



Effects of calcined layered double hydroxides on carbonation of concrete containing fly ash



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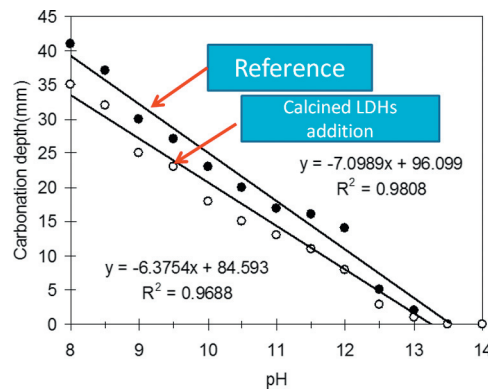
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HIGHLIGHTS

- Carbonation resistance of fly ash blended concrete was investigated.
- LDHs catch CO₂ and fix them in concrete.
- LDHs enhances the carbonation resistance of fly ash blended concrete.
- Carbonation depths were determined and assessed using different methods.
- A linear relationship was found between carbonation depth and pH values of pore solution.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 9 February 2017

Received in revised form 4 June 2017

Accepted 17 November 2017

Keywords:

Layered double hydroxides
Carbonation
Pore solution
Fly ash

ABSTRACT

Carbonation resistance of fly ash blended concrete incorporating LDHs was investigated. X-ray diffraction, IR-spectroscopy and thermal analysis were employed to characterize the component and structural changes of LDHs and cement paste before and after carbonation. Carbonation depths were assessed using the TG-DSC method combining the results obtained from phenolphthalein indicator. The presence of C–O as a basis for determining the presence of CaCO₃ was detected using IR-spectroscopy. The relationships between pH values of pore solution and carbonation depth were discussed.

The experimental results show that LDHs can catch CO₂ and fix them in concrete. LDHs, especially the calcined LDHs, enhances the carbonation resistance of concrete. Test results of TG-DSC indicate that there exists a sharp carbonation boundary. The TG-DSC result uncovers that the carbonation depth is twice of that determined by phenolphthalein indicator. A linear relationship was found between the carbonation depth and pH values of pore solution.

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1. Introduction

Reinforcing bars in concrete are protected against corrosion by a thin oxide and hydroxide layer, which forms on the steel surface

because of the high pH value (pH ≥ 13) of the pore solution in concrete. Corrosion may start when this protective layer is destroyed due to the decrease of pH value. Such a reduction in alkalinity can be ascribed to the result of Ca(OH)₂ carbonation. Carbonation is known as neutralizing process, the chemical reaction of Ca(OH)₂ and calcium silicate hydrate (C–S–H) with CO₂ to form CaCO₃ and water [1]. Carbonation reduces the hydroxide concentration in the pore solution and leads to the destroy of passivity

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layer on the surface of embedded reinforcement bars [2]. The traditional way of determining the depth of carbonation is to spray phenolphthalein indicator onto the surface of a freshly split prism concrete specimen. With the development of the concrete technology, fly ash has become one of the main components of modern concrete in recent years [3]. The secondary hydration of fly ash would consume the $\text{Ca}(\text{OH})_2$, which reduces anti-carbonation of concrete, and leads to decrease in alkalinity and then affects the durability of concrete [4]. Therefore, anti-carbonation was the key factor which restricts the development of fly ash concrete.

In urban and industrial areas, where environmental pollution results in a significant increasing in concentration of CO_2 , carbonation-initiated reinforcement corrosion prevails. Therefore, research has been currently conducted at various institutions to develop new and improved construction materials, rehabilitation and repair technologies, and a better understanding of the physical and chemical mechanisms that lead to deterioration. The improved knowledge will enable designers not only to properly rehabilitate and maintain the current concrete structures, but also to improve the durability of future structures by giving, in the design stage, proper consideration to the exposure environment and conditions. One of the most important strategy is how to control the carbonation process, therefore, establishing and developing new technology for inhibiting carbonation reaction of concrete materials is the guarantee of favorable durability of modern concrete materials contains mineral admixtures such as fly ash.

Among the group of minerals referred to as ‘non-silicate oxides and hydroxides’ [5], the ‘layered double hydroxides’ (LDHs) have physical and chemical properties that are surprisingly similar to those of clay minerals. Their layered structure, wide chemical compositions (due to variable isomorphous substitution of metallic cations), variable layer charge density, ion-exchange properties, reactive interlayer space, swelling in water, and rheological and colloidal properties lead to the wide applications of LDHs. LDHs were referred to as ‘anionic clays’ due to their anion-exchange properties.

Naturally occurring hydrotalcite, $\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_3 \cdot 4\text{H}_2\text{O}$, and synthetic hydrotalcite-like compounds, also called layered double hydroxides (LDHs), have been investigated for many years [6,7]. Taylor [8] described these phases as being structurally related to brucite. The formula of the LDH can be generalized to $[\text{M}_{1-x}^{2+}\text{M}_x^{3+}(\text{OH})_2]^{x+}[\text{A}_{x/n}^{n-}\text{mH}_2\text{O}]^{x-}$, where M^{2+} can be Mg^{2+} , Zn^{2+} , Mn^{2+} , Ca^{2+} , etc.; M^{3+} : Al^{3+} , Ga^{3+} , Fe^{3+} , Cr^{3+} , etc.; and A^{n-} : NO_3^- , Cl^- , CO_3^{2-} , SO_4^{2-} , etc. [9].

The high anion exchange capacity of LDHs-like materials makes their interlayer ion exchanged by organic and inorganic anions versatile and easily achieved [10–12]. LDHs have been studied extensively for a wide range of applications including catalysts [13,14], ceramic precursors [15] adsorbents [16], bio-organic nanohybrids [17,18], and scavengers of pollutant metals and anions [19]. Recent research has shown great flexibility of the LDHs-like materials in tailoring the chemical and physical properties of materials to be used for specific applications, e.g., molecular recognition, optical storage, batteries, etc. [20–22].

LDHs exhibit a selectivity for the anions in the sequence $\text{CO}_3^{2-} > \text{SO}_4^{2-} > \text{HPO}_4^{2-} > \text{F}^- > \text{Cl}^- > \text{B}(\text{OH})_4^- > \text{NO}_3^-$, and therefore, LDHs-like compounds can be used as host materials for a variety of anions of interest to cement scientists such as CO_3^{2-} . LDHs-like materials could be used in cement and concrete for their CO_3^{2-} capturing capacity and to enhance carbonation resistance of concrete.

This study aims to catch carbonate ions in concrete and to fix them in intercalation layers of LDHs, in order to improve carbonation resistance by means of anion-exchange and structure reconstruction of the intercalated layered materials. This work also compares the carbonation depths determined by the TG–DSC and FTIR with the results obtained using the phenolphthalein indicator.

The relationships between the distribution of pH values and the carbonation depth in carbonated fly ash blended concrete are also discussed.

2. Experimental

2.1. Materials

Portland cement (CEM I 42.5) (relative density 3100 kg/m^3 , specific surface area $369.6 \text{ m}^2/\text{kg}$) was used as binder in the work. Fly ash was used as mineral admixture. Their chemical compositions were shown in Table 1. The mineral composition of cement was provided in Fig. 1. The fine aggregate used was river sand with specific gravity, absorption (%) and fineness modulus 2.63 g/cm^3 , 2.41 and 2.2, respectively. The coarse aggregate used was crushed basalt with nominal grain size of 18 mm, the specific gravity were 2.65 g/cm^3 . The density and particle size of starting materials were listed in Table 2. In addition, polycarboxylates superplasticizer (density 1.08 g/mL , solid content 20%) was used to attain the required workability. Fresh water (tap water) was used in this study as mixing water. Table 3 presents the proportions of constituents in the mix for a water–binder ratio of 0.5.

The syntheses of Mg–Al– CO_3 type hydrotalcite were performed experimentally. The different series of LDHs were treated as follows: (1) O-LDHs: raw LDHs was dried at $105 \text{ }^\circ\text{C}$ for 24 h in the oven; (2) C-LDHs: LDHs was calcined at $600 \text{ }^\circ\text{C}$ for 4 h with a heating rate of $3 \text{ }^\circ\text{C}/\text{min}$ from ambient temperature, and cooled naturally to ambient temperature; and (3) R-LDHs: calcined LDHs was mixed with saturated calcium hydroxide solution, and stirred continuously for 24 h under nitrogen gas flow, after which the solid precipitate was collected by filtration and dried at $105 \text{ }^\circ\text{C}$ for 24 h in vacuum.

Content of LDHs was kept at constant 2% by weight of binder, four kinds of specimens were reference sample (MREF), concrete with 2% O-LDHs (MO-2), concrete with 2% C-LDHs (MC-2) and concrete with 2% R-LDHs (MR-2), respectively.

2.2. Casting and curing

The mixing sequence employed consists of dry mixing of the fine and coarse aggregate in the laboratory pan mixer for 30 s and then continued mixing for another 30 s by adding binders including cement, fly ash and LDHs. After ensuring proper mixing of all the solid components, polycarboxylates superplasticizer was added into water and the addition of liquids including water and superplasticizer into the dry solids including aggregates and binders was completed during 30 s. Finally, the mixing between the liquids and solids was continued for further 3 min.

Specimens with the dimensions of $100 \text{ mm} \times 100 \text{ mm} \times 400 \text{ mm}$ were mixed in a pan mixer and then cast in moulds for various tests. The specimens surfaces were covered with polyethylene sheets to prevent loss of moisture. After 24 h, all concrete specimens were demoulded and cured at the temperature of $20 \pm 2 \text{ }^\circ\text{C}$ and the relative humidity of 95% until the testing days.

2.3. Test procedures

2.3.1. Accelerated carbonation

In normal environments (which contain 0.03% to 0.1% CO_2), the evolution of the carbonation depth with time is extremely slow, the carbonation test was done inside a chamber with a CO_2 concentration $\approx 20\% \pm 3\%$, $\text{RH} \approx 70\% \pm 5\%$, and $T \approx 20 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$. Tests were carried out at 56 days of exposure durations. Concrete specimens were used for carbonation property test. After cured in standard curing conditions ($\text{RH} \geq 90\%$, $T = 20 \pm 3 \text{ }^\circ\text{C}$) for 26 days,

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