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Fire resistance test for fire protection materials with high water content

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Abstract

It is known that moist fire protection materials show good fire resistance characteristics. For this reason, these materials are usually made of mixtures of cement mortar and high water content materials such as silica gels or moist perlites. The latent heat of water plays an important role in the resistance of heat propagation in these materials. In this study, a fire resistance test of a material with high water content is conducted and the temperature response of the test is obtained. Also, the water content of the test materials is measured. The test material consists of a mixture of perlite mortar and gel. The gel absorbs the aqueous solution of calcium chloride, which serves as a water storage mechanism. The numerical predictions to simulate the fire resistance test were conducted and the results were compared with the experimentally obtained temperature responses.

1. Introduction

It is well established in the literature that moist fire protection materials have good fire resistance characteristics. Therefore, these materials are usually made of mixtures of cement mortar and high water content materials such as silica gels or moist perlites. The latent heat of water in these materials plays an important role in the resistance of heat propagation. Thus, the walls made of these materials are expected to have good fire resistant characteristics. To investigate this, Jin et al. [1] developed a simple one-dimensional numerical model that predicts the thermal response of walls subject to fires. Using the proposed model, Jin et al. [2] conducted a parametric study to investigate the effect of water content on the thermal responses of the wall. Also, it was analytically confirmed that walls with high water content have good fire resistance characteristics.

The silica gel or the moist perlites are widely used as a water storage mechanism for fire protection materials. The water storage capacity of silica gel is less than 30% by mass. For moist perlites, it is quite difficult to hold water for long periods of time. A super-absorbent polymer gel, can absorb water 200–1000 times more than that of a dry material. However, the absorbed water is quickly evaporated, and the gel dries up in 1–2 days, depending on the ambient temperature or humidity. Therefore,

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Nomenclature

A	absorbency, (kg-solution/kg-polymer)
C	concentration, mass% (kg-$CaCl_2$/kg-solution)
Cp	specific heat, J/kg K
h	heat transfer coefficient at heated surface, W/m^2 K
K	thermal conductivity, W/m K
M	mass, kg
t	time, s or min
T	temperature, K
V	volume, m^3
W	water content, mass% (kg-water/kg)
ε	emissivity
ρ	density, kg/m^3
$\Delta\gamma$	latent heat of adsorbed and crystalline waters

Subscripts

0	value at the beginning of curing process
all	all components
c	cement
ca	calcium chloride
cp	cement paste
eff	effective value
fw	free water
gel	gel
H	heated surface
i	initial
l	region below 373.15 K
p	perlite
pm	perlite mortar
v	region above 373.15 K
w	water including free, adsorbed, and crystalline waters
∞	surrounding

to achieve an optimum fire protection material, research on materials of high water content that can retain water for a long time is required.

Calcium chloride solution has a characteristic of deliquescence. It is expected that a super absorbent polymer gel that absorbs such solution, can regulate its water content by absorbing or releasing water vapor from or to the atmosphere, until it reaches to an equilibrium state with its surrounding. These characteristics make super absorbent polymer gel a good candidate for use as water storage mechanism. Jin et al. [3] investigated the thermal and water content characteristics of an alcoxy-polyalkylen glycol group bridged super-absorbent polymer gel (Nihon Shokubai Co., CN-80M) that absorbs calcium chloride solution. It was found that the gel contains five times the water content of silica gel. Therefore, it was concluded that super-absorbent polymer gel could be used as a water storage mechanism in the fire protection materials.

In this study, fire resistance tests on a material of high water content were conducted, and the temperature response of this material was obtained. The test material was perlite mortar, mixed with gel to increase its water storage capacity. The water content increase was then measured. The numerical predictions for the fire resistance test were conducted and the results were compared with the experimentally obtained temperature responses.

2. Fire resistance test

2.1. Composition of a test material and curing condition

The perlite mortar is widely used as fire protection material. To increase the water content, it is mixed with the swollen super-absorbent polymer gel (CN-80M), which absorbs calcium chloride solution (C = 30% by mass for A = 15 (kg-solution/kg-polymer)). The water storage characteristic of swollen gel was reported by Jin et al. [3]. The dried CN-80M polymer is a powder that swells up to 1 mm in diameter when mixed with the calcium chloride solution. The composition of the fresh perlite mortar and the mixing volume ratio of gels and the fresh perlite mortar are tabulated in Table 1. The perlite used was the class "C". The mixed material was squeezed into equally sized rectangular frames of 210 mm × 210 mm × 60 mm to make two plates (No. 1 and No. 2). The weights of the plates are not exactly the same as that shown in Table 1. The mixed materials were cured in a thermo-hygrostat (Shimazu Physical and Chemical Appliances, HT30W) operated at 25°C and relative humidity of 80% for 21 days. The frame was removed after the second day of the curing process. The weight of both mixed materials were measured using an electric balance (A&D EK-12KA, minimum scale 1 g) and the water content was evaluated from the change in weight.

Table 1
Composition of test materials

	Test material		Perlite mortar	
	No. 1	No. 2	No. 1	No. 2
Cement	1.128 kg	1.142 kg	2.287 kg	2.335 kg
Water	0.598 kg	0.605 kg	1.212 kg	1.238 kg
M_w/M_c	0.53	0.53	0.53	0.53
Perlite	0.147 kg	0.148 kg	0.297 kg	0.304 kg
V_p/V_c	1	1	1	1
Gel (30 mass%)	1.517 kg	1.535 kg	–	–
V_{gel}/V_{pm}	1	1	–	–
Initial mass	3.390 kg	3.430 kg	3.797 kg	3.876 kg
Initial density	1280 kg/m^3	1296 kg/m^3	1435 kg/m^3	1465 kg/m^3

For comparisons, the fire resistance test for a regular perlite mortar that does not include the gel was also conducted. The composition of the perlite mortar is tabulated in Table 1.

2.2. Water content

The change in weight and water content of the test materials during the curing process are plotted in Fig. 1. The weights in the figure are normalized by the initial weight. The average weight of the two plates after the curing process was about 97% of the fresh material and the average water content of the test plates was about 45% by mass. The value of water content is the total amount of water in the material, which includes free water, adsorbed and crystalline water. This will be discussed later.

During the curing process, the cement reacts with the water to form calcium silicate hydrates (C-S-H) and the calcium hydroxide, $Ca(OH)_2$. This chemical reaction is called hydrate reaction. The water molecules in the fresh cement paste changes into the crystalline water as part of calcium hydroxide reaction. Some water molecules are also adsorbed on the surface of the crystals and remains in the cement mortar as physically adsorbed water. The remaining water in the cement paste evaporates. The super-absorbent polymer gel that absorbs calcium chloride solution, is known to absorb water vapor from, or releases it to the atmosphere, until it reaches to an equilibrium state. The water in the gel behaves like free water when heated. This is a desirable characteristic since the amount of mass fraction of free water in the test materials plays an important role in the resistance of the heat propagation (e.g. Jin et al. [2]). To measure this parameter, thermal analyzer (Seiko Densi Co., TG/DTA 300)

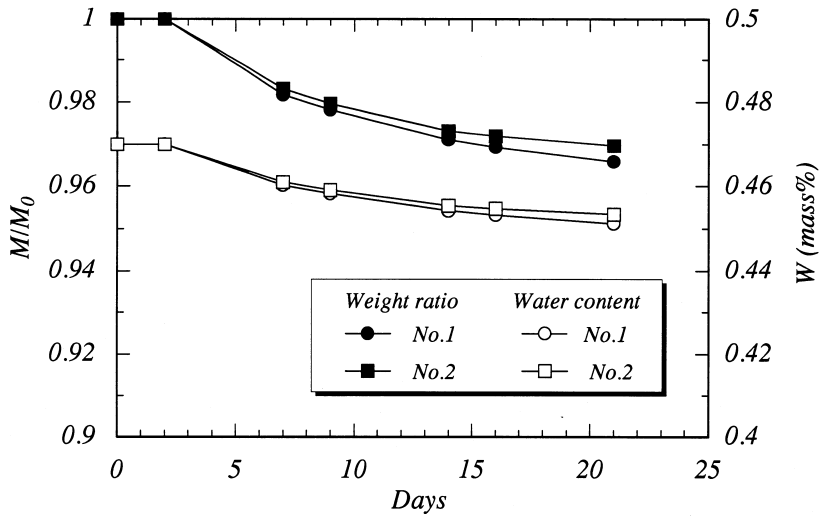

Fig. 1. Change in weight and water content of a test material in a curing process.

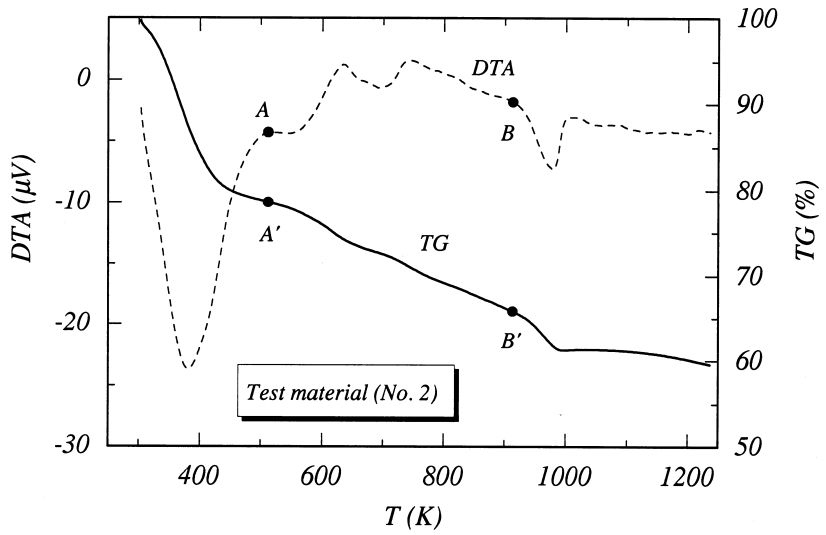

Fig. 2. Results of TG/DTA of a test material (No. 2).

such as the thermo-gravimetry (TG) and the differential thermal analysis (DTA) were used. The sample for the TG/DTA measurement was taken from the surface of the test plate. Since heating rate within the interval of 5–20 K/min does not affect the results of TG/DTA measurement, heating rate of 20 K/min was chosen.

The results of TG and DTA for the sample obtained from test plate No. 2 are plotted in Fig. 2. The solid line in the figure represents the result of TG and the dashed line represents the result of DTA. The endothermic response of the DTA curve from the room temperature to 513 K (point A) is due to the evaporation of the free water. The mass fraction of the free water is evaluated as 21.4% from the TG curve. Several endothermic reactions and the mass reductions can be seen between 513 and 913 K in the figure. In the cement mortar, the decomposition of crystalline water begins at about 513 K and the water decomposition of the calcium hydroxide, $Ca(OH)_2$, occurs at about 773 K. Therefore, the mass reduction between 513 and 913 K (point A to point B), is due to the evaporation of the adsorbed water and the decomposition of the crystalline water and the water decomposition of the calcium hydroxide, $Ca(OH)_2$. The endothermic reaction near 973 K is due to the gas decomposition of $CaCO_3$. The mass fraction of the water

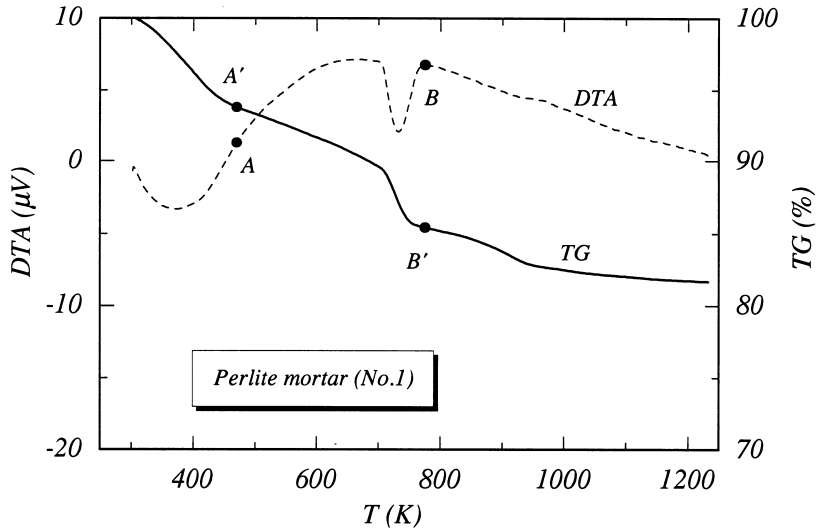

Fig. 3. Results of TG/DTA of a perlite mortar (No. 1).

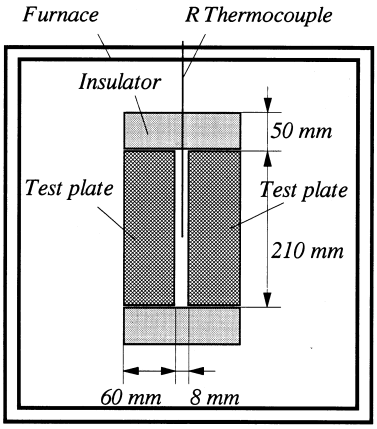

Fig. 4. Experimental setup for fire resistant test.

is 13.0%. Therefore, the ratio of the free water to the total water is 21.4/(21.4 + 13.0) = 62.2% and the ratio of the other waters to the total water is 13.0/(21.4 + 13.0) = 37.8%. Note that these ratios are the results from the surface sample of the test material. However, it is assumed that these ratios are uniform throughout the test plate, so that the average mass fractions of the free water in the test plate will be evaluated from the total water content obtained by the change in weight to be 45%. The evaluated average mass fractions of the free water and the other waters in the test material of plate No. 1 are 28.1% and 17.0%, respectively. For test material of plate No. 2, the same are found to be 28.2% and 17.1%, respectively.

For comparisons with the regular perlite mortar, which does not include the gels, the average mass fraction of the free water in the perlite mortar of V_p/V_c = 1, were evaluated in the same way. The average water content of the perlite mortar after the curing process was 24.3% (by mass). The results of the TG/DTA for the regular perlite mortar are plotted in Fig. 3. The average mass fraction of the free water and the other waters in the regular perlite mortar are 10.2% and 14.1%, respectively. By comparison, it was determined that the average mass fraction of the free water could be increased 18% by mixing the gels.

2.3. Fire resistance test

A schematic diagram of the experimental setup for the fire resistance test is shown in Fig. 4. Two test plates of 210 mm × 210 mm × 60 mm were placed 8 mm apart. The 8 mm gap was filled with ceramic fibers. Calcium-silicate insulators of 50 mm thick and density of 150 kg/m^3 were used to insulate the peripheral of the two test plates. A steel box of 1 mm thick covered the two insulated plates, and the sandwiched ceramic fibers. The box was then set in an electric furnace (Yamato Labotech, FE-100K). The temperature of the furnace was controlled to follow the standard heating curve defined by JIS (Japanese Industrial Standard) S1037. The air temperature in 8 mm gap was measured by R type thermo-couple of 0.65 mm ϕ in 1 min intervals.

2.4. Temperature response of test material

A photograph of the test material after the fire re-

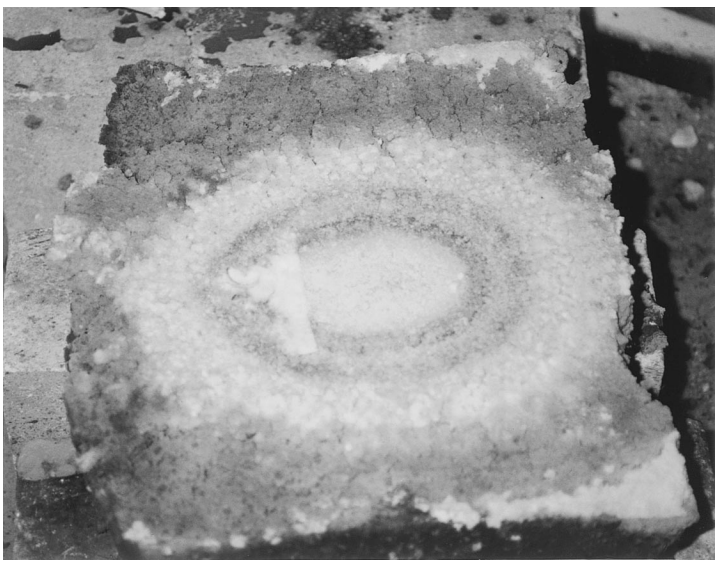

Fig. 5. Picture of the test material after the fire resistance test.

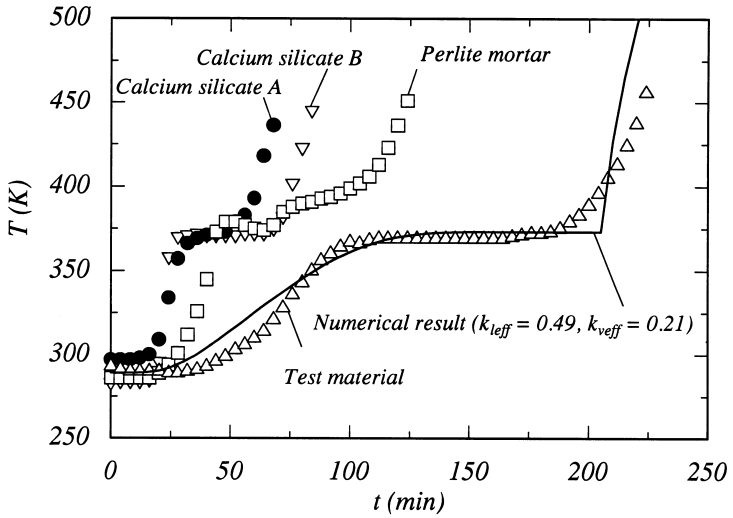

Fig. 6. Temperature responses.

sistance test is presented in Fig. 5. The picture was taken on the side facing the 8 mm gap. The outer gray ring on the picture is the location where the gas decomposition occurred. Such decomposition suggests that the temperature exceeded 973 K during the fire resistance test. The small ring near the center is due to the carbonization of the polymer. The temperature responses in 8 mm gap for the test plate and the perlite mortar are plotted in Fig. 6. The temperature responses for our previous results for the calcium silicate boards A and B conducted by Jin et al. [1] are also plotted in the figure.

The temperature at the 8 mm gap rises and levels off as it reaches 373.15 K. For test plate, it takes about 100 min to reach to 373.15 K. This period is quite long comparing with the other materials. It also takes about 90 min to keep it at 373.15 K. This period is also quite long. The temperature rises again after 185 min from the beginning of the heating. The temperature at the 8 mm gap stays below 450 K (177°C) for a period of 223 min. Overall, the fire resistance time of the test material is 223 min. From this result, it can be concluded that the test material has an excellent fire resistance characteristics comparing with the other fire protection materials.

3. Comparison with numerical result

3.1. Conservation equations and boundary conditions

Governing equations to be considered are the continuity, velocity and energy equations. The flow of vapor in the vapor region is approximated by Darcy's model. The energy equation for the phase change problem was solved using enthalpy method proposed by Cao et al. [4]. If constant thermophysical properties in each region are assumed, the governing equations take the following form [1].

$$\frac{\mathrm{d}(\rho_{\mathrm{v}}U)}{dX}=0 \tag{1}$$

$$0=-\frac{\mathrm{d}P}{\mathrm{d}X}-\frac{v}{k}\rho_{\mathrm{v}}U \tag{2}$$

$$\frac{\partial(\rho_{\mathrm{eff}}E_{\mathrm{eff}})}{\partial t}+\frac{\partial\left(\rho_v\frac{Cp_{\mathrm{v}}}{Cp_{\mathrm{eff}}}UE_{\mathrm{eff}}\right)}{\partial X}=\frac{\partial^2(\Gamma E_{\mathrm{eff}})}{\partial X^2}+\frac{\partial^2 S}{\partial X^2} \tag{3}$$

where

$$\Gamma(E_{\mathrm{eff}})=\begin{cases}K_{\mathrm{veff}}/Cp_{\mathrm{veff}} & E_{\mathrm{eff}}>0\\ 0 & -\gamma_{\mathrm{eff}}\le E_{\mathrm{eff}}\le 0\\ K_{\mathrm{leff}}/Cp_{\mathrm{leff}} & E_{\mathrm{eff}}<-\gamma_{\mathrm{eff}}\end{cases} \tag{4}$$

$$S(E_{\mathrm{eff}})=\begin{cases}0 & E_{\mathrm{eff}}>0\\ 0 & -\gamma_{\mathrm{eff}}\le E_{\mathrm{eff}}\le 0\\ K_{\mathrm{leff}}\gamma_{\mathrm{eff}}/Cp_{\mathrm{leff}} & E_{\mathrm{eff}}<-\gamma_{\mathrm{eff}}\end{cases} \tag{5}$$

above E_{eff} is the effective enthalpy of the test material. E_{eff} equals to zero for the situation where the pours are filled with vapor at saturated temperature. K_{eff}, Cp_{eff} and γ_{eff} are the effective thremal conductivity, the effective specific heat, and the effective latent heat of the free water, respectively.

The wall is initially kept at temperature T_{i}. Thus, the initial conditions are

Table 2
Thermophysical properties used for computation

Temperature range	ρ_{eff} (kg/m^3)	Cp_{eff} (J/kg K)	K_{eff} (W/m K)
Up to 373.15 K	1271	1879	0.49
373.15–913.15 K	1089–0.4706T	$\frac{908300}{1089-0.4706T}+1056$	0.21
913.15 K onwards	659.3	1056	0.21

$$t<0: \qquad T=T_i, \quad U=0 \tag{6}$$

At time $t = 0$, the surface of the wall which faces to the furnace is exposed to the fire flame and the surface is heated by convection and radiation. The surface in front of the 8 mm air gap, can be assumed to be adiabatic. The boundary condition then becomes;

Heated surface:

$$q_{\text{H}} = h_{\text{H}}(T_\infty - T_{\text{H}}) + \varepsilon_{\text{H}}\sigma\left(T_\infty^4 - T_{\text{H}}^4\right) \tag{7}$$

Back surface: $$\mathrm{d}T/\mathrm{d}X = 0 \tag{8}$$

where q_{H} is the heat flux on the heated surface and T_∞ is the ambient temperature in the furnace.

3.2. Properties of test material

The model for the simulation of the fire resistance test proposed by Jin et al. [1] was adopted for the numerical prediction. The thermal properties used for the computation are tabulated in Table 2. The effective density below 373.15 K, ρ_{leff}, is the average value of the two test plates, No. 1 and No. 2 calculated from the measured mass and volume. The test material might have lost most of its water content by the time it reaches 913 K. Beyond this temperature, only the cement, the perlite and the calcium chloride remain in the test plate. The effective densities of the test plate for this condition were calculated and are listed in Table 2. Linear approximation of effective density for temperatures between 373 and 913 K are also obtained.

The effective value of the specific heat below 373.15 K was calculated using mass fractions of the cement paste, the perlite and gels as

$$Cp_{\text{leff}} = Cp_{\text{cp}}\frac{M_{\text{cp}}}{M_{\text{all}}} + Cp_{\text{p}}\frac{M_{\text{p}}}{M_{\text{all}}} + Cp_{\text{gel}}\frac{M_{\text{gel}}}{M_{\text{all}}} \tag{9}$$

where Cp_{cp}, Cp_{p} and Cp_{gel} are the specific heat of the components. Their values [5] are $Cp_{\text{cp}} = 1100$, $Cp_{\text{p}} = 1000$ and $Cp_{\text{gel}} = 2790$ J/kg K, respectively. The estimated mass fraction of the test plate after the curing process is listed in Table 3.

The effective value of the specific heat over 913 K was evaluated under the same assumption as with effective density. Namely, the test plates are assumed to consist of only the cement, the perlite and the calcium chloride. Then, the effective value of the specific heat can be expressed as,

$$Cp_{\text{veff}} = Cp_{\text{c}}\frac{M_{\text{c}}}{M_{\text{all}}} + Cp_{\text{p}}\frac{M_{\text{p}}}{M_{\text{all}}} + Cp_{\text{ca}}\frac{M_{\text{ca}}}{M_{\text{all}}} \tag{10}$$

where Cp_{c}, Cp_{p} and Cp_{ca} are the specific heats of each component. Their values [5] are $Cp_{\text{c}} = 1100$, $Cp_{\text{p}} = 1000$ and $Cp_{\text{ca}} = 958$ J/kg K, respectively.

The latent heat of the free water at 373.15 K under the atmospheric pressure, 2256.9 kJ/kg, is used for both endothermic values of the adsorbed water, the crystalline water and the decomposition of the calcium hydroxide. Therefore, the effective latent heat can be expressed as

$$\Delta\gamma = 2256.9 \times \frac{M_{\text{w}} - M_{\text{fw}}}{M_{\text{all}}} \text{ kJ/kg} \tag{11}$$

Under the assumption that the evaporation of the adsorbed water, the decomposition of the crystalline water and the water decomposition of the calcium hydroxide occur continuously from 373.15 K to T_{B} (913 K), the absorbed heat are included in the effective specific heat as

$$Cp_{\text{veff}} = \frac{\left\{\Delta\gamma/(T_{\text{B}} - 373.15)\right\}\rho_{\text{leff}}}{\rho_{\text{eff}}} + 1056 \qquad 373.15 < T < T_{\text{B}} \tag{12}$$

The effective thermal conductivity for the test plate was measured using the hot-wire probe by Kyoto Electronics Manufacturing Co., PD-31. The effective thermal conductivity of the test plate measured at 298.15 K is 0.49 W/m K. The measured values ranges from 0.20 to 0.22 W/m K for the temperature range of

Table 3
Mass fraction of test materials after curing process

	No. 1	No. 2
$M_{\text{cp}}/M_{\text{all}}$	0.492	0.494
$M_{\text{p}}/M_{\text{all}}$	0.045	0.044
$M_{\text{gel}}/M_{\text{all}}$	0.463	0.462

Table 4
Composition of fresh perlite mortars and volume ratios of gels and fresh perlite mortar

Sample No.	Composition of fresh perlite mortar		Volume ratio of gels and fresh perlite mortar (V_{gel}/V_{pm})
	Perlite/cement ratio (V_p/V_c)	Water/cement ratio (M_w/M_c)	
1	1	0.53	0.5
2	1	0.53	1
3	1	0.53	1.5
4	1	0.53	2
5	2	0.53	0.5
6	2	0.53	1
7	2	0.53	1.5
8	2	0.53	2
9	3	0.7	0.5
10	3	0.7	1
11	3	0.7	1.5
12	3	0.7	2

573.15 K < T < 723.15 K. The value of K_{leff} = 0.49 W/m K was used in the computation, to represent the effective thermal conductivity below 373.15 K. For temperature values above 373.15 K, K_{veff} = 0.21 was used.

3.3. Numerical results

The computation was conducted for the heat transfer coefficient h_H = 50 W/m^2 K and for the emissivity ε_H = 0.9 for the heated surface, and insulated boundary condition for the opposite surface. The same values were used in the paper by Huang et al. [6]. However, some researchers (e.g., Sultan et al., [7]) adopted h_H = 20–30 W/m^2 K for the computation instead of 50 W/m^2 K. The surrounding temperature T_∞, that was calculated from Eq. (13) was also used in the computation. It is noteworthy that Eq. (13) is an approximation of the standard heating curve.

$$T_\infty = 1353 - 340e^{-0.8t/3600} - 130e^{-5t/3600} - 610e^{-19t/3600} \quad (13)$$

The initial temperature of the test plate in this test was 289.15 K. While the calculated temperature of T_∞ from Eq. (13) was lower than the initial temperature

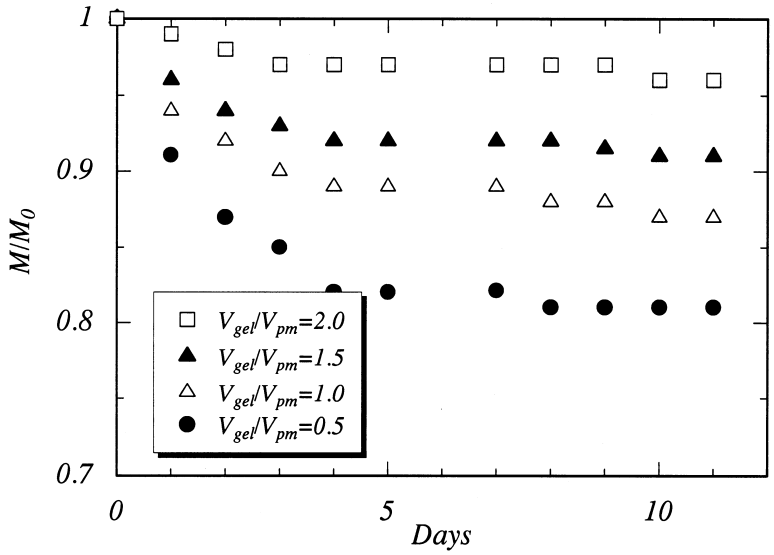

Fig. 7. Change in weight of test materials in a curing process.

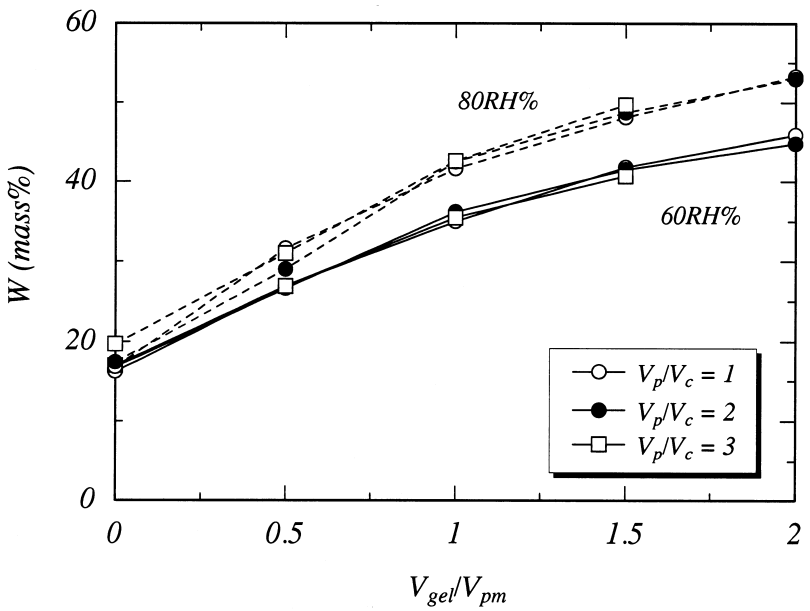

Fig. 8. Effect of mixing ratio, V_{gel}/V_{pm}, on water content.

T_i, $T_\infty = T_i$ was assumed. The results are plotted in Fig. 6. The solid line in the figure represents the numerical result. As seen in the figure, a discrepancy between the numerical and experimental results can be seen at the beginning of heating. However, the time for temperature rise from 373.15 K, almost coincides with the numerical prediction.

4. Composition and water content

From the experimental results above, it could be concluded that the fire resistance characteristics improve with increasing water content. The composition of the tested material was perlite/cement ratio, $V_p/V_c = 1$, and the mixing ratio of the gels/perlite mortar, $V_{gel}/V_{pm} = 1$. It is expected that the water content may change with changing the composition of the material. The correlation between the water content and the composition was also investigated.

4.1. Composition of test materials and curing conditions

The tested materials are mixtures of the same swollen super-absorbent polymer gel (CN-80M), which absorb calcium chloride solution ($C = 30$ mass% for $A = 15$ kg-solution/kg-polymer), and the perlite mortar. The composition of fresh perlite mortars, the mixing ratio of gels and the fresh perlite mortar is tabulated in Table 4. Three perlite mortars with different perlite/cement ratios (V_p/V_c) were prepared. Four mixing ratios of gels and fresh perlite mortar were tested. Therefore, the water content of 12 samples was investigated. These samples were squeezed into petri dishes of 14.7 mm in depth, and 48.7 mm in diameter. The samples were cured in the thermo-hygrostat, operated at 25°C and relative humidity of 80% for 12 days and the change in weight was measured. After the curing process, the relative humidity in the thermo-hygrostat was changed to 60% and the water content under the under this condition was measured.

4.2. Water content

The change-in-weight of the samples No. 1 through 4 during the curing process are plotted in Fig. 7. The weights are normalized by the initial weight of the fresh material. As seen in the figure, the weight decreases in the first few days and levels off after 10 days. This trend is accentuated for sample No. 1 whose mixing ratio of the gels/perlite mortar is $V_{gel}/V_{pm} = 0.5$. The water contents of the samples, which are obtained from the change in weight, are plotted in Fig. 8 with the perlite/cement ratio (V_p/V_c) as the curve parameter. As seen in this figure, the water content increases with increasing mixing ratio of the gels/perlite mortar (V_{gel}/V_{pm}). However, the effect of the perlite/cement ratio (V_p/V_c) on the water content is very little. As expected, the water content for the relative humidity of 60% is lower than that for 80%. This trend is accentuated for materials whose mixing ratios of the gels/perlite mortar (V_{gel}/V_{pm}) are high.

5. Concluding remarks

1. The mass fraction of free water in the perlite mortar is about 10%. However, the mass fraction of free water in the test material reaches to 28%. The water content of free water increases by 18% when mixed with gel.
2. The fire resistance time for 450 K (177°C) of the tested plate is 224 min. This period is extremely long comparing with the other materials.
3. The temperature response predicted by the numerical calculation agrees well with the experimental temperature results.

Acknowledgements

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