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Preliminary characterization of the rice husk ash from the Manabí province for its use in concrete

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Abstract

This article describes the potentialities of ash from controlled burning of rice husks in the province of Manabi (Ecuador), as a substitute for portland cement used in concrete, including selection and preparation of representative samples of rice husk, quantitative and qualitative characterization of the husk, burning procedure and characterization of the resulting ash. The procedures used in the characterization of the rice husk are evaluated, demonstrating the relevance of using the nuclear absorption test to determine the percentage by weight of silica in the mass of the sample and the ash obtained from the burns at different temperatures and times, using fluorescence and X-ray diffraction procedures.

Keywords: rice husk ash; amorphous silica; concrete; pozzolan.

Caracterización preliminar de la ceniza de cáscara de arroz de la provincia Manabí, Ecuador, para su empleo en hormigones

Resumen

Este artículo se describen las potencialidades de la ceniza proveniente de la quema controlada de la cáscara de arroz en la provincia de Manabí (Ecuador), como sustituto del cemento Portland empleado en hormigones, incluyendo la elección y preparación de muestras representativas de cáscara de arroz, caracterización cuantitativa y cualitativa de la cáscara, procedimiento de quema y caracterización de la ceniza resultante. Se evalúan los procedimientos empleados en la caracterización de la cáscara de arroz, demostrando la pertinencia de utilizar el ensayo de absorción nuclear, para determinar el porcentaje en peso de sílice en la masa de la muestra, y se caracteriza la ceniza obtenida de la quema a diferentes temperaturas y tiempos, mediante procedimientos de fluorescencia y difracción de rayos X.

Palabras clave: ceniza decáscara de arroz; sílice amorfa; hormigón; puzolana.

Introduction

Pozzolans are active silica-containing materials that in themselves have little or no binding quality, but mixed with lime in the presence of water, set and harden like portland cement. In general, pozzolans can be divided into two large groups: natural, such as volcanic ash and zeolites; and artificial ones, such as calcined clays, pulverized ashes from stone coal and ashes from the burning of agricultural residues [1].

The greeks, 400 BC, were the first to use pozzolans in lime mortars. Later the Romans not only used powdered pottery, bricks, and tiles to form artificial pozzolans, but also discovered that some volcanic soils mixed with lime were excellent for producing hydraulic mortars. This Roman experience continued to be applied with different alternatives and at present, the use of pozzolans in the production of cements and concretes constitutes an international practice [2]. Researchs had shown a continuity in the study on the use of pozzolans, of natural or artificial origin, as a partial substitute for portland cement in the production of concrete. Bonavetti et al. [3] highlight this experience in the production of self-compacting concrete with high performance and durability, with a low content of portland cement as a result of the addition of natural zeolite as pozzolana.

A pozzolan investigated as a partial substitute for portland cement in the production of mortars and concretes, is the ash from the controlled burning of rice husks. Yanguatin *et al.* concluded that the experienced combinations of rice husk ash with portland cement, allow substituting up to 30% of the cement by ash without affecting the compressive strength. It is also verified that the optimum percentage of substitution fluctuates around 20%, thereby achieving an increase of around 20% in the compressive strength, improvement in the chemical stability of the concrete and an increase in its durability. As a negative effect, there is an increase in the demand for mixing water.

However, regarding the use of rice husk ash in the production of mortars and concretes, there are not many references in Ecuador. In this country, the rice husk is an agro-industrial waste of difficult final deposition that currently has few applications, resulting in a voluminous and polluting by-product. It is a material rich in silica [1,3,9], which has attracted attention in the construction industry as a partial substitute for portland cement used in the production of concrete, but still without conclusive results.

Therefore, in the present paper, a preliminary characterization of the rice husk ash was carried out to possible use in the production of concrete, through the following suppositions:

First, the characterization of the rice husk from the province of Manabí, in particular the determination of its silica content by novel test methods to achieve greater accuracy, comparing with international standards and assessing its viability to be used for the production of ash as a partial substitute for portland cement in the production of concrete.

Secondly, the characterization of the ash resulting, from burning for different temperatures and burning times, using spectrometry and X-ray diffraction techniques.

Experimental

Rice production in Ecuador

In social and productive terms, rice cultivation is the most important in Ecuador, occupying approximately one third of the country's transitory product area [4]. The behavior of the crop cannot establish a trend that is defined as an increase, since in some areas its production increases as well as decreases, being a multivariate of factors the cause of these fluctuations. The provinces with the highest participation in rice production are Guayas, Los Ríos, Manabí, Loja and El Oro. Chronologically in terms of yield, the national average during 2015 was 5.24 t/ha, Loja being the province of higher yield, with an average of 6.75 t/ha. The province that showed the lowest yield was El Oro, with an average production of 3.68 t/ha.

Already in 2016 the survey of surface and continuous agricultural production reflects in its statistics that the national surface planted in that year was 385,039 thousand hectares, a truly significant area, from which 366,194 thousand hectares were harvested, obtaining a production of 1,534,537 t and sales of 1,432,318 t [5].

Although it is difficult to achieve comprehensive statistics due to local consumption omissions, the data indicate that the main rice producing provinces are Guayas, Los Ríos and Manabí, especially the former due to its favorable soils and climatic conditions [6]. However, in Ecuador there are usually increases in the rice production but at the same time reductions in the areas dedicated to cultivation because either they are dedicated to new crops or they are rotation criteria, as can be seen in Table 1. It shows that the three aforementioned provinces have a weight of 96.6% as a trend of the total stacked whole rice in the country. It can also be seen, according to the data obtained from MAGAP [7], that the rice production of the province of Manabí in 2017 was the third most important in the country, reaching 48,604 t. Although it only represents 3.37% of national production, the volume is close to the sum of the rest of the country's provinces except Guayas and Los Ríos.

Table 1. Significant provincial rice productions in Ecuador, 2012-2017, t [7].

Provinces	Years					
	2012	2013	2014	2015	2016	2017
Guayas	1,029,783	1,060,669	902,424	1,187,135	1,035,344	986,397
Los Ríos	444,330	359,569	410,850	383,106	421,483	356,687
Manabí	42,128	63,656	45,607	57,169	55,536	48,604
Others	49,294	32,151	21,073	25,383	22,174	49,179
Total (t)	1,565,535	1,516,045	1,379,954	1,652,793	1,534,537	1,440,865
Surface (ha)	371,170	396,770	354,136	375,117	366,194	286,189
Performance (t/ha)	4.22	3.82	3.90	4.41	4.19	5.03

In the province of Manabí, according to a report from the Central Bank of Ecuador [8], it can be seen that of the cantons of Olmedo, 24 de Mayo, Santa Ana, Rocafuerte, Sucre, Portoviejo, Paján and El Carmen, with 94% of the total. Standing out that they adjoin Santa Ana, 24 de Mayo and Olmedo; and the same happens with the cantons of Portoviejo, Rocafuerte and Sucre. This explains the location of the main pile machines who perform the husking of dry and clean paddy rice in these six cantons, which are located in Santa Ana and Portoviejo. Precisely the rice from Rocafuerte and Sucre is mostly brought to the processing centers of Portoviejo; and to Santa Ana the rice from the cantons of 24 de Mayo and Olmedo. In the case of Pajan and El Carmen, the cultivated rice is processed in pile machines in the same cantons.

This analysis allowed for the purposes of the research to synthesize the four fundamental regions of the province, detailing the average percentage production and location of the main pile machine, for the purposes of sampling the rice husk to be used in the trials:

- Region 1: cantons of Santa Ana, 24 de Mayo and Olmedo; 31% of the provincial production; main pile machine in Santa Ana.
- Region 2: cantons of Sucre, Rocafuerte and Portoviejo; 27% of the provincial production; main pile plant in Portoviejo.
- Region 3: to the south, only the Paján canton, 21% of the provincial production; main pile machine in the center of the canton itself.
- Region 4: to the north, only the El Carmen canton; 16% of the provincial production; main pile machine in the center of the canton itself.

For a better understanding, Figure 1 shows the distribution of the four regions in the province of Manabí and the location of the pile machines in them.

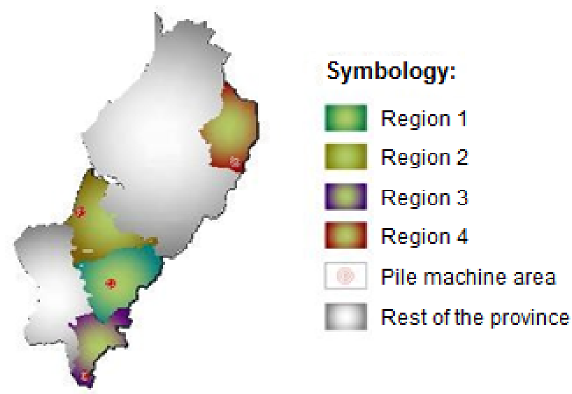

Figure 1. Location of the four large rice growing regions and the pile machines in them [9]

It is also found in the specialized reports that within each one of the synthesized regions the same variety of rice to be cultivated prevails, which constitutes an advantage that facilitates subsequent sampling aimed at the physical and chemical characterization of the rice husk. The most relevant data is the mean estimate of the productive residuals. If rice production in the province were to be maintained or increased, no less than 13.4 thousand tons of rice husk per year can be estimated in the selected regions, which instead of polluting the environment and with a well-directed policy, they can be used as raw material for the production of concrete, as a partial substitute for portland cement, fully justifying this research and its industrial application.

For the purposes of this investigation, the production of dry and clean paddy rice is of interest, since this is the rice that reaches the mills for the husking process. The consultation carried out allows us to synthesize that from the product of the grinding process, approximately 20% of the weight of dry and clean rice husks constitutes the husk, and given the high production volumes the regional potential is high [10].

Determination of silica in the rice husk samples

The previous analysis about rice crop distribution and processing in Ecuador, allowed samples from Paján, Santa Ana, San Eloy, Rocafuerte and Chone pile machines were selected for the tests, guaranteeing representativeness in the north, center and south.

The presence of silica within the structure of the rice husk has been known since 1938. According to referenced research, it oscillates around 20%, occurring in a higher quantity with respect to the rice grain [9,10,11]. Regarding the type of structure, there are several silica polymorphs: quartz, cristobalite, tridymite, coesite, stishovite, lechatelierite and silica gel. Silica or silicon dioxide (SiO_2) as it is also known generally exists in two forms, amorphous and crystalline [11].

For the percentage determination of silica presence in the rice husk samples from the five selected pile machines, the nuclear absorption technique was applied, one of the most innovative and exact for these purposes [12,13], sending them to the laboratories of the Directorate of Chemical and Environmental Sciences, of the Faculty of Natural Sciences and Mathematics of the Escuela Politécnica del Litoral, Ecuador (ESPOL).

Rice husk calcination process

Once the characterization of the rice husk was concluded, as the next step in the investigation, the analysis of the controlled burning procedure and characterization of the resulting ash was undertaken.

Rice husk, when subjected to a calcination process, produces ash in the order of 13 to 29% of the initial weight, composed mainly of silica in a variable proportion between 87 and 97%, plus other amounts of inorganic salts that can be eliminated.

Several authors document that depending on the temperature range and the duration of combustion, crystalline or amorphous forms of silica are obtained [11,14]. It is generally accepted that significant formation of amorphous silica forms in the range of 600 to 800 °C, while crystalline silica occurs above 900 °C [14]. The crystalline and amorphous forms of silica have different properties. For the application required in this research, the relevance is towards ashes with an amorphous structure, an aspect that is more prone to minimum temperatures within the range, although the formation of silica is slightly lower, the relationship being inverse behavior [15,16].

Prior to the calcination process, a drying process was carried out by placing the standard husk samples at a controlled temperature of 105 ºC for 24 hours. After achieving almost zero humidity, the burning procedure was carried out in an oven with a Bartlett brand digital temperature controller (Genesis LT3140 model), generating a total of 70 operating points in the process of calcination of the rice husk, as can be seen in Figure 2, where the vertical columns from left to right, correspond to temperature increases from 600 to 875 ºC with a step of 25 ºC; vertically, from bottom to top, burning times between 15 and 90 min, with a step of 15 min. The color changes in each range can be clearly seen.

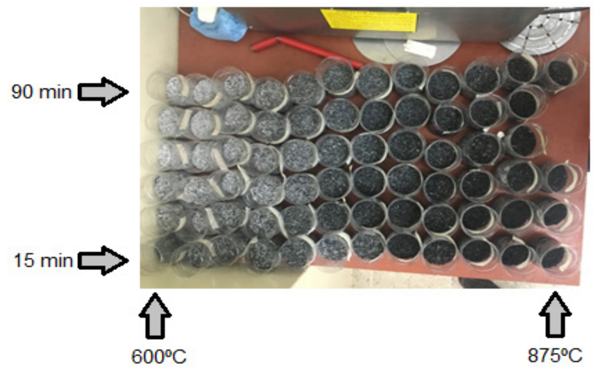

Figure 2. Operational points in the rice husk calcination process

According to Salazar [17], as silica ash is a by-product of a natural compound, in this case rice husk, it requires careful methods or procedures of characterization studies to better understand its nature and be able to determine the necessary modifications in the process of obtaining silica to improve its properties, including adhesion to the matrix when used as a filler in composite materials.

Chemical composition analysis of the rice husk ash

The experimental techniques are based on strictly developed procedures to analyze the presence and formation of silica in rice husk ash, highlighting among them: nuclear magnetic resonance spectroscopy (NMR), scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray fluorescence (XRF), Fourier transform infrared (FTIR) and differential thermal analysis (DTA). In the case of the present investigation, for the determination of the chemical composition of the rice husk ash, the XRF technique [18,19], was used in the applied chemistry laboratories of the cement company CURAZAO SA, of the province of Artemisa, in Cuba; and for the structural characterization of the ash itself, the XRD technique [20,21,22], carried out at the chemistry laboratories of the Yachay Tech University, San Miguel de Urcuquí, Ecuador.

Results and Discussion

The nuclear absorption test on the rice husk samples from the selected pile machines, allows knowing their constitution in percentage of SiO2, as shown in Table 2. The table shows that the results for the percentage of silica from the husk samples are located around the ranges internationally referenced [23]. Soares *et al.* [24] endorse for international practice the presence in the rice husk of an average chemical composition of silica between 16 and 20%. Although two of the reported values are slightly lower in comparison to the rest, their volume is not significant as they are not among the main eight producing cantons of the volume of husk generated and the reported value is only 1.7 and 1.0% of the lower limit of 16% of the presence of silica referenced in the literature. This result allows to scientifically substantiate, the potential for the indistinct use of the husk from any of the cantons.

Table 2. Analysis of the percentage silica (SiO2) in rice husk by $NR_{M.}$

Region	%
Pajan	15.0
Santa Ana	18.0
San Eloy	15.81
Rocafuerte	17.26
Chone	14.30

When initially evaluating the results of the burning, the experience gathered in the international literature was taken into account. In this sense, Ayhan [25] documents that under complete combustion, the rice husk ash with the highest proportion of amorphous structure must present a color that fluctuates in gray/white; but that under partial combustion conditions a black ash is produced, with a rather crystalline structure. This result makes it possible to reduce the operating points obtained as a result of controlled burning to nine, selecting only those in which the coloration is gray/white, corresponding to temperatures of 600, 625 and 650 ºC and burning times of 60, 75 and 90 minutes, shown in Table 3. These were then used to define whether the silica in the rice husk ash obtained has an amorphous or crystalline structure [26,27].

Table 3. Samples selected for characterization by fluorescence and X-ray diffraction (XRD).

Parameters	Samples								
	M1	M2	M3	M4	M5	M6	M7	M8	M9
Time [min]	60	75	90	60	75	90	60	75	90
Temperature [°C]	600	600	600	625	625	625	650	650	650

The results of the XRF to determine the concentration of silica in the ash, can be seen in Table 4. In all cases, a high SiO2 content is obtained, with the maximum value in sample M8, corresponding to a temperature of 650 ºC and a burning time of 75 min.

Table 4. Results for percent silica (SiO2) and alumina (Al2O3) in rice husk ash samples.

	Samples								
	M1	M2	M3	M4	M5	M6	M7	M8	M9
SiO_2/Si	85.00	88.85	87.21	88.76	88.59	89.31	90.05	90.62	90.26
Mean Concentration			88.74		Max Concentration			90.62	
Mean Concentration			85.00		DST			1.74	
Al_2O_3/Al	1.05	0.86	0.89	0.87	0.99	0.92	0.84	0.84	0.85
Mean Concentration			0.90		Max Concentration			1.05	
Min Concentration			0.84		STD			0.07	

STD: standard total deviation

The mineralogical characterization of the rice husk ash samples carried out by XRD, produced very similar diffractograms, practically peakless, as can be seen in Figure 3.

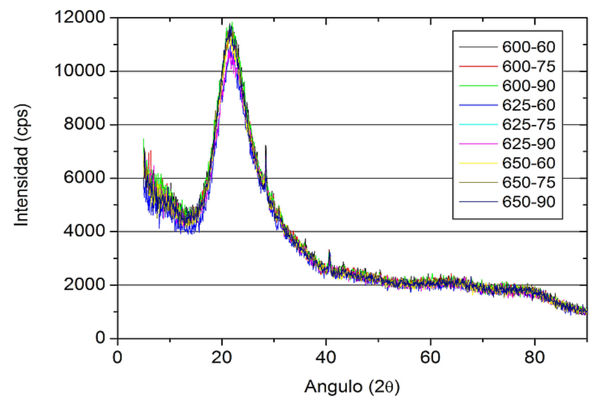

Figure 3. Overlapping diffractograms of rice husk ash samples by XRD.

As recognized in the specialized literature [22, 25, 26], the abundance of sharp peaks correspond to a crystalline or semi-crystalline silica, named cristobalite. In the case of study this is not the case, so it is concluded from the analysis of the diffractograms, that mineralogically the structure of the different samples tested is predominantly amorphous, which is convenient for the final interests of the research.

This allows, in order to give continuity to the investigation, to select M6 as a sample with a predominantly amorphous mineralogical structure, corresponding to a temperature of 625º C and a burning time of 90 min, which constitutes the main result of the investigation in this part.

Conclusions

The research carried out certifies, first, that the industrial by-product of rice production in the province of Manabí, the rice husk, complies with the SiO2 percentage ranges reported in the international literature (between 15 and 18%), enabling this result to give continuity to the investigation.

And secondly, that the controlled burning of it in temperature ranges between 600 and 650 ºC and burning times between 60 and 90 min, produces an ash that is characterized by a predominantly amorphous mineralogical structure, which supports its use as an artificial pozzolan substitute for Portland cement in the preparation of mortars and concretes and makes it possible to proceed to the next stage of the investigation, in this case, the design of dosages with different levels of substitution of cement for ash, taking for this a burning temperature of 625 ºC and time burning for 90 minutes.

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