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Development of two analytical methods for determination of water-soluble chlorides and sulfates in the conservation of concrete heritage buildings

Alberto H. Oroza^{a,*}, Fabio Rojas Pimentel^b, Esmérida Pérez Parra^b,
Luz M. Delgado León^b, Yilena García Amorós^b

^a *Projection, Architecture and City Planning RESTAURA, Cuba No. 316, La Habana 10100, Cuba*

^b *Laboratory of waters and wastewaters "José Isaac del Corral", Extension of Vía Blanca Avenue, La Habana 11000, Cuba*

Abstract

The corrosiveness and pollution of the atmosphere in the Old City of Havana – Cuba, have come to be over the last decades a growing concern regarding the durability and the aesthetics of heritage and historic concrete buildings. The chlorides and sulfates present in the air are capable to deposit over the concrete and have a direct impact on the deterioration of buildings. Chloride anions induce localized corrosion on the reinforced steel, while sulfates produce severe cracking in concrete. Based on Cuban climate parameters, it is important to develop an analytical method suitable to quantify the amount of these substances in the mass of concrete. The aim of this study was to develop two analytical procedures for the quantification of water-soluble chlorides and sulfates in hardened concrete in used buildings. Basic evaluation of the precision, accuracy and uncertainty of the proposed methods was done. In order to determine the content of water-soluble chlorides, mercury(II) nitrate was used as a titrant, and mix of bromophenol/diphenylcarbazone as an indicator. The sulfates content was determined gravimetrically. The study concluded that both methods, as well as the statistical analysis performed are satisfactory, allowing the quantification of the amount of water-soluble chlorides and sulfates in concrete heritage buildings.

Keywords: Heritage buildings; water-soluble chlorides; sulfates; precision; accuracy; uncertainty

1. Introduction

Reinforced concrete has been for decades the most used material for the construction of buildings. It has the advantage of offering high structural capacity, and capable of presenting high performance for durability (Howland 2012). Despite these properties, concrete has the disadvantage of being vulnerable to climate changes, corrosive environments and the presence of air pollutants such as chlorides (Cl^-) and sulfates (SO_4^{2-}) (Meira et al. 2008; Carvajal et al. 2012; Kumar and Imam 2013).

When Cl^- anions reach a critical concentration around the rebar (usually above 0.05% mass of concrete), they are able to damage the passive layer on steel and initiate corrosion (Angst 2011; Silva 2013). In the case of sulfate anions, they can react with the alkalis of the cement and weaken structural elements of buildings due to

* Corresponding author. E-mail address: alberto@diagnosticos.proyectos.ohc.cu

the occurrence of three main chemical reactions within the hardened cement paste: formation of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), recrystallization of delayed ettringite ($\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$), and decalcification of hydrated calcium silicate (CSH) of the cement paste (Diamond 1996; Collepardi 2003).

Since the beginning of the current century, an intensive program of rehabilitation of the heritage and historic buildings in Old Havana City of Cuba has been carried out. Most damages appearing on buildings are revealed as loosing of surface coatings, cracking of the structural elements, corrosion of reinforced steel and blackening of the facades (Brimblecombe and Grossi 2005; Grossi and Brimblecombe 2008). National scientific studies had published mean Cl^- and SO_4^{2-} deposition within the first kilometer inland of 246 $\text{mg}/\text{m}^2\text{d}$ and 33 $\text{mg}/\text{m}^2\text{d}$, respectively, for times of wetness according to ISO 9223 equal to 4966 h/a (Castañeda et al. 2013).

This high level of corrosivity of the atmosphere is significantly affecting the reinforced concrete heritage buildings located within the first kilometer from the northern shoreline. The geography of Cuba describes a long and narrow island, exposed to the corrosive influence of sea spray during most time of the year, because of about 6000 km of shoreline (Corvo et al. 1995).

The damages occurred in the buildings caused a worsening of the aesthetic and architectural values, and an increase of the costs of repair and maintenance in order to preserve the construction heritage of the city. Present Cuban construction regulations establish and debate several aspects of reinforced concrete buildings only for the new-built structures (NC 120; NC 250). As a consequence, heritage and historical buildings remain unprotected and with limited knowledge on the magnitude of damages present in their structural elements. This leads to the creation of new strategies to extend the lifetime of the heritage and historical concrete buildings. A first step toward this purpose is to develop chemical methods capable of quantifying the contents of Cl^- and SO_4^{2-} in the concrete. International organizations and researchers had provided procedures for sampling and analyzing of water-soluble Cl^- in concrete (Carvajal et al. 2010; ASTM C1218/C1218M-99; ASTM D516-11; BS EN 196-2:2013).

The objective of this paper is to present two chemical procedures and statistical results obtained for 110 samples of concrete, in which the Cl^- and SO_4^{2-} anions were determined. Precision, accuracy and uncertainty of the developed methods were also evaluated. The coefficients of variation (CV) obtained for both methods were less than 15%. Considering the possible interferences in the analysis of sulfates by the gravimetric method, the silica content in the samples was studied, as well as the uncertainty associated with the analysis.

2. Experimental

2.1. Analytical method

The structural elements selected for the study were columns and roof slabs fully exposed to the atmosphere with no protection from rain, pollutants and marine aerosol. The extraction of samples was performed using a chisel and hammer from the surface of the concrete up to a depth of 1 cm. The weights of the samples were between 100 and 150 grams. Storage and identification were performed immediately after collection using Ziploc's nylon bags. Each sample was pulverized up to particles size below 1 mm. The following steps summarize the chemical testing performed on the concrete samples:

(1) Obtaining the aqueous extract: The samples were dried at 60°C for 1 hour in a heater. After reaching the room temperature in a desiccator, the samples were weighed to 2 ± 0.01 g. Each test portion was decanted to the beaker of 250 ml and then 100 ml of deionized type III water (BS EN ISO 3696:1995) was added; the sample was then heated to soft boiling during 15 minutes. The residue of the test portion was filtered with filter paper Whatman No. 40, receiving the filtrate in 250 ml volumetric flask.

(2) Determination of water-soluble chlorides: An aliquot was taken from the 250 ml volumetric flask and placed in the beaker. The portion of 0.5 ml of bromophenol and diphenylcarbazone solution was added. After that, the pH was adjusted to 2.5 ± 0.1 , and titration with $\text{Hg}(\text{NO}_3)_2$ with concentration of 0.0141 N (APHA 2005a) was conducted.

(3) Determination of SO_4^{2-} in water: From the aqueous extract, it was taken an aliquot for precipitation in a 250 ml volumetric flask as BaSO_4 in a solution of HCl. The obtained precipitate was filtered using a filter paper with pore size between 4 and 6 μm . The filter paper with the precipitate was incinerated, and BaSO_4 remained was weighed (APHA 2005b). This determination might present some interference if the cement from the concrete contains an excess above 25 mg/l of SiO_2 .

(4) Gravimetric determination of SiO_2 in the aqueous extract of concretes: it was necessary to investigate an independent sample in conditions similar to those for the anions. The aliquot taken from the aqueous extract was evaporated to dryness in a hot plate. As the next step, 2 ml of concentrated HCl was added. Once again, the sample was evaporated to dryness and kept on a hot plate at the temperature $150 \pm 15^\circ\text{C}$ for two hours. After the SiO_2 was dehydrated, separation by filtration and incineration of the filter paper with the SiO_2 in a platinum crucible was performed. Purity test was also performed using HF and H_2SO_4 .

After completion of the chemical analysis of the concrete samples, it was necessary to establish the statistical methods to be used for estimation of precision, accuracy and uncertainty of the results:

(1) Estimation of the precision for the determination of Cl^- and SO_4^{2-} anions in the aqueous extract of concrete: Two methods for estimation of the precision were used. One method was the repeatability using pairs of replicated values from internal quality control, while the other method used n replicates of the same sample (Magnusson et al. 2012).

(2) Estimation of the accuracy for the determination of Cl^- and SO_4^{2-} anions in the aqueous extract of concrete: The most expeditious way to estimate the accuracy is the use of Certified Reference Materials. In the absence of these materials, the recovered sample with addition of known amount of both anions of interest into the solution was used (Magnusson et al. 2012).

(3) Estimation of the uncertainty for the determination of Cl^- and SO_4^{2-} anions: The calculation of the combined uncertainty of the components of the internal laboratory reproducibility and systematic error is used. For the calculation of the systematic error the added-recovered method was applied. The expanded uncertainty was determined for 95% confidence level.

2.2. Results

2.2.1. Estimating precision

As the first step, 110 samples were delivered to the laboratory, from which 23 were doubled according to the internal quality control. These 23 samples were used to calculate precision in conditions of repeatability by the method of standard deviation range (Magnusson et al. 2012). In Tables 1 and 2 the data for both anions, as well as calculations to obtain the precision as coefficient of variation are reported (Eq. 1-2).

The use of the factor 1.128 allows the calculation of the standard deviation (S) or CV when duplicate samples ($n=2$) are available, as reported in Appendix 8 from (Magnusson et al. 2012). The precision obtained, as a coefficient of variation (Eq. 1) in the determination of Cl^- by mercurimetry is satisfactory. To expand the evaluation of the precision, two samples were selected whose Cl^- and SO_4^{2-} anions contents were higher than those reported in Tables 1 and 2, respectively. In this case, 10 replicates of each sample were performed under pure repeatability. Tables 3 and 4 report the results.

The results obtained as the standard deviation and by the use of the statistical Shapiro-Wilk test (W) confirm that in both cases the group of 10 results meets a normal distribution. It can be noted that the estimation of precision in terms of pure repeatability is better than using pairs of values.

2.2.2. Estimating accuracy

For the determination of accuracy, the added-recovered method was used (Magnusson et al. 2012). For this purpose, 1000 mg/l of sodium chloride (NaCl) solution with 99.5% purity and sodium sulfate (Na_2SO_4) with 99.5% purity were prepared. Three additional replicates of the samples for Cl^- and SO_4^{2-} were also prepared. Three test portions deposited in the corresponding beakers were added in the amount of 3, 5 and 7 mg of Cl^- and 200, 400 and 600 mg of SO_4^{2-} ; solutions of 1000 mg/l were prepared. The results for 9 replicates for each anion are presented in the Tables 5 and 6. The average recovered was 103.7% for the Cl^- anion and 96.9% for SO_4^{2-} . In this range, both results seem acceptable.

Table 1. Precision of the results of water-soluble chlorides in concrete. Range from 0.01 to 0.18% m/m.

Sample	X ₁	X ₂	X _{mean} =(X ₁ +X ₂)/2	d=X ₁ -X ₂	r% ₀ = d /X _{mean} ·100
1	0.02	0.01	0.015	0.01	66.66
2	0.01	0.01	0.01	0.00	0.00
3	0.01	0.02	0.015	-0.01	66.66
4	0.01	0.01	0.01	0	0
5	0.01	0.02	0.015	-0.01	66.66
6	0.02	0.03	0.025	-0.01	40.00
7	0.01	0.02	0.015	-0.01	66.66
8	0.01	0.01	0.01	0.00	0.00
9	0.12	0.12	0.12	0.00	0.00
10	0.07	0.07	0.07	0.00	0.00
11	0.04	0.04	0.04	0.00	0.00
12	0.04	0.04	0.04	0.00	0.00
13	0.13	0.13	0.13	0.00	0.00
14	0.09	0.09	0.09	0.00	0.00
15	0.14	0.14	0.14	0.00	0.00
16	0.18	0.18	0.18	0.00	0.00
17	0.07	0.07	0.07	0.00	0.00
18	0.08	0.07	0.075	0.01	13.33
19	0.08	0.08	0.08	0.00	0.00
20	0.07	0.08	0.075	-0.01	13.33
21	0.05	0.05	0.05	0.00	0.00
22	0.14	0.14	0.14	0.00	0.00
23	0.13	0.13	0.13	0.00	0.00
			Mean = 0.0671	Mid-range (%) = 14.4913	

$$CV(\%) = \text{mid-range} / 1.128 = 12.85\% \quad (1)$$

Table 2. Precision of the results of water-soluble sulfates in concrete by gravimetry. Range from 100-250 mg/l.

Sample	X ₁	X ₂	X _{mean} =(X ₁ +X ₂)/2	d=X ₁ -X ₂	r% ₀ = d /X _{mean} ·100
1	121	147	134	-26	19.40
2	127	152	140	-25	17.86
3	133	157	145	-24	16.55
4	139	162	150	-23	15.33
5	148	122	135	26	19.26
6	151	128	140	23	16.43
7	156	134	145	22	15.17
8	161	140	150	21	14.00
9	168	195	182	-27	14.84
10	194	169	182	25	13.74
11	174	201	188	-27	14.36
12	180	206	193	-26	13.47
13	186	211	198	-25	12.63
14	198	214	206	-16	7.77
15	200	175	188	25	13.30
16	205	181	193	24	12.44
17	210	187	198	23	11.62
18	220	227	224	-7	3.12
19	226	230	228	-4	1.75
20	231	219	225	12	5.33
21	237	242	240	-5	2.08
22	243	249	246	-6	2.44
23	250	236	243	14	5.76
			Mean = 185.8	Mid-range (%) = 11.6804	

$$CV(\%) = \text{mid-range} / 1.128 = 10.35\% \quad (2)$$

Table 3. Calculation of the precision of the results of Cl⁻ mercurimetric for 10 replicates of the same sample.

No. replicates	1	2	3	4	5	6	7	8	9	10	Σ	S	X _{mean}	CV (%)	W
Cl ⁻ (%)	0.3311	0.3323	0.3273	0.3323	0.3423	0.3361	0.3373	0.3386	0.3311	0.3336	3.342	0.04379	0.3342	1.31	0.967

Table 4. Calculation of the precision of the results of gravimetric SO_4^{2-} for 10 replicates of the same sample..

No. replicates	1	2	3	4	5	6	7	8	9	10	Σ	S	X_{mean}	CV (%)	W
SO_4^{2-} (mg/L)	378.67	324.34	395.14	355.33	347.39	363.85	319.40	322.69	306.23	316.00	3429,04	29.8059	342.904	8.69	0.929

Table 5. Results of the estimation of accuracy for the determination of Cl⁻ in concrete, by the added-recovered method.

Replicates	Cl (%)		Recovered (%)	Relative error (%)
	Added	Recovered		
1	0.15	0.1656	110.0	
2	0.15	0.1656	110.0	
3	0.15	0.1581	105.0	
X_{mean}	0.15	0.1631	108.7	8.70
1	0.25	0.2455	98.2	
2	0.25	0.2455	98.2	
3	0.25	0.2505	100.2	
X_{mean}	0.25	0.2472	98.9	1.16
1	0.35	0.3616	103.3	
2	0.35	0.3690	105.4	
3	0.35	0.3565	101.9	
X_{mean}	0.35	0.3624	103.5	3.50

Table 6. Results of the estimation of accuracy for the determination of SO_4^{2-} in concrete, by the added-recovered method.

Replicates	SO_4^{2-} (mg/L)		Recovered (%)	Relative error (%)
	Added	Recovered		
1	200.00	170.78	85.4	
2	200.00	197.10	98.6	
3	200.00	225.11	112.6	
X_{mean}	200.00	197.66	98.8	1.17
1	400.00	383.16	95.8	
2	400.00	371.64	92.9	
3	400.00	366.70	92.7	
X_{mean}	400.00	373.83	93.5	6.54
1	600.00	569.20	94.9	
2	600.00	590.61	98.4	
3	600.00	613.76	102.3	
X_{mean}	600.00	591.19	98.5	1.47

2.2.3. Determination of SiO_2 content in concrete

The silica content was determined in 23 samples used for determination of Cl⁻ and SO_4^{2-} . The values obtained were between 0.03% and 0.37% with an average of 0.19% SiO_2 . This is an excessive interference of gravimetric determination of SO_4^{2-} . Table 7 presents the equivalence between the minimum, maximum and average SiO_2 (%), related to the corresponding SO_4^{2-} content in mg/l.

Table 7. Equivalence between the content of SiO_2 (%) and SO_4^{2-} (mg/l) soluble in concrete.

SiO_2 (%)	SO_4^{2-} (mg/l)
0.03	5
0.19	31
0.37	61

The systematic error due to the excess produced by the SiO_2 , depends on the content of SiO_2 and the tolerance established for the specific use. On the other hand, lacking from the separation of the silica leads to saving of time and resources in the execution of the determination of sulfates.

2.2.4. Estimation of uncertainty

For the estimation of the uncertainty in the measurement of water-soluble chlorides in concrete, the repeatability within the internal reproducibility of the laboratory (R_w) was taken into consideration. It was obtained from the standard deviation range from Table 1. In this case, $S = 0.0030\%$ and the relative error is 4.5%. The variability of the concentration of the titrant $\text{Hg}(\text{NO}_3)_2$ solution was included from time to time to this source of variation. This has been estimated on the base of the laboratory experience as $S = 0.25\%$. These two components are expressed as the combined uncertainty of repeatability within the laboratory as follows:

$$u(c) = [4.5^2 + 0.25^2]^{1/2} \approx 4.5\% \quad (3)$$

The uncertainty corresponding to the systematic error, and evaluated by the added-recovered method, was calculated as follows: three test portions of the same sample were supplemented with different equivalents. The concentrations of the solutions were 0.15, 0.25 and 0.35% of Cl⁻. The preparation of samples was done using a burette with capacity of 10 ± 0.02 ml. In Table 5 the percentage of recovered is reported. The average recovered was 103.7%. The value of $u(\text{vol})$ was taken from the metrological calibration of the burette with maximum bias of 2.5% (rectangular range), and a maximum repeatability of 0.8%:

$$u(\text{vol}) = [(2.5 / \sqrt{3})^2 + 0.8^2]^{1/2} = 1.4\% \quad (4)$$

$$u(\text{conc}) = \text{certified} \pm 0.5\% \text{ (95 \% confidence interval)} = 0.25\% \quad (5)$$

$$u(\text{crec}) = [u(\text{conc})^2 + u(\text{vol})^2]^{1/2} = [0.25^2 + 1.4^2]^{1/2} = 1.4\% \quad (6)$$

Quantifying the systematic error of the method and the laboratory by means of the square root of the media square (RMS):

$$\text{RMS} = [(5.2^2 + 4.8^2 + 0.2^2) / 3]^{1/2} = 4.1\% \quad (7)$$

Converting the components of the uncertainty standard $u(x)$:

$$u(\text{bias}) = [\text{RMS}_{\text{E.S.}}^2 + u(\text{crec})^2]^{1/2} = [4.1^2 + 1.4^2]^{1/2} = 4.3\% \quad (8)$$

The combined uncertainty u_c (Eq. 9) is calculated by the R_w of the laboratory and the systematic error of the added-recovered. The result obtained is:

$$u_c = (4.5^2 + 4.3^2)^{1/2} = 6.2\% \quad (9)$$

$$\text{Expanded uncertainty} = 2 \cdot 6.2 = 12.4\% \quad (10)$$

The estimation of the R_w was made in similar way for sulfates. In this case $S = 8.1$ mg/l and relative uncertainty is 4.4%. It was assumed that the standard deviation range is the only component of the uncertainty in the measurement of sulfates amount measured gravimetrically. The component of the uncertainty of systematic bias was also assessed for the recovered. It was supplemented with three test portions from the same sample with 200, 400 and 600 mg/l of SO_4^{2-} . The average recovered is 96.9%.

$$u(\text{conc}) = \text{certified} \pm 0.5\% \text{ (95\% confidence interval)} = 0.25\% \quad (11)$$

$$u(\text{vol.}) = [(2.5 / \sqrt{3})^2 + 0.8^2]^{1/2} = 1.4\% \quad (12)$$

$$u(\text{crec}) = [u(\text{conc})^2 + u(\text{vol.})^2]^{1/2} = [0.25^2 + 1.4^2]^{1/2} = 1.4\% \quad (13)$$

$$\text{RMS} = [(1.9^2 + 3.4^2 + 1.6^2) / 3]^{1/2} = 4.2\% \quad (14)$$

Converting the components of the uncertainty standard $u(x)$:

$$u(\text{bias}) = [\text{RMS}_{\text{E.S.}}^2 + u(\text{crec})^2]^{1/2} = [4.2^2 + 1.4^2]^{1/2} = 4.4\% \quad (15)$$

The combined uncertainty u_c (Eq. 16) is calculated by the R_w of the laboratory, and the systematic error of the added-recovered. The obtained result is:

$$u_c = (4.4^2 + 4.4^2)^{1/2} = 6.2\% \quad (16)$$

$$\text{Expanded uncertainty} = U = 2 \cdot 6.2 = 12.4\% \quad (17)$$

In the determination of SO_4^{2-} samples, the average content of 0.19% of SiO_2 corresponds to 31 mg/l of SO_4^{2-} , which is equivalent to 8.3%, relatively. Expressing it as a component of the combined uncertainty using a normal distribution and a confidence interval of 95%, according to Ellison and Williams (2012) would be 4.2% (Eq. 18). This new uncertainty value is incorporated into the expression:

$$u_c = (4.2^2 + 4.2^2 + 4.2^2)^{1/2} = 7.3\% \quad (18)$$

$$\text{Expanded uncertainty} = U = 2 \cdot 7.3 = 14.5\% \quad (19)$$

If the determination of the concentration of SO_4^{2-} is performed without separating the SiO_2 , it increases uncertainty by 2.6%. If this does not affect the use of the result, it can be obtained in shorter time and with 25% decrease in cost.

3. Conclusions

The precision obtained by the test method developed proved to be satisfactory for Cl^- and SO_4^{2-} anions with calculated CV values of 12.85% and 10.35% respectively. The accuracy of the method was acceptable with an average recovered of 103.7% for Cl^- and 96.9% for SO_4^{2-} .

The increased uncertainty in the determination of SO_4^{2-} by no separation of SiO_2 proved not to be significant. This is presented as an advantage since laboratory response time and costs decreases.

Two methods were developed for determination of the content of water-soluble chlorides and sulfates as a new strategy to extend the lifetime of heritage and historical concrete buildings in Cuba. Precision, accuracy and uncertainty for both measurements were evaluated with positive results.

This study is an alternative to the methods established by the Cuban Standard NC 120:2014 in regards of the durability of reinforced concrete buildings.

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