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Phase evolution of fly ash calcium constituent at early alkali activation reaction age



Ningning Shao, Ze Liu*, Jianjun Fan, Yu Zhou, Dongmin Wang

School of Chemical and Environmental Engineering, China University of Mining & Technology, Beijing 100083, China

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ABSTRACT

Calcic phases are well documented to be the key-strength-giving components of alkali activated materials (AAMs). The clearness of phase evolution processes of fly ash calcium constituent during alkali activation reaction (AAR) will be of great significance to both the cognition and synthesis control of AAMs from fly ash. By tactfully combined the AAR kinetics study with XRD, SEM, and TEM-EDX investigations, different calcic phases at different AAR periods were well detected in this study, which has given important indicative information for the clearness of calcium phase evolution. Based on that, a reasonable mechanism in terms of fly ash calcic phase evolution process was proposed in this paper.

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1. Introduction

Chemical alkali activation technology is an advanced and effective technique for processing of many industrial solid wastes (fly ash, slag, etc.), since alkali-activated materials (AAMs) usually possess excellent mechanical and environmental friendly properties [1]. Similar to calcium silicate hydrate (C-S-H) gel as the key-strength-giving component of cement based materials [2], alkali aluminosilicate hydrate (N,C-A-S-H, N=Na, C=Ca) gels are well recognized to be that of AAMs [3]. For high calcium precursors (such as Class C fly ash, blast furnace slag), C-A-S-H gel was demonstrated to be the main product of AAMs, whereas low calcium precursor favors the N-A-S-H gel [4,5]. Therefore, calcic phases play the lead role in determining the physical properties of AAMs from high calcium content materials, the clearness of calcic phase evolution during alkali activation reaction (AAR) will be very significant and essential for both cognitive level and application fields of AAMs.

On the other hand, circulating fluidized bed combustion fly ash (CFA) is a kind of prevailing but low reactive fly ash, mainly due to its unique thermal history and desulphurization process [6,7]. Unlike conventional pulverized coal combustion fly ash (PFA), the calcium constituent of CFA mainly exist in the form of crystalline CaSO_4 [7,8]. Hence, the CFA calcium constituent phase evolution will be quite distinguishing from that of PFA during AAR. To the

author's knowledge, there's no article available was on that so far.

Here for the first time, the calcium constituent phase evolution from CFA was studied and time-resolved calcic phases, including crystals and amorphous phases, were well observed at typical AAR periods, giving clear indication for the calcium constituent phase evolution processes. At last, a conducive mechanism about the calcic phase evolution during AAR was presented in this study.

2. Experimental

The high calcium raw materials used in this study was CFA with mean particle size of $13.3\ \mu\text{m}$, and its chemical composition in terms of main oxides were as follows: 42.1% SiO_2 , 31.3% Al_2O_3 , 13.1% CaO , 6.3% SO_3 , and 4.2% Fe_2O_3 . Under the premise that: i) the total molar ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3=3.8$ and the net concentration of OH^- is 4 M [9]; ii) residual hetero-ions (mainly Na^+) will be washed out from AAR product system; it is feasible to make high liquid-solid ratio alkali activated CFA slurry with high fluidity in this study, which will be of great convenience for timely cleaning of hetero-ions. Hence, via calculation, Water glass (molar ratio $\text{SiO}_2/\text{Na}_2\text{O}=2.4$) and sodium hydroxide (A.R., > 99% pure), and de-ionized water were mixed together by mass ratio of 1.9:1:5 to form the alkaline activator (AA) in this study, while the mix ratio of AA/CFA (by weight) was 2.67.

This study firstly investigated the AAR isothermal heat flow curves of CFA at $60\ ^\circ\text{C}$ for the initial 24 h. Measurement was performed by a Mettler TG/DSC I simultaneous thermal analyzer and

* Corresponding author.

E-mail address: lzk1227@sina.com (Z. Liu).

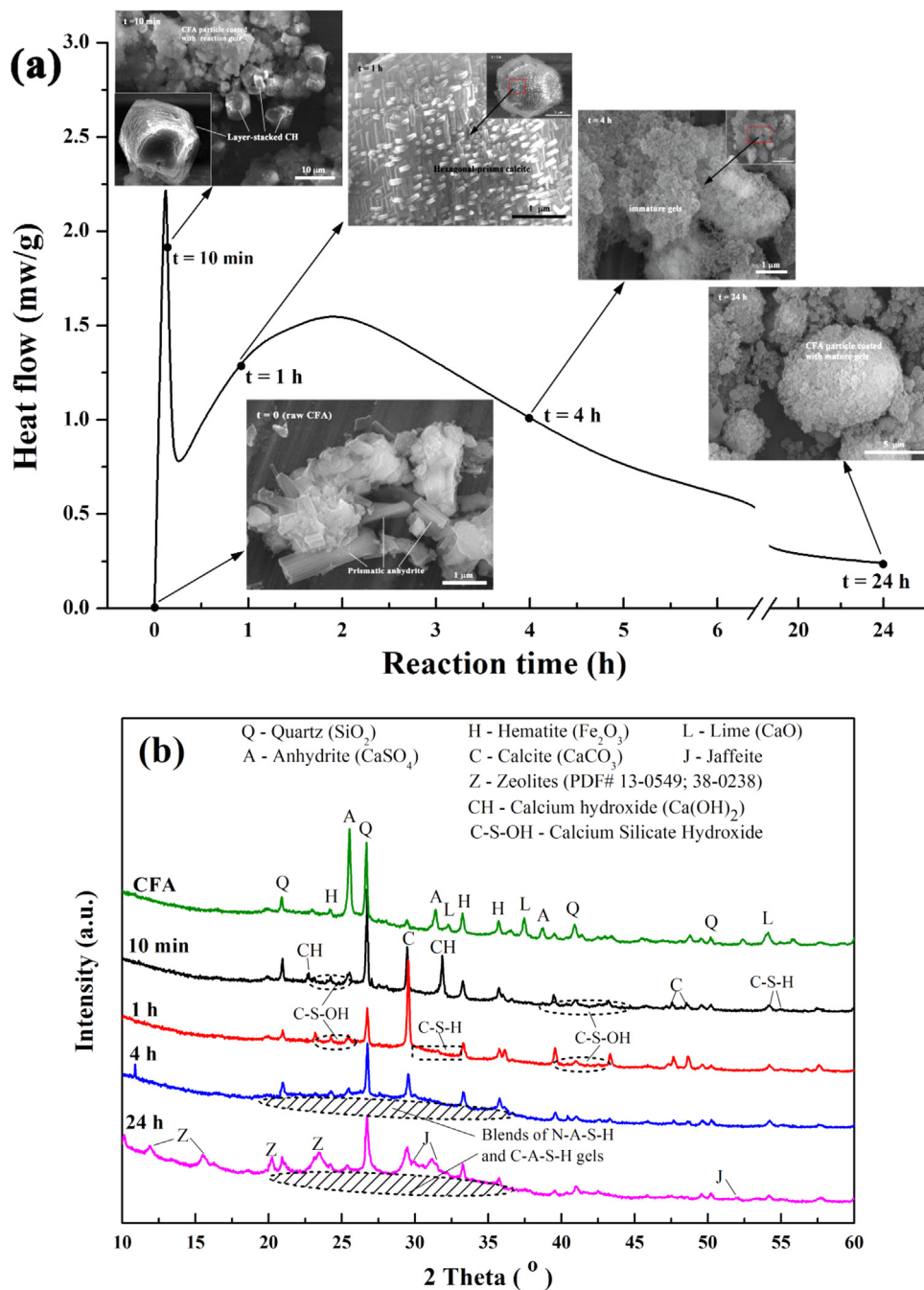


Fig. 1. (a) Alkali activated CFA reaction heat flow at 60 °C along with typical SEM images of AAFAM observed at corresponding chosen time; (b) XRD observation of AAFAM at the chosen time.

the obtained heat flow curve is shown in Fig. 1a. Thus, $t=10$ min, 1 h, 4 h, and 24 h were chosen as the typical time points of AAR different periods for the calcium phase evolution study. The next-step experimental procedures were as follows: 1) the AA was firstly heated up to 60 °C to ensure the starting reaction temperature be 60 °C; 2) rapidly mixed the CFA with AA in a beaker, record the right reaction time as $t=0$, and immediately transfer the beaker into a 60 °C oven; 3) whenever the reaction time arrived at chosen points ($t=10$ min, 1 h, 4 h, or 24 h), a portion of AAR matrix would be drawn out from the whole system and immediately washed and separated with sufficient de-ionized water by a centrifugal machine until the supernatant pH < 8, the AAR would be basically terminated after this procedure; 4) cast away the supernatant and soak the residual with anhydrous ethanol for

1 day to further terminate AAR. After that, drying the residual and then get the intermediate product of AAR corresponding to the chosen time points.

At last, the obtained intermediate products were conducted for further characterization. The morphology was determined using a scanning electron microscope (SEM, JEOL JSM-7001F) and Transmission electron microscopy (TEM) was performed by a field emission transmission electron microscope (FEI Tecnai G2 F30) equipped with an Energy Dispersive X-Ray Spectroscopy (EDX). The TEM-EDX was done in area mode and the detection results at each reaction time were the average from three chosen areas. Moreover, X-ray diffraction (XRD) patterns were recorded using an X'TRA high-performance powder X-ray diffractometer (Smartlab 9000) with Cu K α radiation generated at 200 mA and 40 kV, and

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