

Research paper

The influence of aluminum from metakaolin on chemical binding of chloride ions in hydrated cement pastes

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ABSTRACT

In this study, chloride ions binding capacity of Portland cement blended with 10–30% of metakaolin was investigated. Chemical binding of chloride ions was found depending more on phase assemblage of pastes rather than merely on available aluminum content in the blends. More monosulfoaluminate formed at low replacement levels, while at high replacement levels of metakaolin, strätlingite formation in the blends was favored. When exposed to 0.53 M NaCl solution at ambient temperature, monosulfoaluminate converted to Friedel's salt completely, and played a principal role in chemical binding of chloride ions while ettringite and strätlingite did not participate in the chemical binding of chloride ions. Considering the exposure condition, the contribution of monocarboaluminate in chemical binding was less significant than that of monosulfoaluminate. Results demonstrated that well formulated Portland cement-metakaolin blends with appreciate metakaolin replacement level and sulfate supply are expected to have good chloride binding capacity.

1. Introduction

Chloride ion is the main cause of deterioration of reinforced concrete structures exposed to marine environments and de-icing salts. Some of the chloride ions that penetrate into concrete are held by hydration products, this is called chloride binding. Chloride binding has a significant effect on chloride penetration and on the time to corrosion initiation (Martin-Perez et al., 2000). Chloride binding reduces the free chloride concentration in the vicinity of the reinforcing steel bars which will reduce the chance of corrosion; removes chloride from the diffusion flux and forms Friedel's salt, thus retarding the penetration of chloride to the surface steel bars (McGrath, 1996; Yuan et al., 2009).

Two types of chloride binding, i.e. chemical binding and physical binding, in cementitious materials are commonly recognized. According to recent published literature (Plusquellec and Nonat, 2016; Zhou et al., 2016; Zhou et al., 2018), physical binding mainly results from accumulation of chloride ions in the diffuse layer of calcium silicate hydrate (C–S–H), and both the Ca/Si ratio of C–S–H and Ca²⁺ concentration near to surfaces of C–S–H particles have important impacts on chloride binding of the phase. Chemical binding is the chemical reaction between chloride ions and aluminum bearing phases to form Friedel's salt (Ca₃Al₂O₆·CaCl₂·10H₂O) or its analogue (Justnes, 1998). The incorporation of alumina-rich supplementary cementitious materials (SCM), fly ash and blast furnace slag for examples, enhances

the chloride binding capacity of cementitious materials efficiently (Arya and Xu, 1995; Dhir et al., 1997; Luo et al., 2003; Thomas et al., 2012), and the enhancement is believed to be a function of the alumina content, because more calcium aluminate hydrate phases derive from the reaction of alumina-rich SCM. However, there are some evidence that points to the contrary, and the effect of the inclusion of alumina-rich SCM on chloride binding is not merely rely on the change in alumina content (Dhir et al., 1997; Zibara et al., 2008).

When industrial byproducts such as fly ash and slag are already short in supply in some parts of the world, heat-treated clay minerals represent a promising source of SCM duo to their high alumina and silica content, lower carbon footprint as compared to Portland cement, and high abundance in the Earth's crust (Shi et al., 2017). Metakaolin (Al₂O₃:2SiO₂) was used as a model compound for a number of years among clay-based SCM, for its high pozzolanic reactivity, purity and easy availability (Dai et al., 2014). The incorporation of metakaolin in Portland cement leads to changes in structure and composition of calcium-silicate-hydrate, i.e. increased Al/Si ratio and aluminosilicate chain length associated with decreased Ca/Si ratio (Dai et al., 2014), consumption of portlandite derived from hydration of cement. Furthermore, the reaction of metakaolin forms additional amounts of AFm phases due to its high aluminum content, and the forming of specific AFm phases depends on available anions in the pore solution (Kunther et al., 2016). In turn, the impacts of metakaolin on AFm phase

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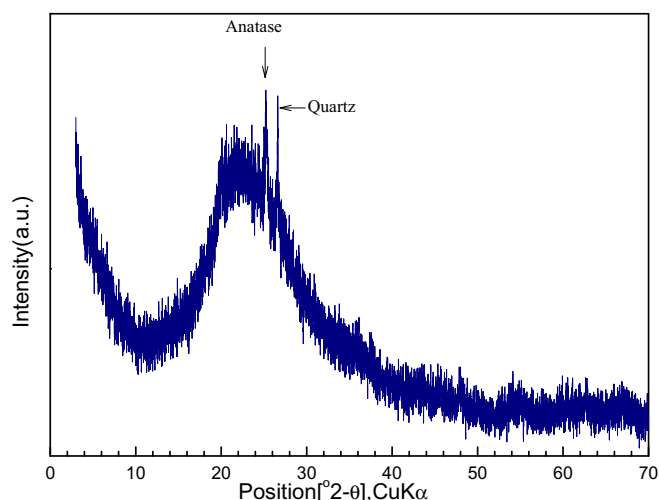


Fig. 1. XRD pattern of used metakaolin.

assemblages have influences on chloride chemical binding.

Thus, the objective of the work is for a better understand on the role of alumina from metakaolin on chemical binding in hydrated cement paste. Influences of metakaolin replacement level on AFm phases assemblage of hydrated Portland cement-metakaolin blends before and after exposed to 0.53 M NaCl solutions are examined experimentally, then the influences are related to chemical binding capacity of chloride of the studied blends to know the exact mechanism through which alumina from metakaolin affects chemical binding capacity of chloride. The reported work is expected to facilitate the application of heat-treated clay minerals in cementitious materials to improve chemical binding capacity of chloride in a more efficient way.

2. Materials and methods

2.1. Materials

Type PI Portland cement (similar to CEM I) and commercial available metakaolin were used for preparing samples used for the experiments. X-ray diffraction pattern of the used metakaolin is given in Fig. 1. Except for minor reflections of residual quartz and anatase present in the metakaolin, a broad reflection characterizes a highly amorphous structure of the used metakaolin. Except for metakaolin, silica fume which contains nearly no alumina, and quartz were selected for comparison purpose. Quartz was used as filler allowing for the same dilution effect, i.e. maintaining the same Portland cement replacement level. Reagent type Sodium Chloride (99% purity) was used to prepare the external solution of 0.53 M. The chemical compositions of cement and SCM used for the experiments are given in Table 1.

2.2. Methods

The experiments involved examining six different blends with different replacement levels. Blends with Portland cement, meta-kaolin, silica fume and quartz at different replacement levels with a water to

Table 1
Chemical composition of materials used (%).

	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	SO ₃	CaO	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	L.O.I
Quartz	1.4	98.0	0.0	0.0	0.0	0.1	0.0	0.0	0.0	–
Silica fume	1.4	97.2	0.0	0.0	0.1	0.3	0.0	0.2	0.0	–
Metakaolin	45.3	51.1	0.7	0.0	0.3	0.1	0.2	0.3	0.0	0.7
Portland cement	4.6	19.1	2.2	2.4	61.2	2.9	0.0	0.8	0.1	1.4

Table 2
Mix formulations of investigated paste mixture.

Mix	W/B ratio	Portland cement (%)	Metakaolin (%)	Silica Fume (%)	Quartz (%)
10MK-OPC	0.5	90.0	10.0	–	–
20MK-OPC	0.5	80.0	20.0	–	–
30MK-OPC	0.5	70.0	30.0	–	–
10SFQ-OPC	0.5	90.0	–	5.3	4.7
20SFQ-OPC	0.5	80.0	–	10.5	9.5
30SFQ-OPC	0.5	70.0	–	15.8	14.2

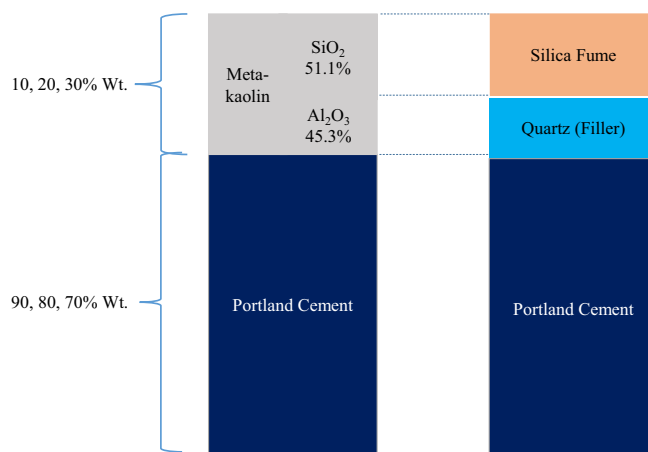


Fig. 2. Schematic illustration of mix formulations of investigated paste mixture.

binder mass ratio (W/B) of 0.5 were used for the experiments. Metakaolin replacement levels of 10%, 20% and 30% were used respectively. In the reference samples, silica fume substituted the SiO₂ content while the alumina content was replaced by quartz to allow for the same dilution effect. Mix formulation of investigated paste mixture is shown in Table 2. The mix names are in the form: the number in the paste name represents the combine SCM replacement level, follow by the initial of SCM and then Portland cement e.g. 10SFQ-OPC means 10% (Silica Fume, and Quartz) replacement level and 90% Portland cement. The schematic for paste mixture is shown in Fig. 2.

All mixes were prepared with distilled water. Prior to mixing, the blends were homogenized for 4 h on a roller powder mixer. Superplasticizer was used for some mixes to get proper fluidity for casting. The pastes were cured in sealed vials (50 mm in diameter) at 20 ± 1 °C in a climate chamber.

After curing for a period of 90 days, the pastes were cut into slices of ~2 mm in thickness. The sliced samples were immersed into isopropanol for 7 days to stop the hydration. According to Thomas (1989), Zhang and Scherer (2011), solvent exchange with isopropanol best preserves the microstructure and least affect the pore size distribution and phase compositions of the samples. The isopropanol was renewed after a period of 24 h' immersion to facilitate the diffusion process. After drying in a desiccator, the slices were broken into smaller fragments. According to work of Thomas et al. (2012), samples weighing about 25 g were placed in a 100 ml cylindrical bottle. The bottle was later filled with 0.53 M NaCl solution, same with chloride ion concentration

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