

## The Effects of ZrO<sub>2</sub> Nanoparticles on Physical and Mechanical Properties of High Strength Self Compacting Concrete

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In this work, strength assessments and coefficient of water absorption of high performance self compacting concrete containing different amounts of ZrO<sub>2</sub> nanoparticles have been investigated. The results indicate that the strength and the resistance to water permeability of the specimens are improved by adding ZrO<sub>2</sub> nanoparticles in the cement paste up to 4.0 wt. (%). ZrO<sub>2</sub> nanoparticles, as a result of increased crystalline Ca(OH)<sub>2</sub> amount especially at the early age of hydration, could accelerate C-S-H gel formation and hence increase the strength of the concrete specimens. In addition, ZrO<sub>2</sub> nanoparticles are able to act as nanofillers and recover the pore structure of the specimens by decreasing harmful pores. Several empirical relationships have been presented to predict flexural and split tensile strength of the specimens by means of the corresponding compressive strength at a certain age of curing. Accelerated peak appearance in conduction calorimetry tests, more weight loss in thermogravimetric analysis and more rapid appearance of the peaks related to hydrated products in X-ray diffraction results, all indicate that ZrO<sub>2</sub> nanoparticles could improve mechanical and physical properties of the concrete specimens.

**Keywords:** ZrO<sub>2</sub> nanoparticles, water permeability, strength, pore structure, thermogravimetric analysis

### 1. Introduction

Advancements in concrete technology have resulted in the development of a new type of concrete which is known as self compacting high performance concrete (SCHPC). The qualities of SCHPC are based on the concept of self compacting high performance concretes. Self compacting concrete (SCC) is a fluid concrete that spreads through congested reinforcement, fills every corner of the formwork, and consolidated under its weight<sup>1</sup>. SCC necessitates excellent filling ability, good passing ability, and adequate segregation resistance. But it does not include high strength and good durability as significant performance criteria. On the other hand, high performance concrete (HPC) has been defined as a concrete which is appropriately designed, mixed, placed, consolidated, and cured to provide high strength and low convey properties or good durability<sup>2</sup>. HPC exhibits good segregation resistance, but does not provide excellent filling and passing ability, and therefore needs external means such as rodding or vibration for suitable consolidation. Hence, a concrete that fulfills the performance criteria of both SCC and HPC can be referred to as SCHPC. An SCHPC is that concrete, which offers excellent performance with respect to filling ability, passing capability, segregation resistance, strength, transport properties and durability.

Virtually all research has used SCC which includes active additions to satisfy the great demand for fines needed for this type of concrete, thereby improving their mechanical properties in comparison with NVC. Köning et al.<sup>3</sup> and Hauke<sup>4</sup> registered strength increase in SCCs made with different amount of fly ash. According to Fava et al.<sup>5</sup>, in SCCs with granulated blast furnace slag, this increase is also evident. On the other hand, when limestone filler is used, Fava et al.<sup>5</sup> and Daoud et al.<sup>6</sup> achieved a tensile strength in SCC lower than the other normal types of concrete.

Permeability of concrete is defined as the movement of liquid and/or gas through a mass of concrete under a constant pressure gradient. It is an inherent property of concrete that chiefly depends

upon the geometric arrangement and characteristics of the constituent materials. The permeability of concrete is mainly controlled by the solidity and porosity of the hydrated paste present in bulk paste matrix and interfacial transition zone. In the hydrated paste, the capillary and gel pores can be distinguished. The gel pores are very small. Although they constitute a network of open pores, the permeability of this network is very low. Conversely, the capillary pores are relatively large spaces existing between the cement grains. It is the capillary porosity that greatly affects the permeability of concrete<sup>7</sup>. The permeability of SCHPC is typically lower than that of ordinary concrete. The previous research showed that SCHPC results in very low water and gas permeability<sup>8,9</sup>. This is mostly attributed to the superior flow properties, dense microstructure and refined pore. Good flow properties result in superb packing condition due to better consolidation, and thus contribute to reduce the permeability of concrete.

Since strength assessments and water permeability of concrete are joined together to affect the final performance of concrete, considering mechanical properties in terms of various types of strengths together with physical properties of concrete specimens seems essential. Hence, in this work, both physical and mechanical properties of concrete specimens have been studied.

As our knowledge, there are few works on incorporating nanoparticles about SCCs to achieve improved physical and mechanical properties. Only, there are several reports on incorporation of nanoparticles in NVCs which most of them have focused on using SiO<sub>2</sub> nanoparticles<sup>10-19</sup> and TiO<sub>2</sub> nanoparticles<sup>20,21</sup>. There are a few studies on incorporating nano-Fe<sub>2</sub>O<sub>3</sub><sup>22</sup>, nano-Al<sub>2</sub>O<sub>3</sub><sup>23</sup>, and nanoclay particles<sup>24,25</sup>. Additionally, a limited number of investigations are dealing with the manufacture of nanosized cement particles and the development of nanobinders<sup>26</sup>. Previously, a series of works<sup>27-34</sup> has been conducted on cementitious composites by adding different

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**Table 1.** Chemical and physical properties of Portland cement wt. (%).

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	Loss on ignition
Cement	21.89	5.3	3.34	53.27	6.45	3.67	0.18	0.98	3.21

Specific gravity: 1.7 g.cm<sup>-3</sup>.

nanoparticles evaluating the mechanical properties of the composites. Nanoparticles can act as heterogeneous nuclei for cement pastes, further accelerating cement hydration because of their high reactivity, as nano-reinforcement, and as nano-filler, densifying the microstructure, thereby, leading to a reduced porosity. The most significant issue for all nanoparticles is that of effective dispersion.

Incorporating of other nanoparticles is rarely reported. Therefore, introducing some other nanoparticles which probably could improve the mechanical and physical properties of cementitious composites is inherent. The aim of this study is incorporating ZrO<sub>2</sub> nanoparticles into SCCs to study compressive strength and water permeability of self compacting high strength concrete. In addition, pore structure, thermal properties and microstructure of the concrete specimens have been evaluated. Several specimens with a constant amount of polycarboxylate superplasticizer (PC) have been prepared and their physical and mechanical properties have been considered when, instead of cement, ZrO<sub>2</sub> nanoparticles were partially added to the cement paste.

## 2. Materials and Methods

Ordinary Portland Cement (OPC) conforming to ASTM C150<sup>[35]</sup> standard was used as received. The chemical and physical properties of the cement are shown in Table 1.

ZrO<sub>2</sub> nanoparticles with average particle size of 15 nm and 45 m<sup>2</sup>/g Blaine fineness producing from Suzhou Fuer Import & Export Trade Co., Ltd was used as received. The properties of ZrO<sub>2</sub> nanoparticles are shown in Table 2.

Crushed limestone aggregates were used to produce self-compacting concretes, with gravel 4/12 and two types of sand: one coarse 0/4, for fine aggregates and the other fine 0/2, with a very high fines content (particle size < 0.063 mm) of 19.2%, the main function of which was to provide a greater volume of fine materials to improve the stability of the fresh concrete.

A polycarboxylate with a polyethylene condensate defoamed based admixture (Glenium C303 SCC) produced from Muhu (China) Construction Materials Co., Ltd was used. Table 3 shows some of the physical and chemical properties of polycarboxylate admixture used in this study.

Totally, two series of mixtures were prepared in the laboratory trials. C0-SCC series mixtures were prepared by cement, fine and ultra-fine crushed limestone aggregates with 19.2% by weight of ultra-fine ones and 1.0% by weight of polycarboxylate admixture replaced by water. N-SCC series were prepared with different contents of ZrO<sub>2</sub> nanoparticles with average particle size of 15 nm. The mixtures were prepared with the cement replacement by ZrO<sub>2</sub> nanoparticles from 1.0 to 5.0 wt. (%) and 1.0 wt. (%) polycarboxylate admixture. The superplasticizer was dissolved in water, and then the nano-ZrO<sub>2</sub> was added and stirred at a high speed for 3 minutes. Though nano-ZrO<sub>2</sub> cannot be dissolved in water, a smaller amount of nano-ZrO<sub>2</sub> can be dispersed evenly by the superplasticizer<sup>11</sup>. The water to binder ratio for all mixtures was set at 0.40<sup>[36]</sup>. The binder content of all mixtures was 450 kg.m<sup>-3</sup>. The proportions of the mixtures are presented in Table 4.

The mixing sequence for SCCs was consisted of homogenizing the sand and cementitious materials for one minute in the mixer

**Table 2.** The properties of nano-ZrO<sub>2</sub>.

Diameter (nm)	Surface volume ratio (m <sup>2</sup> .g <sup>-1</sup> )	Density (g.cm <sup>-3</sup> )	Purity (%)
15 ± 3	165 ± 12	<0.15	>99.9

**Table 3.** Physical and chemical characteristics of the polycarboxylate admixture.

Appearance	Yellow-brown liquid
% solid residue	Approximately 36%
pH	5.2-5.3
Specific gravity (kg.L <sup>-1</sup> )	Approximately 1.06
Rotational Viscosity (MPa)	79.30
% C	52.25
ppm Na <sup>+</sup>	9150
ppm K <sup>+</sup>	158

**Table 4.** Mixture proportion of nano-ZrO<sub>2</sub> particles blended self compacting concretes.

Sample designation	ZrO <sub>2</sub> nanoparticles (%)	PC content (%)	Quantities (kg.m <sup>-3</sup> )	
			Cement	ZrO <sub>2</sub> nanoparticles
C0-SCC1	0	1.0	450	0
N1-SCC1	1	1.0	445.5	4.5
N2-SCC1	2	1.0	441.0	9.0
N3-SCC1	3	1.0	437.5	13.5
N4-SCC1	4	1.0	432.0	18.0
N5-SCC1	5	1.0	427.5	22.5

Water to binder [cement + nano-ZrO<sub>2</sub>] ratio of 0.40.

and then approximately 75% of the mixing water were added. The coarse aggregate was introduced and then the superplasticizer was pre-dissolved in the remaining water and was added at the end of the mixing sequence. The total mixing time including homogenizing was 5 minutes.

Several types of tests were carried out on the prepared specimens:

- *Strength evaluation tests:* Cubic specimens with 100 mm edge length for compressive tests. Cylindrical specimens with the diameter of 150 mm and the height of 300 mm for split tensile tests and Cubic specimens with 200 × 50 × 50 mm edges length for flexural tests were made. The moulds were covered with polyethylene sheets and moistened for 24 hours. Then the specimens were demoulded and cured in water at a temperature of 20° C in the room condition prior to test days. The strength tests of the samples were determined at 2, 7 and 28 days of curing. Compressive tests were carried out according to the ASTM C 39<sup>[37]</sup> standard, split tensile tests were done in accordance to the ASTM C 496<sup>[38]</sup> standard and flexural tests were performed conforming to the ASTM C 293<sup>[39]</sup> standard.

After the specified curing period was over, the concrete cubes were subjected to related test by using universal testing machine. The tests were carried out triplicately and average strength values were obtained.

- *Water permeability tests:* Water permeability tests are performed with several methods such as percentage of water absorption, rate of water absorption and coefficient of water absorption. In this work, to evaluate the water permeability of the specimens, percentage of water absorption is an evaluation of the pore volume or porosity of concrete after hardening, which is occupied by water in saturated state. Water absorption values of ZrO<sub>2</sub> nanoparticle blended concrete samples were

measured as per ASTM C 642<sup>[40]</sup> after 2, 7 and 28 days of moisture curing.

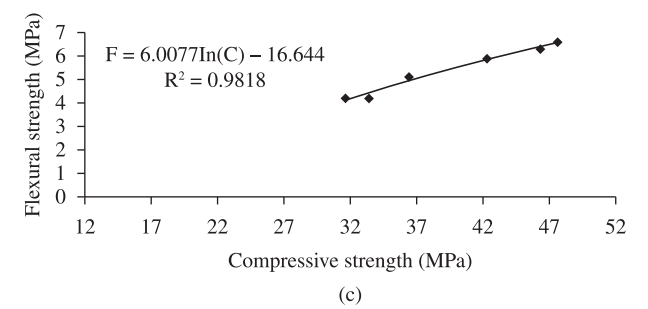
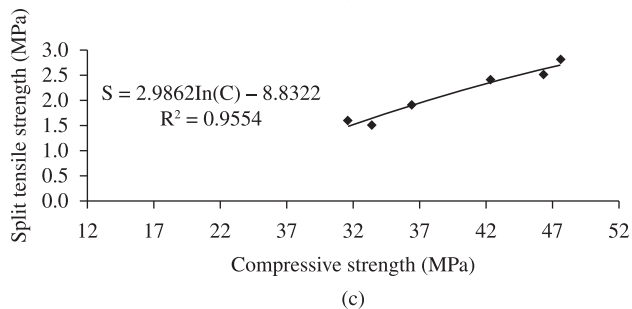
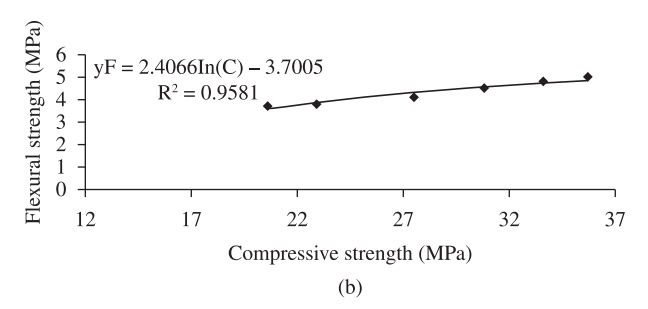
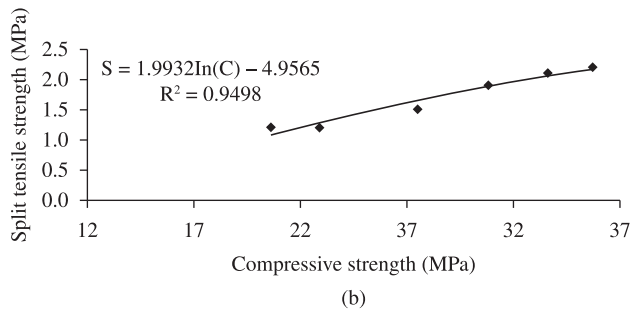
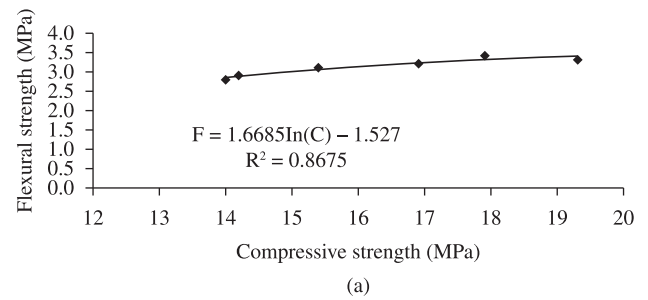
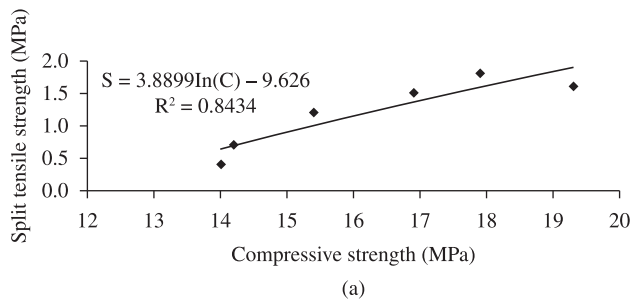
- *X-ray diffraction (XRD):* A Philips PW-1730 unit was used for XRD analysis which was taken from 4 to 70°.

### 3. Results and Discussion

Table 5 shows the compressive strength of C0-SCC and N-SCC specimens at 2, 7 and 28 days of curing. The results show that the compressive strength increases by adding ZrO<sub>2</sub> nanoparticles up to 4.0 wt. (%) replacements (N4-SCC series) and then it decreases, although adding 5.0 percent ZrO<sub>2</sub> nanoparticles produces specimens

**Table 5.** Strength assessments and water permeability of C0-SCC and N-SCC specimens.

Sample designation	SiO <sub>2</sub> nanoparticles (%)	Compressive strength (MPa)			Split tensile strength (MPa)			Flexural strength (MPa)			Percentage of water absorption (%)		
		2 days	7 days	28 days	2 days	7 days	28 days	2 days	7 days	28 days	2 days	7 days	28 days
C0-SCC1	0	14.0	20.6	31.6	0.4	1.2	1.6	2.8	3.7	4.2	2.30	4.28	3.89
N1-SCC1	1	14.2	22.9	33.4	0.7	1.2	1.5	2.9	3.8	4.2	4.09	2.10	1.93
N2-SCC1	2	15.4	27.5	36.4	1.2	1.5	1.9	3.1	4.1	5.1	4.41	2.00	1.69
N3-SCC1	3	16.9	30.8	42.3	1.5	1.9	2.4	3.2	4.5	5.9	4.78	1.87	1.40
N4-SCC1	4	17.9	35.7	47.6	1.8	2.2	2.8	3.4	5.0	6.6	5.18	1.58	1.16
N5-SCC1	5	19.3	33.6	46.3	1.6	2.1	2.5	3.3	4.8	6.3	4.96	1.68	1.29



**Figure 1.** The relationship between split tensile strength and compressive strength of the specimens cured at a) 2 days; b) 7 days; and c) 28 days. C denotes compressive strength and S denotes split tensile strength.

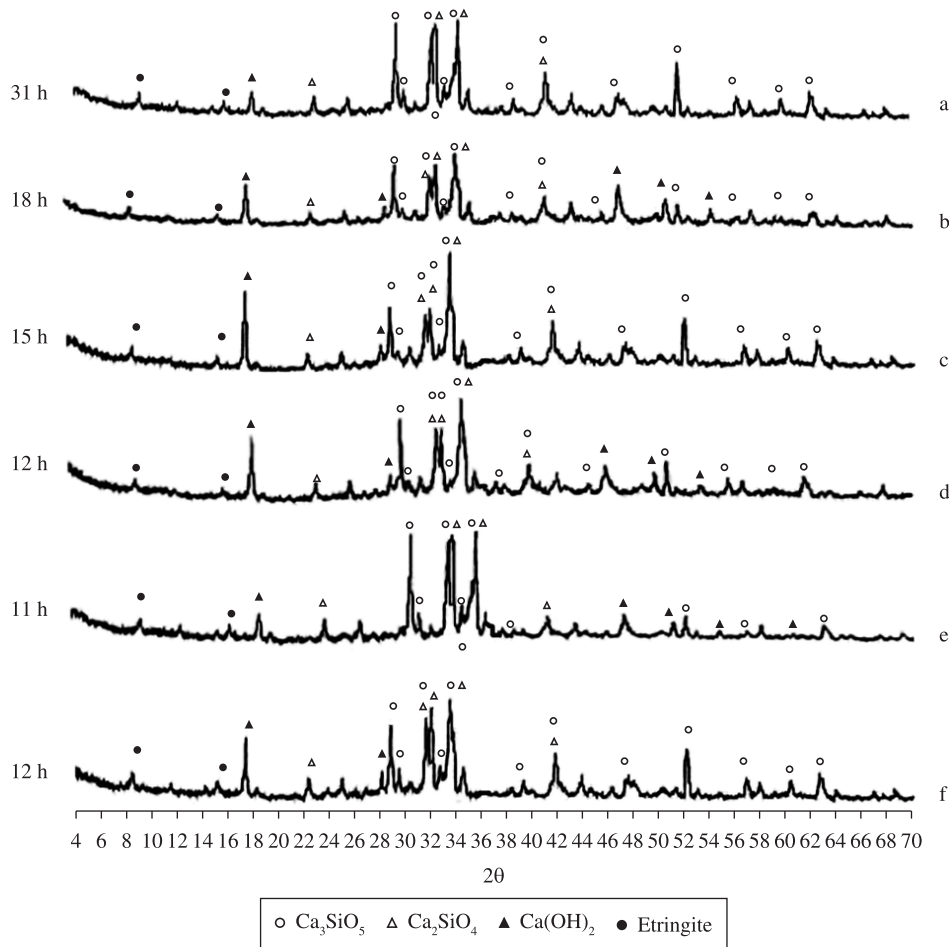
**Figure 2.** The relationship between flexural strength and compressive strength of the specimens cured at a) 2 days; b) 7 days; and c) 28 days. C denotes compressive strength and F denotes flexural strength.

with much higher compressive strength with respect to C0-SCC and N-SCC specimens with 1.0, 2.0 and 3.0 wt. (%) ZrO<sub>2</sub> nanoparticles. The reduced compressive strength by adding more than 4 wt. (%) ZrO<sub>2</sub> nanoparticles may be due to this fact that the quantity of ZrO<sub>2</sub> nanoparticles present in the mix is higher than the amount required to combine with the liberated lime during the process of hydration thus leading to excess silica leaching out and causing a deficiency in strength as it replaces part of the cementitious material but does not contribute to strength. Also, it may be due to the defects generated in dispersion of nanoparticles that causes weak zones. The higher compressive strength in the N-SCC series mixtures with respect to C0-SCC series is due to the rapid consumption of crystalline Ca(OH)<sub>2</sub> which quickly are formed during hydration of Portland cement specially at early ages as a result of high reactivity of ZrO<sub>2</sub> nanoparticles. As a consequence, the hydration of cement is accelerated and larger volumes of reaction products are formed. Also ZrO<sub>2</sub> nanoparticles recover the particle packing density of the blended cement, directing to a reduced volume of larger pores in the cement paste.

Table 5 shows the split tensile strength and the flexural strength of C0-SCC and N-SCC series concretes. Similar to the compressive strength, the split tensile strength and the flexural strength of all N-SCC specimens is more than those of C0-SCC specimens. In addition, the split tensile strength and the flexural strength of N-SCC series is increased by adding ZrO<sub>2</sub> nanoparticles up to 4.0 wt. (%) and

then it is decreased, similar to the compressive strength results. Since evaluations of strength with different tests are not affordable, here, the relationship between compressive strength and split tensile strength, and the relationship between compressive strength and flexural strength is presented. Figures 1a, 1b and 1c show the relationship between the splitting tensile strength and compressive strength of all mixes cured for 2, 7 and 28 days, respectively. In addition, Figures 2a, 2b and 2c show the relationship between the flexural strength and compressive strength of all mixes cured for 2, 7 and 28 days, respectively. In all curves, a logarithmic relation has been adopted to show this relationship. The R-squared values are also given in the figures and show a good compatibility between two specified strength. As figures show, at every age of curing, one may predict a specified strength by knowing at least one of the specimens' strength.

Table 5 shows the percentage of water absorption of the specimens. As Table 5 shows, the percentage of water absorption in C0-SCC specimens at 2 days of is lower than that of N-SCC series while at 7 and 28 days of curing, this value is lower for N-SCC series concrete. This may be due to more formation of hydrated products in N-SCC series at the early ages of curing. As mentioned above, ZrO<sub>2</sub> nanoparticles accelerate formation of cement hydrates and hence the specimens needs more water to produce these products. Therefore, at 2 days of curing, the consumption of water in N-SCC series is more than in C0-SCC series concrete. At 7 and 28 days of curing, the pore structure of N-SCC series concrete is improved and



**Figure 3.** XRD results indicating the formation of hydrated products for different N-SCC specimens: a) C0-SCC1; b) N1-SCC1; c) N2-SCC1; d) N3-SCC1; e) N4-SCC1; and f) N5-SCC1.



water permeability of these series is decreased with respect to the C0-SCC series concrete.

Table 5 also shows that the percentage of water absorption in N-SCC series at 7 and 28 ages of curing is decreased by increasing the ZrO<sub>2</sub> nanoparticles content up to 4.0 wt. (%) and then it is increased. Once again, this may be due to unsuitable dispersion of the nanoparticles in the cement paste when the content of the nanoparticles goes beyond 4 wt. (%). On the other hand, at 2 days of curing, more water requirement by increasing nanoparticles content up to 4.0 wt. (%) results in the decreased the coefficient of water absorption. Therefore, it can be suggested that with prolonged curing, increasing the ages and percentages of ZrO<sub>2</sub> nanoparticles can lead to reduction in permeable voids. This is due to the pozzolanic action and filler effects of ZrO<sub>2</sub> nanoparticles. Another finding is that the interfacial transition zone in concrete had improved due to pozzolanic reaction as well as filler effect of the ZrO<sub>2</sub> nanoparticles. This finding is partially in confirmation of the results of the study by Bui et al.<sup>41</sup>.

Figure 3 shows XRD analysis of C0-SCC and N-SCC specimens at different times after curing. As Figure 3 also shows, the peak related to formation of the hydrated products shifts to appear in earlier times indicating the positive impact of PC on formation of Ca(OH)<sub>2</sub> and C-S-H gel at early age of cement hydration.

#### 4. Conclusions

The results obtained in this study can be summarized as follows:

- As the content of ZrO<sub>2</sub> nanoparticles is increased up to 4 wt%, the compressive strength, split tensile strength and flexural strength of SCC specimens is increased. This is due to more formation of hydrated products in presence of ZrO<sub>2</sub> nanoparticles.
- ZrO<sub>2</sub> nanoparticles could act as nanofillers and improve the resistance to water permeability of concrete at 7 and 28 days and curing. At 2 days of curing, the coefficient of water absorption is increased by increasing the nanoparticles content up to 4.0 wt. (%) since the specimens require more water to rapid forming of hydrated products.
- Some empirical relationships in terms of logarithmic equations were provided to correlate the split tensile strength and flexural strength of a certain mixture to its compressive strength.

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