

Study of the Mechanical Behaviour of Mortars Modified with Rice Husk Ash

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How to cite this paper: Sanou, I., Sawadogo, M., Seynou, M., Zerbo, L. and Ouedraogo, R. (2019) Study of the Mechanical Behaviour of Mortars Modified with Rice Husk Ash. *Journal of Minerals and Materials Characterization and Engineering*, 7, 373-384.

<https://doi.org/10.4236/jmmce.2019.76025>

Received: September 2, 2019

Accepted: October 19, 2019

Published: October 22, 2019

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Abstract

The purpose of this work is to study the influence of Rice Husk Ash (RHA) on the mechanical strength of mortars. For this purpose, ash was produced by calcining rice husk at 680°C for 5 hours to produce reactive pozzolan. The chemical and mineralogical composition studied by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) and X-Ray Diffraction (XRD) showed that this ash is rich in amorphous silica. The ash presents a good pozzolanic activity with a lime fixation rate of 100% after only 7 days of treatment. The addition of RHA to cement contributes to the formation of calcium silicate hydrate (CSH) and calcium aluminate hydrate of C_3ASH_6 type. The presence of RHA in the mortars improves their mechanical strength. This improvement is due to the filler effect of RHA and to formation of the CSH resulting from the pozzolanic reactivity between the amorphous silica of the RHA and the portlandite released by hydration of the cement.

Keywords

Mortar, Rice Husk Ash, Mechanical Strength, Filler Effect, CSH

1. Introduction

Cement is a strategic product in the civil engineering for the construction of reinforced concrete structures. But its production generates around 5% of toxic gases such as CO_2 responsible for the ecological environmental degradation of the planet [1]. Moreover the hydration of cement releases portlandite, which is

responsible for the low resistance of mortars against aggressive environment like acid and sulfate solutions. In order to resolve the ecological and durability problems, partial substitutions of cement by so-called pozzolanic materials could be undertaken. A pozzolan is defined as a siliceous and aluminous material which, in itself, possesses little or no cementitious value but which will, in finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties [2]. The presence of pozzolan in a cement matrix causes a pozzolanic reaction between the portlandite of the cement and the amorphous silica of the vitreous phase of pozzolan. This leads to an increase in the formation of CSH, hydrates which are responsible for the resistance of cement products [3] [4] [5]. Among these pozzolanic materials, rice husk ash (RHA) has been the subject of several studies for its valorization as a supplementary cementitious material in concretes and mortars [6] [7] [8]. Rice husk ash is a by-product from the calcination of vegetable raw material of the rice husk. The rice husk obtained after hulling is most often abandoned in the fields and therefore constitutes an environmental waste. In order to address this storage problem and at the same time improve the properties of cement products, rice husks have been valued in this study. The objective of this paper is to study the influence of RHA on the mechanical strength of mortars. An emphasis will be put on the pozzolanic potentialities of RHA before it is used as pozzolan in the mortars formulation.

2. Raw Materials and Experimental Methods

2.1. Characterization of Raw Materials

The rice husk ash used in this study was obtained by calcining at 680°C a sample of rice husk taken from the rural commune of Bama, Burkina Faso. The calcination was conducted in a NABERTHERM C250 type furnace with a rise rate of 10°C/min for 5 hours. The lime used for the saturated lime test consists mainly of calcium hydroxide (95% pure). The cement whose chemical and mineralogical composition is recorded in **Table 1**, comes from the company Diamond Cement of Burkina Faso [9].

The chemical analysis of RHA was performed by ICP-AES. The chemical composition given in **Table 1** shows that the rice husk ash consists mainly of silica for 96.84% by mass [10]. This value is slightly higher than the one given by the literature, which indicates a maximum of 96% by mass of silica [11]. RHA is low in alkalis because the sum of the alkaline oxides Na₂O and K₂O, 0.84% is lower than the minimum value of 0.95% given in the literature [11]. In addition, the sum of the oxides SiO₂, Al₂O₃ and Fe₂O₃ is greater than the minimum value of 70% set by ASTM C-618 standard [12]. The glass content (SiO₂ - 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 trace level. This low carbon content of RHA improves its pozzolanic responsiveness. The presence of

significant amounts of carbon in the materials causes pore formation, and negatively influences the durability of the materials developed [8].

The RHA diffractogram (Figure 1) shows a large bump (halo) centered at about 22° (2θ) indicating the presence of amorphous phases. Silica, the main element of rice husk ash, is then in amorphous form and will give good pozzolanic reactivity to RHA [13].

Before being used as a substitute for cement, the rice husk ash must have a certain so-called pozzolanic activity enabling it to react with the portlandite released by cement. This activity can be monitored by several methods.

Table 1. Chemical and mineralogical composition of the cement and RHA.

Oxides	Chemical composition (%)		Mineralogy of cement (%)	
	Cement	RHA		
SiO ₂	20.12	96.84	C ₃ S	55.7
Al ₂ O ₃	5.73	1.03		
Fe ₂ O ₃	4.06	0.38	C ₂ S	15.68
MgO	1.18	0.32		
CaO	64.82	0.47	C ₃ A	8.31
P ₂ O ₅	0.39	-		
SO ₃	2.68	-	C ₄ AF	12.34
Na ₂ O	0.08	0.03		
K ₂ O	0.17	0.81		
TiO ₂	-	0.1	F.L	0.8
L.O.I	0.27	-		

L.O.I: Loss On Ignition and F.L: Free Lime.

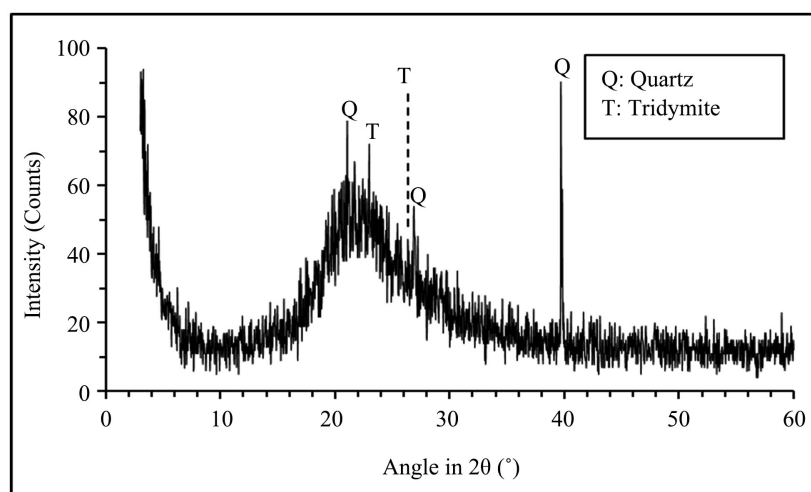


Figure 1. X-ray diffraction pattern of RHA.

2.2. Pozzolanic Activity

2.2.1. Chemicals Methods

The Frattini test and the saturated lime test are the two chemicals methods used to determine the pozzolanic activity. First, the procedure of the Frattini test consists in preparing a mixture of 20 g, consisting of 80% artificial portland cement (CPA) with 20% of RHA added to 100 mL of distilled water. The mixture is kept at a temperature of 40°C for eight days in hermetically sealed plastic boxes. After these eight days, 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 $10^{-1} \text{ mol}\cdot\text{L}^{-1}$ using methyl orange as color indicator. Then the calcium ions (Ca^{2+}) will be as well dosed by an Ethylenediaminetetraacetate (EDTA) solution at $0.03 \text{ mol}\cdot\text{L}^{-1}$ using Patton and Reeders as a color indicator. The results obtained are illustrated by a graph giving the Ca^{2+} ions concentration expressed in $\text{mmol}\cdot\text{L}^{-1}$, which is equivalent to the amount of CaO according to the concentration of OH^- ion also expressed in $\text{mmol}\cdot\text{L}^{-1}$ [14].

The saturated lime test consists in preparing a mixture consisting of 1 g of RHA and 75 mL of saturated lime solution initially prepared by dissolving 2 g of hydrated lime in 1 L of distilled water. The sealed mixture is kept during the test period set at 1, 3, and 7 days. The test is carried out from 10 mL of filtered mixture. The OH^- and Ca^{2+} ions are respectively dosed with dilute hydrochloric acid solution and EDTA using the same procedure as in the Frattini test. Thus, from the quantity of calcium ions contained in the initial mixture, the amount of CaO fixed by the material is determined.

2.2.2. Mineralogical Reactivity

After the Frattini test, the residues obtained were finely ground and then subjected to analysis. Qualitatively, thermogravimetric analysis (DTA/TG) was used to monitor not only the evolution of pozzolanic reactivity but also to identify the products resulting from pozzolanic reactivity. The thermograms DTA/TG are recorded using a Linseis apparatus operating under an air atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$.

2.3. Formulation of Mortars

First the cement, the RHA ground to 80 μm and water (225 g) are introduced into a mixing machine to be mixed for one minute at a speed of 140 revolutions per min. After that, the standardized sand (1350 g) is added and the mixture is then mixed again for 3 minutes at a speed of 280 revolutions per min. The mixture is introduced into $4 \times 4 \times 160 \text{ cm}^3$ prismatic molds and mechanically compacted using a shock wave apparatus. The molds containing the samples are covered with plastic film and stored in the cold storage with a temperature of $20^\circ \pm 1^\circ\text{C}$. Demolding is carried out after a period of 24 hours and the specimens are stored in the laboratory at $20^\circ\text{C} \pm 1^\circ\text{C}$ in a vessel containing water until the day of the test set at 28 days [15]. **Table 2** and **Figure 2** respectively show the composition and the images of the various mortars produced.

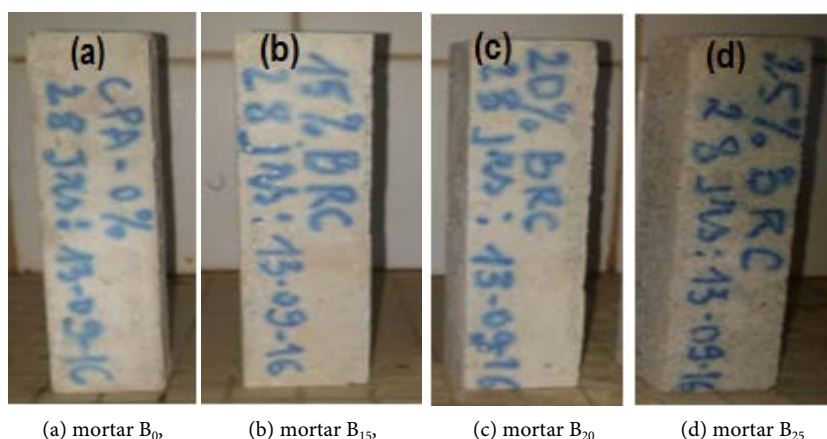


Figure 2. Images of the mortars produced.

Table 2. Composition of mortars.

Mortars	Cement (g)	RHA (g)
B ₀	450	0
B ₁₅	382.5	67.5
B ₂₀	360	90
B ₂₅	337.5	112.5

2.4. Mechanical Strength

The compressive and flexural strength are the mechanical parameters of our mortars that have been evaluated. The flexural strength is carried out on the $4 \times 4 \times 16 \text{ cm}^3$ mortars with a hydraulic press equipped with a 200 kN load cell at a controlled displacement rate of 0.5 mm/min. Equation (1) makes it possible to determine the limit stress in flexural strength [16]:

$$\sigma = \frac{3FE}{2le^2} \quad (1)$$

F is the intensity of the force applied, E is the distance between the two specimen supports, l is the width of the specimens, e is its thickness and σ is the stress at break.

In order to determine the compressive strength, the half-prism resulting from the flexural strength is subjected to a monotonously increasing load until breaking. Thus, the compressive strength is the ratio of the breaking load to the cross section of the specimen. The value of the resistance R_c is obtained from Equation (2):

$$R_c = \frac{P}{S} \quad (2)$$

With: S average value of the section in cm^2 and P the load in kN. The flexural and compression strength tests are carried out in accordance with standard NF P 15-471 [17]. The images examination of mortars was performed with the Hitachi S2500 scanning electron microscopy (SEM).

3. Results and Discussion

3.1. Pozzolanic Activity

3.1.1. Frattini Test

Figure 3 gives the solubility curve of the portlandite and the position of RHA and CPA cement with respect to this curve. From this figure it follows that the CPA located above the solubility curve has no pozzolanic activity. While the RHA located below this same curve has a pozzolanic character [18].

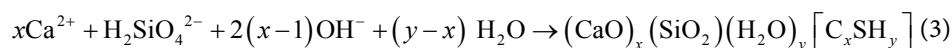
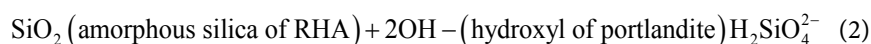
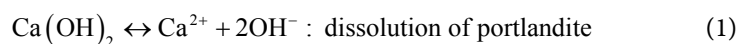
The Frattini test shows that the RHA has a pozzolanic activity. In order to follow the kinetics of the pozzolanic activity of the RHA, the saturated lime test was carried out.

3.1.2. Saturated Lime Test

The results of the saturated lime test are shown in **Figure 4**. At a young age (1 and 3 days), the amount of lime fixed by the RHA is important, even approaching 95% on the third day. This indicates rapid kinetics. From only 7 days onwards, all the lime is fixed by the ash. The high pozzolanic reactivity of this material is mainly due to the combined effect of the high silica content and the fineness of the RHA. According to Massazza, the pozzolanic reactivity is mainly controlled by the composition of active oxides of silica and alumina [19]. However some authors suggest that the chemical composition does not really affect pozzolanic reactivity [20]. Moreover, the increase in its specific surface causes a decrease in the size of its particles, which exposes its upper surface to the chemical reaction thus favoring its pozzolanic reactivity. The amorphous character is also a parameter influencing pozzolanic reactivity. The diffractogram of the RHA (**Figure 1**) by the broad amorphization hump shows that it consists of amorphous phase. According to Walker and S. Pavia, the dominant factor that determines pozzolanic activity is the amorphous character [20].

3.1.3. 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. Thus, after the Frattini test, the residues obtained were finely ground and their DTA/TG thermograms were realized. The different endothermic peaks observed on the thermograms (**Figure 5** and **Figure 6**) can be attributed to a hydrated phase. Thus the first two endothermic respectively at temperatures at 70°C 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 [21]. 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 [4]:



With: $\text{CaO} = \text{C}$; $\text{SiO}_2 = \text{S}$ and $\text{H}_2\text{O} = \text{H}$.

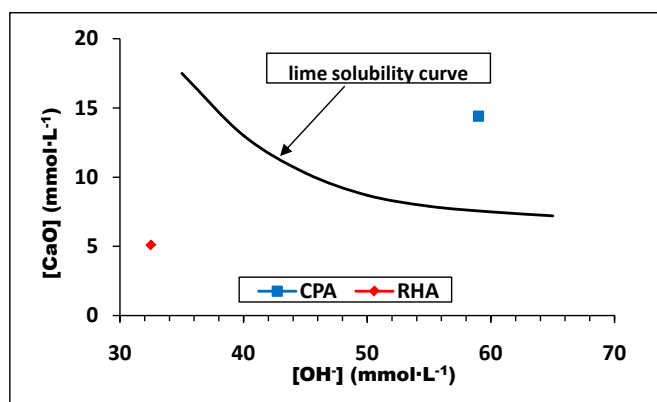


Figure 3. Frattini test results.

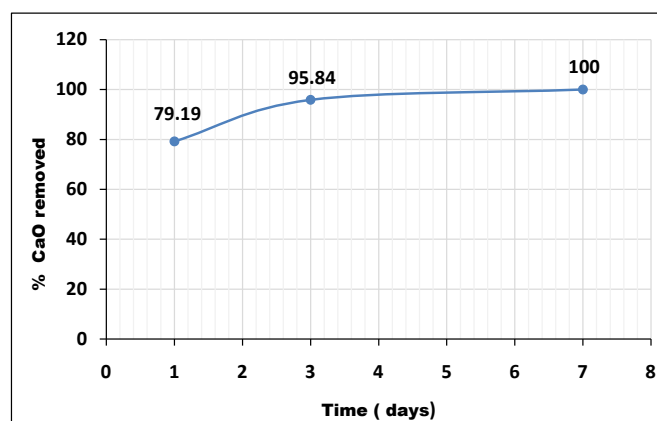


Figure 4. Saturated lime test results.

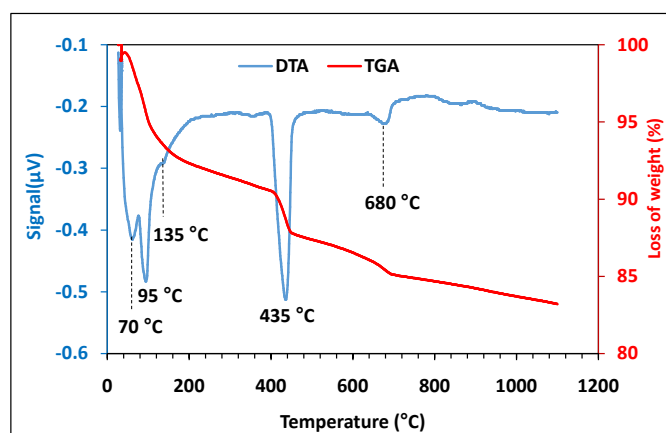


Figure 5. Thermograms DTA-TGA of residues containing cement after Frattini test.

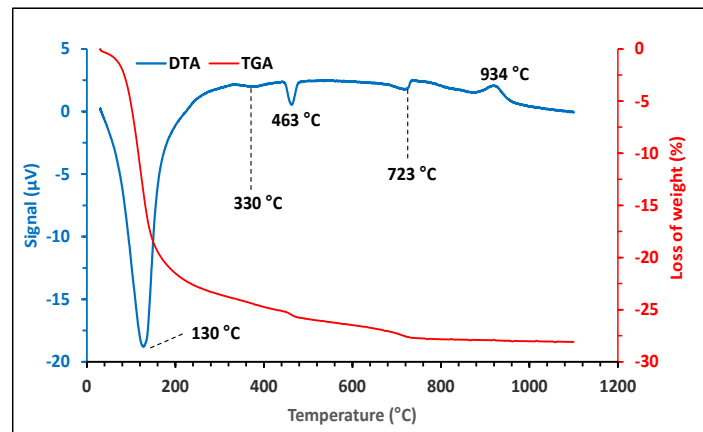
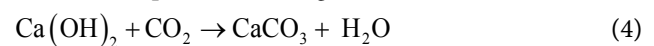


Figure 6. Thermograms DTA-TGA of residues containing cement-RHA after Frattini test.

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. Between 435°C 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 [22]. 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 [8]. 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 in order 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°C and 720°C are due to decarbonation [23]. 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 [10]:



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

Given that RHA has a pozzolanic activity, it can then be used as pozzolan in the production of the mortars.

3.2. Evolution of the Mechanical Strength of Mortars

In order to monitor the development of the mechanical behaviour of the mortars, the flexural and compressive strength of the mortars (Figure 7) was determined 28 days after their formulation. The analysis of Figure 7 shows that the reference mortar B_0 that does not contain RHA has a low mechanical strength

compared to the mortars modified with the RHA. Thus the addition of the RHA contributes to the mechanical strength increase. In addition, the mechanical strength of mortars increases with the rate of RHA up to 20% substitution. With 25% substitution the resistance drops very slightly. The optimal substitution percentage for a better resistance is therefore 20%. This result is in agreement with those obtained by several authors [24]. To have a better understanding of the mortars mechanical behaviour, the SEM images were made on the pieces of mortars obtained after mechanical strength (Figure 8) and then on the powders of the same mortars (Figure 9).

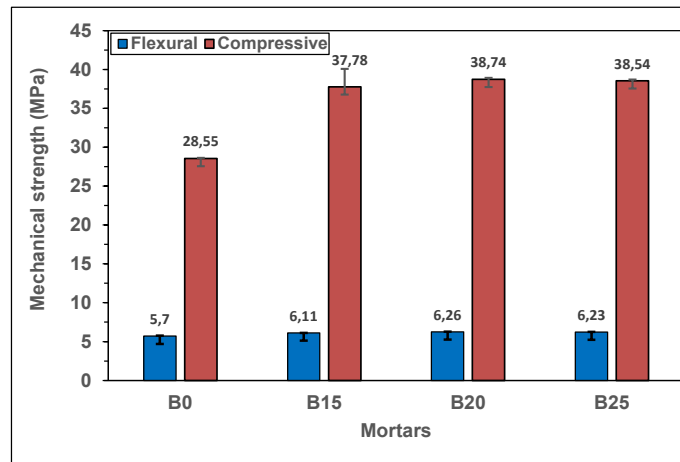


Figure 7. Flexural and compressive strength of mortars after 28 days of curing.

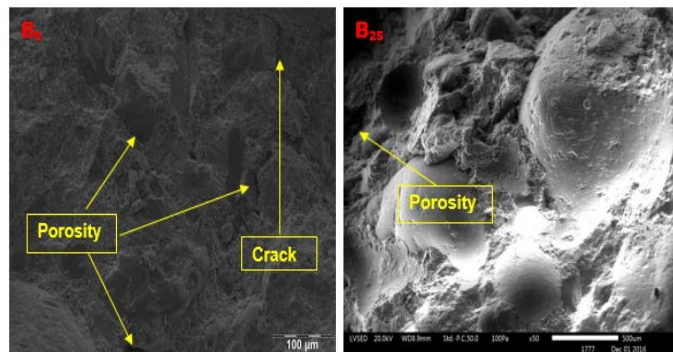


Figure 8. SEM micrographs of mortars B0 and B25.

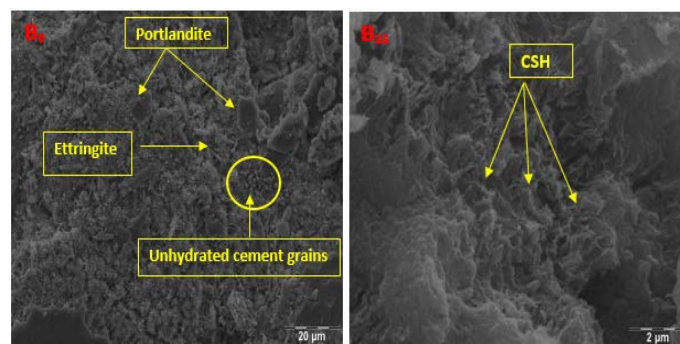


Figure 9. SEM images of mortars B0 and B25 powders.

The SEM image of the B₀ mortar (**Figure 8**) shows a material with a few pores and cracks. On the other hand, the image of mortar B₂₅ shows a fairly homogeneous material having less pores and cracks than B₀. As for the powders (**Figure 9**), some hydration products of clinker compounds such as portlandite (CH) and ettringite (C₆A $\overline{\text{S}}$ H₃₂) as well as unhydrated cement grains on B₀ may be observed. However, CSH is present in the form of honeycombs in the B₂₅ mortar.

These observations provide indication that the improvement of the mechanical properties of the mortars could be due to the filler effect of RHA and the presence of CSH. First RHA can be inserted between the grains of the cement because of its fine size, which reduces the pores and so makes the material more compact and resistant [4]. Moreover, according to several authors, the mechanical strength of cementitious materials is due to the presence of CSH [4] [10] [21]. Indeed, CSH results from the pozzolanic reaction between the portlandite released by the cement and the amorphous silica of the RHA. Their presence makes the material homogeneous with fewer pores.

4. Conclusion

The calcination of rice husk at 680°C for 5 hours has permitted to obtain ash rich in amorphous silica (96.84%). This RHA has a pozzolanic activity with a lime fixation rate of 100% after only 7 days of treatment. The partial substitution of the cement by the RHA results in the formation of calcium silicate hydrate (CSH) and aluminates of C₃ASH₆ types due to the pozzolanic reactivity between the amorphous silica of RHA and the portlandite released by the hydration of the cement. The presence of RHA in the mortars improves their mechanical strength due to the filler effect of RHA and the presence of CSH.

Acknowledgements

We are grateful to Younoussa Millogo of University Nazi Boni of Burkina Faso for his implication in the characterization of samples and for his advice during the writing of this article.

Conflicts of Interest

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

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