

CODE 2.2

ACTIVITY OF FLY ASHES FROM WASTE INCINERATORS AND THE SUGAR INDUSTRY IN LIME MORTARS AND PASTES**Lima Figueiredo, Radson^{1*}; Pavía, Sara²**

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ABSTRACT

Fly ashes (FAs), generated as by-products, can be active in the presence of lime generating cementing hydrates through pozzolanic reaction. These can enhance the strength of hydrated-lime mortars still preserving their physical and chemical compatibility with traditional and historic masonry.

This paper assesses the properties and reactivity of FAs from different sources in order to evaluate their potential as pozzolanic additions in lime mortars.

The results evidenced that the incinerator and sugar bagasse ashes are reactive and combine substantial lime. All ashes significantly increased the flexural strength of hydrated lime alone and only one of the incinerator ashes lowered the compressive strength of the lime at 90 days. The significant strength increase is probably due to the generation of pozzolanic cements and to the increase in nucleation sites for C-S-H precipitation.

The finest (incinerator) ash (IFA2) which also has the greatest specific surface area and the highest calcium content, shows the greatest activity with the mechanical test however slightly disagreeing with the chemical test. The compressive strength reactivity rating follows the trend of the BET results where the most reactive ash is IFA 2, followed by IFA 3 and finally IFA 1.

The ashes consist of a mix of irregular porous and vitreous particles. The chemical composition and the presence of cubic crystals suggest the presence of halite (NaCl) in the incinerator ashes which can adversely impact material durability. The mineral composition evidenced the presence of silicates, oxides, carbonates and salt minerals.

1. INTRODUCTION

As a result of environmental pollution and climate change, there is a need to supply alternative materials that can lower the environmental impact of construction. The environmental impact of construction can be reduced by lowering the embodied energy of building materials. To this aim, traditional binders of high embodied energy and carbon emissions are often partially substituted with supplementary cementitious and/or pozzolanic materials. Many of these are agricultural and/or industrial by-products that were considered waste and dumped in landfills for decades. However, it has been long known that some of these materials have an amorphous siliceous /aluminous content that can react with lime in the presence of water producing cementing hydrates similar to those generated upon hydration of cement and hydraulic limes.

Some ashes display pozzolanic properties so that they create additional cementing minerals that accelerate setting and strength development of limes and cements. Hydrated-limes ($\text{Ca}(\text{OH})_2$ - European designation: CL - calcium lime) have been enhanced with pozzolans since antiquity. It is generally accepted that pozzolans enhance lime mortars while still preserving their physical and chemical compatibility with traditional and historic masonry. CL hardens slowly by carbonation, however, pozzolans impart a hydraulic set, accelerating hardening and increasing strength. The capacity of binding lime and the rate at which the binding takes place is referred to as pozzolanic activity and it is determined by the type and amount of reactive phases, specific surface area of the particle components and other factors.

The chemical and physical characteristics of fly ashes from power stations have enabled their use as partial cement replacement for decades. However, incinerator fly ashes (IFAs) from the sugar industry and urban waste incinerators have been less investigated. IFAs are mainly composed of SiO_2 , Al_2O_3 , Fe_2O_3 , and CaO with minor constituents such as MgO , Na_2O , K_2O , SO_3 , MnO and TiO_2 . According to their chemical composition, fly ashes can be classified as silico-aluminous (having pozzolanic properties) or silico-calcareous, with hydraulic properties [1, 2]. Mostly composed of spherical particles, fly ashes also contain irregularly shaped particles of varying sizes depending on the sources [2, 3]. The spherical shape of IFAs lessens the frictional forces of larger angular particles by filling in the spaces, enhancing packing and reducing the yield stress (“ball bearing” effect) [4].

Several studies have explored the adsorbent properties sugarcane fly ash on the removal and recovery of heavy metals and other hazard chemicals from aqueous solutions [5-8]. The efficacy of bagasse fly ash as a supplementary cementitious material has been investigated to a lesser extent [9]. Arenas-Piedrahita et al. (2016) [9] studied the effects of sugarcane fly ash as a partial cement replacement (10% and 20%) and reported that, there was a decrease on the compressive strength of the mortars at 28 days, especially for mortars with 20% replacement, however, at 56 days, the compressive strength for both levels of replacement reached equal strength than the control.

Researchers have studied the use of fly ash in building materials [4, 10-13]. Felekoğlu et al. (2009) [10] stressed the importance of fineness to achieve a higher compressive strength, and noted that increasing fineness may also result in a higher water demand due to increasing surface area. Payá et al. (1995) [11] argue that the enhancement of compressive strength is mainly related with the content of fly ash particles under $10\ \mu\text{m}$. They also noted that a significant loss of flexural and compressive strengths is associated with the use of coarser fly ash fractions. Siddique (2004) [13] found that using fly ash as a partial Portland cement replacement reduced strength at the age of 28 days, but there was a continuous and significant improvement of strength beyond 28 days. Garcia-Lodeiro et al. (2016) [14] argue that matrices incorporating fly ashes lowered the leaching of metals, resulting in a physical and chemical barrier. Jiménez-Quero et al. (2013) [4] explored the rheological behaviour of cement pastes and mortars with sugarcane IFA replacement, finding that pastes with 20% and 30% fly ash showed a smaller yield stress than a cement paste with superplasticizer (12 mL/kg of cement). They also noted that the use of superplasticizer on mortars with fly ash is not necessary, as there was a decrease of shear viscosity with the increase of shear rate.

Recycling FAs into building materials has environment and economic benefits as it lowers production energy and non-renewable raw materials and reduces carbon dioxide emissions and dumping in landfills. Furthermore, the application of FAs has been reported to enhance the workability of pastes reducing water demand and heat evolution, therefore minimising expansion and cracking at early ages [3]. This paper studies the properties and reactivity of three FAs from waste incineration and sugar industries in Portugal, the Czech Republic and Brazil. The study intends to evaluate their potential as pozzolanic additions in an effort to produce sustainable masonry mortars that reuse waste and have low embodied energy, lower raw material consumption and lower CO_2 emissions than traditional lime mortars.

2. METHODOLOGY

A hydrated lime (CL90-S) complying with EN 459-1 [15] and a siliceous sand with grading and composition complying to the European CEN standard sand were used. The ashes were sourced from a waste incinerator plants in Portugal (IFA 1) and Czech Republic (IFA 2), and from a sugar cane industry in Brazil (IFA 3). A particle size reduction of dried ashes was carried out on IFA 3 using a TEMA T100 Disc Mill (1000 RpM; max sample 150 g). Homogenisation was ensured by subsampling the studied materials with a Rotary Sample Divider laborette 27 to guarantee representativity when small quantities were required for analysis.

2.1. Grading, specific surface area (BET) and microstructure of the ashes

The particle size distribution was measured by laser diffraction using a Mastersizer 2000, composed of the Mastersizer 2000 unit; the Hydro 2000G wet dispersion and the Autosampler 2000 units. This method measures the angular distribution and intensity of the light by particles in suspension and utilises the Mie theory of diffraction in the prediction of laser particle size results.

The specific surface area of the samples was determined with a Quantachrome Nova 4200e and the BET method, a model isotherm based on adsorption of nitrogen gas on a surface. The mineralogical composition and amorphous character of the pozzolans were analysed by X-Ray Diffraction (XRD), using a Phillips PW1720 XRD with a PW1050/80 goniometer and a PW3313/20 Cu k-alpha anode tube at 40kV and 20mA. All measurements were taken from 3 to 60 degrees (2θ) at a step size of 0.02 degrees/second. The microstructure of samples was observed by scanning electron microscope (SEM), Tescan Mira XMU equipment, revealing the surface topography, morphology and pore system. Backscattered electron (BE) signal detector was preferred for observation and image acquisition.

2.2. Chemical composition

The analysis of major oxides was performed by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) analysis. A prepared sample (0.200 g) is added to lithium metaborate/lithium tetraborate flux (0.90 g), mixed well and fused in a furnace at 1000°C. The resulting melt is then cooled and dissolved in 100 mL of 4% nitric acid/2% hydrochloric acid. This solution is then analysed by ICP-AES and the results are corrected for spectral inter-element interferences. Oxide concentration is calculated from the determined elemental concentration, and the result is reported in that format. The trace elements were analysed by ICP-AES with the samples prepared by a four-acid digestion.

Loss on ignition (LOI) was used to determine the unburnt carbon content by thermal decomposition furnace, where a prepared sample (1.0 g) is placed in an oven at 1000°C for one hour, cooled and then weighed. The percent loss on ignition is calculated from the difference in weight.

2.3. Reactivity of the ash by electrical conductivity

The conductivity variation of a lime-ash suspension over time was measured to assess the ability of the ash to combine lime - $\text{Ca}(\text{OH})_2$ – and thus its reactivity. A sample (0.8 g) of ash was added to a saturated hydrated-lime solution (0.2 g of lime in 100 mL of water). The solutions were kept in sealed flasks to avoid water evaporation and carbonation. A thermostatic bath was used to maintain the flasks at constant temperature (20°C). The loss in conductivity was then measured at intervals over 168 hours using a WTW Conductivity Meter ProfiLine Cond 197i.

2.4. Reactivity of the ash by strength development

The reaction between pozzolanic material and $\text{Ca}(\text{OH})_2$ forms strength-developing calcium silicates and aluminates. To assess strength development, the compressive and flexural strengths were measured using 6 prisms (figure 1), sized 40x40x160 mm, produced with a ratio by mass (lime: ash: aggregate: water) of 1:1:3:1.5. A reference specimen was also produced at 2:3:2 (lime: sand: water). They were demolded after three days and cured for 90 days at 20 °C and 60% RH. The strengths were assessed

following EN 459-2 [16] and EN 196-1 [17] using a Zwick loading machine. The water content was calculated with the function established by Walker and Pavía [18] which sets a relationship between specific surface area and water demand, based on experiments with 9 pozzolans.



Figure 1. The ashes slightly alter the lime mortar colour (prisms used for flexural strength testing).

3. RESULTS AND DISCUSSION

3.1. Physical properties, chemical composition, mineralogy and microstructure of the ashes

One of the main variables that determine the suitability of a fly ash for use as cement replacement is fineness as it influences the rate of reaction, affecting the short-term activity of the pozzolanic reaction [19] i.e. the early production of cements. Therefore, the particle size distribution of the materials was analysed. Figure 2 shows the particle size distribution of the IFAs compared with the lime. The grading reveals that the lime presents a narrower distribution (1.445 μm – 138.038 μm) than the fly ashes, with a reasonably wider range of particle sizes - from 0.316 μm to 954.99 μm . However, most particles in the ashes (> 80%) are sized similarly to the lime.

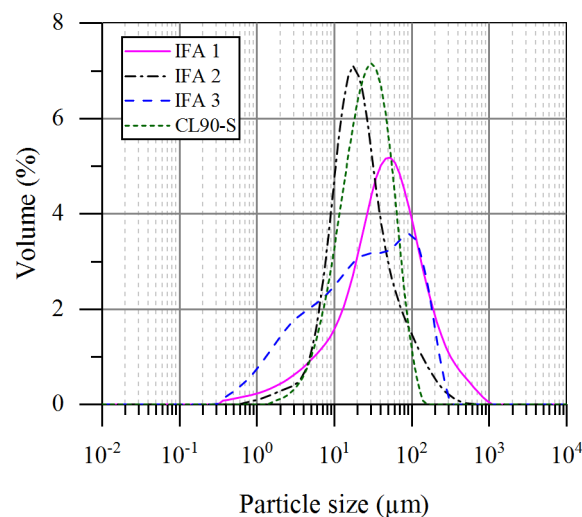


Figure 2: Particle size distribution of the ashes.

The gas adsorption measurements indicate that the specific surface areas (BET) of the ashes range between 4.50 and 5.27 m^2/g (Table 1). The values are greater than those typical of other supplementary cementitious materials such as GGBS and PFA however lower than RHA or MS [18]. Table 1 also includes the undersize particle distribution of the ashes: D10 (10%-cumulative), D50 (median diameter) and D90 (90%-cumulative). Considering the median diameter (D50- 50% of particles under this size), IFA 2 contains the greatest amount of fines as 50% of particles are under 18.69 μm , followed by IFA 3 (22.88 μm), both ashes being finer than the lime (24.13 μm).

Table 1: Cumulative particle sizes and BET.

	D10	D50	D90	BET (m ² /g)
IFA 1	6.74	42.19	167.70	4.50
IFA 2	7.04	18.69	68.98	5.27
IFA 3	2.16	22.88	120.53	5.25
CL90-S	8.15	24.13	57.98	-

Unlike the fly ashes from fossil fuel combustion which are typically spherical, the incinerator and sugar ashes are a mix of irregular and vitreous particles. Spherical hollow particles seldom appear and most particles are very porous. The Scanning Electron Microscope (SEM) showed that incinerator ashes IFA 1 and 2 are a heterogeneous mixture of irregularly shaped particles in clusters, cubes and needles. The qualitative composition and morphology of the cubic crystals suggest the presence of halite (NaCl)- IFA 1- Figure 3. Agglomerates around larger particles building up porosity (fig. 3) were also recorded, in agreement with previous studies [3, 10, 20-22]. IFA 3 presents less agglomerates when compared with the other ashes.

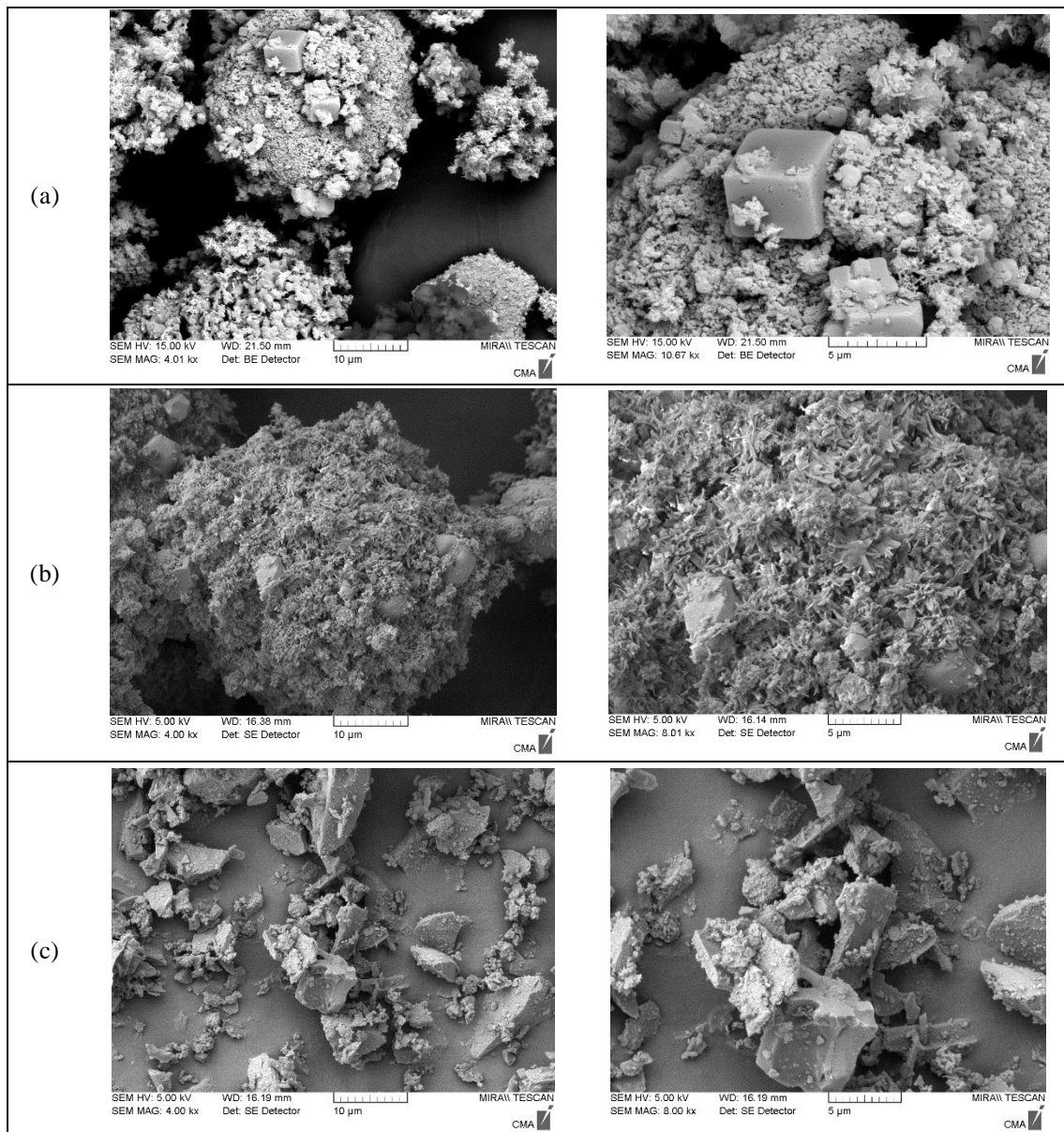


Figure 3: SEM photographs of (a) IFA 1, (b) IFA 2 and (c) IFA 3.

The X-Ray Diffraction analysis (XRD), figure 4, evidenced silicates, oxides, carbonates and salt minerals in the fly ashes. The fly ashes produced by waste incinerator plants (IFA 1 and IFA 2) present similar mineral composition with significant calcium carbonate (calcite). All ashes include ferric iron oxide (hematite). The incinerator ashes contain sodium chloride (halite)- detected by SEM in IFA1 (figure 3) and XRD in IFA2. All ashes contain silica in crystalline form.

There is a difference in the carbonate minerals present in the ashes. The incinerator ashes contain calcite whereas the sugar cane ash contains aragonite (CaCO₃). These are polymorphs of calcium carbonate but calcite is more stable than aragonite at ambient temperatures and pressures [23]. The formation of the polymorphs of CaCO₃ depend on the kinetics of metastable forms, rather than the equilibrium conditions. Also, the formation of aragonite is favoured by the incorporation of larger-sized cations, Sr²⁺, Ba²⁺ or Pb²⁺, into the CaCO₃ lattice [24].

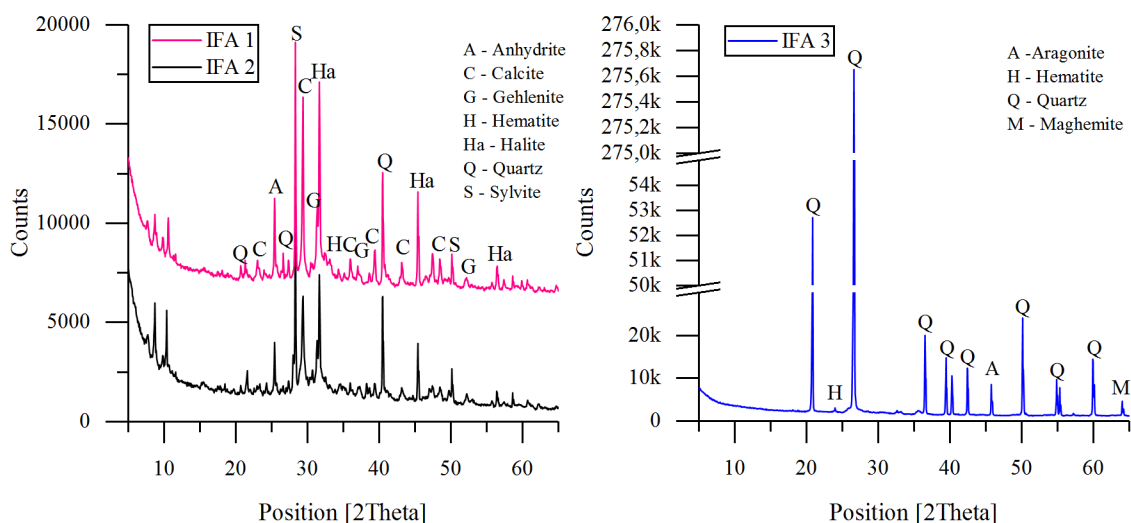


Figure 4: XRD diffractograms of IFAs.

Other minerals such as gehlenite -Ca₂Al[AlSiO₇]- and halite -NaCl- are present in the incinerator ashes IFA 1 and IFA 2. The sugar cane ash IFA 3 is richer in Fe, including significant hematite and maghemite (γ-Fe₂O₃) which is produced synthetically by oxidizing magnetite at a low temperature. Table 2 presents the mineralogical composition of the ashes.

Table 2: Mineralogical composition by XRD.

	Mineralogical composition
IFA 1	Calcite, quartz, hematite, halite, anhydrite, sylvite, gehlenite
IFA 2	Calcite, quartz, hematite, halite, anhydrite, sylvite, gehlenite
IFA 3	Quartz, maghemite, hematite, aragonite

Table 3 shows the chemical composition of the studied ashes. There are major differences in the Si and Ca content between the incinerator and the sugar ashes. The incinerator ashes (IFA 1 and IFA 2) are rich in Ca. This is attributed to the practice of adding carbon followed by Ca(OH)₂.2H₂O into the hot flue for the flue gas neutralisation. In contrast, the sugar fly ash- IFA 3, contains little Ca and high Si. The high Si content can be partially due to contamination. sugar industries have a high level of sand contamination in ashes due to sand adhered during harvesting that remains attached to the cane despite washing [25, 26]. The chemical composition clearly shows that IFA 1 and IFA 2 are fundamentally different from IFA 3, which allows one to classify the former two as silico-calcareous or high-calcium fly ash, and the latter as silico-aluminous or low calcium fly ash.

The loss on ignition (LOI) is substantially different in the ashes. Brazilian standard NBR 12653 [27] classifies pozzolanic materials as Class N (natural pozzolans), Class C (fly ashes from coal burning) and Class E (others). The standard limits for the LOI is 6% for classes C and E. IFA 1 and IFA 2 don't comply with the standard as they have LOI over 15%, while IFA 3 is within the limits, with a loss of 1.33%. Also, the standard's chemical requirement demands a sum of major oxides ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) of at least 50% for Class E and at least 70% for Class C pozzolanic materials. Given the results of the chemical composition of the ashes, shown in Table 3, it is observed that IFA 1 and IFA 2 do not comply with this standard requirement either.

Attention needs to be drawn to the amount of chloride, as chlorides are detrimental for the materials' durability and can cause fracturing by expansion, leaching of portlandite and corrosion of embedded metal reinforcement consequently reducing strength [18, 28]. However, the strength of lime-pozzolan mixes are improved by adding KOH, NaOH, Na_2SO_4 or CaCl [18].

Table 3: Chemical composition (%)

	IFA 1	IFA 2	IFA 3
SiO_2	12.6	7.64	89.9
Al_2O_3	6.21	3.71	2.34
Fe_2O_3	1.19	1.15	3.46
CaO	38.3	43.7	0.42
MgO	2.59	1.3	0.25
K_2O	4.5	4.84	0.52
TiO_2	1.37	0.69	1.41
P_2O_5	2.26	0.84	0.17
SO_3	2.94	2.26	0.027
Na	1.44	0.76	-
Cl	6.92	7.24	0.013
Mg	0.241	0.072	0.094
LOI	15.9	16.65	1.33

3.2. REACTIVITY

The ashes were subjected to chemical and physical tests to evaluate reactivity. The chemical test performed on lime-ash solutions at 20°C demonstrated that all ashes combined lime causing a reduction in electrical conductivity. Table 4 presents the summary of the conductivity variation of lime/ash solutions for a period of 7 days. It is observed that after 168 hours IFA 3 achieves the highest percentage loss in conductivity, followed by IFA 1 and IFA 2.

Tashima et al. (2014) [29] assessed the pozzolanic reactivity of amorphous and crystalline rice husk ashes (RHAs) at different temperatures (40, 50 and 60 °C). At 40 °C the crystalline rice husk ash, with only 10.1% amorphous phase, presented no significant change on the conductivity, while the amorphous RHA yielded about 90% loss in conductivity for the same proportions used in this study (2:8 pozzolan:lime).

It should be noted that variation in temperature lead to considerable variations in conductivity. For instance, variation as low as 0.3 °C causes variations of about 0.5 mS/cm for NaOH [30]. As the results show, the losses in conductivity for the fly ashes (up to 21.84%) confirms the sensibility of the method for temperatures lower than 40 °C, and also confirms reactivity of the ashes.

Table 4: Summary of the conductivity variation of lime/ash solutions.

	Initial conductivity (mS/cm)	Conductivity variation (mS/cm)	Loss in conductivity (%)
IFA 1	15.53	2.61	16.78
IFA 2	17.08	2.43	14.21
IFA 3	9.8	2.14	21.84

To further measure the reactivity of the ashes, the strength development of lime:IFA mixes in relation to a standard lime mix over 90 days was monitored. The strength values for compressive strength reached up to 2.79 N/mm² for IFA 2, surpassing the control mix by 14 %. IFA 3 reached a considerable strength, achieving 92% of the strength of the control. IFA 1 presented the lowest value, achieving 55% of the strength of the reference mix.

All the ashes enhanced the flexural strength of the paste, confirming the pozzolanic character of the ashes. IFA 2 nearly doubled the strength of the control mix while IFA 1 enhanced the strength by 43% and IFA 3 by 33%.

Table 5: Mechanical activity index.

	Compressive strength (MPa)	Flexural strength (MPa)	Activity index (%)	
			Compressive strength	Flexural strength
Control mix	2.45	0.39	100	100
IFA 1	1.36	0.56	55	143
IFA 2	2.79	0.74	114	190
IFA 3	2.26	0.52	92	133

3.3. DISCUSSION

Previous literature has reported that high-calcium fly ashes are finer than low-calcium fly ashes [3, 31]. This is true for the ashes in this study however, the IFA 3 was grinded to achieve a fineness comparable to IFA 1 and IFA 2 which were used as supplied.

The ground IFA 3 has a specific surface area higher than IFA 1, which agrees with the particle size distribution (Figure), as the specific surface area depends on the proportion of fine particles present in the materials [28]. IFA 2 is the finest and has the greatest specific surface area (5.27 m²/g) followed by IFA 3 – 5.25 m²/g – and finally IFA 1.

Specific surface area determines reactivity as a greater surface increases the dissolution rate and solubility of the ash, thus favouring lime combination [19, 25]. The results agree with this, as the strength results follow the reactivity results set by BET, agreeing with what is reported in the literature [10, 18, 19]. This relationship is further supported by a regression fit showing the correlation between compressive strength and specific surface area (Figure 5). The slope is significantly different from zero at the 0.05 level indicating that the results are statistically significant to reject the null hypothesis (H0) so that the hypothesis that compressive strength increases proportionally to the surface area may be supported. Analysis of Variance (ANOVA) was undertaken to determine the results significance.

As aforementioned, the compressive strength follows the trend of the BET results where the most reactive ash is IFA 2, followed by IFA 3 and finally IFA 1. However, the conductivity results, despite being very similar, slightly disagree rating the reactivity of the ashes in decreasing order as: IFA 3, IFA 1 and IFA 2. This is probably due to the low calcium content of the IFA 3 ash.

The significant strength increase by the high-calcium fly ash IFA 2 is probably due to a greater generation of pozzolanic cements and to the increase in nucleation sites for C-S-H precipitation (it is well known that during hydration, pozzolanic particles offer nucleation sites for the precipitation of C-S-H [19]). High-calcium fly ashes lead to a more rapid strength gain as the large amounts of CaO, when released in solution, can react with the glass phase rendering these fly ashes as self-pozzolanic [32]. However IFA 3, with much lower Ca than IFA 1 (0.42 % vs 38.3 %), has significantly greater compressive strength and comparable flexural strength to IFA 3. This may be due to the alumina content, which causes evolution and entrapment of hydrogen gases reducing strength and increasing permeability [33]. A negative correlation was found between

the strength development and the alumina content of the samples. At a confidence level of 95% a Pearson correlation of -0.718 (p-value = 0.029) was established within the studied samples.

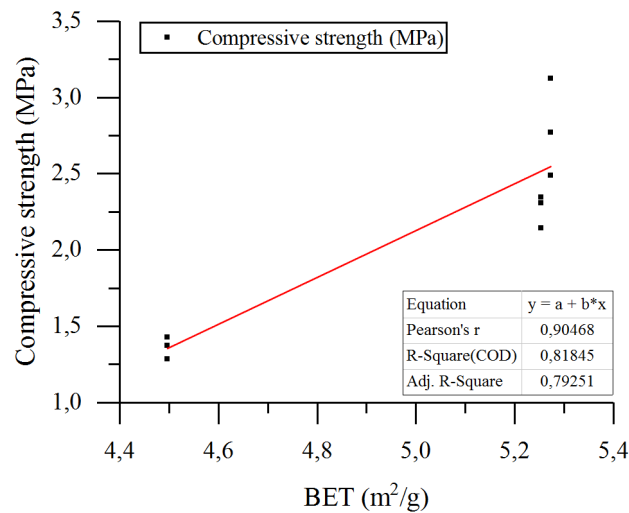


Figure 5: Correlation between compressive strength and specific surface area by BET.

4. CONCLUSIONS

The results evidenced that the incinerator and sugar bagasse ashes studied are reactive and combine lime. All ashes significantly increased the flexural strength of hydrated lime alone and only one of the incinerator ashes lowered the compressive strength of the lime at 90 days.

The tests place the ashes in order of reactivity as follows:

- Conductivity (% loss): similar reactivity, in slightly decreasing order: IFA 3, IFA 1 and IFA 2;
- BET and Compressive strength: IFA 2, IFA 3 and IFA 1;
- Flexural strength: IFA 2, IFA 1 and IFA 3.

The mechanical test (compressive strength) places the ashes in the same order of reactivity as the BET. The compressive strength development shows a positive linear relationship with the specific surface area of the ashes.

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