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Effectiveness Assessment of Superplasticizer Admixtures: Case of Self-compacting Concrete Reinforced with Fine Mineral Fillers

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Abstract

The use of superplasticizers in concrete production has become a common practice, especially when a high fluidity is required. On the other hand, the risk of segregation and the stability of these mixtures is ensured by the incorporation of a large volume of fine mineral additions. The present investigation is devoted to the study of the influence of three types of superplasticizers of different chemical compositions, namely: Combined Synthetic Polymers (CSP), Poly-Carboxylate Ether (PCE), and Modified Poly-Carboxylate Ether (MPCE) on the rheological behavior of self-compacting concrete (SCC) as well as on the mechanical properties at 3, 7, and 28 days of curing. Natural pozzolana (NP) and ground granulated blast furnace slag (GGBFS) were used as additions to stabilize the mixtures. The results revealed that the ether-based superplasticizer PCE gave the best workability and mechanical performance with low amounts (high efficiency). In addition, blast furnace slag promotes the obtaining of better properties whether in the fresh or hardened state compared to natural pozzolana.

Keywords: self-compacting concrete, superplasticizers, strength, workability, mineral fillers

1 Introduction

Many current structures are characterized by the complexity of their architecture (variable shapes and multiple curvatures), as well as by a high density of reinforcement, which often makes it difficult to use conventional plasticity concrete. In order to adapt the concretes to these structures, research has been carried out in recent years to develop concrete formulations characterized by high workability, while being stable (segregation, spreading and entrained air), with good mechanical properties and durability.
Self-compacting concrete (SCC) represents a new generation of concrete with invaluable properties. Thanks to its high fluidity, the implementation is affected without external vibration. This concrete allows the perfect filling of the formwork without any segregation of its components [1, 2]. In the fresh state, the main characteristics of this material are high flowability, passage capacity, and resistance to segregation [3, 4].
Several additions can be incorporated into the production of self-compacting concrete such as metakaolin (MK) [5, 6], fly ash (FA) [7, 8, 6], and ground granulated blast furnace slag

(GGBFS) [7]. These by-products play a significant role in the development of SCC properties whether in a fresh or hardened state [5, 6]. Thanks to their high specific surface area (higher compared to Portland cement) due to thorough grinding, some of these additions have a pozzolanic effect during cement hydration reactions. Indeed, in the presence of moisture, they can fix the portlandite (CH) to form new calcium hydrosilicates (CSH) thus contributing to the improvement of the mechanical properties of concrete [9, 10].

Natural pozzolana is among the mineral additions used as a cementitious additive to replace Portland cement. The use of natural pozzolana as an addition is motivated by its availability (low cost) as well as its advantageous properties such as reduced heat release, decreased permeability, and high chemical resistance, e.g., to acids [11, 12].

Ground granulated blast furnace slag (GGBFS) is a co-product of the metallurgical industry. This by-product is also used as an additive in concrete due to these multiple technical advantages [13, 14]. The incorporation of GGBFS into self-compacting concrete greatly enhances its compactness and consistency. Moreover, it also contributes to the protection of concrete against sulfate attack and the penetration of chloride ions [15]. Indeed, thanks to the lower density of the GGBFS compared to cement, the equivalent mass substitution gives rise to a larger volume of paste, which will consequently promote resistance to segregation and improve the workability of the concrete mixture. The study carried out by Oner and Akyuz [16] showed that the incorporation of ground granulated blast furnace slag in self-compacting concrete significantly improves the rheological behavior of the mixture. The same authors have shown that its incorporation with an optimal content reduces the water/binder ratio, which increases the compressive strength of the hardened SCC.

The high fluidity of self-compacting concrete mixtures is obtained using superplasticizer admixtures. Despite their incorporation with low contents, the latter have a significant influence on the fresh and hardened properties of SCCs [17, 18]. Depending on their chemical structure, there are several types of superplasticizers; among them the polycarboxylate (PC) based superplasticizer. Three (3) important parts make up its structure: a polyethylene backbone, grafted polyoxyethylene (POE) chains, and carboxylic groups as adsorbents. In the presence of this type of superplasticizer (PC), the dispersion is ensured essentially by the steric hindrance (thanks to the long-grafted side chains) instead of the negatively ionized groups (COO^-) which usually adsorb on the outer surface of cement grains [19].

The chemical structure, anionic monomer type, molecular mass, length of the main chain, and side chains, the ratio of anionic / non-ionic groups, and density of superplasticizers have a significant influence on the properties of cementitious materials. In recent years, the objective of several researchers has been to increase the effectiveness of chemical admixtures by modifying these parameters [20, 21, 22].

Evangeline K and Neelamegam [23] examined the influence of 3 types of superplasticizers, namely Sulfonated Naphthalene Formaldehyde (SNF), Poly-Carboxylate Ether (PCE), and Modified Poly-Carboxylate Ether (MPCE) on mechanical performance and workability of self-compacting concrete. The results showed that the workability retention of concrete with PCE allows a longer fluidity of the concrete. In addition, concrete containing MPCE gives better and longer flowability. Therefore, MPCE was found to be a better superplasticizer than the three other selected superplasticizers considered in the study, considering the characteristic behavior of fresh and hardened concrete.

Zhang et al., [24] developed a mathematical model for predicting the optimal mixture of self-compacting concrete depending on the properties of the cement additives (powders) and the

type of admixture (superplasticizer) used. The effectiveness of superplasticizers has also been tested in alkali-activated materials (geopolymers) [25].
This paper reports a comparative investigation of the effect of natural pozzolana (NP) and ground granulated blast furnace slag (GGBFS) on the fresh and hardened properties of self-compacting concrete (SCC) using three types of superplasticizers; the effectiveness of each admixture is assessed.

2 Materials and Methods

2.1 Raw Materials

2.1.1 Cement

The cement used in the present investigation is CEM II 42.5R containing 20% limestone fillers. The density and fineness of this binder are equal to 3.08 g/cm^3 and 3394 cm^2/g respectively. The chemical composition of the cement was carried out through X-ray fluorescence analysis while the mineral contents were calculated using Bogue's formulas. The results are displayed in Table 1.

Table 1: Chemical and mineralogical compositions of the CEM II 42.5R

Oxide	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**K_2O**	**Na_2O**	**SO_3**	**CaO_f**	**L.O.I**
(%)	15.71	4.15	2.52	60.71	2.38	0.59	0.18	1.79	1.42	1.63
Mineral	**C_2S**			**C_3S**		**C_3A**				
(%)	21			55		7				

2.1.2 Aggregates

Crushed coarse aggregates (3/8 and 8/15 mm) with a density equal to 2.73 g/cm^3 were used in this study. Their water absorption is estimated at 0.45%. River sand with a density of 2.67 g/cm^3 is used as a fine aggregate.

2.1.3 Mineral Additions

The mineral additions used in this study are natural pozzolana (NP) and ground granulated blast furnace slag (GGBFS). Their chemical compositions were obtained by XFR analysis and some of their characteristics are given in Table 2.

Table 2: Chemical compositions and characteristics of the additions used

Parameter **Chemical composition**	**Natural pozzolana (NP) (%)**	**Ground granulated Blast furnace slag (GGBFS) (%)**
SiO_2	43.67	50.76
Al_2O_3	14.30	9.90
Fe_2O_3	9.64	1.43
CaO	11.33	30.06

MgO	9.89	3.01
K_2O	1.60	1.47
Na_2O	3.10	1.37
P_2O_5	0.54	0.02
TiO_2	2.05	0.20
Loss on ignition (L.O.I)	3.72	1.65
Characteristics		
Fineness (cm^2/g)	5367	5847
Activity index at 28 days (%)	63.39	74.76
Density (g/cm^3)	2.70	2.67

2.1.4 Admixtures

Three different types of superplasticizers namely: Poly Carboxylate Ether (PCE); Modified Poly Carboxylate Ether (MPCE) and Combined Synthetic Polymers (CSP) were used in this experimental investigation. These admixtures were recovered from the GRANITEX Company. According to the product data sheets, their characteristics are given in Table 3.

Table 3: Characteristics of the admixtures (superplasticizers) used

Superpla-sticizer	**Form**	**Color**	**Density (g/cm^3)**	**pH**	**Active ingredient content (%)**	**Trade name**
PCE	Liquid	Light brown	1.07	6 - 6.5	30	Medaflow 30
MPCE	Liquid	Light brown	1.06	5 - 6	30	Medaflow 145
CSP	Liquid	Brown	1.12	6.5 - 7.5	36	Medaflow 113

2.2 Preparation, Optimization, and Storage of Samples

The volume of the paste is considered a basic factor when preparing the different SCC mixtures. European guidelines for the production of self-compacting concrete have been followed [26]. Using a Water/Powder (W/P) ratio of 0.35, six (6) concrete mixes were prepared. The total mass of powder considered in the formulation is around 450 kg/m^3. The content of the mineral additions, either natural pozzolana or ground granulated blast furnace slag (GGBFS) represents a ratio of 10% relative to the mass of cement. The proportions of the concrete mixtures are summarized in Table 4.

Table 4: Mix proportions

W/P ratio	**Water (L/m^3)**	**Cement (kg/m^3)**	**Addition (kg/m^3)**	**Sand (kg/m^3)**	**Coarse aggregate 3/8 (kg/m^3)**	**Coarse aggregate 8/15 (kg/m^3)**
0.35	157.85	410	41	926	289.83	566.8

The following codes (abbreviations) are assigned to the different mixtures according to:

a) Self-compacting concrete type;
b) The first letter of the addition incorporated, and
c) The type of superplasticizer (SP) used.

SCC-P-PCE: Self-Compacting Concrete with Pozzolana and Poly Carboxylate Ether.

SCC-P-MPCE: Self-Compacting Concrete with Pozzolana and Modified Poly Carboxylate Ether.
SCC-P-CSP: Self-Compacting Concrete with Pozzolana and Combined Synthetic Polymers.
SCC-G-PCE: Self-Compacting Concrete with Ground Granulated blast furnace slag and Poly Carboxylate Ether.
SCC-G-MPCE: Self-Compacting Concrete with Ground Granulated blast furnace slag and Modified Poly Carboxylate Ether.
SCC-G-CSP: Self-Compacting Concrete with Ground Granulated blast furnace slag and Combined Synthetic Polymers.

Optimization and dosage of superplasticizers are based on the slump flow test (Figure 1(a)). The optimal dosage of each superplasticizer, called saturation dosage allows the concrete to reach a slump diameter of 710 mm as shown in Figure 1(b). The recorded saturation dosages are given in Table 5.



Figure 1: a) Slump flow test, b) Slump diameter measurement

Table 5: Optimal (saturation) dosage of each superplasticizer according to the SCC mixture

	Superplasticizer dosage (%)	
SCCs	**SCC-P**	**SCC-G**
PCE	1.6	1.5
MPCE	2.5	2
CSP	3.3	2.6

The adsorbed content of each superplasticizer on the binder grains was evaluated using Altun et al. procedure [27]. A Total Organic Carbon (TOC) analyzer was employed. The adsorbed content was calculated using the formula (1):

$$Ad = \frac{(C_0 + C_c - C_1)V/x}{m} \quad (1)$$

Where *Ad* is the adsorbed content of the superplasticizer on binder (mg/l), C_0 is the TOC content of the superplasticizer solution without a binder (mg TOC/l), C_C is the TOC content of

the binder solution (mg TOC/l), C_1 is the TOC content of the superplasticizer solution when the latter is equivalently adsorbed on the binder surface (mg TOC/l), x is the mass TOC content of superplasticizer (mg TOC/mg), V is the superplasticizer solution volume (in l), and m is the mass of binder (in g).
The preparation of the samples intended for the mechanical tests is carried out by pouring the fresh concrete into molds of standard dimensions (160 mm × 320 mm), (either cylindrical or prismatic). The demolding is done after 24 hours; all the specimens obtained were stored in a climatic chamber (T = 20 °C; relative humidity RH = 90% ± 5) until testing. These controlled conditions ensure the protection of concrete specimens against desiccation.

2.3 Concrete Test Procedures

2.3.1 Fresh Concrete Tests

In order to evaluate the filling ability of the different fresh-state concretes, the slump-flow test and the T_{50} flow time were carried out according to British-adopted European standards [28]. Moreover, the L-box test and sieve segregation tests were performed according to SB EN 12350: 2010: Part 10 and Part 11 [29].

2.3.2 Hardened Concrete Tests

Mechanical characterization of the hardened SCCs was carried out after 2, 7, and 28 days of curing. Compressive strength, splitting tensile strength, and flexural tests were performed according to BS EN 12390 standards [30]. The samples intended for the compressive and tensile strengths are cylindrical specimens with a diameter of 160 mm and a height of 320 mm while those assigned for flexural tests are of prismatic shape having dimensions of 70 mm × 70 mm × 280 mm. The selected result represents the average of three trials.

3 Results and Discussion

3.1 Adsorption Content of Superplasticizers

The content of each superplasticizer (admixture) adsorbed on the cement particles is mentioned in Table 6.

Table 6: Content of superplasticizers adsorbed on cement grains

Superplasticizer (admixture)	Content of adsorption (mg/g binder)
PCE	0.927
MPCE	0.819
CSP	0.704

It can be seen from the results displayed in Table 6 that the amount of PCE adsorbed on the cement grains is higher than with the other two superplasticizers (MPCE and CSP). Generally, the adsorption characteristic of the superplasticizer on the cementitious particles is strongly related to the length of the main and side chains of the admixture. According to the literature [22, 31], the adsorption performance of the admixture improves with the decrease in the

length of the side chain; on the other hand, the steric hindrance effect becomes weaker. Also, as the main chain length increases, the number of anionic functional groups increases, and hence the electrostatic effect increases.
However, the elongation of the main chain of the superplasticizer leads to limited interaction with the cement and consequently low adsorption, due to the intertwining of the long main chains [22, 31].

3.2 Correlation of Superplasticizer Dosages

Figure 2 highlights the correlation between the optimal superplasticizer dosages obtained when the spreading diameter has reached 710 mm, and this, according to the mineral addition (pozzolana or granulated slag) contained in the SCC mixture.
It can be noted that the superplasticizer dosages are lower in the mixtures of SCCs containing granulated blast furnace slag compared to those containing natural pozzolana, and this is valid for all types of superplasticizers used. The dosage increases in a quasi-linear manner and the regression analysis gave a coefficient of determination R^2 equal to 0.992, which means that the established linear mathematical model is almost perfect.
It should be noted that the saturation dosages of superplasticizers obtained using the slump flow test depend on the type of mineral addition contained in the SCC mixture. Indeed, low dosages were recorded with SCCs formulated with granulated slag compared to those of natural pozzolana. Thus, the lowest dosage was recorded with the poly-carboxylate ether (PCE) with a minimum amount of 1.5%; this is due to its important dispersing effect.

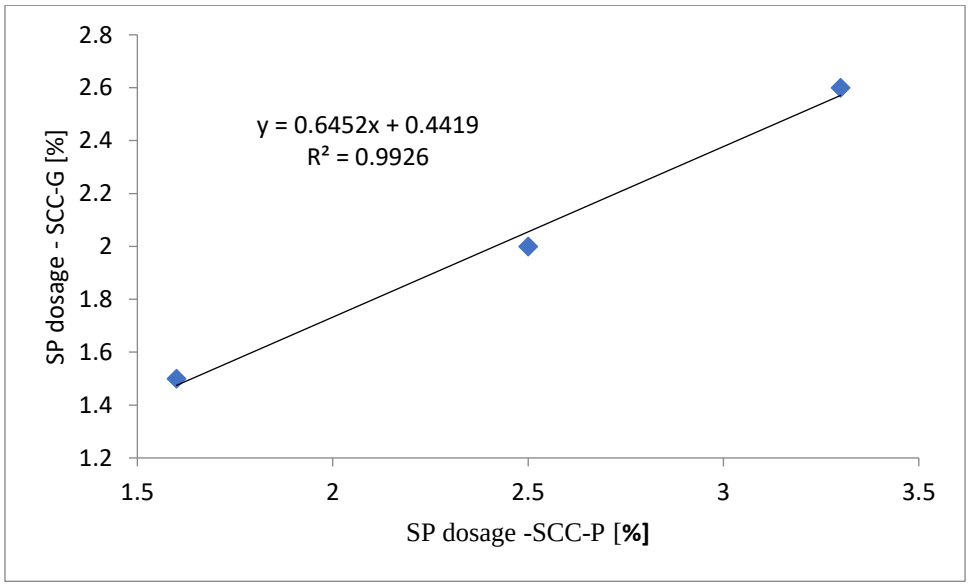


Figure 2: Relationship between the saturation dosages of superplasticizers according to the mineral addition contained in the SCC mixture

3.3 Fresh Properties of Concrete

The evaluation of the rheological properties (fresh state) of the different mixtures of self-compacting concretes is carried out using standard tests, namely L-box, sieve segregation and T_{50} time tests. The results are given in Table 7.

Table 7: Results of tests in the fresh state

	L-box test			**Sieve segregation test (%)**			**T_{500} test (s)**		
	PCE	MPCE	CSP	PCE	MPCE	CSP	PCE	MPCE	CSP
SCC-P	H_1/H_2 = 0.84	H_1/H_2 = 0.87	H_1/H_2 = 0.83	6.29	6.17	3.54	2	2	2.06
SCC-G	H_1/H_2 = 0.88	H_1/H_2 = 0.88	H_1/H_2 = 0.90	4.5	4.6	5.9	2	2.26	2.18

The results of the L-Box tests showed that all concrete mixtures exhibit high height ratios (H_1/H_2), exceeding the minimum value of 0.80 required by EFNARC [32]. In addition, it can be seen that the best results are obtained with SCC containing ground granulated blast furnace slag as an additive.

For self-compacting concrete formulated with natural pozzolana, it can be noted that the best results (lowest risk of segregation) are obtained in concrete mixtures dosed with the combined synthetic polymer CSP, then MPCE, and finally PCE. On the other hand, opposite results were recorded in the concrete mixtures based on granulated blast furnace slag. The lowest rate (4.5%) was obtained in the concrete containing PCE.

Overall, the results obtained by the sieve segregation test make it possible to qualify all the compositions of the self-compacting concretes as stable because the values were in the range (0 to 15%) recommended by EFNARC [32], which means a minimum risk of segregation. This good stability recorded for all SCCs is probably due to a large amount of paste (cement + addition) contained in the concrete and eventually to the percentage of viscosity agent incorporated in the superplasticizer during its synthesis. Furthermore, it is useful to point out that better stability is obtained with the ground granulated blast furnace slag concrete compared with those containing natural pozzolana. This can be partly explained by the high specific surface of the GGBFS.

From the results displayed in Table 7, it is observed that the time (T_{50}) measured for the concrete to reach a spreading diameter of 500 mm is almost the same for all the compositions. The average recorded value is about two (2) seconds, a common slump time, often recorded in self-compacting concretes. The results obtained for all SCC mixtures are in line with the European guidelines for self-compacting concrete [26, 32].

3.4 Hardened Properties of Concrete

3.4.1 Compressive Strength

The results of the compressive strength of the different self-compacting concrete at 3, 7, and 28 days of curing are presented in Figure 3.

At curing ages of 3 and 7 days, it can be noted that all the self-compacting concretes dosed with MPCE superplasticizer give the best compressive strength, followed in the second

position by those dosed with PCE and finally the samples containing CSP admixture. On the other hand, at 28 days of curing, the concrete dosed with PCE admixture showed the highest value estimated at 58.90 MPa. CSP-based concretes often recorded the lowest values at all curing ages.

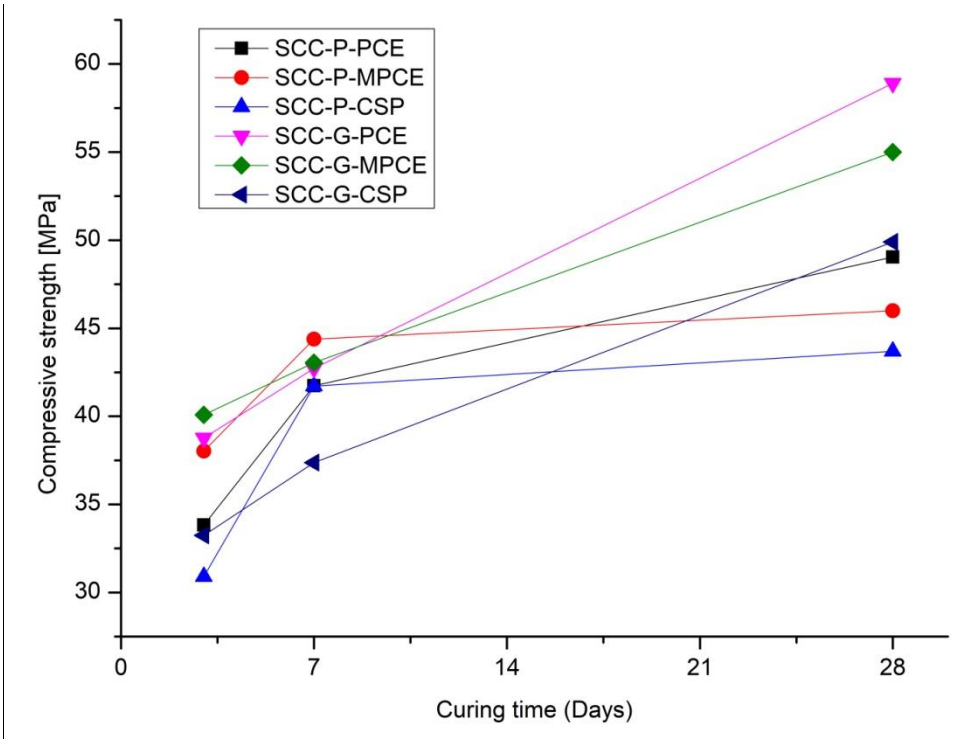


Figure 3: Compressive strength results at different curing times

On another register, except at 7 days of curing, where the compressive strengths are almost similar, it can be noted that the self-compacting concrete produced with blast furnace slag gives better mechanical performances compared with the concrete formulated with natural pozzolana. This can be explained by the filling effect (very fine particles which are inserted between the cement grains thus decreasing the porosity). This densification of the structure is valid for the two mineral additions. However, the blast furnace slag also improves the compressive strengths of concrete through the pozzolanic effect (ability to fix to the lime) [33]. Indeed, the calcium hydroxide (CH) formed during the cement hydration process reacts with the GGBS to produce secondary hydration which will again form CSH gel thus improving the mechanical resistance of the samples [34]. This reaction is favored by the large amount (more than 50%) of the silicon oxide present in the GGBFS as well as its high activity index (74.76%). In addition, the high specific surface of the slag, in particular, because of, the thorough grinding, contributes effectively to improve its reactivity.

3.4.2 Flexural Strength

The results obtained for the flexural strength at different curing ages are given in Figure 4. It can be noted that the values of the flexural strength of the different samples are lower than those recorded by the compressive strength tests. However, the trend of the evolution of these

two mechanical properties (flexural and compression) is practically similar, and this is valid for all the variants studied (according to the type of superplasticizer and the addition used).

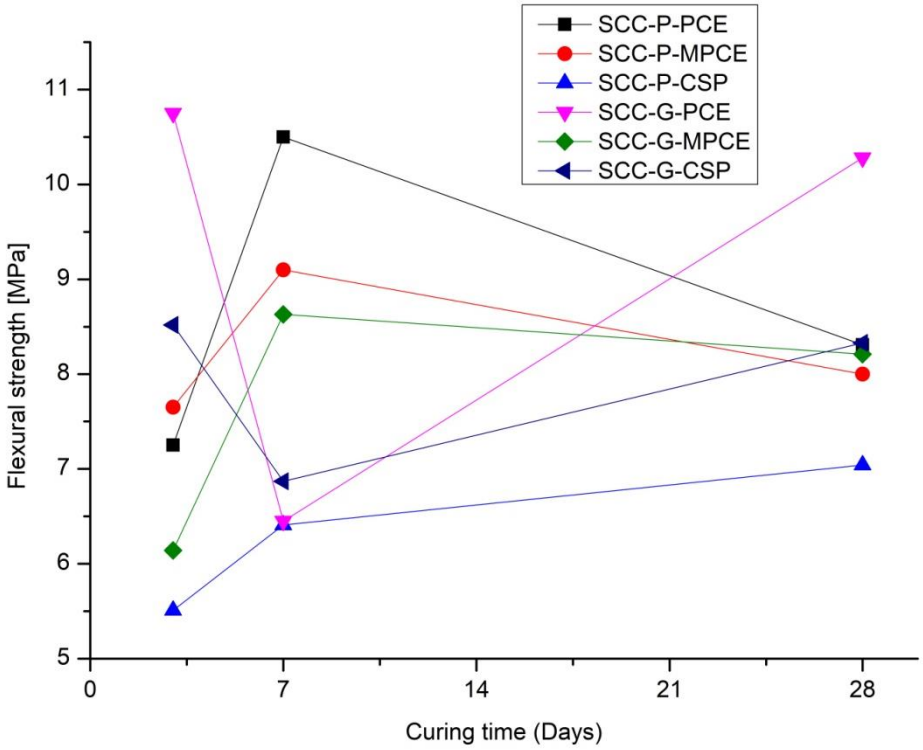


Figure 4: Flexural strength results at different curing ages

3.4.3 Splitting Tensile Strength

The results of the tensile splitting test are shown in Figure 5.

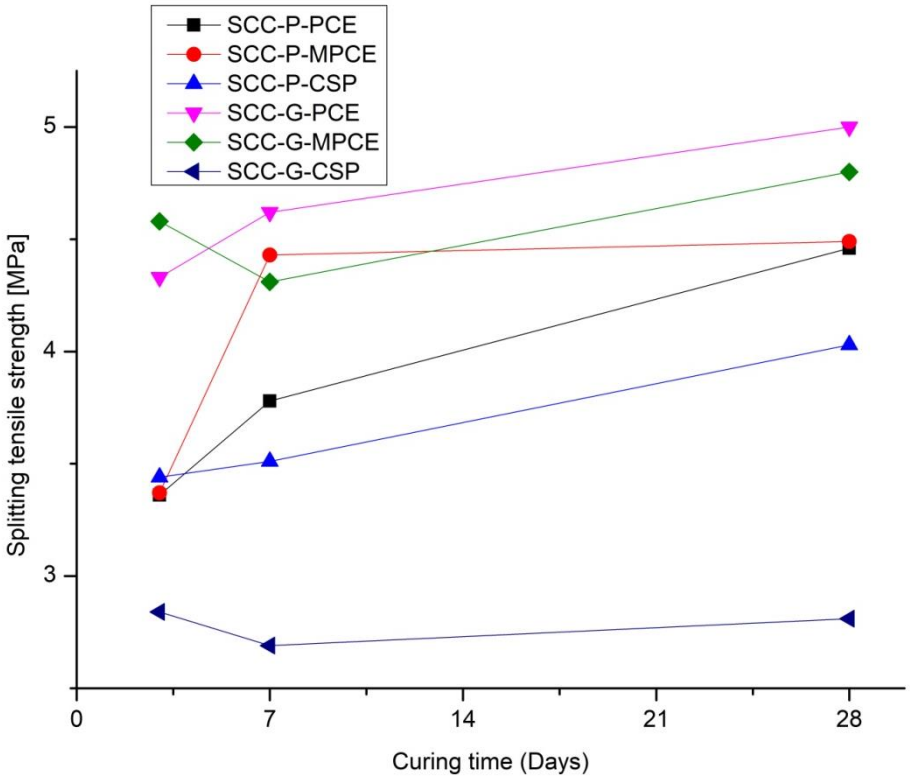


Figure 5: Splitting tensile strength results at different curing times

From the results, it can be deduced that concretes containing ground blast-furnace slag give better resistance to splitting compared to natural pozzolana concretes, especially at 3 and 28 days of curing. On the other hand, low splitting resistance was recorded at all curing ages (3, 7, and 28 days) for the samples where slag addition and CSP admixture are combined.
For concretes blended with natural pozzolana, the best splitting tensile strength is given by concrete containing MPCE admixture at all curing ages; however, for concretes containing GGBS, the use of PCE admixture gave the best performance except after 3 days of curing.

3.5 Relationship between Compressive and Flexural Strength

To highlight the relationship between the compressive and flexural strength of SCCs, the experimental values of the strengths obtained from the different samples at 28 days of curing were exploited. Trend curves were plotted and presented in Figure 6. A regression analysis is performed, and mathematical models (linear and polynomial) have been established.

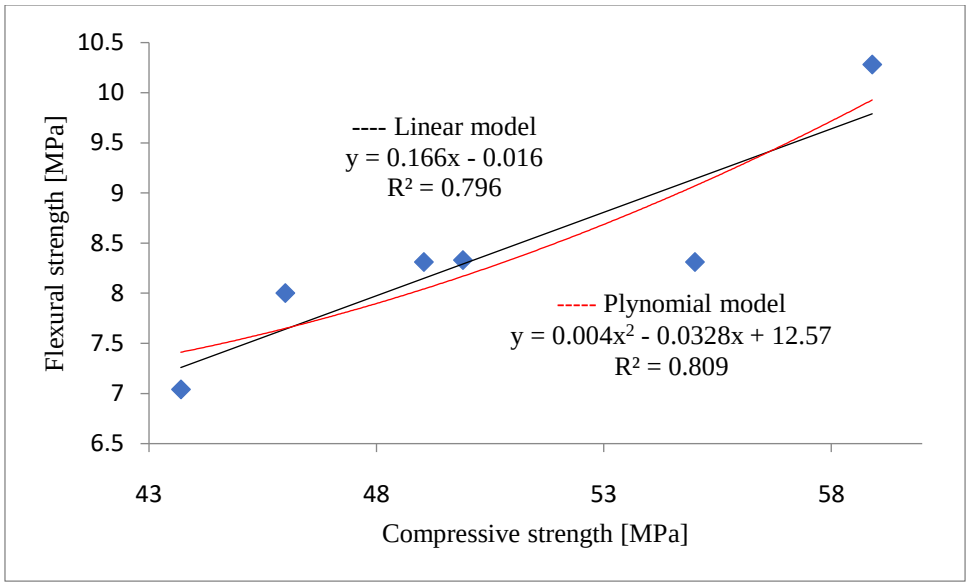


Figure 6: Relationship between compressive and flexural strength at 28 days of curing

The regression analyses, shown in Figure 6, highlight a positive correlation between the two mechanical properties. Indeed, the two properties evolve in the same direction, in other words, the compressive strength increases with the increase in flexural strength.
In order to link these two mechanical properties by equations, two mathematical models were designed according to the trend curves. The results of the calculation of their coefficients of determination R^2 show that the polynomial model is slightly more accurate than the linear model. However, the two models remain relatively imprecise given the value of R^2 which is quite far from 1.

4 Conclusions

The main conclusions from the present experimental study can be summarized as follows:

- Ground granulated blast furnace slag and natural pozzolana can be used as mineral additions in the production of self-compacting concrete (SCC). Their incorporation at 10% by weight relative to the mass of cement has made it possible to achieve properties that meet the standards required for SCCs, whether in the fresh or hardened state.
- Poly Carboxylate Ether (PCE) proved to be the most effective superplasticizer when compared to MPCE and CSP. Moreover, its use with low dosage enabled a better rheological behavior of SCC mixtures.
- The TOC analysis results revealed that Poly Carboxylate Ether (PCE) showed the highest adsorption on the cement particles, while the adsorption of CSP was the lowest. This can be explained by the structure of their main and side chains.
- The saturation dosage of the admixtures varies according to the mineral addition present in the self-compacting concrete mixture, in fact, the lowest saturation dosages

have been recorded in the SCCs containing granulated blast furnace slag as an additive, and this compared to natural pozzolana.

- Self-compacting concretes based on granulated blast furnace slag give better rheological and mechanical properties compared to concretes containing natural pozzolana as mineral addition.
- Compared to PCE and CSP admixtures, Modified Poly Carboxylate Ether (MPCE) makes it possible to obtain high mechanical properties of SCCs at curing ages of 3 and 7 days (short term).
- Our future research direction is to study the effect of these superplasticizers on the different properties of self-compacting concretes in the long term (durability).

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