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Rheological study of cement paste with metakaolin and/or limestone filler using Mixture Design of Experiments

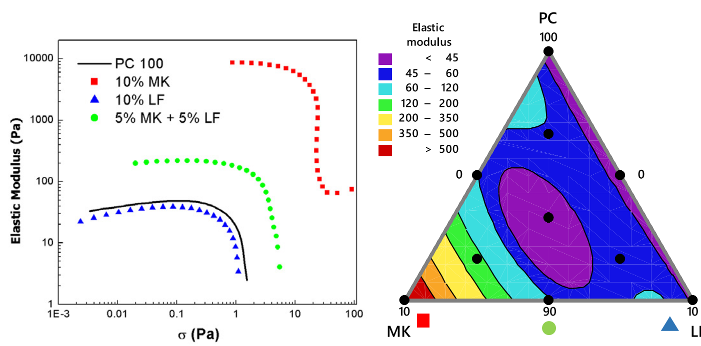
Fabiano Nazário Santos [a], Sara Raquel Gomes de Sousa [b], Antonio José Faria Bombard [b,*], Sheila Lopes Vieira [c]

[a] Natural Resources Institute (Instituto de Recursos Naturais – IRN), Federal University of Itajubá (Universidade Federal de Itajubá – UNIFEI), Itajubá, MG, Brazil
[b] Physics and Chemistry Institute (Instituto de Física e Química – IFQ), Federal University of Itajubá (Universidade Federal de Itajubá – UNIFEI), Itajubá, MG, Brazil
[c] Durham, NC, USA

HIGHLIGHTS

- Metakaolin acts as a thixotropic additive when a superplasticizer is used in cement paste.
- Limestone filler does not interfere with the viscosity or plasticity of the paste with superplasticizer admixture.
- The ideal metakaolin content depends on the paste desired viscosity, thixotropy and workability.
- The use of up to 5–10% metakaolin improve the compressive strength after 7 days.

GRAPHICAL ABSTRACT



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ABSTRACT

Several cement pastes with different amounts of metakaolin (MK) and/or limestone filler (LF) were prepared. The water/cementitious materials ratio was maintained constant at 0.3, with addition of 0.5% wt/wt of poly-carboxylate ether (PCE) superplasticizer admixture. The following parameters of the fresh cement pastes were evaluated: the slump and spread, the Marsh funnel time, the plastic viscosity, yield stress, viscoelastic properties and thixotropy. After the curing of 7 day old pastes, compressive strength tests were performed according to the Brazilian standard using 50 × 100 mm cylinder specimens. We conclude that LF alone is not able to avoid segregation or bleeding, and there is no difference between cement pastes mixed with LF and pure OPC pastes, in terms of rheology. On the other hand, if one needs low slump and low spread, the use of MK is recommended because this material creates a strong, thixotropic interconnected net inside of the paste, increasing the yield stress and the thixotropy of the cement paste. By adding 5–10% wt/wt MK, the average increase of compressive strength is approximately 45% at 7 days, compared to the control (only OPC, water and PCE). The maximum recommended amount of LF or MK substitution in our case was 10% wt.

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1. Introduction

The use of superplasticizer (SP) additives (also known as water reducers) in civil construction has increased in recent years, especially in large-sized works or the ones that require the use of special concretes, such as self-compacting concrete or high performance concrete, among others [1,2]. Poly-carboxylate ether (PCE) is among the additives that has superior performance in terms of viscosity reduction compared to common plasticizers, such as lignosulfonates and naphthalenesulfonates [3]. On the other hand, if poly-carboxylate ether is very effective in reducing cement paste

* Corresponding author.
E-mail address: bombard@unifei.edu.br (A. José Faria Bombard).

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viscosity, extra caution with additive overdose is a must. For instance, a dose of 1 wt.% of water reducing admixture to the cement leads to cement particle segregation, with cement powder settling very fast and causing phase separation with a layer containing Portland cement (precipitate) at the bottom of the recipient and a supernatant containing a lot of water and finer cement particles. Another negative effect of poly-carboxylate ether overdose is the loss of thixotropy (and consequently the workability) of the paste.

Siddique & Khan [4], describe that the use of supplementary cementitious materials (SCM), such as blast furnace slag, fly ashes, microsilica, metakaolin, limestone filler, rice husk ashes, among others, is increasingly growing. The use of such materials may be advantageous not only by the reduction of economic and environmental costs of Portland cement manufacturing but also because it can greatly increase the final performance of structures, such as compressive strength. In addition, some materials may help solving issues of segregation and workability loss caused by superplasticizers overdose. Martins & Bombard [5], showed that the use of nanosilica in combination with adequate doses of poly-carboxylate ether allows the acquisition of a relatively low apparent (plastic) viscosity without workability loss (it maintains the yield stress and thixotropy of the paste). Pera [6], reports that the first documented use of metakaolin in a large-scale work was in the construction of the Jupiá Dam in 1962. Antoni et al. [7], assessed the replacement of part of a Portland cement segment with a combination of metakaolin and limestone filler, resulting in "45% of substitution by 30% of metakaolin and 15% of limestone gives better mechanical properties than 100% OPC". In addition to that, they argue that *"stoichiometric formation of monocarboaluminate hydrate (MC)... corresponds to an addition with a weight ratio of 2:1 metakaolin:limestone."* But the authors did not study the rheology of mixtures.

A partial literature revision about rheological aspects of cement pastes with supplementary cementitious materials follows. Cyr et al. [8], investigated the shear thickening effect of superplasticizers on the rheological behavior of cement pastes containing or not mineral additives. They compare the effect of: metakaolin (MK), quartz (Qtz), fly ash (MFA) or silica fumes (SF). Their superplasticizers (SP) included five different types, but without any detail about the chemistry of each SP. These authors studied three substitution amounts of Portland cement by the four supplementary cementitious materials (SCM) above: 0% (only cement), 10% or 25%.They concluded that in terms of shear thickening effect "can be amplified (metakaolin), unchanged (quartz, fly ashes) or reduced (silica fumes)". Provis et al. [9], studied "the role of particle shape" (morphology) of some SCM: "spherical particles of fly ash", "platy particles of metakaolin", and the "angular particles of blast furnace slag", "both in the context of its effect on paste rheology and on water demand". The authors focused their report on particle shape effects in fresh pastes, particle packing and mix design in geopolymer pastes and geopolymer concretes. However, they did not mention any water reducer, plastifier or superplastifier. Banfill and Frias studied the rheology of blends cement with metakaolin or cement with paper sludge wastes, calcined at 700 °C by 2 h [10]. The authors employed a sulfonated naphthalene formaldehyde condensate as superplasticizer. They concluded that "the use of low concentrations of calcined paper sludge as a supplementary cementitious material... offers a route for utilising this waste material, as an alternative to the... environmental burden associated with the production of metakaolin from natural kaolinite resources." Moulin, et al. reported about the effects of "OPC blended with 30% (by weight of blend) calcined clay and its rheology. However, they also did not use any superplasticizer [11]. Poulesquen et al. studied the rheology of geopolymers prepared with metakaolin, fumed silice and "Waterglass activating solutions", but they did not employ Portland cement, neither any superplasticizer [12]. Janotka et al., investigated in deep the rheology, compressive strength, isothermal calorimetry and setting time of mixtures of Portland cement with "metakaolin sands", a type of SCM that was not pure metakaolin [13]. Their water/cement ratio was 0.5, without addition of any water reducer plasticizer. They concluded: *"...the presence of the metakaolin sands reduces the heat released during the hydration process with respect to non-blended-cement pastes. The incorporation of metakaolin sand induces a decrease of the mechanical strength, with the decrease being higher as the metakaolin sand content increases even though they also produce a refinement in the pore structure and a decrease of the permeability*". Sonebi et al. made an "Optimization of rheological parameters and mechanical properties of superplasticized cement grouts containing metakaolin and viscosity modifying admixture", employing "Central composite experimental design (CCED)", a statistical tool. They employed the same type of superplasticizer we are studying, PCE. However, they stated: "The viscosity of cement grout was determined using a coaxial rotating cylinder viscometer Fann (smooth cylinders, no serration)." Therefore, slippage could have occurred during their measurements [14]. Vance et al. published a paper with the exact same materials that we are studying. The title of their paper is: "The rheological properties of ternary binders containing Portland cement, limestone, and metakaolin or fly ash" [15]. However, different from us, they also did not employ any water reducer admixture. Besides this, in their study, the water-to-solid ratio (w/s) mass/mass were 0.40 and/or 0.45. In our study, with the use of PCE SP, we prepared pastes with fixed water/solids (w/s) ratio = 0.30. Favier et al., compared the rheological properties of a geopolymer paste prepared mixing metakaolin with sodium silicate solution (water glass) versus cement paste. But not blends of OPC + MK [16]. More recently, Vance et al. compared the rheology of suspensions (pastes) prepared with "interground Portland limestone cements" × "three blended limestone cements" They described a ratio w/s = 0.45 and again, without any superplasticizer [17]. Shahriar and Nehdi reported blends of special cement (oil well API Class G OWC) mixed with four types of SCMs: MK, SF, (rice husk ashes) RHA, and low calcium FA, with replacement ranging from 5 to 15%. They also employed a poly-carboxylate-based high-range water reducing admixture, but with water-to-binder mass ratio (w/b) of 0.44, which is the usual w/b recommended for oil well cement formulations. In their study, they used Design of Experiments too. [18].

For the reader interested in reviewing the significant literature on the rheology of cement pastes, as well as hundreds of scientific papers published after 2001, the classical books by Tattersal [19] and Banfill [20] are advised.

Metakaolin is a material with high pozzolanic activity. In addition to being advantageous economically and environmentally, it has the effect of improving mechanical resistance, as compressive strength, since keeping low amount substitution of OPC by MK (~10%) by such way the hydration heat is similar to 100% OPC [21].

Limestone filler addition to cement accelerates hydration of Portland clinker grains at early ages, improves the particle packing, can increase the hydration rate from 1 day to 3 months and produces the formation of calcium carbo-aluminates (hemicarboaluminate or monocarboaluminate), as a result of the reaction between $CaCO_3$ and C_3A of Portland clinker or metakaolin (in case of ternary blends) [22]. However, if partial substitution of OPC by LF can be advantageous (same reasons as MK: economic and environmental aspects), the formation of carbo-aluminates is a drawback, in the case of a sulfate and chloride environment. [23].

Around one hundred papers can be found reporting mixtures of "limestone AND cement AND metakaolin". However, very few [15,17,22,24–27], focus on the rheological properties of ternary blends of these three cementitious materials. Most of these works,

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