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## Linking fresh and durability properties of paste to SCC mortar



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#### ABSTRACT

In the last years many approaches to design SCC have been developed, but it remains a very complex process since it is necessary to manipulate several variables and understand their effects on concrete behaviour (fresh and hardened state). The prediction of concrete or mortar behaviour based on paste properties will be a significant contribution to simplify SCC design. With this purpose, two statistical experimental designs were carried out, one at paste level and the other at mortar level, to mathematically model the influence of mixture parameters on fresh and durability properties. The derived numerical models were used to define an area, labelled by self-compacting zone at paste level (SCZ), where fresh properties of the paste enable the design of SCC mortar. Furthermore, in order to extend this link to durability properties, the effect of including aggregate in cement paste was evaluated by means of the electrical resistivity test.

#### 1. Introduction

Durability of concrete structures is presently looked at with great concern as it represents a challenge to achieve sustainable development in construction. Self-compacting concrete (SCC), initially developed in Japan, corresponds to an advanced special concrete type as it leads to technological, economic and environmental benefits. The main advantage of this sustainable technology lies in the unneeded compaction during placing, thus leading to a homogeneous and more durable material. In the fresh state, SCC must show filling ability, resistance to segregation and passing ability [1]. The selection of constituent materials and the design of mix proportions are essential factors to achieve adequate fresh properties. To produce SCC, a good balance between deformability and resistance to segregation has to be accomplished, which can be made possible by the use of chemical admixtures (superplasticizers, viscosity agents, etc.) combined with high concentrations of fine particles (cement and mineral admixtures) [2]. In addition, the characteristics of fine and coarse aggregate are also very important. With the growing variety of materials available to produce concrete, the mix design process has become complex since it is necessary to manipulate several variables and their interaction is difficult to predict. Indeed, to achieve the adequate performance in fresh and hardened states further work is needed to better understand the effect of mixture parameters governing material performance [3].

Several different mix-design methods have been developed by many academic institutions and construction industry companies, but still, there is no standard method for SCC mix design [1]. Typically, mixture optimization is based on a trial an error approach where each parameter is changed one at a time to assess its influence on concrete properties. This process does not permit understanding interactions of the mixture parameters, may involve carrying out a large and unpredictable number of trial batches and does not guarantee an optimal general solution. One of the methodologies that lately has been applied in the SCC mix design is the statistical experimental design. The derived statistical models established on the basis of a factorial design, highlight not only significance of the mixture parameters but also their interactions on concrete properties. Using such numerical models, a multi-parametric optimization can be carried out, with the user controlling the goal of the optimization and the significance of each experimental parameter. In fact, this approach increases the efficiency in selecting the optimum mix proportions for a given set of constraints (related to fresh and hardened properties and economic constraints) based on a limited number of experimental data points [3-5]. An additional advantage of the factorial experimental design is that there is some freedom to define the mixture parameters (it can be applied to paste, mortar or concrete) and to select the responses to be analysed (e.g. rheological parameters, empirical fresh tests results, durability properties, etc.) [6]. This mix-design approach was followed in the present work.

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Several different design methods found in the literature consider SCC as a suspension of aggregates in paste, separating optimization of the granular skeleton grading, paste volume and paste composition. In fact, paste plays a major role in concrete workability and therefore it is reasonable to expect that there is a direct relationship between paste and concrete flow behaviour [7]. The prediction of the concrete behaviour based on paste properties facilitates the design of SCC, reduces the volume of material required for testing and takes advantage of greater accuracy and precision of paste rheology tests [7,8]. The rheological behaviour of cement paste is controlled by the same factors that control any other suspension, namely, by macro-level factors like particle size, size distribution, shape, texture, density, water content, etc., and by micro-level forces such as colloidal, Brownian and viscous forces. Depending on the size of the particles, on their volume fraction in the mixture and on external forces (applied stress or strain rate), one or several of these interactions dominate (referred by Flatt [9]). Colloidal particles forces dominate, to a large extent, the complex and time dependent behaviour of cement paste, while for most aggregate sizes, only viscous forces are relevant [6]. According to Wallevik and Wallevik [10] rheology of fresh concrete or mortar is much simpler than rheology of cement paste, due to the fact that the time-dependent behaviour (thixotropy and structural breakdown) is more pronounced in cement paste because of the absence of aggregates, which act as a very effective grinder and

The objective of this paper is therefore to establish a link between paste and mortar which exhibit adequate fresh properties (deformability and viscosity) to produce self-compacting concrete. Furthermore, in order to extend this link to durability properties, the effect of including aggregate in cement paste was evaluated by means of the electrical resistivity test. With this purpose, two statistical experimental designs were conducted, with the same set of materials, one at paste level and the other at mortar level (including reference sand). At paste level, numerical models were established relating mixture parameters to rheological properties (vield stress and plastic viscosity), empirical fresh properties (flow diameter and free water content) and a durability property (electrical resistivity). At mortar level empirical fresh properties (flow diameter and flow time), a mechanical property (compressive strength) and the same durability property evaluated in paste (electrical resistivity) were assessed. The mortar numerical models were used to find optimal solutions that satisfy SCC fresh requirements, i.e. to determine the range of mortar mixture parameters where deformability and viscosity coexist in a balanced manner. By using the mixture parameters of the optimized mortars and the derived numerical models to describe paste properties, it was possible to define an area, labelled by self-compacting zone [11,12] at paste level, where the rheological properties of the paste enable the design of a self-compacting mortar. Furthermore, the influence of the aggregate in the electrical resistivity of mortar was studied, which allowed to draw contour plots that aid in the design of SCC mortar with defined durability requirements, based on tests at paste level only. Additionally the correlation between rheological and empirical tests results was discussed and the evolution of paste rheological behaviour during the hydration process was assessed.

#### 2. Experimental programme

#### 2.1. Materials characterization

The mortar and paste mixes investigated in this study were prepared with ternary mixtures including white cement (CEM II/A-L 52.5N according to EN 197-1 [13]) and two mineral additions,

metakaolin and limestone filler. The chemical composition and some physical properties of the cement and the two mineral additions are presented in Table 1. The particle size distribution performed by a laser diffraction granulometer is shown in Fig. 1. A polycarboxylate type high range water reducing admixture was used having a specific gravity of 1.07 and 26.5% solid content. In mortar mixes a reference sand was employed, conforming to EN 196-1 [14]. Reference sand used is a siliceous round natural sand (0.08–2 mm) with a specific gravity of 2.63 and an absorption value of 0.30%. Distilled water was used for all paste and mortar mixes.

#### 2.2. Experimental design

The experiments were designed according to a statistical design approach known as two-level factorial design  $(2^k)$ . This is a process of planning experiments in order to collect appropriate data that can be analyzed by statistical tools, such as analysis of variance, resulting in a valid basis for deriving an empirical numerical model that expresses the relationship between the input variables (e.g. mixture parameters) and response variables (e.g. fresh or hardened properties of paste/mortar). Factorial design is frequently used in experiments involving large number of parameters (input variables) and when it is important to study not only the isolated significance of each parameter in response but also their interaction [15]. According to studies conducted by some authors [4,16–18] the response surface of mortar and paste properties, fresh and hardened properties, exhibit some curvature, therefore, in this study experiments were designed according to a factorial central composite design adequate to fit a second order model. The generic form of a second order model is:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i < j} \sum_{i < j} \beta_{ij} x_i x_j + \varepsilon$$
 (1)

where y is the response of the material;  $x_i$  are the independent variables;  $\beta_0$  is the independent term;  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the coefficients of independent variables and interactions, representing their contribution to the response and  $\varepsilon$  is the random residual error term representing the effects of variables or higher order terms not considered in the model.

To define paste composition, four independent variables  $x_i$  were selected, namely, water to powder volume ratio  $(V_w/V_p)$ , water to cement weight ratio (w/c), superplasticizer to powder weight ratio  $(S_n/p)$  and metakaolin to cement weight ratio (mtk/c). For mortar mixtures it was necessary to include an additional variable to define the composition completely, which was sand to mortar volume ratio  $(V_s/V_m)$ . The selection of these parameters, used widespread in the design of SCC mixtures, was based on the method developed by Okamura et al. [2]. A complete 2<sup>4</sup> factorial statistical design, corresponding to four factors at two levels, was selected for the studies carried out at paste level whereas for the study of mortar mixes a 2<sup>5-1</sup> fractional factorial statistical design was adopted. The fractional factorial design was selected for mortar experimental design since it involves fewer runs than the complete set of  $2^5 = 32$  runs, while it can still be used to obtain information on the main effects and on the two-factor interactions. Axial points and central points were added to both experimental designs (Central Composite Design), allowing, thereby a second-order model fitting. The effect of each factor was evaluated at five different levels, codified in  $-\alpha$ , -1, 0, +1, + $\alpha$ . In order to make the design rotatable (i.e. the standard deviation of the predicted response is constant in all points at the same distance from the centre of the design) the  $\alpha$  value should be taken equal to  $n_f^{1/4}$ , where  $n_f$  is the number of points in the factorial part of the design [5] [15]. The absolute value of each variable corresponding to a given level

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