



# Influence of dispersing agents on the rheology and early heat of hydration of blended cements with high loading of calcined marl



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## ABSTRACT

The dispersing effectiveness of five commercial plasticizers; lignosulfonate (LS), naphthalene sulphonate–formaldehyde polycondensate (NSF) and three polycarboxylate ethers (PCEs) were quantitatively investigated in blended cements where ordinary Portland cement (OPC) was partly replaced by calcined marl (CM) up to 60%. CM drastically viscosified and decreased the flow of the cement due to high absorption of water. The performance of plasticizers mimics that in the OPC system except when PCEs were added. PCEs possessing long side chains were less effective as dispersants due to the consumption of these polymers via the ability of their PEO side chains to intercalate between the remaining layers in the calcined clay. Higher dosages were thus needed for effective dispersion. The decrease in PCE–OPC interaction led to little retardation in cement hydration except at high polymer dosages, whereas the performance of NSF and LS in CM blended cement is driven by clinker content.

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## 1. Introduction

The building sector is one of the most dominating industries due to the constant and growing demand of new construction materials in response to the needs in the housing and infrastructure business. Among which, cement is globally produced with an annual amount of ~3.5 billion tons in 2011 [1,2] and contributes ~6% CO<sub>2</sub> emission annually worldwide, making the cement industry the third largest CO<sub>2</sub> emitter after housing and transport [3,4]. To reduce this, greener and more environmentally friendly binders are sought after. The aim is to reduce the level of CO<sub>2</sub> emissions by ~50% by 2050 as stated by the World Business Council for Sustainable Development. One of the many approaches in tackling this for the cement industry is to increase replacement of clinkers by supplementary cementing materials (SCMs). The advantage of such blended cements is a direct reduction in CO<sub>2</sub> emission, which is reflected in the percentage of substitution by the SCM materials, making them a popular choice in the cement industry.

Generally, many natural, as well as synthetic SCMs can be utilized as suitable materials. These include industrial by-products such as granulated blast furnace slag (GBFS) from iron manufacturing and fly ash (FA) from coal fired power plants. Among the many SCMs, one of the upcoming materials which has gained much attention in the construction world nowadays is calcined clay.

Clay can be found in great abundance all around the world and it has been found that upon calcination, these materials becomes pozzolanic in nature and can/may act as effective replacements of cement in the industry. The most well-known is metakaolin. However, due to its high cost, pure metakaolin is not desired to be a mainstream cement replacement material. Within the same group, calcined marl is a derivative of a contaminated clay product. Marl by itself is often known as 'bad clay' and is rendered unsuitable for production of burnt clay products such as bricks and lightweight aggregate due to the high content of calcium carbonate it possesses. Due to its low applicability in conventional usages, marl is often considered a waste product which can be obtained cheaply in large quantities. However, upon calcination, this material becomes an effective pozzolan containing both calcined clay and calcium containing compounds [5,6]. For this reason, calcined marl can be a large source of unexploited SCMs which can be used effectively in replacing OPC clinkers, but potentially also the existing SCMs which are already available in the market that may become scarce as the demand is increasing.

Previous investigation has shown that after one day of hydration, a compressive strength of 10 MPa sufficient for demoulding is achievable for mortar where 50% OPC is replaced by CM [6]. Nevertheless, incorporation of calcined marl into cements can result in changes in the properties of these binders and thus compatibility with other additions could be potential challenges, particularly at higher replacement values. Two such challenges are the impact on rheology and early hydration development of

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binders. In cement science, rheology of concrete and cement paste is commonly altered and improved by the addition of admixtures such as superplasticizers, and retarders. The introduction of admixtures improves the user friendliness of cementitious materials on site, enables enhanced final properties and aesthetic of the composite or buildings, and also reduce cost and labor force for the construction industry. This demand of materials with optimum workability is expected to continue and grow with the rising usage of construction materials, such as in the area of self-consolidating concrete (SCC) to ease placement without the need of vibrators.

The largest group of rheology regulators among the admixtures consists of (super)plasticizers which act by improving dispersion of particles during the early hydration ages of cement [7]. With normal OPC, extensive research has been performed, both in determining their behaviours and effectiveness on rheology, and also the improvement of their additions on the final strengths and mechanical properties of concretes. The mechanisms underlying the effective dispersion of OPC by these admixtures as driven by electrostatic repulsion or steric hindrance have also been widely discussed. Their effectiveness is generally accounted for by the attributes of the clinker and hydrate phases [8,9], and the specific characteristics (both structural and chemical) of the admixtures added [10,11].

To calcined marl blended cements, little has been studied on the performance of rheology regulators on these binders. This can be attributed to the infancy of this field of research, where rheology regulators such as plasticizers have often been used just to produce a suitable flow for the concrete system [12]. There, the emphasis has always been on the feasibility of using clay as binder replacement and the durability [13] and physical properties of the resulting concrete/mortar [14]. However, exactly due to this surge in research interest and potential usage, a better understanding of the applied rheological effectiveness of these plasticizers is required to ensure optimal usage of the admixtures in such system. Additionally, the influence of these admixtures on blended cements possessing higher replacement amounts of SCMs with the drive in creating a greener construction industry is also to be expected.

For this purpose, five different commercial plasticizers were selected to test for their dispersing effectiveness in calcined marl blended cements. The selection included a high molecular weight Na-lignosulfonate (LS), a naphthalene sulphonate–formaldehyde condensate (NSF) and three different sets of polycarboxylate ether (PCEs) polymers possessing different side chain lengths. High amount of calcined marl (CM) replacement, namely 20%, 40% and 60% replacement of ordinary Portland cement (OPC) by weight were investigated. Rheological analysis was performed employing the principles of a parallel plate rheometer, while the packing density and water demand were followed by the centrifugal consolidation method. Lastly, the impact of varying systems on the heat of hydration of the cement slurries was investigated by isothermal calorimetry and a correlation between this heat evolution profile and the rheological properties of the cement pastes was sought.

## 2. Experiments and methods

### 2.1. Materials

An OPC and a CM (calcined at 850 °C) were supplied by Norcem AS (Brevik, Norway) and Saint-Gobain Weber (Oslo, Norway) respectively. The CM contains mainly smectite (>50%) and calcite (~25%). Minor amounts of kaolinite (~8%), quartz (~4%), siderite (~3%) and pyrite (~1%) are also present. The specific Blaine surface of OPC is 382 m<sup>2</sup>/kg. In the case of CM, the specific surface area as measured by BET is 15.1 m<sup>2</sup>/g and it possesses an average particle

size of 7 µm. Blaine and BET cannot be compared directly as Blaine being based on air flowing through a pack of powder is related to the “outer” surface of particles (i.e. voids between them), while BET measures both outer and inner surface of non-connected pores and cracks. Table 1 displays the oxide compositions of the OPC and CM.

As dispersing agents, five commercial plasticizers are investigated, namely a purified sodium LS (Borregaard AS, Sarpsborg/Norway), a NSF (Sika AS, Sketten/Norway), and three sets of PCEs (Mapei Escon AS, Sagstua/Norway). The NSF has a solid content of 40 ± 0.5%. The LS possessed a high molecular weight and contained very low amount of sugar and organic acid. The three sets of PCEs are denoted SX, NRG and SRN. SX and SRN are both based on polymethacrylate containing polycarboxylates. Industrially, SX is commonly employed in ready-mix systems and contains only one PCE possessing long side chains with low charge density. SRN on the other hand possesses intermediate workability and plasticizing effect, and only holds a methacrylate based PCE with short side chains and high charge density. NRG is made up of a combination of one polymethacrylate based PCE and a polyacrylate based PCE in the ratio of 1:9. The polymethacrylate based PCE here possesses long side chains and low charge density and is similar to that in SX. The polyacrylate based PCE possesses very long side chains and very low charge density. NRG is commonly used in the element industry and generally generates pastes with the shortest workability.

All materials were utilised as per obtained. For preparation of the blended cements, the OPC and CM were manually mixed in the ratio of 4:1, 3:2 and 2:3 to produce blended cements with CM contents of 20%, 40% and 60% respectively. These blended cements were denoted as CM20, CM40 and CM60 respectively. Pure OPC and CM were investigated too. For the dispersing agents, all samples were dissolved in deionised water to obtain solutions possessing solid contents of between 20% and 30% for increased accuracy of dosing. 4 different dry polymer dosages were employed; 0.1%, 0.2%, 0.4% and 0.8% dry polymer bwob (by weight of binder), respectively.

### 2.2. Experimental procedures

The cement slurries were prepared at a low w/b of 0.36 to amplify the effectiveness of the plasticizers. The required dosage of plasticizers was homogenized in the water and added to the dry powder mix over 30 s before blending under high shear for 1 min utilizing a high shear mixer (Philips, 600 W, capacity = 200 mL), let stand for 5 min and a final high shear mixing of 1 min to avoid false setting. The high shear mixing was to mimic the high shear energy in a concrete partly imposed by coarse aggregate [15]. In each mix, the amount of slurry prepared was ~205 g to minimise weighing errors. The

**Table 1**  
Oxide compositions of OPC and CM.

	OPC	CM
SiO <sub>2</sub>	20.8	49.6
Al <sub>2</sub> O <sub>3</sub>	4.6	18.1
Fe <sub>2</sub> O <sub>3</sub>	3.5	10.6
CaO	61.6	14.1
MgO	2.4	2.9
P <sub>2</sub> O <sub>5</sub>	0.2	0.2
K <sub>2</sub> O	1.0	2.4
Na <sub>2</sub> O	0.5	0.7
SO <sub>3</sub>	3.5	–
Alkali	1.1	–
Total	98.1	98.6

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