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Microwave Curing Techniques for Manufacturing Alkali-activated Fly Ash

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ABSTRACT

Alkali-activated fly ash (AAFA) has been identified as a low-carbon alternative to Portland cement (PC). However, conventional thermal oven curing, typically at 85^{o}C, is commonly required to firstly initiate and then accelerate the chemical reaction, which may result in more energy consumption, offsetting the environmental benefits which could be obtained from AAFA.

In this study, the potential of using microwave as an alternative low-energy thermal curing method for manufacturing AAFA has been explored. A microwave curing regime based on a control strategy using temperature feedback data was developed and evaluated in the manufacture of AAFA. The temperature profile within the AAFA sample produced under microwave curing was obtained by using an embedded optical fibre temperature sensor, which was thus used to adjust the microwave power in real-time in order to achieve a desired internal temperature. The AAFA samples manufactured by microwave curing were characterized using compressive strength, XRD, NMR, MIP and SEM, which were also compared with the samples from thermal oven curing. The results indicated that the AAFA manufactured with microwave curing not only showed an equivalent performance, similar reaction products and microstructure, but also showed dramatic reduction in energy consumption.

1. INTRODUCTION

Alkali-activated fly ash (AAFA) is a non-Portland cement binder manufactured by using alkaline activator to activate fly ash, a byproduct from coal power plants. This clinker-free cementitious material is now considered as a promising alternative to Portland cement (PC). Not only are the CO_2 emissions reduced, but the properties of AAFA remain similar to PC. However, thermal curing, typically at 85^{o}C in an oven, is commonly applied to firstly initiate and then accelerate the chemical reaction. Although beneficial to the development of the early strength of AAFA, the energy involved in thermal curing may offset the environmental benefits from this clinker-free system.

In the past, several investigations have been carried out by different researchers to explore the potential of using microwave energy to cure PC-based products (Wu et al., 1987, Leung and Pheeraphan, 1995, Leung and Pheeraphan, 1997, Rattanadecho et al., 2008a). However, the research on curing AAFA with microwave is scarce. The first application of microwave curing on AAFA was reported by Somaratna et al. (Somaratna et al., 2010). In their study, the AAFA mortars (50mm×50mm×50mm) were cured in a domestic microwave oven with different curing durations and under different percentages of full power level. After the microwave heating, the highest temperature found in the core reached 140^{o}C or even 210^{o}C, which would cause severe cracking inside the sample due to over evaporation of the water and overheating inside the sample. The compressive strength of AAFA mortar with microwave curing for 120 min (20% of full power) reached 58 MPa, which is higher than that from thermal oven curing at 75^{o}C for 48 hours (38 MPa).

To avoid the crack formation during microwave curing, Shi et al. (Shi et al., 2014) applied pulsed microwave curing regime on AAFA pastes (25mm×25mm×25mm) with a domestic microwave oven. Their results showed that with pulsed

microwave curing, the AAFA samples could gain higher early strength within shorter curing duration than conventional thermal oven curing. After pulsed microwave curing, the highest cross-sectional temperature of AAFA samples was around 120^{o}C and the highest compressive strength reached 49 MPa.

Attempts have also been made to optimize the microwave curing regime by combining different power levels in order to increase the strength of PC-based pastes (Makul and Agrawal, 2011). In Makul and Agrawal's study, they found that the optimum microwave curing process consisted of 390W for 15 min, 811W for 15 min and 390W for 15min which resulted in the highest compressive strength of OPC sample. These findings indicate that the power control is significant for the strength development of OPC products.

Most of the previous studies were carried out with domestic microwave ovens, but the power control has its own limitations. In addition, although the temperature control inside the sample was considered crucial during microwave curing process, most of temperature measurements in the previous studies were taken after microwave curing, which would result in the inaccuracy due to the heat loss. Therefore, in this paper, the feasibility of applying automatic microwave power control based on real-time temperature feedback for curing the AAFA samples was studied. The compressive strength and the reaction products of the AAFA manufactured in this system were also characterized and discussed.

2. EXPERIMENTAL

2.1 Materials

The fly ash used in this work was from Weihe coal power plant, Shaanxi, China. The chemical composition of FA is as follows: SiO_2 - 49.29%, Al_2O_3 - 30.55%, Fe_2O_3 - 5.55%, CaO - 5.96%, MgO - 0.81%, Na_2O - 0.74%, K_2O - 1.38%, SO_3 - 0.63%, Ti_2O - 1.08%, and LOI - 1.87%. Sodium Hydroxide of industrial grade with a purity of 95% was supplied by ReAgent.

The custom-made microwave oven was supplied by Industrial Microwave System, Ltd, UK. It is capable of generating power at any level from 0 to 1000 Watts. Equipped with fibre optic temperature sensor, the microwave oven can provide real-time monitoring of temperature changes inside the sample during microwave curing process and the temperature feedback is used to adjust the microwave power accordingly.

2.2 Preparation of samples

The AAFA paste was made by mixing fly ash with 8M NaOH solution with a solution to solid ratio of 0.33. The dimension of the samples was 25mm by 25mm by 25mm.

The samples were then cured by two accelerated curing methods respectively as described in detail below:

85^{o}C thermal curing: As control samples, the samples were cured in 85^{o}C thermal oven up to 24 hours immediately after casting.

Microwave curing: AAFA samples were cured under microwave curing process with proportional-integral-derivative (PID) control. The temperature rising procedure consisted of four steps, corresponding to four targeted maximum temperatures (65^{o}C, 85^{o}C, 105^{o}C, 125^{o}C).

2.3 Tests

The compressive strength of AAFA samples was tested after 2 hours followed by accelerated curing. Reaction products were characterised by means of X-ray diffraction (XRD) and nuclear magnetic resonance (NMR). The microstructure of hardened AAFA was studied by mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM).

3. RESUTLS AND DISCUSSION

3.1 Temperature profile of AAFA sample and power profile of microwave curing process

Figure 1 shows that the total curing process consists of four stages and the set targeted temperatures of each stage are 65^{o}C, 85^{o}C, 105^{o}C and 125^{o}C respectively, which are chosen as target for the feedback control with a maximum power level of 400W. The PID controller is used to achieve the feedback control, during which the power level will be adjusted automatically to maintain the targeted temperature.

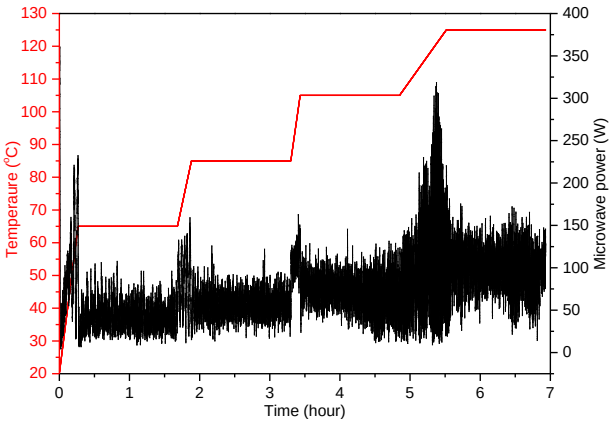


Fig. 1 Temperature profile and power profile of AAFA sample under microwave curing

3.2 Compressive strength

The development of compressive strength of AAFA materials with thermal oven curing and microwave curing is shown in Table 1. It was observed that

with less than 7 hours of microwave curing, the compressive strength of AAFA is almost identical to the AAFA under thermal oven curing for 24 hours.

Table 1 Compressive strength of AAFA under different curing methods

Curing method	Compressive strength (MPa)
85°C Oven	***40.67***
Microwave	***40.91***

3.3 X-ray diffraction (XRD)

Figure 2 shows the XRD spectra of raw fly ash and AAFA samples under thermal oven curing and microwave curing respectively. It can be seen that fly ash is an amorphous material (halo: 17-33° 2θ) with a few crystalline phases, such as quartz, mullite and hematite. The diffraction spectra changed appreciably after alkali activation of fly ash. The crystalline phases detected in the raw fly ash remained apparently unaltered after alkali activation, while the area of the hump representing amorphous phase decreased, indicating that amorphous phase had taken part in the reaction. It was observed that two new crystalline phases of zeolites appeared in both of the two AAFA samples. The peaks appeared at 13.8° and 24° 2θ were attributed to hydroxysodalite (atomic Si/Al ratio = 1), while the other crystalline reaction product appeared at 34.2° 2θ for AAFA with microwave curing was assigned to a zeolite phase of chabazite-Na (atomic Si/Al ratio = 2) (Criado et al., 2007, Bakharev, 2005).

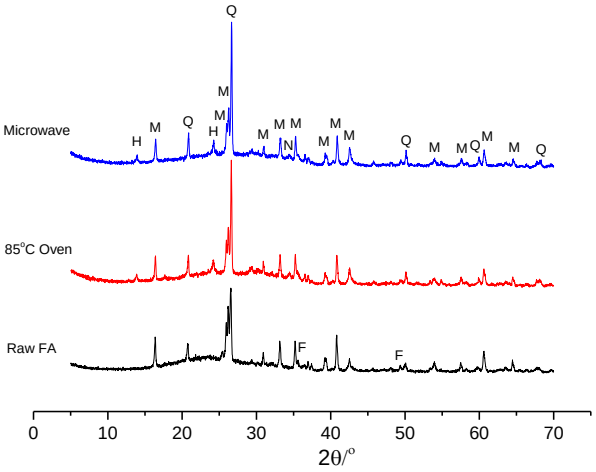


Fig. 2 XRD patterns of raw fly ash and AAFA under thermal curing and microwave curing (Q-quartz, M-mullite, F-hematite, H-hydroxysodalite, N-chabazite-Na)

3.4 Nuclear magnetic resonance (NMR)

The ^{29}Si NMR spectra of the raw fly ash and the AAFA samples under thermal oven curing and microwave curing are illustrated in Fig. 3.

In the raw fly ash, the peaks detected at -84, -94, -98, -101, -105 ppm were attributed to different Si environments of the fly ash. The chemical shift at around -90 ppm was attributed to the Si environment in mullite, while the chemical shifts above -108 ppm were attributed to crystalline phases of silica in quartz (Fernandez-Jimenez et al., 2006, Criado et al., 2008).

After alkali activation, the broad resonance band in raw fly ash shifted in both AAFA samples, indicating the generation of reaction products in the AAFA samples.

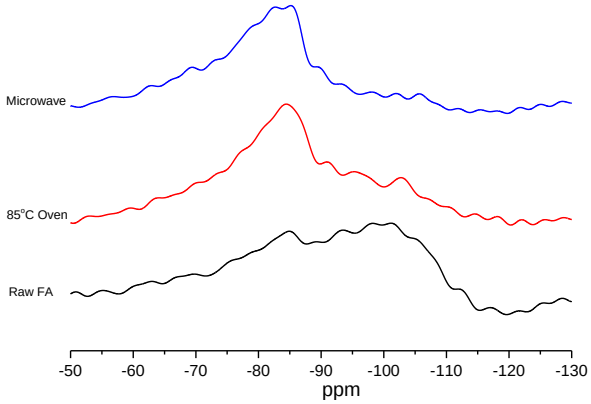


Fig. 3 ^{29}Si NMR spectra of raw fly ash and AAFA under thermal oven curing and microwave curing

3.5 Mercury intrusion porosimetry (MIP)

The pore size distribution of hardened AAFA samples under two different curing methods are presented in Fig. 4.

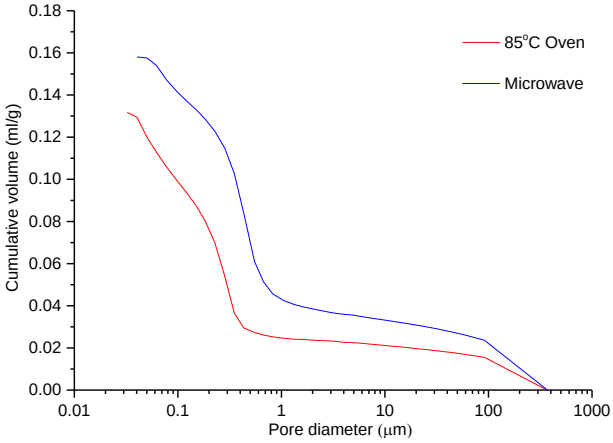


Fig. 4 Pore size distribution of hardened AAFA under thermal oven curing and microwave curing

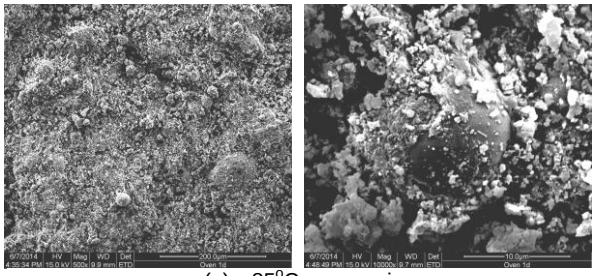
It can be seen that compared with thermal oven cured AAFA sample, microwave cured AAFA has more pores in all pore size ranges, indicating that microwave curing would have the potential to generate a coarser microstructure, which has been verified by the total porosity of hardened AAFA samples under two curing methods as shown in Table 2.

Table 2 Total porosity of AAFA under thermal oven curing and microwave curing

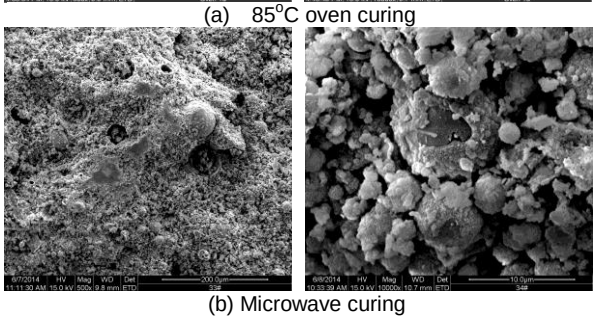
Curing method	Total porosity (%)
85°C Oven	20.39
Microwave	24.13

3.6 Scanning electron microscopy (SEM)

Figure 5 presents the fracture surface of the AAFA with two different curing methods which featured heterogeneous microstructures. Compared to oven cured AAFA sample, microwave cured AAFA sample had more pores, which is consistence with the MIP results. Sodium aluminosilicate gel formed on the surface of fly ash particles and the gap between the particles was observed in both of the AAFA samples, which contributes to the development of the strength (Rattanadecho et al., 2008b).



(a) 85^{o}C oven curing



(b) Microwave curing

Fig. 5 SEM images

3.7 Comparison of energy consumption of AAFA under thermal oven curing and microwave curing

The energy consumption of AAFA under different curing methods is shown in Table 3. Compared with 85^{o}C thermal curing for 24 hours, a significant reduction in energy consumption has been achieved from microwave curing method. In addition, equivalent strength was also achieved from the microwave curing method. Further comparison based on per unit MPa (compressive strength) also clearly indicated the remarked benefits from the microwave curing.

Table 3 Energy consumption (EC) of AAFA under thermal oven curing and microwave curing

Curing method	Duration (hour)	Total EC (kJ)	Strength (MPa)	EC (kJ/MPa)
85^{o}C Oven (1000W)	***24***	86400	***40.67***	2124
Microwave (See Fig. 1)	***5.7***	1835	***40.91***	44.8

4. CONCLUSIONS

From the results of the strength, the AAFA with microwave curing can obtain equivalent strength and microwave curing can shorten curing duration remarkably. It is highlighted that with much shorter curing duration, the compressive strength of AAFA sample under microwave curing is comparable with that under thermal oven curing. The XRD and NMR results demonstrated the slight difference of the reaction products generated under thermal oven curing and microwave curing. The microstructure analysis demonstrated that microwave curing could lead to the generation of a more porous structure of hardened AAFA sample compared to thermal oven curing.

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