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Influence of temperature and moisture on the shelf-life of cement admixed with redispersible polymer powder

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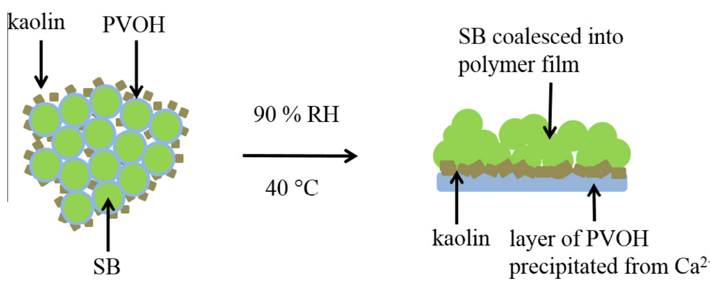
HIGHLIGHTS

- Neat cement and cement containing RDP were aged at 40 °C/90% RH.
- As latex RDPs, EVA and carboxylated SB were used.
- RDPs improve shelf-life of cement via partial film formation.
- Mortars containing RDP retain their mechanical properties better than plain mortar.

GRAPHICAL ABSTRACT



Mechanism of partial film formation in cement / RDP blend exposed to high humidity

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ABSTRACT

The shelf-life of cement blended with redispersible polymer powder (RDP) used in drymix mortars was studied after exposure to an atmosphere of elevated temperature (40 ± 1 °C) and moisture (90 ± 3% RH) simulated in a climate chamber. As RDP samples, ethylene–vinylacetate and carboxylated styrene–butadiene were used. The behaviour of the cement/RDP blends was studied with respect to film formation of the RDPs and the mechanical properties of the resulting mortars. It was found that the presence of RDP decelerates the prehydration of cement in humid environment and thus improves its shelf-life. At the same time, cement triggers a partial premature film formation from RDP powder during aging. Furthermore, mortars containing aged cement/RDP blends retained their mechanical properties much better than mortars casted from cement which had been aged individually. Apparently, RDP can protect cement from rapid aging.

1. Introduction

Cement presents the most commonly used binder material in the world, with an annual consumption of 4.2 billion tons in 2014 [1]. Its low cost, easy application and high compressive strength are the main reasons for its universal acceptance.

A mortar is a composite material which includes a binder phase and sand. Hydrated cement, a product resulting from the chemical reaction between cement and water, coats the sand and forms a rock-like solid matrix. During the hydration reaction the cement hardens and gains strength.

For decades researchers have been working to improve the microstructure of hydrated cement in an attempt to optimize its

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workability, strength and durability, for example *via* the incorporation of polymers into the mixture. Nowadays, several types of polymers used as chemical admixtures are available for the construction industry.

Among those additives are film forming latex polymers such as ethylene–vinyl acetate–versatic acid and styrene–butadiene copolymers. Numerous studies have been published about these materials [2–5]. When cement mortar is modified with latex, some of its undesired properties such as brittleness and low flexural strength are then improved. Additionally, the cohesion during the fresh state and the adhesion strength in the hardened state are enhanced [6–10]. These effects are owed to the formation of latex films in the cement paste [11–14]. The polymer films result from coalescence of individual latex polymer particles after dehydration of the mixture [15–18].

Mortars modified with redispersible latex polymer powder (RDP) are available as drymix blend and applied, for example, as tile adhesives and tile grouts, as adhesive mortar for external thermal insulation systems and as water-proofing membranes. RDPs are usually obtained by spray-drying the liquid latex dispersion containing a colloidal stabilizer and an anti-caking agent. The influence of these spray-drying aids on the properties of the mortar has to be taken into account when using RDPs [19].

It has been reported in previous literature that, in order to form a film in a cementitious matrix, the latex has to undergo particle coalescence which occurs in four main stages [11,18]. Initially, the latex is present as an aqueous dispersion (stage I), then forms a dense particle packing (stage II) until the latex particles start to deform and arrange in hexagonal shape in order to minimize their interfacial tension (stage III). Finally, the polymers diffuse across primary latex particle boundaries and the particles coalesce into coherent polymer films. (stage IV). Relative to this process, it has been observed that the spray-drying aids present in RDPs affect the film formation. For example, polyvinyl alcohol (PVOH) used as colloidal stabilizer in a carboxylated SB latex was found to reduce the negative charge of the SB particles by coating some of the free carboxylate groups that might react with calcium ions present in the cement pore solution. This way, the electrostatic repulsion between latex particles is reduced and the transition from stage II to stage III is accelerated [19]. A similar effect was observed for kaolin which is added as an anticaking agent to RDPs [20].

The main difference between a liquid latex dispersion and a RDP is that in the latter, at first the spray-drying aids which are attached to the surface of the polymer powder particles need to dissolve or disperse and separate from the surface to allow coalescence and film formation [20]. Limited shelf-life is a problem well-known to the drymix mortar industry [21]. In this context, shelf-life means the time period over which a drymix mortar blend remains usable and is fit for the intended application. During storage, the cement contained in the drymix mortar can age and the RDPs may form a film prior to application. Cement aging is the consequence of a reaction between anhydrous cement, water vapor and CO_2 present in air that leads to partial hydration of the surface of the cement grains. It has deleterious effects on some engineering properties such as compressive strength, workability and setting time of the cement paste. For example, *Whittaker* et al. [22] studied the effect of aging on the engineering properties of a CEM I Portland cement sample exposed to relative humidities (RH) of 60% and 85% respectively over periods of 7 and 28 days. They demonstrated that aging results in the formation of a barrier of hydrates with few nm thickness covering the surface of the cement grains. Its thickness depends on the values of RH, temperature and exposure time. This was confirmed by *Dubina* et al. [23] who studied the aging of pure clinker phases and found that only the surface of the clinker particles was hydrated, as was confirmed later by X-ray photoelectron spectroscopy (XPS) [24]. Other studies focussed on the prehydration behaviour of OPC and oil well cement *Dubina* and *Plank* [25–29].

Dubina and *Plank* [30] have also investigated the long-term stability of drymix mortars during storage. They found that the water sorption of each phase started at different RHs. CaO and C_3A were found to present the most reactive in respect to water sorption. For example, CaO starts to sorb water at 14% RH only and binds it chemically while orthorhombic C_3A sorbs water both chemically and physically beginning at 55% RH. For the calcium silicate phases, water sorption starts at 74% RH and the amount of water absorbed is very minor.

Meier and *Plank* [31] found that exposing anhydrous cement to humid environment greatly impacts its hydration behaviour. After aging a Portland cement sample for 1 day only, the heat flow from the hydration measured *via* isothermal calorimetry was decreased severely. Moreover, when the cement was aged for more than 3 days, the main hydration peak characteristic for C-S-H formation had disappeared entirely. Instead, a new peak at early hydration time (0–3 h) attributed to delayed ettringite formation had occurred. These authors also studied the performance of PCE superplasticizers and retarders in aged cement that was previously exposed to humidity. They found that the dispersing performance of the PCEs decreased with prolonged aging time of the cement and that the effectiveness of the retarders was different when applied to aged cement. These examples demonstrate that moist air can have a significant impact on the behaviour of cement and on the performance of some admixtures.

None of those previous researches have studied the influence of aging on RDPs commonly used in drymix mortars. These polymers might also age or undergo premature film formation before the mortar is applied. When this happens, the advantages attributed to them such as e.g. higher flexural and adhesive strength might be lost.

The aim of this research was to understand potential processes and reactions occurring during moisture exposure of a drymix mortar modified with RDP. It was sought to elucidate the processes relating to the latex powder (e.g. film formation, cement/RDP interaction) on a molecular scale and to connect those with the macroscopic properties of the drymix mortar. Another goal was to find out whether the shelf-life of the drymix mortar is linked to the chemistry of the RDP (EVA versus SB polymers) present in the blend and how the spray-drying aids contained in the RDPs may influence the process.

2. Materials and methods

2.1. Materials

The cement used in this study was an ordinary Portland cement CEM I 52.5N from HeidelbergCement, Geseke plant, Germany which is commonly used as base cement in many drymix mortar formulations. Its phase composition as determined

Table 1
Phase composition of the CEM I 52.5N sample as determined by Q-XRD using *Rietveld* refinement.

Phase	wt%
C_3S, m	54.1
C_2S, m	26.6
C_3A, c	3.3
C_3A, o	4.3
C_4AF, o	2.5
Free lime (*Franke*)	0.1
Periclase (MgO)	0.0
Anhydrite	2.6
$CaSO_4$ hemihydrate*	1.2
$CaSO_4$ dihydrate*	0.02
Calcite	3.6
Quartz	1.2
Arcanite	0.5

* Determined *via* thermogravimetry.