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Effect of superplasticizer and shrinkage-reducing admixtures on alkali-activated slag pastes and mortars

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Abstract

This paper shows how several superplasticizers (polycarboxylates, vinyl copolymers, melamine and naphthalene-based) and shrinkage-reducing (polypropylenglycol derivatives) admixtures affect the mechanical and rheological properties and setting times of alkali-activated slag pastes and mortars. Two activator solutions, waterglass and NaOH, were used, along with two concentrations—4% and 5% of Na_2O by mass of slag. All admixtures, with the exception of the naphthalene-based product, lost their fluidifying properties in mortars activated with NaOH as a result of the changes in their chemical structures in high alkaline media. The difference in the behaviour of these admixtures when ordinary Portland cement is used as a binder is also discussed in this paper.

Keywords: Alkali activated cements; Admixtures; Mechanical properties; Workability

1. Introduction

Organic admixtures are added to Portland cement concrete to enhance its properties. Superplasticizers, for instance, yield concrete with high rheological requirements, while maintaining a low water/cement (w/c) ratio to guarantee excellent mechanical properties and long durability [1]. There is a wide variety of superplasticizer admixtures, such as lignosulphonates, naphthalene and melamine-based, vinyl copolymers and the so-called latest generation of superplasticizers, polycarboxylate derivatives. These admixtures are adsorbed on the cement particles, causing electrostatic or steric (in the case of polycarboxylate admixtures) repellency that hinders flocculation.

Shrinkage-reducing admixtures, in turn, tend to decrease the surface tension of the water in the concrete pores, thereby lowering the capillary tension within the pore structure and therefore decreasing shrinkage when the water evaporates [1,2]. The chemical composition of such shrinkage-reducing admixtures is based on polypropylenglycol.

Whereas the effect of these superplasticizers and shrinkage-reducing admixtures has been extensively studied in Portland cement concretes, mortars and pastes [1,3–8], their effect on other binders, such as alkali-activated slag (AAS) pastes and mortars, has received less attention. Blast furnace slag is an industrial by-product that can be alkali-activated to yield adhesive and cementitious compounds, whose production is less energy-intensive and emits less CO_2 than ordinary Portland cement manufacture. Compared to Portland cement, these activated slag cements generate less heat of hydration, reach high strengths at early ages and provide excellent durability in aggressive media [9,10]. Wang et al. [11], studying the inclusion of water-reducing admixtures, such as sodium lignosulphonate and a naphthalene-based superplasticizer in alkali-activated slag mortars, concluded that such admixtures caused a decrease in compressive strengths without improving workability. Other authors [12,13] studied the effect of a water-reducing and set-retarding admixture on properties of blast furnace slag concrete when the slag was activated with a NaOH and

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Table 1
Chemical composition (percent in mass)

	Slag	OPC
LOI	2,02	0.78
SiO_2	34,95	19.97
IR	0,11	0.29
Al_2O_3	13,11	5.17
Fe_2O_3	0,69	3.85
CaO	41,37	64.41
MgO	7,12	1.30
SO_3	0,04	2.64
S^{2-}	1,92	–
Na_2O	0,27	0.39
K_2O	0,23	0.78
CaO free	–	0.22

LOI: loss on ignition; IR: insoluble residue.

Na_2CO_3 mix. In this case, mortar workability was enhanced at the expense of concrete strength measured after 1 day of curing, with strength loss increasing with admixture content. Bakharev et al. [10] prepared alkali-activated slag concrete to which different admixtures, such as superplasticizers or water-reducing or air-entraining compounds, were added. These authors observed a decrease in mechanical strength in the first 28 days when the admixture used was a naphthalene-based superplasticizer or a lignosulphonate derivative water-reducing compound. The naphthalene-based admixture was observed to increase workability in the early stages, although the concrete set very rapidly thereafter, while the lignosulphonate-based admixture had a more prolonged beneficial effect on workability. Moreover, although the air-entraining compound decreased the strength slightly in the first 7 days of curing, subsequent development was similar to that observed in alkali-activated slag concrete without admixtures, and concrete workability was significantly enhanced. Puertas et al. [14] studied the effect of two superplasticizer admixtures—based on vinyl copolymers and polycarboxylates—on waterglass-activated slag mortars and pastes. They concluded that the vinyl copolymer-based admixture decreased mortar mechanical strengths after 2 and 28 days without increasing paste workability, while the polycarboxylate admixture had no effect on the mechanical performance of the mortar but did improve paste workability.

The variability in the research results reported, as illustrated earlier, is due to differences in the conditions in which pastes, mortars and activated concrete were prepared (composition of the slag, nature and concentration of the activator, type and dosage of admixture, etc.). Moreover, none of these studies has explained the differential behaviour of such organic admixtures in Portland cement on the one hand and activated slag cement on the other. The purpose of this research, then, is to study the effect of different superplasticizer and shrinkage-reducing admixtures on the mechanical strengths, slump and setting of alkali-activated slag paste and mortar, compared to their performance in Portland cement pastes and mortars, and explain the behaviour of these admixtures in high alkaline media.

2. Experimental

2.1. Materials

The chemical composition of blast furnace slag and Portland cement type I 42.5 R used in this study is given in Table 1. The specific surface area of the slag and Portland cement were 325 and 360 m^2/kg, respectively, and the vitreous phase content of the blast furnace slag was 99%. Two different alkali activators, waterglass [$(Na_2O \cdot nSiO_2 \cdot mH_2O$ $(SiO_2/Na_2O=3.4)$+NaOH] and NaOH, were used in the alkali activation of the slag.

Six types of chemical admixtures were used (see Table 2):

- five superplasticizers
  - two polycarboxylate admixtures (PC1, PC2)
  - one melamine-based (M)
  - one naphthalene-based (NF)
  - one vinyl copolymer (V)
- one shrinkage-reducing
  - polypropylenglycol derivative (SRA)

One percent of each admixture by mass of binder (slag, cement) was added to the solution.

2.2. Testing procedures

2.2.1. Liquid to solid ratio

Alkali-activated slag and Portland cement mortars were prepared with an aggregate to binder ratio of 2:1; silica (99% SiO_2, quartz) aggregate was used. Two types of alkaline solutions were used to activate the blast furnace slag mortars, NaOH and waterglass. In this case, the waterglass solution had 30% by mass of $SiO_2 \cdot nNa_2O \cdot mH_2O$. Moreover, two different concentrations of Na_2O were used, 4% and 5% by mass of slag.

The liquid to solid (l/s) ratio was determined by slump test Spanish standard UNE-80-116. In this test, a truncated conical mould (70×100×60 mm) was filled with the mortar. The mortar is shaking 10 times in the jolting table and four diameters has been measured. The final value was

Table 2
Physical and chemical properties of the admixtures

Admixture	PC1	PC2	M	NF	V	SRA
Solid content (%)	38	38	40	40	25	–
pH	5.40	4.65	8.22	7.86	6.80	10.70
Density (g/cm^3)	1.09	1.09	1.23	1.20	1.14	1.00
Rotational viscosity (MPa)	79.50	70.37	57.27	58.67	24.13	26.93
% Carbon (C)	52.49	52.61	29.03	46.59	34.05	57.27

Table 3
Liquid/solid ratio obtained by slump test UNE-80-116-86

Binder	Activator solution	Concentration Na_2O (%)	Admixture						
			R	V	M	NF	SRA	PC1	PC2
Slag	Waterglass	4	0.58	0.55	0.55	0.55	0.55	0.55	0.56
		5	0.60	0.58	0.58	0.58	0.55	0.59	0.58
	NaOH	4	0.50	0.49	0.48	0.43	0.48	0.48	0.48
		5	0.51	0.49	0.49	0.45	0.49	0.50	0.49
OPC	–	–	0.42	0.36	0.38	0.36	0.41	0.31	0.31

the arithmetic mean of these measurements, and this diameter must be in the range 105±5 mm.

2.2.2. Mechanical strengths

Mortar specimens (4×4×16 cm) described in the previous section were prepared according to European standard EN 196-1 and were cured at 20±2 °C and 99% relative humidity until the day of the test. Mechanical strengths—flexural and compressive—were determined on these specimens after 2, 7 and 28 days.

2.2.3. Minislump tests

Minislump tests were conducted to determine paste flowability, with and without admixtures, after 3, 10, 30 and 60 min. For these tests, pastes with a l/s ratio of 0.5 were prepared in the mixer for 3 min and subsequently poured into a truncated conical mould (19×38.1×57.2 cm). The diameter was measured in four directions after 10 blows with the jolting table. The final value was the arithmetic mean of these measurements.

2.2.4. Setting tests

The initial and final setting times were determined according to European standard EN 196-3. These tests were run on the same pastes that were used in the minislump tests.

2.2.5. Admixture stability tests

Stability tests were conducted on the organic admixtures in three alkaline solutions, $Ca(OH)_2$ (pH 12.40), NaOH (pH 13.6) and waterglass (pH 13.40), to ascertain how they were affected by highly alkaline media. The alkaline solution to admixture ratio was 1:1.

Approximately 1 g of admixture was taken at different ages and dried in a vacuum dryer to study the changes in its chemical structure with Fourier transform infrared spectro-

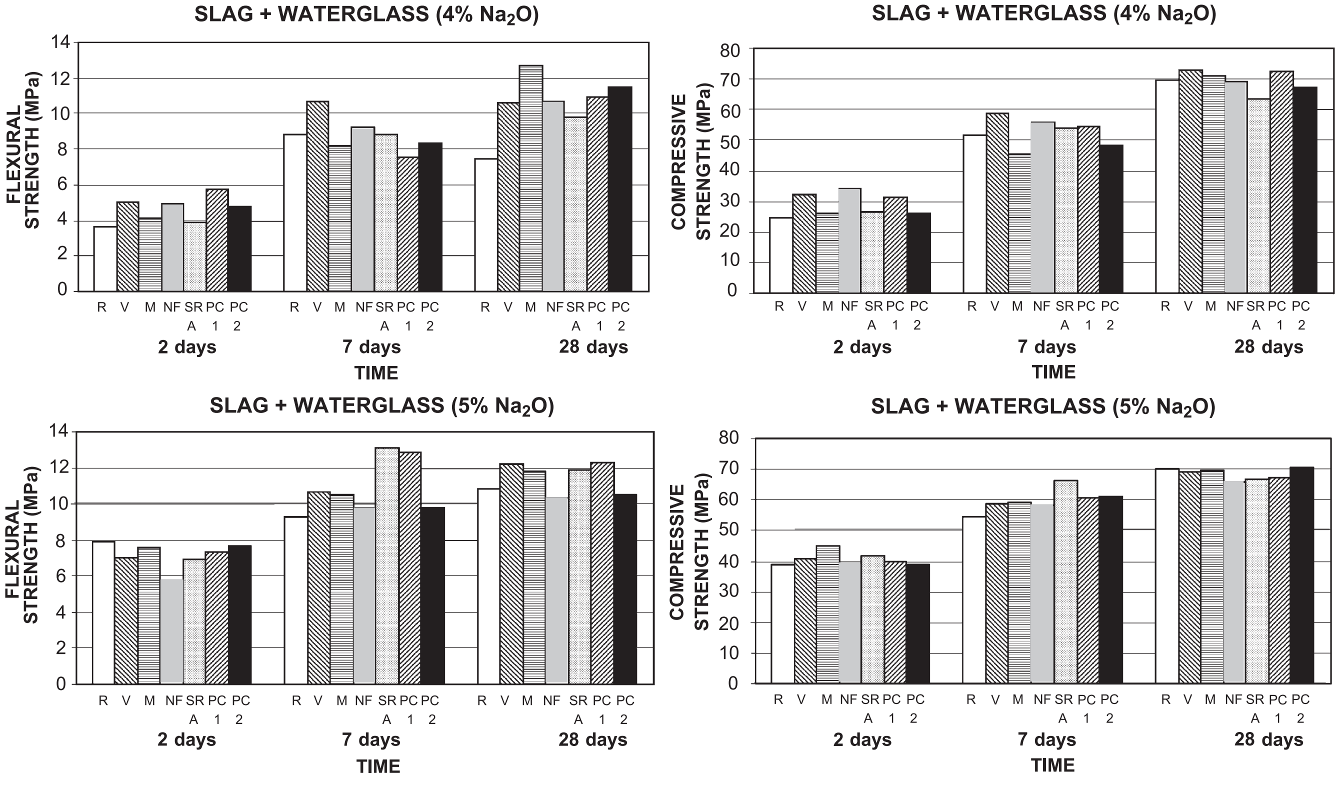


Fig. 1. Flexural and compression strengths of waterglass-activated slag mortars.

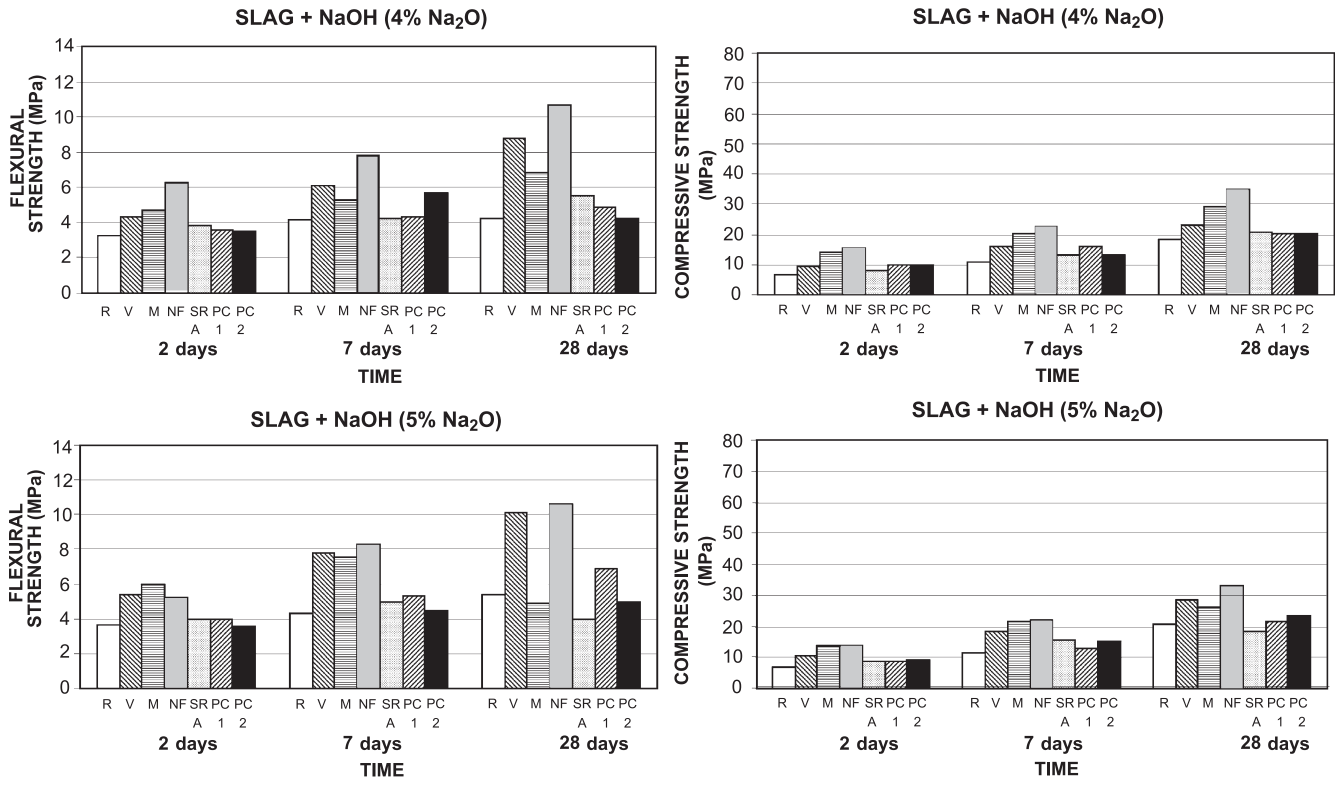


Fig. 2. Flexural and compressive strengths of NaOH-activated slag mortars.

scopy (FTIR). An ATIMATTSON GENESIS spectrometer was used. The spectra were recorded from KBr pellets, weighing 300 mg of KBr and 1 mg of admixture.

3. Results

3.1. Liquid to solid ratio

In all cases, the admixtures reduced the liquid to solid ratio, a reduction that proved to be highly significant when the binder used was Portland cement type I. In the case of alkali-activated slag mortars, the greatest reduction was observed when the activator solution was NaOH and the admixture was a naphthalene derivative (see Table 3).

3.2. Mechanical strengths

Figs. 1–3 show the compressive and flexural strength values over time of waterglass- and NaOH-activated slag, and Portland cement mortars.

Both the flexural and compressive strength values of the NaOH-activated slag mortars were found to be much lower than waterglass-activated and Portland cement mortars.

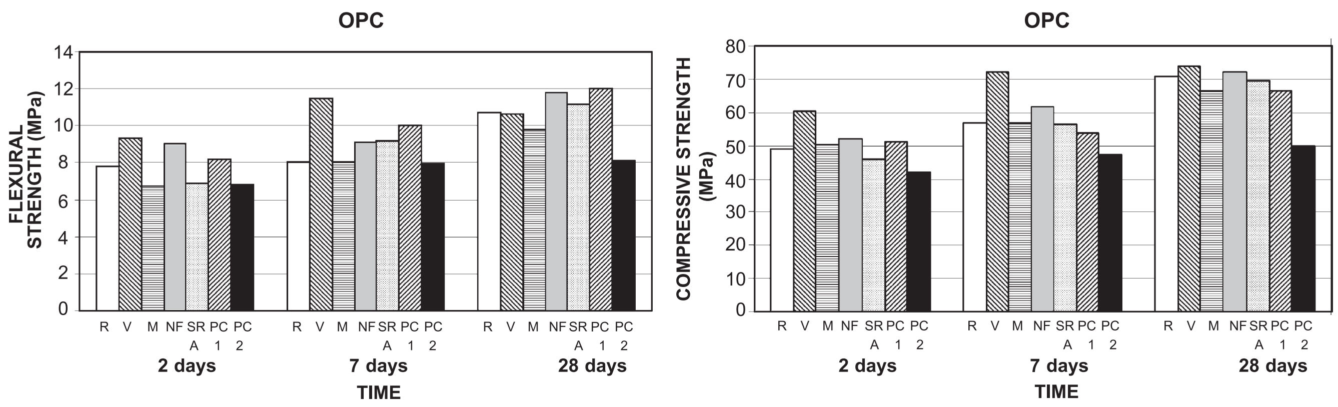
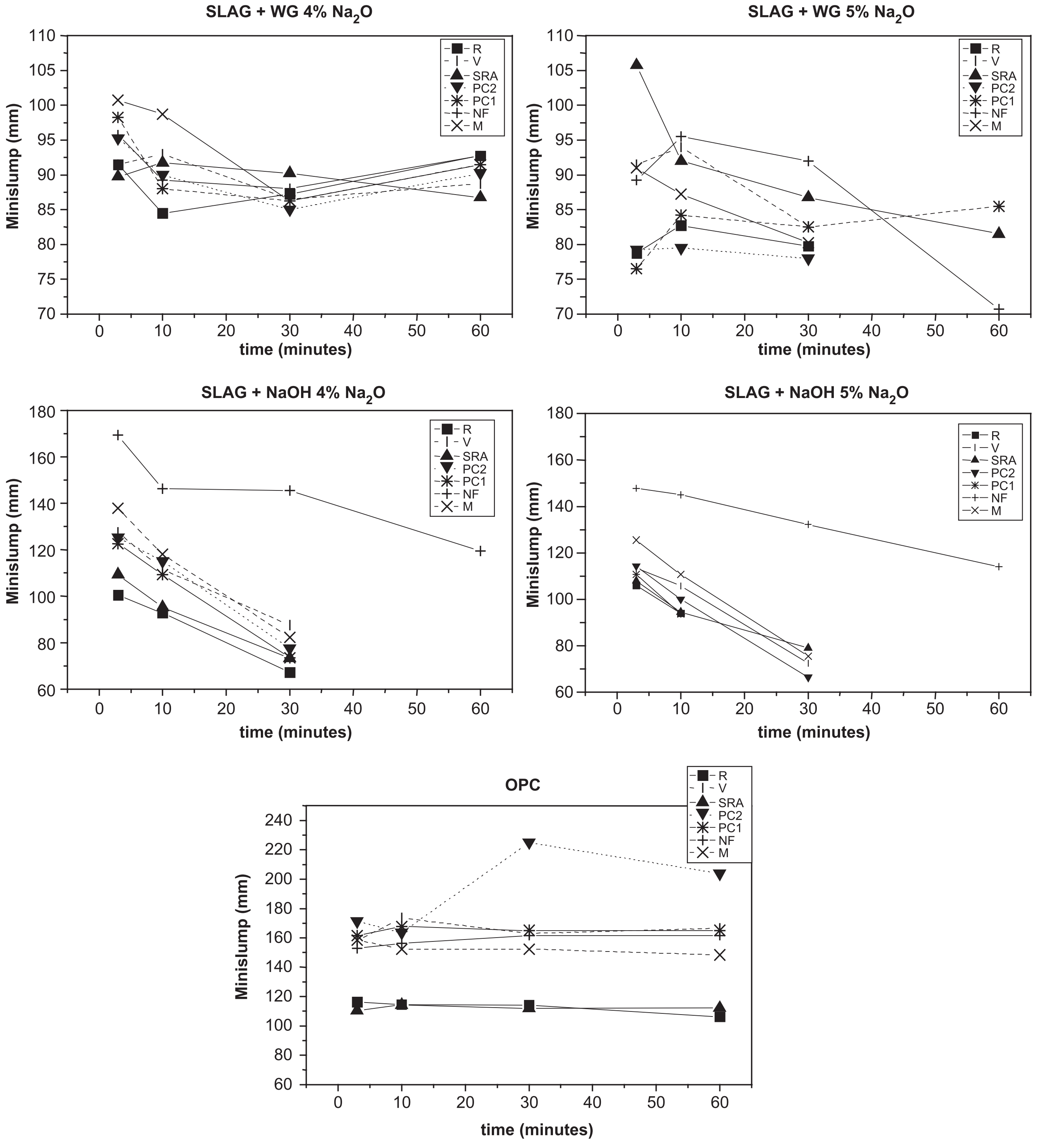


Fig. 3. Flexural and compressive strengths of type I cement mortars.

The strengths of waterglass-activated slag mortars with 4% Na_2O content, in the absence of admixtures (R), was lower than the values obtained for Portland cement at all ages of curing. The same effect was observed in mortars containing 1% of the different admixtures after 2 days of curing, while at greater ages, the mortars with PC1, V and NF admixtures equalled or even surpassed the cement mortar strengths.

Finally, in the case of waterglass-activated slag mortars with 5% of Na_2O, after 2 days of curing, the strength values were lower than those for Portland cement but higher than for waterglass-activated slag with 4% of Na_2O; after 7 and


SLAG + WG 4% Na_2O

SLAG + WG 5% Na_2O

SLAG + NaOH 4% Na_2O

SLAG + NaOH 5% Na_2O

OPC

Fig. 4. Development in time of the slump of the alkali-activated slag and Portland cement pastes.

28 days, the strength values achieved with all the suplerplastizers were similar to or in many cases even higher than the values found for Portland cement mortars.

3.3. Minislump tests

The results of the minislump tests are shown in Fig. 4. When waterglass was the activator solution, none of the admixtures increased the slump of slag pastes. When the activator was NaOH, flowability increased slightly in the first 10 min with PC1, PC2, V and M, while the naphthalene-based admixture increased flow rate significantly during the full 60 min that the test lasted. The shrinkage-reducing admixture had no impact on paste slump.

By contrast, all the superplasticizers enhanced Portland cement paste workability, with the highest rise in flowability observed when the polycarboxylate (PC2) was added to the mix.

3.4. Setting tests

The initial and final setting times for alkali-activated and Portland cement pastes are given in Table 4. The initial and final setting times of the activated slag pastes were much shorter than for the Portland cement pastes. These results agree with those reported by other authors [12,13,15].

In the case of waterglass-activated slag pastes with 4% of Na_2O, the admixtures had no significant effect on setting, with the exception of the vinyl copolymer, which retarded the initial set by nearly 40 min and the final set by over an hour.

In the case of waterglass-activated slag pastes with a 5% Na_2O content, in turn, the setting times were affected by the presence of some admixtures. The vinyl copolymer, for instance, accelerated the initial set slightly but lengthened the final setting time by nearly 5 h. The admixtures based on melamine and polycarboxylate (PC1) retarded the initial set by 1 h and the final set by about 2 h, as compared to the slag paste without admixtures. The naphthalene-based admixture shortened both initial and final setting times.

NaOH-activated slag pastes with 4% and 5% of Na_2O were observed to have very similar setting times, although compared to the waterglass-activated pastes, the initial times were slightly longer and the final times shorter in most cases. However, the setting times for the pastes containing the naphthalene-based admixture were significantly longer.

The impact of the various admixtures on cement paste setting times was much greater than in alkali-activated slag pastes. The initial and final setting times were substantially longer in the presence of suplerplasticizer admixtures, and in the case of the vinyl copolymers and the two polycarboxylates, the time between the initial and final set was very short. The shrinkage-reducing admixture, however, increased the setting time slightly.

Table 4
Setting times of alkali-activated slag and cement Portland pastes

Binder	Admixtures	Initial setting	Final setting	Δt
Slag+waterglass (4% Na_2O)	R	41 min	1 h 36 min	55 min
	V	1 h 21 min	2 h 46 min	1 h 25 min
	M	30 min	1 h 18 min	48 min
	NF	29 min	2 h 14 min	1 h 45 min
	SRA	54 min	2 h 12 min	1 h 18 min
	PC 1	21 min	1 h 38 min	1 h 17 min
	PC 2	36 min	1 h 36 min	1 h
Slag+waterglass (5% Na_2O)	R	56 min	2 h 21 min	1 h 25 min
	V	27 min	7 h 32 min	7 h 5 min
	M	1 h 54 min	4 h 14 min	2 h 20 min
	NF	32 min	1 h 5 min	33 min
	SRA	45 min	1 h 58 min	1 h 8 min
	PC 1	2 h 6 min	4 h 16 min	2 h 10 min
	PC 2	47 min	1 h 30 min	43 min
Slag+NaOH (4% Na_2O)	R	1 h	1 h 24 min	24 min
	V	1 h 14 min	2 h 8 min	54 min
	M	1 h 2 min	1 h 35 min	33 min
	NF	3 h 12 min	5 h 20 min	2 h 8 min
	SRA	49 min	1 h 10 min	21 min
	PC 1	1 h 4 min	1 h 43 min	39 min
	PC 2	1 h 3 min	1 h 51 min	48 min
Slag+NaOH (5% Na_2O)	R	51 h	1 h 25 min	34 min
	V	57 min	1 h 20 min	23 min
	M	49 min	1 h 15 min	24 min
	NF	1 h 49 min	2 h 25 min	36 min
	SRA	47 h	1 h 11 min	34 min
	PC 1	43 min	1 h 7 min	30 min
	PC 2	51 min	1 h 21 min	30 min
Portland cement	R	3 h 31 min	5 h 36 min	3 h 31 min
	V	12 h 13 min	12 h 43 min	30 min
	M	6 h 33 min	7 h 21 min	48 min
	NF	10 h 6 min	12 h 21 min	2 h 15 min
	SRA	4 h 4 min	5 h 59 min	1 h 55 min
	PC 1	17 h 42 min	18 h 12 min	30 min
	PC 2	12 h 13 min	12 h 43 min	30 min

3.5. Admixture stability tests

The infrared spectra for the admixtures in different alkaline media are given in Fig. 5. These spectra show that the chemical structure was not modified in any of the admixtures when they were kept in a $Ca(OH)_2$ solution, whereas with the exception of the polypropylenglycol and the naphthalene derivatives (in NaOH), they all underwent formulation changes when kept in the other two solutions.

For instance, in the infrared spectra for the polycarboxylate and vinyl copolymer admixtures, the band at 1730 cm^{-1} corresponding to the C=O of the carboxylic derivatives disappeared, while bands appeared around 1575 and 1418 cm^{-1}, which correspond to carboxylate groups (COO^-). The bands observed to appear in the vinyl copolymer spectrum at 3440, 3385 and 1601 cm^{-1} were attributed to NH_2 vibrations.

The melamine-based also underwent alterations in its formulation when kept in high alkaline media. The infrared

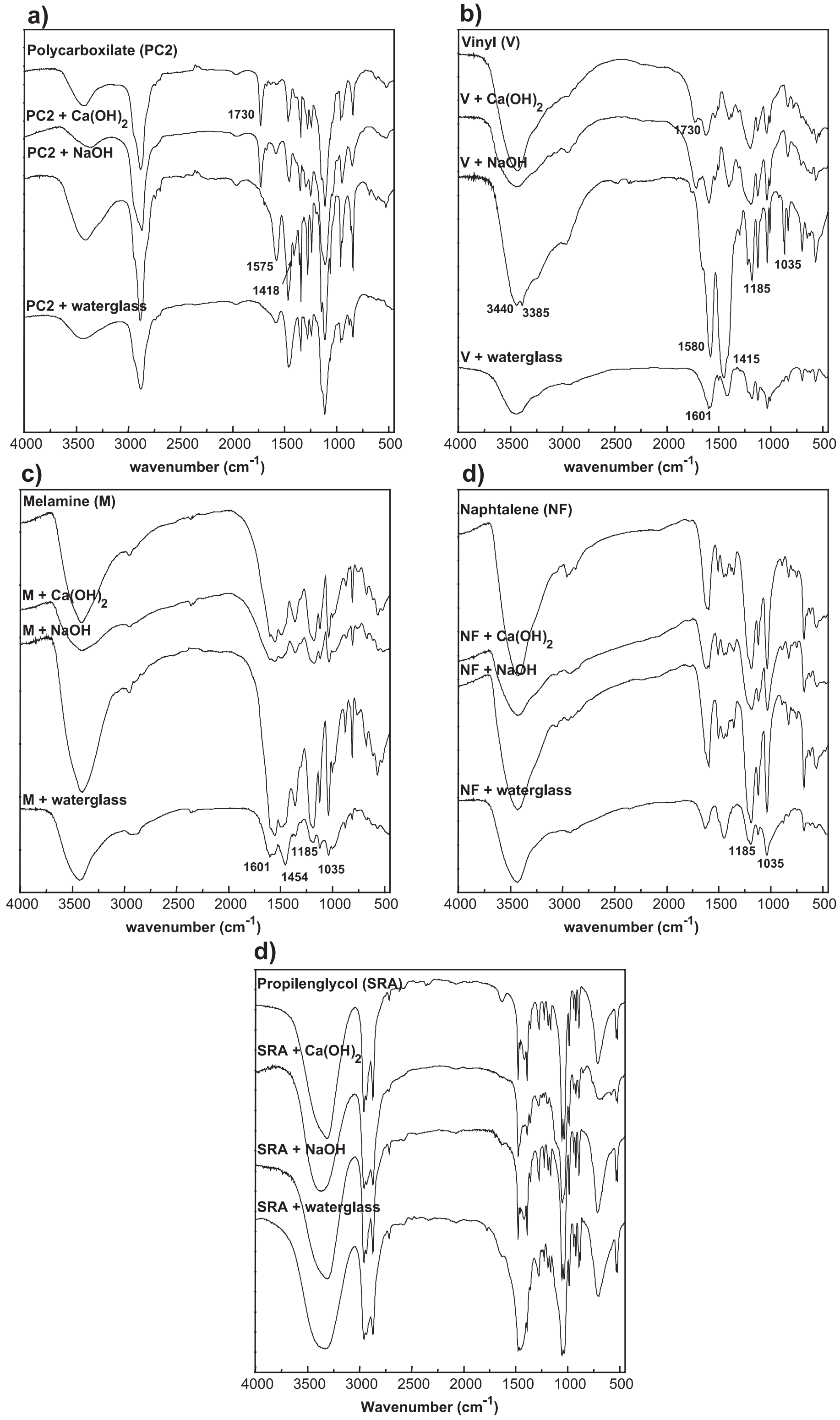


Fig. 5. Infrared spectra of admixtures in different basic media.

spectrum for this compound showed a small rise in the intensity of the bands at 1601 and 1454 cm^{-1}, which correspond, respectively, to the vibrations of the N–H bond in the amine and the C–H bond in the CH_2 adjacent to a heteroatom.

When the naphthalene and melamine-based and the vinyl copolymers were kept in waterglass solutions, changes were observed in the infrared spectra in the zone between 1185 and 1030 cm^{-1}, which were attributed to the vibrations of SO_3 groups.

4. Discussion

The effect of the admixtures used in this study on alkali-activated slag mortars and pastes differed substantially from their effect on Portland cement mortars and pastes.

Much higher mechanical strengths were observed in waterglass- than in NaOH-activated slag mortars. These results agree with reports by other authors [16,17] who carried out factorial experimental designs from which they concluded that the nature of the activator solution is the most statistically significant variable in the alkali activation of blast furnace slag.

According to Fernández-Jiménez et al. [18], when slag is activated with waterglass, the hydrate calcium silicate formed consists of chains of highly condensed silicate anions, as confirmed by the high proportion of units of Si Q^2 and Q^3 observed in ^{29}Si-NMR testing. This facilitates the formation of cross-linked structures that give rise to high strength values. When NaOH is the activator, however, since the hydrate calcium silicate contains a high proportion of Si Q^2 but no Q^3 units, the mechanical strength values are lower.

The effect of the admixtures on the mechanical properties of the different mortars tested depends on the type of binder and admixture used and, in the case of alkali-activated slag mortars, the nature of the alkaline activator.

In waterglass-activated slag, at any of the percentages of Na_2O used, all the admixtures reduce the l/s ratio minimally, occasioning a slight increase in strength with respect to mortars with no admixtures. However, when a 5% concentration of Na_2O is used in combination with the shrinkage-reducing admixture (SRA), the l/s ratio dropped from 0.60 to 0.55, providing for a significant rise in strength after 2 and 7 days, but not after 28.

The naphthalene-based admixture has a substantially different effect than any of the other admixtures on NaOH-activated slag: it significantly reduces the l/s ratio, thereby greatly enhancing mechanical strength.

The reduction in the l/s ratio caused by superplasticizers is much greater when ordinary Portland cement is used as the binder; under these circumstances, the polycarboxylate admixtures PC1 and PC2 are the compounds that reduce the l/s ratio most significantly, from 0.42 to 0.31. However, despite its excellent performance as a water reducing agent, the presence of PC2 in mortars lowers mechanical strengths due to the high entrained air content generated.

The admixtures used do not affect the flowability of waterglass-activated slag pastes, although when slag is NaOH activated, the naphthalene-base admixture has a clearly visible superplasticizing effect with both Na_2O concentrations tested. These results show that the superplasticizers used, with the exception of naphthalene, have no impact on the fluidity of activated slag pastes.

The explanation for these results lies in the modification of the chemical structure of the admixtures in high alkaline media, such as the two alkaline solutions used [19]. This assertion can be made on the basis of the FTIR results obtained for these admixtures in different alkaline media, namely, $Ca(OH)_2$ (pH 12.40), NaOH (pH 13.6) and waterglass (pH 13.40).

When these admixtures are dissolved in a $Ca(OH)_2$ solution, their chemical formulations show no structural alteration, which justifies their good performance as superplasticizers when the binder is Portland cement.

In high alkaline media (pH 13.0–13.6) however, these admixtures undergo structural change. In PC1 and PC2, the alkaline hydrolysis of the ester groups gives rise to carboxylate salts and the respective ethers [20,21]. This may be deduced from the disappearance, in infrared spectra, of the band at 1730 cm^{-1} corresponding to the C=O groups in the esters and the appearance of two bands close to 1575 and 1418 cm^{-1} that correspond to carboxylate groups (COO^-; Fig. 5a). As a result, the main chain, which contains the carboxylate groups, is adsorbed on to the surface of the slag particles, while the lateral chains, comprising ethers, break away from the main chain. Consequently, the steric hindrance that these ether chains impose on ordinary Portland cement particles is practically nonexistent in the case of AAS particles; the result is that the flowability is not improved in pastes with no admixtures, and the superplasticizer properties of these admixtures practically disappear. Fig. 6a shows the reaction scheme for polycarboxylate admixtures.

Similarly, these high alkaline media modify the structure of the vinyl copolymer. As can be seen in the respective infrared spectra (see Fig. 5b), the amine contained in the sulphonic group and its corresponding carboxylate salt are formed as a result of the alkaline hydrolysis of the amide that forms a part of this admixture [20]. The process described for this admixture is outlined in Fig. 6b. The bands close to 1580 and 1415 cm^{-1} observed in the infrared spectrum are attributed to carboxylate groups, and the bands at 3440, 3385 and 1601 cm^{-1} correspond to the vibrations produced by the NH_2 groups in the amine formed. This alteration in the admixture explains the loss of its superplasticizing properties.

The melamine-based admixture undergoes slight alterations in its formulation when kept in high alkaline media. These modifications are more acute when the alkali activator is waterglass. This explains why this compound does not improve the workability of waterglass-activated slag pastes when the alkaline activator is NaOH. However, the melamine-based admixture improves the flowability of the pastes during the first 10 min, which nonetheless declines drastically thereafter. The naphthalene-based admixture, on the contrary, is very stable in the NaOH solution, with no changes observed in its formulation. This is the reason why the superplasticizing effect of this admixture compares favourably to its performance in Portland cement. When the alkaline solution is waterglass however, this compound, like the vinyl copolymer and the melamine-based, undergoes alterations in the SO_3 groups. While this development has

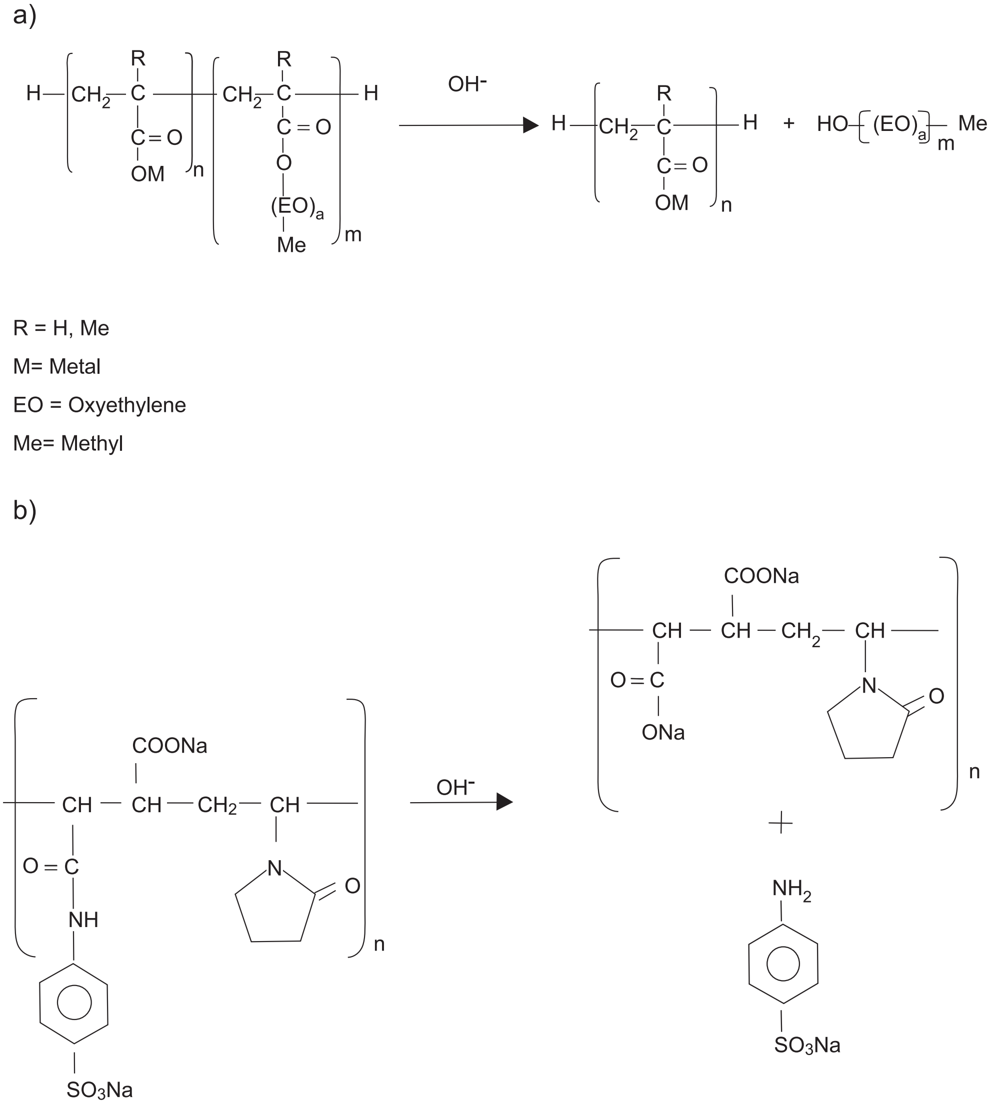


Fig. 6. Alkaline hydrolysis of polycarboxylate and vinyl copolymer superplasticizer admixtures.

yet to be justified, it may be the reason why none of these three admixtures behave as a superplastificizer when the alkaline activator used is waterglass.

Finally, the polypropylenglycol admixture is stable in both alkaline solutions, although since it is not a superplasticizer, it is not able to improve paste flowability, on the grounds of the slump tests performed. However, it does reduce the l/s ratio of waterglass-activated slag mortars.

The instability of these admixtures in the alkaline solutions used as activators affects the setting times of alkali-activated slag pastes. When the activator solution is waterglass, slight variations are observed in the initial and final setting times of the pastes containing these admixtures compared to slag pastes with no admixtures, confirming their scant or no suplerplasticizing effect. When the slag is NaOH-activated however, the naphthalene-based behaves very differently from the rest of the superplasticizers. This admixture retards the initial set by 2 h, and the final set by up to 4 h when a 4% dosage of Na_2O is used. When the Na_2O concentration is 5%, setting times are increased by 1 h.

5. Conclusions

The effect of superplasticizers and shrinkage-reducing admixtures on alkali-activated slag pastes and mortars differs entirely from the effect on ordinary Portland cement mixes. The reason for this difference in behaviour lies in the modifications that high alkaline media, such as waterglass and NaOH solutions, induce in the chemical structures of some of the admixtures used. The only admixture whose formulation is not altered—when the alkaline solution used is NaOH—is the naphthalene-based, which consequently originates a rise in mechanical strength values, improved workability and retardation of the initial and final sets times compared to slag pastes and mortars with no admixtures. And, it is in this case only that the performance of the admixture is comparable to its superplasticizing effects on Portland cement pastes and mortars.

The high alkaline media used do not alter the formulation of the shrinkage-reducing admixture. This compound reduces the l/s ratio more in waterglass-activated slag mortars with 5% Na_2O than in Portland cement mortars, producing an increase in the mechanical strength of mortar in the early stages but no improvement in paste workability.

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