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Use of biomass fly ash for mitigation of alkali-silica reaction of cement mortars

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ABSTRACT

The degradation of large concrete structures over time is well known. One of the main reasons is the reaction that occurs between the cement paste and some reactive siliceous aggregates, which causes a significant expansion that depends on the employed materials and exposure conditions of the structure. This process is known as alkali-silica reaction (ASR) and affects several structures worldwide, including major dams and bridges in long time run. In this work the effect of fly ashes from biomass combustion in the mitigation of the ASR was investigated. The fly ashes were collected from two industrial plants located in the central area of Portugal: (i) a thermal power plant (BFA_1), (ii) co-generation process of a pulp and paper industry (BFA_2). The fly ashes were characterised by different techniques to determine the following properties: particle size distribution (laser interference), loss on ignition and thermal behaviour (TG/DTA), chemical (XRF) and phases (XRD) composition and pozzolanic activity (EN 196-5:2005). These biomass fly ashes were irregular in shape and fine in size. The chemical characterisation revealed significant differences in CaO and SiO_2 contents, but both fly ashes can be considered as class C fly ashes if compared with those generated from the coal combustion.

Accelerated mortar-bar tests were conducted according to ASTM C1260/ASTM C1567 to evaluate the behaviour of the biomass fly ash in the ASR inhibition mechanism. The expansive behaviour was studied on mortars where the cement was partially replaced (20–30 wt%) by the biomass fly ashes. This substitution tends to reduce the expansion upon accelerated curing conditions, and BFA_2 is more effective than BFA_1. But the incorporation of biomass fly ash in the blend along with metakaolin (MK), 20% BFA + 10% MK did a significant improvement in the expansion results, indicating the effective use of biomass fly ash along with metakaolin in mitigating the ASR.

1. Introduction

Since a long time, it has been observed that some aggregates incorporated in concrete develop reactions that damage the structures. In concretes and mortars, the occurrence of internal expansive phenomenon is normally recognized as alkali-aggregate reaction (AAR). According to the nature of the constituents of the aggregate, these reactions can be classified as: (i) alkali-silica reaction, (ii) alkali-silicate reaction, and (iii) alkali-carbonate reaction. In particular, the alkali-silica reaction (ASR) is responsible for the deterioration of concrete structures [1]. This phenomenon involves long, complex and costly repairs. Damage due to ASR in concrete is a phenomenon that was first recognized in 1940 by Stanton in North America [2]. However, ASR mechanisms are not yet enough understood. The influencing factors that favour ASR are: (i) use of reactive aggregates, (ii) strong alkalinity, (iii) water or high moisture levels. Other factors can play a significant role, such as the porosity of the concrete, external temperature and alkalis introduced by some mineral additives or admixtures or by the water [3].

Several studies have been published about the ASR, and it is widely accepted that ASR gel product is formed by the reactions between alkali cations (K^+, Na^+) and hydroxyl groups (OH^-) present in the pore solution and poorly crystallized siliceous minerals found in some aggregates that results in the expansion and cracking of the concrete [4]. The use of industrial wastes, such as fly ash or blast-furnace slag have been proved effective means of controlling the deleterious expansions due to ASR [5,6]. Researchers have proved the significant possibility of using biomass fly ash, another industrial waste material for cement replacement [7–9]. However, biomass fly ash is excluded from additions in concrete according to the current standards because of its non-coal origin. Recently, the new standard EN-450-1 included fly ash obtained from co-combustion of specific co-combustion biomass materials up to 20% by mass of the total fuel. Class C fly ashes with high CaO content are also reported to be useful in mitigating ASR and biomass fly ashes usually are high calcium fly ashes. [7,10].

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Table 1
Mortar formulations (W/B = 0.47, B/A = 0. 44).

Samples	Substitution (wt%)	Biomass fly ash (g)	Metakaolin (MK) (g)	Cement (g)	Super plasticizer (ml)	Consistency (mm)
Ref.	0	0	0	440	0.0	124
20 BFA_1	20	88	0	352	4.4	131
30 BFA_1	30	132	0	308	8.8	131
20 FA_1 + 10 MK	30	88	44	308	8.8	125
20 BFA_2	20	88	0	352	6.2	129
30 BFA_2	30	132	0	308	10.6	115
20 BFA_2 + 10 MK	30	88	44	308	17.6	137

The power production from the combustion of biomass or pulp and paper wastes generates millions of tons of fly ash and slag. The use of these by-products is essential, because they can cause serious environmental problems when disposed of in landfills or ponds. Currently, these fly ashes are partially recycled in the fertilization of agricultural fields and/or forestry [7].

Normally, the biomass ashes are characterized by the high content of calcium oxide, silica, alumina, and low amount of iron oxide [8,9], when compared with fly ashes from the coal combustion. Like the coal fly ashes, we anticipate some capacity of the biomass burning fly ashes to chemically react with calcium hydroxide, producing a more stable calcium silicate hydrate (CSH) gel [8,11–14]. In addition, the fineness of fly ash particles enhances the compactness of the mixtures (filler effect) and reduces the water permeability. When the diffusion of alkaline species, such as sodium and potassium, in the interstitial region of the cement paste becomes high some ASR inducing conditions can be affected [15,16]. Finally, the partial replacement of cement by the fly ash minimises the heating of the mixtures at the beginning of the hardening process, which also positively contributes to reduction in the ASR [17]. However, the high alkali content in biomass ash seem to be a big concern of ASR in concrete and detailed investigation is needed on the role of biomass fly ash in the ASR expansion of concretes.

2. Experimental procedure

2.1. Preparation and characterisation of fly ashes

The biomass fly ashes used in this work were collected from the electrostatic precipitator of two distinct industries: (i) thermal power plant (BFA_1); (ii) co-generation of pulp and paper production (BFA_2). Both plants use forest wastes as main fuel (mainly eucalyptus wastes, resulting from logging and wood processing activities).

The biomass fly ash BFA_1 was collected from a biomass thermal power plant dedicated to electricity production, which uses forest residues for energy production. The fuel is burnt in a boiler with a water cooled vibrating grate, and nominal thermal capacity of 30 MWth. Whenever needed natural gas is also used for maintaining proper temperatures in the boiler. Typical temperatures in the furnace are around 1000 °C. The biomass fly ash BFA_2 was collected from a biomass co-generation plant, from a pulp and paper industry. The fuel is burnt in a boiler with fluidized bed technology, and nominal thermal capacity of 90 MWth. Typical temperatures in the furnace is around 800 °C.

The biomass fly ashes were dried in a laboratory oven at 100 °C to remove excess moisture. The biomass fly ash BFA_1 was then grinded to destroy the coarse particles, while the biomass fly ash BFA_2 was sieved through at 75 μm mesh prior to incorporation in mortars. The materials were washed several times, to remove their soluble components, like alkalis, chlorides and sulphates, and then they were dried at 100 °C.

The particle size distribution of sieved biomass fly ashes, before and after washing, was determined by laser interference (Coulter LS 230). The specific surface area was evaluated by the BET (Brunauer, Emmett, and Teller) method, using N_2.

The chemical composition of the biomass fly ashes was studied using X-ray fluorescence spectroscopy (XRF, Panalytical Axios Dispersive Spectrometer). Crystalline phases were detected by X-ray diffraction (XRD, Rigaku Geigerflex D/max-Series, power 40 kV/30 mA, scan mode continuous/speed-3° 2θ min^{-1}). Gravimetric and differential thermal analyses (TG/DTA) of the biomass fly ashes were performed up to 1000 °C with a heating rate of 10 °C/min (STA 409 EP).The pozzolanicity of the ashes was determined using the modified Chapelle test [18]. In this test we placed 1.000 g of mineral admixture + 2.000 g of calcium oxide in a water volume of 250.0 ml. The solutions were kept for 16 h in an oven at 85 ± 5 °C. At the end of the period, the CaO content was determined for titration with hydrochloric acid (HCl) solution and using phenolphthalein as indicator. The results were expressed in terms of fixed CaO, which is equal to the difference between 1.000 g and the mass of CaO obtained from titration.

Table 2
Chemical composition (wt%) of the raw materials.

Composition	Biomass fly ash BFA_1	Biomass fly ash BFA_2	Metakaolin
SiO_2	52.1	25.1	54.7
Al_2O_3	13.3	11.3	38.0
Fe_2O_3	5.30	5.18	1.22
CaO	15.9	40.1	0.01
MgO	3.31	6.63	0.05
SO_3	0.45	1.12	0.02
K_2O	4.14	2.07	0.01
Cl^-	0.10	0.25	–
Loss on Ignition	10.4	3.50	0.94
Pozzolanicity (NF P 18-513)	618 mg/g	701 mg/g	1200 mg/g

2.2. Mortar formulations and characterisation

The cement mortar bars (275 mm × 25 mm × 25 mm) were prepared meeting ASTM C 1260/ASTM C 1567 specifications with a water/binder (W/B) ratio of 0.47 and binder/aggregate (B/A) ratio of 0.44 (weight ratios). A highly reactive quartzitic fine gravel of alluvial origin (0.15–4.75 mm) is used as the aggregate. Table 1 shows the prepared formulations. As reference, we used an ash-free mortar. Then, 20BF and 30BF mixtures were prepared by replacing 20 and 30 wt% of cement by each biomass fly ash. A blend of 20% of each biomass fly ash and 10% metakaolin was also tested. The metakaolin was obtained by the calcination of a Portuguese kaolin (Mibal) at 750 °C. The components were weighed and mixed thoroughly in a laboratory mixer. Flow table measurements were done to check the workability. According to ASTM C 1567 the spread diameter of the mortar composition should be ± 7.5% of reference mortars, i.e., around 115–133 mm. A super-plasticizer, Glenium 26 SCC was used to make the mortars in workable condition. The mortar formulations are shown in Table 1.

The mortars were prepared with gauge studs at the ends for the length change measurement. The mortar bars were cast at an aggregate to cement ratio of 2.25:1. The mortar bars were cured in the moist room at 23 ± 2 °C and Relative Humidity of (RH) >95% for the first 24 h, demoulded and then kept in a storage container with sufficient amount of water in it. After removing from the moulds the initial expansion readings for the mortars were taken using a digital length comparator accurate to 1 μm. After this, the mortars were preconditioned for 24 h in water maintained at 80 ± 2 °C. The length of the mortar bars was measured and the bars were immediately transferred to storage containers filled with 1 N NaOH solution maintained at 80 ± 2 °C. Length of the mortar bars were periodically measured over a 28 day period. The expansion value was calculated as the average percentage length change of 3 mortar bar samples based on length change since initial immersion in NaOH.

At the end of ASR tests, the microstructure of the samples was studied by scanning electron microscopy (SEM, Hitachi SU 70) and the flexural and compressive strengths were also determined (Shimadzu, model AG-25TA).

3. Results and discussion

3.1. Characterisation of biomass fly ashes

The biomass fly ashes BFA_1 were black in colour, indicating a significant amount of unburnt matter. BFA_2 ashes were medium

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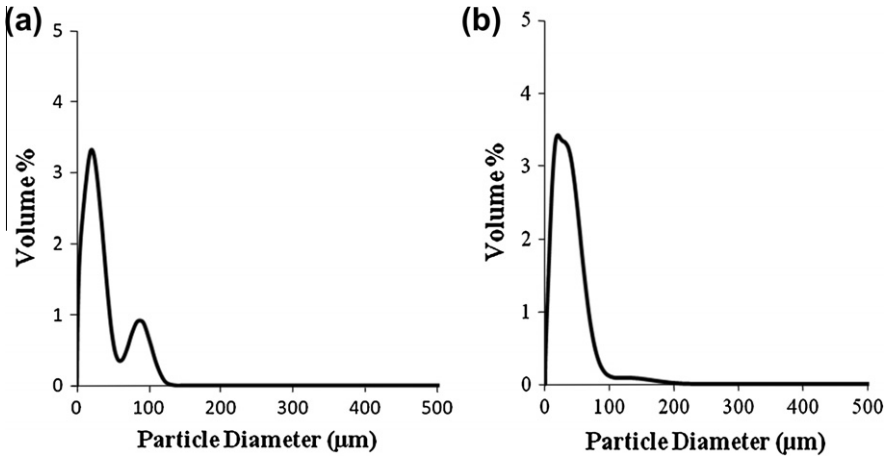

Fig. 1. Particle size distribution of fly ashes: (a) BFA_1 and (b) BFA_2.

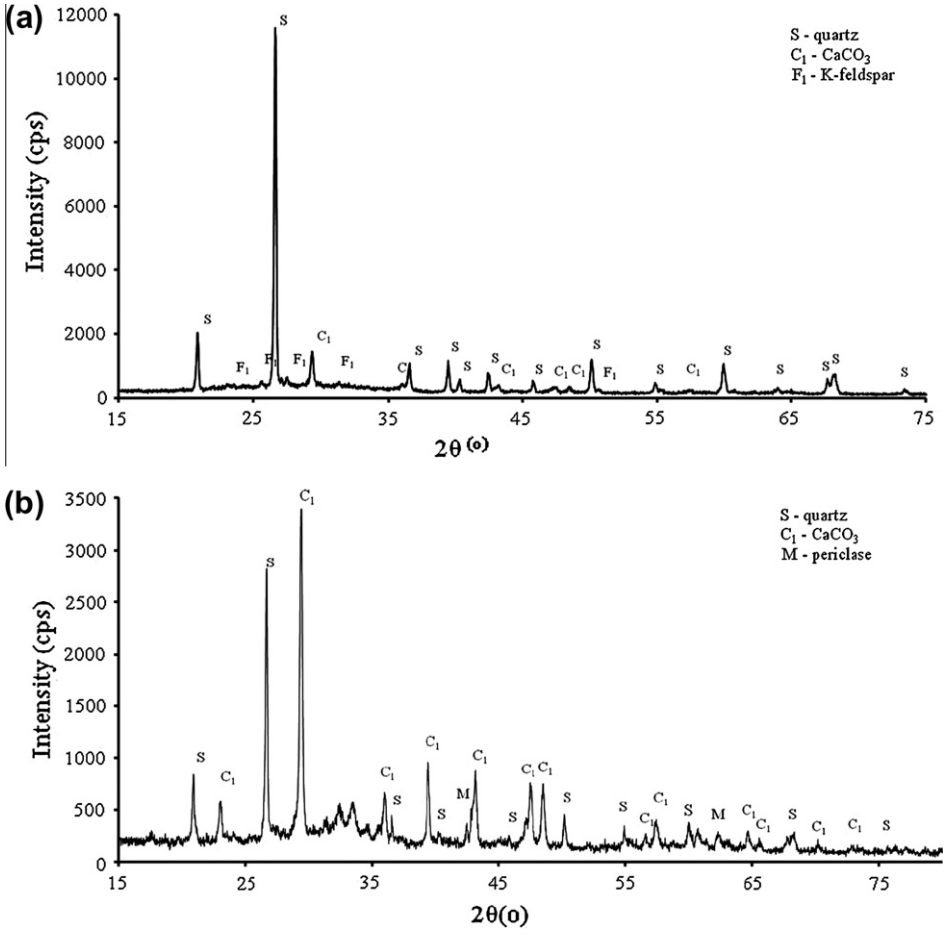

Fig. 2. XRD patterns of the biomass fly ashes: (a) BFA_1 and (b) BFA_2.

grey. This difference suggests that combustion process is less efficient in the thermal power station, despite the use of higher burning temperatures. This observation agrees with values of loss on ignition expressed on Table 2. Above a certain limit, the presence of unburnt material might hinder the correct hardening of the cement, so its amount should be minimised.

The particle size distribution of the unwashed ashes is shown in Fig. 1. Particles were typically finer (<100 μm). The average particle diameter of the fly ashes BFA_1 was 17 μm, slightly less than fly ashes BFA_2, 21 μm. Both showed the existence of agglomeration, most abundant in the case of BFA_1. This is noticed by the existence of a second peak, located at higher diameter values.

Despite the similarities in the size distribution, the values of BET specific surface of the ashes BFA_1 and BFA_2 are very different (28.52 m^2/g and 1,74 m^2/g, respectively). The higher value for biomass fly ash FA_1 can be explained by the higher amount of unburnt material, and also by the irregular particle shape [9].

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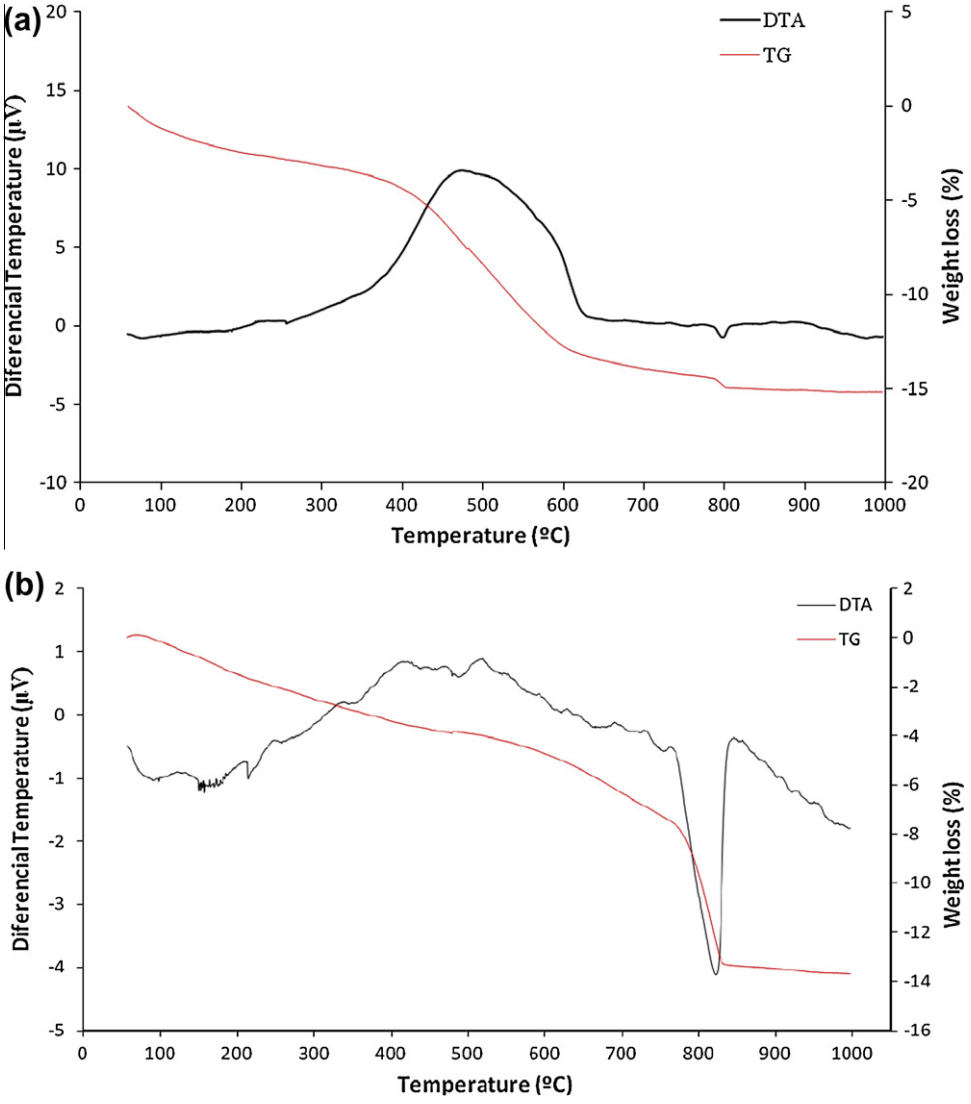

Fig. 3. TG/DTA curves of fly ashes: (a) BFA_1 and (b) BFA_2.

The XRD patterns of the biomass fly ashes are shown in Fig. 2. The main crystalline phases on BFA_1 are quartz (SiO_2), calcite ($CaCO_3$) and microcline ($KAlSi_3O_8$). BFA_2 shows quartz, calcite and periclase (MgO). Differences are in accordance with distinctions in the chemical composition, shown in Table 2. Both ashes contained more than 10% CaO, so they can be classed as type C when compared with those generated by coal combustion. In that sense, it would expect they react both pozzolanically and hydraulically. However, differences in the CaO and SiO_2 contents are relevant. BFA_2 was almost three times richer in lime than BFA_1, while the amount of silica is half compared to that of BFA_1.

TG/DTA curves are shown in Fig. 3. Weight losses in the range of 300–600 °C were caused by exothermic reactions, certainly due to the burning of organic matter confirming the lower efficiency of the combustion process in the thermal power plant from where the biomass fly ash BFA_1 was collected. At about 800 °C, the endothermic peak is due to the decomposition of $CaCO_3$, and now this reaction is more severe in the BFA_2 ash, as predicted by looking to the XRF analysis.

The modified Chapelle test showed a CaO fixation of 618 mg/g and 701 mg/g for the biomass fly ashes BFA_1 and BFA_2, respectively, confirming a considerable pozzolanic character.

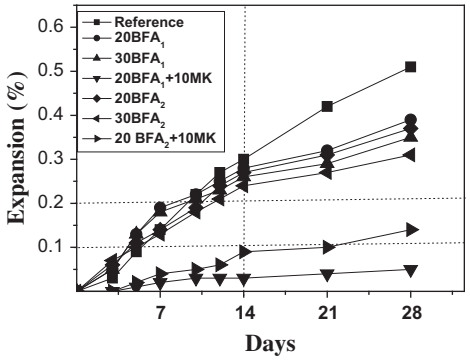

Fig. 4. Time evolution of the expansion of mortars under ASR accelerated curing.

3.2. Characterisation of mortars

Accelerated expansion results for the mixes are shown in Fig. 4. The 14 day and 28 days expansion values are also shown in Fig. 5. According to the ASTM C 1260 test-method, an aggregate is considered reactive if the average expansion of the three bars of mortars

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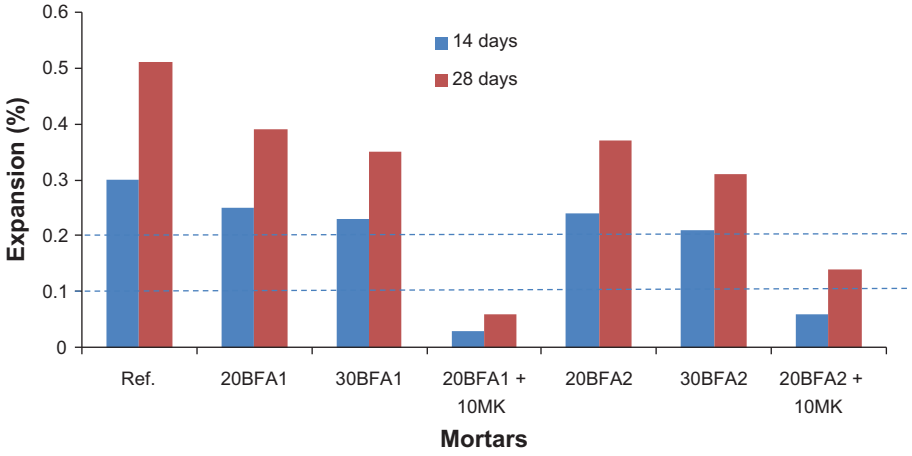

Fig. 5. Expansion values shown by the mortars at 14 and 28 days according to ASTM C 1260 method.

at the end of 14 days of immersion in NaOH is greater than 0.20% [19]. The expansion of the reference mortar prepared with the OPC CEM I type was 0.31% at 14 days and 0.51% at 28 days. This confirms the high reactivity of the aggregate used in these experiments.

The expansion results for mortar mixtures containing 20% and 30% biomass fly ash showed a distinct reduction in the expansion compared to the plain cement mortars. It was observed that the expansion rate of biomass fly ash mortars was high in the initial stages and after that it seemed to decrease. The less alkalinity is favouring the biomass fly ash BFA_1 substituted mortars compared to that of BFA_2. And for BFA_2 the slight increase in pozzolanicity compared to BFA_1 is resulting into reduction in the reaction rate However, the values are above the 0.10% at 14 days limit prescribed in the ASTM C 1567 [20]. But the incorporation of metakaolin in the blend with biomass fly ash did a significant improvement in the expansion results, indicating the effective use of biomass fly ash along with metakaolin in mitigating the ASR. The metakaolin was much effective in inhibiting the ASR even when used at lower rates of cement replacement than the biomass fly ash. The greater relative effectiveness of metakaolin when compared to the biomass fly ash is likely related to its smaller particle size and primarily alumina-siliceous chemical composition which results in its greater reactivity [21,22]. The Biomass fly ash mortars blended with metakaolin showed a mixed behaviour of both metakaolin and biomass fly ashes. The mortar 20 BFA_2 + 10 MK was less effective than mortar 20 BFA_1 + 10 MK in the suppression of the ASR expansion, which could be attributed to the large particle size and chemical composition of the ashes used though BFA_2 showed a comparable reduction in expansion in other compositions. (BFA_2– is poorer in silica and alumina, and richer in alkalis than BFA_1).

SEM/EDX of ASR gels formed in 20 BFA_1, 20 BFA_2, 20 BFA_1 + 10 MK and 20 BFA_2 + 10 MK mortars after 28 days of ASTM C 1260 tests are shown in Fig. 6. The cracks due to the ASR gel can be clearly viewed around the voids and aggregates in the SEM pictures. The ASR gels formed were not having any significant differences in appearance when comparing based on the difference in the chemical compositions of the biomass fly ashes. The gels were mostly of sodium silicate in composition. The similar pozzolanicity of both the fly ashes can be an indication for the similar expansion behaviour of the fly ashes even though they are having significantly different chemical compositions.

The Ca/Si ratio and Na_2Oeq was determined from the quantitative EDX analysis from a series of points in the outer CSH gel in the paste. The values are plotted in terms of expansion vs Ca/Si ratio, as in Fig. 7. It can be observed that the expansion values and Ca/Si ratios were proportional which confirmed the earlier reports on the ASR mitigation effectiveness of supplementary cementing materials that the CSH with a low Ca/Si ratio is able to retain more alkali (Na+K) compared to hydrates of higher lime to silica ratios [23]. There is no apparent correlation between expansion and Na_2O eq., as expected from the experimental protocol we followed, since along the test we add NaOH solution in order to keep the alkaline solution volume constant.

Table 3 shows the values of flexural (R_f) and compressive strengths (R_c), together with the density of hardened mortars cured in accelerated conditions, as prescribed by ASTM C 1260 standard. The differences in strength reflected mostly the variations in density (ρ), as suggested by the R_c/ρ ratio in the last column of Table 3. This suggests that the effect of physical parameters (microstructure and compactness) is conditioning the hardening process and concomitant development of properties. For example, 30 BFA_1 sample is the one showing maximum R_c and is the densest mortar. Density variations justify the strength differences between the samples, containing the ashes of different origin. Being thinner, the biomass fly ashes BFA_1 improve the compactness of the mixtures (stronger "filler" effect).

In general, there is a loss of strength with the increase of cement replacement (This effect was predicted since the mixtures contain lower amount of binder. Even so, R_c values of mortars containing 40 wt% substitution are acceptable, since they are over 40 MPa.

The values of mechanical strength are not related in an obvious way with the extension of the ASR. In fact, if the degradation was very serious, we should expect a visible lost of mechanical strength in the samples that suffered major expansion. So, we should for example expect that R_c of sample 20 BFA_1 + 10 MK is over the value of sample 30 BFA_1. This means that is probably too early (28 days) to notice the deleterious effect of ASR on the mechanical strength, even after curing the samples in conditions that accelerate the expansive phenomena.

4. Conclusions

The biomass fly ashes BFA_1 and BFA_2 were examined for their ability to resist damage by ASR using the Accelerated Mortar Bar Test (AMBT) method where a highly reactive fine aggregate was incorporated in the mortars. BFA_2 fly ash showed less amount of organic matter compared to BFA_1 fly ash. BFA_1 contains higher amount of SiO_2 and Al_2O_3 compared to BFA_2 fly ash, and this last one is much richer in CaO. Both materials are considered as type C, if assuming the classification of coal burning fly ash, meaning

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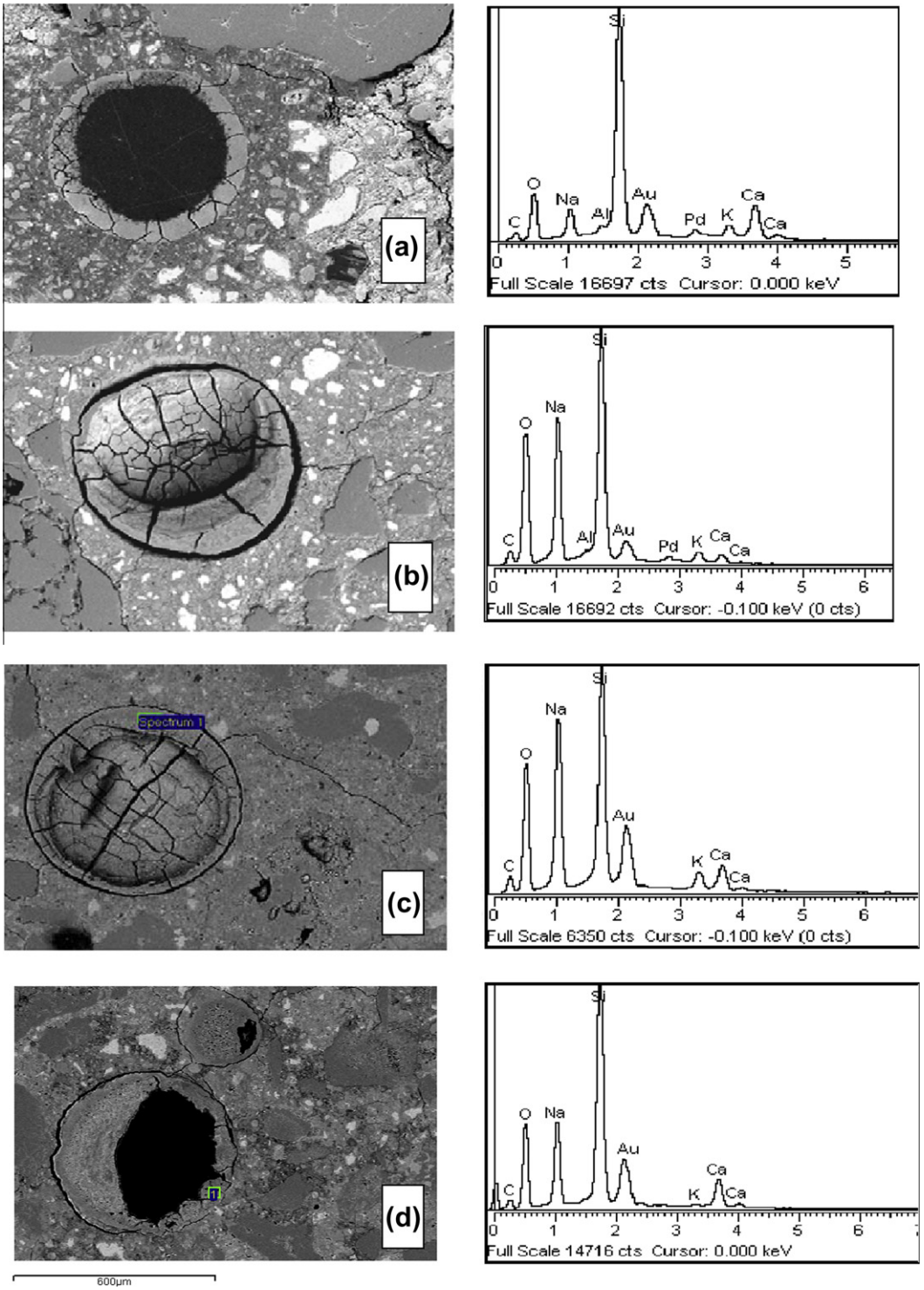

Fig. 6. SEM/EDX of ASR gels formed in (a) 20 BFA_1 + 10 MK, (b) 20 BFA_2 + 10 MK, (c) 20 BFA_1, and (d) 20 BFA_2 mortars after 28 days of ASTM C 1260 tests.

expectable hydraulic character in addition to the expected pozzolanic action. Before using in mortar formulations BFA_1 was milled and sieved (75 µm) while BFA_2 was just sieved (75 µm mesh). Consecutive washing steps in water were also applied to remove the soluble salts. Mortars containing 20 and 30 wt% of cement substitution by fly ash were prepared and cured in ASR accelerated conditions. From the data it was observed that the biomass fly ash incorporation is useful to resist the alkali silica reaction, in concretes. A considerable reduction in expansion was observed on the biomass fly ash incorporated mortars and concretes. The less alkalinity is favouring the biomass fly ash BFA_1 substituted mortars compared to that of BFA_2. And for BFA_2 the slight increase in pozzolanicity compared to BFA_1 is resulting into reduction in the reaction rate. The expansion decreased with the increasing content of cement replacement. However, the values are above the 0.10% at 14 days limit prescribed in the ASTM C 1567. But the use of metakaolin was significantly effective in limiting the AMBT expansion when used with biomass fly ashes (20% BFA + 10% MK). The relative effectiveness of metakaolin was primarily due to its finer particle size and the chemical composition of favouring a high

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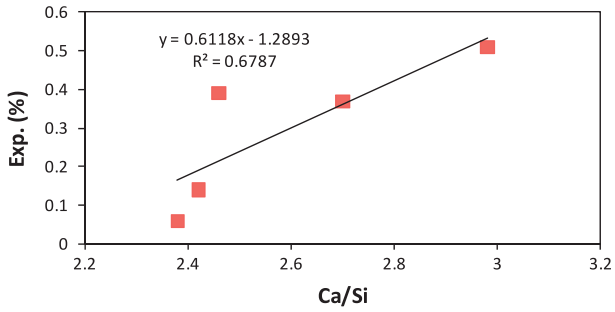

Fig. 7. Expansion vs Ca/Si ratio of the biomass fly ash blended cement mortars of AMBT test after 28 days of curing.

Table 3
Mechanical strength and density of mortars cured for 28 days under AMBT conditions.

Samples	Flexural strength (MPa)	Compressive strength (R_c) (MPa)	Density (ρ) (g/cm^3)	$R_c/(\rho)$ (MPa cm^2/g)
20 BFA_1	4.16	46.08	2.53	18.21
30 BFA_1	4.50	50.19	2.63	19.08
20 BFA_1 + 10 MK	7.28	41.81	2.30	18.17
20 BFA_2	3.63	42.45	2.25	18.86
30 BFA_2	3.57	32.20	2.05	15.70
20 BFA_2 + 10 MK	6.34	36.36	2.07	17.56

reactivity. These results indicate the scope of the effective use of biomass fly ashes along with a minimum amount of metakaolin to mitigate the ASR reactions in concretes.

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