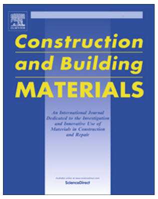
Construction and Building Materials 98 (2015) 44–50

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat



Assessing the pozzolanic activity of cements with added sugar cane straw ash by synchrotron X-ray diffraction and Rietveld analysis

Guilherme A. Calligaris [a], Margareth K.K.D. Franco [b,*], Laurence P. Aldrige [c], Michelle S. Rodrigues [d], Antônio Ludovico Beraldo [d], Fabiano Yokaichiya [b,e], Xavier Turrillas [f], Lisandro P. Cardoso [a]

[a] *Universidade Estadual de Campinas (UNICAMP), Instituto de Física Gleb Wataghin (IFGW), 13083-859 Campinas, SP, Brazil*
[b] *Comissão Nacional de Energia Nuclear (CNEN), Instituto de Pesquisas Energéticas e Nucleares (IPEN), Reator Multipropósito Brasileiro (RMB), Brazil*
[c] *Institute of Material Engineering, ANSTO, PMB 1 Menai, New South Wales 2234, Australia*
[d] *Universidade Estadual de Campinas (UNICAMP), Faculdade de Engenharia Agrícola (FEAGRI), Campinas, SP, Brazil*
[e] *Helmholtz-Zentrum Berlin für Materialien und Energie (HZB), Germany*
[f] *Consejo Superior de Investigaciones Científicas, Institut de Ciència de Materials de Barcelona, Spain*

HIGHLIGHTS

- Sustainability is the key word in the development of new technologies.
- The use of alternative materials in the cement paste is focused.
- Cement pastes and cement pastes blended with Sugar Cane Straw Ashes were studied.
- Our findings contribute in the phase identifications of cementitious pastes.
- The pozzolanicity of the agroindustrial wastes was proven using synchrotron radiation.

ARTICLE INFO

Article history:
Received 6 June 2014
Received in revised form 7 August 2015
Accepted 10 August 2015
Available online 24 August 2015

Keywords:
Synchrotron radiation characterisation
Sugar cane straw ash
Pozzolan
Waste management

ABSTRACT

Sugar and alcohol industries generate large amount of wastes that could produce ashes of great reactivity with pozzolan properties. The objective of this paper is to evaluate the pozzolanicity of Sugar Cane Straw Ashes (SCSA), thermal treated, at different curing times. Employing Synchrotron X-ray radiation for XRD measurements, scans from 10° to 110° ($\theta - 2\theta$ setup) allowed the quantification of several phases of the cement pasts through Rietveld analysis. The SCSA substitution of 20% (weight) in Ordinary Portland Cement (OPC) has improved the AFt (Ettringite) formation up to 47% for 90 days curing time. The Portlandite concentration analysis allowed concluding that this addition of SCSA in OPC has caused a delay in the cement setting time. Moreover, the behaviour of the C3S and Calcite contents in both OPC and OPC/SCSA samples were determined by refinement of the XRD pattern using the Rietveld method.

1. Introduction

Industrial production of cement in Brazil started in 1920, reaching a maximum of 40.2 million tones in 1999 [1]. Despite of a series of up and downs due to economic upheaval, another peak of 65 million tons was attained in 2011. Preliminary information about cement consumption on April 2013 reached 5.9 million of tons [2].

Nowadays, the Brazilian cement industry is among the 10 largest cement world producers and consumers, has a modern industrial park, is internationally recognised for recycling waste minerals and by preserving the environment (low CO_2 emissions) and also by the use of biomass for power generation. Despite these mitigating factors, the expansion of construction activity in Brazil motivates the search for materials that can replace cement. This replacement is necessary due to the environmental and social impacts caused by the production of cement.

Researches involving the use of pozzolans in cementitious matrices have increased significantly in recent years. In the absence of natural pozzolan, cement industries look for new materials in agroindustry. Several authors have reported the use of agroindustrial ashes to partially replace Portland cement [3–7].

The use of sugar cane straw, as forage, contributes to decrease the soil erosion and to reduce the carbon emission from soil to atmosphere [8]. Furthermore, the wastes of sugar cane production can be recycled by biorefinaries to get energy with a substantial

---

* Corresponding author.
E-mail address: margareth_franco@yahoo.com.br (M.K.K.D. Franco).

http://dx.doi.org/10.1016/j.conbuildmat.2015.08.103
0950-0618/

generation of ashes. Since Brazil is the world's largest producer of sugar cane having a huge organic by-products surplus of bagasse and straw [8,9] has a strong interest in finding a way to recycle this waste. One possible solution could be its combustion. Indeed, some authors have reported that these wastes when burned under controlled temperature may show high reactivity, reacting as a pozzolanic material [10,11].

To assess a pozzolanic material it is necessary to carry out a complete series of chemical, physical and mechanical tests to evaluate its performance. One of them involves the direct identification of calcium silicate hydrates (CSH) phases, promoted by pozzolanic reaction, as a function of time [12–14] up to ninety days. This can be carried out by X-ray diffraction, quantifying both the initial crystalline phases and the hydration products over long periods (months), since the pozzolanic reaction is normally slow.

The aim of this paper, consequently, is to evaluate the pozzolanic reaction in blended cement pastes, both without (control) and with sugar cane ashes added, treated at 700 °C, for periods of three, seven, twenty-eight and ninety days. To achieve that, synchrotron X-ray diffraction data acquired at the Brazilian facilities of LNLS, have been studied and crystalline phases quantified by Rietveld analysis in order to have a picture of the major phases evolution during the cement pastes hydration.

2. Experimental procedure

2.1. Materials

2.1.1. Sugar Cane Straw Ashes (SCSA)

The sugar cane straw was harvested from Centro de Tecnologia Canavieira (CTC), placed at Piracicaba, São Paulo – Brazil. It was collected directly from the ground and exposed to the natural environment to naturally dry for 24 h. The ash was obtained from the burning control in an electrical furnace with 10 °C/min heating rate during 3 h at 700 °C X(hereafter SCSA). The production of the ashes was divided in two levels in order to obtain a homogeneous burning, according to Fig. 1. On reaching the first temperature level, 400 °C, the system remains 20 min in order to burn all the organic matter to get the best homogenisation of the ash. In the second level, the material was maintained at 700 °C for 1 h. At the end, the muffle furnace was turned off and a slow and natural cooling process began. The muffle furnace model 10013 from Jung, used to treat the samples, has the dimension of 27 cm × 40 cm × 100 cm. Afterwards, it was used a rotor mill from Tecnal to mill the ashes at 200 rpm during 120 min.

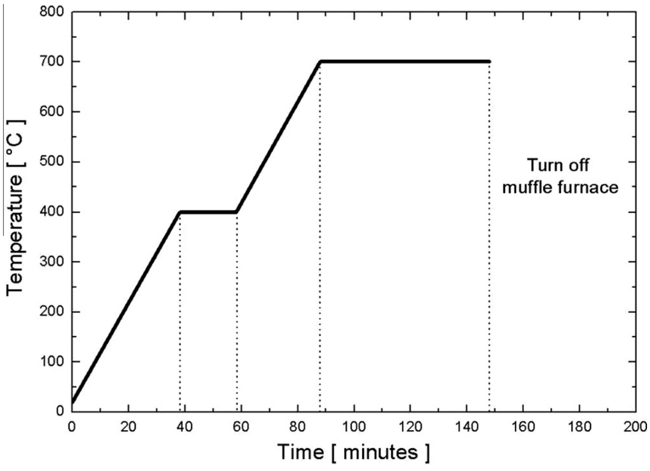


Fig. 1. Temperature treatment of the sugar cane straw ash.

2.1.2. Ordinary Portland Cement (OPC) and OPC/SCSA

The highly initial resistance cement, Ordinary Portland Cement (OPC), (CPV-ARI, Cauê) was utilised in this study, according to the ABNT NBR 5733 (1991) [15]. This cement does not show mineral additions. On the other hand it shows higher quantities of C3S and C2S than others, resulting more reactivity. It was prepared two pastes, the control (pure OPC) and OPC/SCSA (80%/20% by weight). OPC, without mineral addition, calcium hydroxide (95% of purity) and deionised water were used in blended cement pastes. Firstly, OPC and ash was mixed and then it was added deionised water; the water/binder ratio was 0.5. After mixing the materials, pastes were stored in sealed plastic bottles and then left in a curing room at 20 °C until the tests applied. At ages test manual grinding pulverised the samples. The hydration of the samples was stopped by putting the samples into an acetone solution. Afterward, samples were dried at 60 °C for 30 min in furnace.

2.2. Experimental characterisation techniques

2.2.1. SCSA

The chemical composition of the ash was determined using the X-ray fluorescence method, Axios from Panalytical, and the loss on ignition (LOI) was measured according with ASTM C 114 method [16]. X-ray diffraction (XRD) of SCSA was performed at D10B-XPD [17] and D12A-XRD1[18] in the Brazilian Synchrotron Laboratory (LNLS) using 4 + 2-circle Huber diffractometers in a high-resolution mode, with Ge 111 analyser crystal, at 8 keV. This mode was chosen in order to minimise the superposition of neighbouring Bragg peaks allowing for more reliable solution of the mineralogical characterisation. Data were obtained at room temperature, in a $\theta - 2\theta$ geometry (Bragg–Brentano configuration), with a flat plane sample holder, over a 2θ range of 10–70° with a step size of 0.01° and a step time of 1s. Granulometric distribution of Ordinary Portland Cement and ash were measured using a Malvern Mastersizer 2000 apparatus which allows an analysis of particles by laser diffraction from 0.02 to 2000 μm in liquid mode as dispersant, with 10–15% of obscuration and ultrasonic agitation for 60 s. The fineness of the material is an important parameter to evaluate the pozzolanicity. Higher the contacts surface of the pozzolan with calcium hydroxide, greater the rate of pozzolanic reaction.

2.2.2. Ordinary Portland Cement (OPC) and OPC/SCSA

Synchrotron X-ray powder diffraction has become a well-established technique, being suitable for applications in characterisation of cementicious materials [19,20]. This technique that presents the advantage of large photon flux of a synchrotron source and high resolution compared with the conventional X-ray diffractometers, allow us to evaluate with more accuracy the composition of blend paste cement in different curing time. In order to monitor the reaction between SCSA and $Ca(OH)_2$, XRD analysis at 3, 7, 28, 90 days was applied to the pastes.

3. Results and discussion

3.1. Chemical and physical characterisation of SCSA

The chemical composition and LOI of the ash are shown in Table 1. A pozzolan should present in its chemical composition silica as the predominant phase and at least 50% of the sum of the oxides: SiO_2, Al_2O_3 and Fe_2O_3. The presence of certain chemical elements such as sodium and potassium may be undesirable, because they provoke alkali-aggregate reaction in the cement matrix. The SO_3 content should be a maximum of 5% [16]. The chemical composition of SCSA is presented in Table 1, contain 61.0% of SiO_2,

Table 1
Chemical composition and loss on ignition (LOI) of the ash, in mass percent.

SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	SO_3	P_2O_5	K_2O	Cl	LOI
61.0	9.20	4.99	1.29	4.40	2.79	0.15	3.85	2.29	6.98	0.55	2.08

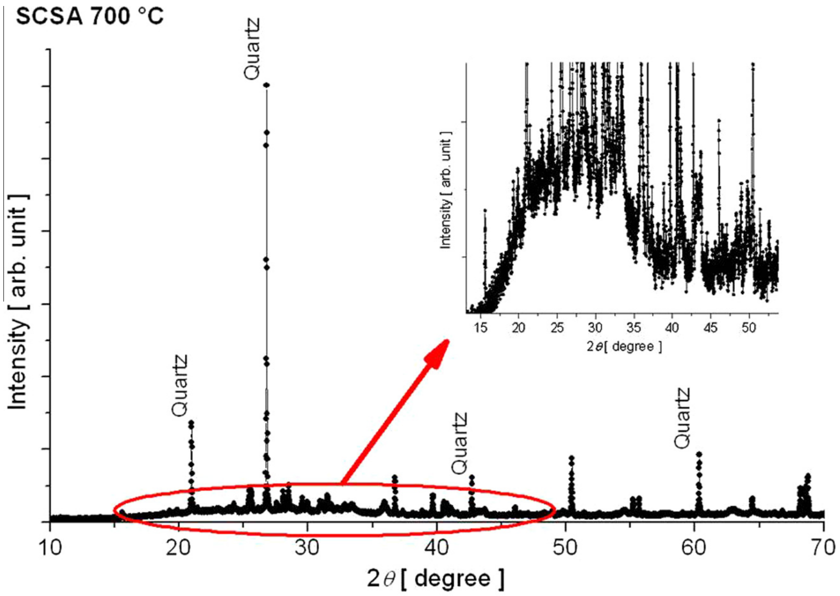


Fig. 2. XRD pattern of SCSA.

and low content of undesirable elements. The loss on ignition is also consistent with the maximum value stipulated by the standard, which is 6.0%.

X-ray diffraction (XRD) of ash were obtained at room temperature, in a $\theta - 2\theta$ geometry, over a 2θ range of 10–70° with a step size of 0.01° and a step time of 1s. Fig. 2 shows the mineralogical characterisation of the SCSA.

Through the technique of X-ray diffraction (XRD), it becomes possible to identify differences in the formation of amorphous or crystalline silica; moreover amorphous material presents high reactivity. XRD pattern of SCSA indicates the presence of quartz and amorphous structure for the silica as displayed in Fig. 2.

In the inset of Fig. 2, we observe an amorphicity halo in the angular 2θ range of 20–30° further crystalline phases. Vitreous materials show a central halo at the 2θ around 22° which it is related to the overlap of amorphous forms of silica [21]. Thus, this amorphicity halo indicates the ash reactivity.

The fragmentation of the particles of mineral additives is required to activate chemical reactions, and these reaction rates are proportional to the specific surface of the material, which varies as the inverse of particle size [22]. The particle size (expressed in μm) distribution parameters of ash are: 1.545, 9.022, 46.995 and 17.40 for D_{10}, D_{50}, D_{90} and the average size, respectively. Also in Fig. 3 the graphical distribution can be seen.

Materials with D80 (passing size) below 60 μm and specific surface area above 300 m^2/kg result in materials that can be classified as pozzolans [23]. Therefore, the SCSA has a high fineness, which is an indication of reactivity.

3.2. XRD analysis

X-ray diffraction is a well-known and very useful tool for structural analysis, since the interaction of this kind of radiation with the analysed material provides important information for a good characterisation, allowing qualitative studies just by observing "fingerprints" of the sample components. The association of this knowledge with the advantage of the high intensity and resolution of the beamline employed here [17], it was possible to identify all primary phases in the pure OPC and OPC/SCSA pastes, such as Alite, Belite, Portlandite, Calcite, Ettringite and Brownmillerite, identified on Table 2. Therefore, Fig. 4 shows the comparison between the pure OPC and treated OPC/SCSA at 700 °C, aged 3, 7, 28 and 90 days of curing time, as well as the above mentioned phases, besides the presence of other distinct phases, although they are not identified directly on Fig. 4, just the first three ones and quartz. It should be noted that quartz was only observed on samples containing SCSA provided by extraction ground site residues.

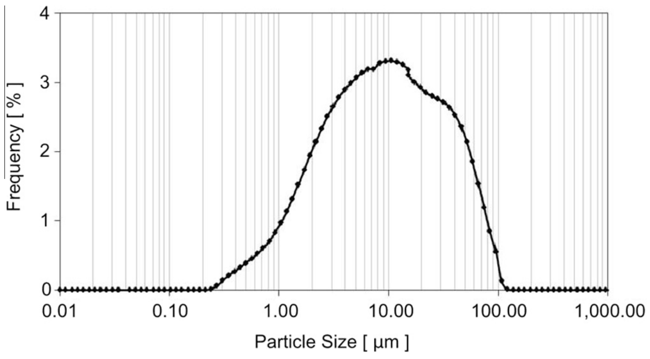


Fig. 3. Particle distribution size.

The presence of Portlandite (CH) contribution in pure OPC and OPC/SCSA (700 °C) samples was not observed for SCSA sample (Fig. 2), which confirms its presence occurs only through the mixture of OPC and SCSA. Quantitative analysis is always a good improvement for analyses and, for X-ray diffraction, it can

Table 2
Phase description of the SCSA/OPC pastes component.

Phase	Formulae	Crystal system notation	Nomenclature
Alite [39]	C_3SiO_5	Trigonal R3m	C_3S
Belite [38]	C_2SiO_4	Monoclinic $P12_1/c1$	C_2S
Portlandite [36]	$Ca(OH)_2$	Trigonal P-3m	CH
Ettringite [34]	$Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$	Trigonal P31c	AFt
Calcite [35]	$CaCO_3$	Trigonal R-3c	$CaCO_3$
Brownmillerite [37]	$Ca_2(Al,Fe)_2O_5$	Orthorhombic Pcmn	C_4AF
Quartz [40]	SiO_2	Trigonal P32 2 1	SiO_2

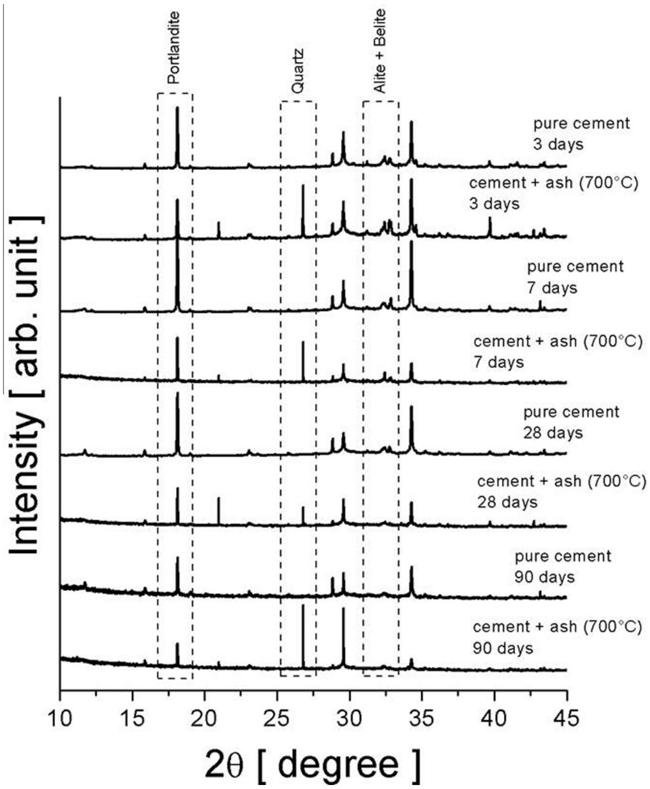


Fig. 4. Selected region of XPD patterns for pure cement cured at 3, 7, 28 and 90 days compared to cement with SCSA cured at the same ages.

normally be achieved by discriminating the major identified phases and then, taking the ratio between their more intense peak areas [24]. This represents a simple approach or a first approximation method, which is not so robust when applied to overlapping peaks and that should be replaced with several advantages by the Rietveld structure refinement method [25–28]. Rietveld analysis is able to distinguish among overlapped peaks from distinct phases, using published structures from the literature, also allowing a full pattern analysis for the XRD data essential for the quantification of all included phases of the investigated samples.

In this study, Rietveld analysis was done with the help of TOPAS [29]. Usually, the refinement result appears as both experimental and calculated data, as well as the graph of the difference between them plotted below, in a single diagram, as shown in Fig. 5 for the OPC (5.a) and OPC/SCSA 700 °C (5.b) cured for 3 days. The Bragg peak positions of the crystalline phases used in the refinement are also depicted at the bottom of Fig. 5. A good briefing to the refinement status is the reliable factors (R_{exp}, R_{wp}) and the GOF (Goodness of Fit), shown in Table 3. Although all results are exhibited in the table, only the 3 days OPC and OPC/SCSA analyses are plotted.

Fig. 6 shows the phase analysis of a control sample (OPC). One can observe that the quantity of CH increases 20% in the cure period from 3 to 90 days. On the other hand the sum of the polymorphs C3S and C2S decrease approximately 21%. The AFt has two behaviours: (1) the percentage remains around 3–4% from 3 to 7 days; (2) the percentage increases to 6% from 7 to 28 days and to around 9% from 28 to 90 days. According to the literature [30], AFt delays the degradation process of the polymorphs alite and belite, and consequently the production process of portlandite.

Fig. 6 displays all phases observed in OPC and OPC/SCSA at different curing times, for easier comparison of the samples. Note that only for OPC/SCSA samples it is observed the low-quartz. The portlandite (CH) behaviour, for the first 28 days, is quite different for OPC and OPC/SCSA samples. For OPC, its concentration has a great increase for this period, while for OPC/SCSA it appears roughly stable.

Distinct assumptions can be considered regarding C3S, C2S and calcite. One can observe from Fig. 6(a) and (b) that, while C2S content is almost the same for both phases after 90 days, C3S exhibits a three times higher content for the OPC/SCSA in comparison to OPC sample. Then, one can assume that OPC/SCSA is more brittle than the OPC paste. However, a closer observation of these results shows that, the calcite phase also presents an increment (two times) in its concentration ratio. This difference in calcite concentrations should give to the OPC/SCSA sample more strength or, in other words, a better resistance to compression [30]. Actually, these two distinct observations should compensate each other and, the investigation of the resistance to compression by means of the Tukey method [31], for both OPC and OPC/SCSA samples have shown similar results. Furthermore, a broader vision of the calcite content observation is shown in Fig. 7, where a distinct behavior for both samples up to 28 days happens, while it increases for OPC/SCSA, decreases for OPC, corroborating the above mentioned conclusions.

According to the literature [32,33], the addition of reactive pozzolan, as gypsum and other sulphate compounds, react with calcium aluminate in the cement to form ettringite within the first few hours after mixing with water. Within 24 h, essentially all of the sulphur in the cement is consumed to form ettringite. However, Fig. 8 reveals that the reaction to produce AFt continues with the curing time in a different way for OPC and OPC/SCSA pastes. For the OPC/SCSA, the AFt portion increases by approximately 47% if compared to the AFt concentration in OPC for 28 days curing time.

4. Conclusions

The objective of this paper is to investigate important issues such as ash addition in OPC, the effect of curing time in pure Ordinary Portland Cement (OPC) and Sugar Cane Straw Ashes (OPC/SCSA – @700 °C), that needs to be studied to evaluate its industrial applicability. The idea of introducing agroindustrial wastes, as sugar cane ashes into the OPC, opens a wide field of

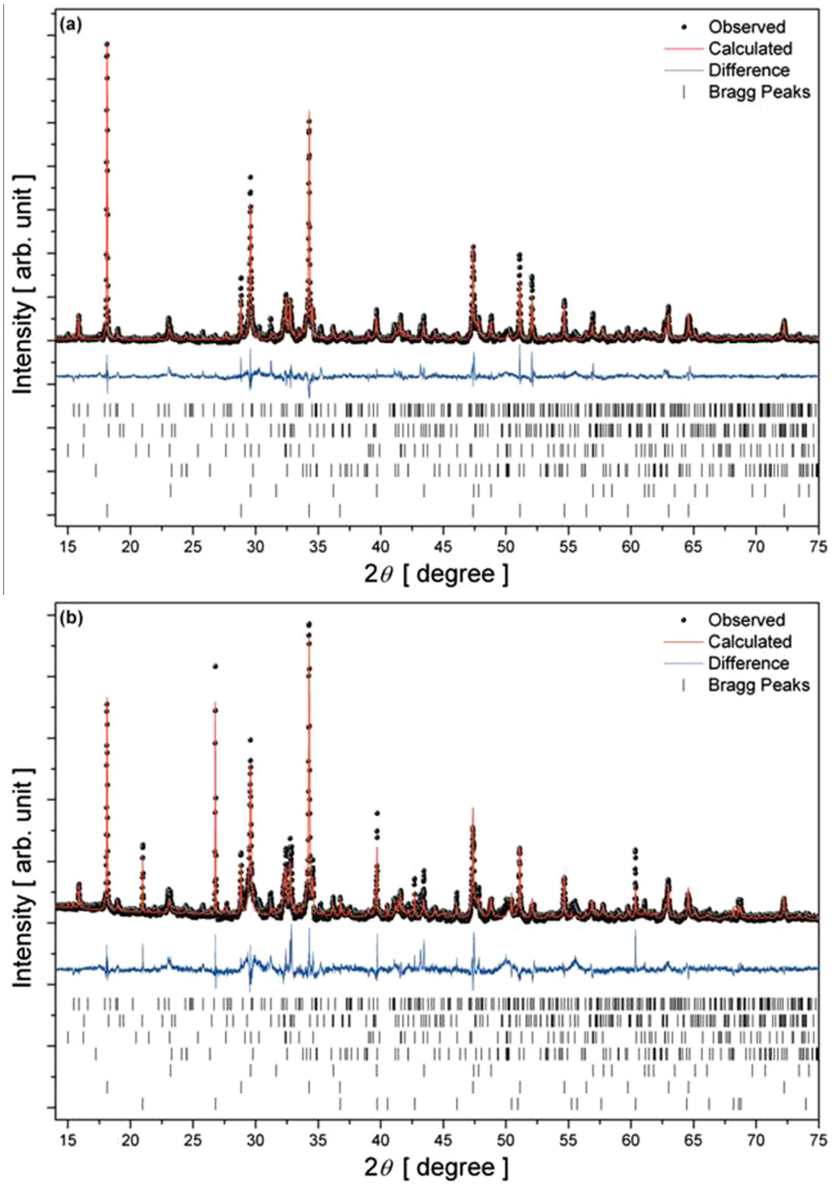


Fig. 5. Diffractograms of OPC (a) and OPC/SCSA (700 °C) (b) samples appear also with the angular position of the identified phases from top to bottom: Ettringite, Belite, Alite, Brownmillerite, Calcite, Portlandite and Quartz (present only in OPC/SCSA sample). Both samples were taken for 3 days of curing time.

Table 3
Rietveld reliability factors and GOF for all analysed samples.

Sample	R_{wp}	R_{exp}	GOF (R_{wp}/R_{exp})
OPC 3 days	13.10	8.12	1.61
OPC 7 days	13.45	6.51	2.07
OPC 28 days	13.36	6.04	2.21
OPC 90 days	17.38	14.40	1.21
OPC/SCSA 3 days	12.77	7.43	1.72
OPC/SCSA 7 days	15.18	11.99	1.27
OPC/SCSA 28 days	14.28	11.49	1.24
OPC/SCSA 90 days	14.24	12.59	1.13

applicability. Therefore, it is necessary to know its behaviour according to the mineralogical variability.

The existence and behaviour of the main compounds such as alite, belite, portlandite, ettringite, calcite, brownmillenite and quartz observed into OPC and OPC/SCSA(@700 °C) were analysed using the association of the synchrotron radiation at LNLS, Campinas, SP, Brazil, and, the Rietveld refinement method. Nevertheless, it should be mentioned that, the present paper is focused in the crystalline phases of the compounds and therefore, the well-known amorphous component C–S–H was not taken into account in the analyses.

The comparison between the Portlandite behaviour in both OPC and OPC/SCSA samples, allowed to conclude that the addition of the SCSA in OPC cause a delay in the cement setting time (hardness process), through the analysis of the early stages, i.e., 3 days cured samples. This delay is the consequence of the sum of the effects induced by existing gypsum ($\leqslant$3%, not computed in Rietveld analysis) in the cement Portland and by the silicates, aluminates and ferrites that also composed the SCSA. The observed difference in the evolution of AFt for the two OPC and OPC/SCSA samples (up to 47%) gives rise to the conclusion that the substitution of 20% of cement by SCSA, provokes a significantly increase of AFt, as expected from pozzolanic materials. The similar observed behaviour for the C3S (more brittle) and calcite (more strength) of higher concentrations for OPC/SCSA samples in comparison to the OPC, after 90 days, leads to opposite conclusions regarding

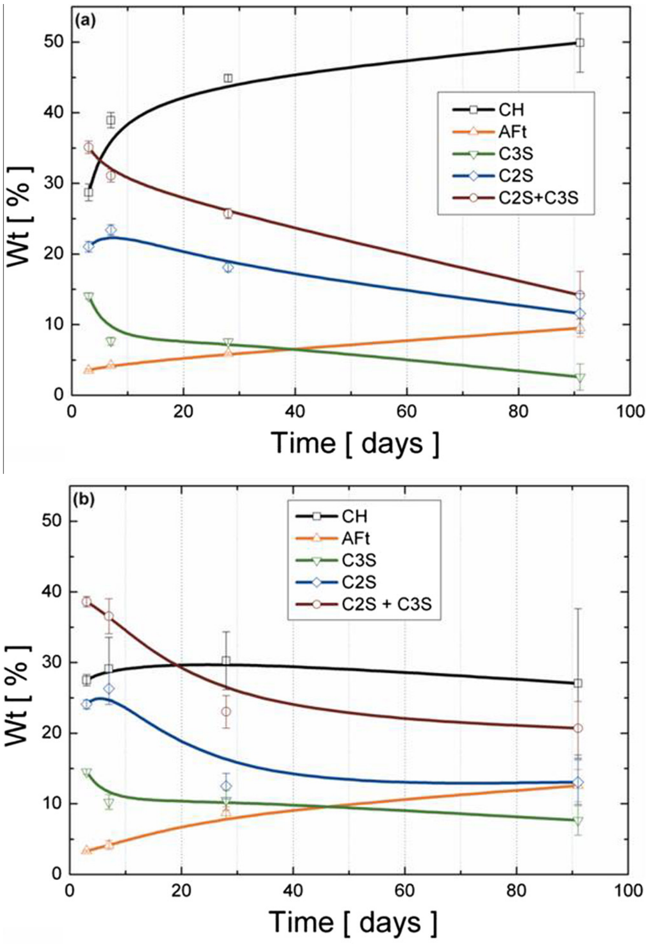


Fig. 6. Observed phases at (a) OPC and (b) OPC/SCSA: CH, C2S, C3S and AFt.

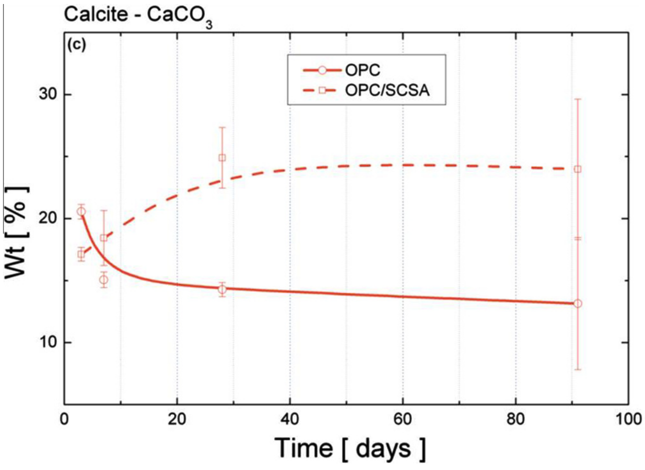


Fig. 7. The evolution behaviour of calcite from OPC and OPC/SCSA.

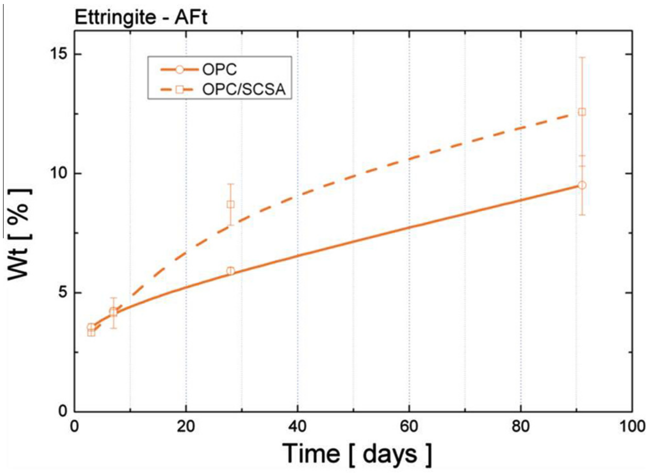


Fig. 8. Evolution of the Ettringite phase for OPC and OPC/SCSA.

the resistance to compression. Nevertheless, it was concluded that they compensate each other, a result that was corroborated by Tukey method, as well as by the distinct evolution of the calcite content in the early stages (up to 28 days). The cement industry is responsible for around 7% of CO_2 emissions in the planet due to the combustion and to the decarbonation process. The use of sugar cane ashes in the Portland cement should contribute as a significant improvement in the sustainability. Furthermore, the pozzolan from the agroindustrial wastes frequently is cheaper than the clinker, notwithstanding its substitution to be limited. The conclusion of this study is that the replacement of 20% of the clinker by the SCSA, shows the same mechanical resistance as a good option to recover the agroindustrial wastes. The OPC/SCSA pastes demonstrate a similar mechanical behavior as the OPC pastes, including the 7 days curing time. Here, it has been proven the pozzolan activity of the SCSA through the X-ray diffraction measurements associated with Rietveld analysis.

References

[1] Associação Brasileira de Cimento Portland, http://www.abcp.org.br/conteudo, 2012.
[2] SNIC Sindicato Nacional da Indstria do Cimento, Relatrio Anual 2010, Brazil, http://www.snic.org.br, 2012.
[3] J.F. Martirena Hernández, B. Middendorf, M. Gehrke, H. Budelmann, Cem. Concr. Res. 28 (1998) 1525–1536.
[4] S.K. Malhotra, N.G. Dave, Cem. Concr. Compos. 21 (1999) 285–291.
[5] F. Martirena, B. Middendorf, R.L. Day, M.P. Gehrke, P.L. Roque, L.S. Martnez, S. Betancourt, Cem. Concr. Res. 36 (2006) 1056–1061.
[6] J. Payá, J. Monzó, M.V. Borrachero, L. Díaz-Pinzón, L.M. Ordóñez, J. Chem. Technol. Biotechnol. 77 (2002) 321–325.
[7] V.N. Dwivedi, N.P. Singh, S.S. Das, N.B. Singh, Int. J. Phys. Sci. 1 (2006) 106–111.
[8] FAPESP Fundação de Amparo à Pesquisa do Estado de São Paulo, Biorrefinarias Polivalentes, in: Agência FAPESP, http://www.agencia.fapesp.br/15613, 2012.
[9] CONAB: Companhia Nacional de Abastecimento. Cana-de-açúcar, http://www.conab.gov.br, 2012.
[10] M. Frías, M.I.S. de Rojas, J. Cabrera, Cem. Concr. Res. 30 (2000) 209–216.
[11] M. Frías, E. Villar-Cociña, E. Valencia-Morales, Waste Manage. 27 (2007) 533–538.
[12] L.J. Parrott, M. Geiker, W.A. Gutteridge, D. Killoh, Cem. Concr. Res. 20 (1990) 919–926.
[13] M.F. Rojas, M.I. Sánchez de Rojas, Cem. Concr. Res. 35 (2005) 1292–1298.
[14] F. Ridi, E. Fratini, P. Baglioni, J. Colloid Interface Sci. 357 (2011) 255–264.
[15] Brazilian Technical Standard Association Rio de Janeiro. ABNT NBR 5733 High early strength Portland cement. Specification, Rio de Janeiro, 1991.
[16] American Society for Testing And Materials, Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete: ASTM C 618, Philadelphia, 2012.
[17] F.F. Ferreira, E. Granado, W. Carvalho Jr, S.W. Kycia, D. Bruno, R. Droppa Jr, J. Synchrot. Radiat. 13 (2006) 46–53.
[18] C. Cusatis, M. Kobayashi Franco, E. Kakuno, C. Giles, S. Morelhao, V. Mello Jr, I. Mazzaro, J. Synchrot. Radiat. 5 (1998) 491–493.
[19] A.G. De La Torre, S. Bruque, J. Campo, M.A.G. Aranda, Cem. Concr. Res. 32 (2002) 1347–1356.
[20] V.K. Peterson, B.A. Hunter, A. Ray, J. Am. Ceram. Soc. 87 (2004) 1625–1634.
[21] M.G. Silva, Cimento Portland com adições minerais, Materiais de construção civil e princípios de ciências e engenharia de materiais, 2007.
[22] Z. Zhu, X. Wang, S. Dai, B. Huang, Q. He, J. Mater. Civil Eng. 2563–69 (2013).
[23] C.G. Cordeiro, R.D. Toledo Filho, L.M. Tavares, E.M.R. Fairbairn, Cem. Concr. Compos. (2008) 30410–30418.
[24] R.A. Young (Ed.), The Rietveld Method. International Union of Crystallography, Oxford University Press, New York, 1993, p. 298.
[25] H. Rietveld, J. Appl. Crystallogr. 2 (1969) 65–71.
[26] M. Paul, Microstruct. Anal. Mater. Sci. 15–17 (2005).

[27] G. Le Saoût, V. Kocaba, K. Scrivener, Cem. Concr. Res. 41 (2011) 133–148.

[28] F. Guirado, S. Gaí, Chinchón, Cem. Concr. Res. 30 (2000) 1023–1029.

[29] A.A. Coelho, J. Evans, I. Evans, A. Kern, S. Parsons, Powder Diffr. 26 (2011) 22–25.

[30] H.F.W. Taylor, Cement Chemistry, Academic Press, London, 1990.

[31] M.S. Rodrigues, Avaliação de Cinzas de Palha de cana de açúcar e sua utilização como adição mineral em matrizes cimentícias (Ph.D. thesis), University of Campinas (UNICAMP), Campinas, 2012.

[32] F. Goetz-Neunhoeffer, J. Neubauer, P. Schwesig, Cem. Concr. Res. 36 (2006) 65–70.

[33] G. Renaudin, Y. Filinchuk, J. Neubauer, F. Goetz-Neunhoeffer, Cem. Concr. Res. 40 (2010) 370–375.

[34] F. Goetz-Neunhoeffer, J. Neubauer, Powder Diffr. 21 (2006) 4–11.

[35] D. Li, B.H. O'Connor, H. Sitepu, J. Appl. Crystallogr. 38 (2005) 158167.

[36] D.M. Henderson, H.S. Sgutowsky, Am. Mineral. 47 (11-1) (1962). 1231.

[37] A.A. Colville, S. Geller, Acta Crystallogr. Sect. B Struct. Crystallogr. 27 (DEC15) (1971). 2311.

[38] W.G. Mumme, R.J. Hil, G. Bushnell-Wye, E.R. Segnit, Neues Jahrb. Mineral. Abh. 169 (1) (1995) 35–68.

[39] A.M. Ilinets, I.A. Malinovski, N.N. Nevski, Dokl. Akad. NAUK SSSR 281 (2) (1985) 332–336.

[40] S.M. Antao, I. Hassan, J. Wang, Feldspars 2007 Session held at the Frontiers in Mineral Sciences Symposium, Canadian Mineralogist, 46, 1501–1509, 2008