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Response of alkali activated fly ash mortars to microwave curing

Jeevaka Somaratna, Deepak Ravikumar, Narayanan Neithalath *

Department of Civil and Environmental Engineering, Clarkson University, Potsdam, NY 13699, United States

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ABSTRACT

Volumetric heating provided by microwave curing results in faster property development as compared to conventional heat curing that relies on heat conduction from the skin to the core. This paper discusses the compressive strength and microstructure development of microwave cured NaOH activated fly ash mortars, and relates them to the microwave energy absorption by the material which is a function of its dielectric properties. Microwave curing parameters are chosen so as to eliminate the effects of thermal runaway. Strengths that are comparable to or greater than those of mortars heat cured for 48 h at 75 °C are obtained in less than 120 min of microwave curing. The rate of energy absorption by the mortars is found to be relatively constant for a considerable fraction of the microwave curing duration, attributable to the compensation for the drop in dielectric loss factor as a result of moisture loss by the increase in internal electric field. Compressive strength is shown to be related to the microwave energy absorbed by the specimens, especially during the time when free water is present in the system.

1. Introduction

Alkali activation of aluminosilicate materials as binder systems for concretes are being extensively studied because of the several advantages they offer in terms of enhanced material properties as well as sustainability [1–4]. Fly ash or ground granulated blast furnace slag are the aluminosilicate materials commonly used because of the presence of soluble silica and alumina species in these materials that undergo dissolution, polymerization with the alkali, condensation on particle surfaces, and solidification that is responsible for the strength and stability of these binders. The influence of the source material and activator chemistry on the reaction mechanisms, reaction product and pore solution composition, and the microstructure of such systems have received considerable attention [5–12]. Activation of aluminosilicates with alkaline solutions generally requires the supply of external energy as heat for the formation of alkali aluminosilicates, especially when the activating solution does not contain soluble silica. A wide range of temperatures ranging from 40 °C to 90 °C have been reported in order to produce alkali activated binders with appreciable mechanical properties [5,6,11]. The influence of thermal curing conditions on the reaction mechanisms, carbonation of reaction products, and mechanical and microstructure development of alkali activated fly ash binders has been extensively reported [13–15].

Conventional heat curing techniques deliver thermal energy to the surface of the material by radiant or convective heating, which is transferred to the bulk through conduction. This creates thermal gradients in the material and thus non-uniform heating that might result in less than desirable properties. An alternative is microwave curing, where microwave energy is delivered directly to the material through the interactions at the molecular level with the electromagnetic field. Microwaves penetrate the material and provide energy, resulting in volumetric heating. The electromagnetic energy is converted to thermal energy which in turn is used to enhance the reaction kinetics and accelerate the strength gain. Hence microwave curing relies on energy conversion rather than heat transfer [16,17]. Since the energy transfer does not rely on diffusion of heat from the surfaces, rapid and uniform heating is possible [18]. A few studies on microwave curing of concretes have been reported, mostly dealing with the strength development of concretes under microwave curing [19–22]. Microwave assisted zeolite synthesis has also been studied [23–25], with the conclusion that the activation time was drastically reduced when compared to conventional hydrothermal synthesis. This paper attempts to provide information on the response of alkali activated fly ash mortars to microwave curing. In the quest for sustainable binders for concretes, it is essential to consider the energy implications of concrete production, and hence it is believed that an understanding of the mechanisms and efficiency of microwave curing of such binders would be greatly beneficial. Microwave energy absorption and its rates in activated fly ash mortars, and the influence of dielectric properties are also provided.

1.1. Material properties of significance when subjected to an electromagnetic field

The interaction of the electromagnetic field with the material structure results in energy transfer, and the mechanisms are

* Corresponding author. Tel.: +1 315 268 1261; fax: +1 315 268 7985.
E-mail address: nneithal@clarkson.edu (N. Neithalath).

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described in detail elsewhere [18,26–28]. Microwave-material interactions result in translational motions of the free or bound charges and the rotation of dipoles. The inertial, elastic, and frictional forces that resist these motions causes volumetric heating of the material. The effect of the electromagnetic field on the material is primarily dependent on the complex dielectric permittivity (ε) of the material, given as:

$$\varepsilon = \varepsilon' - i\varepsilon'' \tag{1}$$

where ε′ is the dielectric constant, and ε″ is the dielectric loss factor. ε′ describes the ease with which a material is polarized by the electric field, and ε″ measures the efficiency with which the electromagnetic radiation is converted to heat. The ratio of dielectric loss factor to the dielectric constant is the loss tangent (tan δ). This parameter depends on the frequency of the microwave radiation and the temperature. The effectiveness of heating a material using microwave energy depends on the amount and rate of energy absorption by the material. The rate of energy absorption, expressed as the power per unit volume (P) is one of the important parameters for microwave processing of materials, and is given as:

$$P = \omega\varepsilon_0\varepsilon''|E_{\text{int}}|^2 = \omega\varepsilon_0\varepsilon' \tan\delta|E_{\text{int}}|^2 \tag{2}$$

where ω is the angular frequency, ε_0 is the permittivity of free space (8.854×10^{-12} F/m), and E_{int} is the intensity of the internal electric field. As can be observed from Eq. (2), the dielectric properties (ε′ and ε″) exert a significant influence on the absorbed power, and thus the volumetric heating in the material. A significant portion of the absorbed power is converted to heat within the material as [28]:

$$\frac{\Delta T}{\Delta t} = \frac{P}{\rho C_p} = \frac{\omega\varepsilon_0\varepsilon''|E_{\text{int}}|^2}{\rho C_p} \tag{3}$$

where T is the temperature, t is the time, ρ is the density and C_p is the heat capacity. The dielectric parameters also influence the depth to which microwaves penetrate into the material, defined as either the depth at which the incident power is reduced by half [28], or the depth at which the power density drops to e^{-1} of the surface value [19,29]. The latter definition is used in this paper.

2. Experimental program

2.1. Materials, mixtures, and specimen preparation

The alkali activated mortars used for this study were prepared using a Class F fly ash that conforms to ASTM C 618 as the binding material. The chemical composition of fly ash is as follows: SiO_2 — 50.2%, Al_2O_3 — 28.7%, Fe_2O_3 — 5.72%, CaO — 5.86%, MgO — 1.74%, Na_2O_e — 0.96%, SO_3 — 0.51%, and LOI — 2.80%. Fly ash is rich in the aluminosilicate phases, which can undergo dissolution–polymerization–condensation reactions in the presence of alkalis to form the binding gel. Analytic reagent grade sodium hydroxide was used to prepare the alkaline activating solutions having concentrations of 4 M, 6 M, 8 M, and 10 M. River sand (d_{50} of 0.6 mm) was used as the fine aggregate. The mortar mixtures were proportioned to contain a paste volume of approximately 50%. Activating solution-to-binding material ratio of 0.40 was used for all the mixtures. Fly ash and the fine aggregates were mixed thoroughly in a laboratory mortar mixer. Requisite amount of the alkaline activator solution of the chosen concentration was gradually added while mixing until the components were homogenized. The mortar was filled in cubical acrylic molds of 50 mm size, and compacted using a table vibrator. The specimens were cured for 12 ± 1 h in the molds at 23 ± 2 °C, demolded, and then subjected to either the microwave curing or heat curing regime. Heat curing was carried out in a laboratory oven at 75 °C for either 24 or 48 h after the initial 12 h at ambient conditions, based on a previous study [5].

2.2. Determination of microwave curing parameters

As stated earlier, microwave curing was started only after 12 h of ambient curing, primarily due to difficulties in subjecting the mold material to microwave radiations for extended periods of time. The average incident microwave power of the oven used was tunable from 0 to 1200 W, at increments of 10%, and the microwave frequency used was 2.45 GHz (this is the most commonly used frequency). Three 50 mm cubical specimens corresponding to a particular mixture were kept in the microwave cavity (37.5 cm × 37.5 cm × 25 cm) for curing. A few initial experiments carried out at higher incident power levels such as 1200 W and 600 W led to severe cracking and deterioration of the specimens within a few minutes. At higher incident powers, in a dielectrically inhomogeneous material, the electromagnetic waves encounter a variety of boundary conditions, resulting in extreme local variations in the electric field intensity. As shown in Eq. (2), absorbed power is proportional to the square of the electric field intensity, and hence local variations in the field results in local temperature fluctuations within the material. It is possible that the temperature of a local area increases rapidly under non-uniform fields, resulting in "thermal runaway" [16], leading to very high internal stresses and material fracture. With rapid increase in temperature, ε″ also rises rapidly, consequently increasing the absorbed power also.

An example of temperature development in 8 M NaOH activated mortar cubes subjected to 240 W, 360 W, and 600 W of average incident power are shown in Fig. 1. Temperatures were measured using an infrared thermal scanner with its emissivity set to 0.95, which is the optimum to read concrete temperatures as suggested in standard emissivity charts (0.94–0.96 for cement and concrete). With increasing incident power, the specimen temperature is seen to increase. At an average incident power level of 600 W, the specimens fractured between 5 and 10 min of microwave curing while at 360 W, the fracture occurred after 60 min and before 120 min. No specimen cracking was observed when the specimens were subjected to a power level of 240 W. Hence an average incident power level of 240 W (corresponding to 20% of the maximum rated power) was used for the remaining experiments. A maximum microwave curing duration of 120 min was chosen because the compressive strengths of the specimens did not increase significantly beyond this curing duration, and random surface cracking began to be observed after this time.

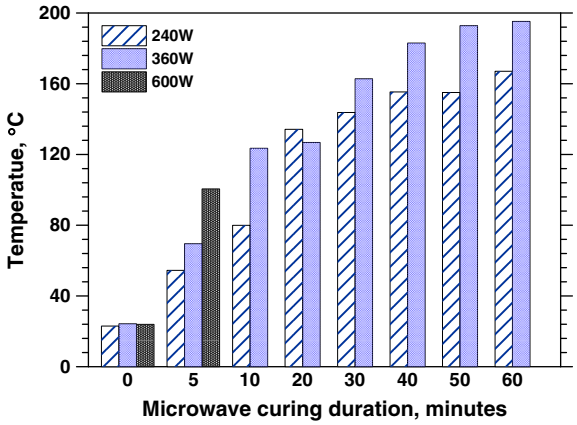

Fig. 1. Temperature development as a function of microwave curing time and incident power for 8 M NaOH activated mortars. Specimens disintegrated between 5 and 10 min at 600 W power and between 60 and 120 min at 360 W power.