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RESEARCH ARTICLE

Investigations on the Pozzolanic Effect of Sugar Cane Bagasse Ashes Used in Cementitious Composites

Paula G. Lamezon de Pádua^{a,*}, Túlio H. Panzera^b, Roberto B. Figueiredo^c, A. M. Gomes^c and Maria T. Paulino Aguiar^c

^aDepartment of Structural Engineering, Federal University of Minas Gerais, Belo Horizonte, Brazil

^bDepartment of Mechanical Engineering, Centre for Innovation and Technology in Composite Materials, Federal University of São João Del Rei, Minas Gerais, São Paulo, Brazil

^cDepartment of Materials and Construction Engineering, Federal University of Minas Gerais, Belo Horizonte, Brazil

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Abstract: Brazil is a major producer of alcohol from sugar cane, a fuel with low environmental impact. The production of alcohol generates a large amount of bagasse, the biggest waste of Brazilian agriculture. This bagasse is usually burned for energy production providing nearly 3% of residual ashes. The potential use of these ashes like mineral admixture of cementitious composites depends on calcination conditions. The present work identifies the physical and chemical characteristics of ashes from the furnace exhauster, obtained in an industry located at southeast region of Brazil. The ashes were obtained from bagasse of sugar cane harvest in two different seasons. Chemical composition analysis, X-ray diffraction, grain size distribution, loss on ignition, thermogravimetry analysis (TGA), differential scanning calorimetry (DSC), specific surface measurements (BET) and Fourier transform infrared spectroscopy (FTIR) tests were used to characterize the ashes. The pozzolanic activity was estimated by pozzolanic activity index tests with cement and lime, by the modified Chapelle test, electrical conductivity in lime solution tests, TGA and FTIR. The ashes presented different chemical compositions and degree of amorphicity. The ashes with a higher content of silica, the lower organic material content and high degree of crystallinity (1st harvest), for the same size, have higher pozzolanic on electrical conductivity in lime solution tests than the ashes with lower silica content and higher amorphicity (2nd harvest). However, the results of differential scanning calorimetry (DSC) and modified Chapelle method would indicate the pozzolanicity of the ashes of the 2nd harvest. The results of pozzolanic activity index (with cement or lime) indicated the ashes were not pozzolanic.

Keywords: Cementitious composites, Differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR), Modified chapelle, Physical-chemical characteristic, Pozzolanic activity, Sugar cane bagasse ash.

1. INTRODUCTION

Brazil is one of the biggest producer of sugar cane. The production of sugar cane estimated in 2013 was approximately 712 million tons [1]. This production tends to increase due to government politics that encourages the construction of new plants to supply the production of alternative fuel [2].

The juice extracted from sugar cane is used for the manufacture of sugar and ethanol, and then the bagasse is produced. The bagasse is the biggest waste of Brazilian agriculture [3]. A ton of sugar cane generates around 280 kg of bagasse [4]. Usually, this product is used as a fuel in the process of power co-generation. The incineration of bagasse generates steam in high pressure and it is expanded in turbines to produce mechanical/electrical energy [5]. The incineration of sugar cane bagasse also generates about 2.4% ashes, collected at different points of the furnace. The fly ashes are collected in the exhauster of the furnace. In general, these ashes contain silica and coal dust [6]. In 2013, it

* Address correspondence to this author at the Department of Structural Engineering, Federal University of Minas Gerais, Belo Horizonte, Brazil; E-mail: paulalamezon@hotmail.com

was estimated that 17million tons of bagasse ash was generated in Brazil. Initially, these ashes were considered environmental liabilities. However, they have been used as fertilizer, as adsorbents to remove raw material or petrochemical materials of the wastewater [7].

Another alternative use of the fly ashes from sugar cane is the mineral admixture of concrete. Other types of ashes are already being used as fillers or pozzolanic materials. In both cases, they are important materials for the manufacture of durable concrete [8]. The ashes of sugar cane bagasse (CBCA) can be used as fine aggregate [9] or as a partial substitute for Portland cement [10]. According to Karade [11] the industrial residues may have pozzolanic properties. Therefore, the temperature and time of burning must be controlled. In Brazil, the studies started in 1998 and indicate the potential use of sugar cane bagasse ash in the production of concrete [12].

According to Mehta and Monteiro [8] and Battagin [13], the pozzolanic material is a siliceous or siliceous-alumina material, which by itself has little or no cementitious value, but when finely divided and in the presence of moisture chemically reacts with calcium hydroxide at ambient temperature to form compounds possessing cementitious properties (hydrated calcium silicates and hydrated calcium aluminates). The pozzolanic activity depends on the levels of non-crystalline silica and alumina and the specific area of the material [13, 14]. Different tests have been used to evaluate the pozzolanic activity of the materials. Some methods are based on the measurement of the presence of $\text{Ca}(\text{OH})_2$ in the sample. Others evaluate a chemical or physical parameter related to the extent of the pozzolanic reaction [15].

The aim of this research is the chemical-physical characterization of the sugar cane bagasse ashes from the furnace exhauster collected in two different seasons, besides assessing its reactivity with calcium hydroxide. The chemical-physical characterization of the ashes was performed using chemical composition analysis, grain size distribution, specific surface measurements (BET), Fourier transform infrared spectroscopy (FTIR), loss on ignition and X-ray diffraction. Different tests were used to estimate the reactivity of the ashes with calcium hydroxide: pozzolanic activity index with Portland cement and lime; modified Chapelle test; electrical conductivity, thermal thermogravimetry analysis (TGA) and differential scanning calorimetry (DSC).

2. MATERIALS AND EXPERIMENTAL METHODS

Two kinds of sugar cane bagasse ashes were collected from the furnace exhauster in different harvest in Alpha Distillery Company (Brazil). Firstly, the chemical composition of the ashes was determined by a Shimadzu EDX-800 X-ray emission spectrograph. The ignition loss and thermogravimetric tests were able to qualitatively assess the carbon content presented in the ashes. The loss ignition test was performed according to NBR 5743 [16]. The thermogravimetry analysis evaluates the variation of mass of a sample heated in controlled conditions. The differential scanning calorimetry measures the difference in the amount of heat required to increase the temperature of a sample and reference as a function of temperature. The equipment used in both tests was a Shimadzu, Model 50-TGA. The samples were heated to 1000°C at a heating rate of 10°C/min under N_2 (inert atmosphere) at a flow rate of 20 ml/min.

The fineness of the ash was evaluated by two methods: the technique of nitrogen adsorption (BET method) and the laser diffraction particle sizing technique (laser granulometry). A Cilas model 1064 grain size meter was used to determine the grain size distribution of the ashes by laser diffraction. This test allows to determine particle size distribution of the analysed material with a particle size ranging from 0.04 to 500 micrometers. A Quantachrome Autosorb-1 was used to the nitrogen adsorption test. The structure of the atomic arrangement of the ashes was analysed by a X-ray diffractometry with CuK α radiation, 30 kV and 30 mA, time constant of 0.6, crystal graphite monochromator, and scanning speed of 2°/min. The structure was also studied by Fourier transform infrared spectroscopy. A Perkin Elmer spectrometer-Spectrum 1000 was used for FTIR. For this test the powdered samples were pressed with potassium bromide (KBr).

Initially, the reactivity of the ashes with calcium hydroxide (pozzolanicity) was evaluated by DM-32 Digimed model bench conductivity meter according to the method proposed by Luxan *et al.* [17]. This method consists of measuring conductivity variations of a solution (200ml) saturate with $\text{Ca}(\text{OH})_2$ maintained at 40°C, before and after adding 5g of the material to be evaluated.

The pozzolanicity was also evaluated by modified Chapelle method and pozzolanic activity index. The modified Chapelle test [18] measures the amount of CaO which reacts with the material evaluated. The mixture of 1 g of calcium oxide, 1 g of pozzolan and water was boiled for 10 hours. The result is determined by titrimetry with HCL and it is expressed by the amount of calcium oxide or fixed consumed per gram of pozzolanic material (mg CaO / g pozzolan).

Pozzolanic activity index (PAI) of the ashes was performed with cement and lime. This test allows the indirect measurement of the pozzolanicity degree based on the comparison of axial compressive strength of mortar specimens with and without ashes at 28 and 7 days of age, respectively.

The reference mortar formulation for testing with cement was prepared with one part of cement and three parts of normalized sand, by mass. In the mortar with ashes, 35% weight of cement was replaced by an equivalent volume of fly ash. The specific gravity of the cement and ashes were determined by Le Chatelier flask. The water amount in the mortar was chosen to correspond to the consistency index of (225 ± 5) mm in the flow table test. The water and binder (cement + ashes) ratio to PAI cement test was set at 0.5. The test for lime reactivity is similar to the test for cement, but the samples are prepared using lime instead of cement (one part of lime and nine parts of normalized sand by mass). The amount of ashes was twice as much as that from lime. The water amount in the mortar was chosen to correspond to the consistency index of (225 ± 5) mm in the flow table test [19]. According to the standard the relation between water and binder (cement + ashes) content must be equal to 0.50 (m/m). Furthermore, to maintain this consistency of mortar for IAP with lime test, the relationship between the mass of water and solid was 0.14, which is the same ratio used by Cordeiro [20].

Cement pastes with and without (reference) ashes (10% by weight of cement was replaced by an equivalent volume of ashes, and water / binder was set at 0.5) were made for FTIR, TGA and DSC tests. After curing for 28 days, the pastes were immersed in acetone for 5 minutes in order to stop the hydration processing. After immersion, the samples were grinding. The reactivity of the ash with hydrated hydroxide presented in the mortar was also evaluated by TGA and DSC using the same methodology and equipment conducted for the thermal analysis of the ashes. The FTIR results allow a qualitative comparative analysis of the structure of the atomic arrangement of the cement pastes with and without ashes. The methodology and equipment were the same used in the study of the ashes.

3. RESULTS AND DISCUSSION

Table 1 shows the chemical compositions of the ashes from the 1st and 2nd harvests, according to the model commonly adopted by the cement industry. The silica and alumina content in the ashes from the 1st harvest are approximately 3.5 times greater than the ashes of 2nd harvest. The amount of CaO, K₂O, Cl⁻ and SO₃ found for the 2nd harvest is superior to the 1st harvest. The high K₂O content could cause alkali-aggregate reaction. The higher content of lime and SO₃ would be related to an incomplete burning [21]. According to Brazilian Standard [22], a pozzolanic material of class N must have a minimum of 70% of SiO₂ + Al₂O₃ + Fe₂O₃ and the SO₃ content should not be more than 4%. However, for Neville [23], the SO₃ content should be less than or equal to 5%, to avoid the formation of ettringite expansion. Thus, the ash of the 1st harvest can be considered a pozzolanic material, while the fly ash from the 2nd harvest is not considered a pozzolanic material.

Table 1. Chemical composition of the ashes.

Compounds	1 st harvest ashes (%)	2 nd harvest ashes (%)
SiO ₂	61.53	17.02
Al ₂ O ₃	17.37	6.77
Fe ₂ O ₃	10.57	13.41
CaO	2.92	11.76
MgO	1.31	2.20
SO ₃	0.45	7.00
K ₂ O	2.63	33.00
P ₂ O ₅	1.78	3.51
Cl ⁻	0.14	4.52
TiO ₂	0.912	0.00
SrO	0.276	0.00
SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	89.47	37.20

Table 2. Results of ignition loss of the ashes.

Samples	(wt%)
1 st harvest ashes	20

(Table 2) contd....

Samples	(wt%)
2 nd harvest ashes	50

Table 2 presents the loss on ignition results of the ashes. The data indicate the ashes of the 2nd harvest have almost more than double of organic matter than the first. Whereas the saccharose content is basically the same in the ashes of both harvests, the difference in the loss on ignition results could be related to the higher carbon content in the 2nd harvest ashes due to incomplete burning or higher concentration of reduced sugars (fructose, glucose and maltose).

Fig. (1) shows the results of thermogravimetric analysis of the ashes from the first and second harvest. The data indicate that the ashes from the 1st harvest heated from 40 °C to 400 °C show a gradual mass loss, thereafter the loss is enhanced up to 640 °C, when it stabilizes. The heating of the ashes from the 2nd harvest also presents two pronounced intervals of losses: 40 °C to 400 °C and 400 °C to 600 °C. After this temperature, the weight loss seems to stabilize. However, the weight loss for these ashes is significantly higher. In total, the ashes of the 1st harvest lost 24wt% and the 2nd lost 38wt%. These results are consistent with those obtained from the loss on ignition test. These differences could also be related to the presence of higher content of reduced sugars in the 2nd harvest and the fact that these sugars present different burning temperature.

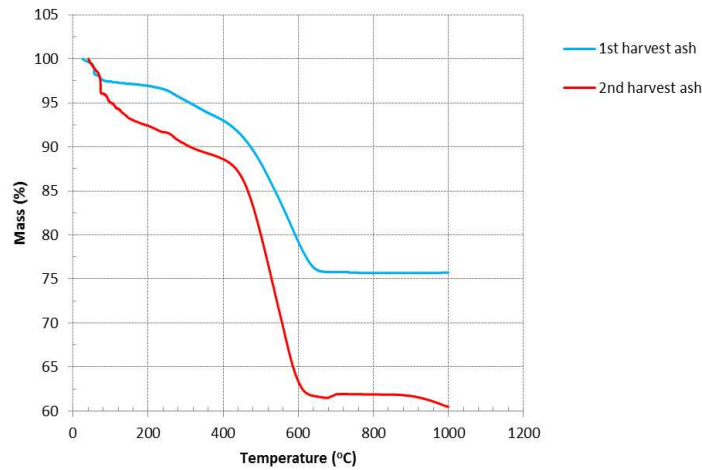


Fig. (1). Thermal thermogravimetry analysis (TGA) of the ashes.

Fig. (2) shows the differential scanning calorimetry of ashes. It is observed that the combustion reactions of residual sugars are endothermic. There are several peaks on the results of the 2nd harvest ash, probably because of the different sugars present in the sample.

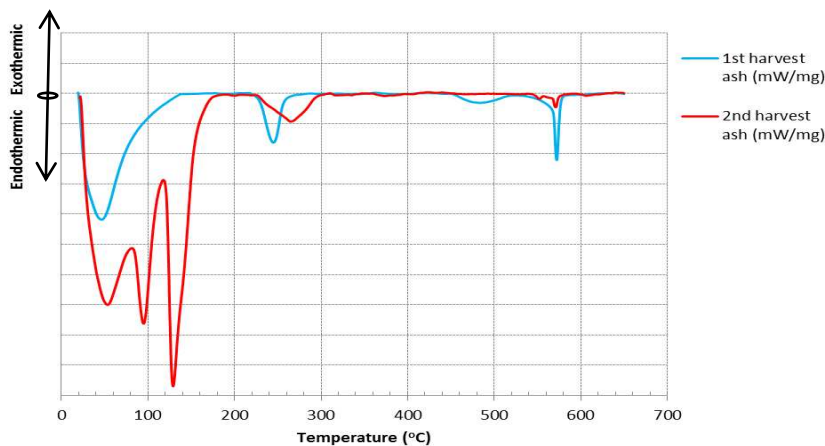


Fig. (2). Differential scanning calorimetry (DSC) of the ashes.

The laser results of the particle size analysis of the ashes are shown in Fig. (3), which displays the size distribution of the sample. By these data, Table 3 shows the average diameter and the values of the particle size under 10%, 50% and 90% by weight of material (d10, d50 and d90). According to NBR 12.653 [22], a pozzolanic material would have at least 66% of material passing a sieve of 45µm. In this context, both ashes would be pozzolans (79% and 78% of the ashes passing a sieve of 45µm). It is observed that 90% of the ashes of the first and second harvest have a diameter of 84.43 µm and 89.44 µm, respectively. According to John [24], the pozzolanic activity would be compromised, requiring a grinding of these ashes [25].

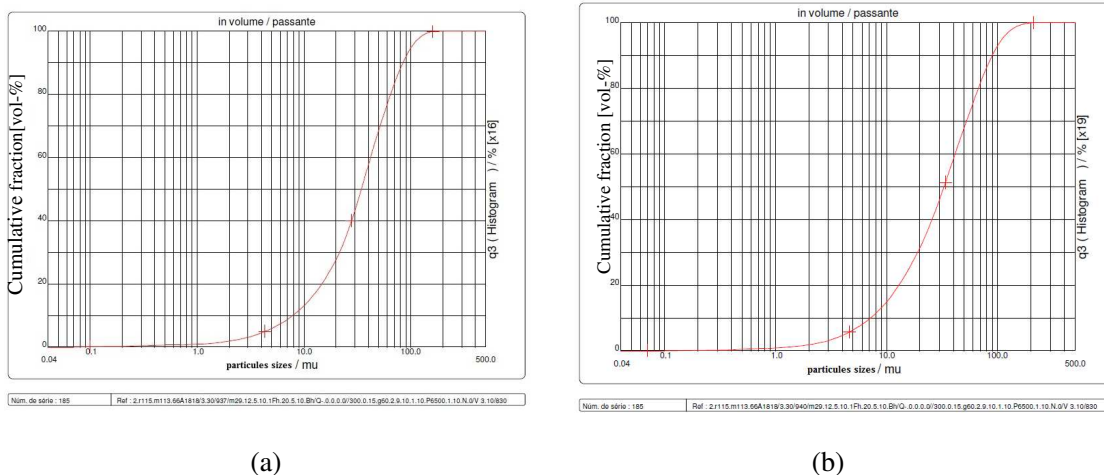


Fig. (3). Granulometric curve of ashes 1st (a) and 2nd (b) harvests.

Table 3. Average diameter and percentage diameter by-pass particle size obtained by laser granulometry method of the ashes.

Samples	Diameter (µm)			average
	10% (d10)	50% (d50)	90% (d90)	
1 st harvest ashes	7.94	34.52	84.43	41.26
2 nd harvest ashes	7.13	32.99	89.44	41.93

The specific surface results of the ashes are shown in Fig. (4). Each figure presents a variation of volume of nitrogen adsorbed and desorbed related to pressure. There is a hysteresis in the range adsorb and desorption curves. These data allow the estimation of the specific surface of the ashes from the 1st and 2nd harvests, being 39.5 and 46.8 m²/g, respectively.

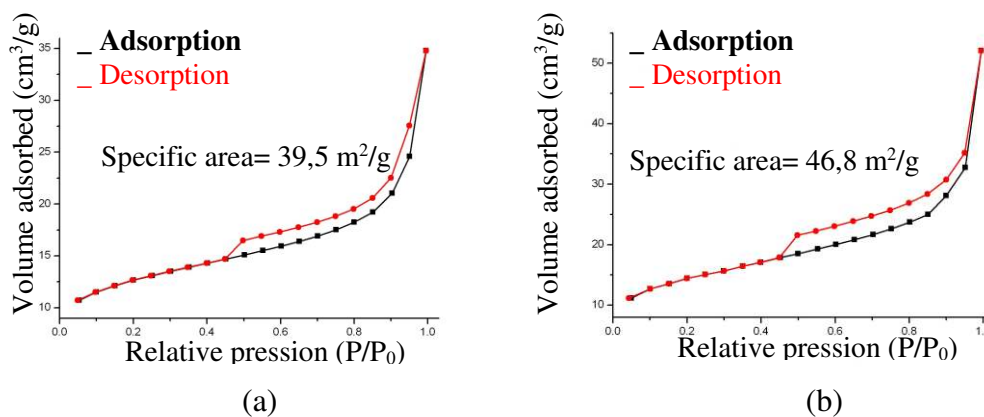


Fig. (4). Nitrogen adsorption test (BET) of the ashes from 1st (a) and 2nd (b) harvest.

Fig. (5) shows the infrared absorption of the ashes on the spectral range 300-4000 cm^{-1} . Both ashes show well defined bands with the presence of the band 1000 amorphous quartz and quartz crystalline at 3500 band, and the presence of vibration due to carbon-carbon bonds. The presence of quartz in the ashes could be related to the contamination of the soil [22].

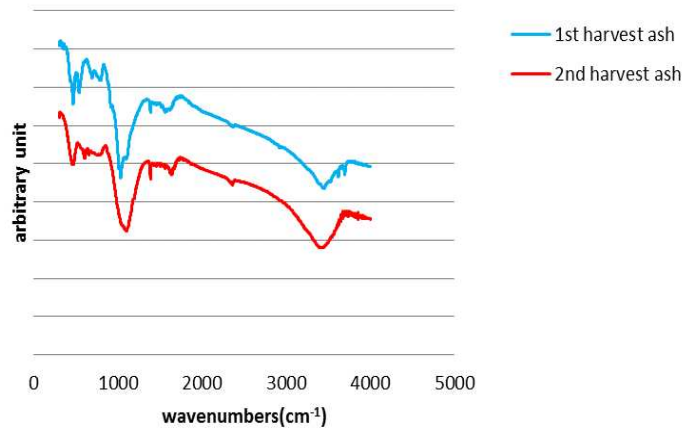


Fig. (5). Fourier transform infrared spectroscopy (FTIR) of the ashes.

Fig. (6) shows the XRD patterns of the ashes. The data clearly indicate that the ashes of the second harvest present predominantly non-crystalline structure, while the ashes of a first show less amorphicity. Thus, it was not possible to infer which of the samples has a higher content of amorphous silica.

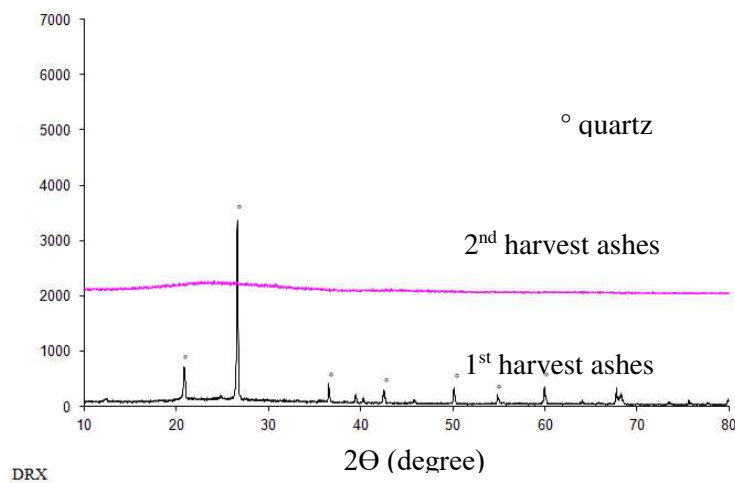


Fig. (6). X-ray diffraction of the *in natura* ashes.

Table 4 presents the results of pozzolanic activity index with cement (cement PAI) of the ashes from 1st and 2nd harvest. No differences were observed in PAI with cement from the ashes of both harvests. According to the IAP Brazilian standards the ashes would not be considered a pozzolanic material.

Table 4. Results of IAP with cement of the ashes.

Samples	IAP
1 st harvest ashes	59.71%
2 nd harvest ashes	59.79%

Table 5 presents the pozzolanic activity index with lime (lime IAP) of the ashes. Notice that there was not much difference in the results. Both ashes could be considered non-pozzolanic because their compressive strengths are under the minimum required by NBR 5751 [26], which is a compressive strength of 6 MPa. The IAP testing cement and these results are consistent. This result has not been reported in the work performed by Gobbi [27], which have used both IAPs to evaluate the metakaolin. In IAP testing with lime, this material was classified as highly reactive, however, the same method using cement revealed a non-pozzolanic material.

Table 5. Results of IAP with lime.

Samples	IAP
1 st harvest ashes	4.5
2 nd harvest ashes	4.0

Table 6 presents the test results of Modified Chapelle [28]. The data indicates that the 1st harvest ashes did not consume CaO, while the ashes from 2nd consumed a greater amount. Replicas of the tests were performed and the same results were found. The standard does not specify a parameter value. According to Raverdy [29], pozzolanic materials should react at 330 mg/g. However, the experiment lasted 10 hours, while the pattern requires 16 hours. Thus, considering that the reaction rate usually varied exponentially with time, it could roughly consider the 2nd harvest ashes as a pozzolanic material.

Table 6. Results of modified Chapelle test.

Samples	Results (mg/g)
1 st harvest ashes	0.00
2 nd harvest ashes	221.76

Table 7 presents the results obtained from the electrical conductivity test ashes. The ashes of the first harvest promoted a reduction in electrical conductivity of the solution around 0.456 mS/cm, while the ash of the second harvest increased the conductivity of the solution at 0.423 mS/cm. According to Luxan [17] the ashes of the 2nd harvest cannot be considered a pozzolanic material while the harvest 1st ashes would be a pozzolanic material. Soares [30] has found that burned ashes at a temperature of 800°C did not show pozzolanic activity. These results tests must be considered keeping in mind that it may be that Ca²⁺ ions are attracted to the fine particles of ashes and the reaction between ions and amorphous silica does not necessarily occur. Thus, the decrease in conductivity would be related to the specific surface of the ashes and the results of the test may not reflect the degree of pozzolanic activity of mineral admixture [31].

Table 7. Conductivities of Ca(OH)₂ saturated solutions with ashes.

Samples	Conductivity mS/cm		
	Initial	Final	Results (Initial - Final)
1 st harvest ashes	6.143	5.687	0.456
2 nd harvest ashes	7.225	7.648	-0.423

Fig. (7) presents the results of differential scanning calorimetry (DSC) of the paste made with and without (reference) 10% ashes, after 28 days of curing. An endothermic peak between 90°C and 110°C has been observed and a second peak between 460°C to 480°C in the three pastes subject to warming. It seems that the absorbed heat in these temperatures is equal to the cement paste and the latter with ashes from the 1st harvest. However, the absorbed heat from the ashes of the second harvest is higher. The heat absorbed in the interval of 460°C and 480°C are very similar to the pastes of cement and pastes with the ashes from the first harvest. In this interval, the heat absorbed from the paste with ashes from the 2nd harvest is lower. However, at intervals of 90° and 60° the heat absorbed by the pastes with ashes is higher.

Fig. (8) shows the results of thermogravimetry (TGA) of pastes made with and without 10% of ashes after 28 days of curing. In paste without ash of the 1st harvest a similar TGA was obtained as the one by Hernandez [32]. The results allow evaluating the reactivity of the constituents of the ash with cement. It is observed that the pastes from the 1st harvest show a gradual loss of mass of 40°C to 400°C. The curve of the pastes from the 1st harvest is reminiscent of the pastes reference to the 450°C, starting with this temperature the pastes from the 1st harvest lose a small amount of mass and shortly, at 560°C gains mass, with a decreasing after 760°C. However, the reference pastes undergo a gradual

decrease of mass to be stabilized. In the case of the 2nd harvest pastes, it is observed that the curve is very similar to the 1st harvest pastes, but with a higher weight loss. The mass gain occurs at the same temperature of the paste from the 1st harvest, but in smaller quantities. A mass gain at the same temperature was reported by Hernandez [32]. In total, reference pastes lost only 30wt%, while a 2nd harvest of pastes lost 35wt% mass and pastes of the 2nd harvest lost 40wt%. According to Rodrigues [33], between 100°C and 180°C there is the dehydration of ettringite and the CSH (calcium silicate hydrate), between 180°C and 240°C occurs the dehydration of the hydrate calcium aluminate and hydrated alumina silicates, and between 520°C and 580°C occurs the dehydroxylation of portlandite. The weight loss after 700°C indicates the release of CO₂ from the decomposition of CaCO₃ [32]. Thus, it is expected that the more pozzolanic ashes have approximately 200°C, higher water loss and the lower dehydroxylation of portlandite. Whereas reaction of dehydroxylation portlandite begins at 440°C, it is possible to roughly assess the amount of CH in each mixture. The sample without ash showed 14.4% of Ca(OH)₂ and one with ash on the order of 10.3%.

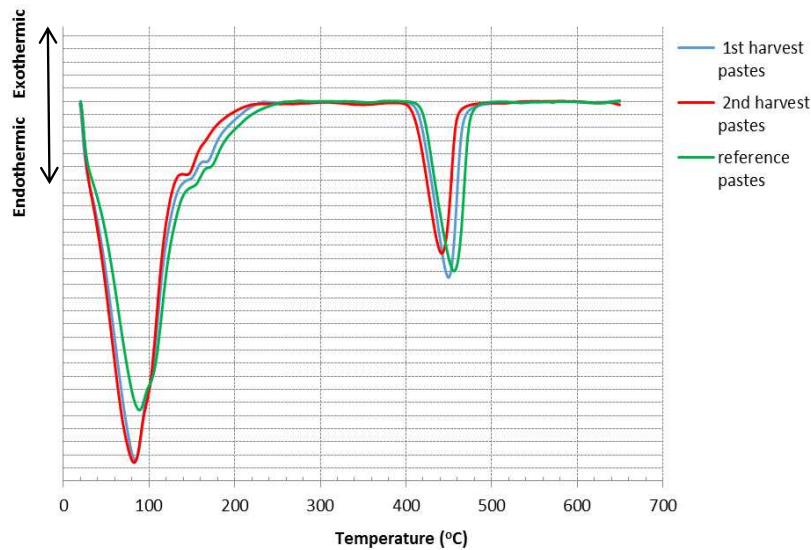


Fig. (7). Differential scanning calorimetry of pastes with and without (reference) ashes.

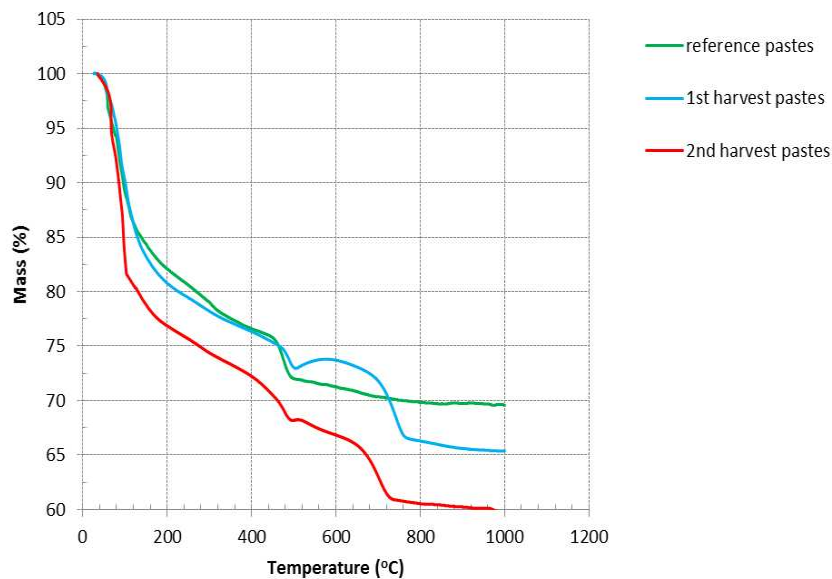


Fig. (8). Thermogravimetry analysis (TGA) of pastes with 90% of the cement and 10% of the *in natura* ashes 1st and one with 2nd harvest and one without ashes (100% cement).

Fig. (9) presents the results of Fourier transform infrared spectroscopy (FTIR) of the pastes made with and without the ashes of harvest 1st and 2nd after 28 days of curing. The data show that the pastes with and without ash have quite similar bands. However, it was not possible to identify the compounds due to the absence of reference parameters.

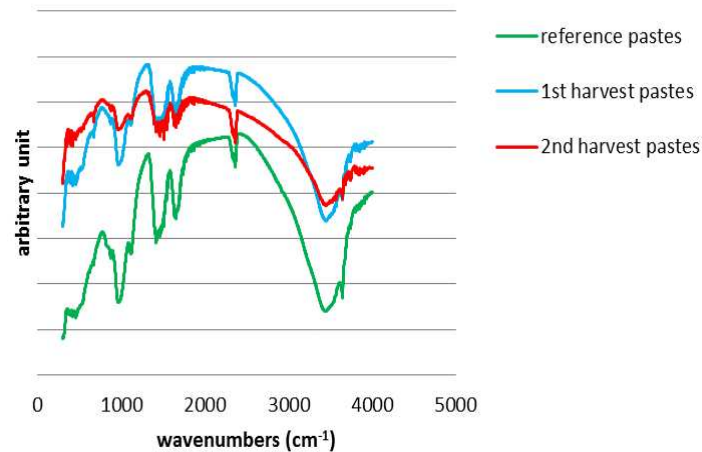


Fig. (9). Fourier Transform Infrared spectroscopy (FTIR) of pastes with 90% of the cement and 10% of the *in natura* ashes 1st and one with 2nd harvest and one without ashes (100% cement).

CONCLUSION

The ashes from different harvests have different chemical compositions and degree of amorphicity. However, the higher content of CaO and SO₃ in one of the ashes and their higher loss on ignition indicate that the incinerations conditions would be responsible for these differences. According to chemical composition and Luxan test the ashes from the 1st harvest can be considered a pozzolanic material. The size distribution shows that both ashes could be pozzolanic materials. The results of differential scanning calorimetry (DSC) and modified Chapelle methods implied the presence of pozzolanic effect by the ashes from the 2nd harvest. The results of pozzolanic activity index (with cement or lime) indicated that the ashes are not pozzolanic. No differences were observed in IAP with cement from the ashes of the 1st and 2nd harvests. The ashes showed similar pozzolanic activity in thermogravimetry analysis. The methods used to evaluate the pozzolanicity of the materials do not present coherent results.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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