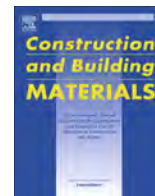




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Technical note

## Influence of dry ice on the performance of Portland cement and its mechanism

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### HIGHLIGHTS

- The standard consistency and the setting time of dry ice on Portland cement were studied.
- The dry ice can better improve the compressive strength of Portland cement paste at 28 days.
- The dry ice retards the early hydration of Portland cement and improves the later hydration.
- Results provide a reference for future application of dry ice in Portland cement-based materials.

### ARTICLE INFO

#### Article history:

Received 6 June 2018

Received in revised form 7 August 2018

Accepted 17 August 2018

#### Keywords:

Dry ice

Portland cement

Hydration

Setting time

Compressive strength

### ABSTRACT

This study reports on the effect of dry ice on the hydration and hardening behavior of Portland cement. Dry ice was directly added to fresh Portland cement paste, with its dosage fixed at 0 wt%, 0.3 wt%, 0.6 wt%, 0.9 wt%, 1.2 wt% and 1.5 wt%. Results showed that dry ice had little effect on the standard consistency and setting time of cement paste when its dosage was less than 0.9 wt%, but otherwise the standard consistency as well as the setting time was increased. The compressive strength of the paste at 7 days increased slightly when the dosage of dry ice was less than 0.9 wt%, but the compressive strength was reduced with the addition of 0.9 wt% to 1.5 wt% dry ice. For the compressive strength at 28 days, the cement paste with 0.6 wt% dry ice showed the greatest value, being increased by 30.9% compared to the control cement paste, and the compressive strength of the samples with dry ice was all higher than that of the control. In addition, hydration heat, XRD, DTA-TG and SEM results showed that the incorporation of dry ice retarded the early hydration of Portland cement, improved its later hydration, and optimized the hardened structure of the cement paste. These results can provide a reference for the future application of dry ice in Portland cement-based materials.

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## 1. Introduction

In order to reduce environment impact of CO<sub>2</sub>, a number of measures have been taken, such as reducing CO<sub>2</sub> emission [1,2] and sequestering CO<sub>2</sub> by using cementitious materials [3]. However, the construction industry annually consumes 4.3 billion tons of ordinary Portland cement (OPC) for making concrete, resulting in a great amount of CO<sub>2</sub> emission that accounts for around 7% of global CO<sub>2</sub> emissions. To reduce carbon footprint of cement industry, a number of studies focused on developing low-carbon cement [4–7]. In addition, other relevant research has been performed, which included CO<sub>2</sub> curing [8–13], CO<sub>2</sub> strengthening recycled aggregates [14–19], as well as the transformation of carbon dioxide

to carbonate as chemical additives [20–22]. X.Y. Pan [11] studied the strength and permeability of CO<sub>2</sub> surface treatment on one-day age of cement mortar. After 24 h of CO<sub>2</sub> treatment, the results showed that the compressive strength was increased slightly, the impermeability of cementing material was improved, while water absorption was reduced by 15–30%. KOU [23] studied the effect of carbonization on recycled aggregates, and their results showed that CO<sub>2</sub> curing increased the physical properties of recycled aggregates: the longer the curing time, the better the degree of carbonation and the higher the quality of aggregate. W. Kunther [24] investigated the effect of bicarbonate ions on the deterioration of mortar bars in sulphate solutions and found that the presence of bicarbonate ions significantly reduced mortar swelling. J.G. Jang [25] studied the effect of sodium bicarbonate on the performance of cement slurry, and they found that the addition of NaHCO<sub>3</sub> caused the internal carbonation of cement slurry, resulting in the consumption of Ca(OH)<sub>2</sub>. Besides, the compressive strength was

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increased with the addition of 5% NaHCO<sub>3</sub>; however, the strength degraded for higher concentrations. Rodger [26] found that Li<sub>2</sub>CO<sub>3</sub> accelerated the early hydration rate of sulphoaluminate cement. Other studies [27–29] found that lithium carbonate significantly shortened the setting time of sulphoaluminate cement, and improved its early compressive strength and flexural strength. Although there have been a number of studies on the influence of CO<sub>2</sub> curing or carbonate salts on hydration and hardening of Portland cement, little research has been carried out on how dry ice, as an alternative form of CO<sub>2</sub>, affects the process of Portland cement hydration.

This study investigates the effect of dry ice on the hydration kinetics, compressive strength, chemical compositions, and microstructure of Portland cement paste.

1.1. Raw materials

Ordinary Portland cement 42.5 grade (conforming to Chinese national standard GB175-2007) [30] was used. The main properties and chemical composition of cement were listed in Tables 1 and 2, respectively. Dry ice was crumbled, and its content of carbon dioxide was 99.99%. Tap water was used throughout this study.

1.2. Testing methods

The procedure for preparing cement paste is as follows: First, cement and water were weighed, and then the crumbled dry ice was weighed. To prevent the volatilization of dry ice, cement was immediately added into agitating pan and thereafter dry ice was added. Finally, water was added to the mixture which was first left for stirring at a slow speed for 90 s and then subjected to a fast stirring for 90 s after an interval of 30 s stop.

Standard consistency and setting time of Portland cement were measured according to Chinese National standard GB/T1346-2011 “cement standard consistency water, setting time, stability test method” [31]. Compressive strength was tested by using 40 mm × 40 mm × 40 mm cubic samples after subjected to standard curing for 7 and 28 days.

The exothermal curves were achieved using the Heat conduction calorimetry and SETARAM hydration exothermal analyzer. 500 mg powder was used and the water-to-powder ratio was 1:1. The exothermal curves were recorded continuously for 30 h. X-ray diffraction analysis (XRD) was used to identify the crystalline components of hydration products at different ages. Diffraction patterns were collected between 5° and 70° with a step size of 0.02°. After hydrated cement paste was taken out from the standard curing room at the corresponding age, and its hydration was terminated by anhydrous ethanol. The sample was dried in a vacuum drying oven at 40 °C, and then ground and passed through a 75 μm sieve before it was tested. Jade 5.0 software with the Powder Diffraction File database was employed to elucidate the mineralogy of the samples based on the diffraction patterns. Differential Thermal Analysis (DTA) was carried out under Ar atmosphere using HCT-3 instrument at 10 °C/min up to 800 °C. The samples that were also dried at 40 °C in the vacuum drying oven were used to study the shape and microstructure of the hydration products with 250FEG SEM.

Table 2  
Chemical composition of cement/%.

SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Loss
21.79	63.09	5.36	3.35	2.73	0.34	0.21	0.16

2. Results and discussion

2.1. Performance of cement paste

2.1.1. Standard consistency and setting time

The influence of dry ice on the standard consistency and setting time of cement was studied when dry ice was mixed into the cement at dosages of 0 wt%, 0.3 wt%, 0.6 wt%, 0.9 wt%, 1.2 wt% and 1.5 wt% by the weight of cement, and testing results are shown in Figs. 1 and 2, respectively.

It can be seen from Fig. 1 that the standard consistency first decreases but it then increases as dry ice content increases from

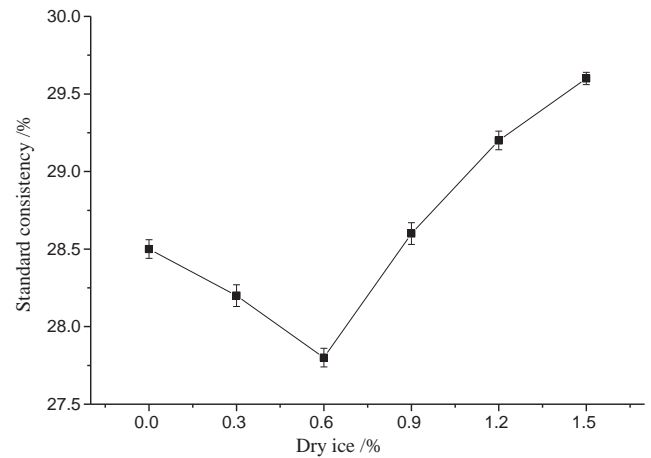


Fig. 1. Effect of dry ice on the standard consistency.

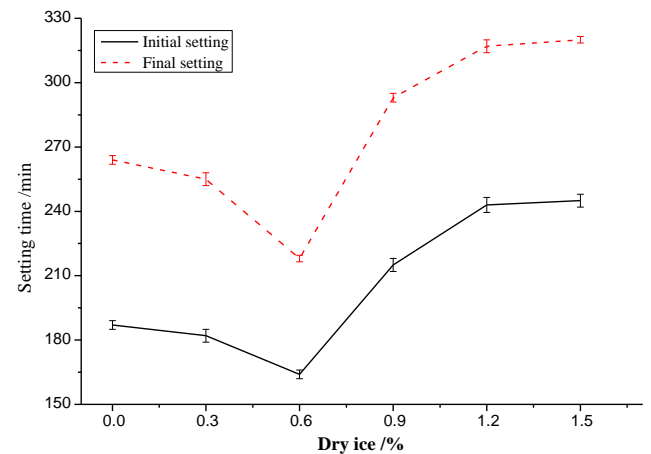


Fig. 2. Effect of dry ice on the setting time.

Table 1  
Main properties of P.O42.5.

Cement	Finen-ess/%	Stability	Setting time/min		Flexural strength/MPa		compressive strength/MPa	
			initial setting	final setting	3d	28d	3d	28d
P.O42.5	1.5	qualified	187	392	5.5	9.6	33.5	50.8

0 wt% to 1.5 wt%. At 0.6 wt%, the standard consistency was amongst the smallest. Compared to the control sample, when the dosage of dry ice was less than 0.9 wt%, dry ice reduced the standard consistency, while when it exceeded 0.9 wt%, dry ice increased the standard consistency. The standard consistency was increased by 3.86% when it went up to 1.5 wt%.

As can be seen from Fig. 2, the effect of dry ice on the setting time of cement was consistent with its effect on the standard consistency. Both the initial setting time and the final setting time first decreased but then increased. When the dry ice dosage was 0.6 wt%, the setting time was the shortest, which means that compared to the control sample, the initial setting time and the final setting time were reduced by 12.3% and 17.4% respectively. When the dosage of dry ice was increased from 0.6 wt% to 0.9 wt%, the setting time had a sharp increase, but when its dosage continued to increase, the increase rate of setting time became slow.

### 2.1.2. Compressive strength

The effect of dry ice on the compressive strength of hardened cement paste at the ages of 7, 28 days was studied. The water cement ratio was fixed at 0.4. The dosage of dry ice was 0 wt%, 0.3 wt%, 0.6 wt%, 0.9 wt%, 1.2 wt% and 1.5 wt%. The results were plotted in Fig. 3.

As shown in Fig. 3, as the dosage of dry ice increases, the compressive strength of cement paste at 7 days first increased little and then decreased. At the dosage of 0.6 wt%, the compressive strength reached the maximum, being increased by 7.2% compared to the control cement paste. When its dosage exceeded 0.9 wt%, the compressive strength at 7 days decreased. Likewise, as the dosage of dry ice increased, the compressive strength of cement paste at 28 days showed a similar change trend as that of cement paste at 7 days; first increased but then decreased. When the dosage of dry ice was 0.6 wt%, the compressive strength of cement paste reached the maximum value with an increase of 30.9% compared to the control sample. Furthermore, even when the dosage of dry ice was 1.5 wt%, the compressive strength of cement paste at 28 days was higher than that of the control one. These results indicate that dry ice had little effect on the early compressive strength of Portland cement paste, but it was beneficial to the compressive strength at the later stage, especially when the dry ice content was 0.6 wt%, the effect was very obvious.

## 2.2. Characterization

According to the above analysis, the setting time and compressive strength of Portland cement paste were changed when dry ice

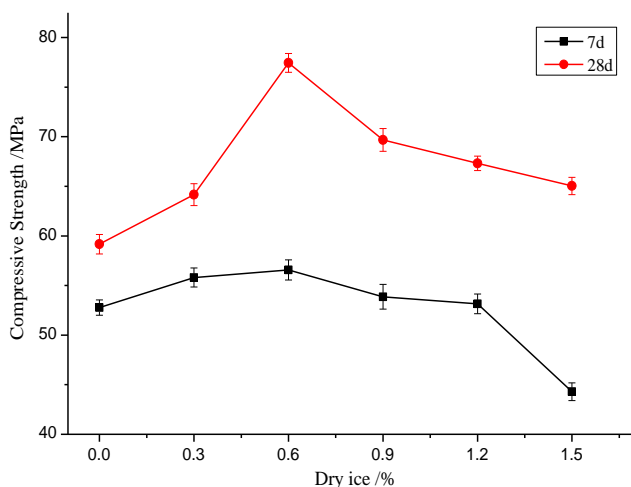


Fig. 3. Effect of dry ice on the compressive strength of cement paste.

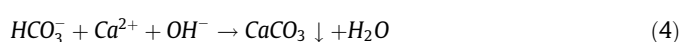
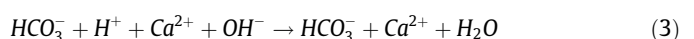
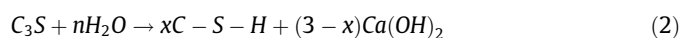
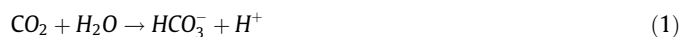
was present in the paste, especially for 0.6 wt%~0.9 wt% dry ice. To better understand the mechanism controlling the effect of dry ice on the hydration process of Portland cement, a series of microscopic tests were performed.

### 2.2.1. Hydration heat test

It can be seen from Fig. 4(a), the value of first peak is 92.452mW, 106.313mW, 119.493mW, 122.402mW, 103.880mW and 93.906mW when the content of dry ice increased from 0 wt% to 1.5 wt%, respectively, indicating that with the increase of dry ice content, the value of first exothermic peak increases and then decreases, and the first peak is greater when the content of dry ice is 0.6 wt%~0.9 wt%. The first exothermic peak can be attributed to the formation of ettringite (Aft) [32,33], so the results suggest that dry ice accelerates the early formation of Aft, especially when its content is 0.6 wt%~0.9 wt%, which is also consistent with the results of standard consistency, initial setting time and compressive strength.

It can be seen from Fig. 4(b) that the value of the second peak is 2.564 mW, 2.343 mW, 2.407 mW, 2.389 mW, 2.168 mW and 2.155 mW when the content of dry ice increases from 0 wt% to 1.5 wt%, respectively. The second peak can be assigned to the hydration of tricalcium silicate ( $C_3S$ ), and the value of second peak gradually reduces as the content of dry ice increases, which indicates that the addition of dry ice may retard the early hydration of  $C_3S$ , moreover, the more the content, the more obvious the effect. A peak shoulder appears after almost 17 h behind the second exothermic resulting from the conversion of Aft to calcium monosulfoaluminate (AFm), and the peak shoulder becomes much smaller as the content of dry ice increases. This observation shows that dry ice can prevent the transformation of Aft to AFm to some extent.

When dry ice dissolves in water, the chemical reaction shown in Eq. (1) occurs. The dissolution of dry ice increases the acidity of the solution, which results in an increase of the solubility of gypsum, thus improving the early formation of Aft; however, too much dry ice can cause too much acidity of aqueous solution, which is unfavorable for the early formation of Aft. Accordingly, the hydration heat of Aft shows a trend of decreasing after increasing. Calcium hydroxide will be produced when  $C_3S$  hydrates (Eq. (2)), and at the same time bicarbonate radical ( $HCO_3^-$ ) and calcium ion ( $Ca^{2+}$ ) (Eq. (3)) are generated. As the hydration of  $C_3S$  continues, the increasing amount of  $Ca(OH)_2$  will result in generation of calcium carbonate (Eq. (4)) that could cover the surface of  $C_3S$ . Both the increment of  $Ca^{2+}$  and the cover of  $CaCO_3$  retard the early hydration of  $C_3S$ . It can be seen in Fig. 4 that the generation peak of C-S-H and  $Ca(OH)_2$  gradually reduces as the dosage of dry age increases. At the same time,  $CaCO_3$  can react with hydrated calcium aluminate (CAH) to form hydrated calcium carboaluminate ( $C_3A-CaCO_3 \cdot 11H_2O$ ), which could prevent Aft transforming into AFm [33]. This result is consistent with the results of the third exothermic peak.



### 2.2.2. DTA results

DTA-TG is used to investigate the effect of dry ice on cement hydration products. The results are shown in Fig. 5. From Fig. 5, we can see that DTA-TG mainly includes three endothermic peaks

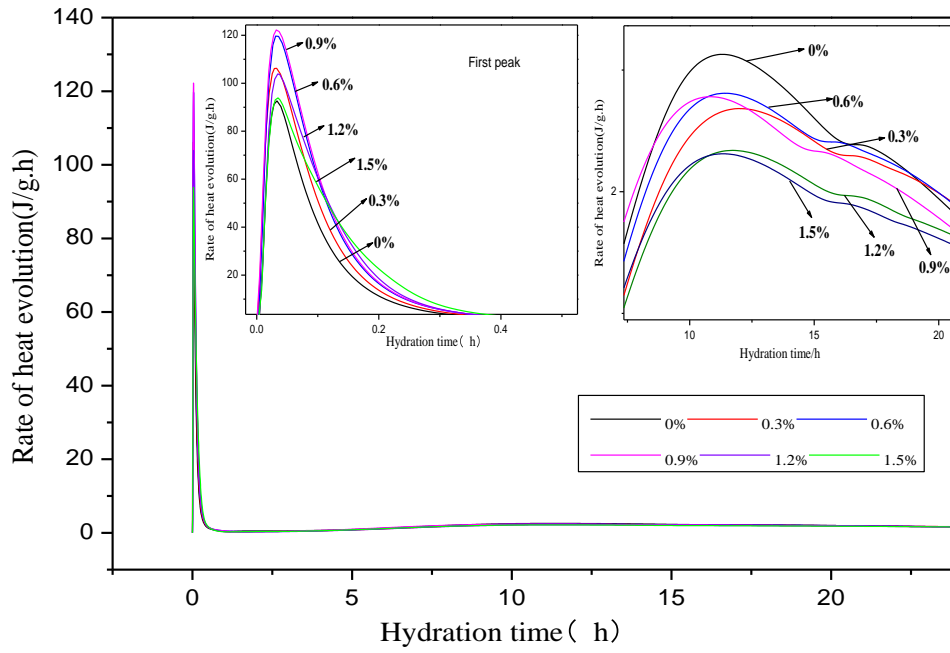


Fig. 4. Hydration heat release curve.

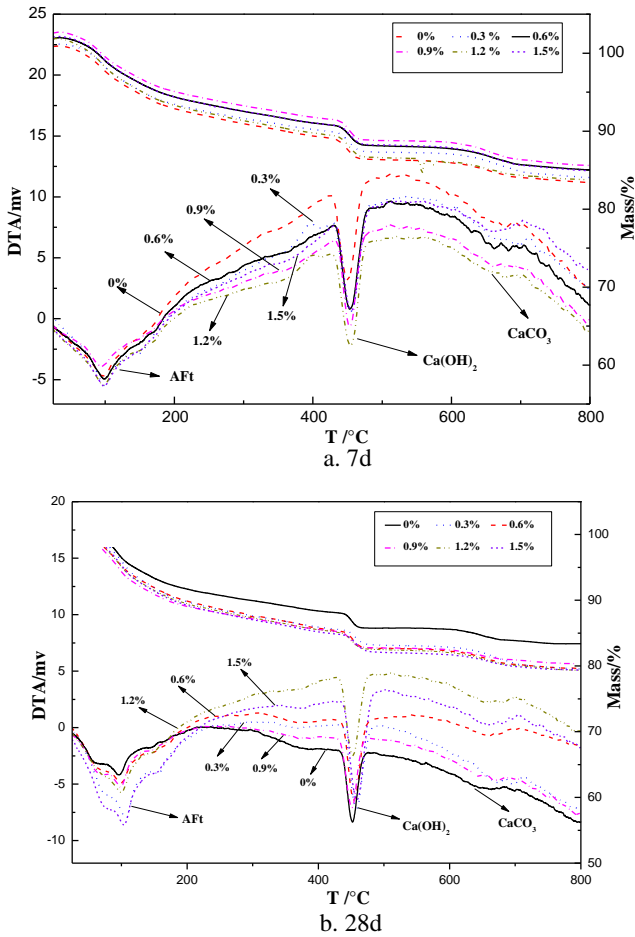


Fig. 5. DTA-TG diagram of the effect of dry ice on cement hydration.

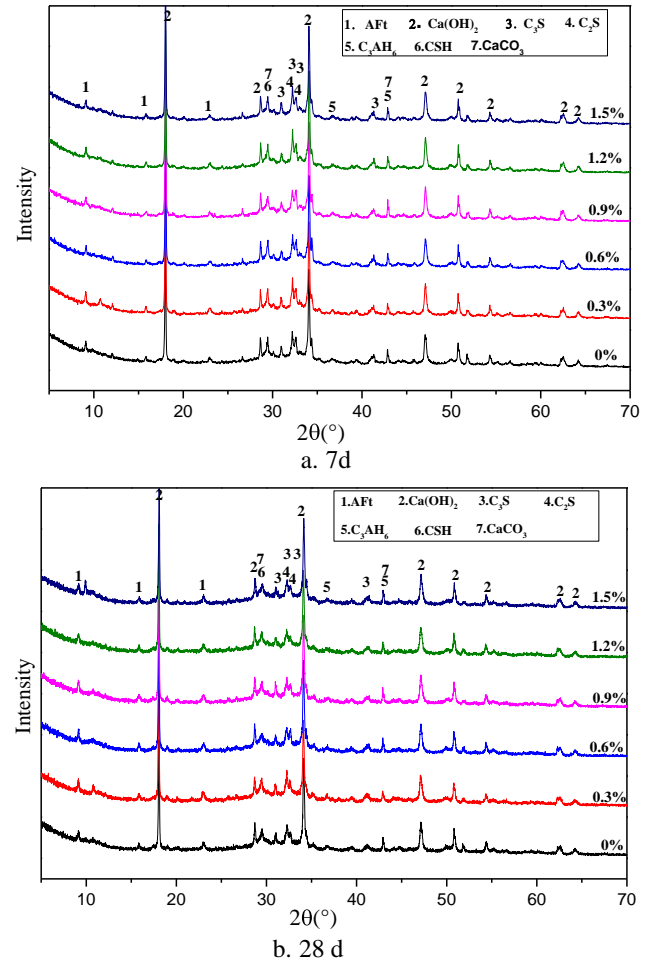


Fig. 6. XRD patterns of hydrated samples at 7 and 28 days.



at 110 °C, 460 °C and 650 °C. Compared with DTA-TG atlas, it can be seen that the endothermic peak of Aft is at 110 °C, the endothermic peak at 460 °C is attributed to  $\text{Ca}(\text{OH})_2$ , and the endothermic peak at 650 °C is assigned to  $\text{CaCO}_3$ .

It can be seen from Fig. 5(a) at 7 days that at 110 °C – 130 °C the sequence of weight loss rate of Aft is 2.411%, 2.347%, 2.268%, 2.191%, 2.144%, and 1.866%, when the dosage of dry age is 0.3 wt%, 0 wt%, 1.2 wt%, 1.5 wt%, 0.6 wt%, and 0.9 wt%, respectively, which shows that dry ice reduces the early amount of Aft, except 0.3 wt%; the sequence of weight loss rate of  $\text{Ca}(\text{OH})_2$  is 8.61%, 8.546%, 8.551%, 8.248%, 8.164%, and 7.649% showing a decrease trend when the dosage of dry age is 0.3 wt%, 1.2 wt%, 1.5 wt%,

0 wt%, 0.6 wt%, and 0.9 wt%, respectively, which shows that dry ice improves the early hydration of  $\text{C}_3\text{S}$  when its dosage is 0.3 wt%, 1.2 wt%, and 1.5 wt%, compared to the control sample. As can be seen from Fig. 5(b) at 28 days that at 110 °C – 130 °C the sequence of weight loss rate of Aft is 3.866%, 3.656%, 3.205%, 3.053%, 2.44%, and 2.298% from big to small when the dosage of dry age is 1.5 wt%, 0.3 wt%, 1.2 wt%, 0.9 wt%, 0.6 wt%, and 0 wt%, respectively, which shows that dry ice enhances the amount of Aft at late ages; the sequence of weight loss rate of  $\text{Ca}(\text{OH})_2$  is 9.49%, 7.346%, 7.181%, 7.15%, 6.195%, and 5.897% from big to small when the dosage of dry age is 1.5 wt%, 0.3 wt%, 1.2 wt%, 0.6 wt%, 0.9 wt%, and 0 wt%, respectively, which shows that dry ice

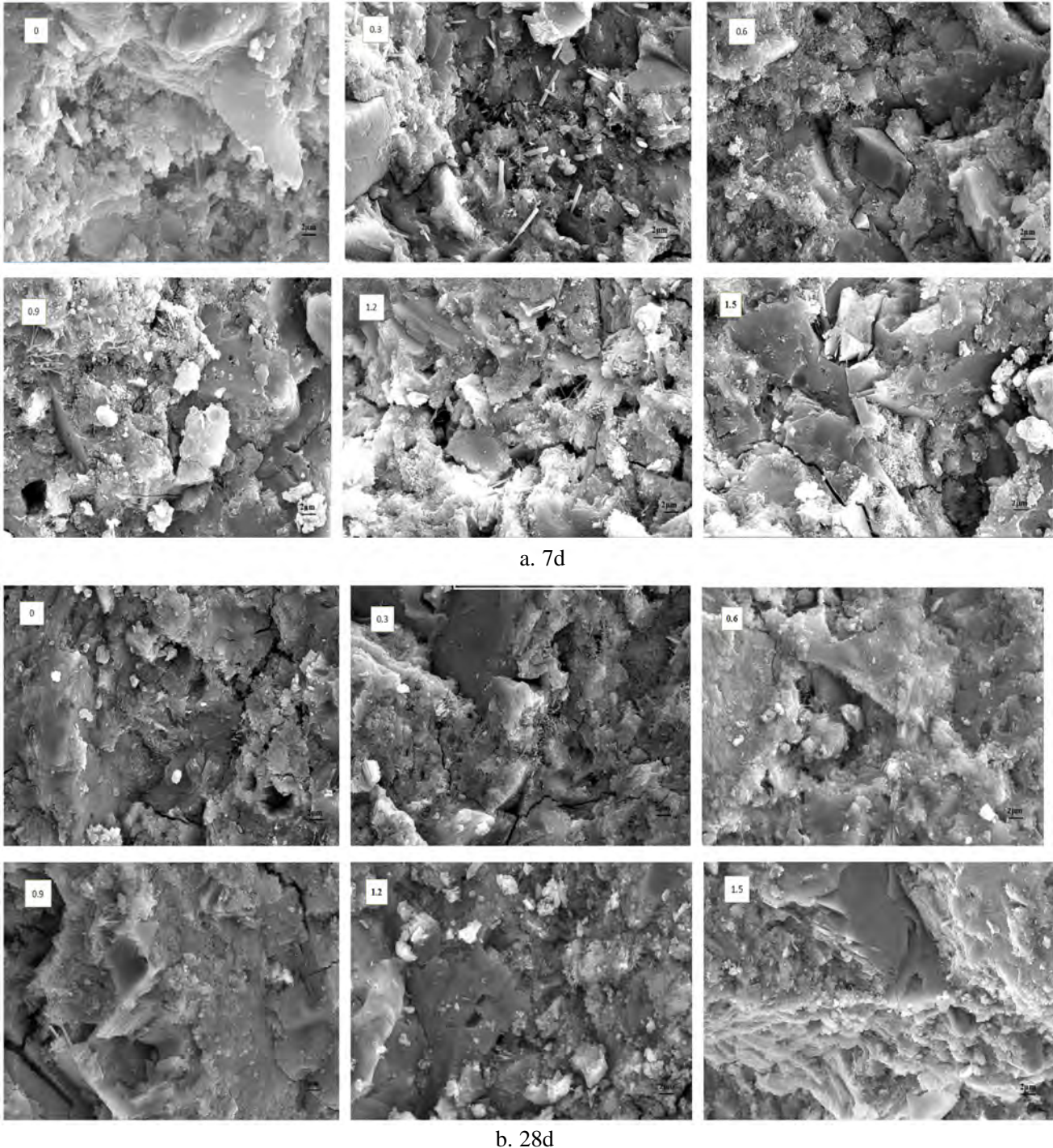


Fig. 7. SEM images of cement paste with different dry ice content at 7 and 28 days.

improves the hydration of C<sub>3</sub>S at 28 days compared to the control sample. In addition, the peak of CaCO<sub>3</sub> at 650 °C is less obvious, indicating that the influence of dry ice on generation of CaCO<sub>3</sub> can be negligible at the given dosage.

Based on the analysis, at the early age, dry ice restricts the early hydration of Portland cement, and generally, the greater its dosage, the more obvious the inhibiting effect, which agrees with the results of compressive strength of cement paste at 7 days. As the age increases, the hydration products of cement paste with dry ice gradually increases, resulting in higher compressive strength of the cement paste with dry ice compared to the control sample.

### 2.2.3. XRD results

The influence of dry ice on the cement hydration products were studied by XRD when the dosage of dry ice was 0 wt%, 0.3 wt%, 0.6 wt%, 0.9 wt%, 1.2 wt% and 1.5 wt%, and the results are shown in Fig. 6.

Fig. 6 shows the diffraction peaks of Aft, Ca(OH)<sub>2</sub>, C-S-H, CaCO<sub>3</sub> and C<sub>3</sub>AH<sub>6</sub> that are the hydrated as well as unhydrated C<sub>3</sub>S and C<sub>2</sub>S of cement pastes at the age of 7 and 28 days. As can be seen from Fig. 6(a), the incorporation of dry ice does not cause significant changes to the XRD peaks of the aforementioned hydration products, indicating that at 7 days, the effect of dry ice on the product of cement hydration process cannot be characterized by XRD, which is consistent with the slight change in the strength of hydrated sample at 7 days. However, as shown in Fig. 6(b), the diffraction peak ( $2\theta \approx 9^\circ$ ) of hydrated product Aft at 28 days increases with the increase of dry ice content. The diffraction peak of Ca(OH)<sub>2</sub> also increases with the increase of dry ice content, which indicates that the incorporation of dry ice improves the hydration of Portland cement at 28 days.

### 2.2.4. SEM results

SEM characterization was used to compare the effects of dry ice on the morphology and microstructure of hardened cement pastes, and the results are displayed in Fig. 7.

It can be seen in Fig. 7(a) that for cement paste at 7 days, the addition of dry ice changes the morphology of Aft. A typical example is that the morphology of Aft changes from needle-like to rod-like shape when the dosage of dry ice is 0.3 wt%, and Aft crystals interconnect with each other to be like dendrites when it is 0.9 wt%. The hardened cement pastes at the dry ice dosage of 0.6 wt%, 0.3 wt%, 0.9 wt%, 0 wt% are much denser than those at the dry ice dosage of 1.2 wt% and 1.5 wt%, which corresponds to the compressive strength at 7 days.

It can be seen in Fig. 7(b) that the structure of hardened pastes at 28 days becomes more compact compared to that of pastes at 7 days. Furthermore, the hardened pastes with dry ice are denser than those without dry ice, which can be seen clearly from the SEM image of paste with 0.6 wt% dry ice. It can be concluded that dry ice can change the hydration process of Portland cement and optimize the structure of hydrated paste.

## 3. Conclusions

Dry ice was incorporated at 0 wt%, 0.3 wt%, 0.6 wt%, 0.9 wt%, 1.2 wt%, and 1.5 wt% by weight of Portland cement, the effect of dry ice on the standard consistency, setting time, compressive strength, hydration-hardening process and product of Portland cement were studied. Main conclusions are as follows:

- (1) With the increase of dry ice content, the standard consistency of Portland cement first decreases but then increases, reaching its smallest value at 0.6 wt%. Similarly, with the increase of dry ice dosage, the patterns of the initial and final

setting are similar to that of standard consistency. When the dosage of dry ice is 0.6 wt%, the setting time is at its lowest value: the initial setting time decreases by 12.3%, and the final setting time reduces by 17.4% compared to the control cement paste.

- (2) The influence of dry ice on compressive strength at 7 days is subtle when its dosage is less than 0.9 wt%, and the compressive strength at 28 days drops notably when its dosage is more than 0.9 wt%. With the increase of dry ice content, the compressive strength of cement paste at 28 days first increases and then decreases. Compared to the control sample, the compressive strength at 28 days increases by 30.9% when its dosage is 0.6 wt%, and the compressive strength of the cement pastes at the given dosage of dry ice is higher than that of the control sample.
- (3) The results of hydration heat analysis, XRD, and DTA-TG show that the incorporation of dry ice has a retarding effect on the early hydration of Portland cement, however, dry ice improves its later hydration. The SEM results of show that dry ice could change the morphology of Aft and optimize the hardened structure of cement paste.

## Acknowledgements

The authors gratefully acknowledge the National Natural Science Foundation of China for financial support (No. 51678220) and the Postdoctoral Fund from Henan Polytechnic University. (No. 672108/106)

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