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Effect of Accelerant Additives in Concrete with Limestone Aggregate in Warm Weather

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Abstract

Additives are chemical compounds that are added to concrete during its manufacture to modify one or more of its properties. The first additive used in modern concrete was the accelerator, intended to shorten the time during which the material is not capable of supporting stress. Traditionally, accelerators have been made from calcium chloride, but today the trend is to use chloride-free additives to prevent reinforcing steel from corroding and thereby make constructions durable. The objective of this study was to evaluate the effects on the concrete of two types of accelerating admixture, using limestone aggregates, under warm sub-humid climate conditions. The applied methodology consisted of the measurement in the laboratory of some of the main properties of concrete in fresh and hardened states, in samples manufactured both with and without accelerators. The results showed that the accelerator without chloride was less effective than the one based on calcium chloride and that without doses of the accelerator, the effectiveness documented by the manufacturers was achieved.

Keywords

Concrete, Additives, Accelerators, Limestone Aggregates, Durability

1. Introduction

Additives are not essential components of the concrete mix, however, they are of great importance when there is a need to modify the characteristics of this material, in such a way, it adapts to the conditions of the project and/or the requirements of the builder. Sometimes, the use of some admixture may be the only means to obtain the required result, for example, to give resistance to the material against freezing and thawing, to accelerate or retard the concrete setting time, or to obtain exceptionally high strengths.

Accelerators were the first type of admixture to be used in the construction of reinforced concrete structures [1]. These additives have been used, mainly, to advance the stripping formwork and its subsequent construction processes, however, in most cases, their use produces an increase in the shrinkage suffered by the concrete when it dries, which must be compensated with effective curing.

According to the theory of concrete technology, the main effects of accelerators on the properties of concrete are to modify its setting time and mechanical strength [2]. The setting is the process of hardening and loss of plasticity of concrete or mortar, produced by the drying and recrystallization of metal hydroxides, resulting from the chemical reaction between water and metal oxides present in the cement clinker. The effect of accelerators on the plastic state of concrete is the reduction of the initial and final setting times. The degree of this reduction varies depending on the amount of accelerator, the temperature of the concrete and the ambient temperature [3].

Once it has finished setting, the concrete begins to form stronger bonds which produce its ability to resist different mechanical stresses [4]. The effect of the accelerator in this hardened state occurs mainly at early ages, and it is reduced later.

Calcium chloride ($CaCl_2$) has historically been the most widely used accelerator, but due to its chlorinated composition, it has drawbacks when used in concrete elements that contain reinforcing steel since it can cause corrosion, for this to happen, the threshold of chloride initiators of corrosion inside the concrete must be exceeded, which is in an interval between 0.2 and 2.0 kg/m^3 of concrete, according to the salinity of the environment [5]. On the other hand, when large amounts of chloride are present, the concrete tends to retain more moisture, which also increases the risk of corrosion. It is important to keep in mind that chlorides can enter the concrete by addition during its manufacture or by exposure in coastal environments [6]. Because of the above, building codes restrict the use of calcium chloride for durability.

Due to the above, in recent decades, there has been a growing interest in the use of chloride-free accelerators, such as calcium nitrite $Ca(NO_2)_2$ [7]. It has been observed that chloride-free accelerators are effective in reducing the setting time, but in some cases, they experience a tendency to reduce strength in the first three weeks. This phenomenon depends mainly on the type of cement and aggregate used [8]. In general, these additives tend to be more expensive than those containing calcium chloride.

This study was conducted in the Yucatan Peninsula, located south of the Tropic of Cancer. The predominant climate in the region is classified as warm sub-humid, with few variations in temperature between the seasons, and with rains in summer [9], this type of climate is common in most of the countries of Central America and the Caribbean, as well as in several countries of the world located at similar latitudes. The average maximum temperature in this region is 36˚C (97˚F) and occurs in May, the average minimum temperature is 16˚C (61˚F) and occurs in January, and the average annual temperature is 26˚C (79 ˚F) [10]. In this type of climate, conditions have been presented that favor the rapid evaporation of water from the mixtures, which in turn causes cracking due to plastic contraction in the concrete, this is of great importance for the control of using additives in concrete [11].

The concrete aggregates used in the study region are the product of crushing limestone rocks formed from calcareous Cenozoic sediments. Its chemical composition is approximately 80% calcium carbonate, 15% magnesium carbonate and small amounts of other compounds (silicon dioxide, iron oxide, sulfates, nitrates, and chlorides) [12]. These aggregates have main characteristics as their high absorption and many fine particles, the foregoing, together with the angular shape of the particles, favors greater water requirements in the mixtures, and therefore of cement. Some studies have proven that mixtures containing fine limestone experience slower setting speed, compared to other concrete studied, this is probably because the reaction induced by calcium carbonate reduces the impact of the chemical additions of the accelerators [13].

The objective of this study was to verify the effect of using two accelerating additives, one with chlorides and the other free of chlorides, on the properties of concrete at early ages, in a context of a warm sub-humid climate.

2. Methodology

The investigation was of an experimental type and consisted of the measurement of some properties of the concrete, in samples manufactured in the laboratory, using different doses of accelerating additives. In the preparation of the concrete, two ratios were used between water and cement measured by weight (W/C), one of them typically used for structural function and the other for reinforcement and confinement of masonry elements.

The dependent variables that were measured in the experiment, according with American Society for Testing and Materials (ASTMs) standards, were the following: Slump (ASTM C143) [14], Volumetric weight of fresh concrete (ASTM C138) [15], Air content (ASTM C231) [16], Initial and final setting times (ASTM C403) [17], and Mechanical resistance to axial compression (ASTM C39) [18].

The independent variables that were controlled in the experiment were the following:

• The W/C ratio took two values: 0.50, with which it was intended to achieve an f'c of approximately 300 kg/cm^2, and 0.70, with which it was intended to achieve an f'c of approximately 200 kg/cm^2.

• Type of accelerating additive, which took two categories: the first was an additive with chlorides (which will be abbreviated as CL) and the second was a chloride-free additive (which will be abbreviated as CLF).

• Dose of the accelerators, which took three values: 0.5%, 1.0% and 1.5%, calculated in relative terms as a percentage of the weight of the cement used in the mixture. These three dosages are within the range recommended by accelerator manufacturers for use in warm weather.

• The curing treatment took two categories: wet, by means of saturation by immersion in a pool, to favor the hydration of the cement (ASTM C192) [19], and natural in the laboratory, trying to imitate the conditions that occur in the real context of many job sites, where the moist curing process or by means of membranes is generally omitted.

• The compressive strength test age took three values: 3, 7 and 28 days. This allowed us to observe the changes in this mechanical property during the period in which most of the cement hydration reactions take place.

Other variables that were measured were: the temperature and relative humidity measured in the laboratory at the time of unloading the concrete from the mixer, to know the climatic context in which the concrete tests were carried out in the fresh state.

The materials used were the following:

• CPC 30R type cement (Compound Portland Cement with a resistance of 30 N/mm^2) that meets the specifications of the Mexican Standard NMX-C-414-ONNCCE [20] and is equivalent to Portland type I (ASTM C150) [21].

• Crushed stone aggregates of typical limestone from the north of the Yucatan Peninsula. **Table 1** shows the main properties measured in the sample of aggregates used in the study.

• Two types of accelerating additives: CLF and CL, which according to their technical data sheets, comply with the ASTM-C 494 [22] standard. By means of a chloride analysis stoichiometry test, it was determined that the number of chlorides in the CL additive was 263.748 g/l.

The concrete samples studied were conducted as follows: for each W/C ratio studied, seven concrete manufacturing processes were carried out, six with an accelerating additive and one without an additive (control sample). For each combination of W/C, type, and dose of accelerator, 18 standard specimens (ASTM C39) were manufactured to carry out the resistance tests, which were divided into two groups randomly, and each of them was assigned one of the two curing treatments. Each of these groups of specimens was divided into three subgroups to randomly assign the strength test ages. In total, 252 concrete specimens were manufactured (126 for each W/C), of which 84 were tested in compression for each age.

Using the ACI-211 [23] concrete mix design method, the amounts of material for concrete manufactured without additive were obtained, which are shown in **Table 2**. From these amounts, the corresponding adjustments were made to add the amounts of additive in the three percentages already mentioned.

Table 1. Properties of the sample of the aggregates used.

Type	Density	LDUW (kg/m^3)	CDUW (kg/m^3)	MAS (mm)	Fineness Modulus	Absorption (%)
Gravel	2.28	1079	1231	19	-	7.05
Sand	2.46	1303	-	-	2.47	3.31

Table 2. Dose of concrete without additive.

Materials (kg)	W/C	
	0.50	0.70
Cement	460.00	328.56
Water	255.74	255.28
Gravel	860.66	870.40
Sand	572.30	690.96

With the data obtained in the tests, an Analysis of Variance (ANOVA) was performed to evaluate the effect of different factors on the variables of interest, by comparing the means of the samples that resulted from the different levels of the factors. To do this analysis, the variance between the means of the groups and the variance within the groups were compared to determine if the groups can be considered part of a large population or separate populations with different characteristics.

The influence of the two factors: type and dose of accelerator were tested, in the variables initial and final setting times, and concrete resistance to compression. The curing type factor was also tested in the strength of concrete made with the CLF admixture, the most currently recommended.

The Fisher-Snedecor F distribution was used with k-1 degrees of freedom between groups and n-k within groups, where k is the number of samples and n is the total number of observations. The F statistic is defined as the ratio of the mean squares between groups and within groups. The root means squares are defined as the sum of squares divided by the degree of freedom. The sum of squares is calculated as the sum of the squares of the differences from the mean [24].

To reject the null hypothesis, which establishes that there is no difference between the means of the samples, a minimum level of significance of 0.05 was defined, in such a way that probabilities equal to or less than this are interpreted as the factor causing significant differences in the means of the samples studied.

3. Results

3.1. Climatic Context

The manufacture of the mixtures with W/C of 0.50 was carried out during the summer and autumn (from August 28 to October 9), while the manufacturing with the W/C of 0.70 was carried out during autumn (from October 23 to November 16). The measurements of temperature and humidity taken in the laboratory are presented in **Table 3**. In general terms, little variation was observed in these climatic parameters during the manufacture of the mixtures.

3.2. Fresh Concrete Properties

Figure 1 and **Figure 2** show the effect of the accelerating additives studied on the slump of the mixtures prepared with W/C of 0.50 and 0.70, respectively. In both cases, the fluidity of the mixture always decreased as the amount of accelerator increased. The greatest influence occurred for the W/C of 0.50 and a dose of 1.5% of additive, a mixture in which a decrease in slump of 100% was measured, compared to the control sample. For this same dose, both for the concrete with W/C of 0.50 and accelerator CL, and for the two concretes with W/C of 0.70, a maximum decrease in slump of around 60% was observed.

Table 3. Climatic parameters during the preparation of the mixtures.

Climatic Parameters	Mean (˚C)	Standard Deviation (˚C)
	W/C 0.50	
Temperature	31.50	1.13
Humidity	88.17	2.88
	W/C 0.70	
Temperature	32.33	0.49
Humidity	90.50	1.51

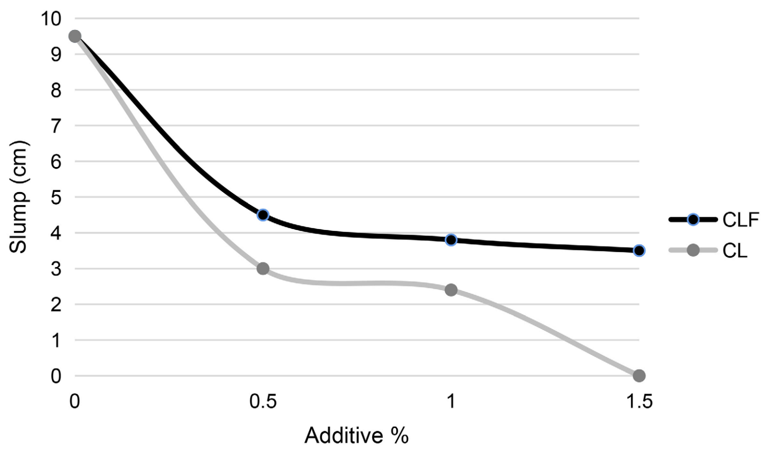

Figure 1. Slump of fresh concrete with W/C of 0.50.

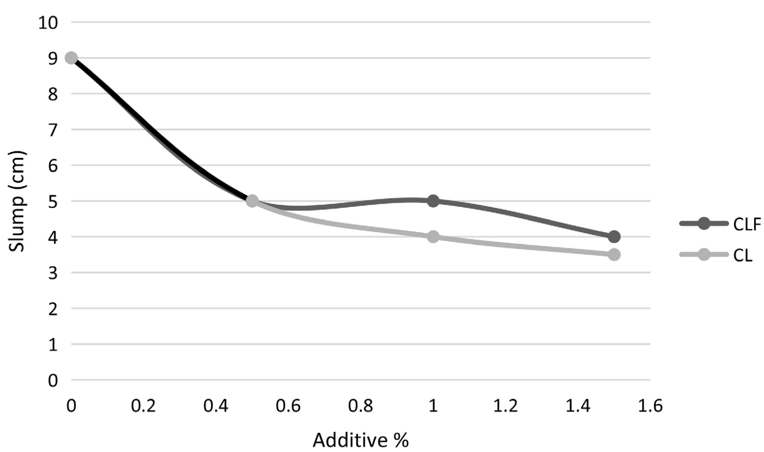

Figure 2. Slump of fresh concrete with W/C of 0.70.

Regarding the air content and the volumetric weight of fresh concrete, in Table 4 and Table 5, these properties were not greatly affected using accelerators. Compare to the values measured in the control samples, the mixtures with additive had maximum differences in air content of 0.3% and 0.2% (in absolute terms) for the W/C of 0.50 and 0.70, respectively, as well as maximum differences in the volumetric weight of 31 and 17 kg/m^3 for the W/C of 0.50 and 0.70.

3.3. Concrete Setting Time

The initial setting times that were measured in the concretes without accelerator were 182 and 178 minutes for the W/C of 0.50 and 0.70, respectively. While the final setting times that were measured in the same concrete were 286 (182 + 104) and 321 (178 + 143), respectively. Figure 3 and Figure 4 show the initial and final setting times that were measured in the concretes with accelerators, for W/C of 0.50 and 0.70, respectively. The times measured in the mixtures without accelerator are also marked with solid lines. In the figures, it is consistently observed that the values are below the times measured in the control samples, measuring for the initial setting greater differences for the W/C of 0.50, and for the final setting, greater differences for the W/C of 0.70.

Table 4. Air content and volumetric weight of fresh concrete with W/C of 0.50.

Additive	Dose (%)	Air Content (%)	Volumetric Weight (kg/m^3)
Control	0.0	2.3	2153.75
CLF	0.5	2.4	2170.78
	1.0	2.5	2170.79
	1.5	2.4	2156.58
CL	0.5	2.4	2170.72
	1.0	2.5	2180.74
	1.5	2.6	2184.87

Table 5. Air content and volumetric weight of fresh concrete with W/C of 0.70.

Additive	Dose (%)	Air Content (%)	Volumetric Weight (kg/m^3)
Control	0.0	2.7	2170.79
CLF	0.5	2.6	2153.75
	1.0	2.8	2156.58
	1.5	2.8	2156.58
CL	0.5	2.6	2153.75
	1.0	2.7	2153.75
	1.5	2.9	2153.75

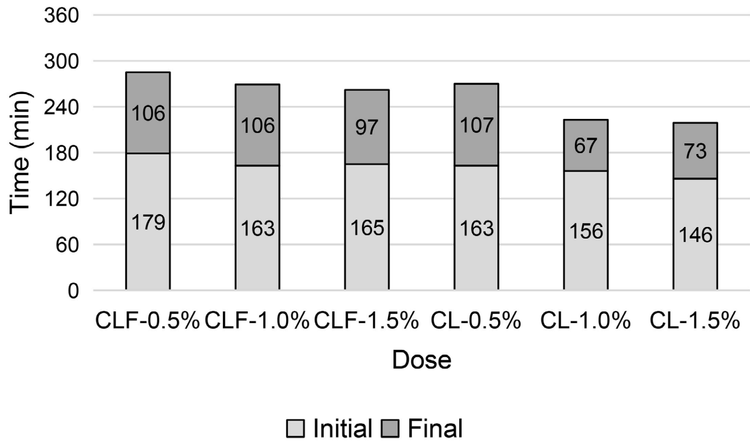

Figure 3. Initial and final setting times of concrete with accelerator and W/C of 0.50.

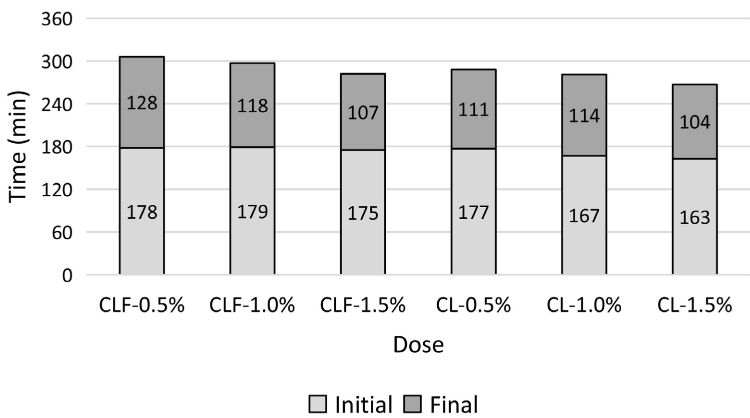

Figure 4. Initial and final setting times of concrete with accelerator and W/C of 0.70.

3.4. Compressive Strength

Table 6 and Table 7 show the results of the compressive strength tests of concrete with W/C of 0.50 and 0.70, respectively. In these tables, it can be observed that the resistance increased systematically as the dose of additive increased, for the three ages, and that the increases were higher for wet curing concretes manufactured with the CL accelerator.

These results have been plotted in Figure 5 and Figure 6, for concrete with wet curing. For the concretes with W/C of 0.50, which are rich in cement, it can be observed that for the highest dose of accelerator there was an appreciable increase in resistance, while for the concretes with W/C of 0.70, a mixture less rich in cement, there was a notable increase in resistance in all doses of the accelerator.

The Analysis of Variance (ANOVA) test was performed to find out if the effect of the accelerator caused differences in the initial and final setting times. The method consisted of testing if the means of the concrete samples' setting times varied when the type of accelerator was changed, the measured data was divided into two samples, one containing all the concrete manufactured with CLF accelerator and the other containing all the concrete manufactured with CL accelerator, for each concrete samples were grouped by the three additives doses and the two W/C.

Table 6. Compressive strength (kg/cm^2) for concrete with W/C of 0.50.

Additive	Age (days)	Curing Method	Dose			
			0%	0.5%	1.0%	1.5%
CLF	3	Wet	266	274	273	281
		Natural	266	283	278	292
	7	Wet	289	289	291	316
		Natural	301	321	310	329
	28	Wet	320	321	327	339
		Natural	335	340	336	344
CL	3	Wet	266	266	271	294
		Natural	267	268	278	288
	7	Wet	289	288	298	306
		Natural	301	319	314	339
	28	Wet	320	320	324	336
		Natural	335	340	335	353

Table 7. Compressive strength (kg/cm^2) for concrete with W/C of 0.70.

Additive	Age (days)	Curing Method	Dose			
			0%	0.5%	1.0%	1.5%
CLF	3	Wet	189	214	213	216
		Natural	207	214	215	225
	7	Wet	228	235	237	240
		Natural	242	256	257	258
	28	Wet	258	288	282	284
		Natural	269	303	301	302
CL	3	Wet	189	192	213	226
		Natural	207	201	238	233
	7	Wet	228	236	245	251
		Natural	207	201	238	233
	28	Wet	258	295	286	300
		Natural	269	314	315	324

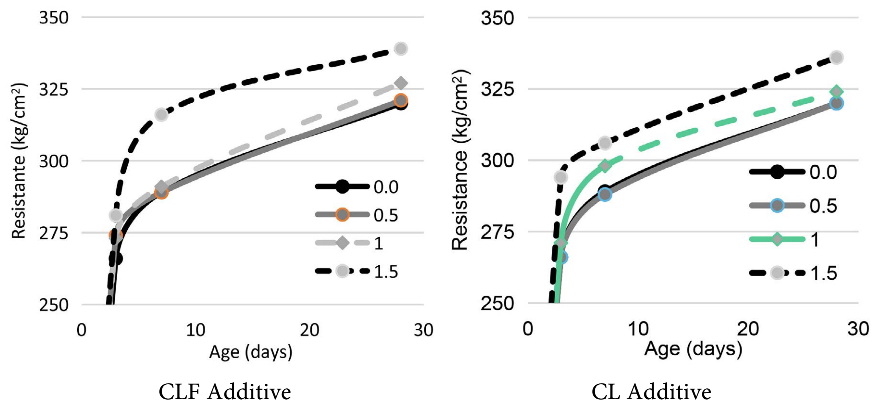

Figure 5. Compressive strength, W/C of 0.50 with wet curing.

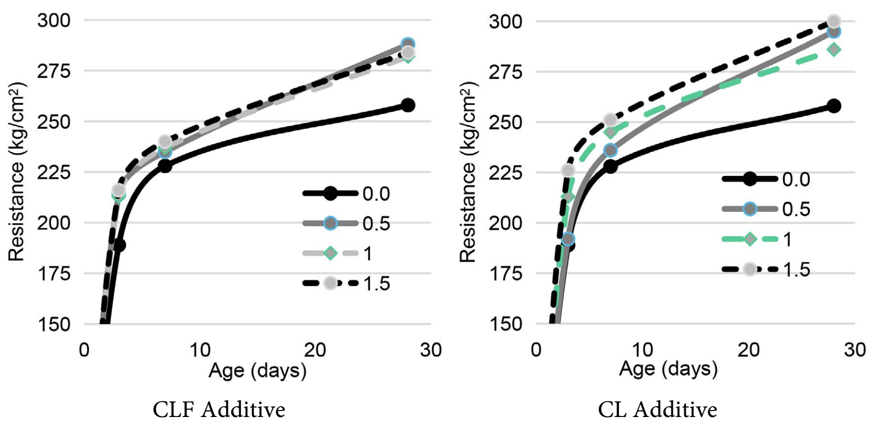

Figure 6. Compressive strength, W/C of 0.70 with wet curing.

Similarly, to test whether the means of the concrete samples' setting times varied when the doses of the accelerators changed, the data was divided into three samples, each containing the concretes manufactured with each dose, in each sample the concretes manufactured with the two types of accelerators and the two W/C were grouped. The results of these analyzes are presented in **Table 8**, in which it can be observed that, statistically, the means of the initial and final concrete samples setting times were marginally different when the type of additive was changed, with probabilities close to the value of 0.05 (0.057 and 0.053). As can be seen in **Figure 3** and **Figure 4**, the CL additive was more effective, that is, the concrete reached shorter setting times.

In the same way, to statistically test whether the effect of the accelerator caused significant differences in the strength of the concrete with moist curing, an analysis of variance tests was carried out for each of the three strength verification ages. The groups were formed in a similar way to those explained for the variables of concrete setting times, that is, when the effect of the type of accelerator was tested, the data was divided into two samples, one containing all the concretes manufactured with CLF additive and the other containing all the concretes manufactured with CL additive, and when the effect of the dose was tested, the data was divided into three samples, each containing the concrete manufactured with each dose.

The results of these analyzes are presented in Table 9, in which, statistically, the means of the resistances of the various groups were not significantly different, since all had probabilities above 0.05. It is important to note that these analyzes group the concretes of the two W/C in the same sample, making a generalization of the results, giving consequently that the differences observed in Figure 5 and Figure 6 are not confirmed in general.

Table 8. Analysis of variance of the setting times, taking as factors the type and dose of the accelerator.

Variable	Factor		Mean	F	Significance
Initial Setting Time (min)	Additive	CLF	173	4.62	0.057*
		CL	162		
Final Setting Time (min)	Additive	CLF	287	4.79	0.053*
		CL	258		
Initial Setting Time (min)	Dose	0.5	174	1.52	0.270
		1.0	166		
		1.5	162		
Final Setting Time (min)	Dose	0.5	287	0.94	0.427
		1.0	267		
		1.5	263		

Table 9. Analysis of variance of the resistance of concrete with moist curing, taking as factors the type and dose of the accelerator.

Variable	Factor		Mean	F	Significance
Resistance at 3 Days	Additive	CLF	244	0.00	1.000
		CL	244		
Resistance at 3 Days	Dose	0.5	237	0.23	0.795
		1.0	240		
		1.5	254		
Resistance at 7 Days	Additive	CLF	268	0.20	0.890
		CL	270		
Resistance at 7 Days	Dose	0.5	264	0.21	0.814
		1.0	265		
		1.5	278		
Resistance at 28 Days	Additive	CLF	307	0.02	0.905
		CL	309		
Resistance at 28 Days	Dose	0.5	307	0.31	0.736
		1.0	301		
		1.5	314		

Finally, to statistically test whether the effect of the type of curing caused significant differences in the strength at 28 days of concrete made with CLF accelerator, Analysis of Variance (ANOVA) test was performed. For this, the data was divided into two samples, one containing the concrete manufactured with CLF accelerator and moist curing, and another containing all the concrete manufactured with the same additive and cured in the environment, in each sample the concretes of the three doses and the two W/C were grouped. The results of these analyzes are presented in **Table 10**, in which no significant differences can be seen.

4. Discussion

In all cases studied, a decrease in slump was obtained as the dose of additive increased. For almost all combinations, the decrease in slump was in a range between 4 and 6 cm, the exception being the mixtures with W/C of 0.50 and accelerator CL, in which decreases in slump were measured in a range between 6 and 9 cm [25] reported that the use of chloride-free accelerators did not have a notable effect on the fluidity of fresh concrete, which was not confirmed in the present study.

The technical sheets of the manufacturers of the two accelerators used in this study recommend a dose of 1.0% for hot weather, to obtain a 50% decrease in the initial and final setting times. This reduction was not achieved in any mixture. The greatest reductions in the setting times were obtained with the W/C of 0.50, accelerator CL and a dose of 1.5%, for this combination, there was a decrease of 20% in the initial setting and 23% in the final setting. The previous behavior could be attributed to the use of limestone aggregates, which, as mentioned by [13], concretes with this type of stone experience lower setting speeds compared to other types of aggregate, probably due to the induced reaction of carbonate calcic. For their part [26] found that chloride-free accelerators have a lower performance as an accelerating agent in the setting reaction compared to those containing chlorides, which was observed in the present study.

On the other hand, the acceleration in the strength gain of concrete is key to advancing the formwork stripping time of the construction elements and their commissioning. The most critical case corresponds to the flexural elements, which can generally be dismantled after 14 days and put into service after 28 days. With the measured data of the compressive strength, in **Table 11** and **Table 12** the number of days in which these two events could occur is presented, using interpolations. For example, for the concrete with W/C of 0.50, accelerator CL and a dose of 1.5%, after 5 days the resistance was obtained that corresponded to 14 days in the concrete without accelerator, and at 16 days the resistance corresponded to 28 days. In general, formwork stripping and commissioning could be carried out in approximately half the time for the 1.5% doses.

In relation to the curing treatment, in this study, as in others carried out in the same region, wet curing did not increase the resistance of the concrete with respect to natural curing. Reference [27] has attributed this effect to the high

Table 10. Analysis of variance of the resistance of concrete with moist curing and NCL additive, taking the type of curing as a factor.

Variable	Factor		Mean	F	Significance
Resistance at 28 Days	Curing Method	Wet	307	1.13	0.313
		Natural	321		

Table 11. Times (days) in which the concrete studied with W/C of 0.50 could be stripped and put into service.

Event	Accelerant	Dose			
		0.0%	0.5%	1.0%	1.5%
Formwork Stripping	CLF	14	14	12	5
	CL	14	14	9	5
Commissioning	CLF	28	28	24	11
	CL	28	28	25	16

Table 12. Times (days) in which the concrete studied could be removed and put into service with W/C of 0.70.

Event	Accelerant	Dose			
		0.0%	0.5%	1.0%	1.5%
Formwork Stripping	CLF	14	8	8	7
	CL	14	7	6	4
Commissioning	CLF	28	17	16	16
	CL	28	15	15	10

porosity of the aggregates in the region, which could produce internal curing, have also attributed the lower strength of wet-cured concrete to the effect of semi-saturation of the specimen at the time of performing the destructive compression tests. According to the results of this study, the use of accelerators did not cause a synergistic effect with moist curing in the phenomenon of maturity in concrete.

5. Conclusions

In the context of the warm region climate:

The two types of accelerators decreased the slump of the mixture, the effect being greater in the accelerator with Chlorides (CLs).

The two types of accelerators decreased the initial and final setting times, with a greater effect on the concrete final setting. However, the reductions contained in the technical sheets of the products were not reached.

The compressive strength at early ages increased with the use of the two accelerators, being more notable for the mixture rich in cement with a W/C of 0.50.

With the data obtained, the significant difference attributable to the accelerators in the setting times was statistically tested, but, in general, the difference in resistance was not confirmed, since the changes in this variable were different for the two W/C.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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