



Acid neutralisation capacity and hydration behaviour of incineration bottom ash–Portland cement mixtures

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Received 10 January 2001; accepted 7 December 2001

Abstract

The present paper deals with the use of incineration bottom ash in cementitious systems. The results of the physical–mechanical characterisation of the solidified products were discussed elsewhere, while the present work focuses on the acid neutralisation capacity (ANC) of the investigated mixtures as a means to evaluate the type of hydration products and their relative amounts in the solid matrix. The approach consists of differential acid neutralisation analysis, which was validated using traditional X-ray diffraction (XRD) methods. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Acid neutralisation capacity; Hydration products; X-ray diffraction; Waste management

1. Introduction

Understanding the type and composition of hydration products in cementitious systems containing waste materials is important, since variations occurring at the microstructural level can account for changes in physical, mechanical, and chemical properties of the solidified materials. In fact, the extent of such changes conditions the possibility of reusing waste materials in cement-based systems.

Acid neutralisation capacity (ANC) is a key property of cement/waste systems, resistance to acid attack affecting matrix durability and thus leaching of contaminants, both because their solubility is strongly affected by the pH conditions and because the integrity of the solid matrix depends on its ability to maintain alkaline conditions [1].

The ANC test [2] is a leaching procedure that is able to provide information on the buffering capacity of a given material, its phase composition, as well as its leaching behaviour under different environmental conditions. In particular, the ANC test allows the resistance to acidification of the material to be investigated and the presence of solid

phases that are stable to be determined and pH ranges to be identified. Such information is of particular interest for solidified products when evaluating either the durability of the matrix or its resistance to chemical attack and therefore its ability towards contaminants’ immobilisation.

If the curve showing the pH of the final eluate is plotted as a function of the amount of acid added, then it can be regarded as a titration curve. If the slope of the curve decreases at some point (i.e., a pH plateau occurs), this can be ascribed to the presence of particular solid phases able to resist a decrease in pH [3,4]. If plotted versus the amount of acid added, the inverse of the curve slope will thus display a spectrum of peaks, each corresponding to a pH plateau [3,5].

The pH value at which a plateau occurs depends on the nature of the solid phase controlling the pH itself, not on its quantity [6], the latter being inferred from the amount of acid required for the corresponding pH variation to be induced. This can be evaluated from the spectrum of peaks by measuring the amount of acid added between the minima at either side of each pH plateau [7].

For the pH plateaux distinctive of the individual solid phases to be identified, the assumption is generally made that the pore solution of hydrated cementitious mixtures is in a steady-state condition close to thermodynamic equilib-

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rium [6], so that the solid phases are in chemical equilibrium with the pore solution. The values of the individual pH plateaux associated with the hydration phases of a cementitious mixture therefore depend on their solubility, and have been identified by different investigators on both pure compounds [4,8–11] and pure systems typical of cementitious materials ($\text{CaO-SiO}_2\text{-H}_2\text{O}$ [12], $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2\text{-H}_2\text{O}$ [13] and $\text{CaO-Al}_2\text{O}_3\text{-CaSO}_4\text{-H}_2\text{O}$ [14–16] systems). On the contrary, very few data have been gathered on the pH stability values of hydration products as found in real hydrated cement paste or concrete. It is emphasised that when dealing with real mixtures, particular care must be taken in understanding how the pH plateaux associated with the different solid phases are modified by the binder composition and presence of impurities.

In the $\text{CaO-SiO}_2\text{-H}_2\text{O}$ system, the high solubility of portlandite conditions a pH ranging about 12.5, in the presence of either portlandite alone [4] or coexistent with Ca-rich ($\text{Ca/Si}=1.8$) C-S-H [12]. Equilibrium pH varies depending on the Ca content of C-S-H. Accordingly, the pH stability value decreases to 12.43 in the presence of high-Ca ($\text{Ca/Si}=1.8$) C-S-H coexistent with intermediate-Ca ($\text{Ca/Si}=1.1$) C-S-H, down to 10.88 for low-Ca ($\text{Ca/Si}=0.8$) C-S-H [12]. Low-Ca C-S-H can be formed as a result of addition of contaminants to cement or partial replacement with other binders, reducing the Ca/Si ratio; this will also decrease as hydration reactions proceed [17].

Different pH stability values have been proposed for portlandite and C-S-H by other authors [13], due to different assumptions on C-S-H composition and the presence of crystalline silicate hydrate forms.

An increase in pH stability values can be ascribed to the presence of alkalis in the unhydrated material [6,12]. Since the anions commonly associated with cementitious materials, like sulphates, silicates and aluminates, are generally sparingly soluble, the charge balance within the pore solution is due to OH^- ions, which are responsible for increasing pH, typically up to 13 to 14 [6]. However, alkalis are not able to exert a buffering capacity, therefore a resistance to acidification is not related to their presence.

The stability values for ettringite are not as easily determined as it is for the other hydrated phases. Nevertheless, some investigators revealed the presence of crystalline ettringite at pH values from 11.5 to 11.8 and a noncrystalline phase similar in composition to ettringite for pH values from 12.5 to 12.8 [1,8,10]. Although the additional stability range of 9.8 to 11.2 has been proposed, recent findings seem to suggest that ettringite may not be stable at pH lower than 11 [1].

The aim of the present research was to validate the results from the ANC test through differential acid neutralisation analysis. In particular, the study was designed to identify the main pH plateaux and correlate them to the hydration products that were likely to be present in the solidified mixtures.

The approach was aimed at assessing the ability of differential acid neutralisation analysis to describe the hydration behaviour of cementitious materials—even in the presence of impurities introduced, such as waste materials—, thus providing an alternative and/or complementary tool to traditional analytical methods (X-ray diffraction [XRD], quantitative XRD [QXRD], scanning electron microscopy [SEM] ...). The work was also focused on evidencing the limitations associated with such an approach.

2. Materials and methods

Three different Municipal Solid Waste Incineration bottom ashes (MBA) and one Hospital Waste Incineration bottom ash (HBA) were used in this investigation. The wastes were collected at different full-scale Italian incineration plants.

The bottom ashes were characterised for their physical properties, including bulk density, water content, loss on ignition (LOI), and chemical properties, including oxide phase and elemental compositions and anion content. Measurements were taken in triplicate and followed ASTM C29 for bulk density, ASTM D2216 for water content, and ASTM C25 for LOI. The elemental composition was determined by an acetylene flame-operated atomic absorption spectrometer (Perkin Elmer mod. 3030 B) after sample digestion according to the APHA 3030H procedure. Anion measurements were taken using ion chromatography (Metrohm mod. 761 Compact IC) after solubilisation according to the Italian UNI 8520 method. The oxide composition was determined from total X-ray fluorescence.

Table 1 shows the physical properties of the bottom ashes and their phase oxide composition compared with that of ordinary Portland cement (OPC), while in Table 2 the elemental and anionic concentrations in BA are presented. The pozzolanic activity of the bottom ashes was also

Table 1
Physical properties and phase oxide composition of BA and OPC

Physical property	HBA	MBABO	MBAMO	MBARE	OPC
Bulk density (kg/m^3)	1511	1425	1138	1365	
Specific gravity	2.56	2.44	2.43	2.46	
Water content (%)	13.65	24.71	21.68	39.28	
Loss of ignition (%)	5.55	2.94	3.47	8.47	
Oxide composition	HBA	MBABO	MBAMO	MBARE	OPC
SiO_2 (% dry wt.)	59.62	56.99	47.76	41.13	25.94
TiO_2 (% dry wt.)	1.09	0.49	0.79	1.23	0.33
Al_2O_3 (% dry wt.)	10.68	9.20	10.55	11.35	5.01
Fe_2O_3 (% dry wt.)	1.95	3.97	8.61	6.77	4.85
MnO (% dry wt.)	0.07	0.08	0.13	0.11	0.07
MgO (% dry wt.)	2.74	3.46	3.67	3.85	2.27
CaO (% dry wt.)	12.22	13.22	16.45	19.77	52.23
Na_2O (% dry wt.)	7.04	5.87	3.51	2.84	0.32
K_2O (% dry wt.)	0.58	1.35	1.41	1.57	1.98
P_2O_5 (% dry wt.)	0.36	0.70	1.29	1.84	0.12

Table 2
Elemental composition of BA

Element	HBA concentration (mg/kg dry wt.)	MBABO concentration (mg/kg dry wt.)	MBAMO concentration (mg/kg dry wt.)	MBARE concentration (mg/kg dry wt.)
Al	8393	55,320	30,280	27,920
Ca	23,000	133,300	103,400	246,400
Cd	<8	<8	<8	<8
Cr	168	79	182	80
Cu	1084	2660	2231	1526
Fe	4659	11,240	33,810	28,750
K	3264	3073	4221	3788
Mg	3177	12,540	8798	31,020
Mn	72	475	608	455
Ni	332	61	151	83
Pb	<80	1287	1197	1473
Zn	201	1443	1709	21,340
Chloride (%)	<0.01	0.12	0.24	0.35
Sulphate (%)	0.09	0.76	0.65	0.77

evaluated from the standard Italian procedure UNI-EN 196 parte 5^a.

Prior to mixing with cement, the bottom ashes were ball-milled to less than 150 μm to (1) reduce inhomogeneity of the material (that is typically characterised by a wide particle size range) and (2) increase the specific surface area, thus enhancing its reactivity.

The bottom ashes were treated with class 42.5 R OPC. Different mixtures were prepared with bottom ash and OPC by varying the waste dosage. The investigated bottom ash/total solids (BA/S) percent ratios were varied over the range 10% to 80% (10%, 20%, 30%, 50% and 80%). Control samples containing only cement were also prepared. Mixing was done according to ASTM C305. Distilled water was used as the mixing water at a water/total solids ratio of 0.3, and the mixtures were cast in 50 \times 50 \times 50 mm moulds and allowed to cure at ambient temperature under controlled humidity conditions (RH > 90%) for 24 h. Then, the specimens were demoulded and kept at ambient temperature and RH > 90% until 56 days.

Sample preparation schedule was made in a random sequence, so as to prevent possible chance variables from skewing the results.

The solidified samples were subjected to ANC test. Prior to the test, the specimens were ball-milled to less than 150 μm ; care was taken to carry out grinding in an inert (N_2 atmosphere) environment in order to prevent carbonation. The material was then dried at 60 $^\circ\text{C}$ to constant weight.

The ANC test was conducted over 48 h in a tumbling machine on 11 subsamples by placing 5 g of the ground and dried material into 30-ml nitric acid solutions with increasing acid concentrations. The acid addition schedule was based on the buffering capacity of the material, which was estimated through a 24-h preliminary test.

After 48 h, the liquid/solid separation was performed through centrifugation; pH, redox potential and electrical conductivity were measured in the leachate.

When necessary, the procedure was modified in order to allow the complete titration curve to be drawn, even when the material showed a strong buffering capacity. In particular, the acid addition schedule was modified by varying the acid concentration of the leaching solutions.

The measured pH values were then plotted against the amount of acid added for each individual mixture, and the curves obtained were analysed through differential acid neutralisation analysis.

In order to easily identify the intervals at which the pH plateaux occurred, the titration curve was usefully transformed [3] into a curve representing the cotangent as a function of the amount of acid added. That way, a spectrum of peaks was obtained, allowing the ranges where the material exerted a resistance to acidification to be identified.

Some of the investigated mixtures were also analysed by means of XRD analysis, and the findings were compared with the results of differential analysis of ANC data. In order to stop the hydration, analytical grade acetone (water content < 0.2%) and ethyl ether were used to grind the material prior to XRD.

3. Results and discussion

From Table 1, it is evident that the bottom ashes are mainly composed of Si, Al and Ca oxides, accounting for 79% to 82% by mass of the material on a dry basis. In particular, the Si and Al oxides content ranges from 52% to 70%. When considering the ternary composition of the four bottom ashes, it appears that it is very close to that typical of pozzolanic materials.

Furthermore, XRD analysis of the bottom ashes, not reported here, revealed no significant crystalline phases in HBA, thus confirming its amorphous nature, and small amounts of quartz and calcite in the three MBAs. The relative amount of crystalline species was obtained by calculating the so-called crystallinity index I_c , given by

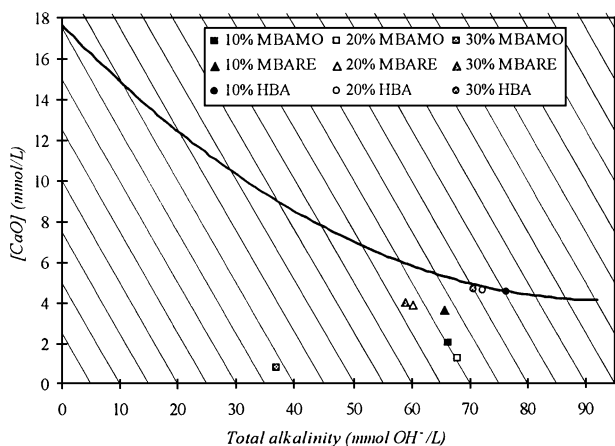


Fig. 1. Pozzolanic activity curves for bottom ashes.

the ratio between the peaks area and total area of the XRD curve; the quantity $(1 - I_c)$ provides an indication on the amorphous content of the material, which for the bottom ashes ranges from 60% to 97% by mass. This suggests that the amorphous siliceous and aluminous species in BA can represent reactive solids, with the potential to confer BA some pozzolanic behaviour. This was assessed from the pozzolanic activity test (see Fig. 1), which revealed that consumption of portlandite produced by cement hydration occurred for all the mixtures.

With respect to the anion content of BA, low chloride and sulphate concentrations were detected, thus confirming very low potential for occurrence of detrimental effects on cement setting, hardening and durability.

Among the trace elements, the major concentration was detected in HBA for Cu and Ni, commonly recognised as lithophilic elements, and therefore likely to be found in bottom ash resulting from waste combustion, and in MBAs for Pb and Zn.

The physical and mechanical properties of the BA–OPC mixtures were reported elsewhere [18], while their

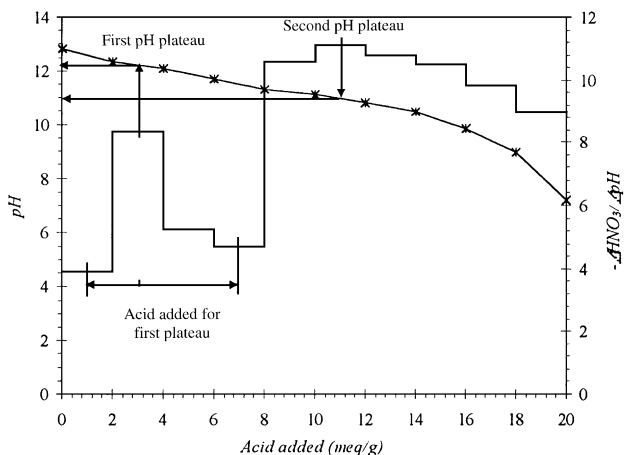


Fig. 2. ANC curve and differential acid neutralisation analysis for the control mixture.

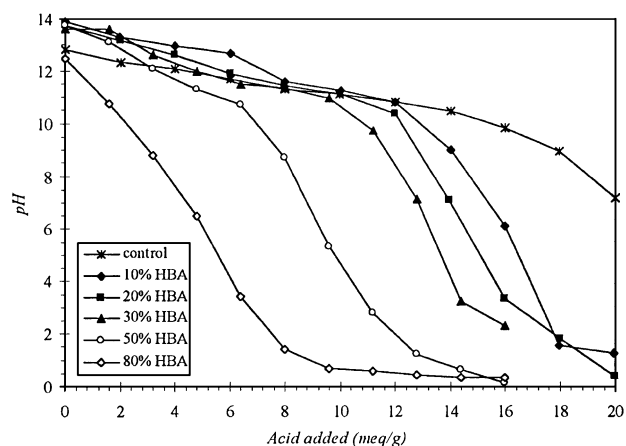


Fig. 3. ANC curves for the control and HBA containing mixtures.

acid neutralisation behaviour is discussed in the proceeding section.

The differential analysis on pH data from the ANC test is shown for the control mixture and the mixtures containing HBA at different BA/S ratios. Fig. 2 depicts the pH versus acid added curve for the control mixture, as well as the related spectrum of peaks, and also shows how differential acid neutralisation analysis was conducted. In Fig. 3 the corresponding curves are plotted for the HBA-containing mixtures. The general feature of the curves was such that an initial series of plateaux is displayed related to the presence of hydration products, followed by a steep drop in pH caused by depletion of such phases at high acid additions. It appears from Fig. 3 that two main plateaux are identifiable for the different HBA–Portland cement mixtures. The first plateau, occurring at a pH value ranging from 12.8 to 13.6, was ascribed to the presence of highly soluble portlandite, while the second plateau, recognised at pH values from 11.0 to 11.4, was ascribed to the presence of C-S-H. The reason for differences in pH values for the two plateaux when compared to those for OPC (12.2 and 11.0, respectively) can be found either in a rise in pH exerted by the alkalis introduced with the waste or in a decrease in Ca/Si ratio of the C-S-H as the bottom ash content increases.

Although some researchers showed that the plateau around pH 11 could be also related to ettringite [1,8,10], for the mixtures investigated, which were tested at 56 days, ettringite was not likely to be found in the presence of bottom ash, as the sulphate content of the blended cement was always far below the critical threshold.

A similar analysis was conducted for mixtures prepared with the other bottom ashes. The curves of pH as a function of acid added are shown in Figs. 4–6 for bottom ash contents of 20%, 50% and 80%.

The results of the differential acid neutralisation analysis are summarised in Table 3 in terms of pH of each plateau and amount of acid required for each plateau to be overcome. As previously explained, the latter is related to the amount of the hydration phase associated to the given pH plateau.

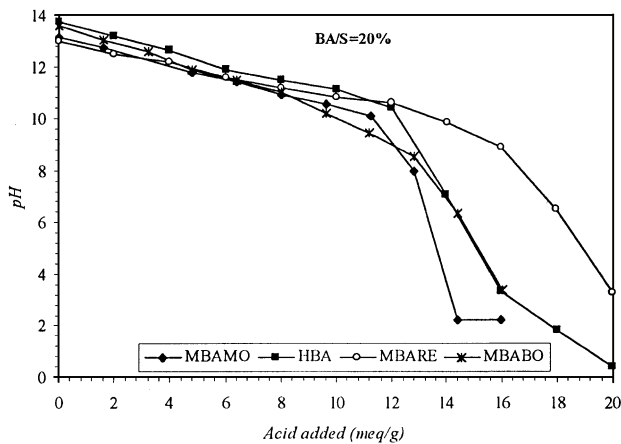


Fig. 4. ANC curves for the mixtures containing 20% BA.

From Figs. 4–6 and Table 3 it appears that the titration curves were very similar for the different mixtures at the same cement replacement level. This is particularly true for pH values varying from 13 down to 9, which is the range where hydration products exert their buffering capacity.

Moreover, the pH values for the two identified plateaux well agree with those found for the HBA–OPC mixtures. Only for the mixture containing 10% MBAMO was a discrepancy evidenced, probably due to improper calibration of the measurement electrode.

A general decrease in the amount of acid required for the first plateau was noticed with increasing the waste dosage of the mixtures, revealing a reduction in the amount of the associated phase (i.e., portlandite). This fact can be explained either from dilution effect due to bottom ash and/or from the additional effect due to its pozzolanic behaviour. Cement dilution by waste material results in a lesser amount of hydration products, including portlandite, being produced, as a consequence of the lower cement content of the mixture. Pozzolanic reactions contribute to a further decrease in the amount of portlandite in the solid matrix compared to that ascribed to cement dilution. Evidence of this was provided by comparison between the XRD patterns at 7-day accel-

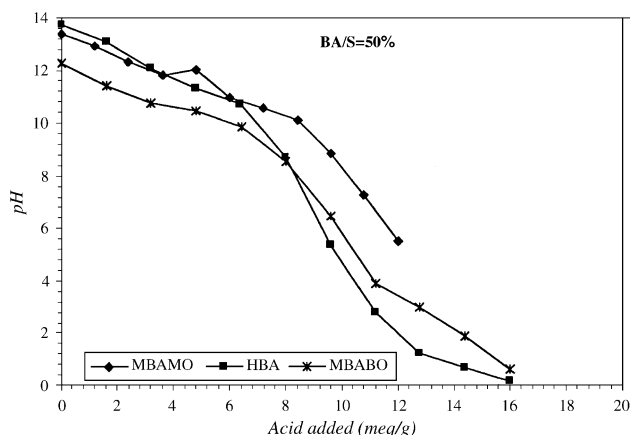


Fig. 5. ANC curves for the mixtures containing 50% BA.

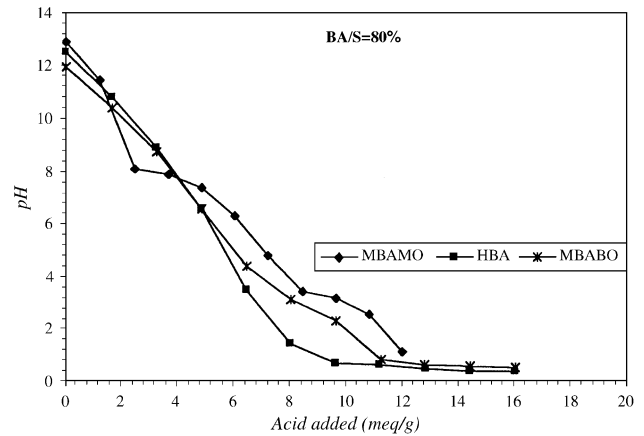


Fig. 6. ANC curves for the mixtures containing 80% BA.

erated curing at 40 °C and RH>90% (see Fig. 7), obtained for three mixtures containing 100% OPC, 30% limestone+70% OPC and 30% MBARE+70% OPC; limestone was considered as an inert material, thereby was added to cement to simulate cement dilution. Although no quantitative analysis was carried out, from Fig. 7 it appears that the relative height of the peaks associated with portlandite decreased when replacing cement both with limestone and bottom ash. However, the decrease was more pronounced when replacing limestone with an equivalent amount of bottom ash. This shows that the portlandite content in the presence of bottom ash was lower than would be expected based on a simple dilution effect on cement. So, it can be concluded that at the investigated cement replacement levels bottom ashes were able to exhibit some pozzolanic reactions,

Table 3
pH plateaux and related amount of acid added

Material	BA/S ratio (%)	pH at 1° plateau	pH at 2° plateau	Acid added for 1° plateau (meq/g)	Acid added for 2° plateau (meq/g)
OPC	0	12.2	11.0	7.0	13.0
	10	11.2	9.6	6.4	8.8
	20	12.0	10.7	4.0	6.4
MBAMO	30	11.8	11.0	4.0	8.0
	50	nd	11.1	—	7.2
	80	nd	nd	—	—
MBARE	10	12.9	10.8	6.0	10.0
	20	12.4	10.7	4.0	14.0
	30	12.9	11.1	4.0	10.0
MBABO	10	12.8	11.5	3.6	4.8 ^a
	20	12.8	11.7	3.2	12.0
	30	11.8	10.6	3.2	9.6
HBA	50	nd	10.6	—	9.6
	80	nd	nd	—	—
HBA	10	12.8	11.4	6.0	10.0
	20	12.8	11.3	4.0	10.0
	30	13.6	11.4	4.0	11.2
HBA	50	13.4	11.0	2.4	6.4
	80	nd	nd	—	—

nd=not detected.

^a The value is affected by the fact that the maximum amount of acid added did not allow the complete titration curve to be plotted.

responsible for portlandite consumption. Further suggestions on this behaviour were provided by comparison of mechanical strength data from the three abovementioned mixtures. After 7-day accelerated curing at 40 °C and RH>90%, the 100% OPC mixture showed an unconfined compressive strength (UCS) of 51.9 MPa, the values for the 30% limestone + 70% OPC and 30% MBARE + 70% OPC mixtures being 25.1 and 40.6 MPa, respectively.

Such conclusions also help interpret the values of acid added for each plateau as shown in Table 3. The amount of each phase can be estimated by calculating the amount of acid added for each plateau, only down to a certain limit, depending on the resolution of the titration curve. This resolution is directly related to the acid addition increment chosen for the ANC test. Since the scope of the ANC test was also to investigate the leaching behaviour of the material in terms of acid neutralisation capacity and contaminants release from the matrix, the acid was added in such amounts that the pH varied within a wide interval (typically from 13 down to 3). This obviously resulted in poor resolution of the peak spectrum, the minimum appreciable acid amount for each plateau being twice the acid increment. If the amount to be calculated was lower than this minimum, the measured value was zero and no peak was detected.

From Table 3 and Figs. 5 and 6 it is evident that for high bottom ash contents one or both pH plateaux disappeared. In particular, for BA/S=50% the plateau associated with portlandite was not recognisable any longer, while for BA/S=80% both those associated with portlandite and C-S-H were absent. On the other hand, when analysing the XRD patterns for mixtures containing 50% bottom ash at 56-day curing (see Fig. 8 for HBA), portlandite was still recognisable. This was related to the poor resolution of the ANC curves obtained from the selected acid addition schedule.

On the contrary, for mixtures containing 80% bottom ash at 56-day curing, even the XRD analysis did not display significant peaks related to portlandite, confirming that

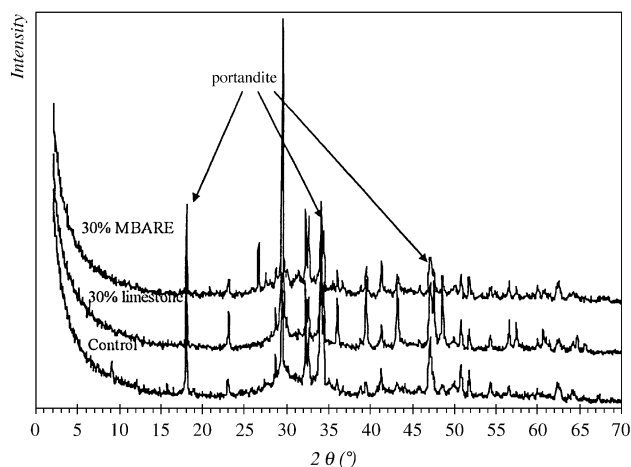


Fig. 7. XRD patterns for the mixtures containing 100% OPC (control), 70% OPC + 30% limestone and 70% OPC + 30% MBARE.

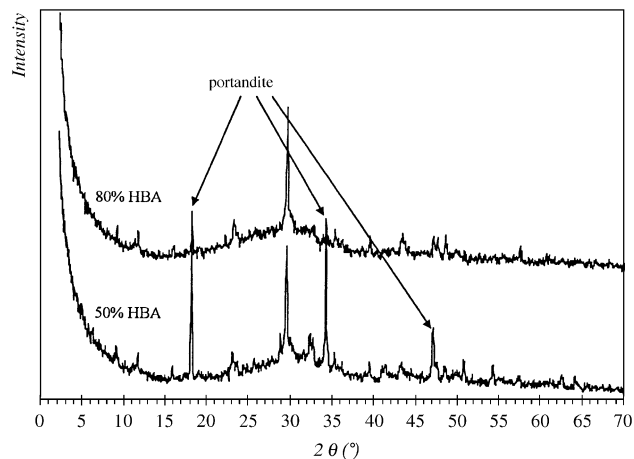


Fig. 8. XRD patterns for the mixtures containing 50% and 80% HBA.

portlandite produced by cement hydration is consumed in pozzolanic reactions (Fig. 8).

As for C-S-H (Table 3), the pH of the second plateau for all the mixtures was in good accordance with the typical range [1,12]. The pH range observed in the present study was 10.6 to 11.7, depending on the bottom ash composition. Unlike the peak distinctive of portlandite, for C-S-H it was not possible to calculate the amount of acid required for such a phase to be neutralised, because in most cases the upper limit of the acid addition interval could not be easily identified due to the shape of the titration curve. Unfortunately, no indication concerning the amount of C-S-H was obtained from XRD, due to the amorphous nature of this phase.

In the case of C-S-H, differential acid neutralisation analysis was therefore able to detect the presence of C-S-H but not estimate its amount. When examining the titration curves for the different mixtures, it was clear that C-S-H was not detectable at 80% cement replacement level, at least with the resolution of the adopted method. Moreover, as previously noticed, at 80% BA content the XRD curves showed the absence of portlandite. Thus, it is tempting to hypothesise that hydration reactions proceeded to some extent even in the presence of 80% BA, as revealed by the strength data. At 56-day curing, the UCS values were 8.1 MPa for HBA, 8.6 MPa for MBABO and 12.9 MPa for MBAMO. In the absence of portlandite, C-S-H can be thought of as the hydration phase responsible for mechanical strength development. However, as a consequence of the low cement content of such mixtures, C-S-H is expected to be formed in small amounts so that it is not able to exhibit a visible pH plateau. In this case, if the pH plateau of C-S-H were to be detected, a lower acid addition increment would have to be adopted in the ANC test.

4. Conclusions

The differential acid neutralisation analysis of ANC data coupled with XRD were used to evaluate the hydration

behaviour of Portland cement mixtures incorporating increasing amounts of incineration bottom ash.

The results from the experimental programme suggest that differential acid neutralisation analysis is able to indicate the presence of hydration products, and also their relative amounts. However, if accurate information is needed, the resolution of the method must be carefully considered.

The findings from the investigation also revealed that the bottom ashes used are capable of exhibiting a weak pozzolanic activity.

Acknowledgments

The experimental work was conducted within the framework of the Industrial and Materials Technologies Programme (Brite-EuRamIII) research project “Neural Network Analysis for Prediction of Interactions in Cement/Waste Systems”, involving Imperial College (UK), Trinity College (I), Universidad de Cantabria (E), University of Rome “La Sapienza” (IT), University of Surrey (UK), British Nuclear Fuels (UK), Euroresiduos (E) and GE.SE.NU. (IT) as partners. Financial support of the European Commission and the industrial partners is gratefully acknowledged.

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