



An ESEM investigation of latex film formation in cement pore solution

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ABSTRACT

Environmental scanning electron microscopy (ESEM) and complementary methods were employed to study the time dependent film formation of a latex dispersion in water and cement pore solution. First, a model carboxylated styrene/*n*-butyl acrylate latex dispersion possessing a minimum film forming temperature (MFFT) of 18 °C was synthesized in aqueous media via emulsion polymerization. Its film forming property was at a temperature of 40 °C, studied under an ESEM. The analysis revealed that upon removal of water, film formation occurs as a result of particle packing, particle deformation and finally particle coalescence. Film formation is significantly retarded when the latex dispersion is present in cement pore solution. This effect can be ascribed to adsorption of Ca²⁺ ions onto the surface of the anionic latex particles and to interfacial secondary phases. This layer of adsorbed Ca²⁺ ions hinders interdiffusion of the macromolecules and subsequent film formation of the latex polymer.

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1. Introduction

Latex dispersions are widely used for modification of cement-based mortars and concretes [1,2]. They allow to improve some undesired properties of hardened cement such as brittleness and low flexural strength. Additionally, cohesion and adhesion of the fresh and hardened mortars and concretes are significantly enhanced. These effects are owed to the formation of polymer films which result from the coalescence of latex particles when water is consumed by cement hydration and evaporation. The latex polymers which are most common in the construction industry are based on ethylene/vinyl acetate or styrene/acrylate chemistry [2].

Studies on the film forming process of latex polymers represent a field of intense research since many years [3,4]. Latex film formation is a process which entails several steps. It starts from an aqueous latex dispersion and ultimately results in a homogeneous polymer film. A schematic representation of the film formation process in water as solvent is shown in Scheme 1. Generally, the film forming process can be viewed as a succession of those four steps:

In Stage I, the latex particles are randomly dispersed in the solvent. When water is removed from this dispersion, ordering and packing of the particles takes place (stage II). The particles are in contact with each other whereby the interstitial spaces are filled with solvent. As water evaporation and consumption progresses, an ordered array of hexagonal deformed latex particles is formed (stage III). For latex dispersions exhibiting a hydrophilic surface, particle deformation results into polyhedral cells separated by hydrophilic layers (transition stage III*)

[5]. Some authors present that between stage II and III, an intermediate stage (II*) exists which is characterized by a randomly packed array of deformed particles which are surrounded by water-filled interspaces [6]. Finally, in stage IV a molecularly homogeneous polymer film is formed as a result of particle coalescence stemming from polymer interdiffusion and break up of hydrophilic interlayers. This process requires that the ambient temperature is above the minimum film forming temperature (MFFT). MFFT depends on the monomer composition of the latex particles and is influenced by other additives (e.g. emulsifier) used in the emulsion polymerization process. Experimentally, it is determined on a so-called *Kofler* bench. Closely linked to MFFT is the glass transition temperature (T_g) of a latex dispersion. It represents the temperature at which an initially soft and flexible latex polymer film becomes brittle and hard. For a given polymer, T_g can be calculated using the *Fox* equation [7], or it is measured by differential scanning calorimetry (DSC).

Today, the film forming process occurring in water is understood sufficiently. There are, however, only few reports on the effect of electrolytes (e.g. salt) on the film formation. Such condition is however prevalent when latex polymers are used in cement-based building materials such as tile adhesives, self-levelling compounds, polymer concrete etc.

According to *Chandra* and *Flodin* styrene methacrylate dispersions lose their film forming ability when blended with Ca(OH)₂ [8]. Using a sorption balance *Erkselius* et al. found a significantly reduced drying rate for a methyl methacrylate-methyl acrylate latex copolymer when surfactants and salts were present [9]. Hence, it becomes obvious that latex film formation is highly sensitive to ambient conditions, such as temperature, pH or type and concentration of ions present in solution. In another work, *Jenni* and *Zurbriggen* present that film formation of a polyvinylalcohol coated redispersible latex powder is accelerated in

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