

## Performance of Chlorides Penetration and Corrosion Resistance of Mortars with Replacements of Rice Husk Ash and Nano-SiO<sub>2</sub>

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The use of nano-silica oxide (nSO) and ash as cement replacement in mortars has allowed improve the performance of their physical, chemical and electrochemical properties. Researchers have used as a replacement or addition of cement in mortars various types of nSO and ash, including rice husk ash (RHA). In the study of the synergy of simultaneous use of nSO and ash, research has focused on improving the physical and mechanical properties of mortar, with few reports directed to the study of the resistance to corrosion in corrosive environments with the presence of chlorides. It was evaluated in the present work the performance of the simultaneous use of RHA and nSO as replacement of ordinary Portland cement (OPC) in mortars with plastic consistency according to ASTM C1437-99 with use of superplasticizer and relation water/ cementitious of 0.50 at 30 and 90 days of age, conducting studies on compressive strength, total porosity, accelerated corrosion test with impressed voltage at 12.0 V and Rapid Chloride Penetration Test according to ASTM C1202-97. OPC was replaced by 15% RHA and 0.50% nSO individually and simultaneously, resulting the most effective simultaneous use with decrements in porosity of 5.7% and charge passed of 77.7% and increases in compressive strength and to corrosion of 6.1 and 34.0%, respectively. The use of superplasticizer facilitated the manufacturing of mixtures with RHA and nSO, but decreased the speed of the hydration process of the mixture, demeriting the performance of the properties of the hardened mortar. The replacement of OPC by 15% RHA and 0.5% nSO in simultaneous use in mortars of plastic consistency has advantages in the performance of the physical, mechanical and electrochemical properties, due to the synergistic effect of ash-nSO and porosity- compressive strength, presenting very low chloride permeability.

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**Keywords:** Plastic-mortar, mortar-nanometric, accelerated -corrosion, chloride

## 1. INTRODUCTION

In reinforced mortar, the arrival of chlorides to the surface of the reinforcement is the major cause of corrosion. These ions move through the water contained in the pore network, its mobility is defined by the porosity and permeability of the cement matrix. As the durability of mortar and concrete is demerited with the corrosion of the reinforcement, it is also a direct consequence of the pore structure, porosity and pore size distribution of the mass of the mortar [1-3]. In order to provide durability to mortars and concrete, researchers have replaced cement with various types of ash, RHA [4, 5], fly ash, blast furnace ash, sugarcane bagasse ash, among others [6-17], improving mechanical, structural, physical, chemical and electrochemical properties, diminishing the effect of the corrosion in the reinforcement in adverse environments [13, 18- 20]. These ashes are the product of various industrial and agricultural processes. For example, rice husk is an agricultural waste used for energy production in power plants, being the resulting RHA in the process discarded in pourers of waste, causing an environmental problem [21]. By virtue of the pozzolanic properties of RHA, it has been used as a replacement of cement, avoiding the problem of waste and reducing the amount of cement used in the mixture. Reduction in the use of cement contributes to the environmental aspect, because in its production high levels of contamination are generated [22, 23]. The replacement or addition of cement is not limited to ashes; different researchers have used nSO exploiting the properties conferred to them by their nanometric size [24].

In recent years, with the help of nanotechnology it has been made great progress in improving material properties, inclusive in mortars by the addition of nSO in the mix. In several studies it has been replaced cement individually, nSO [25- 27], Iron oxide, titanium oxide and zirconium, among others [28-32]. nSO in addition or replacement of cement provide increased durability to mortar and concrete and benefit in the physical and mechanical properties [24]. Furthermore, it has been investigated the synergistic effect obtained by the simultaneous use of nSO with CV and ash of sewage sludge, where the pozzolanic activity is accelerated at an early age due to the high silica content and high surface area of the nSO. It was concluded that the gel C-H-S generated fills in the gaps of the cement matrix, achieving a more uniform and compact matrix, and consequently an increase in the durability of the mortar [34- 38]. However, there are few investigations on the simultaneous use of nSO and ash in the replacement of cement.

In the study of the performance of nSO and ashes incorporated into a cement matrix, reports related to FA predominate [33- 35], with a focus on mechanical, physical and chemical properties of the matrix, studies on corrosion of reinforcement are scarce. According to reports from various authors, incorporating RHA improves the microstructure of the paste and reduces the effects of corrosion [4, 5]. However, there are scientific reports that address the durability and corrosion of mortars reinforced by replacements of OPC by nSO and RHA in simultaneous use [39, 40]. The replacement of OPC by RHA and nSO in simultaneous use should favor the microstructure of the matrix, obtaining mortars with lower porosity and higher performance in mechanical strength, resistance to corrosion and chloride penetration.

The purpose of the present work was to study the performance of porosity, mechanical strength, corrosion resistance and resistance to chloride penetration in mortars, in replacing OPC by

RHA and nSO individually and simultaneously, with 15% RHA replacements and 0.50% nSO referred to the weight of total cementitious. For this, studies of porosity in open pore, simple compressive strength, accelerated corrosion test with impressed voltage (ACTIV) and Rapid Chloride Penetration Test (RCPT) were performed.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Materials

For the production of the specimens it was used fine aggregate, OPC, RHA, nSO, distilled water and superplasticizer (SP). The fine aggregate was siliceous river sand graded according to ASTM C 33-03, fineness modulus of 2.53, mass density 2570 kg/m<sup>3</sup> and 4.2% absorption. The physical properties of cementitious materials are presented in Table 1.

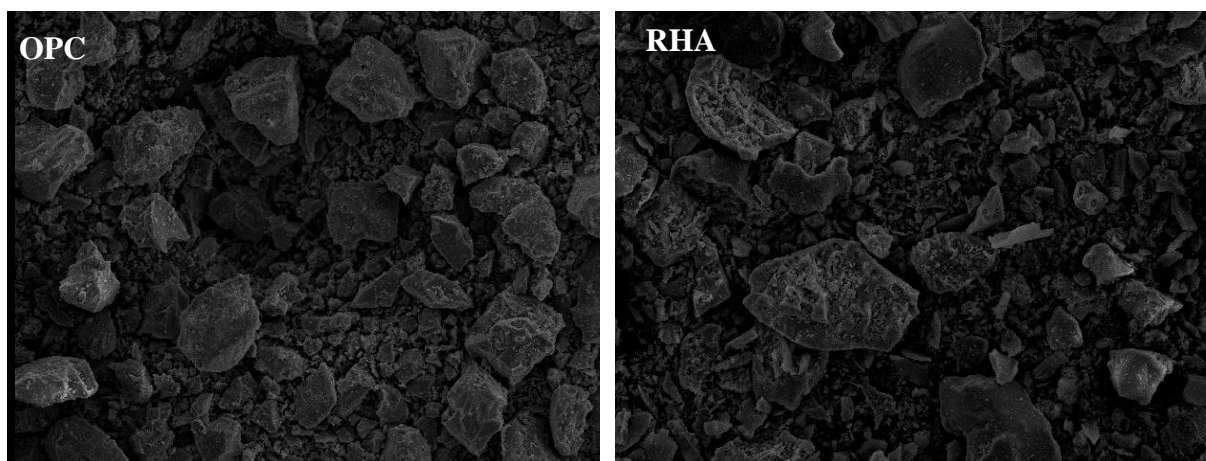


Figure 1. SEM of Ordinary Portland Cement and Rice Husk Ash (x500).

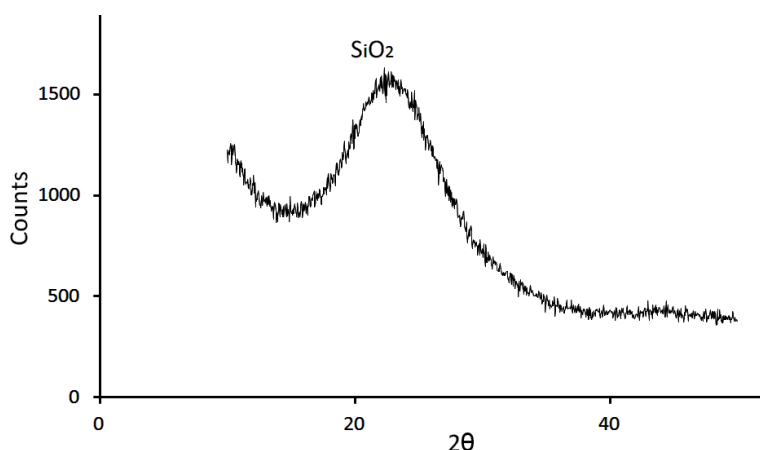


Figure 2. nSO Rx Dispersion.

The SP was a water-reducing admixture of high rank and short delay without chlorides type F and type I, according to ASTM C 494 / C 494M - 99a [41] and C1017 / C1017M - 07 [42]. The OPC was supplied by a local cement company, while the RHA was obtained from industrial wastes from Odisha, India, milled in a Los Angeles apparatus for 2 hr. and sieved at 325 mesh. nSO was commercially obtained without any additional treatment for its use. The chemical properties of OPC, RHA and nSO, according to chemical analysis of Gravimetry (G) and Stoichiometry (S), are presented in Table 2 and Scanning Electronic Microscopy (SEM) of cementitious in Figure 1. Furthermore, the scattering analysis of Rx of nSO is shown in Figure 2.

**Table 1.** Physical properties of cementitious materials.

Property	OPC	RHA	nSO
Mass Density ( $\text{kg/m}^3$ )	3071	2251	-
Superficial Area ( $\text{m}^2/\text{g}$ )	20.23	23.82	777.7
Average particle size ( $\mu\text{m}$ )	27.61	29.88	0.015

**Table 2.** Chemical components of materials

Oxide	OPC	RHA	nSO	Analysis method
$\text{SiO}_2$	20.046	84.375	71.451	G
$\text{SO}_3$	2.589	0.080	-	E
$\text{Fe}_2\text{O}_3$	1.976	0.309	-	E
$\text{Al}_2\text{O}_3$	0.294	0.235	N.D.*	E

\*N.D. Not Detected

## 2.2 Mixture preparation and specimens.

In mortar mixtures a relation Water/Cementitious (A/cm) of 0.50, Sand/Cementitious of 2.75 and SP was used to achieve a fluency of  $110 \pm 5\%$ , the dosage of cementitious in the mixtures are shown in Table 3. The mixing procedure was similar to ASTM C 305-12 [43] with some variations:

- 1) If the mixture contained RHA, this was dry blended with OPC until achieving uniform consistency.
- 2) If the mixture contained nSO, this was added to 95% of the total water of the mixture, and then was immediately sonicated for 10 min.
- 3) The cementitious material was added to 95% of the water; immediately it was mixed for 30s. at slow speed.
- 4) The mixer was stopped and switched to medium speed; immediately it was mixed for 30s. while the sand was added slowly during this period.
- 5) It was mixed for 30 s. at medium speed. SP + 5% water were added slowly in the first 10 s.

6) The mixer was stopped remaining at rest for 90 s. In the first 15 s. the walls of the mixing vessel were rapidly scraped and the mixture was covered the remaining time.

7) Finally, it was mixed for 90 s. at medium speed.

The fluidity of the mixture was verified for  $110 \pm 5\%$  according to ASTM C 1437-99 [44], the mixture was immediately poured into molds of PVC and covered with plastic for 24 hr. until hardened condition.

**Table 3.** Cementitious dosing in mortar mixtures (%)

Mixture Nomenclature	OPC	RHA	nSO
M100-0-0	100.0	0.0	0.0
M85-15-0	85.0	15.0	0.0
M99.5-0-0.5	99.5	0.0	0.5
M84.5-15-0.5	84.5	15.0	0.5

### 2.3 Total Porosity and compressive strength.

The compressive strength was performed according to ASTM C 109 / A 109M-05 [45] in cubic specimens of 2.5 cm side. After the assay, the residues of the specimens were immersed in methanol for analysis by SEM.

The porosity of the hardened mortar was determined according to equation (1). The method used was in a vacuum saturated condition, accepted method successfully by various authors to calculate porosity in cementitious materials [46, 47].

$$P (\%) = 100 * \frac{W_a - W_d}{W_a - W_w} \quad (1)$$

Where:

P= Total Porosity (g).

$W_a$ = The specimen weight in saturated conditions (g). The saturated condition is achieved by 1.0 hr. vacuum in air, 3.0 hr. of vacuum submerged with deaerated water and 20 hr. of submerged at ambient pressure.

$W_d$ = Weight of dry specimen after 24 h. of drying in oven at  $100 \pm 5$  °C (g).

$W_w$ = Specimen weight under saturated conditions submerged in water (g).

Both assays were performed at 30 and 90 days of curing in distilled water, 100% relative humidity and temperature of  $23 \pm 2$  °C. The results reported are the average of 3 assays.

2.4 Accelerated corrosion test with impressed voltage.

ACTIV provides an indirect comparison parameter for the estimation of the resistance to corrosion of cementitious materials [47]. Specimens were 4.0x4.0x16.0 cm, reinforced with steel rods 0.95 cm in diameter and 16.0 cm long as shown in Figure 3. Steel was protected in the interface air-mortar, coating of 1.5 cm and steel surface exposed to a medium of 37.3 cm<sup>2</sup>.

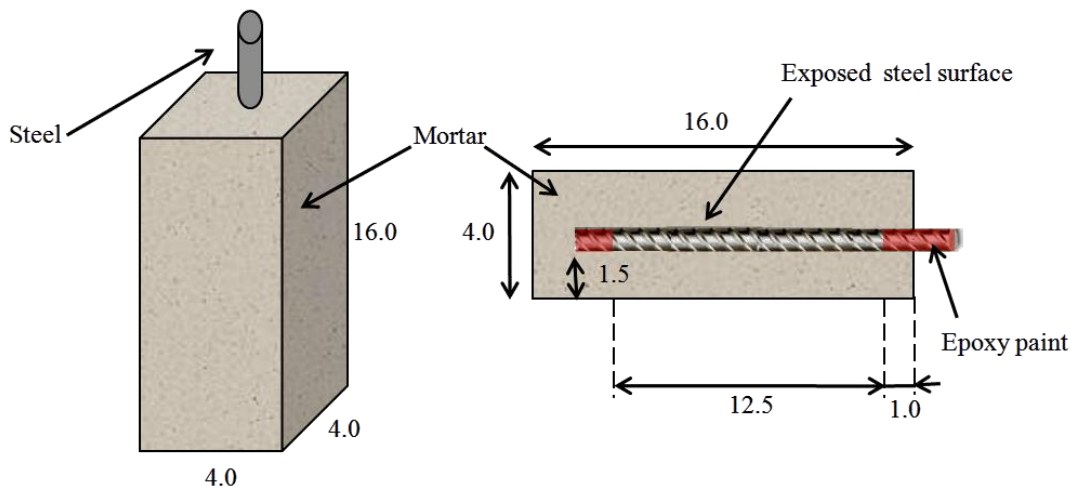


Figure 3. Reinforced mortar specimen.

At 30 and 90 days of age, the specimens were subjected to ACTIV under arrangement presented in Figure 4; they were immersed in 5% NaCl aqueous solution by applying constant voltage of 12 V to  $\pm 0.1$  at  $25 \pm 2$  oC. The condition of the prism was monitored continually until the cracking of the specimen, recording the time of initiation of the first crack (TIFC). The results reported are the average of 3 assays.

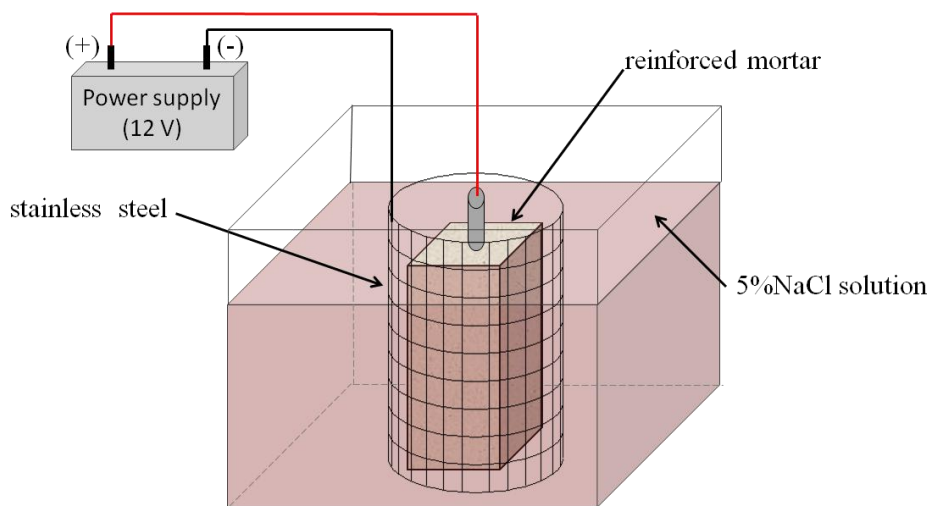
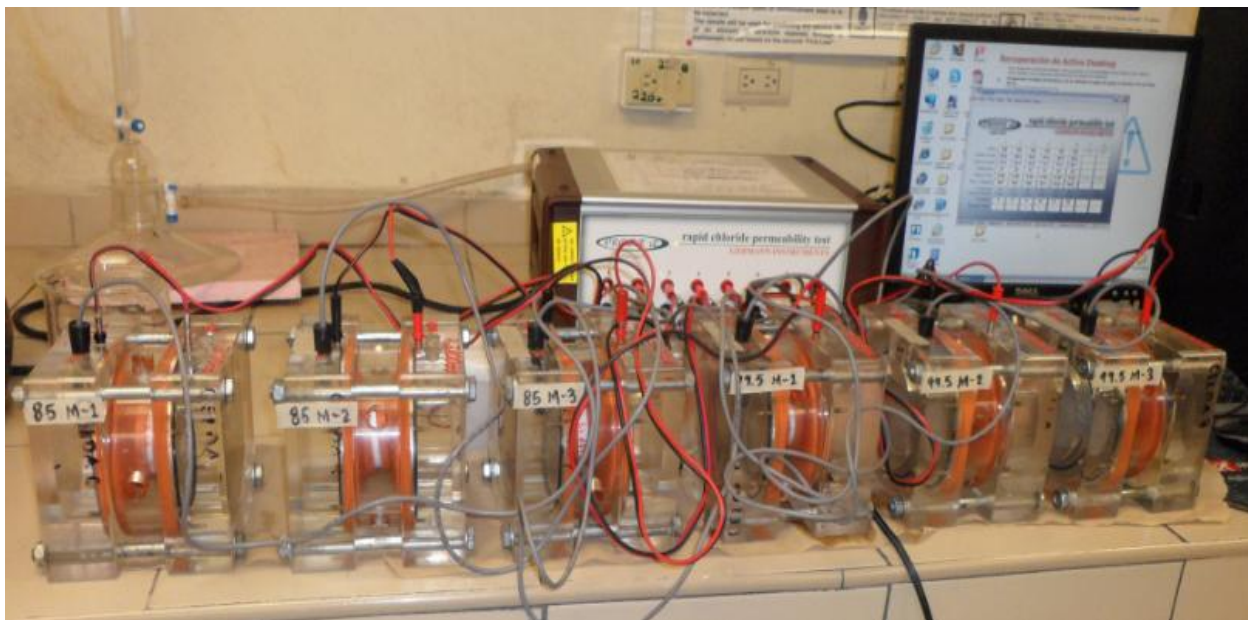


Figure 4. Experimental arrangement ACTIV assay.

### 2.5 Rapid chloride penetration test.

In RCPT constant voltage of  $60.0 \pm 0.1$  V is applied in cementitious cylindrical specimen, obtaining as a result from the test a total passed charge that is used to classify a mortar according to its Chlorine ion permeability. In the present study the RCPT was performed according to ASTM C1202-97 [48] with a variant of the test tube dimensions, dimensions of 5.0 diameter x 4.0 cm height were used at 30 and 90 days age in hardened mortar, the experimental arrangement is shown in Figure 5. The reported results are the average of 3 assays.



**Figure 5.** Rapid chloride penetration test according to ASTM C1202–97

## 3. RESULTS AND DISCUSSION

### 3.1 Materials

The use of RHA and nSO as a replacement of OPC provides higher performance to the properties of mortar and concrete, creating products that serve to fill in the gaps of the matrix [5, 25]. According to SEM presented in Figure 1, the particles conforming RHA are irregular and have sharp edges, size of 1.7 to 45.4  $\mu\text{m}$  and average particle size of 29.88  $\mu\text{m}$ , they are also irregular in OPC but of softer edges with sizes of 1.3 to 63.0  $\mu\text{m}$  and an average particle size of 27.61  $\mu\text{m}$ . OPC has thinner particles than RHA while larger particles. RHA is a natural ash calcined type N according to ASTM C618-99 [49], with  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{F}_2\text{O}_3 > 70\%$  and  $\text{SO}_3 < 4.0\%$ . nSO has a  $\text{SiO}_2$  content of 71.45%, higher to OPC and lower to RHA. The particle size distribution of cementitious, their physical and chemical properties define the microstructure of the hardened mortar.

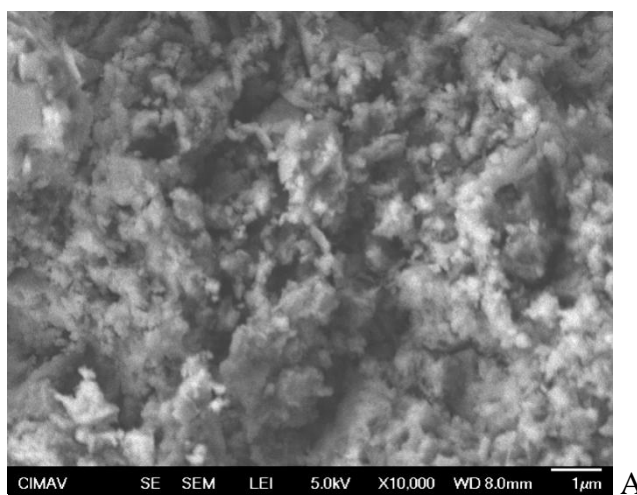
3.2 Mixtures and morphology of hardened mortar

The hygroscopic nature of RHA and its surface area larger than OPC cause mixtures requiring more water [50] or increases the amount of SP used in A/Cm constant [51]. In the case of ash and nSO in simultaneous use, researchers have used A/Cm up to 0.70 [36], it is well accepted the SP use to achieve the desired fluidity of the mixture [47]. In mixtures made in this study, the replacement of OPC by RHA and nSO in combined use increased the amount of SP and the surface area of the cementitious; this is presented in Table 4. The high surface area of the mixture due to nSO and RHA replacements conjunct to the hygroscopic nature of RHA contributed to the increase of SP, reaching percentages up to 5.2% in M68-30-2 and surface area of 36.5 m<sup>2</sup>/g. The properties of the mixture in fresh state and the curing conditions contribute to define the microstructure of the hardened mortar.

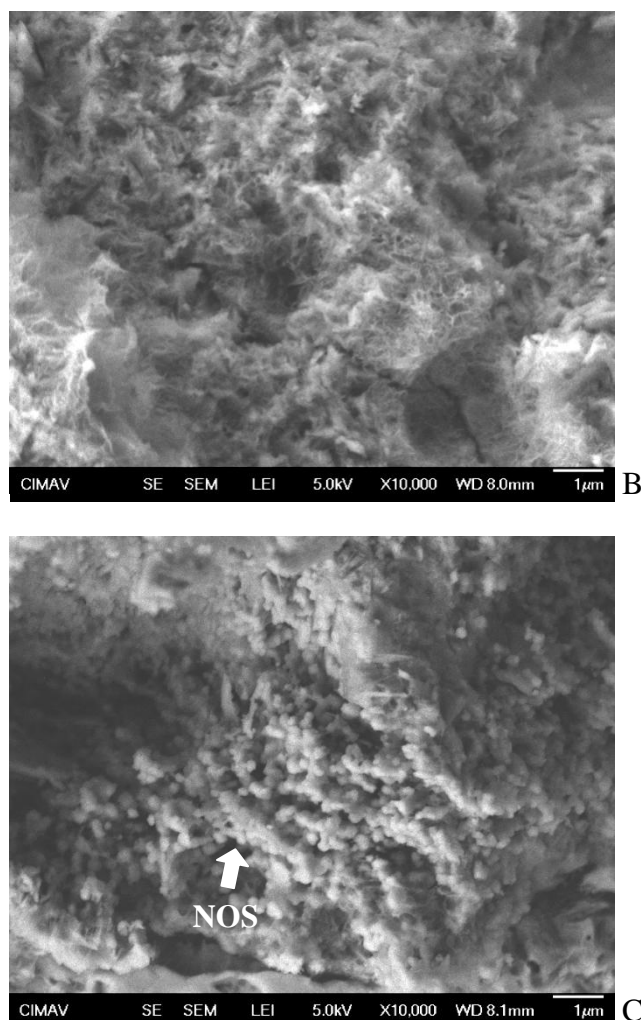
The uniform dispersion of nSO has an important role in improving the microstructure of the cement matrix, contributing to an early hydration [52]. The microstructure of hardened mortars with and without replacements is presented in Figure 6. The morphology of the cement matrix with RHA replacements is more compact than only with OPC or OPC + RHA + nSO. Furthermore, there were not nSO cluster localized, denoting good dispersion. The porosity and mechanical properties of the hardened mortar are mainly defined by the microstructure of the matrix.

**Table 4.** SP and superficial area in mortar mixtures.

Mixture	SP (%)	Superficial area of the cementitious (m <sup>2</sup> /g)
M100-0-0	0.8	20.2
M85-15-0	1.3	20.8
M99.5-0-0.5	1.5	24.0
M84.5-15-0.5	2.1	24.6







**Figure 6.** SEM hardened mortars 30 days of age, a) M100-0-0 b) M85-15-0 c) M84.5-15-0.5

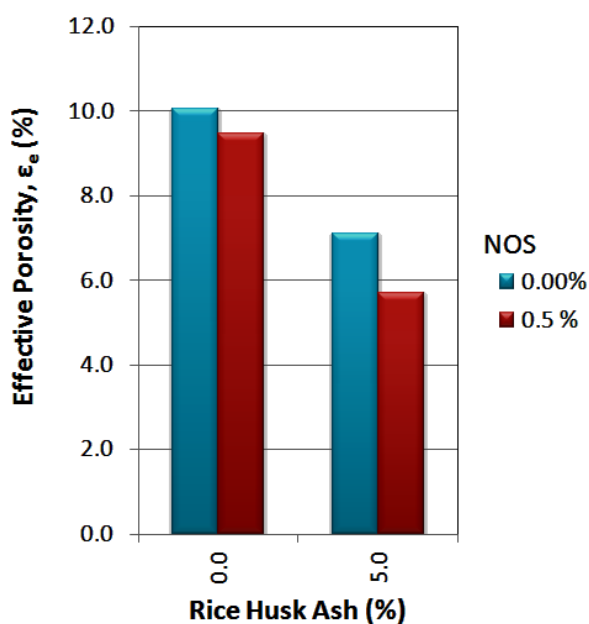
### 3.3 Total porosity and compressive strength.

Total porosity of mortars decreases to major replacement of OPC by RHA, in a certain percentage the porosity again increases. [47] The total porosity resulting from hardened mortars are presented in Table 5. For RHA individual replacement, porosity increased to major RHA respect to M100-0-0. In general, the individual replacement of nSO favored the porosity, presenting at 30 days the lower porosity in M99.5-0-0.5 and in M84.5-15-0.5 for 90 days. The simultaneous replacement of RHA and nSO was more favorable at 90 days of age, presenting the lowest porosity in M84.5-15-0.5, less than M99.5-0-0.5. The simultaneous use of RHA and nSO favors the decrease of porosity in ages superior to 30 days. Furthermore, in the case of effective porosity for 90 days, with the individual replacement of OPC by nSO or RHA performance increased when decreasing 5.8 and 29.4% respectively. Meanwhile, decrease for RHA + nSO was 43.5% with respect to reference, being M84.5-15-0.5 the mixture with greater performance due to the synergistic effect that occurs by replacing the OPC by the RHA and nSO simultaneously. Effective porosity presents greater benefits than total porosity at 90 days, suggesting that the presence of RHA and nSO in single or simultaneous use causes

in the pores network fewer spaces interconnected, favoring the increase in durability when decreasing the possible transport routes of aggressive agents for mortar or reinforcement. Several investigations [4,5,24,25] show the favorable effect of the replacement of OPC by RHA or nSO with respect to physical properties, besides paying dividends in mechanical properties.

**Table 5.** Total porosity of hardened mortars [% (% reference)]

Mixture	Age (days)	
	30	90
M100-0-0	21.3 (100.0)	20.5 (100.0)
M85-15-0	22.4 (105.2)	21.2 (103.4)
M99.5-0-0.5	21.1 (99.1) *	20.4 (99.5)
M84.5-15-0.5	22.1 (103.8)	19.8 (96.6) *



**Figure 3.** Effective porosity, 90 days age.

Compressive strength of hardened mortar increases its performance to a major OPC replacement by RHA; in a certain percentage the compressive strength decreases again [47]. The results of the compressive strength of hardened mortars are shown in Table 6. In general, single or simultaneous replacement of OPC by RHA and nSO showed an increase of resistance with respect to M100-0-0. The individual replacement of OPC by nSO at ages of 30 and 90 days, favored the resistance to compression presenting increments up to 6.4%. Moreover, the simultaneous replacement of RHA and nSO was more effective, showing maximum resistance in M84.5-15-0.5. Based on the results, the replacement of OPC by RHA or nSO in single or simultaneous use favors the performance

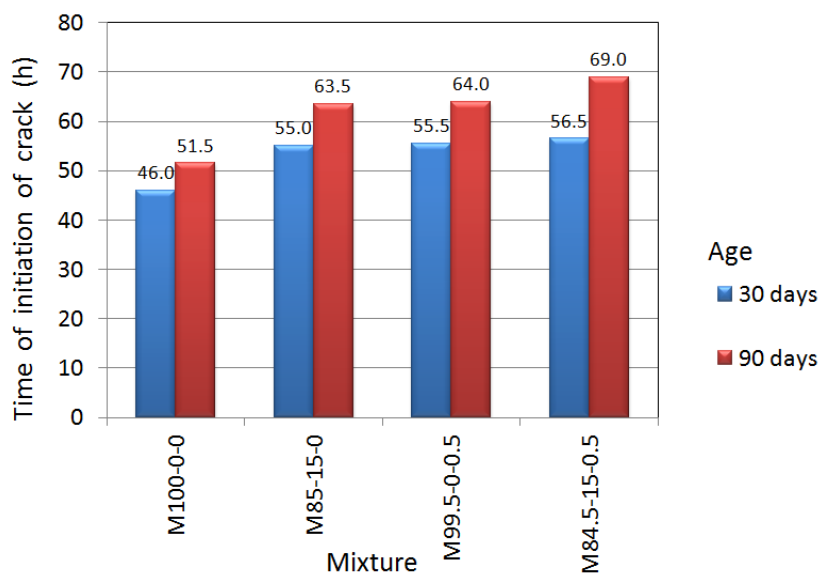
of compressive strength of hardened mortar. In the performance of cementitious materials it is important the performance of the mechanical properties, as is its resistance to corrosion.

**Table 6.** Compressive strength of hardened mortars [MPa (% reference)]

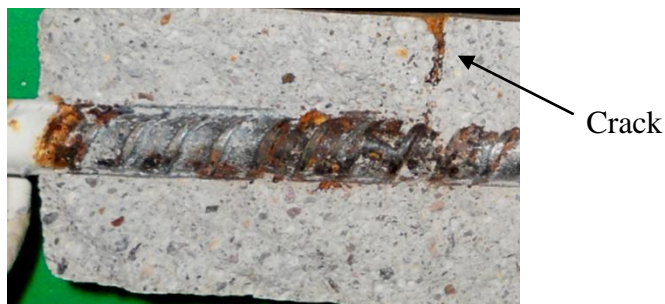
Mixture	Age (days)	
	30	90
M100-0-0	34.4 (100.0)	36.2 (100.0)
M85-15-0	36.6 (106.4)	38.1 (105.3)
M99.5-0-0.5	35.3 (102.7)	37.9 (105.7)
M84.5-15-0.5	36.9 (107.3) *	38.4 (106.1) *

3.4 Accelerated corrosion test with impressed voltage.

The use of rapid tests or accelerated with the application of electric fields to increase the mobility of chlorides in the cement matrix is accepted in the study of reinforced mortars [51], ACTIV provides TIFC produced by corrosion of reinforcing steel in mortars [47]. TIFC results by ACTIV are presented in Figure 8 and ACTIV specimens after ACTIV in Figure 9. In all replacement cases of OPC by RHA or nSO individually or simultaneously, an increase was obtained in TIFC respect to M100-0-0. TIFC increases have been reported up to 89% with the individual use of RHA [47] and the use of RHA + nSO up to 51.1% [39, 40]. At age 30 days, TIFC presented an increase in its performance in all cases, with increases of 19.6 to 22.8%, however at 90 days age the performance increased from 23.3 to 34.0% relative to M100-0-0. The maximum performance was presented with the replacement of OPC by RHA + nSO for both ages, managing to increase 51.5 h. to 69.0 h.



**Figure 4.** TIFC in assay of Accelerated corrosion test with impressed voltage.

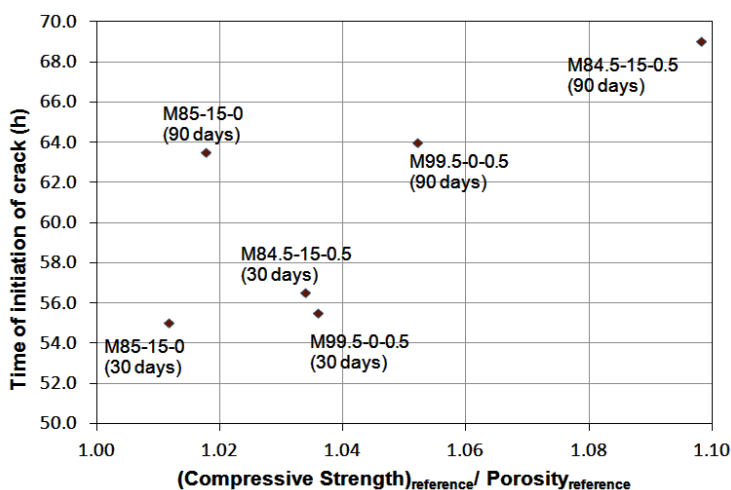


**Figure 9.** Especimens after ACTIV.

The performance at age of 30 days is limited by the presence of SP, at higher ages TIFC is favored presenting increases up to 34% above the reference, which is attributed to the increase in speed of the hydration process. Based on the results, the assay of ACTIV has greater performance with simultaneous replacement of OPC by RHA and nSO, as in the case of porosities in hardened mortar at 90 days of age.

The maximum performance in M84.5-15-0.5 at 90 days of age is attributed to the presence of lower effective porosity and to the maximum mechanical strength that interact in this mixture providing greater resistance to corrosion. Therefore, effective porosity of mortar and compressive strength are key factors to define the corrosion resistance of cementitious materials, on one hand in defining the distribution of the pore network and the resistance to tension forces produced by corrosion products.

The correlation  $(\text{compressive strength})_{\text{reference}}/\text{porosity}_{\text{reference}}$  ( $RC_r/P_r$ ) and TIFC are shown in Figure 10, this relation of variables presents the interaction that porosity and compressive strength have to define the resistance to corrosion or to TIFC where the  $RC_r/P_r$  parameter is generally directly proportional to TIFC. Based on the results, ACTIV provides a parameter of indirect comparison which combines the effect of porosity and compressive strength of the cementitious matrix defining the resistance to corrosion in a reinforced mortar.



**Figure 50.**  $RC_r/P_r$  vs TIFC.

3.5 Rapid chloride penetration test.

The increased in RHA replacement decreases the charge passed of RCPT in a cementitious material [18]. The results of the charge passed are presented in Figure 11, at the age of 30 days the maximum performance is obtained with single replacement of RHA with a decrease of 75.4%, while individual replacement of nSO showed an increase of 13.8%, which is attributed to the effect of SP on the delay in the velocity of the hydration process, in this case RHA + nSO showed a decrease of charge passed of 60.8%. The maximum performance was obtained at 60 days age with individual replacement RHA + nSO with 77.7% decrease in charge passed while the individual replacement of RHA and nSO presented a decrease of 76.5 and 8.7% respectively.

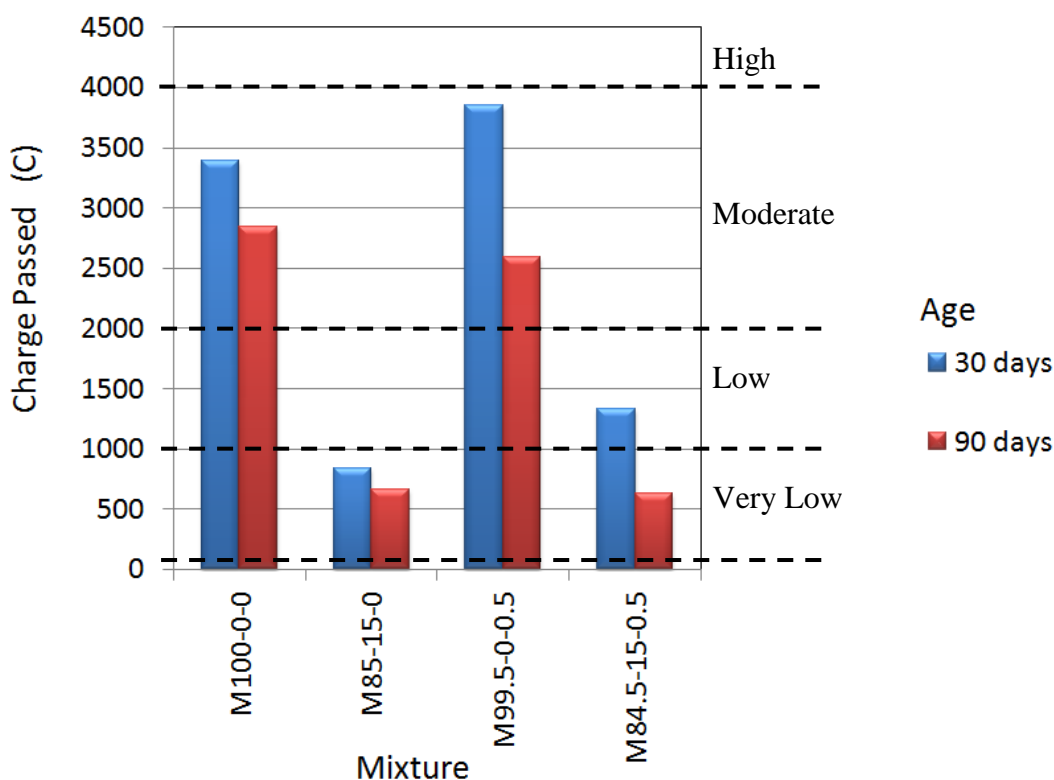


Figure 6. Chloride Permeability according to Charge Passed

Chloride permeability was modified in two mixtures from High to very low, for M85-15-0 at both ages and for M84.5-15.0.5 at 90 days age. Generally the greatest performances are presented in mortars which count with some content of RHA. Based on the results, the assay of RCPT presents greater performance with the simultaneous replacement of OPC by RHA + nSO, as in the case of porosities and ACTIV of hardened mortar at 90 days of age, presenting chloride permeability classified as very low. The decrease of the charge passed at 90 days with the substitution of OPC by RHA and nSO is attributed to the effect of effective porosity in the ions transport through the pores network, with the result of cementitious masses with lower permeability to chlorides.

#### 4. CONCLUSIONS

Results in the present study demonstrate that:

Manufacture of mortars with plastic consistency with replacement of OPC by RHA and nSO is possible with the use of SP and ultrasonic dispersion of nSO. High percentages of the use of SP in the manufacturing of mixtures demerited the performance of the simultaneous use of RHA and nSO in hardened mortars, decreasing the speed of the hydration process.

Replacements of OPC by 15% RHA and 0.5% nSO in simultaneous use in mortars of plastic consistency presents advantages in the performance of physical and mechanical resistance, in the effective porosity, resistance to corrosion and resistance to chloride penetration due to the synergy of RHA and nSO, with respect to individual use of RHA and nSO, managing to reduce chloride permeability from High to very low. These advantages are reflected at higher than 28 days old ages, due to the delay caused by the effect of SP.

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