

RECOVERY OF ASH FROM LIGNOCELLULOSIC WASTE IN THE CEMENT INDUSTRY

BRIKI, K.^{1*} – BENZEROIAL, B.² – ZIDANI, K.³

¹ *Department of Civil Engineering, University of Batna 2, Batna, Algeria.*

² *Department of Geography and Territorial Planning, University of Batna 2, Batna, Algeria.*

³ *Department of Mechanical Engineering, University of Batna 2, Batna, Algeria.*

**Corresponding author*

e-mail: l.briki[at]univ-batna2.dz

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Abstract. In this research we have targeted three objectives: an economic objective of minimizing energy expenditure, a technological objective of manufacturing a new cement by improving the mechanical performance of ordinary Portland cement, and an ecological objective of minimizing the resulting CO₂ emissions from the decomposition of carbonates during cooking. To achieve these objectives, we have developed a new cement composed of a partial substitution of clinker with artificial pozzolans rich in silica, obtained by treatment of lignocellulosic residues, in this case rice husk ashes. This substitution is added to the clinker with percentages ranging from 25 to 75%. These substitutions were chosen based on the presence of silica that can react with portlandite (Ca(OH)₂) resulting from the hydration of the cement, forming more C-S-H which improves the mechanical properties of the cement. The pozzolanic activity of these substitutions was examined by chemical analyzes and mechanical tests on mortar test pieces. Other techniques, namely X-ray diffraction (XRD), were used to visualize the pozzolanic power of the substitution used. Improving this reactivity is obtained by calcining these additions at temperatures of 750°C. This significantly reduces the CO₂ emissions that accompany the production of Portland cement clinker.

Keywords: *eco-cements, resistance, mortar, rice husks, compression, bending*

Introduction

For many years, Portland cement has been the most widely used building material in the world because of its mechanical performance, fire holding and competitive cost. The cement is then a hydraulic binder in the form of a very fine, non-metallic and inorganic powder. When water is added, it forms a more or less fluid binding paste that hardens both under water and in the air thanks to the agglomeration of solid inert materials. This property is due to the formation of calcium silicate hydrates resulting from the water mixing reaction with the cement constituents. However, its manufacture is very energy-intensive and emits a significant amount of carbon dioxide (CO₂) well known for its impact on the greenhouse effect (Collins, 2010; Habert and Roussel, 2008; Flower and Sanjayan, 2007; Parrott, 2002). This CO₂ comes from the expenditure of energy to produce very high temperatures and the decarbonization of limestone (CaCO₃) in lime (CaO) and carbon dioxide (CO₂). CO₂ emissions from the cement industry were estimated at 5-7% of global emissions with 0,9 tons of CO₂ emitted into the atmosphere to produce one ton of cement (Benhelal et al., 2013). With the recent increase in global warming, attention has increased to concerns about greenhouse gas emissions associated with cement production. The challenge for the cement industry is to produce efficient cement at a competitive cost with minimal environmental impact: the production of one

ton of cement requires 1,65 tons of raw materials, the difference evaporates into the atmosphere and about 1 ton of CO₂ is released for every ton of charred limestone (Hendriks et al., 1998).

The cement manufacturing process begins with the decarbonization of calcium carbonate (CaCO₃) at around 900°C, which releases calcium oxide (CaO) as well as carbon dioxide (CO₂). The cement industry has a very large environmental impact, which it is committed to reducing. Indeed, the clinkerization during which calcium oxide reacts at high temperature (1450-1500°C) with silica, alumina and ferrous oxides to form silicates, aluminates or calcium ferrites. These four compounds must be dosed judiciously. When combined at high temperatures, they form the clinker (JRC, 2000). This represents 6% of man-made CO₂ emissions and could exceed 10% by 2050. In addition to consuming a lot of energy, the cement process requires large quantities of raw materials. It takes 1,7 tons of raw materials, including 4/5th of limestone and 1/5th of clay, to make 1 ton of clinker (Gineys, 2011). These materials come from quarries that follow several regulations designed to limit the discomforts induced by extraction: compliance with environmental protection standards, watering of traffic tracks to reduce dust, proximity to the quarry near the cement kiln for transport, quarry rehabilitation plan at the end of the operation. In order to limit the consumption of non-renewable extracted resources, cement producers replace part of the clinker after cooking with secondary products from other industries without altering the quality of cement such as blast furnace dairy, fly ash and plaster recycling gypsum. However, it is important to ensure that waste recovery in cement plants is done in an environmentally sound manner, while maintaining an impeccable quality for clinker and cement products. Whenever possible, these must be concentrated and recycled by appropriate technical means.

It is therefore important and topical to study the possibilities of upgrading materials obtained by treatment of lignocellulosic residues that can reduce pollution related to cement production in order to develop eco-cements that respect the environment. One of the alternatives to reduce the negative impact of the cement industry on the environment is to partially replace the clinker in Portland cement with pozzolanic materials to produce compound cements. These pozzolanic materials are either natural materials such as natural pozzolan (Benkaddour et al., 2009), thermally treated materials such as metakaolin (Rikioui et al., 2011), or industrial by-products such as silica fumes and fly ash (Hosseini et al., 2011; Ayrinhac, 2005), as well as coal mash (Kurama and Kaya, 2008; Cheriaf et al., 1999), silica fumes (Habert, 2014; Kadri et al., 2011), flying ash (Mardani-Aghabaglou et al., 2014; Amato, 2013; Wangchuk et al., 2013), charred clay (Mohammed et al., 2016) and limestone (Holcim Official Portal, 2015). These mineral additions, composed mainly of either silica or silica and alumina, exhibit a certain chemical activity called "pozzolanic" that allows them to react with lime to form compounds similar to cement hydrates. In addition to reducing CO₂ emissions, the use of pozzolanic materials in cement offers several other benefits such as reducing the cost of cement due to the substitution of a part of the clinker that is expensive by natural pozzolan or less expensive and greener industrial by-products. Bajare et al. (2013) do show that the use of 20% crushed mash as a partial substitution for CEM I reduces the cost of conventional concrete with Portland cement by 9,3%. In addition, these pozzolanic materials contribute to the improvement of the mechanical characteristics of concretes through the development of the pozzolanic activity (Aruntaş et al., 2010).

These advantages open up the possibility of converting large quantities of industrial by-products into sustainable, competitively costed building materials.

Early studies of mineral additions in Portland cement focused on the pozzolanic effect, the optimization of the substitution rate, the impact of mineral additions on the properties of fresh and hardened concrete. In recent studies, efforts are increasingly focused on sustainability. The latter is very important, as previous work has shown a reliance on the mineral composition of binders and environmental conditions. Today, it is accepted that silica and alumina in the glass phases are reactive (Semcha, 2006). The incorporation of the rice husk as reinforcement in a cement matrix has been the subject of some work, notably those of Morsi (2011) as well as those that essentially summarize the work of Julian Salas Serrano at the Eduardo Torroja Institute (Spain) (Salas, 1986; 1985). The use of rice husks without calcination in combination with a mineral binder has been little studied. Only a few works open up prospects for the upgrading of this by-product in the manufacture of lighted mortars based on Portland cement (Serrano et al., 2012; Jauberthie et al., 2003; Tamba et al., 2000; 2001; Salas, 1986; 1985; González De La Cotera, 1982). However, most of this work involves a small fraction of rice husks in the matrix and mineral aggregates are sometimes retained. In the building sector, ash from the burning of rice husks has been the subject of much research (Xu et al., 2015; Ganesan et al., 2008; Jauberthie et al., 2000). Rice husk is characterized by a lower organic matter content than most other lignocellulosic resources since it contains about 20% amorphous silica concentrated mainly on its outer surface (convex) (Johar et al., 2012; Jauberthie et al., 2000). As a result, when the rice husk is charred above 500°C, the organic matter disappears and gives way to a very silica-rich nanometric ash (SiO₂). The ashes contain 95% silica and develop a very high pozzolanic reactivity (Xu et al., 2015; Jauberthie et al., 2000). They can therefore be used as a pozzolanic filler in Portland cements to improve the mechanical performance of ordinary concretes in the same way as fly ash or silica fumes.

Characteristics of rice husk ash

Chemical composition

The organic composition of the rice husk is quite similar to that of the majority of lignocellulosic by-products. The particularity concerns the presence of ash (15 to 25%) contain a high amount of silica (*Table 1*).

Table 1. Chemical composition of the rice husk.

Cellulose (%)	Hemicelluloses (%)	Lignin (%)	Silica (%)	Extractables (%)
25-35	18-21	26-31	15-25	2-5

Sources: Tran et al. (2014); Mansaray and Ghaly (1999)

Mineral composition

Differential thermal analyses were conducted by Briki et al. (2022) on the rice ball. He observed that a volatile material was released up to 360°C. From 525°C, crystalline phases have begun to be seen, but the net transformation of amorphous silica into crystalline silica takes place at around 750°C.

Specific surface

The presence of carbon in ash, also dependent on calcination conditions, increases the specific surface area since the carbon is also very porous. This was demonstrated by Bui (2001) when he charred rice husks at different temperatures and combustion times (Table 2). The same conclusion was reached by Sugita and Shoya (1992) following the measurement of specific surfaces after a grinding of 10, 40 and 80 minutes (Table 3) (Bui et al., 2005; Sugita and Shoya, 1992).

Table 2. Specific surface and amount of carbon present in ash samples from rice husks obtained under different conditions.

Temperature (°C)	Duration (h)	Fire loss (%)	Specific surface (m ² /g)
400	24	6.25	76
500	20	4.86	57
600	6	27.15	131

Table 3. Specific surfaces after different grinding times.

Sample (S)	Grinding time (h)	Specific surface (m ² /g)
CBR (0)	0	123
CBR (14)	14	151
CBR (18)	18	137

Pozzolanic activity

The pozzolanic reaction was found to increase with an increase in the amount of amorphous silica and an increase in the specific surface. In addition, Sugita and Shoya (1992) indicated that the relationship between pozzolanic activity and grinding time was the same as the relationship between the specific surface and the grinding time.

Materials and Methods

Preparation, analysis and characterization of the materials used

Cement

The CPA cement used is type CEM I 42.5 N without any addition (95% clinker with 5% gypsum), produced by the GICA group of Ain-Touta located in Batna city (Algeria) and compliant with the standard NF EN 197-1 (NF EN 197-1, 2000) whose clinker is produced and ground in conjunction with gypsum by the GICA group. This cement is used for the formulation of mortars by setting the ratio E/C equal to 0,5. The dosage of natural gypsum (dehydrated calcium sulphate, CaSO₄. 2H₂O) was kept constant at 5% (Table 4). The chemical compositions of the gypsum are given by the Table 5. The cement components according to Bogue's formula are in Table 6. The chemical composition, successively determined by X-ray fluorescence (Table 7). The cement size curve is presented in Figure 1. Analysis by X-ray diffractometry of the anhydrous CPA (Figure 2). Physical characteristics are mentioned in Table 8.

Table 4. The physical properties.

Category	Number
Apparent volumetric mass (g/cm ³)	1100
Absolute volumetric mass (g/cm ³)	3190
Fineness of grind (cm ² /g) [Specific surface]	4200
Density	3138

Take time: Start (hours)	3138
Take time: End (hours)	2h:12
Physical properties	3h:08

Table 5. Chemical composition of gypsum.

Elements (%)	Gypsum
SiO ₂	8.50
Al ₂ O ₃	2.54
Fe ₂ O ₃	1.04
CaO	29.32
MgO	3.07
SO ₃	36.53
K ₂ O	0.53
Na ₂ O	0.03
CL	0.008

Table 6. Mineral composition (Bogue method).

Cement	CPA-CEM 1
C ₃ S	57.83
C ₂ S	16.75
C ₃ A	8.03
C ₄ AF	10.92

Table 7. Chemical composition (% mass) of the cements used.

Cement	CPA-CEM 1
SiO ₂	19.82
Al ₂ O ₃	5.33
Fe ₂ O ₃	3.75
CaO	63.83
MgO	1.16
SO ₃	0.67
K ₂ O	1.07

Table 8. Physical properties.

Cement	Resistance					
	Compression			Bending		
	2D	7D	28D	2D	7D	28D
CPA-CEM 1	31.70	47.05	50.30	5.9	7.7	8.85

Al ₂ O ₃	1
SiO ₂	94.789
Fe ₂ O ₃	0.650
MgO	0.130
K ₂ O	0.300
Cl-	0.030
SO ₃	0.080

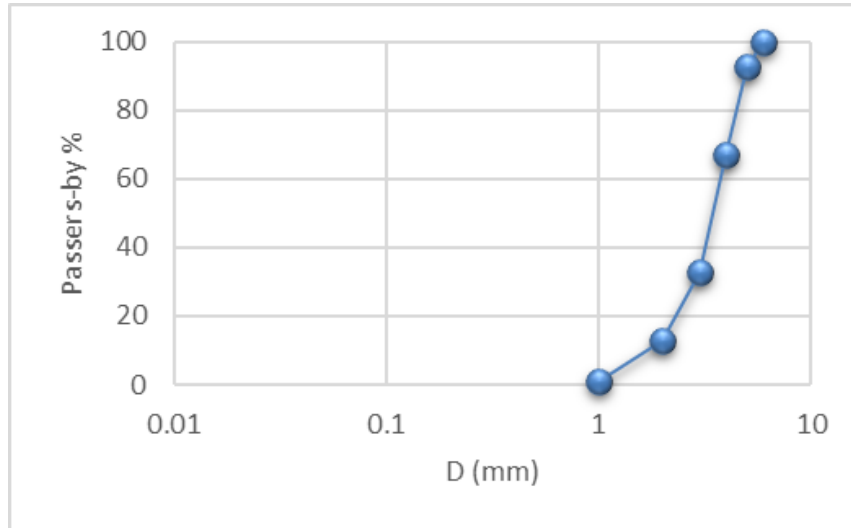


Figure 3. Standardized sand granulometric analysis.

Mixing water

It complies with standards NF P 18-325 and NF P 18-303 (Table 11).

Table 11. Physical-chemical composition of mixing water.

Setting	Content
Calcium (Ca)	99.21
Magnesium (Mg)	44.72
Chlorides (Cl)	97.71
Bicarbonates (CO ₃ H)	285.86
Sulphates (SO ₄)	122.84
Sodium (Na)	/
Potassium (K)	/
Residual Chlorine	/
Nitrates (NO ₃)	8.24
Organic materials	0.00
Oxygen (O ₂)	2.5
Free CO ₂	/
Fe ²⁺	0.00
PH	7.46
Hardness	42.64
Temperature	19,3
Residual chlorine	/
Conductivity	1036
Nitrite	0.00
Ammonium	0.00
Orthophosphate	0.00

Rice husk ash

In order to measure the water content, a certain number of husks were placed in a pan and the evolution of the mass was observed (*Table 12*). The rice husk used therefore has a water content of 16,5%. And in order to know the fire loss of the rice husk, a quantity of 45 grams of husks was weighed after each hour spent in an oven at temperatures ranging from 200 to 1100 degrees Celsius (*Table 13* and *Figure 4*). The pozzolanic activity of ash is determined by the Chapel test, which was performed twice on each of the charred samples at a given temperature (*Figure 5* and *Figure 6*). The average values are shown in the following *Table 14*. The mineralogical composition of the ash was determined by XRD. The XRD was carried out on three samples of rice husk ash calcined at different temperatures: 600, 700 and 800 °C (*Figure 7*, *Figure 8* and *Figure 9*). The average density obtained for the ash used is 2635 g/cm³ for the ash obtained under the chosen conditions. The granulometric curve for rice husk ash is shown in *Figure 10*. The specific surface has been determined by the Blaine method (EN 196-6) and it is 16455 cm²/g. Burning bulls made of rice husks (80% and 70%) clay (20% and 30%) was only tested by Villar-Cociña et al. (2003) and the result was an ash containing 57.30% to 62.69% silica, 14.83% to 10.36% from Al₂O₃, 8.60% to 6.11% from Fe₂O₃ and a fire loss of 2.04% to 14.21% (Jaturapitakkul and Roongreung, 2003; Villar-Cociña et al., 2003; Bui, 2001; Hernández et al., 1998).

Table 12. Water content.

Duration in the oven (h)	Weight crystallizer (g)	Total weight (g)	Rice husk weight (g)	Water content (%)
0	79.41	89.41	10	0
12	79.41	88.6	9.19	8.1
24	79.41	87.77	8.36	16.4
72	79.41	87.76	8.35	16.5

Table 13. Fire loss.

Calcination temperature (°C)	Fire losses (%)
200	8.26
300	50.78
400	62.46
500	73
600	73.34
700	73.76
800	73.04
900	74.72
1000	77.06
1100	77.62

Table 14. Chapel test results.

Starting pH of the solution	Addition of HCl 0.1N (mL)	Free time remaining (g)
500	309	0.7725
600	320	0.8
700	310	0.775
800	330	0.825
900	350	0.875

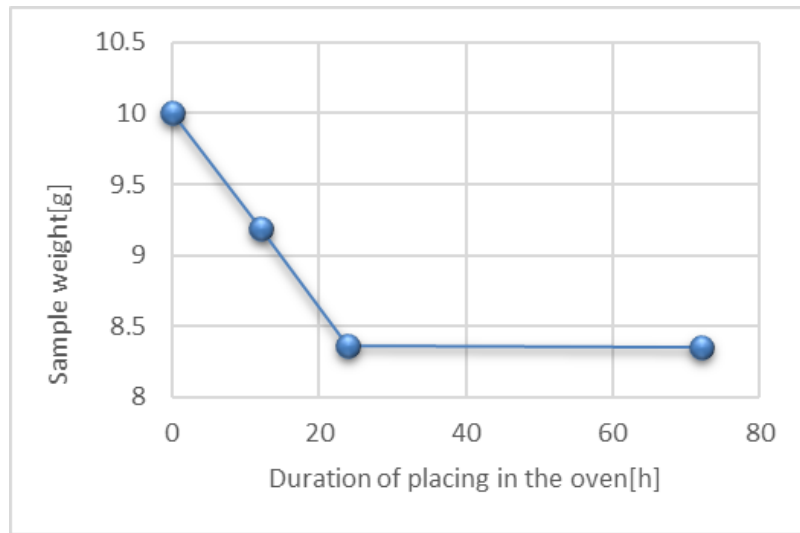


Figure 4. Water content within the rice husk.

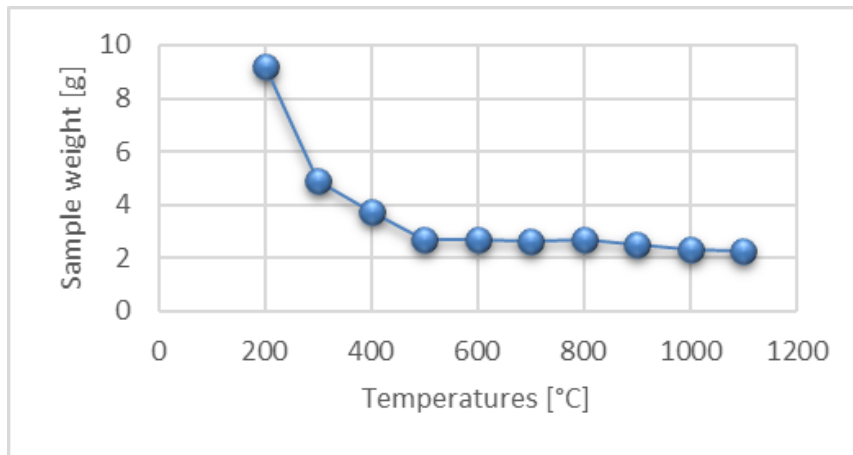


Figure 5. Fire loss of rice husk depending on temperature.

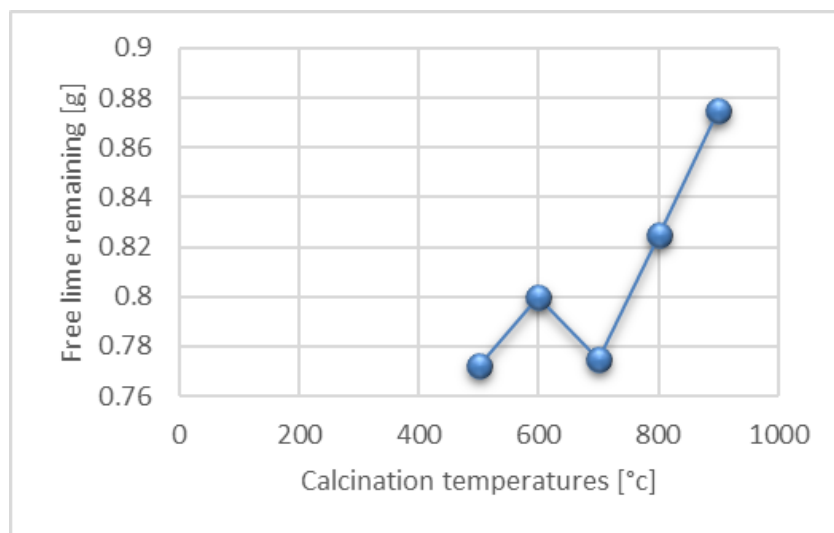


Figure 6. Chapel test: Remaining free lime depending on temperature.

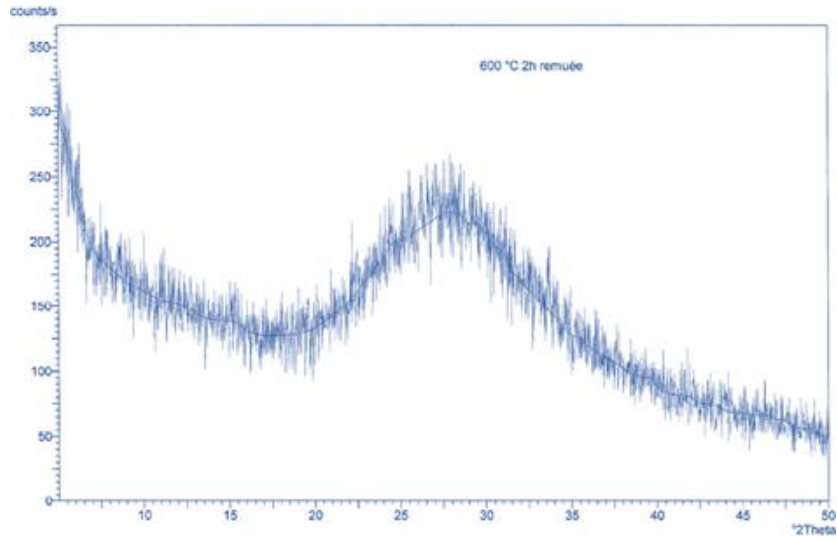


Figure 7. DRX for rice husk ash (600 °C).

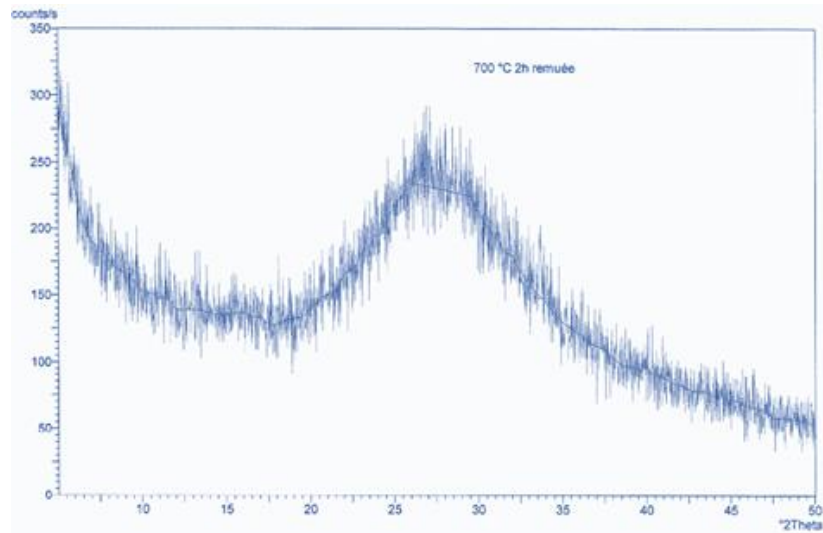


Figure 8. DRX for rice husk ash (700 °C).

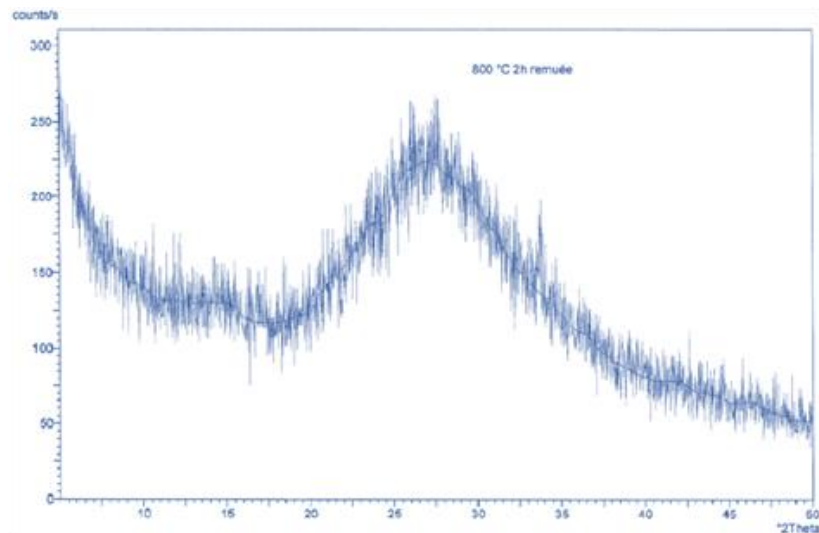


Figure 9. DRX for rice husk ash (800 °C).

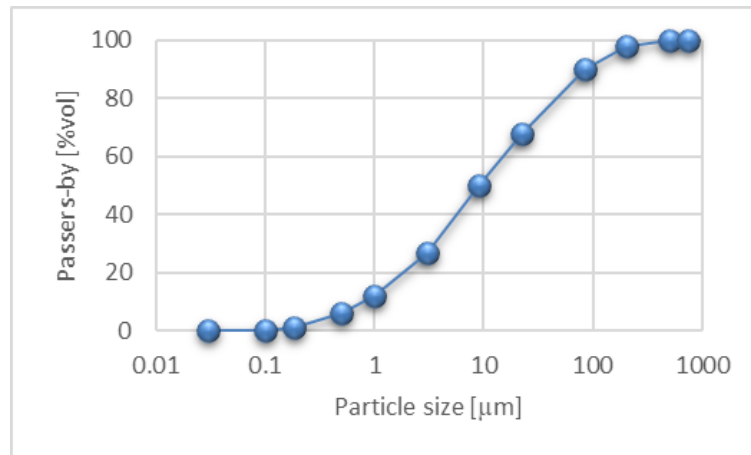


Figure 10. Granulometry of the ash of the rice husk.

Composition of mortars

The mortars prepared in this experimental work meet the European standard NF EN 196-1. For each of them, 450g of binder and 1350g of sand were used, i.e. a C/S=1/3 ratio. The water/binding ratio of all the mortars implemented was kept constant equal to 0,5 for each of them. An additional amount of water in addition or less can be replaced in some compositions to obtain sufficient workability. Of the four blends, a first is composed only of Portland artificial cement CPA, it serves as a reference in this experimental program and three were formed with a substitution of clinker by 25%, 50% and 75% of rice husk ash CBR1, CBR2 and CBR3 respectively. The mass proportions of the various cement constituents are mentioned in *Table 15*. In the anhydrous state, cements underwent chemical treatments to determine density as well as density mass and SSB tests according to standards EN 196, NF EN 196-1 and EN 196-6 respectively. The physical properties studied are: the normal consistency and start-and-end time determined by Vicat's test of pure compound cement pulp used in accordance with NF 196-3. The mechanical properties studied in prepared mortars are compression and bending resistance on standardized 4 x 4 x 16 cm³ test tubes according to NF P-18-406 and NF P18-407 standards. After removal at 24 hours of age, the test tubes are kept in a humid environment (20°C and 100% RH) until the age of the bending test at 2, 7 and 28 days according to the NF P15-402 standard. Both ends of each test tube will be tested in compression to determine the strength of the mortar for each rice husk dosage for different ages.

Table 15. Identification of different mixtures.

Type of cement	Constituents of cement (%)			Constituents of the mortar (g or mL)		
	Clinker	Gypsum	CBR	Cement	Water	Sand
CPA-CEM 1	95.00	5.00	00.00	450	129	1350
CBR 1	70.00	5.00	25.00	450	165	1350
CBR 2	45.00	5.00	50.00	450	240	1350
CBR 3	20.00	5.00	75.00	450	260	1350

Results and Discussion

A influence of normal consistence on synthesized cements

The addition of CBR brings some difficulties in terms of water demand and the working ability of clinker-based mortars. It has been shown that the demand for water is greater when cement is substituted for CBR. This is due to the specific large area due to porous nature and giving a sponge role to the ash. This adverse effect is especially noticeable for CBR mixtures with finer cements with a significant grinding time. In addition, the demand for water is increased when there is a high carbon content contained in ash as the carbon is also porous but thinner than silica (*Figure 11*). The demand for water also increases with the grinding time to reach a maximum and then decreases during a pronounced grinding. This increase in water demand can compensate for the decrease in workability.

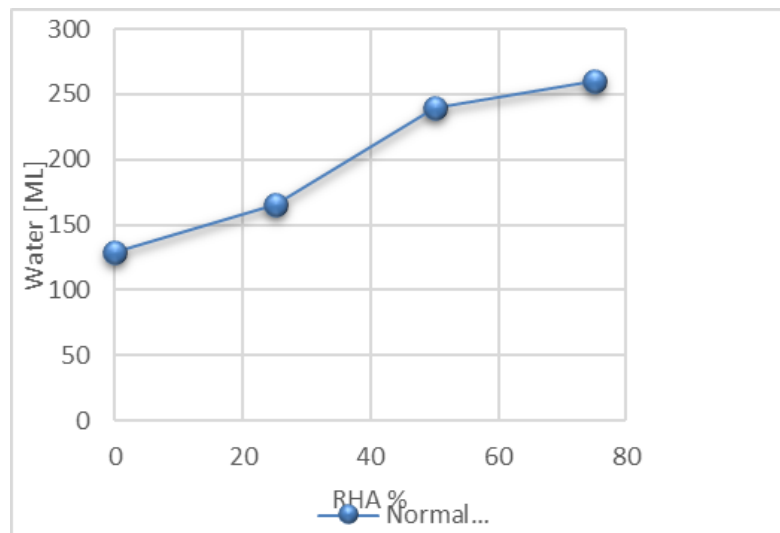


Figure 11. Normal consistency test results.

Influence of CBR substitution rate on the intake of used cements

Figure 12 shows that CBR cements have a much longer time than standard Portland cement. Indeed, we crushed the clinker with 5% gypsum. The take time changes in the same direction as the percentage of the CBR. This take time is due to the addition of gypsum in the mixture of the synthesized cement. This delay can be explained by the existence of CBR-soluble extractables, which decrease the rate of hydration of the alite (C3S). The latter is an obstacle to the progression of hydration (Murakami, 1969). This is due to the slowness of the pozzolanic reaction (Bui, 2001).

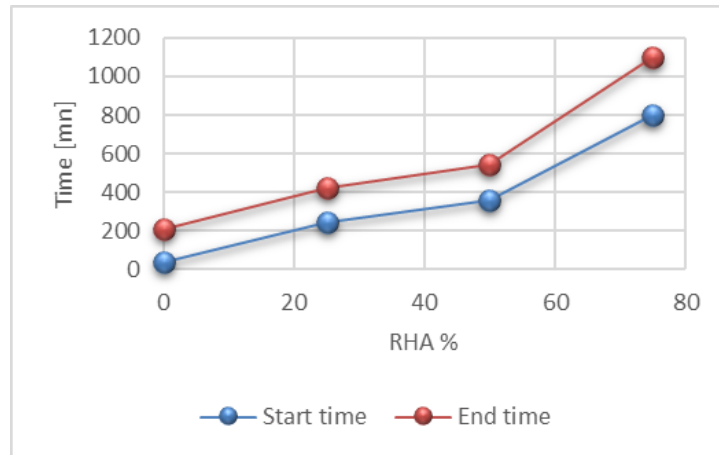


Figure 12. Start-and-end results.

Influence of density on the pulp of the cements used

It is noted that the specific Blaine surface of synthesized cements increases with the increasing rate of clinker substitution, this may be due to the nature of the incorporated substitution (CBR). The density of CBR cements is smaller than that of artificial Portland cement (*Figure 13*). As a result, if a part of cement is replaced with CBR relative to the mass, the volume of the dough of the mixture increases (*Figure 14*). CBR cements prevent cement particles from forming in blocks.

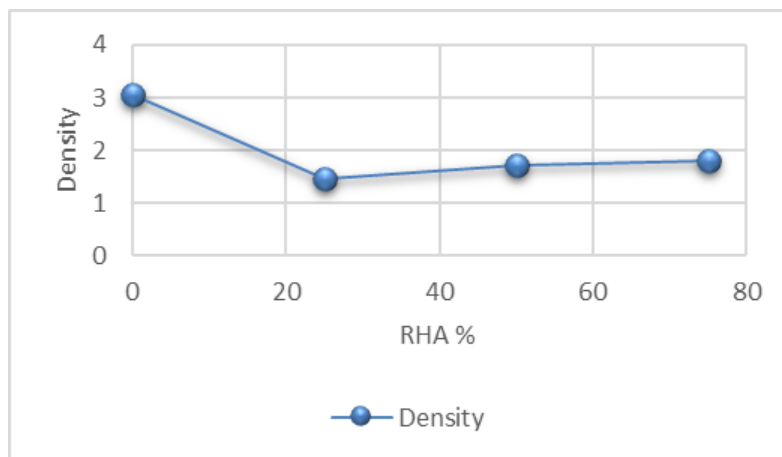


Figure 13. Effect of CBR addition rate on density.

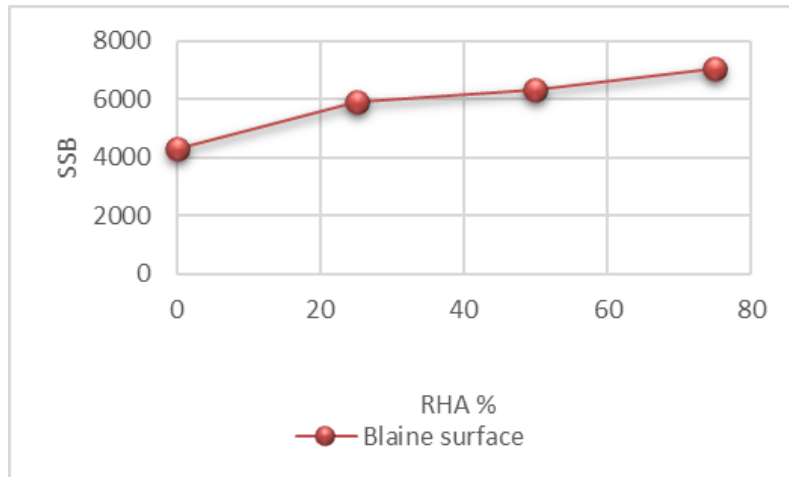


Figure 14. Effect of the CBR addition rate on the specific Blaine surface.

Influence of moisture and fire loss on the pulp of used cements

The moisture content accelerates the hydration kinetics of C2S, which is particularly slow at the heart of synthesized cements. Thus, the effect of the specific large surface on the pozzolanic reaction reacts with the Portlandite $\text{Ca}[\text{OH}]_2$ released during cement hydration to form additional CSH crystals in large quantities (*Figure 15*). The cellulose/lignin matrix of the rice husk is destroyed by fire, represents only 20-25% of the initial weight and leaves behind irregular and angular particles consisting of a porous silica skeleton (*Figure 16*). Although the ash particles of rice husks are not very small, they have a very large specific surface mainly internal due to their porosity. However, this again depends on the calcination conditions, i.e. temperature and duration, since the crystallization of the silica leads to the agglomeration of particles and the transformation into a compact granular structure (Jaturapitakkul and Roongreung, 2003). After grinding, the porous structure breaks and gives rise to fine porous particles with properties similar to those of silica fumes (Briki et al., 2022).

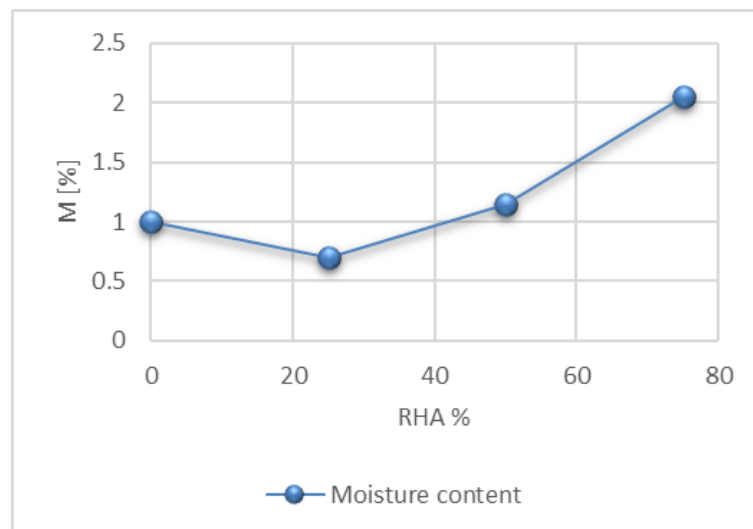


Figure 15. Moisture content of synthesized cements.

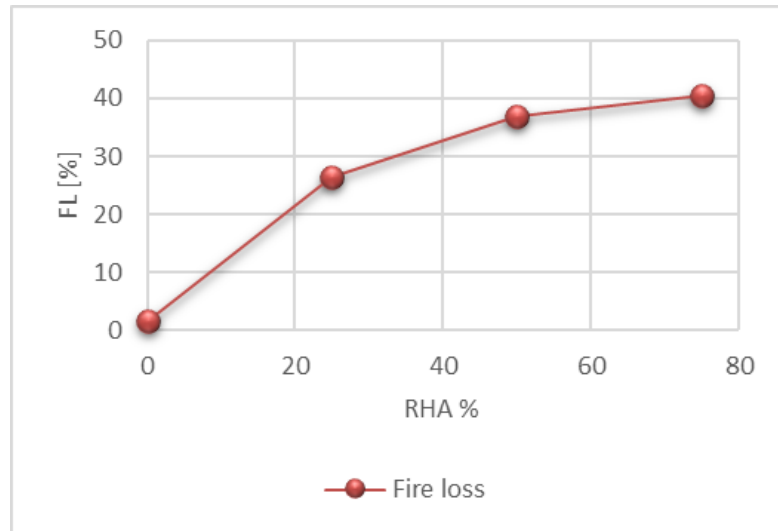


Figure 16. Fire loss of synthesized cements.

Mechanical mortar behaviour

The two previous figures place in certainty that the resistances of mortars evolve by increasing over time and show no falls except the CBR3 which has 75% clinker substitution. These changes are clearly visible on the graphs of the flexing and compression resistances. We can see that the control mortar, the first mixture with 100% artificial Portland cement, has for each age a higher resistance than that of other mortars. At 28 days, the control mortar respects the resistance announced by the technical sheet i.e. a minimum of 50,3MPa in compression. It is noted that the increase in resistance to compression and bending according to the age of hardening is virtually identical for all mortars tested except for the CBR3 compound cement which remains monotonous between 7 and 28 days in compression and decreases in bending traction. *Figure 17* shows us that at a young age cements synthesized with CBR have low strength except for cement with 25% CBR which has an acceptable resistance to CPA cement, while those synthesized with 50% and 75% CBR have lower resistance or even insufficient especially for the compound cement CBR3. The resistances, in the short term (2 and 7 days) are due to the tricalcic silicate (C3S) contained in cements and resistance at 28 days is mainly due to the ram (C2S) (*Figure 18*). In fact, the evolution of resistance depends on the CBR content and shelf life of the test tubes, as well as the C3S. Thus, CBR ash could have an effect on the reactivity of the clinker in the short term, manifested by an increase in take time and a decrease in resistance (Moir, 1983).

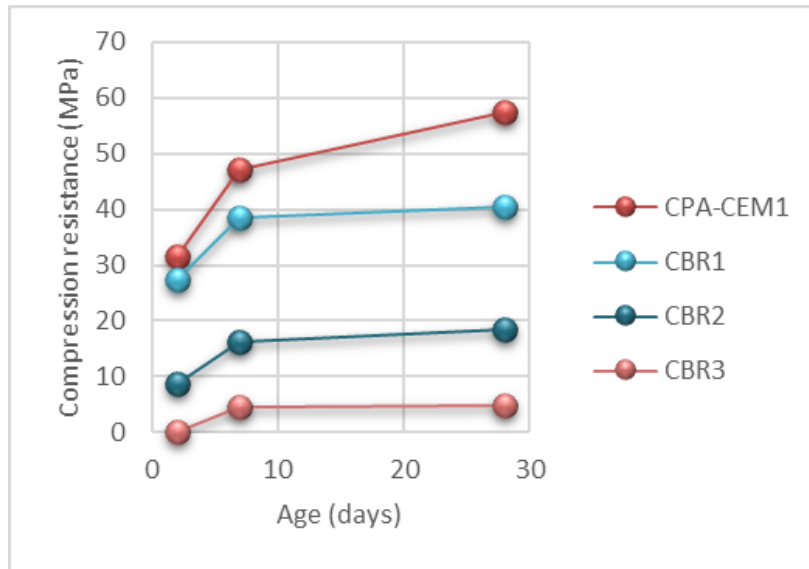


Figure 17. Compression test results.

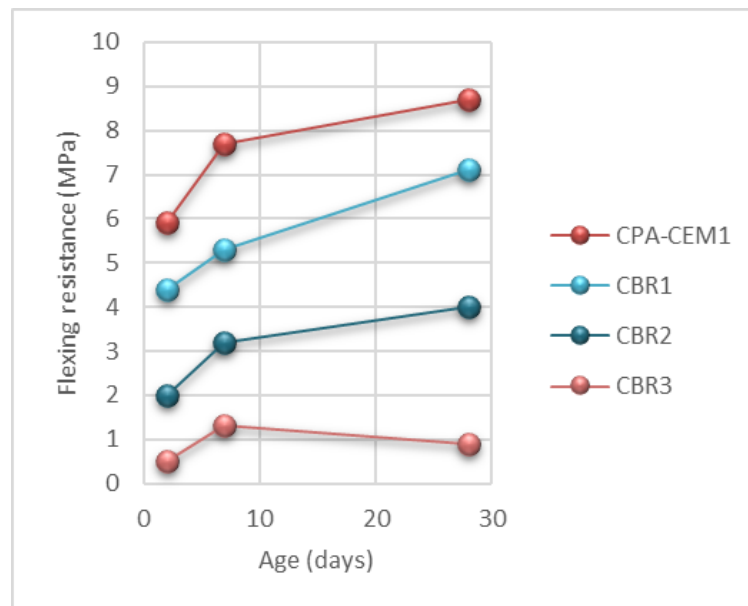


Figure 18. Bend test results.

Conclusion

This article has undeniable technical, economic, and ecological interests. Indeed, the study undertaken in the latter, tells us that it is possible to exploit the ash of rice husks that has proven qualitatively that it could be a good artificial pozzolan. It was revealed that after calcination and following an X-ray diffractometry study showed that the ash was partially amorphous and partially crystalline with visible cristobalite traces. This told us not to burn ash at too high temperatures. This fact is only explained qualitatively thanks to the DRX. Nevertheless, ash shows high pozzolanic activity after grinding in comparison to the literature consulted. This approach of uniting various cement materials (clinker, CBR) is becoming increasingly receptive to the new way of developing mortars. This research has also allowed us to study the influence of rice

husk ash as a substitute on the Portland cement manufacturing process. The results allowed us to limit the percentage of CBR in cement and to find new types of cement with different substitution percentages. Only 25% CBR cement has a standardized resistance.

Today, partial substitution in the formulation of new cements has become a necessity supported by ecological considerations, in order to reduce carbon dioxide emissions, and by their encouraging economic contribution since these substitutions escape the high calcination and offer a considerable energy gain. The cement industry whose manufacture (clinker-gypsum), releases large amounts of CO₂ (greenhouse gases) into the wild. The world's CO₂ emissions from cement are now estimated at more than 375 million tons, or 7% of global CO₂ emissions (Benhelal et al., 2013). So, the ecological objective of this research work is to develop and characterize ash-based cements of rice husks to reduce the CO₂ emissions of the cement industry. This 25% substitution of clinker reduces the energy consumption of the clinker almost to half of 1450 degrees Celsius to 750 degrees Celsius and contributes accordingly to the improvement of the environment by reducing CO₂ emissions.

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Conflict of interest

The authors declare that there is no conflict of interest involve in this research study.

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