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Study on properties of self-compacting concrete modified with nanoparticles

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ABSTRACT

The paper presents the results of studies of a total of 11 series of self-compacting concrete, which were modified with different amounts of the following nanoparticle additives: SiO_2, TiO_2 and Al_2O_3, and also a reference concrete without the addition of nanoparticles. The study included the rheological properties of concrete mixes and the physical and mechanical properties of a hardened self-compacting concrete. The characteristics of air pores obtained using a computer image analyser and analysis of a microstructure with the use of a computer microtomograph are also presented. The paper contains the results of tests of compressive strength, flexural strength, hardness and elastic modulus, which were obtained using the nanoindentation technique. The obtained results were analyzed and commented on.

1. Introduction

Self-compacting concrete (SCC) is increasingly used in the construction industry [1–7]. In comparison with ordinary concrete (OC), the SCC contains a greater amount of fine particles, high dosage of admixtures and has more optimized packing density which provides the desired fluidity and viscosity. It was proven that modification of concrete with very fine particles enhance its both physical and mechanical properties. Addition of fly ash has a beneficial effect on compressive and flexural strength of concrete [8]. Freeze–thaw resistance is improved as well [9]. Active filling material positively influence on delaying the destructive processes in concrete and increase cement composites' fracture toughness [10,11]. Recently possible application of nanoparticles in Portland cement based materials were studied [12,13]. The most commonly used nanoparticles include: SiO_2, Al_2O_3, CuO, TiO_2, ZnO_2, Fe_2O_3 and Cr_2O_3. Their dosage varies from 0.5 wt.% to 12 wt.% of cement depending on the type of nanoparticles, the type of base material (cement paste, mortar, concrete) and aims of the modification. However, based on the review of literature the optimum range appears to be between 1 wt.% and 4 wt.% of the cement [14]. Other nanomaterials added to concrete include also nanoclays, nanotubes and nanofibers [15–17]. The published so far test results show that rheological

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properties of concrete appear to be affected by nanoparticles [18–20]. Partial replacement of Portland cement with nano-Al_2O_3 or nano-SiO_2 worsens workability of SCC which requires higher amount of superplasticizer [21]. However, selected physical properties of concrete modified with nanomaterials are improved. Porosity is reduced especially in the range of meso and macro pores [22]. The improvement of the porosity results in a decreased permeability [23] and lowered absorption [24]. The addition of nanoparticles tends to reduce the shrinkage [23] and has a positive effect on the corrosion resistance of concrete [24,18]. Mechanical properties of concrete modified with nanoparticles are also improved. Aciting as a filler nanoparticles strengthen the microstructure of cement reducing quantity and size of $Ca(OH)_2$ crystals [25] and accelerate C–S–H gel formation [26]. In the presence of nano-additives compressive and flexural strength is increased [27,28] and the abrasion resistance of concrete is improved [29]. The influence of nanoparticles on concrete elastic modulus and hardness can be measured in different ranges from nano through to micro and macro [30,31]. Nanoparticles appear to enhance the concrete hardness elongating the silicate chain of calcium silicate hydrate (C–S–H) [32]. Besides enhancing physical and mechanical properties of cementitious materials, some nanoparticles like nano-TiO_2, may be used to create self-cleaning concrete [33]. Uniform dispersion and formation of agglomerates appear to be the main problem related to incorporation of nanoparticles which requires the usage of surface surfactants to stabilize water dispersions [15,23,34,27]. Nanoparticles are also added in a powder form [29,35] to concrete mix, what has a positive effect on the properties of obtained hardened concrete. Some studies show no significant effects of method of addition on distribution of nano-SiO_2 [36].

The purpose of this study is to increase knowledge in this subject by performing relatively comprehensive studies to show the effect of the addition of nanoparticles (SiO_2, TiO_2 and Al_2O_3), which were added in different amounts in relation to the weight of cement, on the properties of SCC. The study included the rheological parameters of a concrete mix and the physical and mechanical properties of a hardened SCC. The maximum diameters of the slump and subsidence time were measured during the rheological studies of the concrete mix. Tests of the physical properties included the measurement of the characteristics of air pores (the total air content and the content of micropores with a diameter below 300 μm) using a computerized image analyzer. In addition, analysis of the microstructure of the examined concretes was carried out using a computer microtomograph, in which three-dimensional reconstructions were created and the amount of air pores in the tested samples measured. The tested mechanical parameters were compressive strength and flexural strength and also hardness and elastic modulus that were measured using the nanoindentation technique.

2. Experimental setup

A total of 11 self-compacting concrete mixes were prepared, of which one was a reference (S1) without a nano-additive. The following components were used to prepare the self-compacting test concretes: Portland cement CEM I 52.5R produced by Finnsementti Oy, complied with EN 197-1 [37], with a specific surface area of 0.6 m^2/g, specific gravity of 3.15 g/cm^3 and chemical composition presented in Table 1; polycarboxylate based superplasticizer, type Glenium Sky 600 produced by BASF with a specific gravity of 1.06 g/cm^3; tap water; crushed granite aggregate with an average specific gravity of 2.67 g/cm^3, absorption capacity of 0.2%, fractions of 10–5, 5–2, 2–1, 1.2–0.5, 0.6–0.1 mm and a fraction with a grain size <0.1 mm acting as a fine filler. Grading curve of used aggregates is presented in Fig. 1. The applied additives were three different types of nanoparticles in powder form: nano-SiO_2, nano-TiO_2, nano-Al_2O_3. The nano-SiO_2 had a particle size of 10–20 nm (BET), density of 2.4 g/mL at 25 °C, purity of 99.5% and was added as 0.5 wt.%, 2.0 wt.% and 4.0 wt.% of the cement replacement. The nano-TiO_2 had a particle size <25 nm, density of 3.9 g/mL at 25 °C, purity of 99.7%, specific surface are of 50 m^2/g and was added as 0.5 wt.%, 2.0 wt.% and 4.0 wt.% of the cement replacement. The nano-Al_2O_3 had a particle size <50 nm (TEM), specific surface area of 40 m^2/g and was added as 0.5 wt.%, 1.0 wt.%, 2.0 wt.% and 3.0 wt.% of the cement replacement. All used nanoparticles were supplied by Sigma–Aldrich company. The SEM images of the used nanoparticles are shown in Fig. 2. The W/C ratio was equal to 0.42 for all test concretes. Mix compositions are shown in Table 2.

Table 1 – Chemical composition of Portland cement CEM I 52.5R.

Component	[wt.%]
CaO	65.0
SiO_2	22.0
Al_2O_3	5.4
Fe_2O_3	3.3
MgO	3.2
Cl^-	≤0.08
Loss of ignition	3.0

Mixing of concrete was done using pan mixer and consisted of two phases. First, cement was mixed with aggregates and nano-powder for 2 min. In the next step, water with superplasticizer was added and mixed for another 2 min. The workability was assessed by determination of the maximum diameters of the slump flow (D) and subsidence time (T_{500}) during which the concrete mix coming out of the cone reaches 500 mm in diameter. Tests were performed according to the "Specification & Guidelines for Self-Compacting Concrete" prepared by EFNARC (European Federation for Specialist Construction Chemicals and Concrete Systems) [38].

Physical properties were determined with Nikon microscope SMZ800 coupled with Sony DXC950P camera and software Image Pro Plus 4.1 with the Scope Pro module. Analysis was done on 25 mm thick polished slices with a side length of 100 mm. Tests covered pores size range from 4 to 4000 μm. The test procedure was carried out in accordance with PN-EN 480-11:2008 on 90 days old samples [39] maturing in laboratory conditions. The measured parameters were the total air content (A) and content of the micropores having diameter of less than 300 μm (A_{300}).

Microstructural analysis was done using a Skyscan 1172 computer microtomograph equipped with an 11 MPix resolution camera. A lamp voltage of 100 kV was used. Due to the