



# A study on the impact of different clay minerals on the dispersing force of conventional and modified vinyl ether based polycarboxylate superplasticizers



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## ABSTRACT

The impact of three different clay minerals (montmorillonite, kaolinite and muscovite) on the dispersing force of vinyl-ether based PCE superplasticizers was studied. At first, a conventional vinyl-ether PCE was synthesized from maleic anhydride (MA), mono methoxy poly(ethylene glycol) ester of MA and 4-hydroxy butyl vinyl ether (HBVE). Additionally, a PEG-free PCE was prepared by substituting MPEG with hydroxyalkyl groups in the ester. Next, the effect of three different clay minerals on the fluidity of cement paste admixed with both PCEs was investigated. It was found that the conventional PCE is negatively affected by the clays in the order: montmorillonite  $\gg$  kaolinite  $>$  muscovite. Whereas, the PEG-free PCEs were only slightly affected. The study suggests that conventional PCEs containing PEG reasonably tolerate all types of clay minerals except montmorillonite. When montmorillonite is present as a contaminant, then use of a PEG-free PCE such as described here presents a viable mitigation strategy.

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## 1. Introduction

The invention of polycarboxylate superplasticizers (PCEs) as a new generation of concrete admixtures in the 1980s represents one of the most significant breakthroughs in the history of concrete admixtures. However, recently more and more field users report that under certain conditions PCEs can exhibit a strong sensitivity to clay impurities. Most confusing, in some cases the negative impact of clay minerals on the dispersing force of PCE was very pronounced whereas in other cases, the effect was only minor. Apparently, different types of clay minerals show a completely different impact on the performance of PCEs in relation to variations in their physical and chemical properties.

Clay minerals are hydrous aluminosilicates characterized by crystal sizes of less than 2  $\mu\text{m}$  in diameter. They are phyllosilicates constituted by a layer structure made of sheets of Si-centered tetrahedra alternating with sheets of Al-centered octahedra. Alternatively, they may be constituted by sheets of Si-centered tetrahedra alternating with sheets of Mg-centered octahedra. The presence of trivalent cations (e.g.  $\text{Al}^{3+}$ ) or divalent cations (e.g.  $\text{Mg}^{2+}$ ) in the octahedral sheet brings about a different cation occupancy in the sheet. With divalent cations all sites are occupied, while with trivalent cations only two thirds are occupied.

Clay minerals support extensive isomorphous substitutions in both the tetrahedral and octahedral sheets. The most common ones are

$\text{Al}^{3+}$  for  $\text{Si}^{4+}$  in the tetrahedral sheet and  $\text{Fe}^{3+}$ ,  $\text{Fe}^{2+}$  or  $\text{Mg}^{2+}$  for  $\text{Al}^{3+}$  in the octahedral sheet. Where isomorphous substitutions imply cations of different valence, a net charge is acquired by the layer which must be balanced by inter-layer species.

Clays can be categorized depending on the way that tetrahedral and octahedral sheets are combined into layers. The tetrahedral sheets are always bonded to the octahedral sheets through the unshared oxygen vertex substituting a hydroxyl in the octahedral layer. On one side of the octahedral sheet two hydroxyls out of three are replaced by a  $(\text{Si}_2\text{O}_5)^{2-}$  unit. If there is only one tetrahedral and one octahedral sheet in each layer, then the clay is known as a 1:1 clay or a t-o clay. The alternative, known as a 2:1 clay or t-o-t clay, has two tetrahedral sheets, one on each side of the octahedral sheet. The layers are held together by hydrogen bonds (e.g. kaolinite, a 1:1 dioctahedral clay), or by electrostatic forces between the charged layers and the interlayer cations (e.g. illite, a 2:1 dioctahedral clay) [1–3]. Where the layer charge is low in 2:1 clays like in smectites (0.2–0.5 interlayer charge per 11 oxygens, compared to 0.5–0.75 in illites) [4], the interlayer is large and can host variable amounts of water molecules and different kinds of ions. It can easily shrink and swell as the result to changes in relative humidity.

The clay minerals used in the present work have been: kaolinite, a non-swelling 1:1 dioctahedral clay; muscovite, a non-swelling 2:1 dioctahedral clay belonging to the group of illites; and Na montmorillonite, a swelling 2:1 dioctahedral clay belonging to the group of smectites.

The principle structures of the major clay minerals occurring as contaminant in concrete are displayed in Fig. 1.

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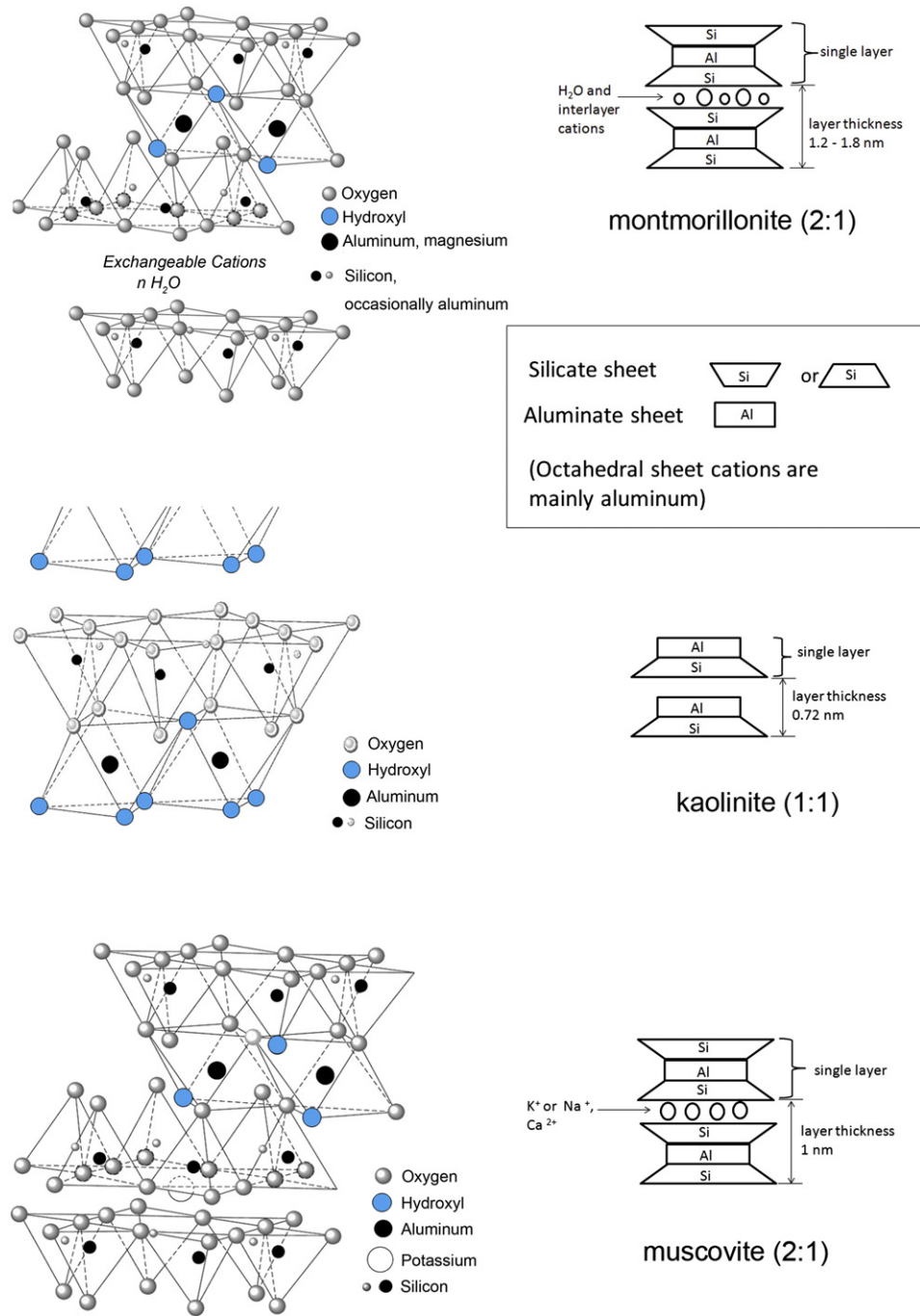


Fig. 1. Principle structure of the major clay minerals occurring as contaminant in concrete, after Grim [5].

The aim of this study was to compare the effect of three most frequently occurring clay minerals (montmorillonite, kaolin, muscovite) on cement pastes admixed with polycarboxylate (PCE) superplasticizers. From this, it was sought to understand the different impacts of differently structured (2:1 and 1:1) swelling and non-swelling clay minerals on PCEs. Furthermore, two types of vinyl ether-based PCEs were synthesized, one with a conventional polyethylene glycol (PEG) side chain, and one with hydroxyalkyl pendant groups. For both polymers, their specific modes of interaction with these different clay minerals were assessed via XRD, adsorption and zeta potential measurements. From this mechanistic study it was hoped to obtain an explanation for the different behaviors of clays with PCEs, as reported by field users.

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Chemicals

4-Hydroxy butyl vinyl ether (>99% purity, purchased from Sigma-Aldrich CHEMIE, Steinheim, Germany), maleic anhydride (>99%, Merck Schuchardt OHG, Hohenbrunn, Germany), methanol, ethanol, n-propyl alcohol (all >98% purity, VWR International, Darmstadt, Germany), sodium hydroxymethane sulfinate (Rongalit C, >98% purity, Sigma-Aldrich), ferrous sulfate (>98% purity, VWR), 2-mercapto ethanol (>99% purity, Sigma-Aldrich), hydrogen peroxide (30% aqueous solution, VWR) were used without further purification.

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