USE OF CALCINATION RESIDUE FROM RICE HUSK AS A SUBSTITUTE FOR CEMENT

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ABSTRACT

In this study, we have developed a new cement composed by a partial substitution of the clinker with artificial pozzolans rich in silica, obtained by treatment of lignocellulosic residues, in this case, ash from rice husk. This substitution is added to the clinker with percentages ranging from 25 to 75%. These substitutions were chosen on the basis of the presence of silica which can react with portlandite $(Ca(OH)_2)$.

The results obtained show that these materials have, after activation, a great pozzolanicity that allows their addition to the Portland clinker with a percentage of up to 25% of the mass of the clinker.

The improvement of this reactivity is achieved by calcinating these additions at temperatures of 750°C. This significantly reduces the CO_2 emissions that accompany the production of Portland cement clinker.

KEYWORDS

Eco-cements, CPA cement, Rice husk ash, Calcination, resistance

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.

However, its manufacture is very energy-intensive and emits a significant amount of carbon dioxide (CO_2) well known for its impact on the greenhouse effect [1-6].

The most effective strategy to reduce the carbon footprint of the cement industry on a worldwide scale is to reduce the clinker factor [7].

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. Limestone calcined clay cements (LC3) are one of the promising alternatives for high performance sustainable cements [8-10]. Previous studies have focused on different stages of the processing of calcined clays, such as grinding [11-13] and color control [14]. These pozzolanic materials are either natural materials such as natural pozzolan [15], thermally treated materials such as metakaolin [16,17], or industrial by-products such as silica fumes and fly ash [18,19], as well as coal mash [20,21], silica fumes [22,23], flying ash [24-26], charred clay [27], limestone [28], mine tailings and polymeric waste [29], bauxite residue [30], river sediment [31,32]. 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.

B. Diana et al. [33] 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 [34].



Today, it is accepted that silica and alumina in the glass phases are reactive [35]. The incorporation of the rice husk as reinforcement in a cement matrix has been the subject of some work, notably those of Morsi [36] as well as those that essentially summarize the work of Julian Salas Serrano at the Eduardo Torroja Institute (Spain) [37,38].

The use of rice husk 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 [37-39]. However, most of this work involves a small fraction of rice husk in the matrix and mineral aggregates are sometimes retained.

In the building sector, ash from the burning of rice husk has been the subject of much research [40-42]. 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) [41,43]. 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 (*SiO*₂).

The ashes contain 95% silica and develop a very high pozzolanic reactivity [43,44]. 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 [35].

This is a strategy emphasized in this paper. The aim is to study the pozzolanic quality of rice husk ash in mortar and the experimental work relates to the development of different mortar compositions integrating the rice husk ash, the mechanical strengths are then determined.

MATERIALS AND METHODS IN MECHANICAL APPROACH

Materials and mixtures

Cement

The Portland Cement used is type CEM I 42.5 N without any addition (95% clincker 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, $CaSO_4$. $2H_2O$) was kept constant at 5%.

Properties of cement are mentioned in Table 1.



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No	Property	Test results
1	Physical properties:	
	Apparent volumetric mass (g/cm^3)	1.100
	Absolute volumetric mass (g/cm^3)	3.190
	Fineness of grind (cm^2/g) [Specific surface]	4200
	Density	3.138
	Take time: Start (hours)	2h:12
	Take time: End (hours)	3h:08
2	Chemical composition (% mass):	
	SiO ₂	20.34
	$Al_2 \tilde{O_3}$	5.37
	Fe_2O_3	3.75
	CaO	63.83
	MaO	1.80
	SO ₂	2.20
	$K_2 O$	1.07
	Insoluble residue	1.12
3	Boque's formula:	
	C_2S	57.83
	C_2S	16.75
	$C_2 A$	8.03
	$C_{A}AF$	10.92
4	Compressive resistance:	
	2 days	31.70
	7 days	47.05
	28 days	50.30
5	Bending resistance:	
	2 days	5.90
	7 days	7.70
	28 days	8.85

Tab. 1 – Physical properties of Portland Cement.

Rice husk ash



Fig. 1 – Rice husks before and after calcining.

After combustion of rice husks, the ash was finely pulverized. Chemical composition and physical properties of the RHA are given in (Table 2).



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No	Property	Rice husk ash
1	Constituents (%):	
	SiO_2	86.98
	Al_2O_3	0.84
	Fe_2O_3	0.73
	Na_2O	0.11
	K ₂ O	2.46
	СаО	1.40
	MgO	0.57
2	Fire loss (%) [quantity of 45 grams]	
	T°p 200°C	8.26
	T°p 300°C	50.78
	T°p 400°C	62.46
	T°p 500°C	73.00
	T°p 600°C	73.34
	T°p 700°C	73.76
	T°p 800°C	73.04
	T°p 900°C	74.72
	T°p 1000°C	77.06
	T°p 1100°C	77.62
3	Specific surface (cm^2/g) [EN 196-6]	16455
4	Absolute density (g/cm^3)	2.635
5	Mean particle size (μm)	5
6	Water content (%)	16.50

Tab. 2 – Chemical composition and physical properties of the rice husk ash.

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. The average values are shown in the following Figure 1:



Fig. 2 – Chapel Test: Remaining Free Lime depending on temperature.

A second test measuring pozzolanic activity is Luxan, based on the measurement of the change of electrical conductivity in a solution saturated with $Ca(OH)_2$ lime after addition of pozzolanic ash.





The average values of electrical conductivity for different temperatures are shown in the following Figure 2.





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.

600°C

	Background	E	Angle		f d-spacing
	(counts/s)		(°2Theta)	(Å) (°2Theta)	
0,28000	172,79	18,24	8,63719	65,61	12.85512
0,12000	131,03	18,06	19,57060	64,96	5,69569
0,48000	155,20	14,28	22,14895	51,36	5,03955
0,12000	214,44	27,80	26,63066	100,00	4,20311
0,20000	147,38	24,79	33,62696	89,16	3,34657
	0,28000 0,12000 0,48000 0,12000 0,20000	Background (counts/s) 172,79 0,28000 131,03 0,12000 155,20 0,48000 214,44 0,12000 147,38 0,20000	Background (counts/s) 18,24 172,79 0,28000 18,06 131,03 0,12000 14,28 155,20 0,48000 27,80 214,44 0,12000 24,79 147,38 0,20000	Angle Background (°2Theta) (counts/s) 8,63719 18,24 172,79 0,28000 19,57060 18,06 131,03 0,12000 22,14895 14,28 155,20 0,48000 26,63066 27,80 214,44 0,12000 33,62696 24,79 147,38 0,20000	Angle Background (°2Theta) (counts/s) 65,61 8,63719 18,24 172,79 0,28000 64,96 19,57060 18,06 131,03 0,12000 51,36 22,14895 14,28 155,20 0,48000 100,00 26,63066 27,80 214,44 0,12000 89,16 33,62696 24,79 147,38 0,20000

Fig. 4 - XRD results for rice husk ash $- 600^{\circ}C$.

700°C

Significance		Background	Background		T d-spacing		
		(counts/s)		(°2Theta)		(Å)	
0,67	0,10000	230,06	15,25	27,48859	29.96	4.07434	
) 0,63	0,10000	228,59	50,89	28,02837	100,00	3,99741	
0,68	0,48000	148,26	23,66	33,72937	46,50	3.33671	

Fig. 5 - XRD for rice husk ash $- 700^{\circ}C$.

800°C

Significance		Background	I	Angle		d-spacing
		(counts/s)		(°2Theta)		(Å)
0,75	0,80000	117,25	7,35	17,10127	16,81	6.51061
0,60	0,06000	118,13	31,23	18.39910	71.39	6.05491
1,66	0,24000	136,05	43,74	33,72838	100.00	3.33680
0,61	0,20000	86,65	8,47	39,03376	19,36	2,89753

Fig. 6 – XRD for rice husk ash – 800°C.

With reference to the tables of the results, only the sample calcined at 700°C. has a peak at 4.07434 Å characterizing the presence of crystobalite whose "d" is between 4.05 and 4.13. The sample of the ash obtained at 600°C. contains a "d" close to 4.2031 Å which can explain the presence of crystobalite. The intensity of this peak is lower than that representing crystobalite at 700°C. this therefore explains the formation of crystalline phases when the temperature increases.

Note that between the XRD characterizing ash calcined at 600°C. and the XRD for ash calcined at 800°C., the quantity of quartz decreases, so quartz is transformed into a crystalline phase.

Sand

The standard sand is packaged in polyethylene bags each containing 1350 g \pm 5g. Physical properties and chemical composition are shown in Table 3.

No	Property	Cumulative percentage retained (%)
1	Sieve size (mm):	
	0.08	99 ± 1
	0.16	87 ± 5
	0.50	67 ± 5
	1.00	33 ± 5
	1.60	7 ± 5
	2.00	0
2	Chemical constitute:	Constituents (%)
	CaO	1.090
	Al_2O_3	1
	SiO_2	94.789
	Fe_2O_3	0.650
	MgO	0.130
	<i>K</i> ₂ <i>O</i>	0.300
	Cl-	0.030
	SO ₃	0.080

Tab. 3 – Physical properties and chemical composition of standardized sand.

Mixture proportioning

Four mixtures of thirty-six mortar test pieces prepared in this experimental work complying with the European standard NF EN 196-1.

A study conducted by Siline Mohammed and Omary Safiuallah [45] with the objective of experimentally searching for the optimum gypsum content of a Portland Cement CEMI was concluded that the optimum of this cement is 5.5%. So, we opted for a Portland Cement CEMI (PC) composed of 94.5% clinker and 5.5% gypsum with a characteristic 28-day strength of 50.3Mpa.

The chemical compositions of the gypsum are given by the following Table 4:

				•		•••			
Elements %	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K ₂ 0	Na ₂ O	CL
Gypsum	8.50	2.54	1.04	29.32	3.07	36.53	0.53	0.03	0.008

Tab. 4 – Chemical composition of gypsum.

Preparation of test specimens

All mixtures with a constant water/binder ratio of 0.5 were manufactured with a binder content of 450g and a sand content of 1350 g, i.e. a C/S ratio 1/3.

The binder content of all other mixtures is kept fixed at 450 g. Of the four mixtures, the first is composed only of PC without using superplasticizer, it serves as a reference in this experimental program, the ashes of the rice husk (RHA) were used to replace 25%, 50% and 75% by mass of PC



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without adding superplasticizer in order to study the influence of RHA on the practicability. These are three RHA1, RHA2 and RHA3 mixtures respectively.

Additional or less water may be substituted for certain compositions to provide sufficient workability. The proportions by mass of the various constituents of the cement are mentioned in Table 5.

Type of cement	Cons	stituents of	Cement	Consti	tuents of th	e mortar
	Clinker	Gypsum	RHA	Cement	Water	Sand
	[%]	[%]	[%]	[g]	[ml]	[g]
PC	94.50	5.50	00.00	450	129	1350
RHA1	69.50	5.50	25.00	450	165	1350
RHA2	44.50	5.50	50.00	450	240	1350
RHA3	19.50	5.50	75.00	450	260	1350

Tab. 5 – Identification of different mixtures.

In the anhydrous state, the cements were subjected to chemical treatments to determine the density and density tests and Blaine Specific Surface BSS tests according to EN 196, NF EN 196-1 and EN 196-6 respectively.

The physical properties studied are: the normal consistency and the start and end time determined by the Vicat test of pure compound cement pastes used in accordance with NF 196-3.

The consistency of the mixtures will be tested as well as the compressive and bending strength on standardized test pieces $4x4x16 cm^3$ according to the standards NF P-18-406 and NF P18-407.

After removal from the mold at 24 hours of age, the test pieces are kept in a humid environment (20° C. and 100% RH) until the age of the compressive and bending test at 2, 7 and 28 days according to the standard NF P15-402.

EXPERIMENTAL, RESULTS AND DISCUSSION

Workability

It is measured according to the standard NF EN 1015-3 and determined by means of shaking table. The spreading diameters for each of the mortars are mentioned in Table 6.

Composition	D1 (mm)	D2 (mm)	Spreading diameter
PC	156	158	157
RHA1	113	119	116
RHA2	148	140	144
RHA3	-	-	-

Tab.6 – Mortar Consistency Test Results.

RHA2 and RHA3 mortars each performed poorly in terms of consistency. For RHA3 mortar, there is no clear formation of a cake after the shaking. The mixture being too dry. It is obviously a lack of water.





Influence of normal consistency on synthesized cements



The addition of RHA brings some challenges to the water demand and workability of clinkerbased mortars. Water demand has been shown to be greater when cement is substituted with RHA. This is explained by the large specific surface area due to porous nature and giving a sponge role to the ash. This unfavorable effect is especially remarkable for RHA mixtures with finer cements with a long grinding time.

In addition, the demand for water is increased when there is a high carbon content in the ash, as carbon is as porous but finer than silica. The demand for water also increases with the grinding time to reach a maximum and then decreases with a pronounced grinding. This increase in water demand can offset the decrease in workability.

Influence of RHA substitution rate on the intake of the cement used



Fig. 8 – Start and End Results.

Figure 7 shows that RHA cements have a much longer time than standard Portland cement. The sitting time evolves in the same direction as the percentage of RHA.





This setting time is due to the addition of gypsum in the mixture of the synthesized cement. This delay can be explained by the existence of soluble extractables in RHA which reduces the rate of hydration of alite (C_3S). The latter forms an obstacle to the progress of hydration. This is due to the slow reaction of the pozzolanic [49].

Density Influence on the pulp of the cement used



Fig. 9 – Effect of RHA addition rate on density.



Fig. 10 – Effect of RHA addition rate on Blaine Specific Surface.

It is found that the BSS area of the synthesized cements increases as a function of the increasing rate of clinker substitution, this may be due to the nature of the incorporated substitution (RHA).

The RHA cement density is smaller than that of PC. Consequently, if a portion of cement is replaced with RHA relative to the mass, the volume of the mixture pastes increases. RHA cements prevent cement particles from forming in blocks.







Influence of moisture and fire loss on cement paste used





Fig. 12 – Fire loss of synthesized cement.

The moisture content accelerates the hydration kinetics of the C_2S , which is particularly slow in the heart of the synthesized cements. Thus, the effect of the large specific surface area on the pozzolanic reaction reacts with the $Ca[OH]_2$ Portlandite released during the hydration of the cement to form other additional crystals of *CSH* in large quantities.

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 siliceous skeleton. 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 the particles and to the transformation into a granular compact structure [46]. After grinding, the porous structure breaks and gives rise to fine porous particles having properties similar to those of silica fumes [48].





Mechanical behaviour of mortars

Crushing tests of the 4x4x16 cubic specimens were carried out in order to determine the average resistance of three specimens at different hardening ages.



Fig. 13 – Compression test results.

Our PC mortar has compressive strength almost the same as the PC30 manufactured by D.D.Bui et al. [47] see even a little more, but other concretes made by the same authors including 10%RHA, 15%RHA and 20%RHA for their high resistance, are mainly due to the use of superplasticizer and in addition the W/B ratio is taken equal to 0.34.

The RHA1 mixture has a compressive strength at 28 days (40.40Mpa) equivalent almost to 3/4 that of the PC control mortar (57.55Mpa), with a water / binder ratio having been kept equal to 0.5.

Similarly, tensile tests on three supports of standardized 4x4x16 specimens were also carried out in order to determine the average tensile strength of three specimens at different hardening ages.



Fig. 14 – Bending test results.



The two preceding figures place in certainty that the resistances of the mortars evolve increasing with time and have no fall apart from RHA3 which has 75% of clinker substitution. These evolutions are clearly visible on the graphs of the flexural and compressive resistances. It can be seen that the control mortar, the first mixture with 100% artificial Portland cement PC, has for each of the ages a resistance higher than those of other mortars. At 28 days, it complies well with the strength stated in the data sheet i.e. a minimum of 50.3MPa in compression.

It should be noted that the increase in compression and bending resistance as a function of the hardening age is practically identical for all mortars tested except for the RHA3 compound cement which remains monotonous between 7 and 28 days in compression and decreases in traction by bending.

Figure 13 shows us that at a young age the cements synthesized with RHA have a low resistance except for the cement with 25% RHA which has an acceptable resistance with respect to the PC cement, while those synthesized with 50% and 75% RHA have lower or even insufficient resistance especially for the RHA3 compound cement.

The short-term resistances (2, and 7 days) are due to the tricalcium silicate (C_3S) contained in the cements and the 28-day resistance is due mainly to the belite (C_2S).

In fact, the evolution of the resistors depends on the RHA content and the storage time of the test pieces, as well as on the C_3S . Thus, RHA could have an effect on the clinker reactivity in the short term, manifested by an increase in setting time and a decrease in resistance.

To provide such a mortar with acceptable mechanical resistance, the water / cement ratio must be considered.

CONCLUSIONS

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 rice husks ash that has proven qualitatively that it could be a good artificial pozzolan,
- that 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.
- that is allowed us to study the influence of rice husk ash as a substitute on the Portland cement manufacturing process,
- that the results allowed us to limit the percentage of RHA in cement and to find new types of cement with different substitution percentages.
- that only 25% RHA cement has a standardized resistance,
- this 25% substitution of clinker reduces the energy consumption of the clinker almost to half of 1450 degrees Celsius to 750 degrees Celsius,
- that it contributes accordingly to the improvement of the environment by reducing CO_2 emissions.

This approach of uniting various cement materials (clinker, RHA) is becoming increasingly receptive to the new way of developing mortars and can be improved and enriched in the near future by recommendations which are:

- The water / binder ratio will have to be increased to allow greater workability.
- Experimentally find the optimum content of the synthesized gypsum cement.
- Improve the density of the mixture-mortar by improving the filling of particles by adding a superplasticizer.
- Influence of pozzolanic activity as a function of grinding time and therefore of the specific surface area of the ash particles.
- Calcine the rice husk and use the residues resulting from this calcination as a complement to a binder such as lime for example.



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