

Characterization of construction and demolition waste in order to obtain Ca and Si using a citric acid treatment

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Abstract

The paper analyses residual concrete (CW) degradation by organic acids and their potential use as nutrient source for degraded soils. The aims of the research were to carry out a chemical and mineralogical characterization of CW and to evaluate the dissolution of calcium and silicon from CW using citric acid in order to determine its potential use as a source of these elements for degraded soils. The chemical and mineralogical characterization made it possible to identify calcium and silicon as major elements associated with the concrete phases in the form of calcite, portlandite, hydrated calcium silicate, and hydrated calcium aluminosilicates. Calcium and silicon extraction was studied through a chemical and mineralogical analysis of CW (smaller than 4 mm), which was submerged in a citric acid solution for two days at 28 °C and 110 rpm under controlled and uncontrolled pH conditions. Based on the concentration of calcium and silicon in solution, it was possible to conclude that after acidulation, this waste can be used as a source of nutrients for soils degraded by human use.

Keywords: construction and demolition waste (CDW), dissolution and organic acid.

Caracterización de un residuo de construcción y demolición para la obtención de Ca y Si mediante tratamiento con ácido cítrico

Resumen

Los ácidos orgánicos y principalmente el ácido cítrico generan daños en estructuras de concreto, comprometiendo su correcto desempeño. Por otra parte, el concreto residual (CR) es un problema ambiental debido a los grandes volúmenes generados requiriendo para su correcta disposición amplias extensiones de terrenos. El objetivo de este estudio fue caracterizar química y mineralógicamente el CR y evaluar la disolución de calcio y silicio con ácido cítrico, con el fin de determinar su uso potencial como fuente de estos elementos para suelos degradados. La caracterización permitió identificar como elementos mayoritarios el calcio y el silicio asociados a las fases del concreto como la calcita, portlandita y aluminosilicatos de calcio hidratados. La extracción de calcio y silicio fue investigada mediante análisis químico y mineralógico sobre concreto residual sumergido en solución de ácido cítrico por dos días a 28 °C y 110 rpm controlando y no controlando el pH. Debido a la concentración de calcio y silicio en solución se pudo verificar que estos residuos luego de la acidulación podrían ser empleados como fuente de nutrientes para suelos degradados antrópicamente.

Palabras clave: residuos urbanos, disolución, ácidos orgánicos.

1. Introduction

The acceleration of urban growth and satisfying the demand that has resulted from the population explosion has led to the demolition of one-story buildings to make way for

vertical buildings, in order to optimize land use. This has generated greater demand for the primary materials used to manufacture concrete, particularly for aggregates, which are its primary component in terms of volume. The mining of these stony aggregates has environmental impacts and causes

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changes in ecosystems that can alter biological equilibrium [1]. Meanwhile, the solid waste generated in construction and demolition processes represents approximately 50% of the total waste produced in urban areas [1,2]. It is, thus, necessary to search for new ways to obtain aggregates, such as using construction and demolition waste (CDW). This could be one way to address the problem of mineral deposit depletion while also decreasing the space used for the final deposition of CDW [3–5].

Concretes manufactured with aggregates recycled from CDW have similar mechanical and durability properties to non-structural concretes manufactured with natural aggregate [3,6,7]. However, the use of fine aggregates (particles smaller than 4mm), which represent approximately 40% of the waste produced [4,8], has not shown good results [1,9]. Thus, it is necessary to establish a recycling process that produces value for CDW, which represents 16% of the total solid waste produced worldwide [1,10–12]. Proper management and recycling of this waste could prevent it from being deposited in uncontrolled landfills, parks, median strips, private lots, legal and illegal dumps, and other places [13].

Meanwhile, global research has been undertaken on the deterioration of buildings, structures, and monuments, since this affects their aesthetics and proper functioning [14–19]. When the materials used in these structures are exposed to substances like water, atmospheric gases, and microorganisms, they become unstable and undergo changes in their physical, chemical and mineralogical properties [14,19–21]. The main groups of microorganisms responsible for this deterioration are bacteria, fungi, and lichen [15,17,22–24]. These microorganisms exude organic acids that are capable of eroding rocks, leading to the eventual deterioration of buildings [25–27]. Additionally, during this biodeterioration process, the action of the organic acids produced by these microorganisms allows different types of plants to take route in urban structures in the absence of soil [19].

One organic acid that is commonly found in soils and deteriorated structures is citric acid. This acid plays an important role in the weathering and dissolution processes of primary and secondary minerals such as aluminosilicates in both soil and urban structures [17]. This increases the possibility that destabilized concrete waste could leave behind elements such as calcium and silicon. These elements are commonly found in soils and, thus, could be used as sources of nutrients in soils degraded by human use.

The objective of this study was to evaluate the dissolution of Ca and Si contained in concrete waste by attacking it with citric acid at different concentrations and pH levels. In doing so, the idea was to establish the dissolution process of these elements and determine their potential to be used as nutrient sources for degraded soils.

2. Experimental work

2.1. Waste samples

The samples used for this study were CW provided by the Cement Chemistry Laboratory at the Universidad Nacional de Colombia in Medellín. The samples were air-dried for one week and then a size reduction process was performed in

order to guarantee a fine particle size distribution between 0.038 and 0.30 mm and a coarse particle size between 0.3 and 2.36 mm. These sizes cannot be used as recycled aggregates.

2.2. Waste characterization

The waste used in the study was characterized by Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and X-ray Fluorescence (XRF).

The SEM images and microchemical analysis were obtained with a JEOL JSM 5910 LV microscope equipped with an EDS OXFORD solid-state detector. The work was performed in back-scattered electron (BSE) observation mode, with a 20 kV acceleration voltage, and a 210 second data collection time. The samples were previously metallized with gold.

In order to determine the element content in the waste, the energy-dispersive XRF technique was used with 2D direct excitation. The spectrometer used was a PANalytical MiniPal 2, 9 w (30 KW, 1 mA) chrome irradiation tube, Si-PIN detector, 12-position sample changer, 100-240 V, 45-65 HZ.

The mineralogical composition of the waste was determined by XRD using a Panalytical Reference X'Pert PRO MPD with Cu radiation of wavelength $K\alpha_1 = 1.5406 \text{ \AA}$. Power: 45 kV and 40 mA. Scanning: a step-size of 0.013° at a speed of 59 s per step with continuous scanning.

2.3. Experimental methods

In order to evaluate the dissolution of the Ca and Si contained in the concrete waste, exploratory tests were performed at three different pH levels to determine the best pH. Subsequently, tests were performed at a constant pH, with and without regulating this parameter, in order to identify its effects. All of the experiments were performed using the same procedure.

The elements of interest were obtained by treating the waste with an aqueous solution containing citric acid. The following concentrations of citric acid were used: 8.5 gL^{-1} (0.04 M), 9.5 gL^{-1} (0.05 M), and 10.5 gL^{-1} (0.055 M). A constant pH of 2.2 was maintained; this was previously determined by exploratory testing. The elements were sterilized in an autoclave at 120°C and 0.1 MPa for 20 minutes before use.

The working system was prepared by putting 5 g of waste into contact with each liter of solution until a 300 mL working volume was obtained. This was poured into a 500 mL reactor. The mixture was placed in an orbital agitation system, where it was agitated for 48 hours at 110 rpm with an average working temperature of 28°C . Samples of 10 mL were taken at certain time intervals. These were sent for chemical analysis to determine their Ca and Si content. During these intervals, the pH was also regulated for the tests in which it was necessary to do so. The recovery of these last elements was obtained by creating a balance in the aqueous solutions.

3. Results and discussion

3.1.1 Initial chemical and mineralogical characterization

XRF analysis was performed to identify the elements present in the waste. The primary elements found were silicon

Table 1.
Chemical composition of concrete waste according to XRF.

Element	%Fine	%Coarse
Si	19.16	20.69
Ca	22.15	17.93
Al	5.07	5.16
Fe	2.57	2.51
P	1.03	0.96
Ti	0.40	0.36
Mg	1.43	0.51
Na	< 0.010	< 0.010
K	0.99	0.98
Mn	0.14	0.14
Cr	0.06	0.06
S	0.93	0.93
Losses on ignition	6.92	9.95

Source The Authors

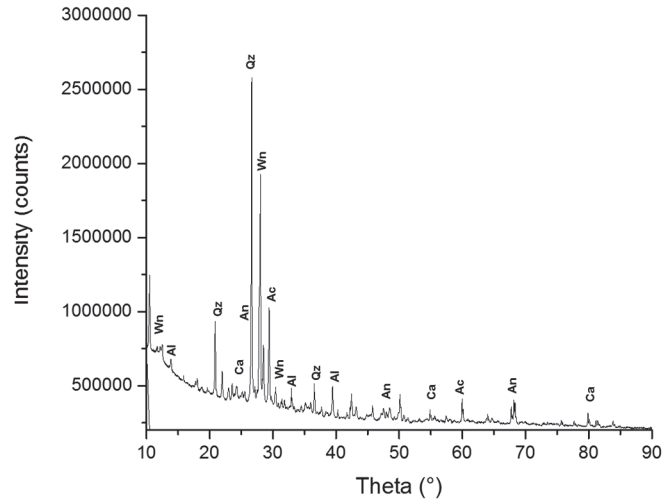


Figure 2. X-ray diffraction of coarse concrete waste, where Qz is quartz, An is anatase, Wn is wollastonite, Ac is actinolite, Al is albite and Ca is calcite. Source: Mejia et al., 2015.

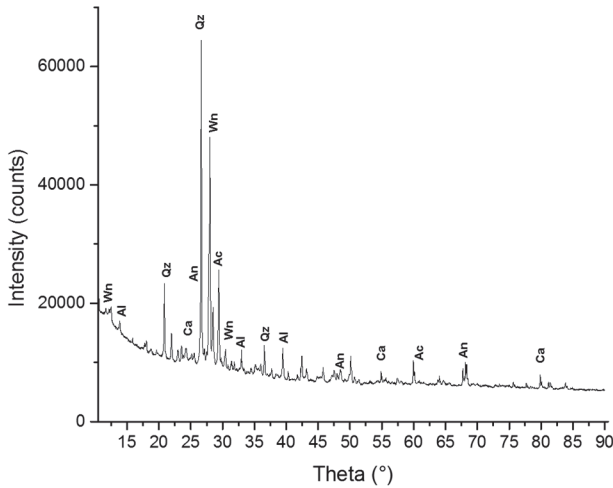


Figure 1. X-ray diffraction of fine concrete waste, where Qz is quartz, An is anatase, Wn is wollastonite, Ac is actinolite, Al is albite and Ca is calcite. Source: Mejia et al., 2015

and calcium, with aluminum and iron present to a lesser extent (Table 1). Additionally, traces of the following elements were found: titanium, magnesium, sodium, potassium, manganese, chromium, phosphorous, and sulfur.

XRD was used to confirm the mineral phases associated with the waste of both particle sizes. The mineral phases detected for the fine material were: quartz (SiO₂), calcite (CaCO₃), albite and actinolite (Ca₂(Mg,Fe²⁺)₅Si₈O₂₂(OH)₂) (Fig. 2). The phases for the coarse material were: quartz, actinolite, albite, and calcite (Fig. 2). It is important to note that the mineralogical phases present in cement paste can be masked by the minerals previously mentioned, which may be the components of concrete aggregates.

SEM analysis and microchemical analysis confirmed the presence of minerals including feldspars and silicates. Fig. 3 shows sub-euhedral, rounded, and elongated particles. The percentage in weight of the elements measured by EDS was very similar to that of albite, calcite, quartz, and aluminosilicates.

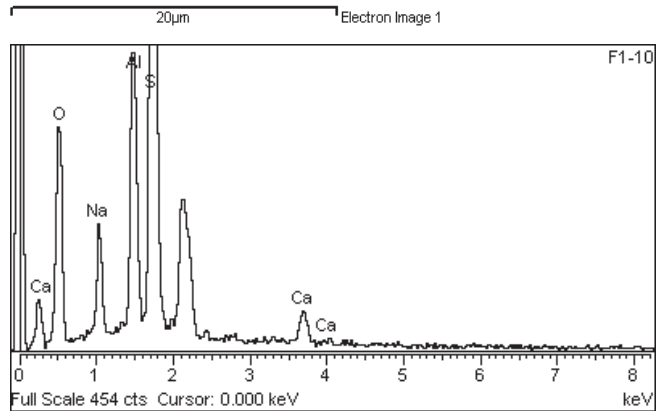
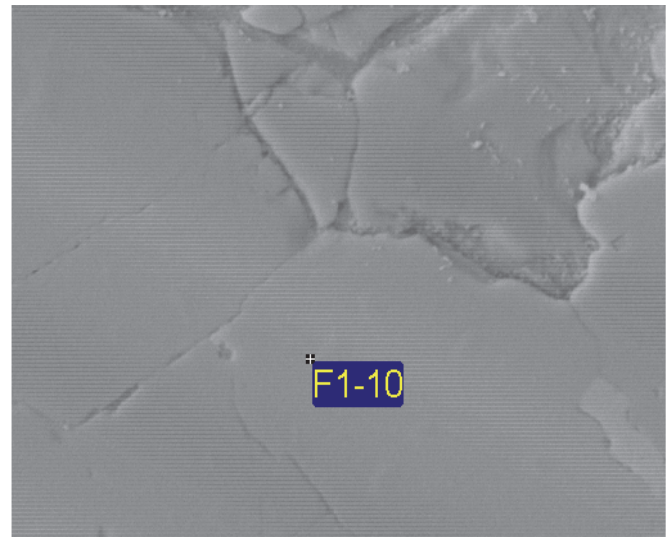


Figure 3. Upper image: SEM micrograph of a calcium-sodium plagioclase grain. Lower image: microchemical analysis spectrum showing Ca: calcium, O: oxygen, Al: aluminum, Si: silicon, Na: sodium. Source The Authors

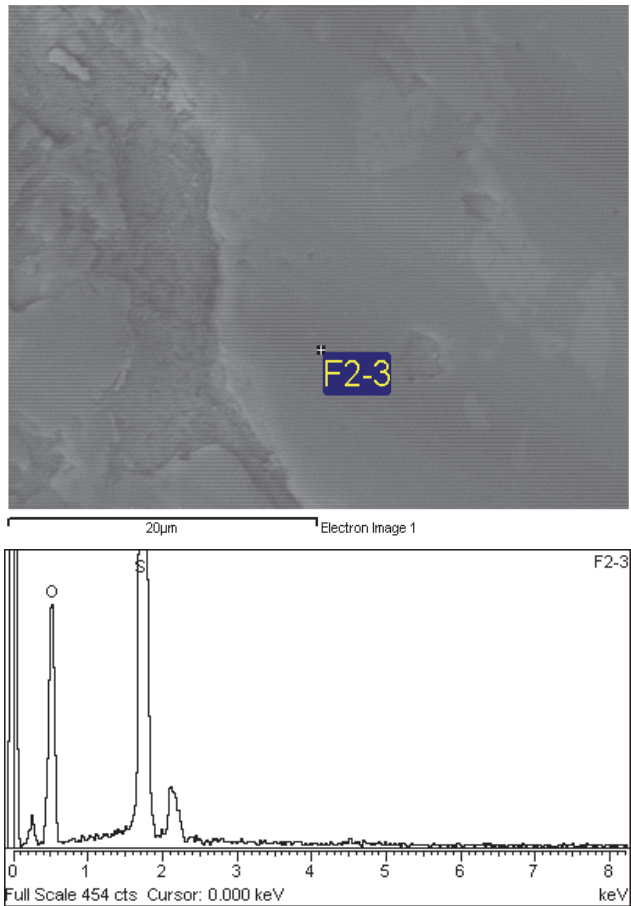


Figure 4. Upper image: SEM micrograph of sample 2, which corresponds to a grain of quartz. Lower image: microchemical analysis spectrum of the grain. O: oxygen, Si: silicon
Source The Authors

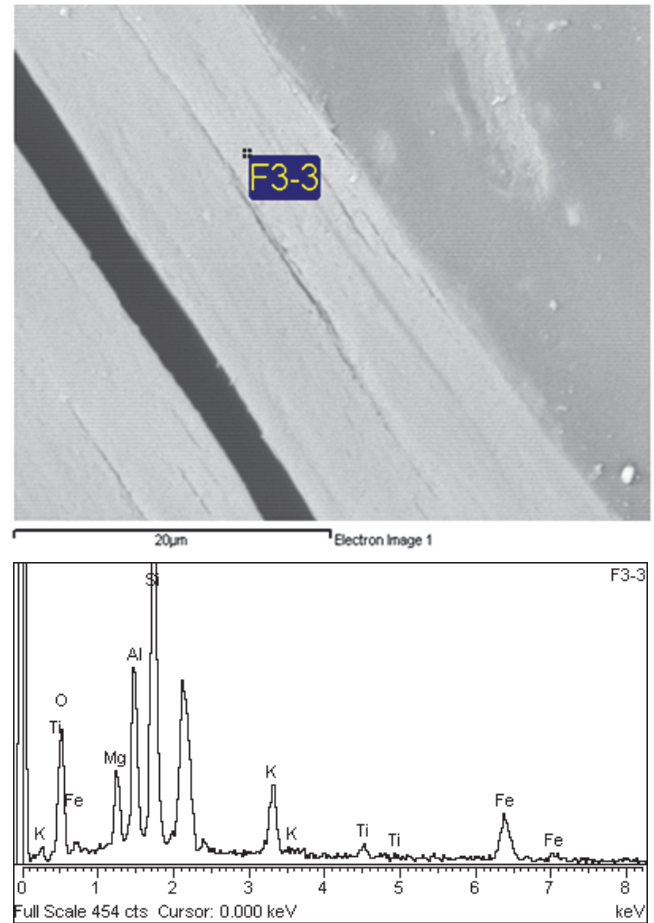


Figure 5. Upper image: SEM micrograph of silicate grains, sample 3. Lower image: microchemical analysis spectrum of the grain. K: potassium, O: oxygen, Al: aluminum, Si: silicon, Mg: magnesium, Ti: titanium, Fe: iron.
Source The Authors

The quartz that was present was interspersed with different minerals including calcium, sodium and magnesium aluminosilicates (Fig. 4).

Fig. 5 shows a grain of magnesium, potassium, and iron silicate, which presents a fibrous tabular structure typical of this type of mineral. The grains are intergrown with aluminosilicate grains.

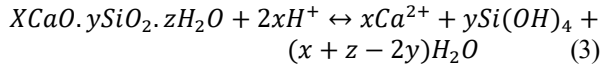
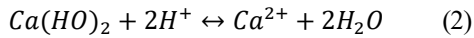
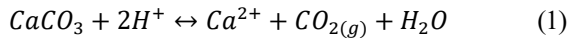
Through chemical and mineralogical characterization of the waste, we were able to determine the presence of elements that could potentially be used as nutrients in degraded soils after being subjected to a structural destabilization process. This is because they are found in minerals that have a very low or no solubility in water. Reusing these elements would generate a recycling of nutrients such as silicon and calcium, and, thus, they would contribute to plant growth [11,28]. According to XRD, XRF and SEM results, the CW samples presented a large percentage of quartz, which is a chemically inert mineral that is useful for improving the physical properties of soil, including texture, aeration, infiltration, and drainage [11]. Additionally, the presence of calcite, wollastonite, and anatase was also observed. These minerals can improve the chemical properties of soils such as their pH regulation. In this case, the finest material presented the highest calcium content [12].

3.2. Effect of pH on dissolution with citric acid

3.2.1. Exploratory tests

In order to evaluate the effect of acidity on Ca and Si dissolution at different citric acid concentrations, the dissolution of these elements was evaluated after 48 hours of processing. We found that the greatest Ca and Si dissolution occurred with 10.5 gL⁻¹ of citric acid and a pH of 2.2 (Table 2). This coincides with other authors' findings [24], who discovered that citric acid hydrolysis permits the dissociation and complexation of Ca²⁺. The dissolved Ca²⁺ probably came from carbonates and portlandite, which are the most soluble minerals. From a lesser extent it also came from hydrated calcium silicates, which are low solubility minerals. Ca²⁺ dissolution produces concrete decalcification and the formation of calcium salt (Equations 1 and 2) [18,24,29]. Meanwhile, [30–34] found that when concrete was subjected to attacks by different organic acids, the effect was accelerated at a lower pH. Moreover, the release of silicon indicated that calcium silicate dissolution took place in addition to portlandite and calcite dissolution; this occurred as shown in Equation 1-3 [29]. However, some authors have

found that after a partial dissolution process, a silica gel forms, which limits the release of silicon and calcium [29,32,33]. Using this test, we were able to determine that the working pH to improve Ca and Si release was 2.2; this condition was used thereafter.



3.2.2. With pH regulation

In order to evaluate the effect of the citric acid concentration, an experiment was conducted in which the acidity of the solution was regulated. The dissolution kinetic of Ca over time at different citric acid concentrations was similar for a short period of time (seven hours), during which the dissolution velocity was high. After 10 hours, the velocity tended towards zero; this effect was more marked for the lowest citric acid concentrations (Fig. 7). Additionally, the curve forms were typical of passivation processes. This could indicate the formation of a precipitate on the waste particles, thus generating a barrier between the citric acid and the waste particles. Ca^{2+} dissolution was greater at a higher acid concentration (10.5 gL⁻¹) (Fig. 6). In the case of silicon, dissolution was lower than 10% and release occurred rapidly during the initial hours of the process (Fig. 7). Calcium release could have been inhibited by the formation of calcium citrate ($Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$); however, this compound is moderately soluble in water (0.096 g for each 100 mL at 23 °C), and the concentration of $Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$ that formed was lower than 0.05 g per 100 mL. Thus, it appears that the process was not inhibited by the formation of calcium citrate. Meanwhile, as previously noted, some authors have found that the formation of a silica gel can limit concrete dissolution, impeding calcium and silicon release. The formation of this gel is, thus, thought to have limited the process [30].

3.2.2. Without pH regulation

In order to determine the effect of pH on calcium and silicon dissolution at different citric acid concentrations, this parameter was left to evolve over the course of the process. The dissolution behavior of calcium and silicon was very similar to that generated when pH was controlled, except that the dissolution percentages were lower (Figs. 8 and 9). Meanwhile, proton consumption was observed that was caused by the increase in pH during the process, which rose from 2.2 to 3.7 in all cases (Table 3). This could be due to the dissolution reactions of the calcium carbonate and the calcium aluminosilicates.

3.3. Final chemical and mineralogical characterization

By conducting SEM analysis and microchemical analysis after completing the process with citric acid, we were able to

identify alterations in the concrete waste particles (Fig. 10). We observed calcium silicate hydrate grains with evidence of flaking caused by dissolution. Moreover, microchemical analysis of the sample of concrete waste with citric acid (10.5g/L, pH controlled at 2.2) showed that the calcium concentration was lower after dissolution process (Table 1). This indicates the decalcification of calcium silicate hydrate. These results coincide with other authors findings who have explained the calcium silicate hydrate leaching was a result of the decalcification process, and silica gel precipitates over the grain [17, 19, 24, 29, 32]

Table 2. Calcium and silicon dissolution percentages at different pH levels and citric acid concentrations.

pH	Ca (%)			Si (%)		
	8.5 gL ⁻¹	9.5 gL ⁻¹	10.5 gL ⁻¹	8.5 gL ⁻¹	9.5 gL ⁻¹	10.5 gL ⁻¹
2.2	45.52	34.52	42.87	0.31	0.83	1.3
3.2	32.42	30.74	40.29	0.23	0.82	0.87
4.2	31.9	29.9	33.54	0.21	0.61	0.83

Source The Authors

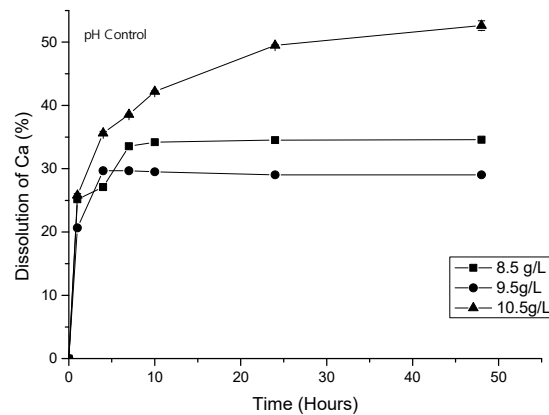


Figure 6. Dissolution of calcium from CW with controlled pH. Source The Authors

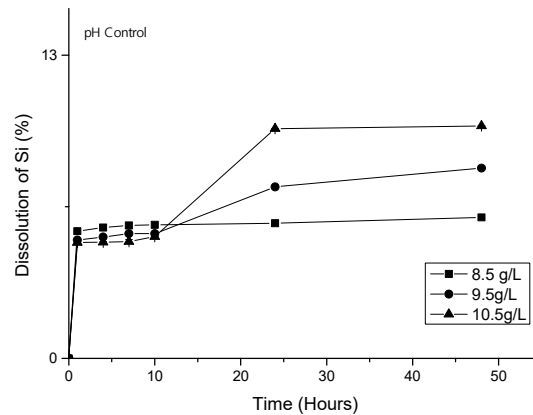


Figure 7. Dissolution of silicon from CW with controlled pH. Source The Authors

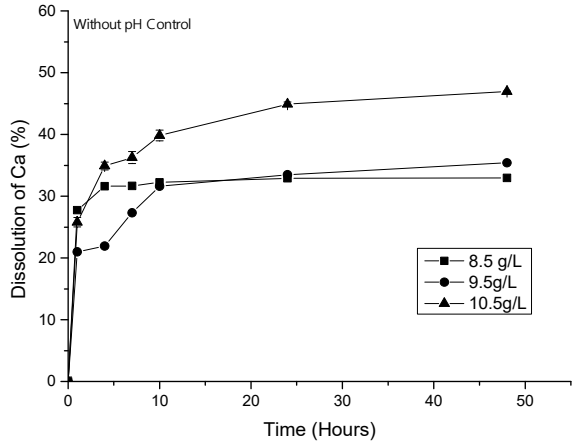


Figure 8. Dissolution of calcium from CW with uncontrolled pH. Source The Authors

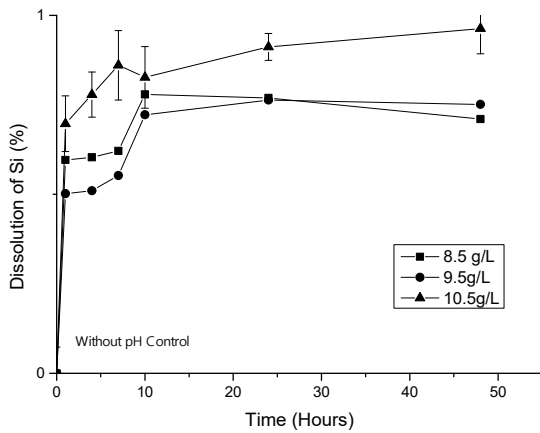


Figure 9. Dissolution of silicon from CW with uncontrolled pH. Source The Authors

Table 3. Initial and final pH in the concrete waste dissolution process using citric acid.

Initial citric acid concentration (gL ⁻¹)	Initial pH	Final pH
8.5	2.2	3.77
9.5	2.2	3.72
10.5	2.2	3.65

Source The Authors

4. Conclusions

CW has the potential to be used as a source of Ca and Si, after undergoing an acidulation process with citric acid. It could be used to provide nutrients for plants in nutrient-deficient soils since; in addition to containing minerals such as calcite, portlandite, and CSH, it can also release Ca and Si in solution after undergoing a dissolution process with citric acid.

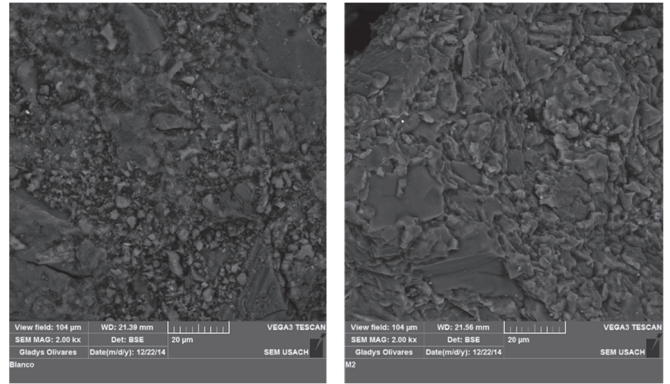


Figure 10. Scanning electron microscopy analysis of the concrete waste after (left) and following (right) the dissolution process with citric acid (10.5g/L-1, pH controlled at 2.2). The image to the right shows the flaking process of the concrete particles after the leaching process.

Source The Authors

Table 1. Scanning electron microscopy microchemical analysis of the sample of concrete waste after and before the dissolution process with citric acid (10.5g/L-1, pH controlled at 2.2).

Element	Weight (%)		atomic (%)	
	Control	Leaching sample	Control	Leaching sample
O	51,78	48,67	68,63	63,65
Ca	18,35	3,95	9,71	2,07
Si	16,58	30,08	12,52	22,42
Al	5,29	7,03	4,15	5,45
Fe	3,75	4,01	1,42	1,52
Mg	2,15	2,87	1,87	2,48
Na	1,39	2,22	1,29	2,03
K	0,62	0,57	0,34	0,31
S	0,09	0,6	0,07	0,07
Total	100	100	100	100

Source The Authors

The minerals identified are present as primary or secondary minerals in the clayey fraction of the soil; therefore, they would not drastically affect the chemical composition of the soil.

By performing dissolution tests with citric acid, we were able to observe calcium silicate hydrate dissolution; this was evidenced by the concentration of silicon in solution.

The best dissolution conditions for calcium and silicon occurred at 10.5 g/L-1 of citric acid and had a pH of 2.2, with a controlled pH.

The SEM characterization allowed us to find an agglomeration of grains and corrosion evidence on the CSH grains.

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