



Study of the influence of water reducing and setting retarder admixtures of polycarboxylate “superplasticizers” on physical and mechanical properties of mortar and concrete

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- ✓ Compressive strength.

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Abstract

The aim of this article is to improve the physical properties and mechanical performance of mortars and concretes in the fresh and hardened state while reducing the amount of mixing water used and extending the setting time and the hardening of concrete. In this work, we have incorporated an admixture of advanced superplasticizers for prolonged slump retention “SP402” into the formulation matrix of concrete at various percentages ranging from 0.5% to 4% by weight of cement with a step of 0.5%, whilst partially substituting the mixing water by the last one in order to reduce the amount of water used in the mixing. The obtained results by different prospected formulations show that the addition of SP402 in the formulations matrix significantly reduces the amount of water used in the mixing and the water/cement ratio (W/C). We noticed that the initial and final times increase when the percentage of SP402 also increases. Similarly, the porosity and capillary absorption decreased as a function of the increases in the mass fraction of SP402. However, the compressive strengths at a young age (2 days), median ages (7 days) and long-term (28 days) were improved. We have succeeded in improving the mechanical performance of mortars and/or concrete, including the porosity /the capillary absorption / the compressive strength and reducing the amount of mixing water used while modifying the setting time according to use requirements.

1. Introduction

The introduction admixture of the superplasticizer in cement mixtures has every interest to be done as late as possible when kneading the concrete, to minimize the amount of mixing water, to improve the properties theology of the cement paste and thereafter to increase its mechanical strength [1,2]. The fluidity and the flow of cement paste have controlled by workability in the fresh state as well as the compressive strength and the durability of mortars and/or concretes [3-6]. These properties depend strongly on the dispersion of the cement paste composed of water, cement, mineral additives and admixtures [7-12]. These admixtures optimize these properties by creating an electrostatic repulsion and/or stress, which causes a deflocculation [13-17]. More specifically, the state of dispersion influences the workability of concrete by trapping water in the agglomerate which does not contribute to the flow properties [18-21]. The admixtures of superplasticizers advanced for prolonged slump retention (SP402) are polymers in liquid form, made especially for the cement industry and concrete. They are the basis of salts of sodium or calcium of poly-naphthalene sulfonic, of sulfonic melamine poly sodium salt, acrylate-ester (polyacrylate) copolymer, or lignosulphonate of high purity [22-28]. The hot weather causes rapid evaporation of water from the surface of the fresh cement paste. The retarders are used to offset the accelerating effects of hot weather on the setting times or retarded the all of the settings of concrete when unusual conditions are different [29]. The retarder mixture is a mixture which retards the setting of the concrete [30,31] this phenomenon can cause one or more of the mechanisms among which; the admixture molecules are adsorbed on the surface of the cement particles, preventing further reactions between cement and water. In other words, when water is added to the cement, the calcium ions and the hydroxyl ions are released to

the surface of the cement particles. However, the hydration products C_2S and C_3S begin to crystallize when the critical value of the concentration of these ions is reached. While the admixture reacts with one or more components of the cement to form a precipitate on the cement particles, which gives a "coating" low permeability on the cement particles. Subsequently, the admixture forms complexes with Ca^{2+} ions which are released by hydration \rightarrow Reduction of available Ca^{2+} . In other words, it causes the formation of complexes with the calcium ions in solution released by the cement during the first minutes of hydration. This formation increases the solubility of the cement, that is to say, increases the concentration of Ca^{2+} , OH^- , Si^{4+} , Al^{3+} , and Fe^{3+} in the aqueous phase of the cement paste which will occur during hydration in the presence of the retarder [32]. Finally, the admixture poisons the nucleation sites of $Ca(OH)_2$ and/or CSH and inhibits the formation of bonds between the hydrated products. In other words, the calcium ions and the hydroxyl ions accumulate in solution and will not precipitate to form calcium hydroxide [32-33].

In this experimental work, we studied the influence of the incorporation of admixtures of advanced superplasticizer for prolonged slump retention 'SP402' in the matrix of formulation of concrete high performance at different percentages ranging from 0.5 to 4% by weight of cement with a step of 0.5%, while partially substituting the mixing water by the SP402. Then, we studied the influence of the addition of SP402 on physical properties of fresh cement paste on one hand and on the other hand on the mechanical performance of concrete and/or mortar. The obtained results from the different developed formulations showed that the addition of the 0.5 and 3.5 of SP402% by weight of cement in our formulation, improving both the physical properties of cement paste and the mechanical performance.

2. Experimental

a. Materials

For evaluating the influence of the admixtures of advanced superplasticizer for prolonged slump retention "SP402", on physical properties of fresh cement paste and mechanical performance of mortars and /or concretes in the hardened state. We have preceded our work by the characterizing of materials used, to understand the phenomena which occur at the moment of mixing and hardening of concrete.

i. Cement

The type of cement used in this work is (CMI / 42.5) from the plant of Amran in Yemen. The chemical and mineralogical compositions of clinker, gypsum, and cement determined by XRF are presented in the Tables (1) and (2), with the absolute density is 3.14 g.cm^{-3} and the specific surface Blaine is $3240.00 \text{ cm}^2.\text{g}^{-1}$.

Table 1: Elementary chemical compositions of clinker, gypsum, and cement in weight of atomic

Chemical name	Chemical formula	Cement nomenclature	Clinker	Gypsum	Cement
Lime	CaO	C	62.76	33.40	61.29
Silica	SiO ₂	S	21.00	0.70	19.99
Alumina	Al ₂ O ₃	A	5.84	0.36	5.57
Ferrite	Fe ₂ O ₃	F	3.00	0.09	2.85
Magnesia	MgO	M	1.96	0.63	1.89
Sulfur trioxide	SO ₃	S	0.90	47.20	3.22
Potassium oxide	K ₂ O	K	1.21	0.03	1.15
Sodium oxide	Na ₂ O	N	0.20	0.10	0.20
Chloride ion	Cl ⁻	Cl	0.02	0.01	0.02

Table 2: Mineralogical composition of clinker

Chemical name	Mineral name	Chemical formula	Cement nomenclature	Content
Tricalcium silicate	Alite	Ca ₃ SiO ₅	C ₃ S	47.70
Dicalcium silicate	Balite	Ca ₂ SiO ₄	C ₂ S	25.10
Aluminate tricalcium	Aluminate	Ca ₃ Al ₂ O ₆	C ₃ A	10.40
Tetracalcium Aluminoferrite	Ferrite	Ca ₄ Al ₂ Fe ₂ O ₁₀	C ₄ AF	9.10

ii. Superplasticizer for prolonged slump retention (SP402)

The superplasticizer "SP402" are polymers in liquid form, prepared especially for the cement industry and concrete. They are based on sodium or sulfonated naphthalene-formaldehyde "SNF" (Figure.1 "a"), sulfonated melamine formaldehyde "SMF" (Figure. 1 "b"), high purity lignosulfonate (Figure. 1 "c") or acrylate ester copolymer (polyacrylate) (figure. 1 "d") [EN 934 - 2].The SP402 used in the formulation of mortars and/or concrete of high performance, is delivered by the company CONMIX Ltd in Sharjah United Arab Emirates. This admixture is the liquid nature, brown color, 1.23 g.cm^{-3} of density, 0.5-1.0 (%) of area training, and Nile of chloride content. They are incorporated during the mixing of the mortar and/ or concrete in equal a dose raining

from 0.5% to 4% by weight of cement with a step of 0.5%, to improve the physical and the mechanical properties of the fresh or hardened state.

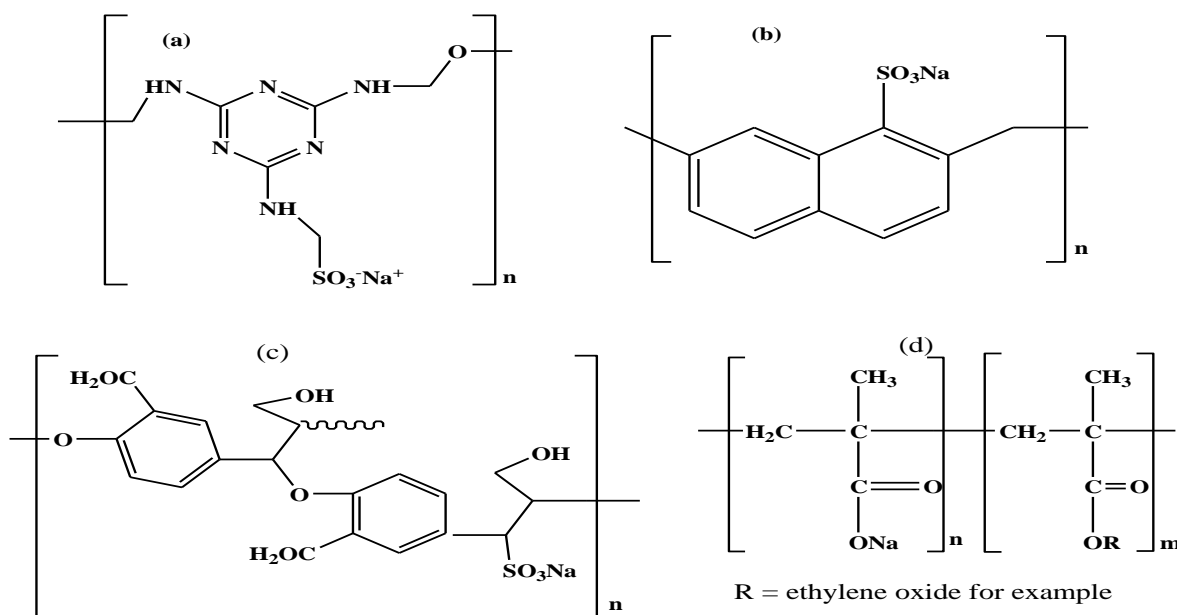


Figure 1: Families of superplasticizers; “a”: Sulfonated melamine formaldehyde (SMF), “b”: Sulphonated naphthalene-formaldehyde (SNF), “c”: Lignosulphonate of high purity and “d”: acrylate ester copolymer (polyacrylates)

iii. Water

The water used to mix the mortar and/or concrete is tap water. The main characteristics of these waters are summarized in Table (3).

Table 3: Main features of the mixing water

Components	pH	Turbidity	CO ₃ ²⁻	HCO ₃ ⁻	Ca ⁺²	Mg ⁺²	Conductivity
Values	7.00	450.00 [mg.l ⁻¹]	216.00 [mg.l ⁻¹]	0.00 [mg.l ⁻¹]	56.40 [mg.l ⁻¹]	52.40 [mg.l ⁻¹]	692.00 [μS/cm]

iv. Sand

To make our mortar, we used standard sand according to the norm EN 196-1, delivered by the new French company of Littoral. Its particle size analysis is illustrated in Figure (2).



Figure 2: Granulometric curve of sand

The particle size analysis presented in Figure (2) shows that used sand grains are distributed in a systematic way according to the specifications of the standard EN 196-1.

b. *Methods of characterizations of materials*

i. *Method of preparation of fresh cement paste*

The experimental protocols used to determine the physical properties of fresh cement paste, namely the standard consistency (W/C report) also the initial and final time are given in the standard EN 196-3. This is to make a paste of cement with 500 g of cement into the bowl of the mixer. The amount of water needed is chosen to take into account on the different percentages of superplasticizer SP402 used in our formulations that show in Table (4). We immediately put the mixer with a slow speed for 90 Sec. Then we stopped the mixer for 15 Sec to bring the paste which is located beyond the mixing zone. Subsequently, we restarted the mixer at slow speed for 90 Sec. After we quickly introduced the paste into the frustoconical mold placed on a glass plate, without excessive compaction or vibration. Then the assembly is placed on the plate of the Vicat apparatus. Afterward, we measured the separation distance between the end of the probe and the base plate. This distance (d) is the consistency of the paste studied (W/C report). And finally, the paste of cement with a standardized consistency will be placed on the plate of the Vicat automatic device to measure the initial and final time.

Table 4: Formulation matrix of fresh cement paste with the SP402

N° of test	Mass of cement (g)	Water (ml)	W/C	% SP402
1	500	130	0.26	0.00
2	500	120	0.24	0.50
3	500	115	0.23	1.00
4	500	110	0.22	1.50
5	500	105	0.21	2.00
6	500	100	0.2	2.50
7	500	95	0.19	3.00
8	500	90	0.18	3.50
9	500	84	0.168	4.00

ii. *Methods of preparation of mortar and/or concrete in the hardened State*

To achieve the objective of our study, we prepared a mortar of reference without and with superplasticizers SP402 which the compositions are inspired by the normal mortar defined by the standard EN 196-1, with a quantity of water and adjusted and a paste with a consistency standard as shown in Table (5). The procedures followed for the preparation of our mortars begin with mixing, the procedures followed for the preparation of our mortars begin with the mixer, and then we filled a mold (4 x 4 x 16) cm³.

The Tightening of the mortar in this mold is obtained by introducing the mortar twice and by applying to the mold 60 shocks each time using the shock device. After the mold is leveled, covered with a plate of glass and stored in the wet room. After 20 h or 24 h from the start of the mixing, the specimens are removed from the mold and stored in water at 20 ° C ± 1 ° C until the time of the test of rupture. The compressive strengths were measured at a young age (2 days) in median age (7 days) and long-term (28 days) using a bending test machine (Figure 3) to break the specimen into two halves and each party is responsible the subject of compressive using a hydraulic compressive testing machine (Figure 4). The value of the resistance is considered as the average of the crushing stress of three test pieces (6 halves).

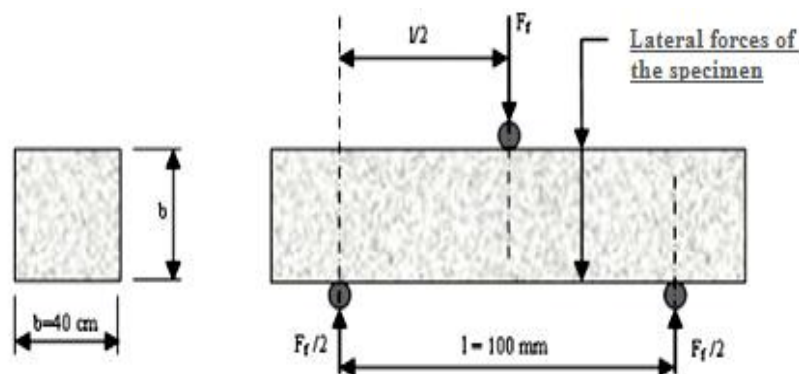


Figure 3: Device of bending load for the mortar specimens

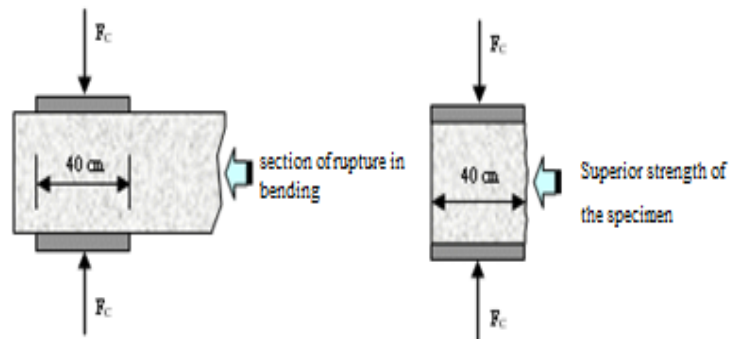


Figure 4: Device in compressive load for the mortar specimens

The compressive strength was calculated using the equation (1).

$$\text{Compressive strength} = \frac{\text{Load, in "N"}}{\text{Area, in "mm}^2\text{"}}; \text{MPa} \quad \text{Equation 1}$$

Table 5: Formulation matrix of mortar and/ or concrete with SP402 in hardened state

N° of test	Mass of cement (g)	Water (ml)	% SP402	Sand (g)	W/C
1	450	225	0.00	1350	0.50
2	450	211.5	0.50	1350	0.47
3	450	200	1.00	1350	0.44
4	450	188	1.50	1350	0.42
5	450	174	2.00	1350	0.39
6	450	160	2.50	1350	0.36
7	450	155	3.00	1350	0.34
8	450	144	3.50	1350	0.32
9	450	141	4.00	1350	0.31

iii. Porosity

The porosity is an essential feature of the mortar and/or concrete at hardened state. It is a part of the factors that determine the durability of concrete. It is obviously calculated using the equation (2).

$$P = \frac{V_P}{V_T} \quad \text{Equation 2}$$

With:

V_P : The pore volume of the specimens;

V_T : The total volume of the specimens, that is to say, the sum of the volume of solid and the volume of the pores.

iv. Capillary absorption

The capillary absorption (CA) of our formulations at different percentages of superplasticizer SP402 was calculated by using the equation (3). It is expressed in $\text{g} \cdot \text{mm}^{-2}$.

$$CA = \frac{M_f - M_i}{S} \quad \text{Equation 3}$$

With:

CA: The capillary absorption ($\text{g} \cdot \text{mm}^{-2}$);

M_f : The mass of the specimen after conservation for 2 days, 7 days and 28 days, in grams;

M_i : The mass of the specimen before conservation under the water in grams;

S: The area of the specimens in (mm).

v. Gain of compressive strength at 28 days

We calculated the gain of compressive strength at 28 days using equation (4), it is expressed in percentage.

$$G = \frac{R_{mcx} - R_{mct}}{R_{mct}} \times 100 \quad \text{Equation 4}$$

Where:

R_{mct} : Control of compressive strength of mortar at 28 days;

R_{mcx} : Compressive strength of mortars with superplasticizer and $X = 0.5\%, 1\% \dots\dots 4\%$.

3. Results and Discussion

3.1. Influence of the quantity of SP402 on the physical properties of fresh cement paste

3.1.1. Influence on the amount of mixing water

The Figures (5 and 6) show the ratio W/C and always the quantity of water reduced as a function of the dosage of SP402.

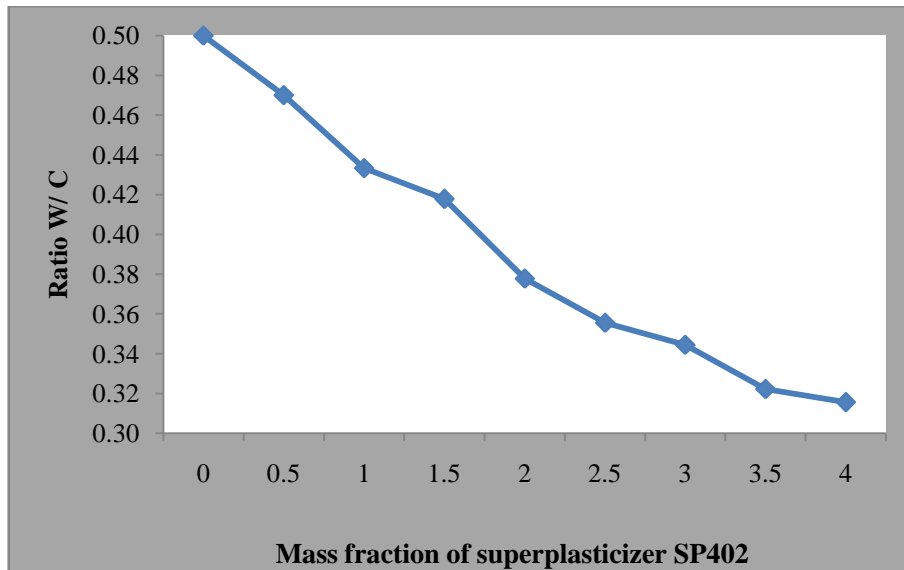


Figure 5: Ratio of W/C as in function of mass % of SP402

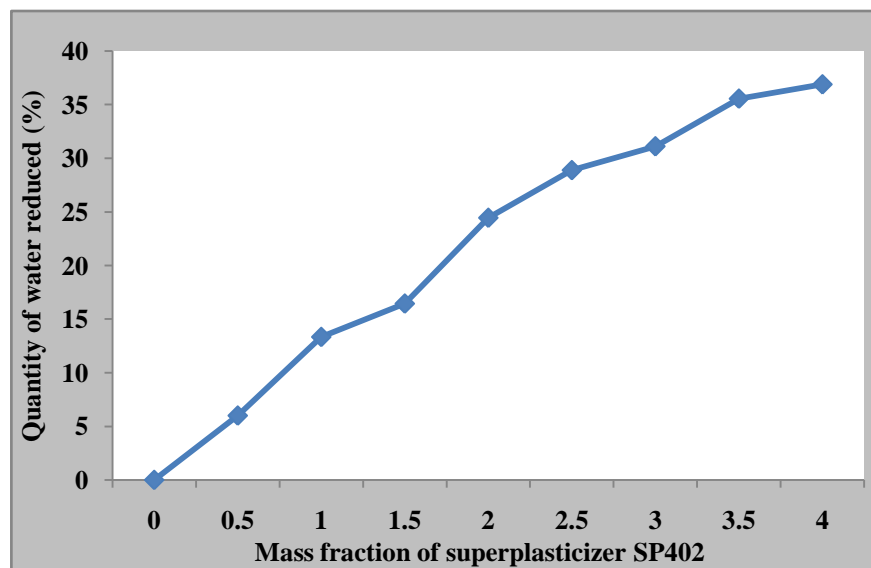


Figure 6: Quantity of water reduced as in function of mass % of SP402

After the Figures (5 and 6), we have noticed that the incorporation superplasticizer for prolonged slump retention (SP402) into the mortar/concrete formulation matrix considerably reduces the amount of mixing water and therefore the W C ratio. This reduction in water quantity is generally due to the action of the dispersion exerted by the adsorption of the molecules of SP402 on the solid surface which modifies the zeta potential of the particles on one hand and which promotes their dispersion due to of a phenomenon of steric hindrance on the other hand. Beyond 3.5% (saturation point) the SP402 has a negative effect, which can create a phenomenon of segregation. Our results corresponded with the other authors, as well [2, 5, 19, 22, 35-37].

3.1.2. Influence on setting time

The Figure (7) reveals the evolution of the time as a function of the different percentages of SP402. At the Figure (7), we have seen that the initial and final times increase as a result of the increase in the addition of SP402 in the concrete matrix formulation. This increase is logically explained by several reasons, among which we found that when water is added to the cement, the calcium ions and the hydroxyl ions are released to the surface of the cement particles.

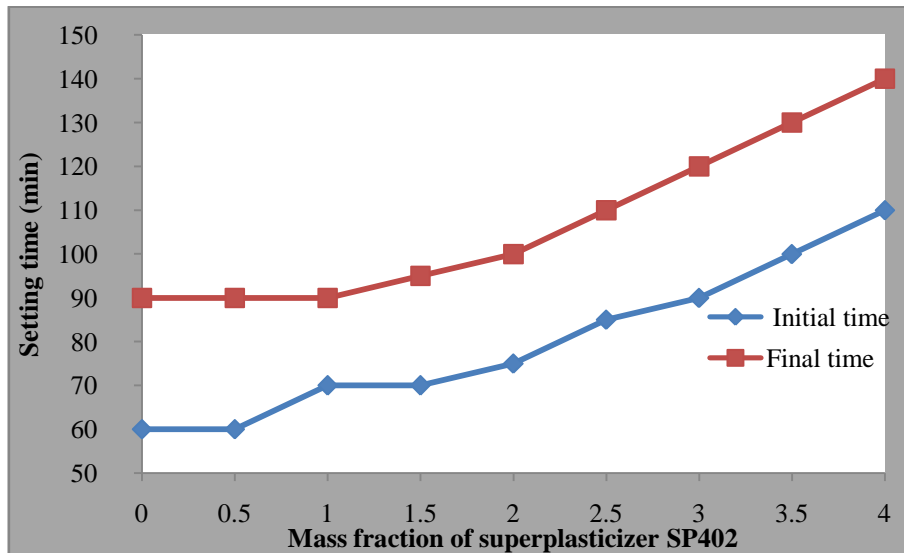


Figure 7: Setting time as in function of mass % of SP402

However, the hydration products C_2S and C_3S begin to crystallize when the critical value of the concentration of these ions is reached. Thereafter, the adjuvant reacts with one or more components of the cement to form a precipitate on the cement particles, which confers a low permeability "coating" on the cement particles. Then, the adjuvant forms complexes with the Ca^{2+} ions which are released by hydration \rightarrow Reduction of available Ca^{2+} . Finally, the adjuvant poisons the nucleation sites of $Ca(OH)_2$ and/or CSH and inhibits the formation of bonds between the hydrated products. In this way, hydration is delayed. Our results resembled with the other authors, as well [17, 25, and 37].

3.2. Influence of the quantity of superplasticizer SP402 on mechanical performance

3.2.1. Influence on porosity

The Figure (8) shows the porosity of mortar and/or concrete formulated by different percentages the addition of superplasticizer SP402 as a function of the mass fraction of SP402.

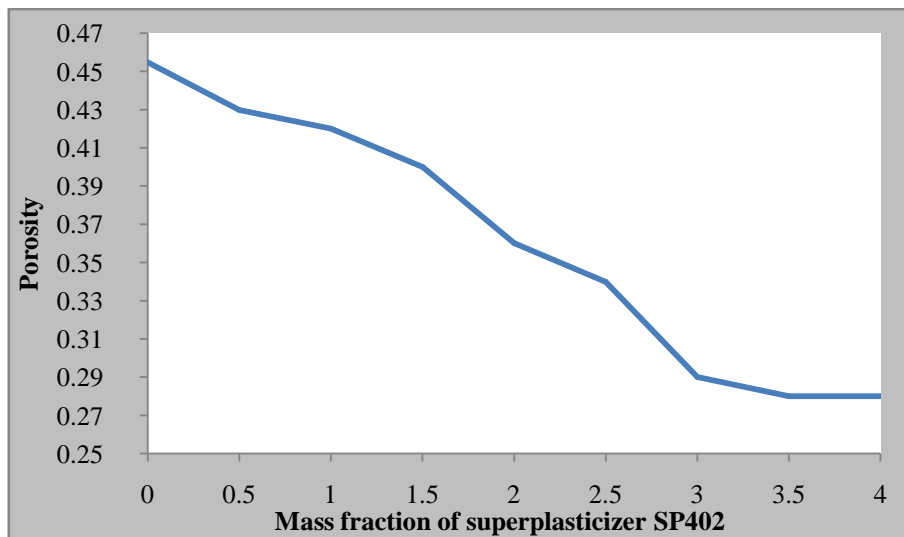


Figure 8: The porosity of mortar and/or concrete as a function of mass % of SP402

From the Figure (8), we found that the porosity of mortars and/or concrete formulated by different percentage the addition of superplasticizer high reducer of water and retarder of setting time (SP402) as a function of the mass fraction of this superplasticizer decreases. This decrease is due to the effect of the molecules of SP402 which disperses the cement grains from one another and which reduces the interstitial void between the particles of cement and those of the aggregates (sand), which will subsequently influence the mechanical of compressive strength. These results resembled with the other authors, as well [2, 5, 35, 36].

3.2.2. Influence on capillary absorption

The Figure (9) shows the capillary absorption of mortar and/or concrete formulated by different percentage the addition of superplasticizer (SP402) as a function of the mass fraction of SP402.

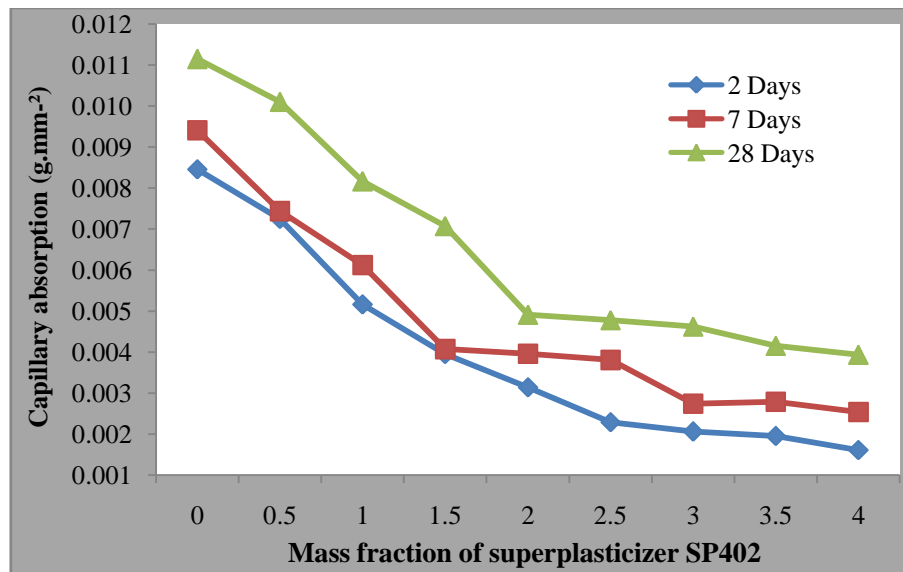


Figure 9: The capillary absorption of mortar and/or concrete as a function of the mass % of SP402

According to the Figure (9), we have observed that the capillary absorption of mortar and/or concrete formulated by different percentage of the superplasticizer (SP402) decreases as a function of the mass fraction of this superplasticizer. This decrease is explained by the fact that the molecule of the superplasticizer disperses the grains of cement from each other on one hand and on the other hand by the effect that the capillary absorption of the mortars is influenced by the structure of porous and the rate of the superplasticizers. The last one has contributed to the reduction of the capillary absorption by the formation of a polymer film and the reduction of the capillary pressure. These effects are similar to the other authors, as well [2, 5, 35].

3.2.3. Compressive strength

The Figure (10) presents the compressive strength of mortar and /or concrete as a function of the mass fraction of SP402.

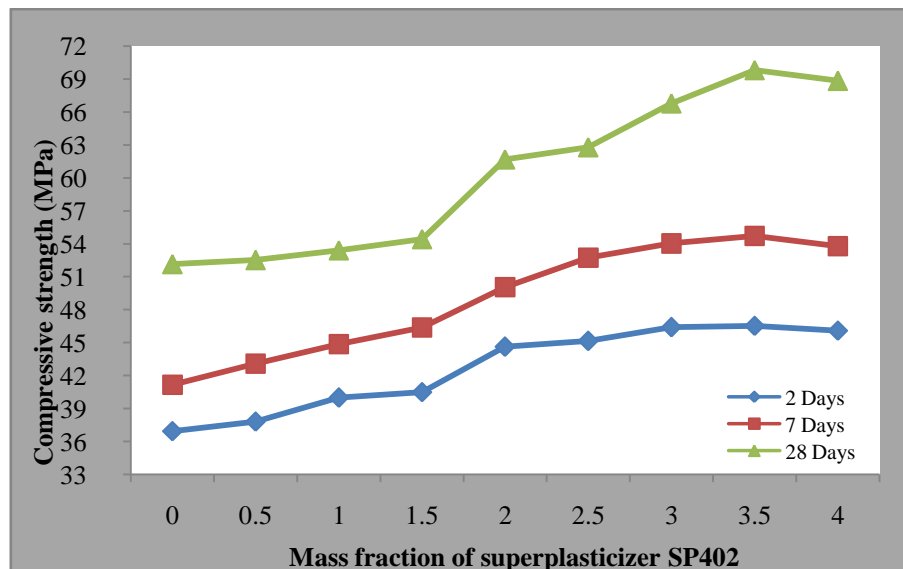


Figure 10: The compressive strength of mortar and /or concrete as in function of mass % of SP402

The experimental results Illustrated in the Figure (10) have shown the compressive strength at the young age (2 days), medium term (7 days) and long-term (28 days) increases as a function of the increase of the mass fraction SP402. This increase can be explained by the effect that the addition of SP402 in the mortar and/or concrete formulation matrix disperses the grains of the cement from one another and fills the voids between the cement particles and aggregate. In other words, it minimizes the porosity of mortar and/or concrete. This porosity is

influenced by the increase in mechanical of compressive strength. Beyond these percentages, decreases the compressive strength considerably. This shows that the 3.5% of the SP402 is the point of saturation. These properties are similar to the other authors, as well [2, 5, 35-37].

3.1. Gain of the compressive strength

The Figure (11) presents the gain in compressive strength of mortar and /or concrete as in function of the mass fraction of SP402.

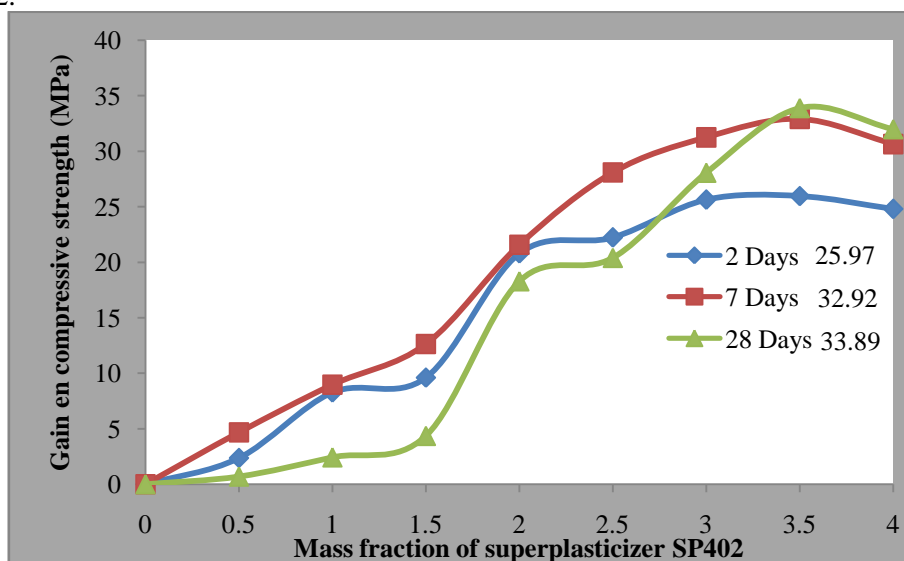


Figure 11: The gain in compressive strength of mortar and /or concrete as in function of mass % of SP402

According to the Figure (11), we have found that the gain in mechanical of compressive strength increases with increasing SP402 in the formulation matrix. For example, the incorporation of 3.5% of SP402 by weight of cement increases the mechanical resistance of compression by 25.97 / 32.92 and 33.89 at a young age (2 days) / medium term (7 days) and long-term 28 (days) respectively.

4. Conclusions

During this work, we have incorporated a new admixture of nature polymerizes, formed by the superplasticizer (SP402) in the matrix of a formulation of mortar and/or concrete. This last one was added at different percentages ranging from 0.5 to 4% by weight of cement with a step of 0.5%, while partially substituting the mixing water by SP402 in view to reduce the amount of mixing water used and to improve the physical properties and mechanical performance.

The obtained results by different formulations developed to show that the dosage between 0.5% and 3.5% of superplasticizer (SP402) by weight of cement in our formulations reduced 36% of the amount of water compared to the control. Similarly, the ratio W/C was decreased. Afterward, we noticed that setting time is increasing, according to the percentage of SP402 in the concrete matrix. The porosity and the capillary absorption have been decreased as a function of the increase in the mass fraction of SP402 in the formulation matrix, which will improve the mechanical of compressive strength at the young age (2 Days), median age (7 days) and long-term (28 d days) will be by of 25.97% / 32.92% and 33.89 respectively.

We have succeeded in improving the mechanical of compressive strength of mortars and/or concrete and reducing the amount of mixing water used by introducing the superplasticizer (SP402) into formulation the matrix at various percentages ranging from 0.5 to 4% by weight of cement with a pitch of 0.5%. We found better physical and mechanical properties between 0.5% and 3.5% SP402 by weight of cement above this percentage, the properties considerably decrease this shows that the percentage of 3.5% of SP402 is the saturation point.

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