



Effects of lactic and citric acid on early-age engineering properties of Portland/calcium aluminate blended cements



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HIGHLIGHTS

- PC/CAC-based blended cement mortars are investigated as a concrete repair material.
- Effects of carboxylic acids on PC/CAC hydration and engineering properties studied.
- Portlandite crystals are the major hydrates in PC/CAC with lactic and citric acids.
- Lactic acid at 2% wt. or below improves strength PC/CAC at earlier ages.
- Citric acid reduces strength of PC/CAC and blocks dissolution of cement grains.

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ABSTRACT

In this study, Portland/calcium aluminate blended cement (PC/CAC) was combined with citric acid or lactic acid as additives to investigate the effects of the aforementioned carboxylic acids on the hydration reactions of PC/CAC as a potential fast hardening and low cost repair material for concrete. Mortar specimens with the carboxylic acid additives of either 0.5%, 1% or 3% by weight, prepared with a binder:sand:water ratio (by weight) of 1:3:0.5, were subjected to flexural and compressive strength tests at early ages up to 28 days. In order to understand the phase composition of the hydrates in the PC/CAC systems, XRD analyses were conducted on ground PC/CAC mortars with and without carboxylic acid at 7, 14 and 28 days. In combination with this, SEM images of selected mortar specimens were also taken at the same times for visual analyses of hydrates. Citric acid did not have any beneficial effect on enhancing the calcium silicate phase as initially assumed and instead reduced the strength of PC/CAC cement at all levels of concentration. The experiment analyses revealed that Portlandite crystals were the major hydrate phase in PC/CAC with lactic and citric acids. Lactic acid below 2% wt. improved both compressive and flexural strength gained at early ages due to improved crystallinity of the calcium hydroxide crystals. Combined with its inherent rapid setting time, PC/CAC blended cements have a potential to be developed into a suitable repair material for concrete.

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1. Introduction

1.1. PC/CAC based mortar

Repair technology has become increasingly important in modern construction practice. Demands for repair and protection of existing concrete infrastructure have dramatically increased in the last few decades which are consistent with greater appreciation of sustainability. A quality repair material often requires rapid setting and hardening, good adhesion, compatibility with existing

concrete, dimensional stability, and corrosion resistance. Calcium aluminate cement (CAC) has the potential to meet these repair requirements [1].

CAC has proven high strength development which is usually attributed to the formation of 2CAH_8 , 3CAH_6 and CAH_{10} [2,3]. CAC was developed during the last decade of the nineteenth century as an alternative to Portland cement (PC) to prevent structural concrete elements from serious sulphate attack. Other major fields of CAC application are in refractory concrete for industrial use in processes involving high temperatures [4] and in so-called 'building chemistry' where it constitutes one part of a complex mixture of mineral and organic ingredients for applications such as self-levelling screeds and tile cement [5]. CAC distinguishes itself from

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PC by containing a much higher concentration of alumina, normally 30–90% by weight [6]. Besides, CAC is also considered as a type of sustainable cement since CAC clinker contains less calcium compounds than PC clinker. In this case, less limestone is used in the manufacturing of CAC clinker, thus reducing the CO₂ emissions derived from the raw materials compared with PC clinker production [7]. CAC clinker is comprised of the same basic oxides as PC clinker such as lime, silica, alumina, and iron oxide, however the proportions of oxides are fairly different in CAC and PC clinkers. All CACs contain mono-calcium aluminate (CA or CaAl₂O₄) as the main hydraulic phase. Other phases may be present based on the type of CAC however it is important to note that C₃A is not a normal component of CACs [2].

Advanced material research has recently focused on ettringite rich cement compounds [8] which are known to be a dominant formation in CAC. Some properties including the hydration inhibition phenomena in these ettringite rich materials from PC/CAC/CS (where CS represents calcium sulphate (CS) systems), have been recently evaluated [9–11] and are of key importance to the engineering community as a more advanced appreciation on the mechanisms needs to be developed. CACs have several unique properties which could make them the materials of choice in concrete repair applications. When CAC is used alone as a conventional mortar, these properties include: 1. rapid hardening, even at low temperatures; 2. high temperature resistance/refractory performance; 3. resistance to a wide range of chemically aggressive conditions; and 4. resistance to impact and abrasion [6,12–14]. Cementitious products based on ettringite have a broad range of uses: formulations with water contents near the minimum requirement to ensure plasticity are widely used in proprietary floor screeds, high performance tile grouts, refractory kiln and sewage lining.

However the use of PC/CAC based systems as a repair material is not considered as often as polymer-modified cementitious mortars which are traditionally strong candidates for repair materials. One reason for this is that the annual production of CAC is very small, around 1% of PC in the UK market. Fondu, the brand of CAC used in this study and produced by Kerneos Ltd, is 3 to 4 times more expensive than PC, while white CAC is 10 times more expensive. This is governed by the cost of alumina derived from the natural mineral bauxite which is also primarily used in aluminium production, making it much more valuable than limestone used for PC production. Due to the high market price of CAC, it has not been economical and hence unpopular to use it alone for larger scale repairs. For this reason, research has led us to investigate its use as a replacement in minor quantities in PC for some desirable applications. If CAC is able to impart its attractive rapid hardening properties to PC, it would make for a very useful repair material with relatively low cost compared with pure CAC-based repair material. Due to its rapid setting, a PC/CAC system could be utilised in rapid repair of PC structures, such as highways, airport runways, and bridge decks. It could reduce the waiting time and cost associated with ordinary PC repair materials. Periods of sustained interruption for example in a busy highway, airport runway, bridge, etc., will cause significant economic loss. By using PC/CAC materials, the potential to reduce interruption to services could be greatly reduced, therefore allowing valuable time and resources to be saved.

The efficient repair and replacement of concrete often requires a rapid setting material that can be placed and cured in a relatively short period of time. Frequently, temporary repairs are made using materials that are later found to be incompatible with the existing pavement, structure, and environment. This practice causes these materials to fail prematurely, frequently requiring re-repair [15]. However research shows that PC/CAC based mortars may attain lower compressive strength resistance than PC mortar [16,17]. Mortar based on this binary system drops in compressive and flex-

ural strength with increased concentrations of CAC [11,16]. Previously, the main areas of research involving CAC in binary and tertiary systems have been related to optimizing setting time, mechanical strength, and fracture toughness [4,18–20]. To the best of the authors' knowledge, research on the combination of PC/CAC blends with chemical admixtures of organic and inorganic nature has not been investigated, allowing this study to create the opening in this field of research.

1.2. Lactic and citric acid addition

Lactic and citric acid are organic admixtures which belong to the carboxylic group of acids. Citric acid has been found to be one of the more effective acids at retarding PC hydration through the adsorption of citrate ions onto the surface of the Portland cement grain surface [21]. Ions of calcium, alumina, silica and iron are all potentially capable of chelating with organic compounds. In practice, the most widely used retarders are chiefly hydroxyl carboxylic acids or their salts [22,23]. Other studies on the interaction of citric acid with calcium sulphate hemihydrate, i.e. CaSO₄ · 1/2H₂O, confirm that the kinetics of hydration and setting time were governed by adsorption of the acids on the surface of the gypsum crystals and reduced interlocking of the microstructure [24]. The addition of citric acid to calcium sulfoaluminate cement increased the setting time, modified the morphology of the ettringite, changed the microstructural configuration and prevented a decrease in compressive strength by delayed ettringite formation [25].

Further investigations have been aimed to understand the mechanisms of the retardation using citric acid. Pore solutions, collected after different times of hydration, have been analysed for ion concentrations and organic carbon. The zeta potential as well as the composition of the hydrated solid phases has been studied [25,26]. Singh et al. [21] found that the zeta potential of cement decreases with increasing additions of citric acid, which was explained by the adsorption of citrate ions onto the positively charged surfaces of the Portland cement grains. Schwarz [26] concluded that citrate ions increase the dissolution rate of the ferrite phase. In the presence of PC/CAC cement, it was found to have a beneficial effect on strength development and appeared to reduce the retardation of the silicate phase hydration [11]. On the contrary, lactic acid has been shown to accelerate the hydration of Portland cement as proved by electrical conductivity measurements indicating that lactic acid formed a salt of calcium lactate during the interaction with Portlandite [30]. This product would also be beneficial in PC/CAC systems.

The main objective of this study was to investigate and evaluate the effects of the addition of lactic acid and citric acid on the compressive and flexural strength development of ternary PC/CAC/CS blends focusing particularly on the microstructure development and hydration products. The intention is also to develop a mortar with high early ettringite precipitation through the addition of lactic acid and reduce the disruption to the hydration of the silicate phases which contribute to the long term strength through the addition of citric acid. For this purpose mortar specimens were prepared using six PC/CAC/CS blends of varying citric and lactic acid additions to determine the compressive and flexural strengths up to 28 days. Experimental techniques such as SEM/BEI/EDX and XRD were employed to understand the mechanism of hydration of the ternary mortar at different ages.

2. Experimental program

2.1. Materials and specimen preparation

The weight percentages of the cementitious materials in the mortar formulations are summarized in Table 3. The additional

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