



# Correlation between hydration of cement and durability of natural fiber-reinforced cement composites



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## ABSTRACT

The influence of cement hydration on the durability of natural fiber-reinforced cement composites and the deterioration of the embedded natural fibers were investigated by incorporating four supplementary cementitious materials (SCMs). Cement hydration was presented to be a crucial factor in understanding natural fiber degradation behavior. The results indicated that the incorporation of SCMs slowed down both mineralization and alkaline hydrolysis of fiber cell walls by promoting the hydration of cement. This is attributed to the reduced alkalinity of pore solution and calcium hydroxide content. Degradation of natural fiber was significantly mitigated and the durability of cement composites was improved consequently.

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## 1. Introduction

Cement based composite materials, especially concrete, play an important role in the history of human development in the last 2000 years. Attributed to excellent compressive strength, low-cost, long service life and relatively low maintenance requirements, cement composites have already been applied in various infrastructures. The main disadvantages of cement composites are their low tensile strength, cracking resistance and fracture toughness. Steel reinforcing bars and various fibers, such as steel fiber, mineral fibers, glass fiber and plastic fibers, have been used to reinforce mortar and concrete to control the initiation and growth of microcracks [1]. The desirable improvements of tensile behavior, flexural strength and toughness, and ductility can all be realized by using fiber reinforcement. The increase in fracture energy is caused by the fiber itself and the bond between the fiber and matrix. To meet the requirement of sustainable development of construction materials, natural fibers attract more attention as reinforcement of cementitious materials.

Owing to its high tensile strength, high modulus and low cost, sisal fiber has proven to be a suitable natural reinforcement to cement based materials. It is well known that natural fiber consists of three main components: cellulose, hemicellulose and lignin

[2–5]. Cellulose, which is a kind of linear hemi-crystal phase is the main structural components for natural fiber [6]. Hemicellulose and lignin are amorphous phases and are binding phases for cellulose [5,7]. The distinct cell structure endows sisal fiber excellent mechanical properties with low density, meanwhile it is also the reason for natural fiber's poor durability in alkaline environments, such as the cement matrix. When exposed to alkaline pore solution and the mineral-rich environment of the cement matrix, natural fiber will experience severe degradation and will become brittle due to two aging mechanisms: alkaline hydrolysis and cell wall mineralization. This significantly affect the fiber's reinforcing role in cement composites. Furthermore, cement composites can suffer premature deterioration including a decrease in post-cracking toughness and cracking when they undergo various aging processes.

Sisal fiber degradation can be mitigated through two ways: fiber pretreatment and cement matrix modification. Pretreatment of natural fiber includes chemical, physical and physicochemical methods. Silane coating, hornification, autoclave, sodium silicate, potassium silicate [8–11] have been shown to improve the mechanical properties and durability of natural fiber in cement based materials. Bastidas et al. [12] and Zornoza et al. [13] reported that the corrosion resistance of steel embedded in mortar exposed to chloride can be substantially improved by incorporating fly ash. Moreover, the partial cement replacement by slag [14] and silica fume [15] has also been proven to be an effective way to mitigate the corrosion of rebar in concrete. So it can be anticipated

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that the aging behavior of natural reinforcements can be arrested through cement modification using these supplementary materials. Tolêdo's work indicates that natural fiber degradation can be effectively mitigated by producing a calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) free cement matrix through high volume cement replacement with calcined clay (metakaolin (MK) and calcined waste crushed clay brick) [16–18] or MK [19]. Ground granulated blast furnace slag [20,21], silica fume, fly ash, and MK [22,23] have similar effects. In the author's previous work, both fiber pretreatment and cement matrix modification by using various supplementary cementitious materials, such as metakaoline (MK), nanoclay (NC), limestone (LS), rice husk ash (RHA), fly ash (FA) and diatomite have been investigated. The results indicate that both of the two methods can effectively arrest the natural fiber's degradation. However, pretreatment of natural fiber may require more effort, can increase cost, and needs to consider the compatibility between modifying agents and the cement matrix, as well as its effect on the interfacial properties of fiber-cement. Therefore, it is more logical to improve the initial mechanical properties and durability of natural fiber-reinforced cement composites through modifying the hydration of cement.

Although cement matrix modification promises to be an effective way to improve the durability of natural fiber in cement. The modification mechanisms of these SCMs in cement and correlation between cement hydration and fiber degradation, which are the foremost objectives of this study, are not yet fully understood. In this study, in order to get the cement matrices with different degrees of hydration and to investigate the influence of the alkalinity of pore solution on fiber degradation behavior, four SCMs, such as MK, RHA, silica fume (SF) and FA, were selected to partially replace cement with different mixing amounts. Given the differences in their chemical composition, particle size and pozzolanic activity, the cement substitution level was adjusted for each SCM to ensure the diversity in cement hydration. The effect of these SCMs on cement hydration kinetics and hydration products, as well as their effect on durability of natural fiber-reinforced cement composites were investigated. The correlation between degree of cement hydration and natural fiber degradation behavior was also determined.

## 2. Experimental

### 2.1. Raw materials

The ASTM Type I Portland cement (PC), used in this paper, with a Blaine fineness of  $385 \text{ m}^2/\text{kg}$ , was sourced from Lafarge North America Inc., Whitehall, PA, USA. Aggregate used to prepare mortar specimens consisted of natural river sand that had a maximum aggregate size of 4.7 mm, specific gravity of 2.6 and fineness modulus of 2.8. Metakaolin (MK) was sourced from Pivorpozz Inc. Rice husk ash was supplied by Agrilectric Research Co., Lake Charles, LA. For comparison, SF and FA were also incorporated into cement as SCM to investigate their effects on cement hydration and fiber degradation. The chemical compositions of cement and SCMs determined by means of X-ray fluorescence (XRF) are summarized in Table 1.

From Table 1, it can be seen that MK has a higher aluminate content than other SCMs. So it can be predicted that MK will enhance the hydration of cement more significantly at early age. The silicate mineral contents of RHA and SF are much higher than that of other SCMs. FA, which is a kind of high-calcium Class C fly ash shows higher lime content than SF. The silicate content of FA is lower than other SCM, but it has a higher aluminate amount than SF and RHA.

The particle size distributions (PSD) of cement and the supplementary cementitious materials (SCM) determined by means

**Table 1**  
Chemical composition of the used materials.

| Compositions                   | Cement | MK     | RHA   | SF     | FA    |
|--------------------------------|--------|--------|-------|--------|-------|
| CaO                            | 63.7   | 0.0707 | 0.51  | 4.89   | 27.31 |
| SiO <sub>2</sub>               | 12.9   | 51.8   | 90.45 | 92.3   | 32.25 |
| Fe <sub>2</sub> O <sub>3</sub> | 7.97   | 4.15   | 0.015 | 0.84   | 6.85  |
| SO <sub>3</sub>                | 5.25   | 0.105  | 0.037 | 0.30   | 2.82  |
| Al <sub>2</sub> O <sub>3</sub> | 4.14   | 42.4   | 0.016 | 0.665  | 17.41 |
| MgO                            | 3.50   | –      | 0.24  | 0.525  | 5.32  |
| SrO                            | 0.915  | 0.0397 | –     | 0.0398 | 0.51  |
| K <sub>2</sub> O               | 0.907  | 0.218  | 1.67  | 0.263  | 0.37  |
| Na <sub>2</sub> O              | –      | –      | 0.031 | –      | 1.67  |
| TiO <sub>2</sub>               | 0.279  | 1.07   | 0.009 | –      | 1.52  |
| ZnO                            | 0.251  | –      | –     | 0.105  | 0.11  |
| ZrO <sub>2</sub>               | 0.119  | 0.0884 | –     | –      | 0.15  |
| P <sub>2</sub> O <sub>5</sub>  | –      | –      | 0.76  | –      | 0.52  |
| MnO                            | –      | –      | 0.07  | –      | 0.10  |
| Cl                             | –      | 0.0457 | –     | 0.0465 | –     |
| C                              | –      | –      | 6.19  | –      | 3.09  |
| Other                          | 1.21   | 0.0125 | 0     | 0.0257 | 0     |

of laser diffraction are shown in Fig. 1. The median particle size of cement ( $D_{50}$ ) is  $9.57 \mu\text{m}$  and its surface weighted mean (the diameter of a sphere that has the same surface area ratio as a particle of interest) and volume weighted mean (the diameter of a sphere that has the same volume ratio as a particle of interest) are  $3.5 \mu\text{m}$  and  $12.3 \mu\text{m}$ , respectively. MK shows a higher fraction of particles with a diameter smaller than  $5 \mu\text{m}$  compared to PC. The Brunauer–Emmett–Teller (BET) specific surface area (surface areas of solids by physical adsorption of gas molecules based on BET multi layer adsorption theory; the specific surface area is obtained from total surface area divided by mass) of MK ( $2.93 \text{ m}^2/\text{g}$ ) is higher than that of PC ( $1.7 \text{ m}^2/\text{g}$ ), which means it can modify the cement matrix in both hydration products and microstructure as pozzolanic substitution material and fine filler, respectively. Although having a similar SiO<sub>2</sub> content, RHA's median particle size and surface area are 8.93 times and 24.02% of those of SF, respectively. It can be anticipated that SF will improve cement hydration and consume  $\text{Ca}(\text{OH})_2$  more effectively than RHA, and consequently enhance the durability of natural fiber-reinforced cement composites. However, the distinct porous structure of RHA (Fig. 2-a) endows it a great potential to serve as external water carrier to realize the internal curing, which is beneficial to enhance the hydration of cement. The particle size distribution of FA is more decentralized than those of other SCMs. A median particle size of  $13.2 \mu\text{m}$  was obtained from FA, with a BET-specific surface areas of  $1.46 \text{ m}^2/\text{g}$ .

Sisal fibers produced from Tanzania, with an average diameter of  $202.5 \mu\text{m}$  and a tensile strength of 605 MPa, was provided by Bast Fibers LLC of Creskill, New Jersey. Fig. 2b shows the microstructures of the surface of a sisal fiber, respectively. The fiber's rough surface is beneficial to enhance interfacial bonding properties between the fiber and cement matrix, but it also provides space for the precipitation of cement hydration products on fiber surface.

### 2.2. Mixture proportion and specimens preparation

Pure cement matrix and blends with cement substitutions by MK, RHA, SF and FA at 30 wt.% (MK30), 20 wt.% (RHA20), 10 wt.% (SF10), and 30 wt.% (FA30) levels, respectively, were investigated in this study. A water/cementitious materials/sand ratio of 0.4:1:1 was used for all mortar mixtures. If necessary, the workability was adjusted when necessary using ADVA 408 superplasticizer up to 3 wt.% of binder, which is not enough to significantly impact the hydration kinetics of cement [24]. The cement pastes were mixed with gradually added sand in a mechanical mortar mixer at 60 rpm for 2 min followed by a 1 min rest and 3 min of further mixing at 120 rpm. Cement pastes prepared for chemical analysis were cast in sealed bottles and then stored at  $23 \pm 2^\circ\text{C}$  until to be tested.

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