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The autoclaved concrete industry: An easy-to-follow method for optimization and testing

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HIGHLIGHTS

- This work describes a simple method to optimize the autoclave interaction of crystalline silica with hydrated lime (CH).
- An amount of 38% CH is consumed in a mixture of 50% SFL and 50% CH.
- A conversion factor of 1.32 to calculate the required silica was obtained.
- A verification test to the conversion factor was carried out using Portland cement.
- The amount of ground sand added to cement was precisely calculated using the conversion factor.

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ABSTRACT

This work describes a simple test method that is used to optimize the amount of ground crystalline sand of a given fineness that reacts equivalently with hydrated lime (CH) under autoclave conditions. The stimulated pozzolanic reactivity of the inertness of crystalline silica under autoclave curing leads to the formation of various amorphous and crystalline C–S–H structures. A series of 10 mixtures incorporating different proportions of ground crystalline quartz and calcium hydroxide with a water-to-binder ratio of 0.5 were prepared, cured under autoclave conditions and investigated using XRD, SEM-EDS and FT-IR analyses. A stoichiometric ratio of 1.32 was found between approximately 57% ground crystalline silica and 43% calcium hydroxide. Applying this ratio as a conversion factor, the optimum content of ground crystalline sand required to entirely consume the generated calcium hydroxide in hydrated cement was calculated to produce an optimized autoclaved concrete. Similarly, the amount of the same ground crystalline sand that should be added to cement was successfully obtained. A verification test was conducted using Portland cement paste to assess the validity of the conversion factor. The microstructural analysis of the optimized autoclaved cement paste with 30% ground crystalline sand reveals the formation of different nano-sized grains of C–S–H with a low Ca/Si ratio of 1.09. The average compressive strength of the 2.5-h autoclaved cement paste with 30% ground sand displays a value equivalent to that of the 28-day normally cured cement paste without ground sand. This result confirmed the validity of the current approach, which was supported by the good correlation among the XRD, SEM-EDS and FT-IR results. Consequently, mass production of optimized autoclaved concrete can be easily and economically achieved.

1. Introduction

Currently, advanced and green technologies necessitate the use of mineral admixtures in concrete. However, mineral admixtures are unavailable in many developing countries, which creates an added cost in concrete production due to the additional cost of importation and transportation processes. Additionally, frequent variation in the physical and chemical properties of mineral admixtures from one shipment to another represents another critical problem to be solved. The importance of mineral admixtures in concrete is indisputable. However, in developing countries, exploring alternative local materials has become unavoidable. The replacement of common mineral admixtures with local materials provides an alternative solution to these problems in specific applications.

Dune sand, abundantly available in many desert regions of the world, was proven to be active in autoclaved concrete. It can be taken as a typical example to the local materials that can potentially replace common mineral admixtures in specific concrete

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applications such as autoclave and steam curing. Several research works have discussed the effect of inert ground crystalline sand on the strength of concrete under normal, steam and autoclave curing conditions [1–4]. These results showed that autoclave curing represents the optimum curing technique, and the strength of autoclaved concrete was observed to increase gradually up to a certain replacement level after which a gradual reduction in strength occurred [1]. The authors refer to this replacement level as the critical replacement level. Moreover, the effect of ultrafine ground crystalline silica on the amount of water required for constant workability under normal curing was investigated [5], and it was shown that an increase in the amount and fineness of the ground crystalline silica decreased the water requirement due to improved particle packing. In another study, the presence of ultrafine inert ground crystalline silica was shown to improve the degree of hydration compared with those of the control mixture [6,7] under normal curing. Nonetheless, the grinding of quartz to an ultrafine level for incorporation with cement under normal curing adds an unavoidable cost. However, this cost can be reduced with another effective curing technique, i.e., optimized autoclave curing, which requires a lesser degree of fineness and accordingly minimizes the cost. To this end, the autoclave curing of ground crystalline silica (such as dune sand) stimulates the pozzolanic reactivity of this material in the presence of the calcium hydroxide (CH) generated during cement hydration [2,4]. This type of stimulated pozzolanic reactivity leads to the formation of different series of tobermorite phases, crystalline calcium silicate hydrates (C–S–H), and other forms of the main binder in concrete to provide improved properties.

Comprehensive research has been carried out by many researchers on the formation and development of tobermorite minerals (C–S–H) resulting from the interaction between inert ground crystalline silica and calcium hydroxide under autoclave conditions [8–11]. In brief, no critical studies were found that optimized and reduced (as much as possible) the amount of ground crystalline sand that should be stoichiometrically added to cement to efficiently reach the critical replacement level. Near the critical replacement level, improved mechanical properties are expected under different curing schemes, i.e., under autoclave curing. In most of the available publications, determination of the critical replacement level of inert ground crystalline silica depends on the trial methods applied. At the same time, other researchers have targeted the type of reaction products (i.e., the tobermorite phases) of the autoclaved mixture of inert ground crystalline silica and the CH reaction.

Therefore, the aim of the current study was to establish a stoichiometric ratio or conversion factor that would provide a reliable estimation of the critical replacement level of finely ground crystalline dune sand with CH under autoclave conditions. The outcomes of this research are expected to contribute to the production of optimized and cost-effective autoclaved concrete. Throughout the current investigation, the results are presented using different characterization techniques to identify the critical replacement level and are critically discussed.

2. Materials and experimental program

The identification and evaluation of the C–S–H phases are generally conducted using SEM-EDS analyses and scanning electron microscope (SEM) imaging coupled with an X-ray micro-analyzer (EDS) to determine the internal Ca/Si ratio characteristic of C–S–H. Additionally, XRD and FT-IR analyses are used to establish the stoichiometric ratio. Finally, the determination of the stoichiometric ratio between SFL and CH aids in determining the conversion factor. Thermogravimetric and differential thermal analyses (TGA/DTA) were performed using a TA instrument (model SDT Q600). A sample weight of 30 mg in powder form was placed in a Pt crucible and heated in a nitrogen atmosphere at a temperature range from ambient to 1000 °C and at a heating rate of 20 °C/min. Thermal analyses were carried out to determine the total amount of liberated CH from the autoclaved hydrated cement. Estimation of the total CH amount generated during cement hydration is necessary for calculating the optimum amount of dune sand that should be added to the cement. Therefore, the conversion factor should provide the optimum amount of ground crystalline dune sand added to cement under the defined autoclave conditions to obtain autoclaved concrete with enhanced strength.

Crystalline ground dune sand also referred to as silica flour powder (SFL) with a median grain size of 4.5 μm and a relative specific gravity of 2.7 was sourced from Riyadh, Saudi Arabia. The physical and chemical properties of SFL are summarized in Table 1. The particle-size distribution of SFL measured using laser scattering particle size distribution analyzer (LA 950V2, Horiba) produced a wide range of 0.01 μm to 3000 μm, as shown in Fig. 1. Analytical grade $Ca(OH)_2$ of 95% purity and general use cement (CEM) in compliance with ASTM C150/C150 M-12 [12] and with a median grain size of 11.5 μm were used. The chemical and physical properties of the SFL and cement are shown in Table 1 and Fig. 1. The main phases of the CEM are shown in Table 1, and the mineralogical composition of the SFL is provided in Fig. 2. The XRD patterns of SFL prove its high crystallinity, demonstrating that it is composed primarily of quartz silica (Qz).

Experimental work was conducted using different mixing compositions to estimate the optimum content of SFL that synergistically reacts with CH under autoclave conditions, as shown in Table 2. The data obtained were used to extract the conversion factor. All mixtures were alpha-numerically identified using the letter M followed by 2 numbers; the first symbol indicates the CH content and the second indicates the SFL content, as shown in Table 2. For example, "M05-95" indicates 5% CH and 95% SFL. A water-to-(SFL + CH) ratio of 0.5 was used for all mixtures, as shown in Table 2. Four verification mixtures of cement paste were prepared to evaluate the effect of the optimum amount of SFL on the strength of cement paste under normal and autoclave curing conditions. The verification mixtures were duplicated and prepared using a W/B ratio of 0.5 with and without the addition of 30% SFL under normal and autoclave conditions. The tested mixtures were identified accordingly as 30SFL-NC, 30SFL-AC, Co-NC and Co-AC. The mixtures with and without the addition of 30% SFL (30SFL-AC, Co-AC) were autoclaved for 2.5 h under the designed autoclave conditions in accordance with the suggested conversion factor. The mixtures with and without 30% SFL (30SFL-NC, Co-NC) were cured under normal conditions of 21 ± 2 °C and 100% RH for 28 days.

3. Preparation of mixtures and testing

Each duplicate mixture listed in Table 2 was prepared using 100 g of blended powder and 50 g of water with manual mixing for 1 min. Subsequently, 2-min mechanical mixing intervals were applied at a speed of 9000 rpm using a variable speed electric hand mixer followed by a pause. After five minutes had elapsed, the paste was poured into two crucibles. These preparation steps guaranteed that all mixtures were well homogenized and discrepancies in the results were avoided. Ceramic crucibles are preferred for such a study because they are able to withstand the testing conditions. Three-quarters of ceramic crucibles with a fixed volume of 25 ml were filled with the prepared paste. Two of the crucibles from each duplicate were held at normal temperature, whereas the other two were transferred to a rack inside the autoclave reaction chamber positioned above the water level. The autoclave was closed and activated to reach a pressure of 5 bars at a temperature of 165 °C for 1 h. The pressure was readjusted to reach 10 bars at a temperature of 180 °C in a period of 1 h, at which time these

Table 1
Physical and chemical properties of SFL and cement powders.

Oxide composition (%)	SFL	CEM
SiO_2	99.45	20.50
Al_2O_3	0.20	5.82
Fe_2O_3	0.02	4.10
CaO	0.02	64.14
MgO	0.005	0.71
Na_2Oeq	0.09	0.26
SO_3	NA	2.44
Loss on ignition (%)	0.22	1.38
Specific gravity	2.7	3.14
Fineness (m^2/kg)	324	373
C_3S	NA	53.40
C_2S	NA	13.25
C_3A	NA	8.49
C_4AF	NA	12.48