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Microstructure analyses of autoclaved ground dune sand–Portland cement paste

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HIGHLIGHTS

- Autoclaved 30% GDS mixture yielded compressive strength comparable to control mixture cured under normal conditions.
- The microstructure analyses reveal how GDS contributes to the strength and physical properties of the autoclaved mixtures.
- SEM and EDX showed the clear formation of dense and newly formed thin crystalline of CSH with a low CaO/SiO_2 ratio.
- XRD, DTA and TGA analyses revealed that the portlandite phase is consumed in GDS autoclaved mixtures.

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ABSTRACT

In this study, SEM, EDX, XRD, DAT and TGA are used to perform microstructure analyses of autoclaved ground dune sand–ordinary Portland cement (GDS–OPC) pastes. Pastes containing 0%, 10%, 20%, 30% and 40% GDS as cement replacement were prepared for this purpose. The microstructure analyses were used to examine the microscale changes that occur in the GDS–OPC pastes with increasing GDS replacement levels. The SEM and EDX results showed the clear formation of dense and newly formed thin crystalline calcium silicate hydrate with a low CaO/SiO_2 ratio. The XRD, DTA and TGA analyses revealed that the portlandite phase had been eliminated. The microstructure analyses reveal how GDS contributes to the strength and physical properties of concrete.

1. Introduction

Microstructure is the main factor affecting the mechanical and durability properties of concrete [1]. Therefore, it is essential to understand the microstructure of cement and hydration products of cement paste. The term "microstructure" refers to the structure that develops in concrete at a micro level. There are various techniques available to investigate the concrete and cement microstructure such as scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), differential thermal analysis (DTA), thermo-graphic analysis (TGA), and Fourier transform infrared spectroscopy (FT-IR). Many attempts have been made to study the microstructure of hydrated cement paste cured under normal condition [2–6]. Amorphous calcium silicate hydrate (CSH-gel), needle-like forms of ettringite and monasulfate, hexagonal crystalline plates of calcium hydroxide (CH) and dark pores are common phases observed in microstructure analyses [7–10]. Typical changes detected by DTA and TGA analyses include (a) the dehydration of evaporable water at 25–120 °C, (b) the dehydration of calcium silicate hydrate (CSH) at 180–300 °C, (c) the dehydroxylation of CH (portlandite) at 450–550 °C, and (d) decarbonation of calcium carbonate ($CaCO_3$) at 700–900 °C [11–13].

In some applications such as the precast concrete industry, elevated curing temperature and autoclave curing are used to accelerate the hydration process [14,15]. The microstructure analysis of concrete cured at elevated temperature is rather different than that of concrete cured at ambient temperature. During autoclaving, two features of reactions observe: the hydration products are crystallized, and silicaceous materials react with CH generated from the hydration of cement compounds to form additional CSH via a pozzolanic reaction. In the absence of added siliceous materials, CSH tends to be replaced by a crystalline phase such as α-C_2SH, which is undesirable for strong and durable of concrete [9,16].

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Previous studies by the authors have reported that ground dune sand (GDS) can be used as a source of silica in autoclave concrete production [17,18]. This study aimed to investigate the microstructures of the hydrated product of pure and blended cement pastes cured under autoclave condition. The GDS was used as a partial cement replacement in different contents (0%, 10%, 20%, 30%, and 40%). Microstructure analyses were performed using SEM, EDX, XRD, DTA, and TGA. By combining the information from these techniques, more detailed information can be obtained concerning the microstructures of cement paste cured under autoclave condition.

2. Experimental work

2.1. Material

Ordinary Portland cement (OPC) complying with ASTM C150 and GDS were used in this study. The dune sand was collected from the desert in the Kingdom of Saudi Arabia and mechanically ground into an approximate fineness of 90% passing 45 μm. The XRD analysis and SEM image of GDS are shown in Figs. 1 and 2, respectively. The chemical composition of OPC and GDS is presented in Table 1.

2.2. Mix proportion

Five mixtures of paste were prepared including a control mixture (CTRL) and four GDS–OPC blended mixtures (Table 2). The CTRL mixture was fabricated with 100% OPC as the binder material, while the blended mixtures were prepared with a combination of OPC and GDS. The GDS was used as a partial OPC replacement. The levels of replacement were 10%, 20%, 30% and 40% (by weight) and the mixtures were labeled as M10, M20, M30, and M40, respectively. The pastes were cast into steel molds (50 × 50 × 50 mm) and stored at room temperature for 24 h before being de-molded. The de-molded pastes were immersed in water at 23 ± 2 °C (normal curing conditions) for 16 h before being placed in the autoclave curing chamber. The temperature of the autoclave chamber was increased from room temperature to 180 ± 2 °C over 1 h. Correspondingly, the pressure was increased from atmospheric pressure to 1.0 MPa and held for 5 h. Next, the autoclave heater was turned off and the room temperature was reached after approximately 1 h. Samples of the CTRL mixture were divided into groups: one group was cured under normal curing (NC) conditions for 28 days, while the other was cured under autoclave conditions (AC).

2.3. Sample characterization

A compressive strength test was carried out in accordance with ASTM C109. The compressive strength results presented are an average of three specimens. For microstructural and microanalytical characterization of the hydrated pastes, Jeol JSM 6610LV coupled with EDX was used. Samples for SEM analysis were prepared by taking fractured surface specimen of the cured pastes. The specimens were glued to carbon stubs with carbon paint prior to the SEM analysis. The SEM analysis was carried out using accelerating voltages of 10 kV and 15 kV and magnifications of 3500× and 8000×. The chemical composition analysis of selected spots (fields of view) was carried out using EDX.

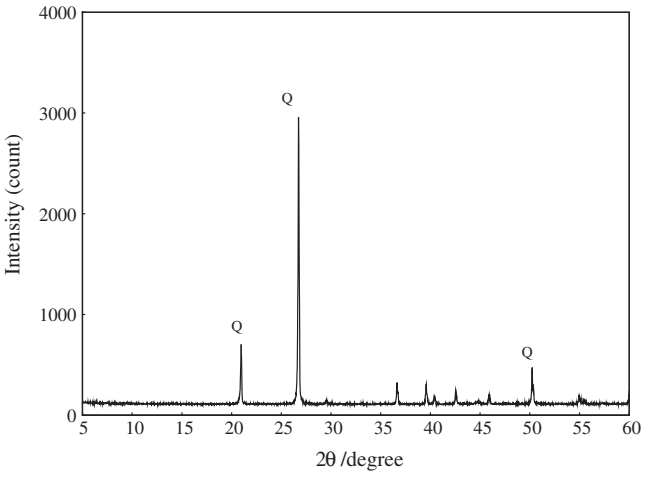

Fig. 1. XRD of GDS.

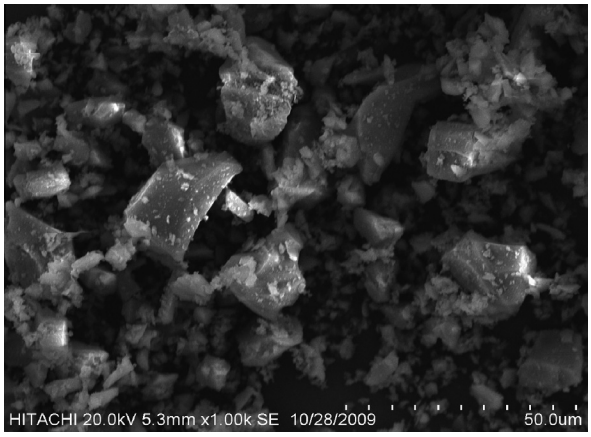

Fig. 2. SEM of GDS.

Table 1
Chemical composition and physical properties of OPC and GDS.

Oxides	Chemical composition (%)	
	OPC	GDS
CaO	57.96	1.68
SiO_2	22.62	91.4
Al_2O_3	6.11	0.99
Fe_2O_3	3.69	0.56
K_2O	0.98	0.21
MgO	2.16	0.18
Na_2O	0.17	0.05
SO_3	2.99	0.06
Loss on ignition (LOI)	3.02	1.15
Specific gravity	3.15	2.64
Specific surface area (cm^2/g)	3012	2574

Table 2
Mix proportions of the pastes.

Mixture designation	Composition (%)		W/B	Curing condition
	OPC	GDS		
CTRL	100	–	0.5	NC and AC
M10	90	10	0.5	AC
M20	80	20	0.5	AC
M30	70	30	0.5	AC
M40	60	40	0.5	AC

A Shimadzu XRD-6000 diffractometer was used for the mineralogical analysis of the hydration products. The samples for XRD analysis were prepared by grinding pieces of hydrated pastes into powder form passing a 75-μm sieve [5]. The scans were executed in the 2θ range from 5° to 60° at a scanning rate of 2°/min.

DTA and TGA analyses were applied to determine the amount of CH present and consumed in the cured samples. A predefined amount of the selected sample in powder form was weighed, placed in a platinum sample pan and then heated from 50 °C to 1000 °C at a heating rate of 10 °C min^{-1} in nitrogen gas flow. DTA and TGA analyses were carried out using a TA instrument (model SDT Q600).

3. Results and discussion

3.1. Compressive strength

The compressive strength results for the CTRL and blended cement pastes are presented in Fig. 3. For the CTRL paste, autoclave curing (AC) decreased the compressive strength by approximately 33% compared with that cured for 28 days under the normal curing (NC) conditions. This could be due to the change in the chemistry of hydration products and formation of non-uniform microsturcutre under autoclave curing conditions [9]. Fig. 3 showed that the