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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.

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# 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 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  $CO_2$ , a number of measures have been taken, such as reducing  $CO_2$  emission [1,2] and sequestrating  $CO_2$  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  $CO_2$  emission that accounts for around 7% of global  $CO_2$  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  $CO_2$  curing [8–13],  $CO_2$  strengthening recycled aggregates [14–19], as well as the transformation of carbon dioxide

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https://doi.org/10.1016/j.conbuildmat.2018.08.109 0950-0618/© 2018 Elsevier Ltd. All rights reserved. to carbonate as chemical additives [20-22]. X.Y. Pan [11]studied the strength and permeability of CO<sub>2</sub> surface treatment on oneday 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  $Li_2CO_3$  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  $CO_2$  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  $CO_2$ , 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  $\times$  40 mm  $\times$  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  $5^{\circ}$  and  $70^{\circ}$  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 um 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	$Al_2O_3$	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. 2. Effect of dry ice on the setting time.

Table 1

Main	properties	of	P.042.5.
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Cement	Finen-ess/%	Stability	Setting time/min	Setting time/min		Flexural strength/ MPa		compressive strength/MPa	
			initial setting	final setting	3d	28d	3d	28d	
P.042.5	1.5	qualified	187	392	5.5	9.6	33.5	50.8	

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