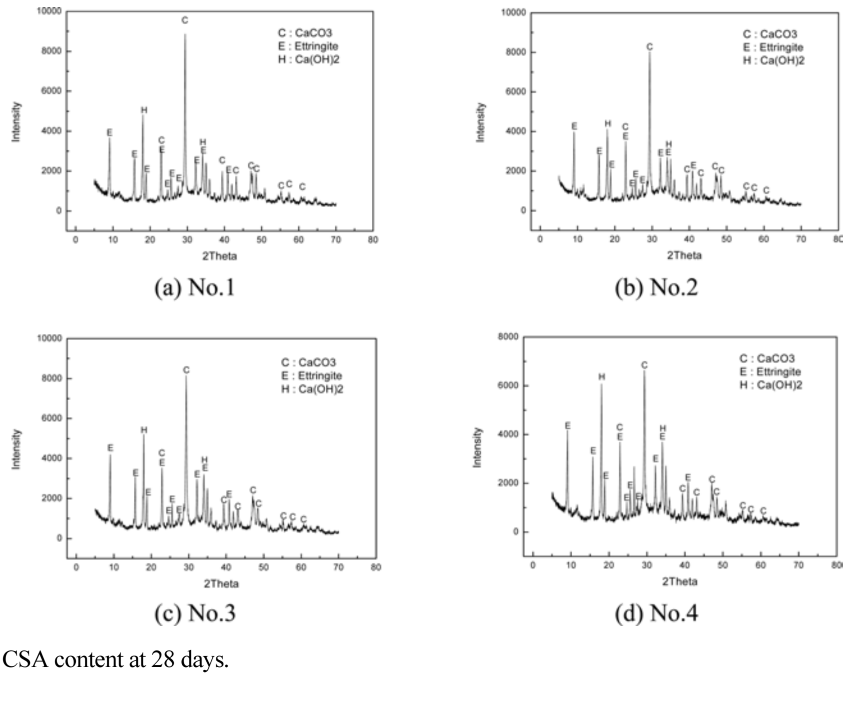


Fig. 8. XRD patterns as CSA content at 28 days.

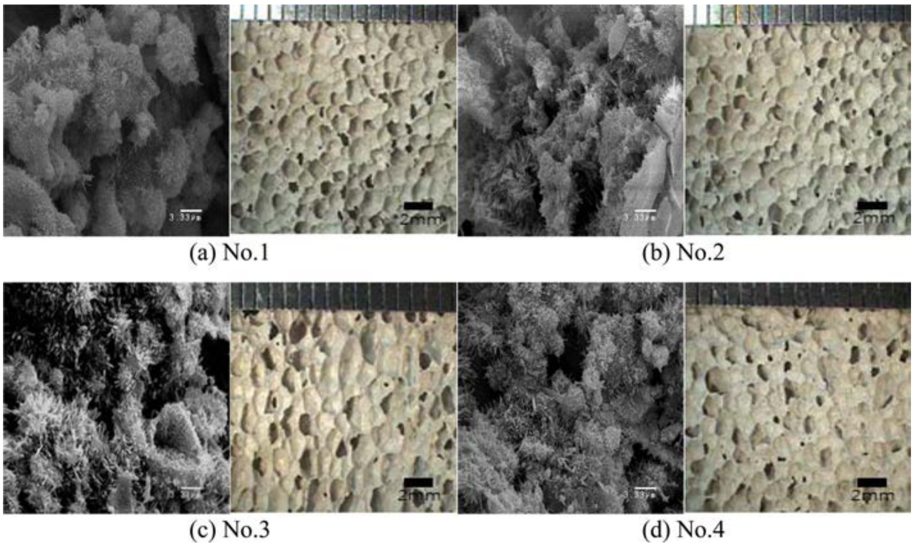


Fig. 9. Microstructure and Pore Characteristics at 28 days.

in Fig. 3.

Fig. 8 indicates XRD patterns of the insulation material made after curing for 28 days. $CaCO_3$, ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$) and $Ca(OH)_2$ are detected as the major crystal in all the specimens. Moreover, as the CSA contents increase a slight increase in the ettringite peak intensity is observed.

Fig. 9 shows pore wall microstructure and characteristics of the pore in the inorganic insulation material with various CSA contents. As discussed above that CSA contents increase the ettringite phase which can found here in the inside of pore wall and suggest that the hydrate is formed by a compact structure. We infer that such a compact pore wall improves the compressive strength of the insulation material.

Furthermore, the specimens were observed through camscope and found that the pore of all the specimens ranges from 1 mm to 2 mm with the average pore size about 1.5 mm. In addition, the shape and size of the pore are kept unchanged regardless of CSA content and the pores usually keep the same shape. Most of the pores in all the insulation materials exist in the form of a connected pore or open pore, but some pores exist in the form of a closed pore. Especially, we infer that the closed pore has a positive effect on the realization of excellent heat conductivity.

Conclusions

This study was carried out with CSA under atmospheric steam curing conditions to improve the compressive strength and reduce its hardening time. We have successfully controlled the CSA contents and atmospheric steam curing in the various ways and drew the conclusion as follows.

In the case of 5% CSA sinking phenomena happened in the process of making a slurry, but the addition of the setting-retarder prevented the further sinking phenomena. This 5% CSA slurry have relatively rapidly hardened and a superior compressive strength was achieved.

The compressive strength of the inorganic insulation material varies depending on the experimental condition of atmospheric steam curing. It is revealed that the optimal curing condition for compressive strength is 80˚C for 8hrs. Furthermore, curing for 10 hrs. about the compressive strength drops due to heat shock which causes the crack.

The main crystals of the inorganic insulation material are $CaCO_3$, ettringite and $Ca(OH)_2$. The ettringite peak intensity increases by increases CSA contents and finally, the inside of pore wall became more compact which improve the compressive strength of the insulation materials.

Next, by using atmospheric steam curing on the condition of 80 °C-8 hrs. together with 5% CSA slurry, the most excellent compressive strength is acquired. As described above in phenomena 3, the compressive strength stands at 0.25 Mpa, heat conductivity is 0.045 W/mK and specific gravity ranges 0.12 ~ 0.13 g/cm^3.

As described above, we can infer that when adding CSA, early hardening of slurry and the resulting reduction of mold removal time are possible. In addition, the findings show that the inorganic insulation material made after proper atmospheric steam curing has the optimal compressive strength and heat conductivity.

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