



Research paper

Role of layered double hydroxides in setting, hydration degree, microstructure and compressive strength of cement paste

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ABSTRACT

The effects of calcined Mg-Al-CO₃ LDH on setting time, hydration degree, microstructure as well as compressive strength of cement pastes were explored. Furthermore, the role of LDH in cement system has been revealed by characterizing the component and structural changes of LDH. The experimental results show that LDH is physically and chemically active in cement paste. 1–3% calcined LDH accelerates the hardening process and improve the setting of cement to some extent. LDH incorporation leads to the slight increasing in non evaporable water content, and the increasing ratio is less than 1% at each curing age. The hydration heat for LDH blended specimens are less than that of control cement because of the dilution effect. LDH accelerates the hydration reaction to some extent even though it is not a dramatic change. LDH has refine effect on pore structure of cement paste and the addition level of LDH should be no less than 2%. Cement pastes with LDH exhibits increased compressive strength regardless of curing time, and the increase rate at 28 days is lower than that obtained at early ages. Fulfillment of structure regeneration of calcined LDH in cement paste environment was also been revealed.

1. Introduction

It is acknowledged that concrete is one of the most used building materials due to a global production of around 10,000 million tons per year (Hasanbeigi et al., 2012; Faella et al., 2016). The concrete production requests huge amounts of energy and fuel consuming (Hasanbeigi et al., 2012; Faella et al., 2016; Meyer, 2009; Jakobsen et al., 2017). In addition, there are CO₂ emissions during the manufacture process with a large volume of raw materials required to produce concrete each year. Therefore, the durability and service life become important issues for concrete materials and structures, its deterioration and degradation over time due to sulfate attack (Albitar et al., 2017), chloride permeability (Shaheen and Pradhan, 2017) and carbonation (Liu et al., 2016) has been widely observed and documented. Concrete durability issues are the major cause of concern all over the world especially in the marine environment, where chloride and sulfate salts exist simultaneously and the structures are undergoing deterioration before their expected life (Moffatt et al., 2017; Valipour et al., 2017).

Consequently, concrete sustainability continues to be of importance to the construction industry. The use of supplementary cementitious

materials (SCMs) in concrete is an important part of existing strategies to improve durability performance, maximum the service life and sustainability (Medina et al., 2017; Zhang et al., 2016; Yang et al., 2015; Labbaci et al., 2017). The SCMs, rich in silicon and aluminum, are widely used as pozzolanic materials and have several advantages including reducing permeability and harm chemical reactions, improving the strength and durability (Evi, 2017). Most of previously published work on blended cement or concrete systems including SCMs focuses on mechanical or durability aspects (Lothenbach et al., 2011). The use of SCMs in concrete is often driven by economics and improvements in the long-term mechanical properties and durability. Usually, the impetus to replace cement with SCMs comes from pressure on the industry to reduce CO₂ emissions from concrete production. Often the high volume cement replacements result in losses in performance at early ages, and thus driving research into balancing sustainability and performance (Juenger and Siddique, 2015).

Therefore, research is currently carried out to develop new modification materials, and better understanding of the physical and chemical mechanisms involve in cement hydration (Eren et al., 2017; Anju et al., 2016; Zhao et al., 2016; Zhou et al., 2017). How to control the deterioration process induced by chemical corrosion by using new

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modification material is the guarantee of good durability of modern concrete containing large amounts of mineral admixtures or SCMs.

Layered double hydroxides (LDH) have been investigated extensively and have shown potential for a number of applications include adsorbents, anion exchangers, medical applications as well as catalysts (Theiss et al., 2016). The formula of the LDH can be generalized to $[M_1^{2+}M_x^{3+}(\text{OH})_2]^{x+}[A_{x/n}^{n-}m\text{H}_2\text{O}]^{x-}$, 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^- , OH^- , CO_3^{2-} , SO_4^{2-} , etc. (Qu et al., 2016).

The structure of LDH is basically built up from the parent structure brucite $\text{Mg}(\text{OH})_2$ by isomorphous substitution of divalent cations. The substitution of divalent Mg^{2+} by trivalent M^{3+} carry net positive charge, which has to be balanced by anions intercalated such as NO_3^- , Cl^- , CO_3^{2-} and SO_4^{2-} along with structural water into the interlayer regions.

LDH/hydrotalcite has ability to exchange cations and anions. This ability was investigated in two areas: the first is using hydrotalcite as sorbent for cation contaminants, e.g. Mg/Fe hydrotalcite (Gasser et al., 2016), and the second as cement additive during the immobilizing hazardous wastes, e.g. Bentonite (Reddy et al., 2010). The reconstruction of the original LDH structure can be achieved by hydration of the calcined LDH. This unique property is ascribable to structural memory effect. LDH can take up a variety of contaminants and toxic substances directly from the environment through anion-exchange and reconstruction. Capturing target anions can be attained by simple anion exchange in solution or by reconstruction of calcined LDH.

The high anion exchange capacity of LDH ensures the fulfillment of interlayer ions exchanged by other anions such as the ions in cement pore solution. Therefore, LDH can be used as host materials for a variety of anions of interest to cement and concrete. Analysis of published literatures indicates that utilization of LDH in cement and concrete receives more attention and interest from researchers. Recent research has shown synthetic $\text{Ca}-\text{Al}-\text{NO}_3$ LDH could be used for the immobilization of chloride ions from pore solution of cementitious materials due to the direct anion exchange of nitrate in the interlayer of LDH by chloride in aqueous solution (Chen et al., 2015). Yoon et al. (2014) founded that calcined LDH not only adsorbed chloride ions in aqueous solution with a memory effect but also had a much higher binding capacity than the original LDH in the cement matrix. Calcined LDH rebuilt the layered structure in cementitious environment and demonstrated the feasibility of applying LDH to the cement and concrete materials. Chen et al. (2016) also revealed the effect of MgO on the formation of LDH and chemical resistance of slag-fly ash blended cement and they believed that sulfate resistance of slag-fly ash-clinker system under wetting-drying cycles increased with MgO content of synthetic slag due to the favored formation of LDH and hence the immobilization of sulfate. Ke et al. (2016) reported that the inclusion of calcined LDH in Na_2CO_3 -activated cements accelerated the reaction, and promoted hardening within 24 h and they concluded that the effectiveness of calcined LDH was associated with removal of dissolved CO_3^{2-} from the fresh cement. Our previously published work (Duan et al., 2013) also indicated that carbonation depth of concrete decreased notably with the addition of LDH, especially the calcinated LDH due to the exchange and immobilization of carbonate ions by LDH. Xu et al. (2009) proposed CaAl -layered double hydroxide as potential concrete hardening accelerators as it exhibited great enhancement in respect of early mechanical properties with addition of LDH. They

found that LDH accelerated the precipitation rate of the main cement hydration products, and thus speeding up hydration. Guan et al. (2016) founded that LiAl -LDH with 3D micro-nano structures led to faster hydration rate, shorter setting time and higher early compressive strength of calcium sulphoaluminate cement clinker. Yang et al. (2014) observed that ion exchange occurred between free chlorides in simulated concrete pore solution and synthesized $\text{CaAl}-\text{NO}_3$ hydrotalcite by coprecipitation method, thereby reducing the free chloride concentration and increasing binding of chloride present in concrete.

However, the understanding of LDH in cement paste is presently hampered by: 1) scarce information on hydration degree with addition of LDH, and 2) lack of detailed knowledge of microstructure changes. A brief summary of the previously published findings reveals that few studies focus on the effects of LDH on the setting, hydration degree and microstructure, and the mechanisms are even not well known. Little information is available about three-dimensional microstructure of cement-LDH system. Furthermore, scarce research has been carried out to use LDH to change the compressive strength of cement paste. Information of effects of LDH addition on hydration of cement paste still requires further investigation.

This study aims to explore the effects of LDH on setting time, hydration degree, micro-analysis as well as compressive strength of cement pastes by means of structure regeneration. Addition level of cement by the employed calcined $\text{Mg}-\text{Al}-\text{CO}_3$ LDH ranges from 1% to 3% with an interval of 1% at a constant water/binder (w/b) ratio of 0.4. Furthermore, the mechanism of the role of LDH in cement system has been revealed. The obtained findings are expected not only to uncover the role of LDH in cement, but also to develop a new kind of modification material for cement hydration.

2. Experimental

2.1. Materials

In this work, Portland cement (CEM I 52.5) (relative density 3100 kg/m^3 , specific surface area 369 m^2/kg) was used as binder. Cement paste samples were prepared for various test and micro-analysis with w/b ratio of 0.5.

$\text{Mg}-\text{Al}-\text{CO}_3$ LDH with average particle size of 15 μm is used in the experiment. $\text{Mg}-\text{Al}-\text{CO}_3$ hydrotalcites was synthesized using a conventional coprecipitation method. A salt solution (200 mL) containing appropriate ratios of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (1.2 mol/L) and $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.4 mol/L) was created. Solution (200 mL) containing NaOH and Na_2CO_3 , at sufficient concentration was also created to precipitate the salt in the first solution. The two solutions were simultaneously added drop by drop into a 200 mL sample of deionized water, which were vigorously stirred. The temperature was fixed at 313 K, and the pH was maintained at 10–11. The resulting slurry was allowed to rest at 338 K for 12 h. The final precipitate was centrifuged several times with deionized water, until the superstratum water was free of Cl^- . The precipitate was then dried at 378 K for 8 h to obtain the $\text{Mg}-\text{Al}-\text{CO}_3$ type hydrotalcite powder.

The chemical compositions of cement and LDH as well as calcined LDH were listed in Table 1. The physical properties of cement were provided in Table 2. The average particle size for cement and calcined LDH are 37.5 μm and 9.5 μm , respectively.

According to the literature (Vagvolgyi et al., 2008), the thermal

Table 1
The chemical compositions of LDH and cement (wt%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	SO_3	K_2O	LOI
LDH	0.02	21.75	/	/	35.66	/	0.07	/	42.02
Calcined LDH	0.05	36.72	/	/	57.27	/	1.02	/	4.17
Cement	21.35	4.67	3.31	64.60	3.08	0.15	0.85	0.34	0.95

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