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# USE OF FINE GROUND DUNE SAND AS A SUPPLEMENTARY CEMENTING MATERIAL

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**Abstract.** The process of Portland cement production is associated with high consumption of energy and resources. Therefore, there is a need to replace the Portland cement with environmental friendly materials. This study was conducted to determine the feasibility of using ground dune sand as cement replacement materials under different curing conditions. Portland cement was replaced by ground dune sand at five levels of replacement (0–40% by weight). The compressive strength of mortar under standard and autoclave curing conditions and the influence of different autoclave temperatures and durations were investigated. The microstructure of selected mixtures was analyzed by XRD and SEM. Results showed that the compressive strength under the standard curing decreased as the level of replacement increased. However, under autoclave curing compressive strength increased as the content of ground dune sand increased. XRD and SEM revealed the absence of calcium hydroxide and the formation of secondary calcium silicate hydrate. The improvement of compressive strength and the absence of calcium hydroxide under autoclave curing indicated that the pozzolanic reaction between silica of dune sand and calcium hydroxide occurred.

**Keywords**: dune sand, autoclave curing, pozzolanic materials, compressive strength, XRD, SEM.

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## Introduction

Manufacturing of Portland cement consumes a considerable amount of energy and natural resources. The production of one ton of Portland cement requires about 4 GJ of energy and about 1.7 tons of raw materials (limestone and shale) which leads to environmental destruction and pollution problem (Malhotra 2000; Sabir *et al.* 2001; Worrell *et al.* 2001). There is a clear need to find materials which can be used as cement replacement material.

Pozzolan materials such as ground granulated blast furnace slag (GGBS), fly ash (FA), silica fume (SF) and natural pozzolan have been used successfully as substituting materials for Portland cement. These materials consist of high silica content in non-crystalline form (Sabir *et al.* 2001). Published data demonstrated that the use of pozzolan materials in concrete would result in ecological, technical and economic benefits (Hassan *et al.* 2000; Khatri *et al.* 1995; Malhotra *et al.* 2000; Malhotra 1993; Mehta 2004; Yazıcı *et al.* 2008).

However, the use of pozzolanic materials lead to various disadvantages including: increased setting time; slow rate of strength development; prolonged period of curing; increased water demand; increased superplasticizer dosage; and placing problem (Ghrici *et al.* 2006; Thomas *et al.* 1999). Furthermore, some pozzolana materials are becoming expensive and unavailable in market (Erdem, Kırca 2008; Cassagnabère *et al.* 2009). Due to above mentioned reasons, researchers are seeking for cheap and easily available cement replacement materials.

Thermal treatment method is used to improve the reactivity of some natural and by-product pozzolans. Metakaolin and rice husk ash were obtained by clacination of kaolin clay and burning of rice husk under controlled temperature, respectively (Sabir *et al.* 2001; Chandrasekhar *et al.* 2003). High strength concrete could be achieved by incorporating ground quartz sand and fine stone dust as a partial cement replacement in an autoclave curing system (Jaafar *et al.* 2002; Yang *et al.* 2000).

A number of studies reported that the use of crystalline silica performed better than amorphous silica under autoclave curing (Luke 2004; Jupe *et al.* 2008). Autoclaving amorphous silica such as silica fume, fly

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ash and natural zeolite were reported to display lesser strength than autoclaving crystalline silica (Luke 2004). Oyefesobi and Roy (1976) found that glassy silica was more reactive than the quartz silica at temperature below 100 °C, however, at temperature above 100 °C quartz silica showed better performance.

Dune sand is an abundant natural material, with high content of quartz silica (Cisse, Laquerbe 2000; Laquerbe *et al.* 1995). Due to fine particles (100% passing 600 μm) and uniform shape, dune sand does not meet the standard limit of the fine aggregate gradation recommended by ASTM C 33 and BS882 (Al-Harthy *et al.* 2007). Therefore, it has been used as partial replacement sand for fine aggregate and backfilling materials only (Al-Sanad *et al.* 1993).

A very limited work has been carried out to study the possibility of using dune sand as cement replacement materials (Guettala, Mezghiche 2011). However, no attempt has been made so far to utilize the ground dune sand as supplementary cementitions materials in mortar or concrete.

The objective of this study was to investigate the optimum level of cement replacement using ground dune sand; optimum curing cycle and provide microstructure analysis of the hydration products. The outcome of study is expected to encourage the use of dune sand as a cement replacement material in concrete industry.

## 1. Material and method

Dune sand (DS) used in this work was obtained from Saudi Arabia. The DS was mechanically ground to approximately cement fineness (95% passing a 45 μm opening size). The SEM image and XRD analysis of ground DS is shown in Figures 1 and 2, respectively. The chemical composition of DS is presented in Table 1. Ordinary Portland cement (OPC) which complies with ASTM C 150 was used in this study. The chemical composition and physical properties of OPC are given in Tables 1 and 2, respectively. Standard sand (Minining sand) with maximum particle size of 2.36 mm was used as fine aggregate in mortar mixtures. The specific gravity and fineness modulus of the standard sand was 2.5 and 2.49, respectively.

Standard consistency and the setting time of cement and dune sand-cement pastes were determined by Vicat probe and Vicat needle apparatus. Control (CTRL) mixture contained only Portland cement as binder material. In the other mixtures, Portland cement was partially replaced with ground DS. The levels of cement replacement by DS were 10, 20, 30 and 40% (by weight) and the mixture were labelled as DS10, DS20, DS30 and DS40

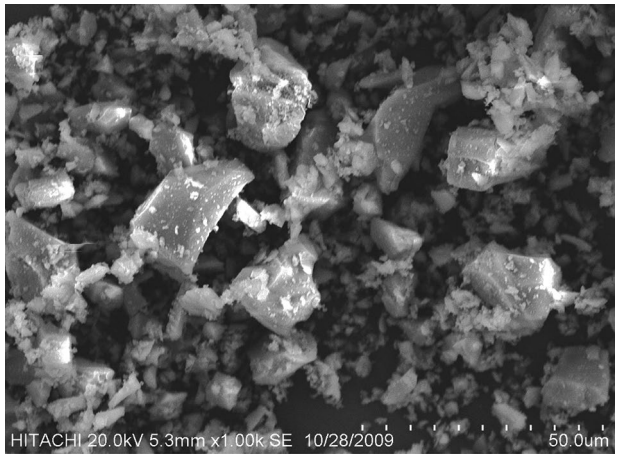


Fig. 1. SEM image of ground dune sand

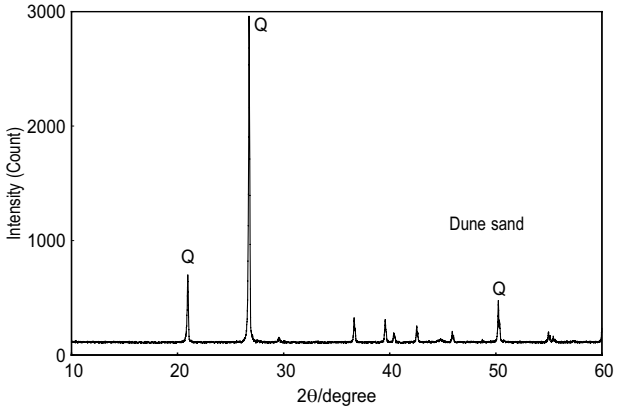


Fig. 2. XRD analysis of Dune sand

respectively (Table 3). The fine aggregate to binder (cement and DS) and water to binder ratios used for all mixtures were 3:1 and 0.5:1, respectively. The mixtures were prepared by the procedure specified in ASTM C 305. Mortar specimens were cast into cube of 50 mm and compacted on a vibration table for about 15 sec.

All specimens were covered with plastic sheets and stored in air at 23±5 °C for 24 hours. The specimens were then demolded and cured under one of two curing conditions: standard curing (immersion in water at 23±2 °C) or autoclave curing. Three specimens were cured under standard conditions for 28 days. The remaining specimens subjected to autoclave curing were first held under standard curing conditions for 16 hours to develop initial strength and then placed in the autoclave chamber.

The autoclave curing was used for two purposes; first, to investigate the optimum level of cement replacement using DS under autoclave curing and secondly to investigate the optimum autoclave curing condition. To

Table 1. Chemical analysis (%) of the materials used

| | $SiO_2$ | $Fe_2O_3$ | $Al_2O_3$ | CaO | MgO | $SO_3$ | MnO | $TiO_2$ | $K_2O$ | $Na_2O$ | $ZrO_2$ | IOI |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| OPC | 22.62 | 4.69 | 7.11 | 57.96 | 2.16 | 2.99 | – | – | 0.98 | 0.17 | – | 1.8 |
| Dune sand | 88.78 | 0.52 | – | 3.66 | – | – | 0.03 | 0.16 | 0.64 | – | 0.03 | – |

Table 2. Physical properties of Portland cement paste and mortar

| Property | Result |
|---|---|
| Normal consistency (%) | 33 |
| Setting time (min) | |
| (a) Initial | 110 |
| (b) Final | 180 |
| Compressive strength (MPa) | |
| (a) 3 days | 21 |
| (b) 7 days | 33 |
| (c) 28 days | 46.6 |

Table 3. Mixture proportions of mortar specimens

| Material | Mortar mixtures | | | | |
|---|---|---|---|---|---|
| | CTRL | DS10 | DS20 | DS30 | DS40 |
| Cement | 500 | 450 | 400 | 350 | 300 |
| DS | – | 50 | 100 | 150 | 200 |
| Fine aggregate | 1500 | 1500 | 1500 | 1500 | 1500 |
| w/(c + DS) | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |

achieve the first objective, autoclave temperature was increased from room temperature to 182 °C in 1 hour. Correspondingly, the pressure was increased from atmospherical pressure to 1.0 MPa and remained constant for 5 hours. After 5 hours the autoclave heater was turned off to cool and the room temperature was attained in 45 minutes. To investigate the optimum curing conditions, different temperatures and periods of curing were used. Temperatures of 182, 200, 210 and 220 °C were applied, whereas the two periods of curing used were 5 and 8 hours.

After completion of their curing periods, specimens were tested under a uniaxial compressive test machine. The rate of loading of the testing machine was 0.75 KN/ sec. The compressive strength is reported as the average of three specimens. The mineralogical composition of the hydration products were determined by X-ray diffraction, using a Shimadzu XRD-6000 diffractometer with a scanning rate of 2º/ min from 10º to 60° (2θ). Scanning electron microscopy (SEM) was used to investigate the morphology of the hydration products. The S-3400N machine was used for that purpose.

## 2. Result and discussion

### 2.1. Normal consistency and setting time of dune sand- cement mixture

The normal consistency and the setting time of dune sand-cement pastes are presented in Table 4. Water demand for the standard consistency was not affected by the replacement of cement by DS (Table 4).

This could be explained by the surface roughness and the angular shape of DS particles as shown in SEM image (Fig. 1) (Garcés *et al.* 2008). The slight increase in the setting time for mixtures containing DS where the replacement is higher than 10% could be attributed to the less $C_3S$ and $C_3A$ content in the total blended mixture. Therefore, fewer products of CSH and ettringite were produced in the early stage of hydration, which leads to slow strength development and retards the setting time (Hewlett 2003).

Table 4. Normal consistency and setting time of dune sand-cement pastes

| Mixture ID | Normal Consistency (%) | Setting time (minutes) | |
|---|---|---|---|
| | | Initial | Final |
| CTRL | 33 | 110 | 180 |
| DS10 | 33 | 110 | 180 |
| DS20 | 32 | 120 | 185 |
| DS30 | 32 | 120 | 185 |
| DS40 | 32 | 120 | 185 |

### 2.2. Compressive strength of mortar

Figure 3 shows the results of compressive strength of mortars after standard and autoclave curing. The compressive strength of mortars cured under standard curing decreased with the increased level of cement replacement by ground DS. At 10, 20, 30 and 40% replacement level the compressive strength was reduced by about 25, 38, 43 and 52%, respectively, compared to the CRTL mixture. The reduction can be attributed to lesser cement content in the mixtures, increased water to cement ratio and DS acted as a filler materials. The results indicate there was no pozzolanic reaction for the DS under normal condition.

For the autoclave curing, all mixtures containing ground DS displayed compressive strength higher than CTRL mixture (Fig. 3). This can be attributed to the fact that under autoclave curing, quartz $SiO_2$ in DS reacted with calcium hydroxide (CH) generated from the hydration of cement to produce additional secondary cementitious matrix.

The maximum compressive strength was obtained at 30% level of OPC replacement by DS (46 MPa). The 30% replacement of cement by ground DS lowered the Ca/Si ratio to be around unity which is associated with

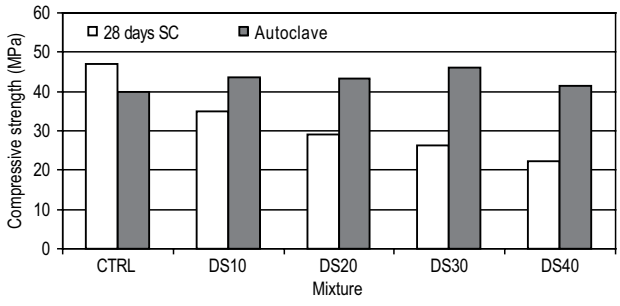


Fig. 3. Compressive strength of mortars cured under standard condition and autoclaved condition

the formation of rich silica calcium silicate hydrate (tobermorite or xonotlite) (Luke 2004; Mindess, Young 1981; Taylor 1997).

The autoclave curing reduced the compressive strength of the CTRL mixture by about 15% compared to the strength achieved by the standard curing. The reduction in the compressive strength after autoclave curing can be explained by the formation of crystalline α- $C_2SH$ which is associated with high porosity and less uniformity of the final products. This finding is in conformity with the literature (Yang *et al.* 2000; Mindess, Young 1981). The results also show that, under autoclave curing cement can be substituted by ground DS up to 40% without significant loss in the compressive strength.

### 2.3. Effect of different autoclave curing cycles on the compressive strength

DS30 mixture was selected to evaluate the optimum autoclaving temperature and duration because it developed the highest compressive strength under autoclave curing. This mixture was cured at 182, 200, 210, and 220 °C for two periods of curing (5 and 8 hours).

Figure 4 shows the effect of different autoclave temperatures and period of curing on the compressive of DS30 mixture. It can be seen that increasing the period of curing from 5 to 8 hours, led to improve the compressive strength of the mixture cured at 182 and 200 °C by 25 and 16%, respectively. However, prolonging the period of autoclaving at 210 and 220 °C did not show any improvement on the compressive strength. The maximum compressive strength was achieved when the specimens were cured at 200 °C for 8 hours. In addition, the compressive strength obtained at 8 hours can be obtained at 5 hours if the temperature is increased from 182 °C to 200 °C (Fig. 4).

### 2.4. Morphological investigation

Scanning electron microscope analysis of the CTRL standard curing and DS30 autoclave curing mixtures was conducted on the fracture surfaces of paste samples prepared with similar composition of the mortars.

Figure 5(a) shows the microphotographs of the CTRL mixture cured under standard curing for 28 days. The image shows that the hydrated CSH and crystalline calcium hydroxide (CH) product were dominant. The image of DS30 mixture cured under autoclave condition is shown in Figure 5(b). Hydrated CSH in needle-like structure (tobermorite) can be observed at the centre of the image and no hexagonal crystalline of CH is appeared. CH might be consumed by the pozzolanic reaction with silica in the DS.

### 2.5. X-ray diffraction (XRD) analysis

Figure 6 presents the XRD pattern of the DS30 cured under standard condition. Strong peak of quartz at 26.64º (2θ) and two peaks of CH at 34.1° and 18° (2θ) can be observed. This result reveals that no pozzolanic reaction between CH and quartz silica took place under stand-

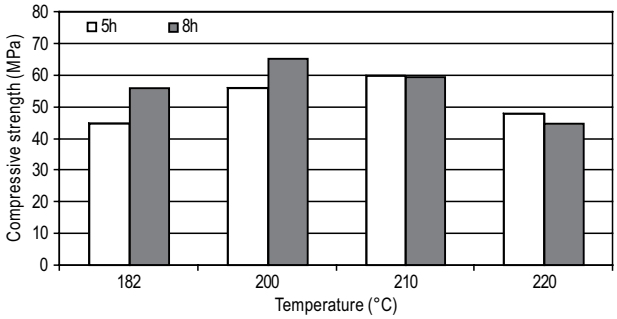


Fig. 4. Effect of different autoclave temperatures and period of curing on the strength of DS30 mixture

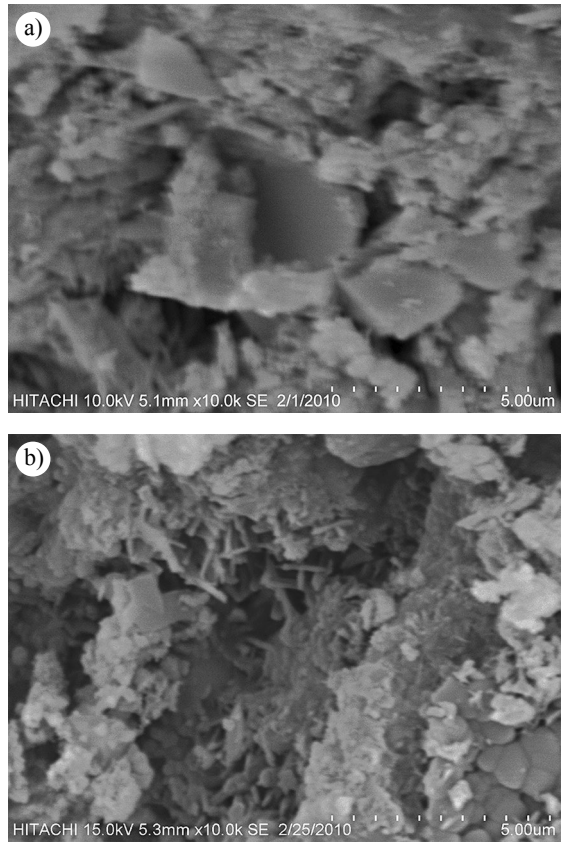


Fig. 5. SEM images: (a) Control mixture under normal; (b) DS30 mixture under autoclave curing

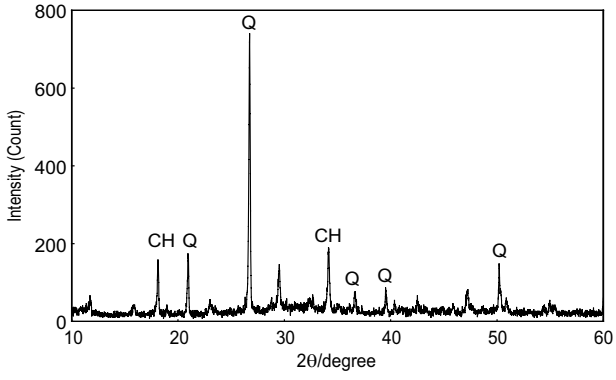


Fig. 6. XRD analysis of DS30 mixture cured under standard condition

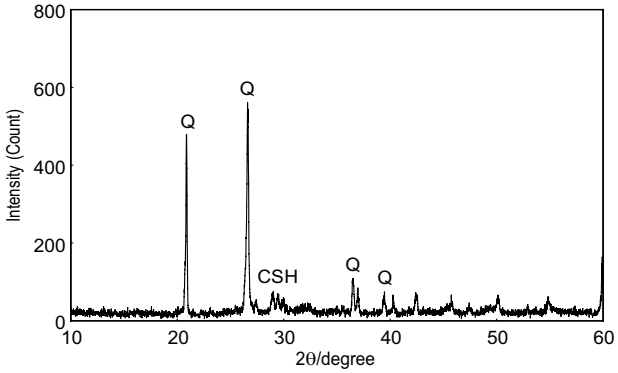


Fig. 7. XRD analysis of DS30 mixture cured under autoclave condition

ard curing and DS acted as a filler material. Ashraf *et al.* (2009) found similar peaks of CH at similar positions in the cement paste mixture cured under standard curing.

Figure 7 shows the XRD pattern of the same mixture but cured under autoclave condition. The high peak for the quartz ($SiO_2$) decreases but still present in the final products. However, the peaks for CH are no longer evident. The absence of CH peaks can be attributed to the pozzolanic reaction between the CH and ground silica in the DS.

This reaction produces additional cementitious products (i.e. tobormorite) resulting in an enhancement in the compressive strength.

## Conclusion

The effect of ground DS as a partial cement replacement on the compressive strength of mortar cured under standard curing and autoclave curing conditions was investigated. The following conclusion could be drawn:

a) For the standard curing, the compressive strength decreased with an increase in the level of cement replacement by ground DS. However, under autoclaved curing, the DS-cement mixtures enhance the compressive strength. The maximum compressive strength was achieved at 30% level of replacement;
b) The replacement of cement by ground DS did not modify the normal consistency and the setting time;
c) The highest compressive strength can be achieved by extending the period of autoclave curing or increasing the temperature. But the autoclave temperature should not be higher than 210 °C;
d) SEM study showed that crystalline CH appeared with standard curing, whereas, tobermorite in needle-like structure can be observed under autoclave curing;
e) After autoclaving, XRD results show that the quartz silica in DS can react with CH to form additional calcium silicate hydrate. The peak intensity for CH was absent but residual peaks from quartz silica were still apparent;
f) With autoclave curing, up to 40% of cement can be substituted by the ground dune sand. This not only enhances the compressive strength but also has a significant impact on the environment.

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