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Cracking potential and temperature sensitivity of metakaolin concrete

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HIGHLIGHTS

- Metakaolin showed higher sensitivity to temperature and increased hydration heat.
- Metakaolin increased splitting tensile strength and restrained tensile stress.
- The use of metakaolin in concrete mixtures showed greater shrinkage and lower creep.
- B3 model was used to model early age creep.
- Conversion of Aft phases to Afm phases with metakaolin increased the amount of pores.

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ABSTRACT

Metakaolin is a pozzolanic material with the potential to reduce permeability and chloride ingress; however, quantification of the effects of metakaolin use on the cracking sensitivity of concrete mixtures is needed to ensure that these improvements in performance are not compromised. This study was conducted to investigate the early age cracking potential due to restraint stresses from incorporating metakaolin in concrete. Calorimetry testing showed that metakaolin was more sensitive to temperature than mixtures with only Portland cement. Results showed more shrinkage, less stress relaxation, and higher restraint stress from the inclusion of metakaolin, potentially increasing cracking sensitivity of mixtures.

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1. Introduction

Concerns over the role of concrete constituent materials on early-age cracking susceptibility have led to research efforts to quantify these effects. Reductions in volume associated with thermal and autogenous shrinkage are the principle driving forces that lead to cracking. As the water-cementitious materials ratio (w/cm) decreases, cracking tendency increases because of higher amounts of autogenous shrinkage. As the amount of water present in the system decreases during hydration, low relative humidity conditions develop in the pores. These low-pressure pockets create tensile stresses on the pore walls as the water migrates through the system. This phenomena is attributed to the meniscus of the water becoming more narrow as the water tries to relocate within the pore network, causing the walls of the pores to pull closer together [1].

To meet durability standards, supplementary cementitious materials (SCMs) are now commonly included in concrete mixes. Materials such as slag, fly ash and silica fume have years of research and structural applications to identify the advantages and consequences of using these SCMs. Metakaolin is a newer SCM that is not being used as commonly in the field despite its benefit of high early strength and lower permeability.

These benefits are the result of the high lime consumption when metakaolin reacts with the hydrating paste and the paste undergoing a process called pore size refinement. This pore size refinement is an occurrence when the porosity of the paste decreases and the size of the pores are reduced. Although this process is useful in reducing chloride penetrability [2,3] and susceptibility to sulfates [4], this alteration in the microstructure of the paste has been shown to impact volume changes such as shrinkage and stress relaxation. It was found that increasing dosages of metakaolin will have positive correlation with free shrinkage rates [5]. Additionally, research on restraint stresses have been conducted on metakaolin but only at a constant temperature through ring testing without restraint on the outer edge of the concrete ring [5]. However, no research has been performed to test concrete with metakaolin for cracking potential when restrained under uniaxial

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conditions or how a predetermined temperature profile simulating semi-adiabatic conditions affects mechanical properties development, shrinkage, phase transformation and pore structure.

The overall research goal for this paper was to determine if any changes to early-age concrete behavior that affects cracking occur in concrete when metakaolin is added, the cause of any potential changes, and if this behavior precludes the use of the modified B3 model to simulate the early-age stress relaxation. In this study, rigid cracking frame tests were performed with a predetermined temperature profile to provide means for measuring uniaxial stress development with concrete being exposed to temperatures comparable to that of a 1 m^3 concrete element. This stress development was investigated using isothermal calorimetry and semi-adiabatic testing to determine the heat sensitivity of the metakaolin mixture, while also using a free shrinkage frame and the testing of cylinders to track the development of shrinkage and mechanical properties when subjected to the same temperature profile [6]. Pore size distribution characterization was performed using nitrogen adsorption and X-ray diffraction was also conducted to examine potential phase transformation that could affect the mechanical properties and stress development.

2. Materials and methods

2.1. As-received materials characterization

A Type I/II Portland cement and commercial metakaolin were used in this study. The as-received materials were characterized for their chemical, mineralogical and physical properties. The chemical oxide composition of the cement and metakaolin was determined by a certified external laboratory. Mineralogical characterization of the as-received materials as well as the hydrated cement-metakaolin paste was determined using a Panalytical X'Pert PW3040 Pro diffractometer coupled with Panalytical HighScore Plus software 3.1. The pattern was collected using Cu Kα radiation at a current of 40 mA and voltage of 45 kV; the step size was 0.02 degrees per step and counting time 4 s per step. Back-loading technique was used in placing the powder in the sample holder to minimize preferred orientation. The procedures of ASTM C1365 [7] were used in preparation of the powder for crystalline phases quantification. Blaine fineness was determined according to ASTM C204-11 [8] following instrument calibration using SRM 114q supplied by NIST. The specific gravity measurements were conducted in accordance to ASTM C188-09 [9].

Metakaolin was used at a 10% replacement level by mass. The concrete proportions for the control (CN) and metakaolin (10MK) mixtures used in free shrinkage, mechanical properties and cracking frame tests are presented in Table 1.

A w/cm ratio of 0.35 was maintained constant by adjusting the mix water for the water content of the chemical admixtures. The same proportions of the cementitious constituents were maintained in paste mixtures for isothermal calorimetry, nitrogen adsorption and X-ray diffraction experiments.

2.2. Isothermal calorimetry

In assessing the effect of metakaolin on the hydration kinetics and temperature sensitivity of the cementitious mixtures, isothermal calorimetry was performed on 10MK paste samples using a TAMAIR eight-channel isothermal conduction calorimeter. Tests were conducted at three different temperatures (23, 30 and 40 °C). The w/cm ratio was maintained at 0.35 and included chemical admixtures at the same proportions adopted for the corresponding concrete mixtures. The test was conducted in accordance to ASTM C1702 [10] Procedure B for external mixing.

Table 1
Mix design for concrete (1m^3).

Material	CN	10MK
Cement (kg/m^3)	445	400
Metakaolin (kg/m^3)	0	44
Coarse aggregate (saturated surface dry) (kg/m^3)	949	949
Fine aggregate (saturated surface dry) (kg/m^3)	624	607
Water (kg/m^3)	154	154
Air entraining admixture (mL/100 kg cementitious)	7	7
Type A/F admixture (mL/100 kg cementitious)	390	390
Type D admixture (mL/100 kg cementitious)	304	304
w/cm ratio	0.35	0.35

The cumulative heat of hydration at the three temperatures was then used to calculate the mixture activation energy using the procedure outlined by Poole et al. [11]. The activation energy was determined following Eq. (1):

$$\alpha(t_e) = \alpha_u \cdot \exp\left(-\left(\frac{\tau}{t_e}\right)^{\beta}\right) \tag{1}$$

where, α (t) = Degree of hydration at age t, α_u = Ultimate degree of hydration, τ = Time parameter (hours), β = Shape parameter, dimensionless, and t = Elapsed time since the contact of cementitious material and water (hours).

In this procedure, the α_u, β, and τ parameters were fit so that the degree of hydration with time fit to that calculated from the cumulative isothermal calorimetry heat of hydration results at 23 °C. The degree of hydration was calculated from isothermal calorimetry results using Eqs. (2)–(4):

$$\alpha(t) = \frac{H(t)}{H_u} \tag{2}$$

$$H_u = H_{cem} \cdot p_{cem} + 461 \cdot p_{slag} + 1800 \times p_{FA-CaO} \cdot p_{FA} + 330 p_{SF} \tag{3}$$

$$H_{cem} = 500 \cdot p_{C_3S} + 260 \cdot p_{C_2S} + 866 \cdot p_{C_3A} + 420 \cdot p_{C_4AF} + 624 \cdot p_{SO_3} + 1186 \cdot p_{FreeCa} + 850 \cdot p_{MgO} \tag{4}$$

where H(t) is the cumulative heat of hydration by mass of cement (J/g) from isothermal calorimetry at time t, p_{cem} is the % of cement by mass in the cementitious system, p_{slag} is the % of slag cement by mass in cementitious system, $p_{FA\text{-}CaO}$ is the % CaO in the fly ash used, p_{FA} is the % of fly ash by mass in cementitious system, p_{SF} is the % of silica fume by mass in cementitious system, p_i is the % of ith component by mass in cement. Since the ultimate heat of hydration contribution from metakaolin is not known, the value for silica fume was used in the analysis. Errors in H_u could cause a change in the calculated α_u fit parameter, but will not change the overall calculated adiabatic temperature development.

After the parameters were fit to the cumulative heat of hydration results at 23 °C, the τ parameter was fit to the cumulative isothermal calorimetry heat of hydration results at the 30 °C and 40 °C temperatures using the α_u and β parameters fit at 23 °C. The τ values from each temperature were then used as the rate constants in an Arrhenius plot to calculate the apparent activation energy.

2.3. Semi-adiabatic calorimetry

Semi-adiabatic calorimeters contain a heavily insulated concrete sample. This system allows a slight amount of heat to escape the enclosed system. Sensors in the calorimeter measure the heat loss rate in the calorimeter with time. The sensors in the calorimeter were calibrated by monitoring the heat loss rate with time using hot water placed in the calorimeter. The measured concrete sample temperature development and heat loss with time was then used to calculate the temperature rise, simulating an adiabatic system [11]. Measurements for semi-adiabatic calorimetry were conducted using instruments assembled at the University of South Florida. Obtaining the adiabatic temperature rise of concrete is a multistep process that involves determining the heat based activation energy of concrete mixture through isothermal calorimetry, calibration of the semi-adiabatic calorimeter and assessment of temperature loss, measurements of concrete temperature development in the semi-adiabatic calorimeter and calculating the fully adiabatic temperature rise. The fully adiabatic temperature rise was fit from the measured concrete temperature development and heat loss following the procedures recommended by RILEM [12]. The heat of hydration curve used in the fitting followed Eq. (5) [13].

$$Q_h(t) = H_u \cdot C_c \cdot \left(\frac{\tau}{t_e}\right)^{\beta} \cdot \left(\frac{\beta}{t_e}\right) \cdot \alpha_u \cdot exp\left(-\left[\frac{\tau}{t_e}\right]^{\beta}\right) \cdot \exp\left(\frac{E_a}{R}\left(\frac{1}{T_r} + \frac{1}{T_c}\right)\right) \tag{5}$$

where $Q_h(t)$ is the rate of heat release with time t, H_u is the total heat available for reaction (J/g), C_c is the cement content (kg/m^3), α_u, τ, and β are hydration coefficients, t_e is the concrete equivalent age maturity, E_a is the activation energy calculated from isothermal calorimetry, R is the universal gas constant, T_r is the concrete reference temperature taken to be 296.15 °K, and T_c is the concrete temperature at time t (°K). H_u can be calculated from the cement composition as shown in Eq. (3) [14] and (4) [13].

2.4. Concrete temperature profile

The temperature profile used in the testing simulates the temperatures that would be exhibited in a concrete mass element. In order to make the predetermined temperature profile, the temperature at the center of a newly placed concrete wall 1.0 m thick with a constant surface temperature was simulated using the experimental data collected from the semi-adiabatic calorimetry [15].