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Influence of Rice Husk Ash on the Durability of Cement Pastes in Hydrochloric Acid Environment

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Abstract: The use of pozzolanic materials as cementitious additives is one of the ways to reduce CO_2 production during cement production and also improve the durability of cementitious materials. The purpose of the present work is to investigate the influence of Rice Husk Ash (RHA) on the performance of cement pastes in hydrochloric acid environment. For this, ash was produced by calcining rice husk at 680°C for 5 hours to produce reactive pozzolan. The chemical and mineralogical composition of the ash has been assessed by ICP-AES and XRD analyses. The results showed that the obtained ash is rich in amorphous silica with pozzolanic activity index higher than the required minimum value of 75% for pozzolanic materials. The use of RHA as an additive to cement promoted the formation of calcium silicate hydrate (CSH) and calcium aluminate hydrates such as C_4AH_{13} and C_3ASH_6. The presence of RHA in the cement paste improved their resistance to hydrochloric acid attack due on one hand to the formation of supplementary CSH, resulting from the pozzolanic reaction between amorphous silica of the RHA and the portlandite released by hydration of the cement, and another hand by the filler effect of RHA.

Keywords: Pozzolanic Materials, Rice Husk Ash, Cement Paste, Durability, Hydrochloric Acid, Calcium Silicate Hydrate

1. Introduction

Cement is a product needed to carry out construction work. It is the third among the commonly used materials in a building, coming only after steel and aluminum [1]. Unfortunately, its manufacturing generates a lot of energy and causes negative ecological impacts. It has been estimated that the production of one kilogram of cement generates about the same amount of CO_2 [2, 3]. Cement industries are then considered to be among the most atmospheric polluters. Hydration of cement also releases portlandite, which is responsible for the low resistance of mortars. Cementitious structures are also subject to sustainability problems, especially in aggressive environments [4].

To resolve the ecological, energetic, and durability problems, partial substitutions of cement by so-called pozzolanic materials could be undertaken. It has been noticed that the production of pozzolans releases less CO_2 and their grinding requires less energy than clinker. Also, the use of pozzolanic materials in cement or concrete leads to an increase in mechanical strength and sulfate resistance and a decrease in alkali-silica activity, chloride permeability, and hydration heat [5]. The pozzolan in a cement matrix is responsible for a pozzolanic reaction between the portlandite and the amorphous silica of the vitreous phase of pozzolan which leads to an increase in the formation of CSH, hydrates responsible for the resistance of cement products [6].

Among these pozzolanic materials, RHA has been the subject of several studies for its valorization as supplementary cementitious material in concretes and mortars [7-9]. The RHA is a by-product of the calcination of the vegetable raw material of the rice husk. It is mainly composed of silica (80-95%) and has a microporous cell structure which contributes to its pozzolanic reactivity. The use of RHA as a pozzolan in cement reduces the amount of carbon dioxide produced by cement manufacturing. It also improves the mechanical strength of mortars and concretes and greatly contributes to the durability of these materials [7, 9]. However, few works have been devoted to the influence of rice husk ash on the durability of cement pastes in hydrochloric acid environment to our knowledge.

In Burkina Faso, the rice husk obtained after hulling is most often abandoned in the fields and therefore constitutes an environmental waste. To address this storage problem and improve the properties of cement products, rice husks have been valued in this study. The objective of this paper is to evaluate the influence of RHA on the performance of cement pastes in a hydrochloric acid environment. First, the characterization and pozzolanic reactivity of the RHA will be analyzed. Then, some cement pastes will be developed and the effect of the hydrochloric acid solution on these pastes will be investigated.

2. Raw Materials and Experimental Methods

2.1. Characterization of Raw Materials

The RHA (figure 1b) used in this study was obtained by calcining at 680°C a rice husk (figure 1a) taken from the rural commune of Bama, Burkina Faso. The calcination was conducted in a NABERTHERM C250 type furnace with a heating rate of 10°C/min for 5 hours. This rice husk ash has been characterized in our previous studies [10, 11]. However we shall give some results from this characterization.

The cement with chemical and mineralogical composition reported in Table 1, is produced by the Diamond Cement Company from Burkina Faso [12].

The chemical analysis of RHA was performed by ICP-AES. The chemical composition given in Table 2 shows that the RHA consists mainly of silica for 96.84%.wt. This value is slightly higher than the results given in the literature, which indicates a maximum of 96% by mass of silica [8]. RHA is low in alkalis because the sum of the alkaline oxides Na_2O and K_2O, 0.84% is lower than the minimum value of 0.95% given in the literature [8]. The sum of the oxides SiO_2, Al_2O_3, and Fe_2O_3 is greater than the minimum value of 70% set by ASTM C-618 standard [13]. The glass content (SiO_2 - CaO) of 96.37% is well above the minimum value of 34%. These results suggest that rice husk ash is a very reactive pozzolan. The total sum of the analyzed oxides (99.98%) shows that it contains almost no carbon even at a trace level. This low carbon content of RHA will improve its pozzolanic responsiveness. The presence of significant amounts of carbon in the materials causes pore formation, and negatively influences the durability of the developed materials [7].

The RHA diffractogram (figure 2) shows a large bump (halo) centered at about 22° (2θ) indicating the presence of amorphous phases. Silica, the main element of RHA, is then in amorphous form and will give good pozzolanic reactivity to RHA [14].

The SEM image of RHA (figure 3) similar to the one obtained by Guilherme Chagas Cordeiro and al [9] has a layered structure which proves that the RHA has an amorphous character as demonstrated by X-ray diffraction.

To be used as a substitute for cement, the RHA must have a certain so-called pozzolanic activity enabling it to react with the portlandite released by cement hydration. This activity can be followed by chemical, mineralogical, or mechanical tests.

(a) (b)

Figure 1. *(a) Rice husk, (b) Rice husk ash.*

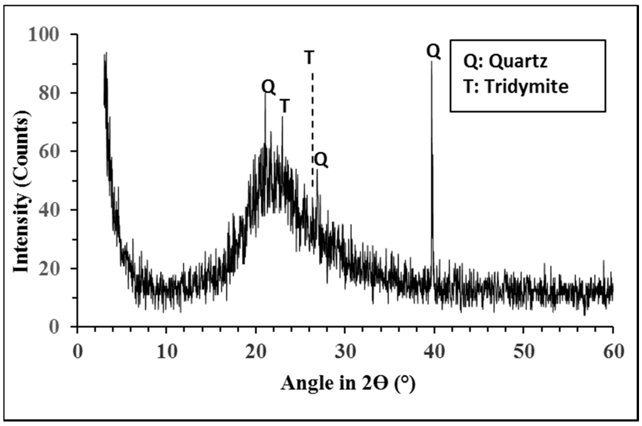

Figure 2. *X-ray diffraction pattern of RHA.*

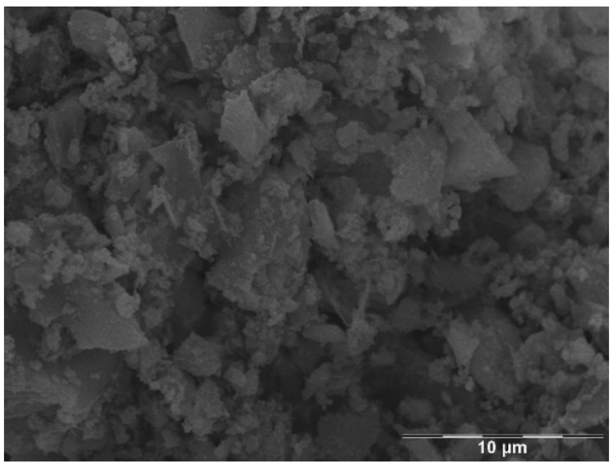

Figure 3. *SEM image of RHA.*

Table 1. *Chemical and mineralogical composition of cement use.*

Composition chimique (%)	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	P_2O_5	SO_3	Na_2O	K_2O	F.L	I.R.	L.O.I.
	20.12	**5.73**	**4.06**	**1.18**	**64.82**	**0.39**	**2.68**	**0.08**	**0.17**	**0.8**	**0.28**	**0.27**
Mineralogie (%)	C_3S 55.7			C_2S 15.68			C_3A 8.31			C_4AF 12.34		

F.L: Free Lime, I.R: Insoluble Residues L.O.I: Loss On Ignition (1000°C).

Table 2. *Chemical composition of RHA.*

oxydes	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	Na_2O	MgO	K_2O	CaO	Total
%	96.84	1.03	0.38	0.1	0.03	0.32	0.81	0.47	99.98

2.2. Pozzolanic Activity

2.2.1. Chemical Method

The Frattini test is the chemical method used to evaluate the pozzolanic activity. It consists of preparing a mixture of 20 g, composed of 80% portland cement CEM-I with 20% of RHA mixed with 100 mL of distilled water. The mixture is kept at a temperature of 40°C for eight (08) days in hermetically sealed plastic boxes. After this, the mixture is filtered through a pore-size filter paper. The hydroxide ions (OH^-) contained in the filtrate are then dosed with a dilute hydrochloric acid solution at 10^{-1} $mol.L^{-1}$ using methyl orange as a color indicator. Then the calcium ions (Ca^{2+}) will be well-dosed by an EDTA solution at 0.03 $mol.L^{-1}$ using Patton and Reeders as a color indicator. The obtained results obtained are represented by a graph giving the Ca^{2+} ions concentration expressed in $mmol.L^{-1}$, according to the concentration of OH^- ion also expressed in $mmol.L^{-1}$ [15].

2.2.2. Mineralogical Reactivity

After the Frattini test, the obtained residues were finely ground and then submitted to physiochemical analysis. Qualitatively, X-ray diffraction, differential thermal and thermogravimetric analysis (DTA/TG) were used to monitor the evolution of pozzolanic reactivity as well as identify the products resulting from pozzolanic reactivity. The used diffractometer is a Bruker AXS, operating at 40 kV-40 mA, using a graphite monochromatic CuKα radiation. The thermograms DTA/TG were recorded using a Linseis apparatus operating under an air atmosphere at a heating rate of 10°C/min.

2.2.3. Pozzolanic Activity Index

The Pozzolanic activity index is the ratio of the mechanical compressive strength at 28 days of the mortars containing RHA and those containing none called reference mortars [13]. The procedure for determining the Pozzolanic activity index consisted of first preparing a reference mortar made of a mixture of 1350 g of standardized sand with 450 g of artificial Portland cement (CPA) and 225 ml of demineralized water. Other mortars were made using the above mixture, substituting 15 to 25% of cement with RHA. All of these were formulated according to standard NF-P-15-403 [16]. The mortars are kept in cold storage and then demolded 24 hours later. They are then maintained in water at 20°C for 28 days to finally be subjected to the test of mechanical compression.

2.3. Diffusion of Chloride Ions in Cement Paste

The chloride ion's diffusion through the cement paste has been evaluated by electrochemical impedance spectroscopy (EIS). The EIS is a reliable tool for analyzing the electrochemical processes that occur between materials and their interfaces with electrically conductive electrodes. The use of EIS as a characterization tool in the study of cementitious materials has been well documented [17-20]. It can be used to study the behavior of fixed or mobile charges through the homogeneous or interfacial parts of solid, liquid, or gaseous materials. The test was performed on parallelepiped test pieces (figure 4) containing various contents of RHA (Table 3). The mortar sample has been kept immersed in HCl solution 1M and then at regular time intervals the electrochemical impedance spectra are recorded for 5 days. The measurements were carried out with a Solartron 1280B potentiostat/galvanostat, controlled by a microcomputer for flexibility in the acquisition and processing of data (figure 5). The explored frequencies ranged between 10 mHz and 20 kHz. The disturbed sinusoidal voltage was fixed to 250 mV. The data acquisition software was Zplot and the processing software of these data was Zview. The effective conductivity of the specimens (σ_{eff}) has been determined from the resistance Rb obtained by the electrochemical impedance spectra in Nyquist representation as indicated in figure 6. The abscissa of the intersection of the two arcs corresponds to the resistance Rb. From this resistance we can deduce the effective conductivity (σ_{eff}) using the equation (1) [21]:

$$\sigma_{eff} = \frac{L}{R_b A} \tag{1}$$

Where L and A are respectively the lengths between the wire and the exposure surface of the sample and the section.

Figure 4. *Images of specimens containing various proportions of RHA.*

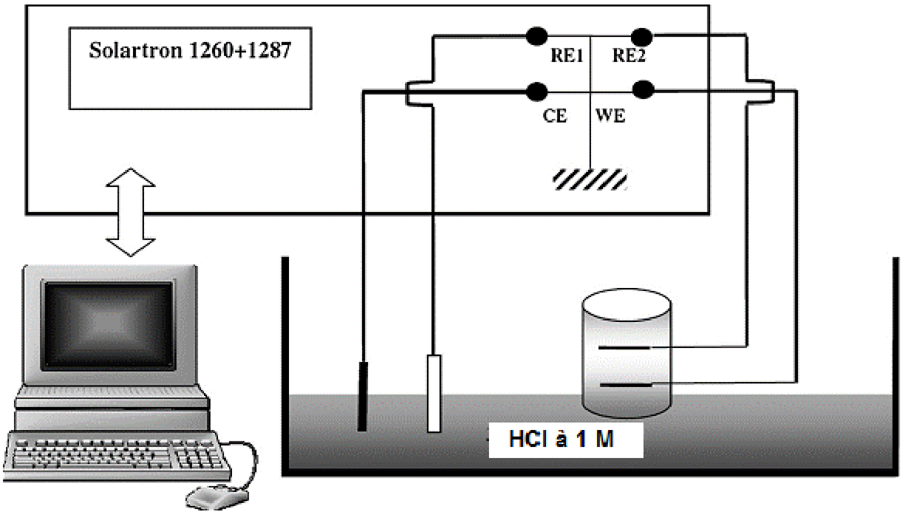

Figure 5. *Electrochemical device use.*

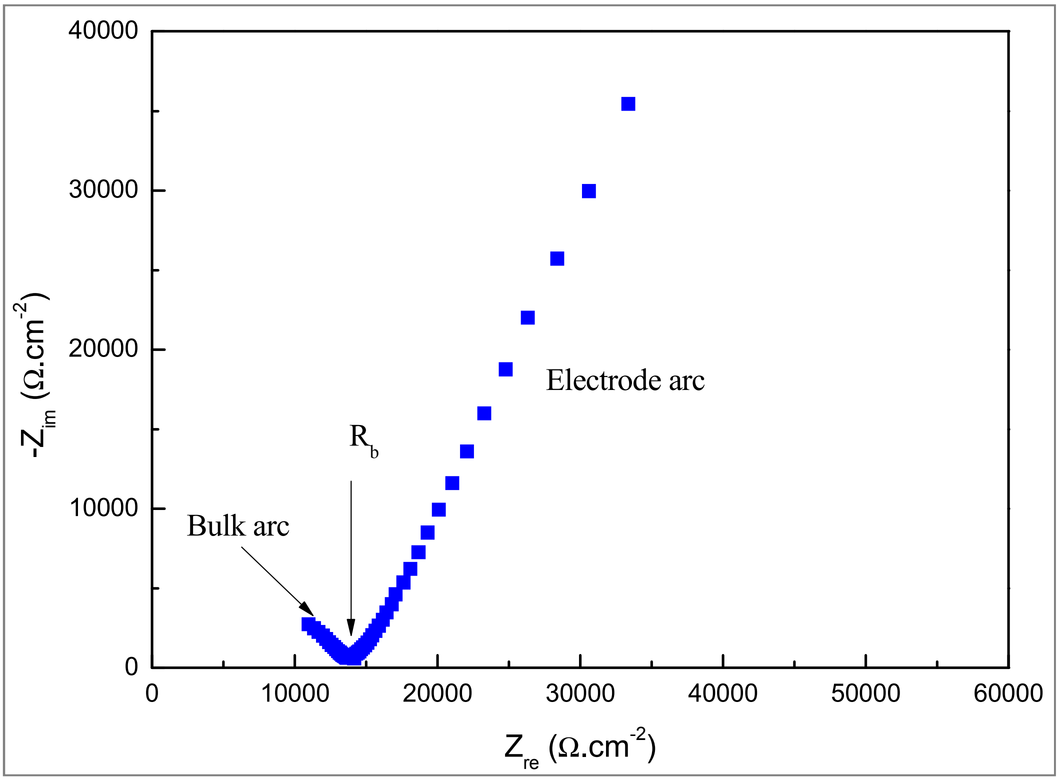

Figure 6. *Nyquist impedance diagram representation.*

Table 3. *Composition of mixtures.*

References	P_0	P_{15}	P_{20}	P_{25}
Cement (g)	50	42.5	40	37.5
RHA (g)	0	7.5	10	12.5
Water (g)	20	22.5	25	27.5

3. Results and Discussion

3.1. Pozzolanic Activity

3.1.1. Frattini Test

The Figure 7 gives the solubility curve of the portlandite and the position of RHA and CPA cement concerning this curve. From this figure, it can be observed that the CPA located above the solubility curve has no pozzolanic activity while the RHA located below this same curve has a pozzolanic character [17, 22].

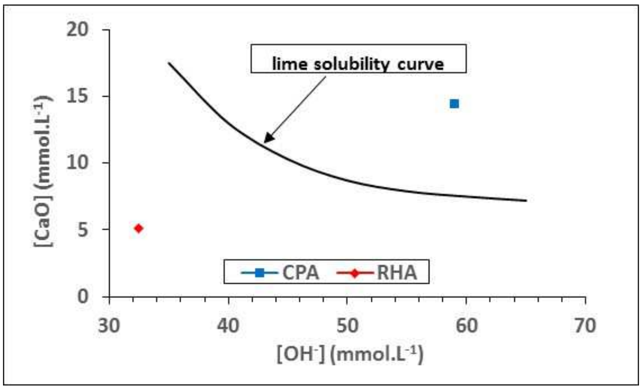

Figure 7. *Frattini test results.*

3.1.2. Mineralogical Reactivity

The reaction of portland cement with water results in the formation of different hydration products. The released portlandite is required for the pozzolanic reaction. This portlandite, in the presence of active pozzolanic materials, can lead to the formation of new hydration products similar to those formed by the hydration of cement. To identify these products, the diffractograms in figure 8 were carried out. The stripping of the diffractograms reveals the presence of portlandite (CH). However with a decrease in the intensity of peaks relative to portlandite in the mixture containing the RHA. This decrease is due to the consumption of portlandite by the RHA by its pozzolanic responsiveness [23]. Also, the presence of ettringite ($C_6A\bar{S}_3H_{32}$) is reported in both diffractograms. Moreover, the addition of RHA to the cement causes the formation of a neoformed phase of hydrated calcium aluminate types of C_4AH_{13} and C_3ASH_6 [22]. Indeed, the presence of these hydrates was only observable in mixtures containing cement and RHA. In addition, the presence of quartz was detected as well. But we notice that its peak was more intense in the cement-RHA mixtures, it is the residual quartz that did not participate in the pozzolanic reaction. Other anhydrous phases of cement such as albite (C_3S) and belite (C_2S) were also present [24, 25].

The XRD allowed us to highlight the pozzolanic reaction from the consumption of portlandite by RHA and the formation of new hydrates. But it remains insufficient for t identification of non-crystallized hydrates such as CSH, hydrates responsible for the resistance of the material which has a nanocrystalline structure, equivalent to an amorphous phase. Therefore, we realized the DTA/TG thermograms to complete the results of the XRD.

The different endothermic peaks observed on the DTA thermograms (figures 9 and 10) can be attributed to a hydrated phase.

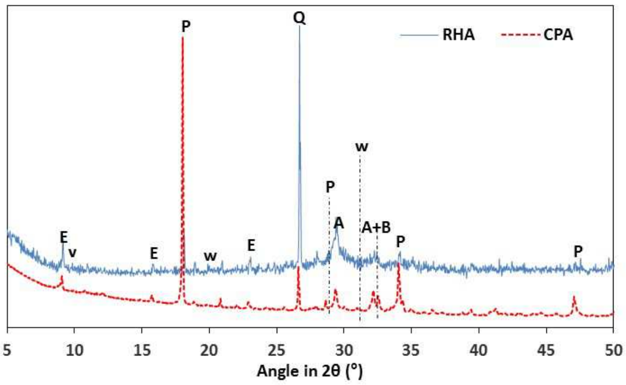

Figure 8. *X-ray diffraction of residues after Frattini test.*

P: portlandite (CH), Q: Quartz (SiO_2), E: ettringite ($C_6A\bar{S}_3H_{32}$); A: Albite (C_3S); V: hydrate (C_4AH_{13}); W: hydrate (C_3ASH_6); B: Belite (C_2S)

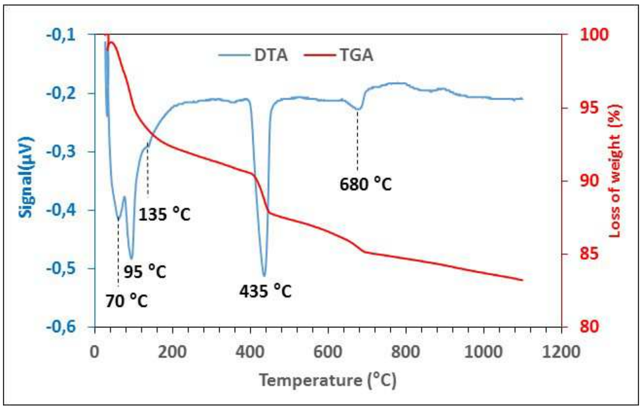

Figure 9. *Thermograms DTA-TGA of residues containing cement after Frattini test.*

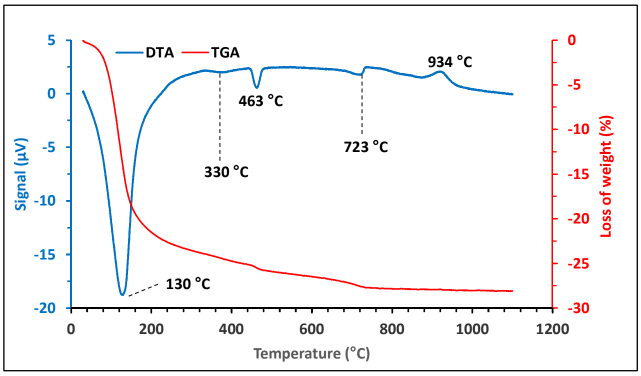

Figure 10. *Thermograms DTA-TGA of residues containing cement-RHA after Frattini test.*

Thus the first two endothermic respectively at 70 and 95°C for the cement alone correspond to the departure of hygroscopic water. The peak around 135°C observed on both DTA thermograms corresponds to the dehydration CSH. This peak is greater in the mixture containing RHA, equally associated with a high mass loss. This suggests a much larger presence of CSH gels in this mixture. The calcium silicate hydrates result from the pozzolanic reaction between the amorphous silica (SiO_2) and the portlandite (CH) released by the hydration of the cement according to the reactions 1, 2 and 3 [10-12]:

Reactions 1:

$$Ca(OH)_2 \leftrightarrow Ca^{2+} + 2\ OH^{-:} \text{ dissolution of portlandite}$$

Reactions 2:

$$SiO_2 \text{ (amorphous silica of RHA)} + 2\ OH- \text{ (hydroxyl of portlandite) } H_2SiO_4^{2-}$$

Reactions 3:

$$xCa^{2+} + H_2SiO_4^{2-} + 2\ (x-1)\ OH^{-} + (y-x)\ H_2O \rightarrow (CaO)_x(SiO_2)(H_2O)_y\ [C_xSH_y]$$

With: $CaO = C$; $SiO_2 = S$ and $H_2O = H$.

The endothermic peak around 330°C indicates the presence of hydrate C_3ASH_6, which shows that the cement-RHA mixture allows the formation of new hydrate as founded by X-ray diffraction. Between 435 and 465°C, we have the peak of dehydration of portlandite. This peak is more intense in the cement than in the cement-RHA mixture, which proves the strong release of portlandite by cement and its consumption by RHA, hence the pozzolanic reactivity of RHA [26]. The fixation of portlandite by pozzolan or the reduction of portlandite in the cement-water-pozzolan system may be due mainly to the dilution effect and the pozzolanic effect. Firstly, the dilution effect is defined as the effect caused by the dilution of the cement in the mixture resulting from the partial replacement of the cement with pozzolan, which results in the release of the portlandite. Then, the pozzolanic effect is defined as the effect caused by the reaction between pozzolan and portlandite released by hydration of the cement to necessarily form CSH gels. Nevertheless, the effectiveness of a pozzolan in such a system is rather dependent of the pozzolanic effect than the dilution effect, since the latter is independent of the nature of the pozzolan used. The peaks at 680 and 720°C are due to decarbonation. That is the formation of calcite following the reaction involving the portlandite produced by hydration of cement and the carbon dioxide of the atmosphere according to the reaction 4.

Reactions 4:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

The only exothermic peak at 930°C at the mixture level could be likened to the presence of tridymite in RHA.

3.1.3. Pozzolanic Activity Index

The Pozzolanic activity index resulting from the partial substitution of 15, 20, and 25% of RHA with cement (figure 11) is greater than the minimum value of 75% required by ASTM C 618 [13]. This is due to the formation of CSH, hydrates which are responsible for the mechanical strength of cementitious materials. RHA, therefore, has pozzolanic properties, so it can be used as a substitute for cement in the formulation of cement paste.

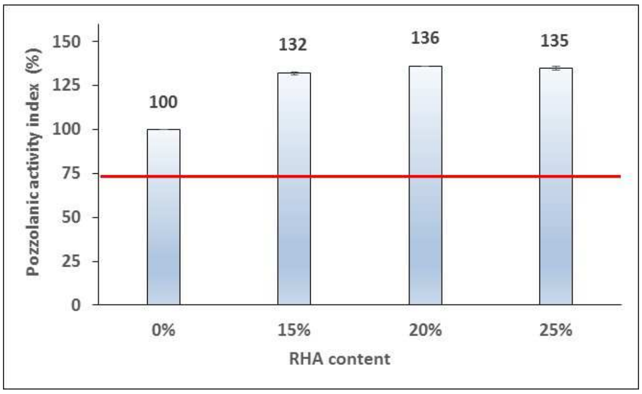

Figure 11. Pozzolanic activity index of RHAs after 28 days for 25% cement replacement.

3.2. Durability of Cement Paste in an Aggressive Environment

The durability of cementitious pastes in an aggressive area was determined by electrochemical impedance spectroscopy. The values of the charge transfer resistances of the materials (R_{mat}) recorded in table 4 were deduced by the Nyquist diagram given in figures 12, 13, 14, 15, and 16. First, it can be seen that the pastes modified with the RHA have better resistances than unmodified paste (P_0). It can therefore be said that RHA played an important role in the resistance of pastes against hydrochloric acid. The low strength of unmodified paste could be due to the conductivity of this material. Indeed the immersion of this material causes the strong release of the portlandite during the hydration of the cement. This release generates the production of Ca^{2+} and OH^- ions, thus making the material more conductive. The presence of portlandite in large quantities has a detrimental effect on the resistance, which makes the material more permeable. Moreover, in the pozzolanic reaction fixing the lime, the capillary pores are reduced by the formation of the CSH gels, thus blocking the absorption of the acid solution, hence the increase of the resistance for all the pastes with additions compared to the paste without addition [27]. Also, the presence of the acid causes the reaction 5 below, allowing the formation of highly soluble calcium salt ($CaCl_2$). These salts are easily removed from the cementitious paste thus weakening the structure of the paste.

Reactions 5:

$$2HCl + Ca(OH)_2 \rightarrow CaCl_2 + 2H_2O$$

In addition, other authors have attributed the resistance to chemical attack of pozzolanic materials to the following phenomena [28, 29]:

(a) pozzolanic materials reduce permeability, thereby preventing the ingress of water and transport of alkali and hydroxyl ions.
(b) pozzolanic materials increase strength and stiffness, resulting in better resistance to cracking and less expansion;
(c) replacing a portion of cement with a less-alkaline pozzolanic material decreases the total amount of alkali present;
(d) pozzolans react with calcium hydroxide to form calcium silicate hydrate with a low CaO/SiO_2 ratio. The formation of CSH depletes portlandite and the low C/S ratio enables the entrapment of alkalis, both of which reduce the amount of hydroxyl ions available to participate in the chemical attack.

The ohmic resistances of the cementitious pastes decrease when the rate of RHA increases. Thus, the paste P_{15} containing 15% RHA has the best resistance at all times of the immersion period. The content 15% then represents the optimal percentage to obtain a better resistance to hydrochloric acid.

Table 4. *Values of the ohmic resistances of the materials (R_{mat}) after immersion in HCl.*

References	Time of immersion				
	1 hour	1 day	3 days	4 days	5 days
	Values of R_{mat} ($\Omega.Cm^2$)				
P_0	-	966	-	-	-
P_{15}	14075	9950	8700	8950	8760
P_{20}	5125	-	6850	7215	6470
P_{25}	4210	4035	3150	3765	3940

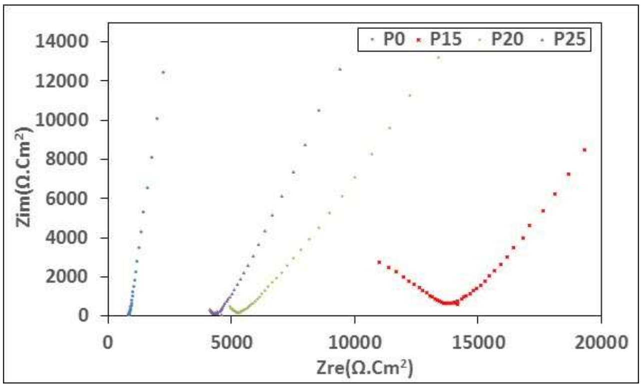

Figure 12. *Nyquist diagram for 1 hour in HCl.*

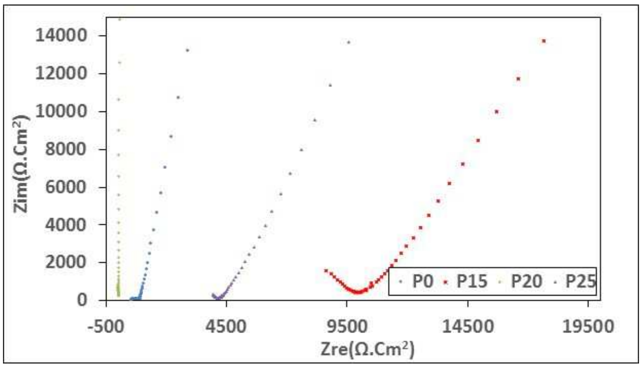

Figure 13. *Nyquist diagram for 1 day in 1 day in HCl.*

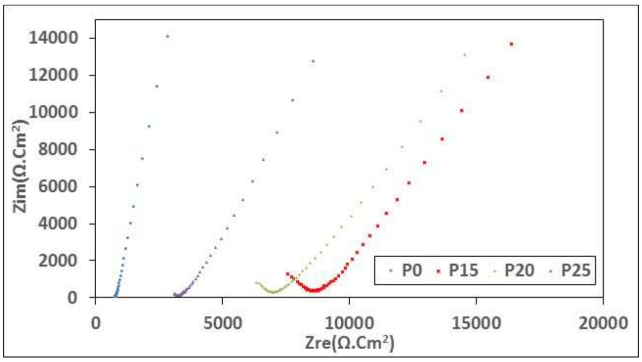

Figure 14. *Nyquist diagram for 3 days in HCl.*

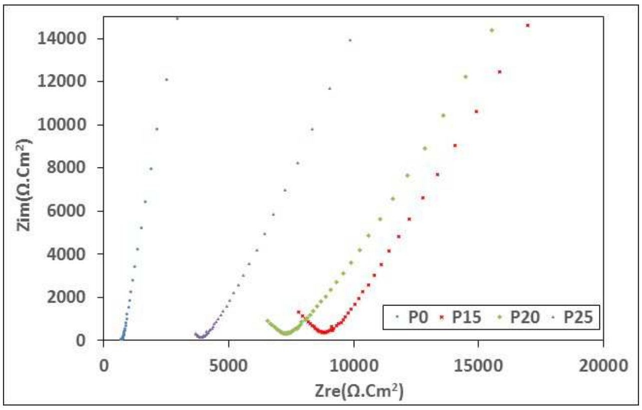

Figure 15. *Nyquist diagram for 4 days in HCl.*

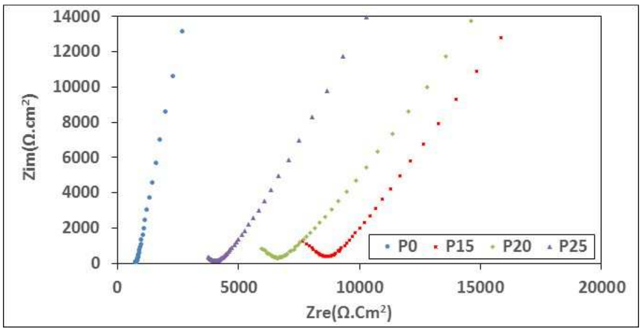

Figure 16. *Nyquist diagram for 5 days in HCl.*

4. Conclusion

In the light of the tests carried out during this work we can draw the following conclusions:

1. The calcination of the rice husk at 680°C for 5 hours has permitted to obtain very reactive ash.
2. The characterization of this ash showed that it is rich in amorphous silica (96.84%) with a high glass content (96.37%) and pozzolanic indices greater than the minimum value of 75%.
3. The partial substitution of the cement by the BRC results in the formation of supplementary calcium silicate hydrate (CSH) and aluminates of C_4AH_{13} and C_3ASH_6 types.
4. The presence of RHA in the cement pastes improves their resistance to hydrochloric acid attack due to the pozzolanic reactivity between the amorphous silica of BRC and the portlandite released by the hydration of the cement.
5. In short, the use of 15% of BRC in the formulation of cement pastes makes it possible to reduce the consumption of cement and improves the resistance to hydrochloric acid.

Conflict of Interest Statement

The authors declare that they have no competing interests.

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