Journal of Ceramic Processing Research. Vol. 19, No. 5, pp. 428~433 (2018)

J O U R N A L O F
Ceramic Processing Research

The characteristics of mineral hydrate insulation material using activated cement prepared from pilot plant activation system

Sung Kwan Seo, Yong Sik Chu*, Tae Yeon Kim and Yoo Kim
Energy & Environmental Division, Korea Institute of Ceramic Eng. & Tech., Jinju 660-031, Korea

In this study, using the pilot plant activation system, the activated cement has been manufactured and then applied to the manufacturing process of mineral hydrate insulating material. The fineness of the activated cement is controlled at 5, 000 cm^2/g and 7,500 cm^2/g and the features of mineral hydrate insulating material, using OPC and the activated cement for each degree of fineness, has been analyzed. As the result of analyzing the crystal habit of the manufactured mineral hydrate insulting material, it is analyzed that the main crystal phase of specimen is tobermorite and some quartz peak has been detected. As the degree of fineness of the activated cement increases, the height of bubble of slurry increases as well, whereas the tendency for the density character to decrease has been detected. Along with it, as the density character decreases, the compression strength has decreases, whereas the tendency for the thermal characteristic to increases has been detected. The main features of mineral hydrate insulating material, using the activated cement with the fineness of 7,500 cm^2/g, the compression strength of 0.36 MPa, and the thermal conductivity of 0.044 W/m·K, presents the excellent features as insulation.

Key words: Activated cement, Activation system, Mineral hydrate insulation material, Thermal conductivity.

Introduction

Energetically modified material (EMM) is a material that uses mechanochemically activated powder and it is designated as energetically modified fly ash (EFA) and energetically modified cement (EMC) in accordance with the type of its powder [1-2]. The mechanochemical phenomenon occurs in the middle of the process of transferring mechanical energy into a solid object. The process of transforming the internal structure of a substance or its physical properties so that it will ultimately be activated into a solid matter is highly complicated and it is affected by the influence of methods of mechanical processes, solidity of a substance, and lattice structure of a crystal [3]. Among the mechanical processes of powder, it is general to use the method to grind it with a grinder, such as a ball mill or a vibratory mill and the reactivity increases as a particle is activated in the process of grinding powder. The extent of activation of a particle of powder, therefore, possesses the intimate correlation with the method of grinding. Thus, there is obvious necessity to develop and apply an apt method of grinding, in accordance with features of each type of powder, and, for such reasons, a type of grinder, media of grinding, and arrangements of media should be examined [4-5].

Meanwhile, in case of fire, in a building, constructed with the organic insulation, the risk of damage of life and property increases even more due to rapid spreading of fire and producing poisonous gas.[6-7] The growing tendency of the usage of the inorganic insulation is, therefore, understandable because of the change of recognition of society and reinforcement of governmental regulation. Mineral hydrate insulation is a typical type of the inorganic insulation.[8] The mineral hydrate insulation is manufacture by hydrothermal synthesis, with the condition of steam curing at high temperature and high pressure, after manufacturing green cake, by adding a blowing agent into mixed slurry of raw materials, such as cement, calcium oxide, and plaster. Before the hydrothermal synthesis, the phenomenon of backing of slurry is frequently and easily detected with the green cake, due to the high content of slurry and a blowing agent and it poses negative influence on the manufacturing of the mineral hydrate insulation [9]. As a measure to control the above-mentioned phenomenon of backing slurry, it is possible to control the speed of reaction by the activation of cement. To apply the activated cement into the actual process great volume of activated cement shall be supplied. In case of domestic, however, there are not enough facilities to grind for the activation of cement.

In this study, therefore, the activated cement will be manufactured, by using the activation system with the pilot plant scale at Korea institute of ceramic engineering and technology (KICET), and then it will be applied to the manufacturing process of mineral hydrate insulation. In addition, to assess the features of insulation for each powder of PP manufactured

*Corresponding author:
Tel : +82-55-792-2463
Fax: +82-55-792-2458
E-mail: yschu@kicet.re.kr

activated cement, the operating condition of activation system has been controlled for the manufacturing the activated cement for each type of powder and the evaluation of characteristics has been conducted after the manufacturing of mineral hydrate insulation.

Experimental Method

By controlling the operating condition of pilot plant activation system (PPAS), the activated cement with the fineness of 5,000 cm^2/g and 7,500 cm^2/g has been manufactured. As it is shown in the Fig. 1, the main facilities of the activation system are composed with a raw material silo, which is a storage of the raw material, a hopper, a continuous vibratory mill, a separator, a bag filter, and a product silo. Besides, the transferring facility for the raw material and its products is constituted with a bucket elevator, a screw conveyor, a Hoist crane, a CVM control panel, which controls the vibratory mill, and a main control system, controlling the entire facility. The gross output of the PPAS, constituted with the above-mentioned facilities, may vary, depending on the type of material, that has been used, with the range of approximate 2 to 8 tons, with 8 operating hrs in a day.

To assess the properties of mineral hydrate insulation for each powder of PPAS manufactured activated cement, the condition of mixture, presented in the Fig. 1, has been designed. In this case, for the No. 1, inactivated ordinary Portland cement has been used and for No. 2 and No. 3, activated cement with the fineness of 5,000 cm^2/g and 7,500 cm^2/g by the Pilot Plant activation system have been used respectively. In case of calcium oxide and plaster, they have been fixed at 12% and 5%, respectively, and the amount of the silicone water repellent was fixed at 0.1%, comparing to the powder of the raw material. By controlling the rate of mixing water by 130% (comparing to the raw material), the slurry has been manufactured and the powder of aluminum has been mixed at the rate of 0.52% for the formation of bubble.

Then, the manufactured slurry has been aged inside of a thermo-hygrostat (CC600, Woojin Precision, Korea) at the temperature of 50 °C and the relative humidity of 60% for five hours. At this point, the aluminum powder, which has been added as a blowing agent, forms pores, expands its volume, and the hydration reaction has been processed. After the process of manufacturing, the green cake has been charged into an autoclave (J-180T, JISICO, Korea). Then, the temperature was risen till 180 °C for an hour, and then the process of the hydrothermal synthesis was proceeded for seven hours at the temperature of 180 °C.

The specimen, after the hydrothermal synthesis, was sliced into pieces, with the measurement of 100*100*100 mm and 200*200*200 mm, was dried till it reached the constant weight, and the analysis of properties of mineral hydrate insulation was conducted. With the specimen of measurements 100*100*100 mm, the size of pores, density, and compression strength were measured, and the observation of matrix and analysis of crystalline were conducted by using a scanning electron microscope (SM-300, Topcon, Japan) and a X-

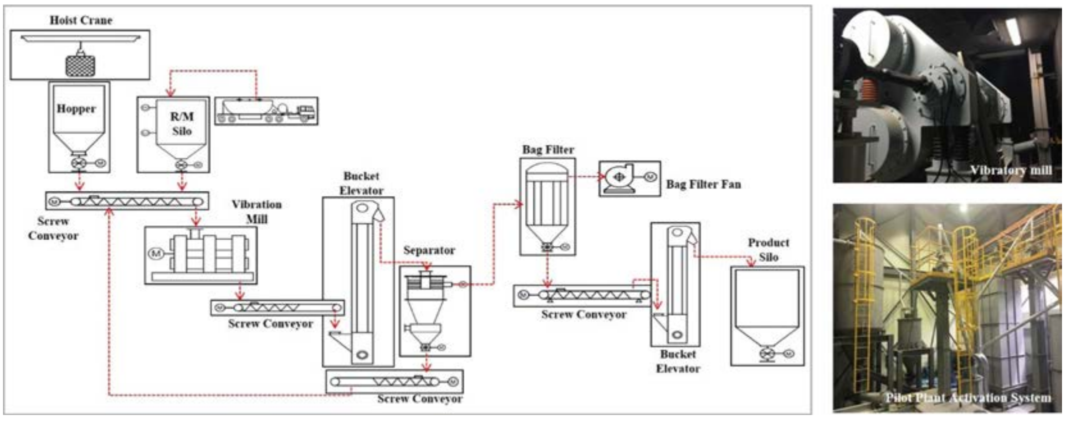


Fig. 1. Pilot plant activation system.

Table 1. Mixing ratios of mineral hydrate insulation used activated cement. [Unit: wt%].

No.	Binder				R/B*	A/B*	W/B*
	Quartz	Cement	Lime	Anhydrite			
1		50(OPC)					
2	33	50(A·C 5000)	12	5	0.1	0.52	130
3		50(A·C 7000)					

*R/B: Repellent/Binder ratio, A/B: Al powder/Binder ratio, W/B: Water/Binder ratio.

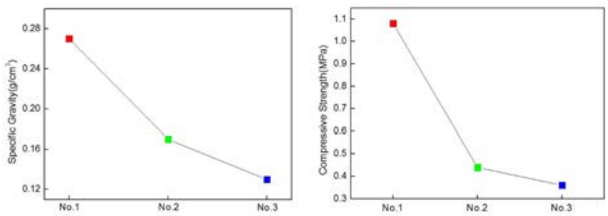
scattering analyzer (D5005D, Ziemens, Germany). In addition, the specimen with the measurement of 200*200*200 mm was used to assess the thermal conductivity by using the thermal conductivity tester with the application of heat flow meter (HC-074, EKO, Japan).

Result and Discussion

After the completion of the hydrothermal synthesis, the specimen was split into a piece with the volume of 100*100*100 mm, was dried till it reached the constant weight at the temperature of 100 °C, and was assessed for the bulk specific density, including open pores. For the specimen, after finishing the measurement of density, was assessed for the compression strength with a universal testing machine. Fig. 2. Presents the result of assessment of density and compression strength of mineral hydrate insulation.

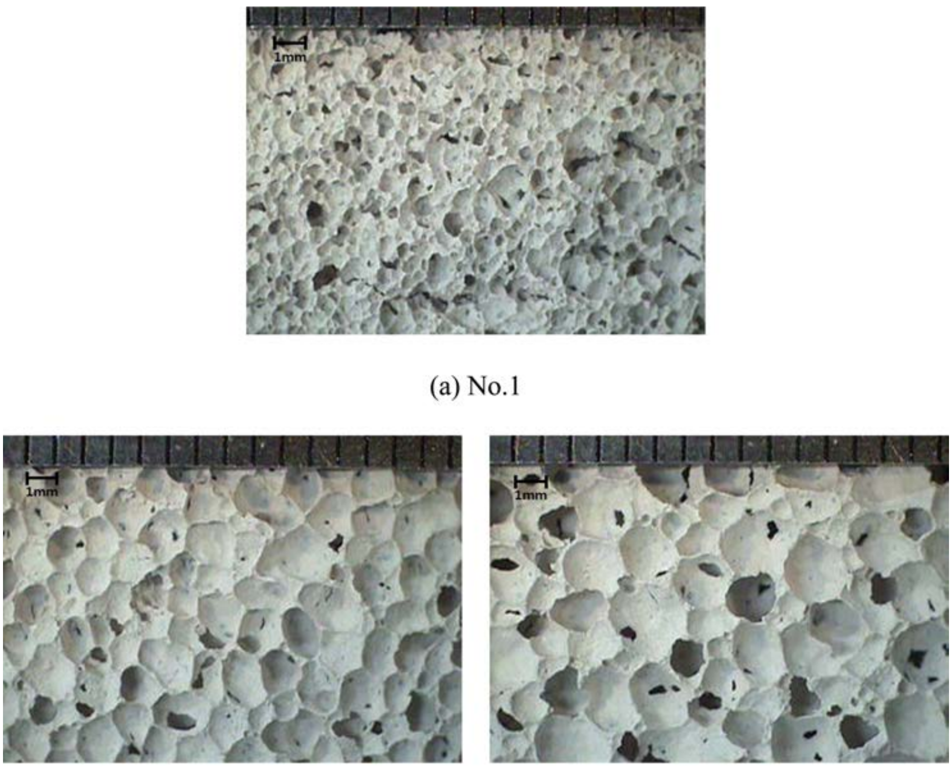
In consideration of the result of assessment of density of mineral hydrate insulation, the density was assessed as 0.13 ~ 0.27 g/cm^3. Additionally, the density of mineral hydrate insulation, using the activated cement with the fineness of 7,500 cm^2/g, showed the lowest amount of density, which was 0.13 g/cm^3. Namely, the higher degree of fineness of activated cement, the more frequent the tendency of lowering of density. It is estimated that the height of the formation of bubble of slurry is related to the fineness of cement. Generally, to manufacture mineral hydrate insulation, there must be high degree of contents of a blowing agent and mixing water. In case of increase of a blowing agent and mixing water, however, the slurry cannot be normally expanded and the phenomenon of backing, which means the slurry sinks down, or the separation of the slurry occurs during the process of expansion or the process of setting after the expansion. In this experiment, the backing phenomenon or the separation of the slurry didn't occur but showed the tendency for the height of bubble of the slurry to decrease as the degree of the fineness of activated cement was lower. In short, it is noticeable that the height of blowing of slurry and the density of insulation has very close correlation.

The compression strength of mineral hydrate insulation was assessed as 0.36 ~ 1.08 MPa. Additionally, as the



(a) Specific gravity (b) Compressive strength

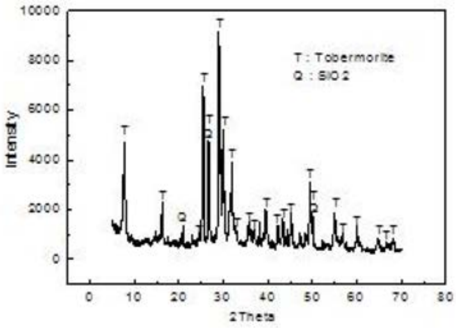
Fig. 2. Physical properties of mineral hydrate insulation in accordance with the fineness of activated cement.



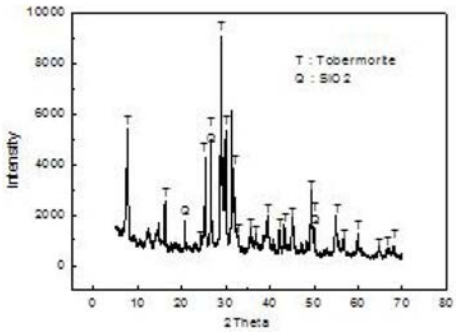
(a) No.1

(b) No.2 (c) No.3

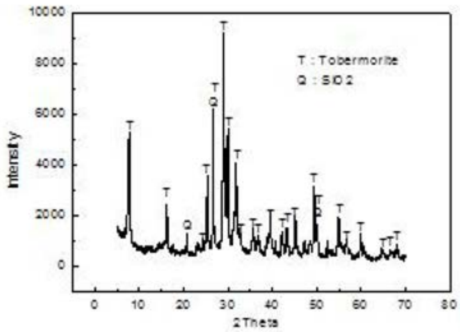
Fig. 3. Properties of pores of mineral hydrate insulation in accordance with degree of fineness of activated cement.



(a) No.1



(b) No.2



(c) No.3

Fig. 4. Properties of crystal of mineral hydrate insulation.

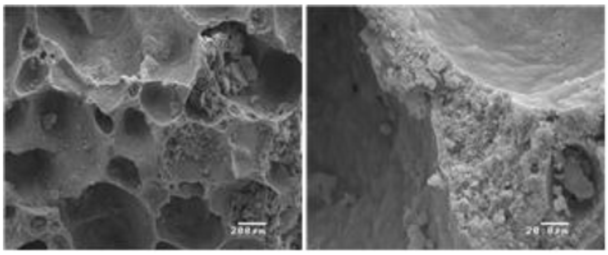
degree of fineness of activated cement increases, it is tended to have lower compression strength, and similar tendency has been observed in the result of assessment of density. That is, the compression strength of mineral hydrate insulation has very close correlation with density and the compression strength increases as the density increases.

By utilizing cam scope, the size of pores of mineral hydrate insulation was assessed and the result has been represented in Fig. 3. As the result of the assessment of the size of pores of each specimen, in case of the specimen with the inactivated OPC, the size was approximately 0.5 ~ 1.0 mm. For the size of specimen No. 2 and No. 3, with the activated cement, the range was approximately 0.5 ~ 2.0 mm and 1.0 ~ 2.0 mm respectively. Also, in all the specimens, connected pore, open pore, and close pore have been observed and it is considered that such closed pores performed to promote the insulation property [10].

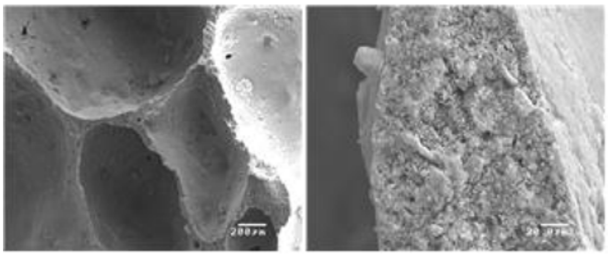
After the analysis of physical properties, the specimens were undergone through the analysis of crystalline by utilizing a X-ray diffractometer and the result can be found on the Fig. 4. As the result of the analysis of crystalline, the main crystal of all the specimens was tobermorite ($5CaO·6SiO_2·5H_2O$) and some of quartz peaks were detected as well. That is, it is considered that the reaction of hydrothermal synthesis has been satisfyingly performed in all the specimens. In terms of intensity of main peak of the crystal of tobermorite, however, showed the tendency to increase as the degree of fineness of activated cement had increased. It is deemed as the result of the active reaction of hydrothermal synthesis according to the increasing reactivity as the particle of cement gets smaller and smaller.

Fig. 5. shows SEM images of pores and matrix of mineral hydrate insulation. In case of the size of a pore of mineral hydrate insulation, specimen No. 3 has the largest one and specimen No. 1 shows the smallest one. It is the same result of the assessment by utilizing cam scope and it is expected to have enhancement effects of the properties of insulation because of the relatively big pores. By considering the density of matrix, it is detected that specimen No. 3 has the highest density of all. Namely, the reactivity grows as the activation degree of cement is higher and it will result in even more active hydrothermal synthesis to make the matrix even denser.

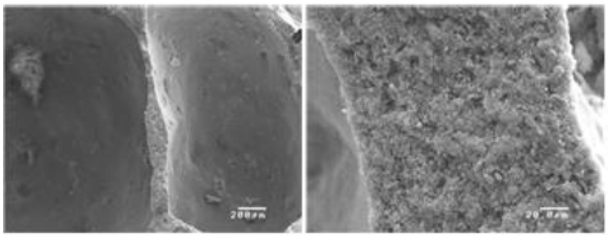
Fig. 6. shows the assessment of thermal conductivity with the application of heat flow meter by using the specimen with the measurement of 200*200*200 mm. The thermal conductivity of the mineral hydrate insulation is 0.044 ~ 0.102 W/m·K and specimen No. 3 showed the lowest value. It is the similar result, comparing to density properties, and it is determined that the increase of the degree of fineness of activated



(a) No.1



(b) No.2



(c) No.3

Fig. 5. Microstructure of mineral hydrate insulation.

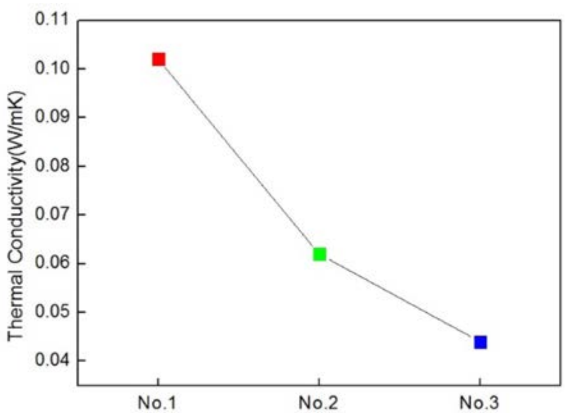


Fig. 6. Thermal conductivity of mineral hydrate insulation.

cement allows the average size of pores and height of bubble of slurry to increase as well so that the insulation property of the material becomes improved.

Conclusions

In this research, it has been aimed to apply to process of manufacturing mineral hydrate insulation after the manufacturing of activated cement by utilizing Pilot Plant activation system. In addition, to determine the properties of insulation for each degree of fineness of PP manufactured activated cement, activated cement with the degree of fineness of 5,000 cm^2/g and 7,500 cm^2/g were manufactured and the following result was drawn from the manufacturing mineral hydrate insulation.

1) As the result of analysis of crystal phase of mineral hydrate insulation, the main crystal is tobermorite and it is deemed that the reaction of hydrothermal synthesis has been favorably performed. Also, the tendency to have active hydrothermal synthesis in accordance with the higher reactivity as the particles of cement get smaller. It is detectable that the similar tendency has been shown in the result of observation of matrix with the usage of a scanning electron microscope.

2) The density of mineral hydrate insulation, with the activated cement with the degree of fineness of 7,500 cm^2/g, is 0.13 g/cm^3 and the tendency to have lower density, as the degree of fineness of activated cement increases, has been observed. Additionally, the range of the compression strength of mineral hydrate insulation is 0.36 ~ 1.08 MPa and it shows the same tendency just as the assessment result of density.

3) As a result of assessment of the size of pores of mineral hydrate insulation, in case of specimen with inactivated OPC shows the approximate range of 0.5~1.0 mm. The size of pores of specimen with activated cement presents the approximate range of 0.5~2.0 mm. The bigger the pores are, the greater thermal properties have been detected and it is possible to confirm it with the result of assessment of thermal conductivity. The thermal conductivity of mineral hydrate insulation with activated cement with the degree of fineness of 7,500 cm^2/g is 0.044 W/m·K, which is lower than the thermal conductivity of other specimens.

Based on the above-stated results, in case of manufacturing activated cement with the level of 7500 cm^2/g, it is considered possible to apply to the manufacturing process of mineral hydrate insulation.

Acknowledgments

The present study was carried out with the support of research fund from the Land, Infrastructure and Transport Technology Regional Specialization Project (15RDRPB103390-04), the Korea Ministry of Land, Infrastructure, and Transport. We would like to extend our sincere gratitude for the support.

References

1. H. Justnes, L. Elfgren, and V. Ronin, Cem. & Conc. Res. 35 (2005) 315-323.
2. H. Justnes, P. A. Dahl, V. Ronin, J. E. Jonasson and L.

Elfgren, Cem. & Conc. Comp. 29 (2007) 533-541.
3. F. Shi and W Xie, Miner. Eng. 70 (2015) 130-140.
4. F. Bellmann and J. Stark, Cem. & Conc. Res. 39 (2009) 644-650.
5. K. Sobolev, Cem. & Conc. Comp. 27 (2005) 848-853.
6. L. Hu and T. Yang, Int. J. Energy Eng. 3[1] (2013) 23-26.
7. K. Manohar, Brit. J. App. sci. & Tech. 2[3] (2012) 227-239.
8. O. Unal, T. Uygunoglu and A. Yildiz, Build. & Environ. 42[2] (2007) 584-590.
9. Z. Pan, H. Li and W. Liu, Constru. & Build. Mater. 72[15] (2014) 256-261.
10. H. Kurama, I. B. Topcu and C. Karakurt, J. Mater. Processing Tech. 209[2] (2009) 767-773.