academicJournals

Vol. 9(24), pp. 1003-1118, 30 December, 2014
DOI: 10.5897/SRE2014.6001
Article Number: 9BCC81A49374
ISSN 1992-2248 © 2014

http://www.academicjournals.org/SRE

Scientific Research and Essays

Full Length Research Paper

Influence of nanosilica and a polycarboxylate superplasticizer on the rheological and electrokinetical properties of cement pastes

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Received 7 June, 2014; Accepted 5 December, 2014

The effect of individual and combined addition of both nanosilica (NS) and polycarboxylate ether plasticizer (PCE) admixtures on cements pastes was studied. The sole incorporation of NS increased the water demand, as proved by the mini-spread flow test. An interaction between NS and hydrated cement particles was observed in fresh mixtures by means of particle size distribution studies, zeta potential measurements and optical microscopy, giving rise to agglomerates. On the other hand, the addition of PCE to a cement paste increased the flowability and accelerated the setting process. PCE was shown to act in cement media as a deflocculating agent, reducing the particle size of the agglomerates through a steric hindrance mechanism. Mechanical strengths were improved in the presence of either NS or PCE, the optimum being attained in the combined presence of both admixtures that involved relevant microstructural modifications, as proved by pore size distributions and SEM observations. The results indicate also the effectiveness of NS and polycarboxylate superplasticizer in producing high packing density and in accelerating the pozzolanic activity to produce more C-S-H gel by consuming calcium hydroxide $Ca(OH)_2$ in order to improve the mechanical properties of cement pastes.

Key words: Cement pastes, polycarboxylate plasticizer, nanosilica, optical microscopy, rheological and electrokinetical properties.

INTRODUCTION

During the mixing, the cement particles are dispersed and suspended in water. However in the presence of a superplasticizer, the cementitious paste becomes more stable and the shortage of water which affects the maniability of cementitious pastes in time will be resolved in the presence of a superplasticizer. In addition, many research studies have shown that several factors affecting the rheological stability of the cement pastes namely; the concentration of solids, the cement characteristics and the nature and dosage of the superplasticizer used. The essential purpose of the use of a superplasticizer is to disperse the cement grains.

Due to the dispersing effect of a superplasticizer, the fluidity of the paste is increased, and shear stress and

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Table 1. Chemical and mineralogical composition of different cements.

Composition	Cement		
	C_1	C_2	C_3
SiO_2	21.65	18.81	22.68
Al_2O_2	03.38	04.91	06.05
Fe_2O_3	04.94	02.95	03.81
CaO	64.39	64.16	60.49
MgO	01.29	01.06	01.26
K_2O	00.54	00.43	00.63
Na_2O	00.14	00.31	00.49
SO_3	01.66	01.34	02.79
CaO_{Free}	00.85	00.69	01.50
Cl^-	0.0014	0.0024	0.0075
C_3S	57	57	56
C_2S	19	18	19
C_3A	02	12	10
C_4AF	15	07	09
Specific Surface (SSB) (cm^2/g)	3300	3726	3624
Setting times; (min)	189-260	149-210	189-260
% limestone addition in clinker	-	11	-
% pozzolan addition in clinker	-	-	08

plastic viscosity are reduced (Tattersall and Banfill, 1983; Justnes and Vikan, 2005; Vom Berg, 2009; Vikan, 2005; Neubauer et al., 1998; Collepardi, 2005; Uchikawa et al., 1997). The superplasticizer is adsorbed on the cement particles and changes the degree of flocculation in one of three ways: the increase in zeta potential and the repulsive forces between the cement particles (electrostatic repulsion of the double layer); improvement of the solid-liquid affinity and presence of a steric hindrance.

Several researchers have studied the effect of the nature and superplasticizer dosage on the rheological behavior of cementitious pastes. The superplasticizer action consists of two physical and chemical phenomena (Vom Berg, 2009; Vikan, 2005; Neubauer et al., 1998). The physical phenomenon includes non-specific effects of adsorption, electrostatic repulsion and steric repulsion (Neubauer et al., 1998; Uchikawa et al., 1997). Chemical phenomenon was assigned to the reactive nature of the cement particles. The preferential adsorption (selective) (Neubauer et al., 1998; Collepardi, 2005; Uchikawa et al., 1997; Kauppi et al., 2005; Flatt and Houst, 2001; Zhang et al, 2001) to chemisorption and chemical reactions to form new hydrated phases (Zhang et al, 2001; Griesser et al., 2005; Plank and Hirsch, 2007). The importance of steric forces dispersed cement suspension was highlighted in recent years, for various types of superplasticizers. This depends on the zeta potential of the suspension. Also, the sulfate concentration (sulfate content in cement, as a setting regulator) in the solution has a significant influence on the adsorption of superplasticizer during the first minutes of the process of hydration. Griesser has proved that the shear stress (yield stress) of the cement paste reaches a minimum with a certain amount of sodium sulphate (Na_2SO_4), which depends on the type of cement and superplasticizer (Griesser et al., 2005; Kheribet et al., 2012). This was explained by the competitive adsorption on the hydro-calcium aluminate C_3A between sulphate ions and molecules of the superplasticizer. This phenomenon is observed much more with the superplasticizers of the polynaphthalenes type (PNS) and polycarboxylates PCE (Houst et al., 2002; Yamada et al., 2001; Alonso et al., 2007; Palacioset al., 2009; Chandra and Björnström, 2002; Mäder et al., 2004; ACI, 2003; Johnson et al., 2000).

For this, the present study comes be given more information about the effect of superplasticizer type on the essentials properties of cement mortar (concrete) such as rheological and mechanical properties and the sustainability. The use of superplasticizer Aeternum1 (containing nanosilice + polycarboxylate) with cements (CEM II/A) will be the object of our study. We propose to integrate the adjuvant Aeternum1 of new generation to cements compounds CEMII / A, in order to assess the influence of superplasticizer on rheological and physico-mechanical properties of these cements in aggressive environments. The results obtained allow us to estimate substitution possibilities of CRS cement by these cements with superplasticizer of Aeternum1.

METHODOLOGY

Materials used

Cement: In order to assess the effectiveness of the superplasticizer Aturnum1, three cement types were chosen. For this, the cement C1 (Artificial Cement Portland (CPA)), cement C2 (Cement Resistant to the Sulfates (CRS)) and cement C3 (Cement with pozzolan addition of type CEMII/A 42.5) were used in this work. The chemical and mineralogical characteristics of cements used are given in Table 1. All used cements are according standard norm NF EN 197-1 (EN 197-1 and 197-2, 2000). The XRD pattern of the used cement confirms that these cements are according to standard norm (Figure 1).

Sand: The sand is standard sand for making mortar. It is also according to standard norm EN-v.

Superplasticizer (SP): The superplasticizer Aeternum-1 is used as a superplasticizer in cement pastes and mortar mixtures at different percentages by cement weight. Figure 2 gives the chemical and mineralogical composition presented in a XRD-pattern of the chosen superplasticizer. Also, the IR spectroscopy analysis is carried on this superplasticizer. The obtained results are given in the Figure 3.

The XRD showed that Aeternum-1 is a new generation superplasticizer, which is an hydration accelerator based on carbon

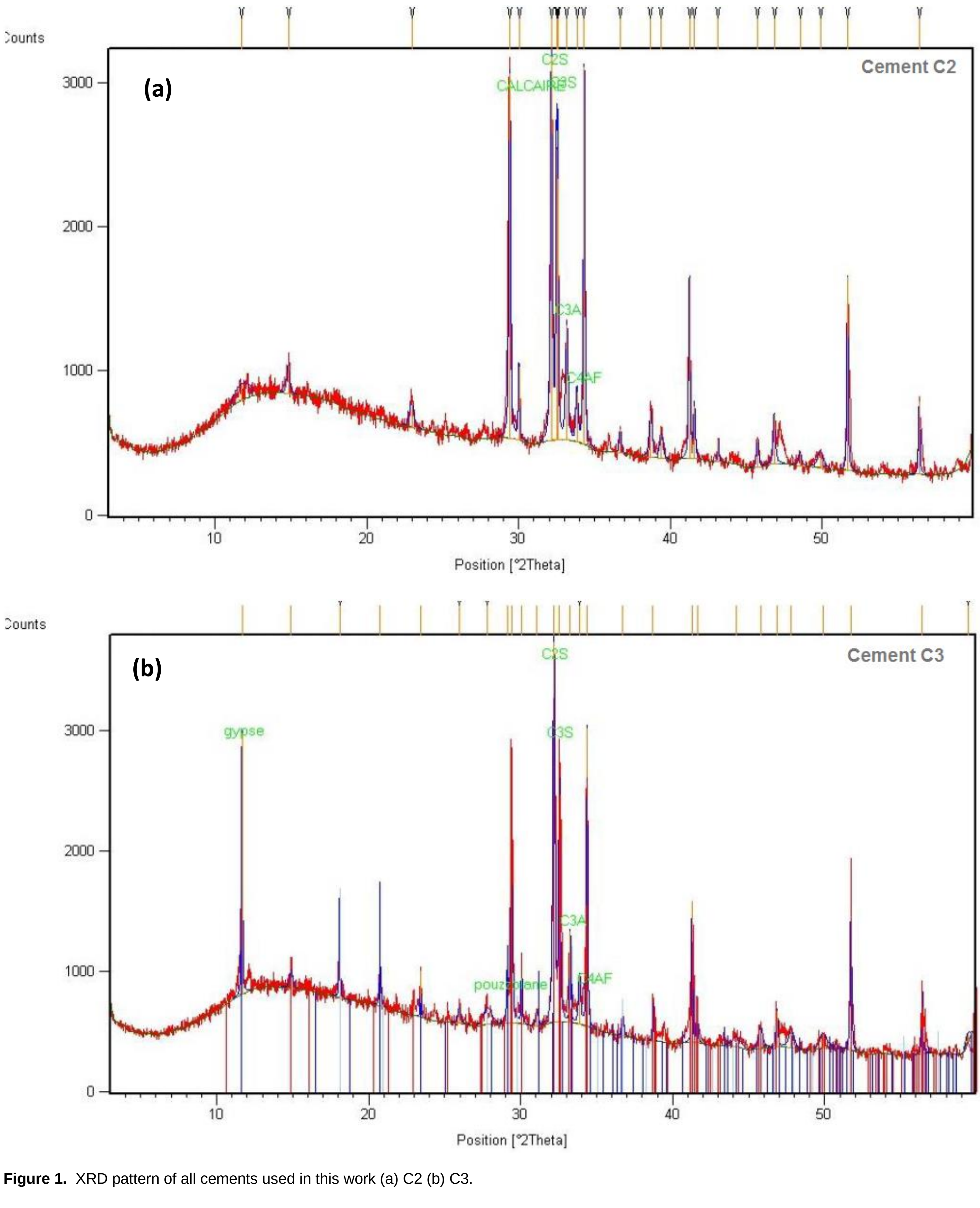

Figure 1. XRD pattern of all cements used in this work (a) C2 (b) C3.

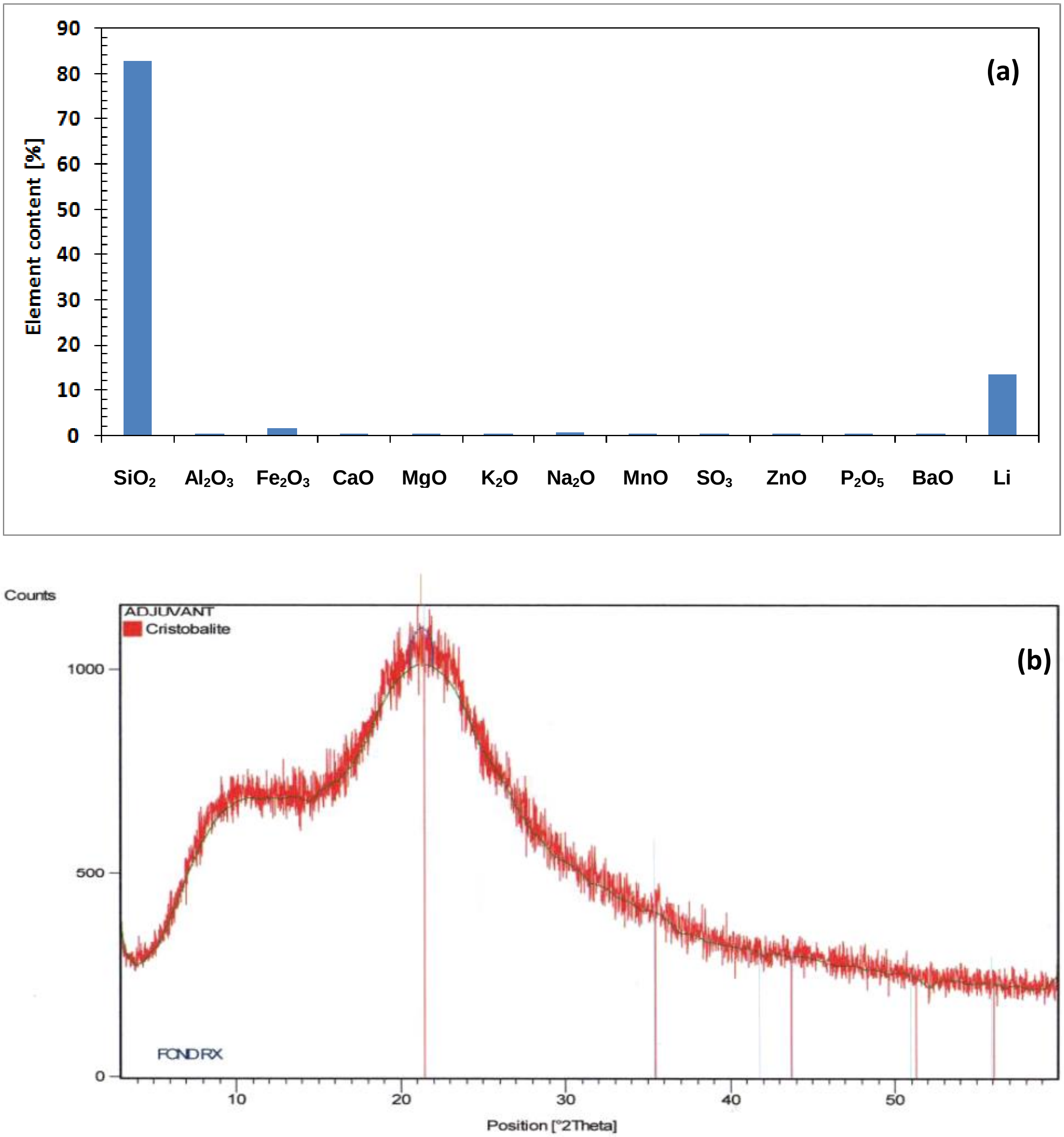

Figure 2. Chemical composition (a) and XRD analysis (b) of the adjuvant Aeternum-1.

and silicate in powder, infused in active nano-micro-silicates, it combines with the high pozzolanic activity of the latters, ensuring good rheological properties, fluidity (without segregation), waterproofness and compressive strength as well as good stability to the chemical aggressions.

The viewpoint of the physical characteristics consists of spherical particles having as size a few tenths of microns, and specific surface area of about 220000 cm^2/g. This feature allows a high dispersion and reaction on the cement particles, and a large capacity to capture and fix the calcium hydroxide ($Ca(OH)_2$) and transform it in a first time to an hydrated silicate and then to a stable and irreversible calcium hydrosilicate (C-S-H) tobermorite type.

The results provided by X-ray fluorescence allowed us to know the content of the various chemical constituents. Silica has an elevated content (82.67%), a high value of loss on ignition (13.7%), and a value of 6% carbon. The other constituents have very low values. The result of the XRD showed that the adjuvant is an amorphous body in high proportion and present a single peak of crystalline silica, as can be be seen on the crystallographic radiogram. This XRD graph is very similar to that of the silica fume.

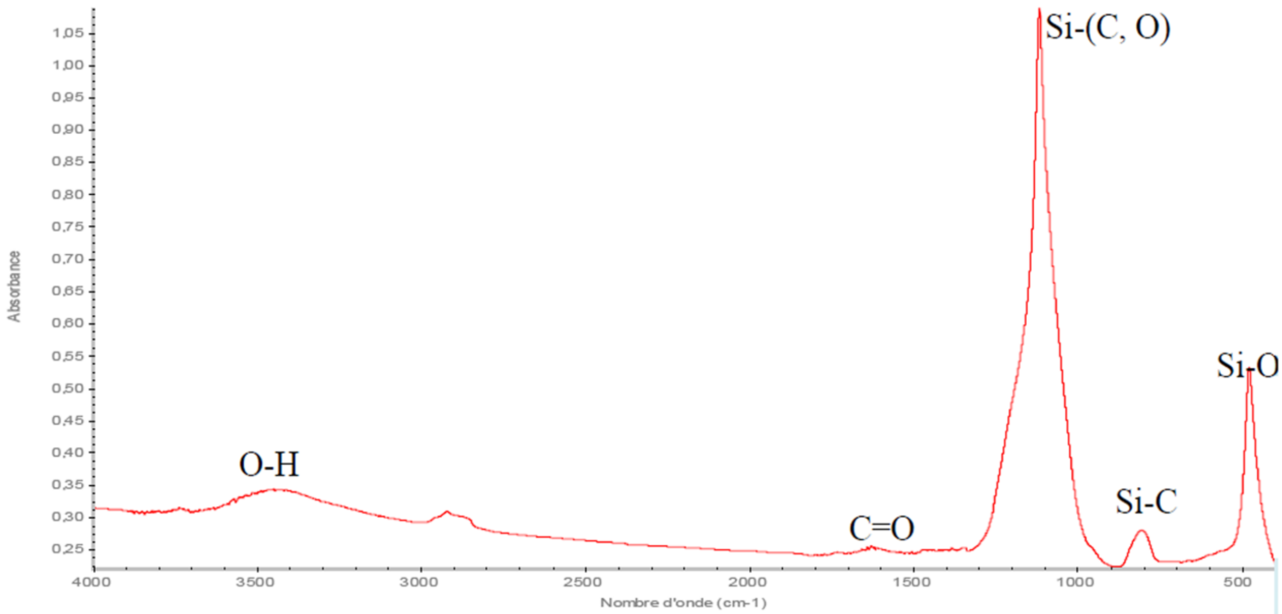

Figure 3. IR spectroscopy analysis of the superplasticizer used.

The Infrared spectrometry consists in irradiating the sample in the range 4000-400 cm^{-1} and detecting the frequencies absorbed by the latter (Figure 3). Only the vibrational normal modes inducing a displacement of the barycenter charges of the atomic group are active in infrared spectrometry. Figure 4 shows the IR spectrum of the adjuvant used, and illustrates some types of links, such as-Si-(O-C), Si-O and Si-C which are the most visible, indicating that this adjuvant is based on silicate and carbon; it is also organic. The C=O link is not significant as revealed in this adjuvant being without a carboxyl function.

Test methods

To perform this work, an experimental study was conducted according to the following work plan.

Rheological tests and fresh properties

Rheological tests: All rheological tests were carried using a Viscosimeter (VT550) equipped by coaxial cylinder geometry. Rheological measurements were conducted according to following protocol used by Kheribet et al. (Safi et al., 2011; Kheribet et al., 2012). It was proved by these authors that this protocol can be used for the cementitious pastes containing the superplasticizer (Safi et al., 2011). To compare the effect of superplasticizer used in this study, the cementitious pastes based on (C2 and C3), were prepared with a ratio W/C = 0.38 which is kept constant. The result obtained were compared to cement pastes with a ratio W/C = 0.5.

Slump test: All studied cement pastes were also tested for flowability immediately after mixing. The slump was measured at 20°C, using a mini-cone it was carried on fresh cement paste for each mixture.

Electrokinetic properties: The effect of different dosages of superplasticizer on the zeta potential of cementitious pastes was carried using a Zetameter (ZETASIZER 2000) of Malvern Instrument which has frequently used for determining the zeta potential of cement particles. For this, 1 cm^3 of the cementitious suspension is diluted in 30 cm^3 of distilled water, after which 5 ml of this suspension is injected into the analyzer (Kheribet et al., 2012).

Physical and mechanical properties

Setting time: The setting time was measured according to standard norm ASTM C191–13 (ASTM C191, 2013) same results as the time of setting of hydraulic cement paste measured by other methods, or the time of setting of mortar or concrete.

Casting and curing of mortars specimens: All the mixtures were mixed and prepared using a mortar mixer. All mortar compositions are prepared with the normal sand, according to the European standard EN 196-1 (EN 196-1, 2000). Before casting, slump-flow test is attempted as workability tests on fresh mortar for each mixture. Thereafter, three (3) specimens were cast in prismatic molds of (40×40×160 mm^3), for each concrete mixture. One day after casting, samples were stored in water under 21±1°C.

Mechanical tests: The mechanical tests (flexural strength and uniaxial compression) were conducted at 28 days of curing age according to European Standard EN 196-1 (EN 196-1, 2000). To study the effect of the superplasticizer on mortars, durability was based on the three cement types; the flexural and compressive strength was also measured on all mortar specimens that have been cured in different environments (trap water, sea water and chemical solution) under 21±1°C. The obtained results were compared to result obtained on the mortar samples stored in water.

RESULTS AND DISCUSSION

Rheological study of cement pastes with and without adjuvant

Rheological tests are performed for cement pastes based

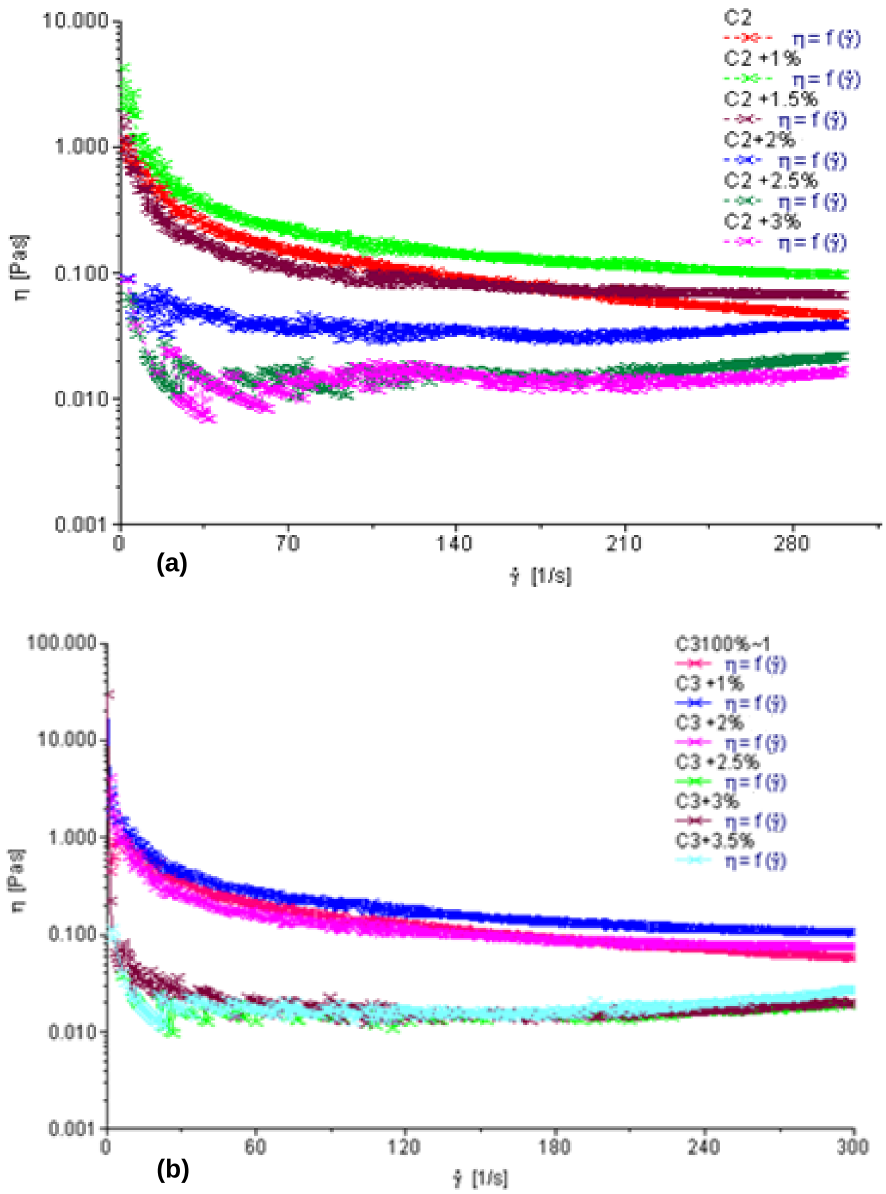

Figure 4. Apparent viscosity according to the shear rate of the cement pastes (C2 and C3) in dosages with adjuvant (0, 1, 1.5, 2, 2.5 and 3%, respectively) (a) C2 (b) C3.

on two types of cement C2 and C3 at different dosages of adjuvant to study the behavior of cement pastes and determining the saturation point for each type of cement. In this part we will also study the workability of cement grout, before ending with a study of the zeta potential measurements.

Determining saturation point of the different cements

To determine the saturation point we proceeded according to the protocol formulation of cement pastes, of fixing the amount of water and varying the amount of adjuvant in the mixture and the rheological measurements using the viscometer VT 550 with imposed speed. A composition of the cement paste was prepared for the witness cement grout with W/C ratio = 0.50 and the other pastas were prepared with a W/C ratio = 0.38. The results obtained are shown in Figure 4.

The saturation point is obtained from the curve of apparent viscosity according to shear rate. After testing the two types of cement C2 and C3 with different amounts of adjuvant (0, 1, 1.5, 2, 2.5, 3 and 3.5%) it was observed that:

The cement slurry with W/C=0.5 at the beginning have a higher viscosity value, but decreases with increasing shear rate, that is the same for both studied cements. For adjuvanted cement grout (cement + water +% adjuvant, with W/C= 0.38), we find that:

The adjuvant Aeternum-1 significantly affects the viscosity of the cement paste, and its content increases more, the viscosity of the paste decreases more. The apparent viscosity decreases with the shear rate of the cements grout with added limestone + 1% adjuvant, but with higher viscosity values relative to that of the grout without adjuvant. For 1.5% of adjuvant, the viscosity decreases initially and increases with increasing shear rate, such that it exceeds the values obtained by the grout without adjuvant.

This behavior was observed for grout with added pozzolan percentages for 1% and 2% successively. For a percentage equal to 2% superplasticizer, the cement slurry with added limestone has a constant viscosity with shear rate, which is a Newtonian flow. For the cement with added pozzolan, the Newtonian flow is obtained with 2.5% adjuvant, where the viscosity is constant as a function of shear rate. Beyond these percentages, it no longer affects the flow of grout. It can be seen that as the percentage of Aeternum-1 increases, the flow approaches the Newtonian flow, until it reaches the saturation point beyond which the adjuvant has no influence on the flow, which can be explained by the supersaturation of the cement grains. The saturation point varies from one cement to another – that of superplasticizer of C2 is 2% and the one of C3 is 2, 5%. The variation of the shear rate is a function of shear rate gradient of cements at different percentages of adjuvant:

According to the results, we observe that all studied pastas have a Binghamian behavior (1) following the model of Hershel-Bulckley described by the equation:

$$\tau = \tau_0 + k.\gamma^n \quad (1)$$

τ_0 is the yield stress of the material; *k* is a parameter of consistency and n is a flow index. If *n* <1, the material is said to be shear-thinning; If *n*> 1, the material is said to be shear thickening.

The Figure 5 shows clearly that there is a correlation between the Hershel Bulckley model, and the results obtained (Banfill, 2003; Collepardi, 2005). The flow of the material is in steady state if it remains homogeneous (no segregation of the particles).

Spreading- test with mini cone

Spreading with mini cone is typically used to study evolution of the handling of the grout as a function of time. This involves measuring the diameter of the spreading of the slurry on the plate at various time intervals after the preparation of the grout (10, 20, 30, 40, 60, 90 and 120 min). All cements were tested for a W/C ratio = 0.38 in the presence of different dosages of Aeternum-1 adjuvant. The obtained results are given in Figure 6.

When the superplasticizer contacts with water, the dissolution of the constituents of cement begins, which increases the concentration of calcium (Ca^{2+}), alkali (K^+, Na^+), hydroxides (OH^-) and sulphates (SO_4^{2-}). These alkali are initially present in the clinker phases (Na_2O and K_2O) or sulfated form (Na_2SO_4, K_2SO_4, K_2Ca (SO_4) 2H2O. The presence of these salts in the pore water, especially alkalis hydroxides (Na, K) induces a decrease in the relative fluidity (Griesser et al., 2005; Kheribet et al., 2012) as have been noticed for C3, which loses its workability over time; and measuring the reduction in dosage of adjuvant due to the high content of alkali (Na_2O, K_2O) and SO_3, unlike the C2 cement that has good workability over time regardless of the percentage of adjuvant. The nature of cement also affects the workability; the cement C3 contains natural pozzolan which has the porous character and C2 cement contains the limestone filler which is known for its physical properties (better handling and workability).

Spread measurement after different times of rest revealed increase in viscosity after application of a stress or shear rate, the system returns to its initial state (reversible phenomenon), sees its viscosity decreases and spread of cement grouts.

Electrokinetic study of cements at different percentages of superplasticizer

The electrostatic surface potential is another important feature of cement suspensions for adsorption of superplasticizers. Zetametry measurements allow determining the potential (zeta) of ζ particle, which is defined as the measured electrostatic potential at the shear plane of the particle. The potential near the isoelectric point (pH for which the zeta potential is zero) are difficult to measure. Slightly positive or negative values can be found in the literature and depend on the cement composition.

From Figure 7 we see that the zeta potential has values that do not exceed 4.9 mV in absolute value. Potential of cement slurries C2 have negative charges with and without adjuvant, for the cement slurries C3, the zeta potential changes its sign from positive to negative, in which the isoelectric point is obtained for a dosage of 1% adjuvant. Based on the results we can see that Aeternum-1 is better adsorbed by active sites.

Effect of superplasticizer content on the physico-mechanical properties

Evolution of physical properties depending on superplasticizer dosage

Setting time: In this graphical presentation (Figure 8), it

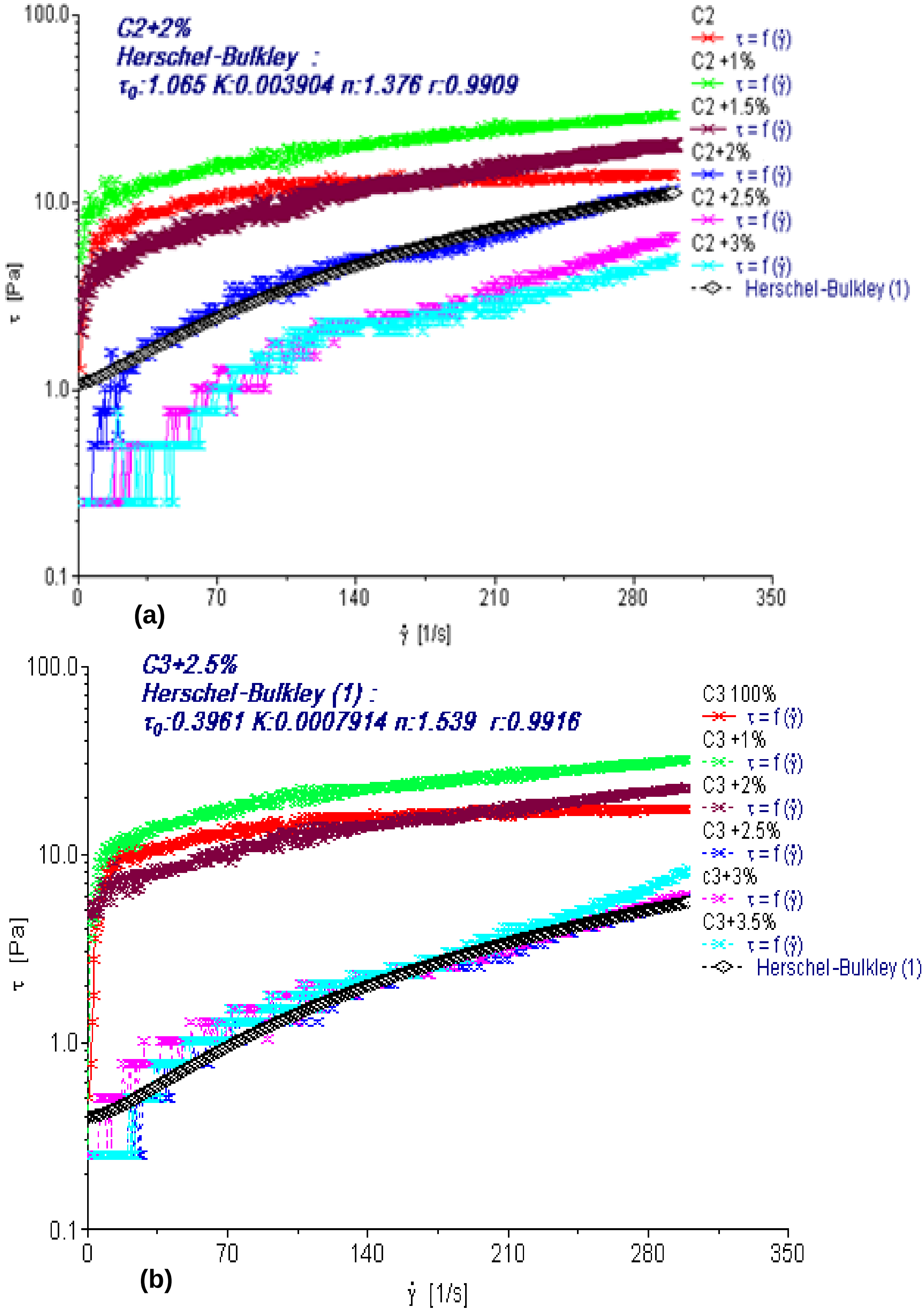

Figure 5. Shear stress as function as shear rate of the cement pastes (C2 and C3) with dosages in adjuvant (0, 1, 1.5, 2, 2.5 and 3%, respectively) (a) C2 (b) C3.

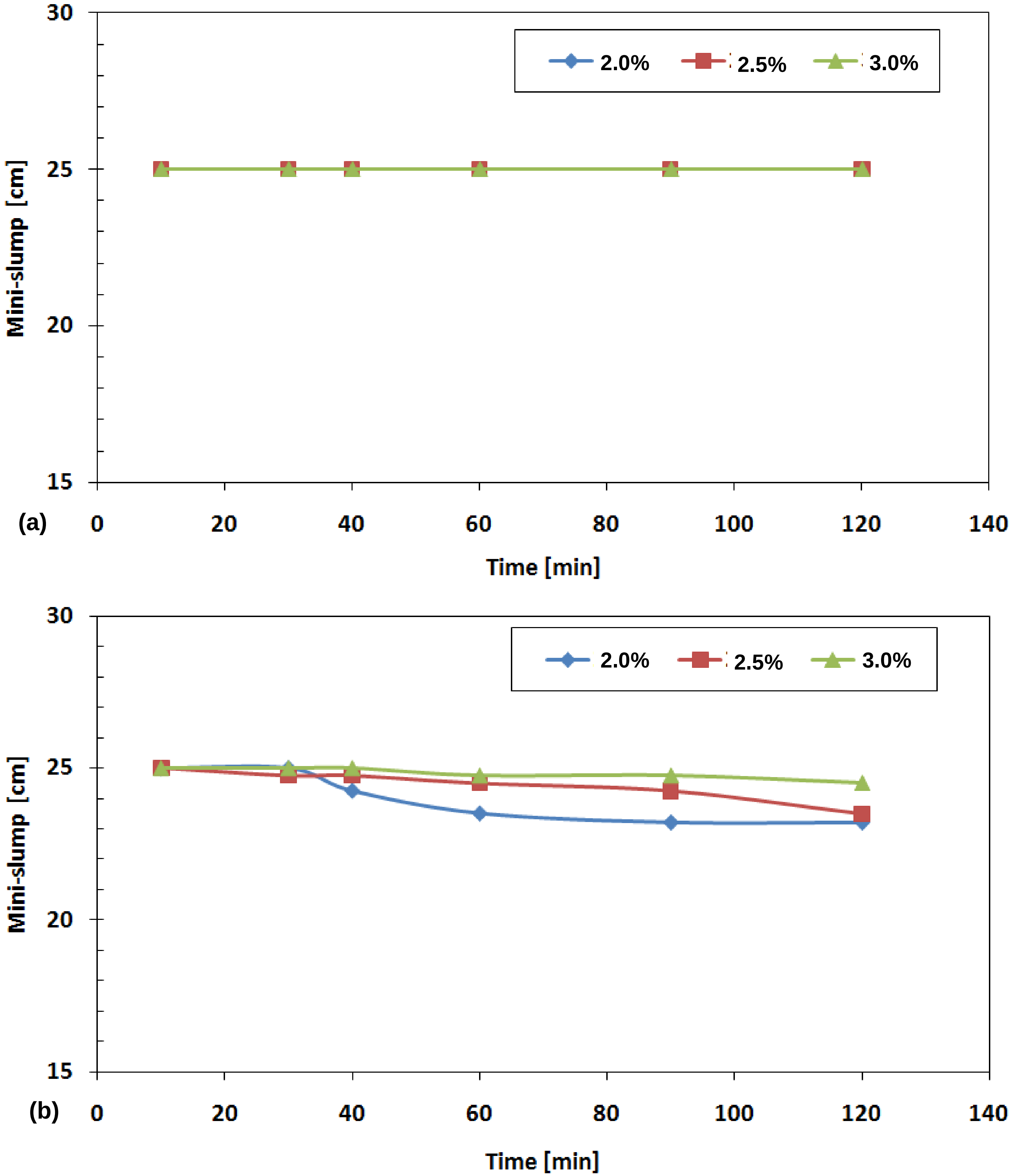

Figure 6. Graphical representations of the results of the spreading with mini-cone (a) C2 (b) C3.

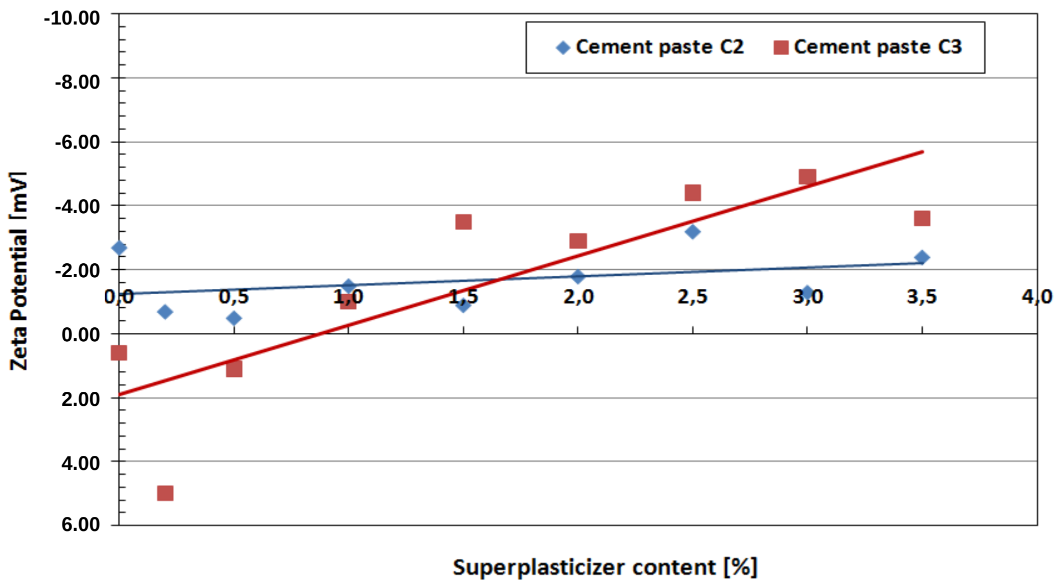

Figure 7. Graphical representation of the results of zeta potential.

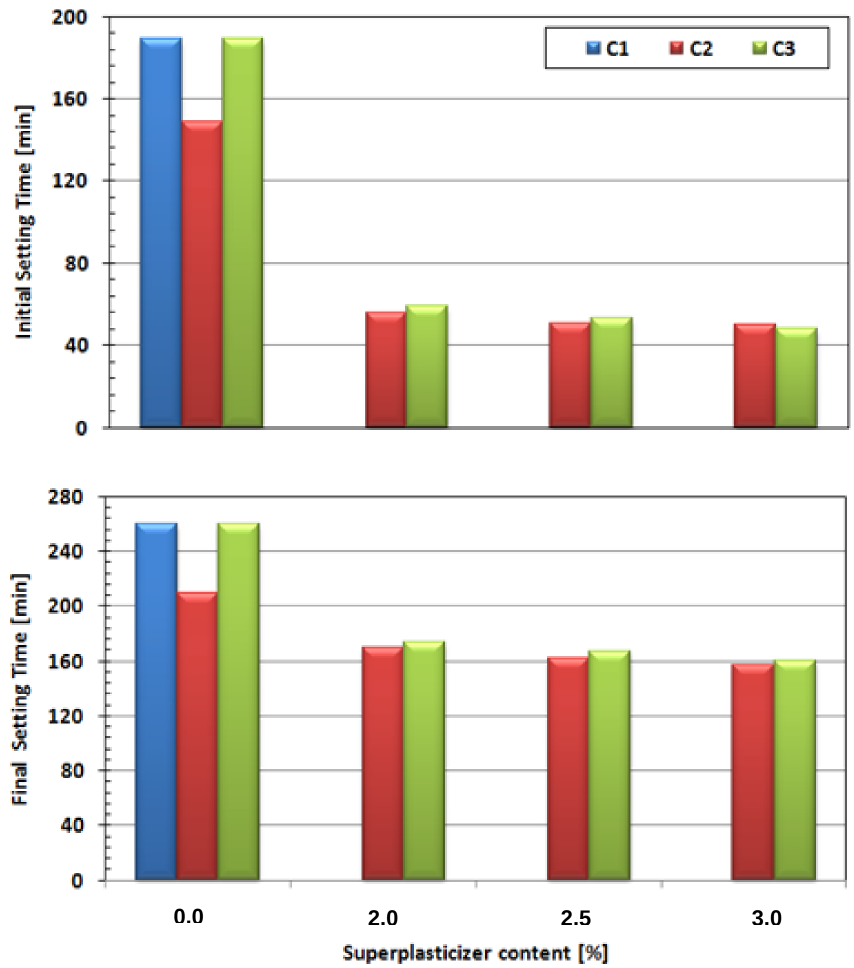

Figure 8. Setting times of cementitious pastes with superplasticizer.

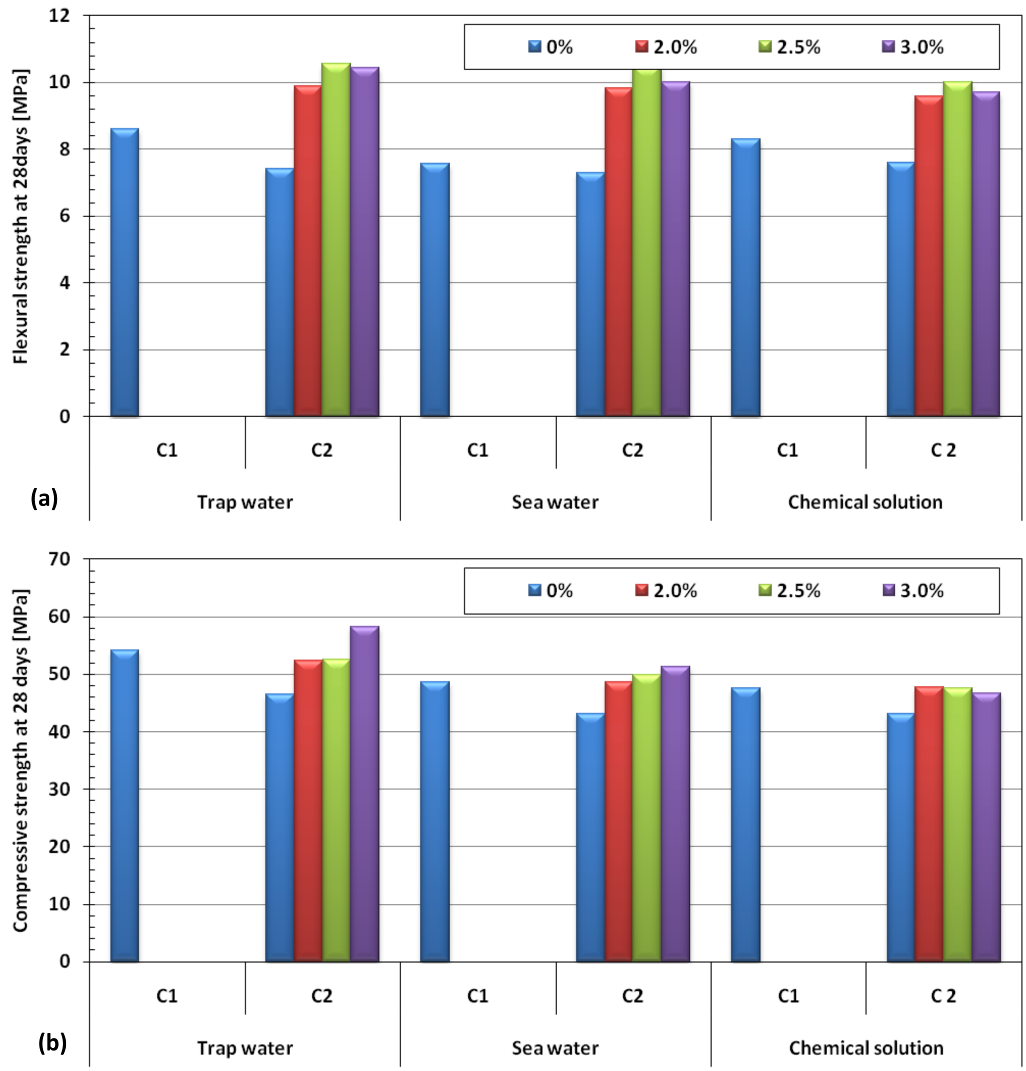

Figure 9. Mechanical strengths at 28 days based mortars C2 (a) Flexural strength (b) Compressive strength.

was noted that: The cement setting time depends on the ratio W/C of temperature and of nature of cement. In the presence of superplsticizer, it appears to take acceleration depending on the dosage of the superplasticizer in cements C2 and C3. The mineralogical composition has a high content of C3S, so there is a large heat of hydration, which involves its rapid dissolution and a precipitation of CSH.

This can be explained by the presence of the superplasticizer which acts as a setting accelerator by nucleation or germination on the surface of the cement grains. This germination causes a rapid setting time, and also by the deflocculating effect of the superplasticizer on cement, thus accelerating the setting and hydration.

Evolution of mechanical strength depending on the age of the test specimens of adjuvanted mortars stored in different environments: To properly study the evolution of mechanical strength in the presence of adjuvant Aeternum-1 various mixtures were carried out whose compositions are as follows: With three storage agressive environments: potable water; seawater; chemical solution, (we consider here a solution dosed at 50 mmol.l^{-1} of magnesium sulphate as aggressive environment. According to NF 18011, such a solution has a degree of aggressiveness A3 by both sulfate and magnesium ions).

Determination of mechanical strengths for the C2 - Comparison between C1 and C2 (with addition of limestone): Figures 9 and 10 show a comparison of flexural and compression strengths of test specimens of a cement mortar with added limestone in the presence of varying percentages of superplasticizer (0, 2, 2.5 and 3%, respectively), with a witness cement (sulphate cement resistant, CRS), preserved in the different environments

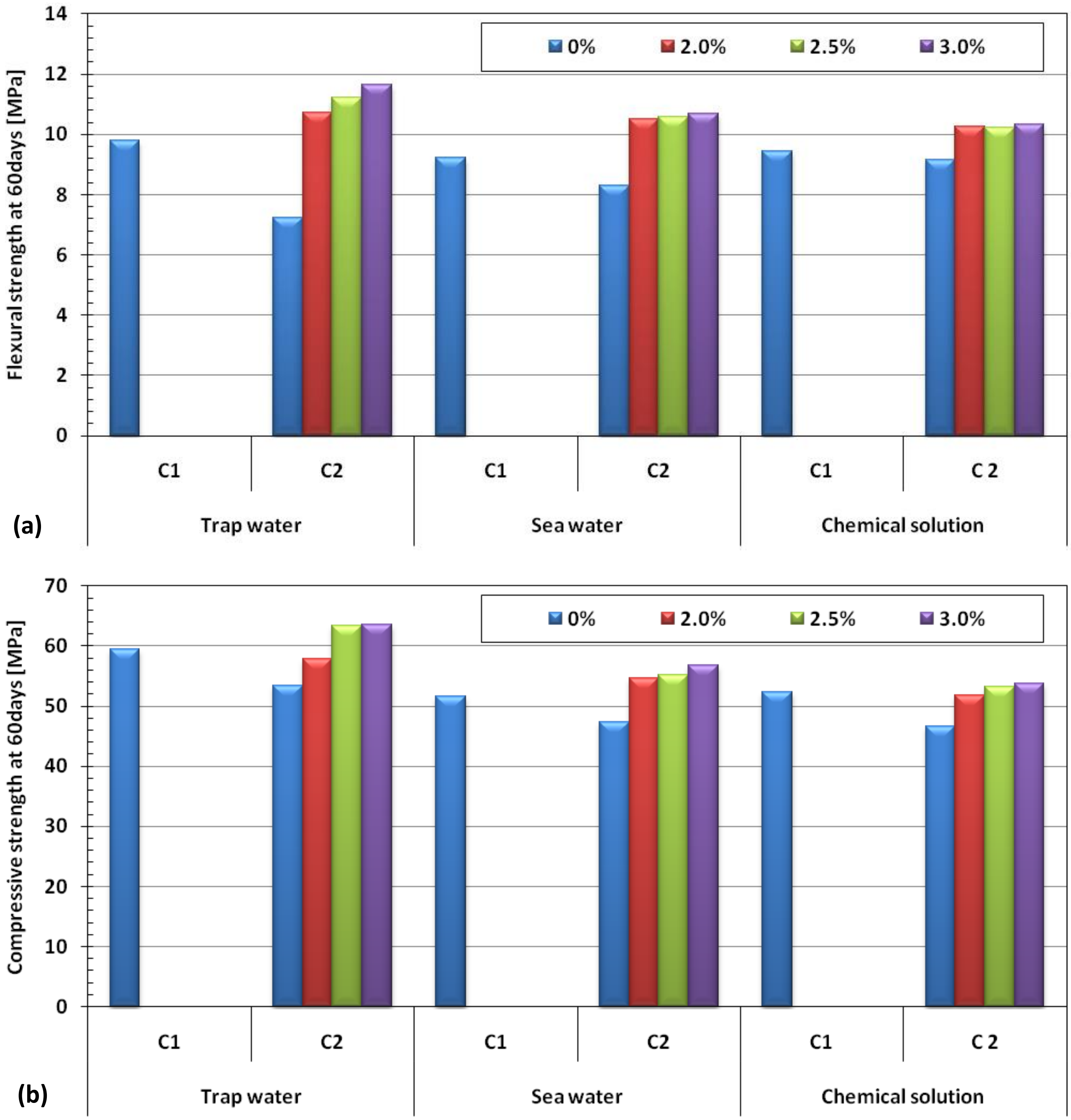

Figure 10. Mechanical strengths at 60 days based mortars C2 (a) Flexural strength (b) Compressive strength.

to different ages (2, 7, 28 and 60 days, respectively).

In the short term, it is noted that flexural and compressive strength of cement C2 is always smaller than the cement witness, but after incorporation of the adjuvant, an increase in resistance is observed. Thus, the test pieces preserved in the drinking water give better strength values than in the sea water and the chemical solution which is due to the aggressiveness of the environment. The increase in resistance in the presence of superplasticizer is caused by making the accelerating effect and rapid hydration of C_3S.

After 28 days of storage, it was noted that the cement witness (C1) shows better resistance in relation cement C2 without superplasticizer, and a decrease in resistance following the aggressiveness of the environment. The adjuvanted mortars give a slight improvement of resistance in aggressive environments; however, we saw very good resistances similar to those obtained by the

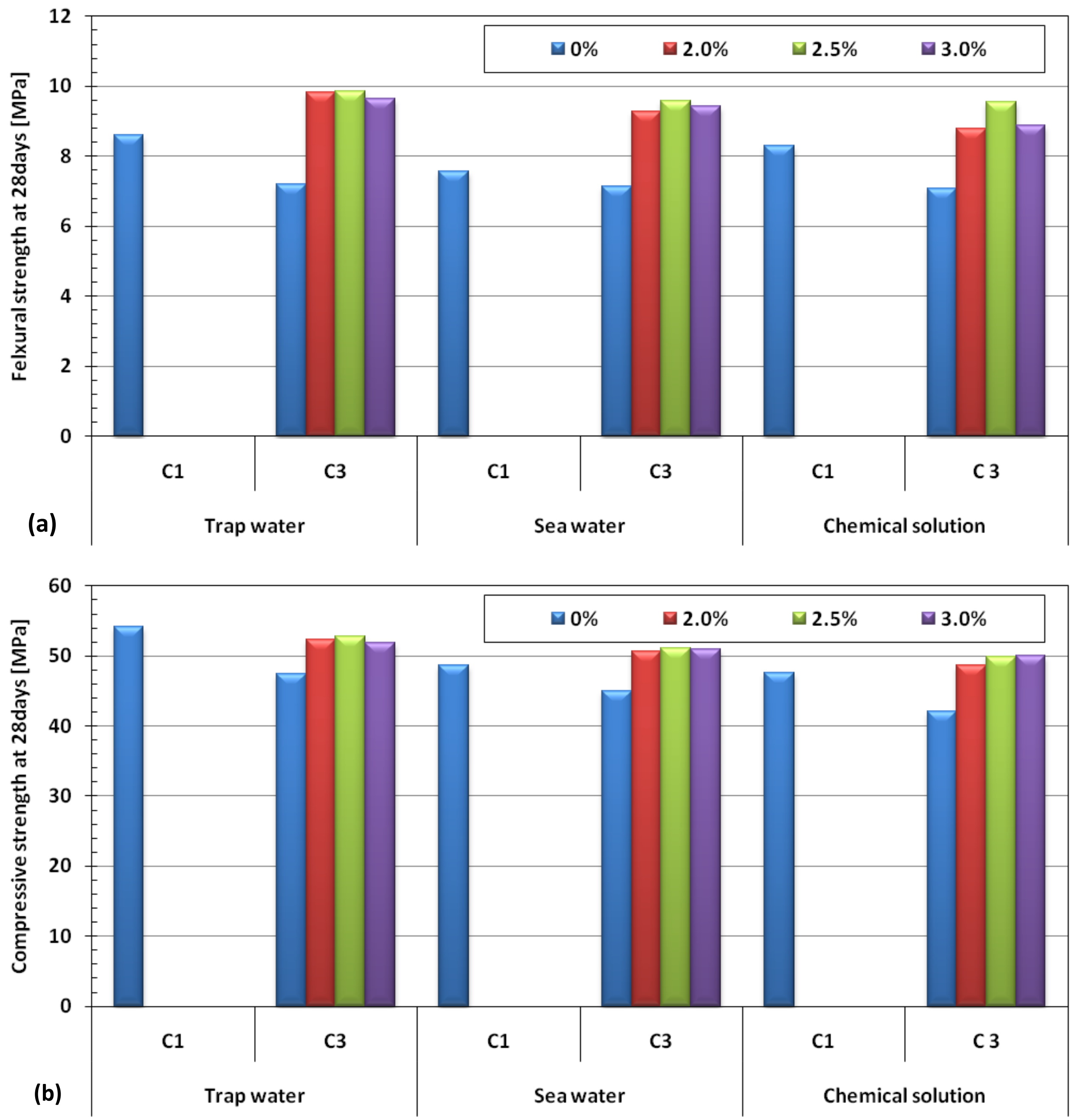

Figure 11. Mechanical strengths at 28 days based mortars C3 (a) Flexural strength (b) Compressive strength.

CRS in drinking water.

At 60 days of storage, we noted that the adjuvanted mortars C2 have better resistances depending on the dosage of superplasticizer and comparable to those of C1 in all environments, which shows the reliability of the plasticizer used.

The two types of cement addition increase resistances to all ages (Kamel et al. 1991; Read et al., 1991; Ghrici et al., 2005). Resistances at early age are due to the acceleration of the hydration of cement, while those at long term develop through the pozzolanic reaction (Kamel et al. 1991; Read et al., 1991) causing refinement of the pores and by replacing portlandite by the CSH.

Determination of mechanical strengths for the C3 (Comparison between C1 and C3 (with addition of pozzolan): Figures 11 and 12 show a comparison of flexural and compression strength of test specimens of cement mortar with addition of pozzolan with various percentages of superplasticizer (0, 2, 3 and 2.5%,

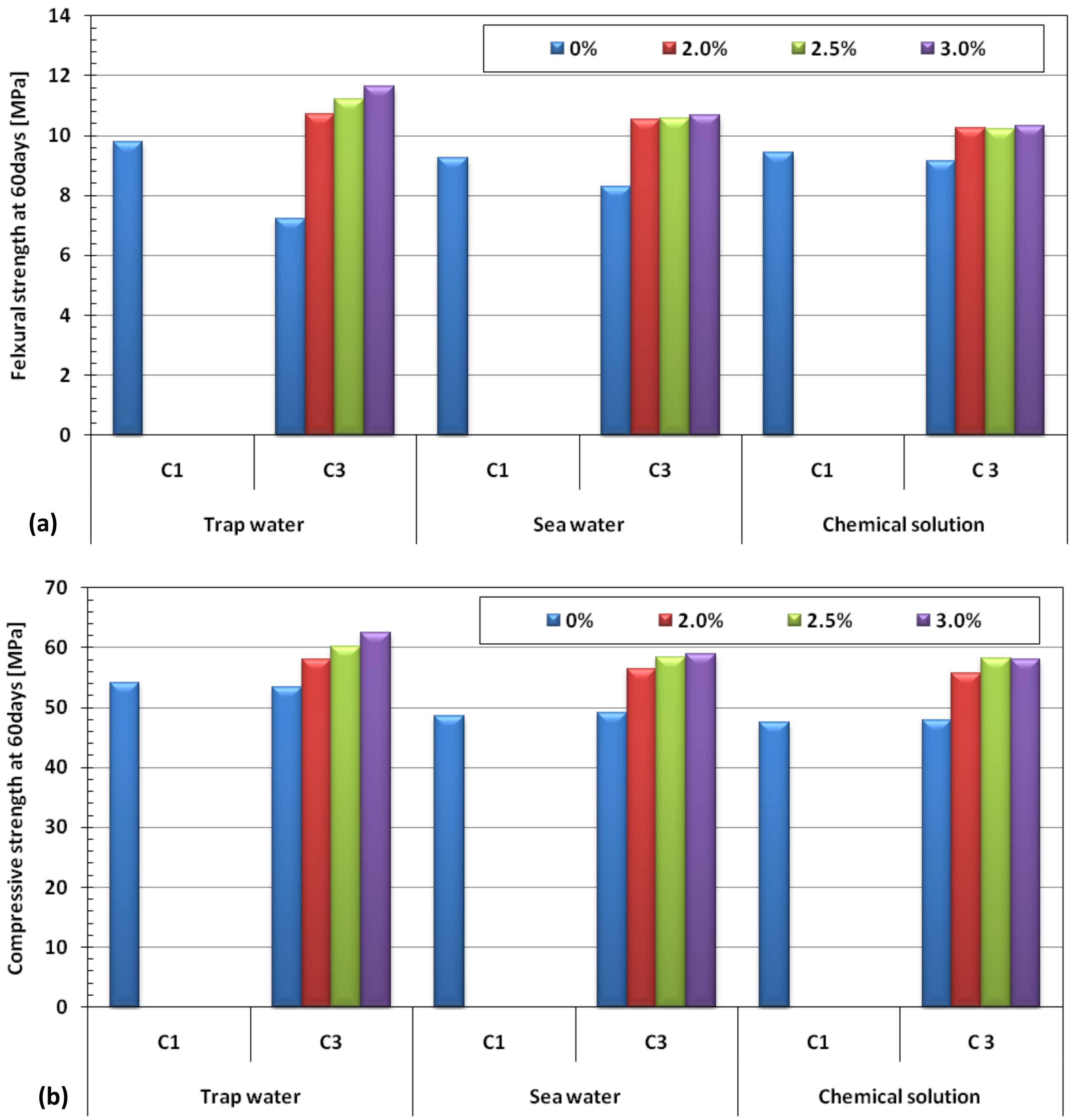

Figure 12. Mechanical strengths at 60 days based mortars C3 (a) Flexural strength (b) Compressive strength.

respectively), with a witness cement (sulphate resistant cement, CRS), preserved in different environments for ages (2, 7, 28 and 60 days, respectively).

From these figures, we note that the non-adjuvanted cement mortars C3 develop flexural and compressive strengths always lower than the witness mortar, and that at all age, and a reduction in the resistance according the environment aggressivity. For samples of mortars containing adjuvant dosages (2, 2.5 and 3%, respectively), mechanical resistances develop as follows:

After 28 days of storage we noted that the adjuvanted mortars have resistances comparable to those of the witness in the different environments.

At 60 days of storage, we note that the adjuvanted mortars C3, have better resistances depending on the dosage of adjuvant and are comparable to those of C1 in all environments, which shows the reliability of the adjuvant used.

The two types of cement addition increase mechanical strengths to all ages. Mechanical strengths at an early age are due to the acceleration of the hydration of cement, while those at long term develop through the pozzolanic reaction causing refinement of the pores and by replacing portlandite by the CSH (Read et al., 1991; Ghrici et al., 2005).

Conclusion

The use of the adjuvant with a percentage (2, 2.5 and 3%), substantially reduces the value of W/C deflocculated the cement particles; this reduction results in an increase in resistance to a maximum value (63.55 MPa). Age is a predominant parameter, given the observed results, particularly on the mechanical resistance. The cement with addition of pozzolan, gives the best mechanical resistances; this is due to the pozzolanic reactivity, (natural pozzolan contained in the cement and amorphous silica present in the superplasticizer).

Beyond 28 days of storage, the mortars containing pozzolan have high mechanical strength compared to the C1 (sulphate resistant cement) through the double pozzolanic activity of the pozzolan and silica fume contained in the adjuvant; also, the structure of the cement paste is modified by the formation of the CSH and decrease in portlandite. That is to say, the additional hydrate formation CSH, which precipitate in the pores, decreases the porosity and increases the compactness of the cement paste.

Cement with limestone addition and adjuvanted, present significant mechanical resistances. Indeed, the presence of fine limestone consolidates and densifies the cementitious matrix by filling the pores of the structure and CSH formation, and graced the pozzolanic reactivity of the amorphous silica of the adjuvant.

The behavior of cement with limestone addition to the inverse of the pozzolan develops its mechanical resistance to short-term (7 days), but they are less important compared to those obtained by the cement with pozzolan addition to long term (28j and 60j).

Finally, this study opens new perspectives on the possibility of using composite cements CEM II-A with adjuvant/superplasticizer (Aeternum-1) in aggressive environments.

Conflict of Interest

The authors have not declared any conflict of interests.

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