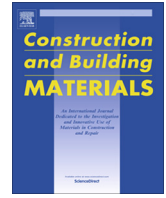




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## Influence of high contents of limestone fines on rheological behaviour and bond strength of cement-based mortars



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### HIGHLIGHTS

- Mortars with cement partially (30, 60%vol) replaced by 2 types of limestone fines.
- Limestone type and use of dispersant affected rheological parameter yield torque.
- At 30%vol, limestone decreased mortar tensile strength but increased bond strength.
- At 60%vol, limestone reduced mechanical properties; bond strength less intensely.
- Mortars with lower yield torque displayed higher bond strength.

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### ABSTRACT

This paper investigates the influence of cement replacement by high contents (30%vol and 60%vol) of limestone fines on rheological behaviour and mechanical properties of mortars as well as on the bond strength of mortar-ceramic interface. Mortars with 75%vol of sand and 25%vol of fines were produced using cement and two limestones having different particle size distributions and surface areas. A similar set of mortar compositions was also prepared using a polycarboxylate-based dispersing admixture. Rheological behaviour of the mortars varied depending on type and content of limestone fines, and whether the admixture was added. Elastic modulus and tensile strength of the mortars decreased with the increase of limestone content. However, for replacement rates of 30%vol, the tensile bond strength between mortar and ceramic block increased, likely because of ITZ densification. The dispersing admixture improved the tensile bond strength of all compositions without necessarily increasing their mechanical properties. The obtained results were compared to others available in the literature.

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## 1. Introduction

The variation of the cement paste microstructure in the vicinity of either an aggregate or an external substrate is called the interfacial transition zone (ITZ) [1]. There is a consensus that this region is the weakest zone in cementitious composites: in concretes, the relevant ITZ occurs mainly around large aggregates; while for

rendering, masonry and adhesive mortars or repairing grouts, the important interfaces are between the cementitious material and the diverse substrates (bricks, blocks, ceramic tiles, walls, reinforcing elements, etc) involved in their applications. The bond strength is an essential requirement of multi-phase materials because the interface zone is generally more porous than the bulk paste, thus affecting significantly the mechanical properties of the systems and their durability [2].

The interface microstructure has a very thin layer ( $\approx 1 \mu\text{m}$ ) of hydrated phases that are formed directly over the substrate or aggregates surfaces [3]. After the very first layer of hydrates at the interface, the region characterized by a deficit of particles (ITZ) starts and, then, extends inwards the paste in a depth of approximately  $50 \mu\text{m}$  [3], which is comparable with the average size of the cement grains [4]. The porosity of this region depends on the packing of the particles against the substrate (or aggregate) surface [4–6] (wall effect), on the local water content [7], and on

*Abbreviations:* ITZ, interfacial transition zone; CPV, Type Cement Portland according to Brazilian standard ABNT NBR 5733:1991; NBR, Brazilian standard; VSA, volumetric surface area; BET, Brunauer-Emmett-Teller; XRF, X-ray fluorescence; ABNT, Brazilian National Standards Organization; RH, relative humidity; PUNDIT, portable ultrasonic nondestructive digital indicating tester.

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the hydration degree of the binder [1,8]. The capillary absorption of porous recycled aggregates or masonry can also modify the porosity of the ITZ [9–12]. If capillary forces are sufficient to pull fine particles towards the interface, there will be a reduction of local water/solids ratio by increasing particle packing and/or by modifying hydrated products [1,3,13], thus improving the contact area between the substrate and the matrix.

According to Scrivener *et al.*, the ITZ porosity and its interconnectivity are most significant in the first 15–20  $\mu\text{m}$  closest to the surface [4]. The increase of packing by particles finer than conventional cements (<10  $\mu\text{m}$ ) [14] can promote a densification of ITZ solely by the filling effect [4], yet the fine particles will also act as nucleation sites and aid hydration [15] as a side effect. Pozzolanic materials, for instance, react with calcium hydroxide and water to form hydrated silicate calcium (C-S-H), thus further densifying the ITZ [16]. There have been a number of studies demonstrating that it is possible to reduce the porosity of the ITZ and improve the strength of concrete by adding supplementary materials such as silica fume, blast-furnace slag and fly ash [17–25]. However, with an increasing demand for cement-based materials and a limited availability of fly ash and blast furnace slag [26], or the high cost of silica fume, there is a tendency to replace binders by almost inert ultrafines such as limestone [27].

$\text{CaCO}_3$  is not completely inert in cementitious materials as it can react with aluminates of the clinker such as  $\text{C}_3\text{A}$  or even with alumina-containing phases to produce carboaluminate products that can participate on microstructure densification [28–30]. The amount of limestone that can react is limited by the availability of aluminium and even when it is added in amounts as small as 5%wt, thermodynamic simulations show that not all of it is consumed [29]. For higher amounts, the filler and dilution effect are predominant. Up to date, there is no published evidence of the effect of binder replacement by high rates of limestone fines on the mechanical performance of ITZ.

In Europe, there are types of cement that contain 6% up to 35% wt of ground limestone [31]. In Brazil, this content varies from 0% to 10%wt [32–36]. Some studies have shown that replacing 15–20% wt of the binder with limestone, by intergrinding with clinker and without any control of the size distribution and surface area of each component, did not have a significant impact on compressive strength [37–39]. From the point of view of hardened concrete, the strength normally is not reduced by replacing 5–10%wt of binder with limestone, if water to solids ratio is constant. Menadi *et al.* have reported reduction of 20% on compressive and flexural strengths (1 year) of both mortar and concrete in which cement had been partially replaced by 20%wt of limestone, because of water/cement ratio was kept constant [40]. Similarly, Das *et al.* have obtained decrease of 10% on 28-days strength of mortars with replacement rate of 35%vol and constant water/powder ratio [41]. In contrast, according to Proske *et al.*, the addition of 75–150  $\text{kg}/\text{m}^3$  of limestone powders in concretes with binder content of 150  $\text{kg}/\text{m}^3$  can increase the compressive strength in 44% and this additions is essential to provide an optimum paste volume [27]. Moreover, Palm *et al.* verified that for a constant water/clinker ratio concrete made with cements with 50%wt limestone have systematically a higher compressive strength. It can be attributed to the reduction of total water in mixes with limestone rich cements that leads to a higher solid volume in the concrete and a lower porosity [42].

The use of high contents of fine particles in concentrated cementitious suspensions involves much more intense surface forces. The yield stress generally decreases with the content of fine particles in cementitious pastes [43–45], but the viscosity varies greatly with different types and quantity of fines. Zhang and Han [45] found that the viscosity of cement paste can decrease with the use of silica fume, fly ash and limestone when the quantity

added is smaller than or equal to 15%. Vance *et al.* [44] verified that the plastic viscosity is independent of the limestone replacement level up to 35%vol and dependent solely on the volumetric solid loading and the particle size distribution of the solids in the suspension.

Surface area and particle morphology – besides the effects of particle size distribution – also play an important role on the rheological behaviour of concentrated suspensions. Rubio-Hernández *et al.* [46] demonstrated that the replacement of cement by fly ash (up to 20%wt) reduced both Herschel–Bulkley consistency  $k$  (for water/solids ratio between 0.35 and 0.45) and yield stress (especially at lower water/solids ratio) of the pastes, not only because of fly ash smaller particle size than that of the cement, but also due to the spherical shape of its particles [46]. For concretes with approximately 30%wt of calcium carbonate fine, Esping [47] showed that both yield stress and plastic viscosity increased considerably due to the use of particles with higher surface area. Some of these particles were even coarser than others used, but because of their roughness and/or particle shape, the effects of surface area were dominant on the rheological behaviour of the studied systems. The results indicated that an increase of specific surface area ( $\text{BET}_{\text{water}}$ ) of 1  $\text{m}^2/\text{g}$  corresponds to an additional water quantity of approximately 0.8% by mass of limestone content to keep concrete flowability constant [47].

Simulation results have shown that the benefits derived from the addition of fine particles can be reduced or even eliminated if the particles are flocculated in the system [48]. Flocculation has also major effects on rheological behaviour of cement suspensions, since it reduces particle mobility and increases phase separation tendency [49]. An alternative to maintain fluidity without increase the water demand is the use of dispersant admixtures. The addition of these superplasticizers reduces the yield stress, i.e., inter-floc interaction, since these polymers stabilized the particles, hindering their agglomeration. For limestone particles, this effect is more evident with the use of naphthalene or polycarboxylate admixtures in comparison to melamine or lignosulfonate types [50].

Rubio-Hernández *et al.* [51] showed that for highly dispersed pastes (containing simultaneously polycarboxylic acid and lignosulfonate) both yield stress and plastic viscosity decreased considerably with increasing replacement content of cement for finer limestone filler up to 70%wt [51]. More recently, based on an extensive experimental work with 11 different types of particles (limestone, quartz and granite) in cement pastes, Damineli *et al.* concluded that the dispersing admixture (polycarboxylate) consumption and rheological properties of pastes could vary greatly depending on the physical characteristics of the particles such as particle size distribution, surface area and morphology [52]. In some formulations, both yield stress and viscosity decreased with 50%wt of substitution of cement for fine particles, while in others the reduction was observed only in the yield stress. Two extreme cases displayed a considerable increase of both rheological parameters even with a higher content of dispersing admixture (compared to the pure cement paste), owing to irregular shape and high surface area of particles [52].

Adding limestone fines with suitable physical features in replacement of cement can therefore be beneficial to improve the packing of the system and consequently improve ITZ mechanical properties. So far, there is no direct evidence of the effect of rheological behaviour or flocculation (by using high content of limestone fines) on ITZ properties. Besides considering the ongoing scenario with high demands for housing and infrastructure alongside increasing sustainability needs, the improvement of the properties of low-cement compositions by choosing appropriate inert (or almost inert like limestone) filling materials, appears as an important alternative to the reactive supplementary materials.

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