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Ceramic waste as aggregate and supplementary cementing material: A combined action to contrast alkali silica reaction (ASR)

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ABSTRACT

Recently, many efforts have been made to recycle waste of different nature as constituents of sustainable concrete. This practice produces large environmental benefits that can be further extended if deleterious chemical side-reactions, deriving from the use of some types of waste and/or raw materials, could be prevented and suppressed. This paper presents the combined action of different ceramic wastes partially replacing natural sand and cement, respectively. Alkali silica reaction (ASR) promoted by boron–silicate and lead–silicate glass used as fine aggregates ($\leqslant$4 mm) is limited and controlled by using a new type of blended cement based on a siliceous residue coming from sludge produced by the polishing of porcelain stoneware tiles. The results of expansion tests carried out in accelerated conditions together with mechanical and microstructure characterisations of mortar samples highlight the combined action of the investigated wastes. Indeed, the blended cement containing porcelain stoneware polishing residue can be effectively exploited as valid alternative to pozzolan cement.

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1. Introduction

Construction materials, concrete in particular, have attracted great interest as a possible way to recycle industrial and/or urban waste. The relatively simple concrete mixing technology, the large volumes of produced material, the uniform and widespread diffusion of building sites on the territory can easily furnish accessible sites without drawbacks deriving from long transport routes. Fine/coarse aggregates and cement can be successfully replaced by different kind of waste. Recycled aggregates coming from construction and demolition waste have been investigated [1–4] as well as scrap tyres [5,6], plastic waste of different origin [7–9] and waste glass [10] as fine aggregates. As a partial substitution of ordinary Portland cement (OPC), carbon fly-ash has been the precursor of a new type of supplementary cementing materials (SCMs), followed by the use of blast furnace slag [11], pulverized soda lime glass [12], residuals of glass separate collection [13], treated bottom ashes [14] and biomass fly ash [15], activated slag [16,17], rice husk ash [18], metakaolin and calcined clays [19] and even ceramic residues [20]. In the above reported studies waste is proposed as secondary raw material, thus avoiding the costs associated to waste disposal and the negative impact on the environment (landfill saturation, land pollution, etc.). However, the use of non-traditional raw materials can lead to unexpected disadvantages as undesired chemical reactions can occur between traditional and recycled constituents. Indeed, recycled SCM can interfere with cement hydration reactions and mechanical strength development. For instance, metallurgical slags (blast furnace slag, steel slag, etc.) partially replacing OPC, usually result to blended cements with lower early strength and longer setting time [21,22]. Municipal solid waste incineration (MSWI) fly ash has some cementitious activity, but the reactivity is relatively low and its addition to cement may lead to retardation of cement hydration [23]. Special treatments are usually carried out on MSWI fly ash to make them suitable as SCM [24,25]. Furthermore, recycled aggregates can promote the formation of delayed ettringite and/or alkali silica reaction (ASR) leading to the growth of expansive products with disruptive consequences on concrete elements [26–28].

ASR is a deleterious reaction between the amorphous silica of aggregates and cement alkalis, leading to expansive gel formation. Several studies are addressed to better understand ASR when glass is used as sand or gravel replacement in concrete production. Indeed, it has been recently demonstrated that the expansion extent strongly depends on glass chemical composition [26]. For example, lead–silicate and boron–silicate rich glass when used as sand replacement (25 wt.%) in mortar samples induce ASR. The elevate content of ions such as Pb^{2+} and B^{3+} lead to a high solubility of the relevant glass in cement environment and the development of highly expansive products. As recycled glass used for natural aggregate replacement may derive from many sources (urban separated collection, industrial waste, pharmaceutical glassware, television screen, fluorescent lamps, etc.) the possibility that glass rich

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in Pb or B ions might be used in concrete production is rather high, unless a very accurate selection of cullet is not carried out.

One of the most frequently applied methods to inhibit ASR is to use pozzolan cement (CEM IV) or blended cement with pozzolan supplementary cementing materials [29–32]. However, these types of cements are not commonly used as they are generally more expensive than OPC. Certainly, CEM IV pozzolan cement represents only about 6% of the total European cement production [33].

Recently [20,34], a solid residue (hereafter named as PR, polishing residue) resulting from porcelain stoneware tiles polishing sludge has been investigated as a new type of SCM. PR is the solid part of the relevant sludge formed during porcelain stoneware polishing step. This operation is performed on fired tiles by abrasive devices made of silicon carbide (SiC) and magnesium-based (MgOHCl) binder. In Europe, polishing sludge is classified as non-hazardous waste (European Waste Code 10.12.99), but the presence of some compounds (CaO, MgO, SiC and chlorine compounds coming from the abrasive tools) prevents sludge re-introduction into the ceramic production cycle compromising a closed loop recycling. Italy, the 3rd world tile producer, disposes to landfill more than 20,000 ton of polishing sludge every year [35]. Ceramic materials production is largely diffuse in Europe where the major producing countries are Spain, Italy, Germany, UK and France. Between the new Member States of the EU, production is well developed in the Czech Republic, Poland and Hungary, which all have strong ceramics sector [36]. Indeed, ceramic industry also generates large amounts of waste that can be fruitfully exploited for the production of sustainable building materials [37,38].

Polishing residue is obtained by drying the relevant sludge and eliminating the fraction greater than 0.106 mm, that is usually less than 5 wt.%. Neither crushing milling treatments nor washing procedures are necessary, thus ensuring a low cost of the residue as an alternative cement constituent.

The new binder made up of 25 wt.% PR + 75 wt.% CEM I 52.5 R exhibits mechanical and durability performances comparable/better than those shown by mortar based on OPC (100% CEM I 52.5R) and a chemical activity of PR towards calcium hydroxide was determined [20].

The aim of this work is to evaluate the effect of PR based binder as an ASR inhibitor when recycled glass rich of Pb or B ions are used as 25 wt.% replacement of natural sand, thus exploiting different ceramic wastes largely produced. Promoting PR as SCM able to mitigate the deleterious effects of ASR means encouraging the use of blended cement with low clinker content and, consequently, moving towards the use of sustainable construction materials.

The behaviour of PR based binder has been compared with that of OPC (CEM I) and pozzolan cement (CEM IV/A). With each types of cement, mortar samples where prepared using natural sand and lead–silicate or boron–silicate glass as fine aggregate. The results of ASR test, evaluated in accelerated condition (ASTM C1260 [39]), as well as mechanical properties and microstructure of mortar samples cured in standard conditions are reported and discussed disclosing the role played by the investigated wastes.

2. Experimental part

2.1. Materials

A porcelain stoneware tiles polishing sludge was used to obtain PR residue. A highly homogenous sample of sludge representative of a single collecting site was selected (Hera S.p.A. Service for the Environment, Modena, Italy). The sludge was dried ($T = 105$ °C for 24–36 h) and ground in a laboratory ball mill to obtain a fine grain size distribution ($D_{10} = 1.5$ μm, $D_{50} = 8$ μm and $D_{90} = 30$ μm), as characterised by laser particle-size analyser (Master Sizer 2000, Malvern Instrument). As this residue was investigated as cement constituent, the fraction >106 μm was eliminated by sieving. The resulting material, henceforth named PR, has a specific surface area of 25.9 m^2/g (by BET analysis Micrometrics Gemini Series, 2360 with N_2 at 77°K). By use of an X-ray fluorescence spectrometer (PW1414, Philips) and X-ray powder diffraction (PW3710, Philips), the chemical and mineralogical composition of PR was determined. PR is mainly constituted by $SiO_2 = 62.2$; $Al_2O_3 = 15.8$, $MgO = 6.8$, $Na_2O = 3.7$, $CaO = 2.2$, $K_2O = 1.5$ and $ZrO_2 = 1.2$ wt.%. Fig. 1 schematically shows where PR is located in the $CaO–SiO_2–Al_2O_3$ system in comparison with the most used SCM.

By mineralogical analysis, PR discloses the presence of quartz, mullite and albite calcian, deriving from porcelain stoneware body. Amorphous SiO_2 phase, traces of calcite and silicon carbide, deriving from the abrasive tools, have also been determined. A complete chemical–physical characterisation of PR is reported elsewhere [20].

PR based binder was prepared by dry mixing 25 wt.% PR and 75 wt.% of CEM I 52.5 R: the amount of PR was set on the basis of CEM II blended cement compositional range reported in EN 197-1 [40]. Mixing procedure was carried out in a mixer for 30 min to ensure a repeatable mix. Chemical (chloride and sulphate content) and physical (initial setting time and soundness) parameters of PR based binder were previously determined [20] to ascertain the fulfilling of cement requirements set by EN 197-1.

Reference binders were 100% OPC CEM I 52.5 R and 100% pozzolan cement CEM IV/A 42.5 R (Italcementi, Italy)

Silica sand, with normalized particle size distribution according to EN 196-1 [41], was used as aggregate for mortar preparation. Three different types of glass waste were used as a partial substitution (25 wt.%) of silica normalized sand: lead–silicate glass (CG), coming from production of tableware, giftware and home décor items in crystal (kindly supplied by CALP, Colle di Val d'Elsa (Italy), boron–silicate glass (BG) and amber boron–silicate glass (ABG) coming from pharmaceutical containers (kindly supplied by Bormioli Rocco, Fidenza (Italy). The chemical composition (wt.%) of glass is reported in Table 1: values below 50 × LLD have been omitted. The investigated glass were dry ground in a laboratory steel jaw crusher to get particles between 0.075 and 2.00 mm, with size distribution close to that of normalized sand (EN 196-1). Fineness modulus, determined according to EN 12620 [42], is 2.7 for BG, 2.6 for ABG, 2.8 for CG and 2.8 for normalized sand, respectively.

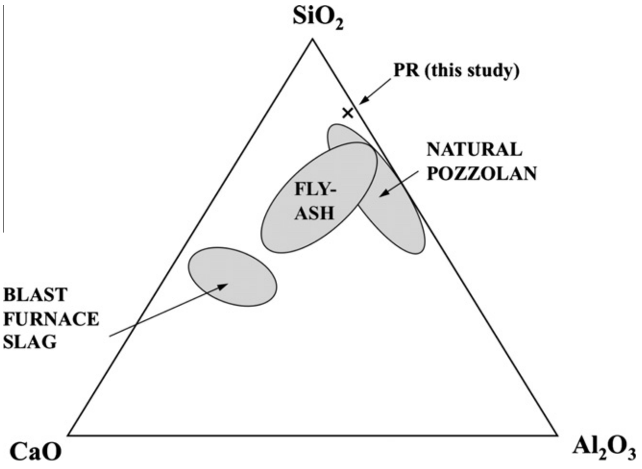

Fig. 1. Ternary diagram of mostly used SCM.

Table 1
Chemical analysis (wt.%) of the investigated glass.

	BG[a]	ABG[b]	CG[c]
SiO_2	68.12	65.30	61.70
Al_2O_3	5.64	5.88	0.04
TiO_2	–	2.89	–
Fe_2O_3	0.02	0.78	–
CaO	1.53	1.42	0.01
MgO	–	–	0.03
K_2O	1.24	1.16	–
Na_2O	8.06	8.02	3.96
ZrO_2	–	–	–
NiO	–	–	–
ZnO	0.82	0.78	0.90
BaO	2.95	2.86	–
PbO	–	–	25.30
P_2O_5	–	–	–
B_2O_3	11.60	10.70	0.72

[a] BG: boron–silicate glass.
[b] ABG: amber boron–silicate glass.
[c] CG: lead–silicate glass.

2.2. Mortar preparation

The investigated mix designs and relevant acronyms are reported in Table 2: mortar samples containing 100 wt.% of silica sand or 75 wt.% of silica sand +25 wt.% of glass, were alternatively prepared with 100% CEM I, 100% CEM IV/A and 25% PR + 75% CEM I 52.5. Mortar samples have been named M, followed by CEM I, CEM IV or PR according to the binder used in the mix design and, when necessary, by BG, ABG and CG indicating the glass type replacing 25 wt.% of silica sand (e.g. MPR-BG is the mortar prepared with PR based binder and boron–silicate glass replacing 25% of silica sand).

Mortar samples were prepared by means of a Hobart planetary mixer of about 5 L capacity, according to the normalized mix-design (binder, sand and water in a weight ratio of 1:3:0.5) and procedure for cement mechanical strength determination (EN 196-1). Mortar prisms (40 × 40 × 160 mm) for mechanical strength determination were cured at 20 °C and R.H. > 95% for 28 days. Mortar prisms (40 × 40 × 160 mm) for accelerated expansion tests were cured in NaOH 1 M at 80 °C for 14 days, according to ASTM C1260.

2.3. Characterisation

Mortar workability was determined by using a flow table according to EN 1015-3 [43]. Compressive strength of mortar samples was determined by an Amsler–Wolpert machine (100 kN) at a constant displacement rate of 2400 ± 200 N/s. Mechanical data are reported as average of at least five measurements. Expansion measurements were determined by means of a mechanical comparator (0.001 mm accuracy) on samples cooled at room temperature: the detailed procedure is elsewhere reported [26] and close to that prescribed by ASTM C1260. Pore size distribution measurements were carried out by mercury intrusion porosimeter (MIP, Carlo Erba 2000) equipped with a macropore unit (Model 120, Fison Instruments). Porosimeter samples, about 1 cm^3, were cut by a diamond saw, dried under vacuum and kept under P_2O_5 in a vacuum dry box till testing. Scanning electron microscopy (SEM) analysis was carried out by means of XL 20 Philips instrument. Sample fresh fractured surfaces were coated by graphite. Phase recognition was acquired by energy-dispersive X-ray spectroscopy (EDX Genesis 2000).

3. Results and discussion

3.1. Fresh state behaviour

The use of the investigated waste has a direct effect on mortar workability as reported in Fig. 2. Regardless the aggregate type and comparing only the effect of the binder, CEM IV based samples always exhibit the highest values of workability, whereas PR based samples always lead to the lowest results. Such behaviour can be ascribed to the different shape of binder particles (Fig. 3). PR particles, deriving from mechanical abrasion processes, mainly have angular shape thus hindering particles flowing. On the contrary, natural pozzolan of volcanic origin and/or silica fly ash, usually both present in CEM IV, mainly exhibit rounded contours particles, which favour workability.

Glass introduction as 25% sand replacement further contributes to a general decrease in workability. The workability loss is more evident when BG and ABG are used according to their lower value of fineness modulus. Moreover, all the glass types have angular shapes with very sharp edges due to their brittle nature, thus explaining the results detected for the relevant mortar samples.

3.2. Expansion, mechanical and microstructure results after accelerated curing condition

With the aim to prove if a combined action occurs between glass waste and PR based binder, expansion test has been carried out in accelerated conditions on mortar specimens alternatively prepared with BG, ABG, CG and normalized sand as fine aggregate and CEM I, CEM IV and 75% CEM I + 25% PR as binder. Fig. 4 reports the expansion data after 14 days of curing in NaOH 1 M solution at 80 °C.

Table 2
Mix-design of mortar samples.

	Binder			Aggregate			
	CEM I	CEM IV	25%PR + 75%CEM I	Silica sand	BG	ABG	CG
MCEM I	450.0	–	–	1350.0	–	–	–
MCEM I-BG	450.0	–	–	1012.5	337.5	–	–
MCEM I-ABG	450.0	–	–	1012.5	–	337.5	–
MCEM I-CG	450.0	–	–	1012.5	–	–	337.5
MCEM IV	–	450.0	–	1350.0	–	–	–
MCEM IV-BG	–	450.0	–	1012.5	337.5	–	–
MCEM IV-ABG	–	450.0	–	1012.5	–	337.5	–
MCEM IV-CG	–	450.0	–	1012.5	–	–	337.5
MPR	–	–	450.0	1350.0	–	–	–
MPR-BG	–	–	450.0	1012.5	337.5	–	–
MPR-ABG	–	–	450.0	1012.5	–	337.5	–
MPR-CG	–	–	450.0	1012.5	–	–	337.5

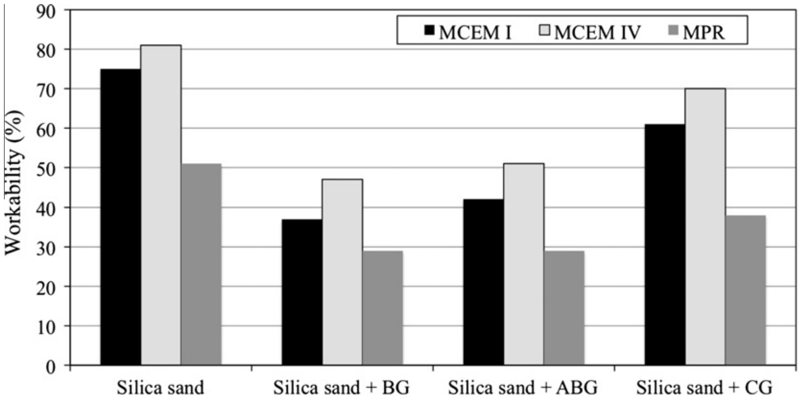

Fig. 2. Workability of mortar samples.

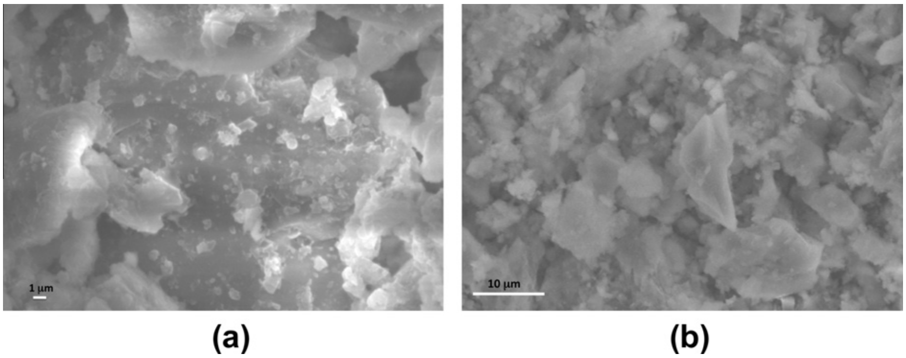

Fig. 3. SEM images of CEM IV (a) and PR (b).

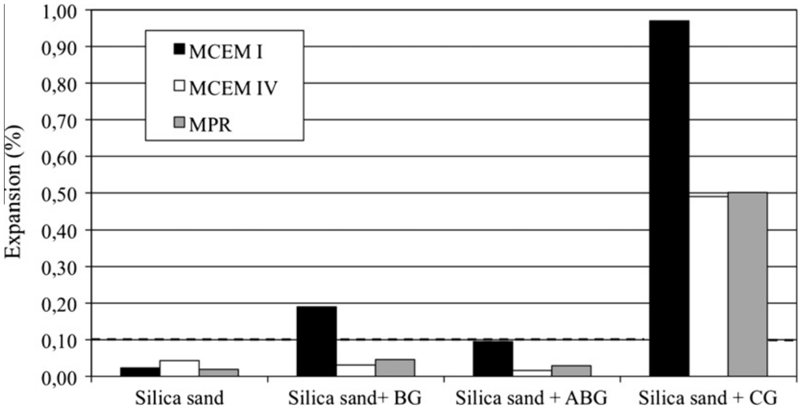

Fig. 4. Expansion of mortar samples after 14 days of curing in NaOH 1 M solution at 80 °C (dotted line shows the limit set up by ASTM C1260; expansion values reported are the average of three measurements).

According to previous results [26], when CEM I is used with BG, ABG and CG, expansion processes occur in the relevant mortar samples generally overcoming the limit (0.10%) fixed by ASTM C1260 (MCEMI-ABG shows expansion equal to 0.10%). CG causes the largest mortar expansion. When CEM IV and PR based binder are adopted, a strong reduction of expansion process is evident. Mortar samples containing BG and ABG are safely well below the limit and an expansion decrease of about 50% is determined when CG is used. Thus, PR based binder works as inhibitor of alkali silica reaction as commercial pozzolan cement does.

Fig. 5 shows compressive strength values of the investigated mortars after expansion test.

According to the results above discussed, the highest compressive strengths are determined when no expansion occurs. When 100% silica sand is used as aggregate, the mortar compressive strengths are ⩾52.5 MPa and ⩾42.5 MPa in good agreement with the mechanical strength class of the cement used (CEM I 52.5 R, CEM IV 42.5 R, to 75% CEM + 25% PR a 52.5 R class has been previously assigned [20]). When glass is used as 25% sand replacement, the decrease in compressive strength is strictly related to the extent of the expansion. For MCEM I, the highest reduction is detected when CG is adopted, followed by samples containing BG and ABG. For MCEM IV and MPR the compressive strength reduction is also more evident when CG is used according to expansion results that

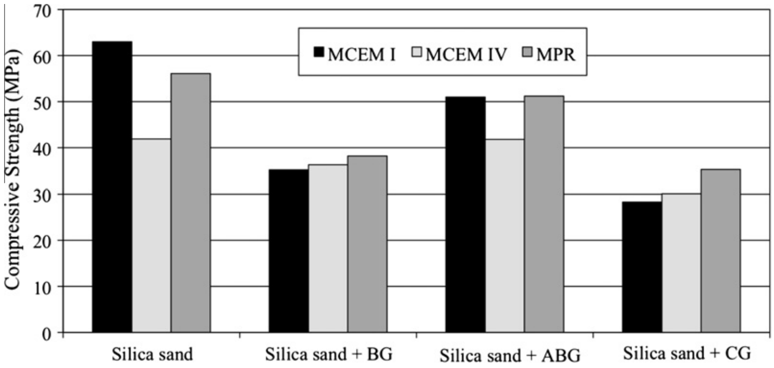

Fig. 5. Compressive strength of mortar samples after expansion test.

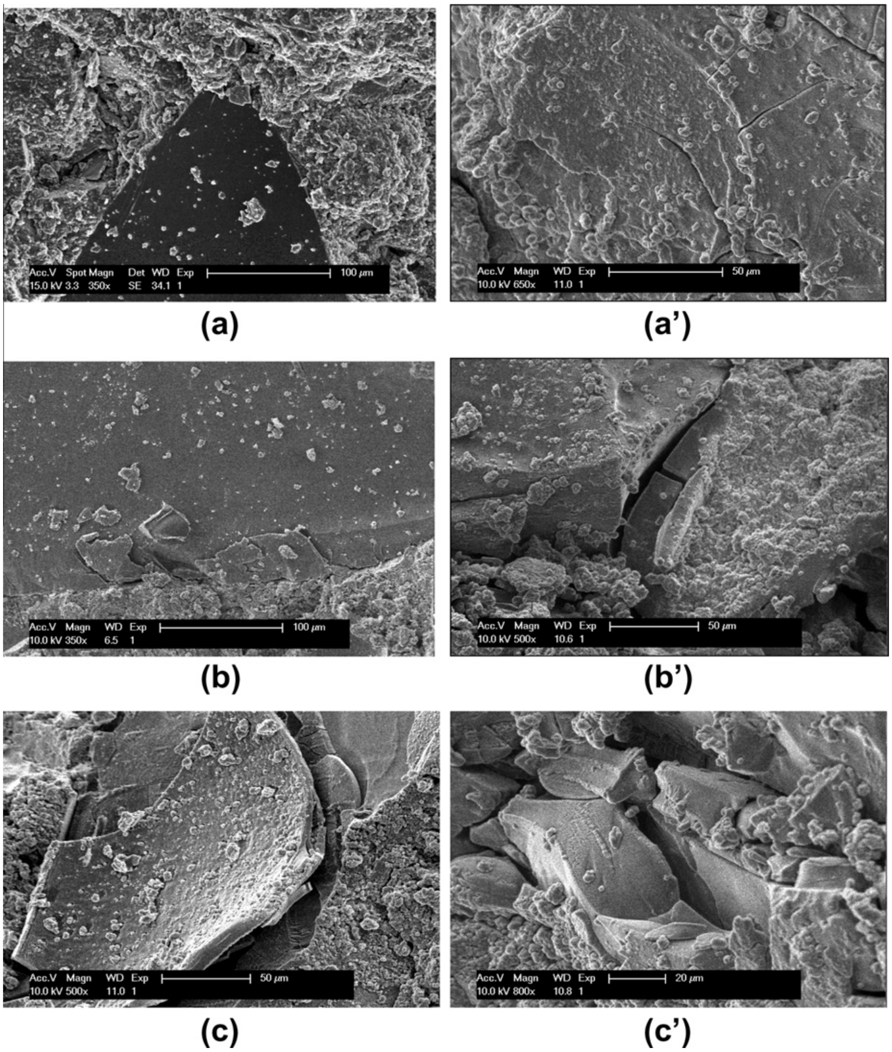

Fig. 6. Microstructure of MPR-BG (a) and MPR-CG (a′), MCEM IV-BG (b) and MCEM IV-CG (b′), MCEM I-BG (c) and MCEM I-CG (c′) after expansion test.

still overcome the limit of 0.10%. However, for MCEM IV and MPR containing BG and ABG the lowering of the compressive strength is less evident according with the very low expansion determined.

SEM analysis provides an insight in mortar microstructure after the expansion test. Fig. 6 shows BG and CG in the three different cement matrices. The sharp, un-reacted contour of BG and the good adhesion with PR based matrix confirms the suppression of ASR (Fig. 6a). A similar behaviour for BG is also observed when it is immersed in CEM IV matrix (Fig. 6b), whereas BG reactivity is clearly evident when CEM I is used (Fig. 6c). The evidence of BG reacted particles and cracks in OPC matrix are further proofs of ASR occurring in MCEM I sample.

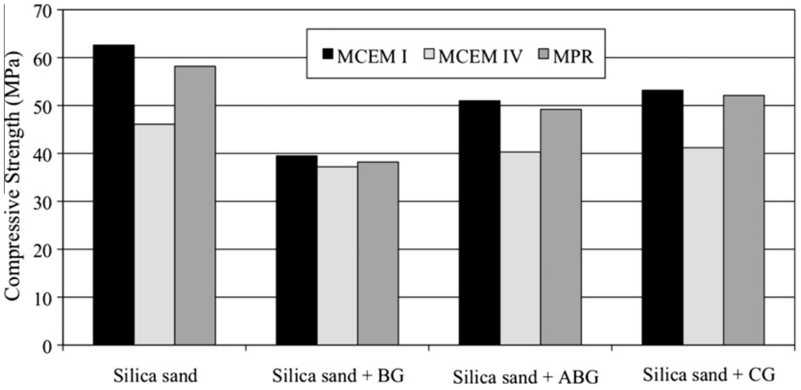

Fig. 7. Compressive strength of mortar samples after 28 days of curing in standard condition.

The reactivity of CG, which always forms expansive products, becomes more and more evident passing from MPR (Fig. 6a) and MCEM IV (Fig. 6b) to MCEM I matrix (Fig. 6c). For the latter, the extended presence of expansive products on CG surface leads to the formation of very large cracks thus exhibiting the typical effects of ASR. Indeed, PR based binder is able to reduce ASR caused by BG and ABG under the limit and mitigate ASR consequences when CG is used. PR based binder behaviour is then close to that of commercial pozzolan cement and, consequently, PR use as a SCM is promising.

3.3. Mechanical and microstructure results after standard curing condition

The investigated types of waste are attractive as their use can be exploited for green/sustainable building. For this reason mechanical characterisation and microstructure analyses have been carried out on mortar samples cured in standard conditions too.

Compressive strengths are reported after 28 days of curing at 25 °C and R.H. $\geqslant$ 90% in Fig. 7. Compressive strength of mortar samples strictly follows binder strength class. In fact, MCEM I and MPR (strength class: 52.5 R) show the highest values comparing to MCEM IV (strength class: 42.5 R). Glass introduction, regardless the type, leads to a compressive strength lowering and such reduction is particularly evident when BG is used. Compressive strength decrease is probably due to the low workability detected when glass is used as aggregate which leads to a general increase in total open porosity (water absorption of 100% silica sand mortar samples is about 10% lower than relevant mortars containing glass). Moreover, less effective adhesion of the binder matrix to the smooth surface of the glass has been elsewhere reported [44,45].

Compressive strengths data obtained with standard curing condition are generally equal or superior than the relevant results obtained in accelerated curing condition. The different behaviour in mechanical performances is particularly evident when CG is used as aggregate, according to CG ability to promote ASR.

Figs. 8 and 9 report pore size distributions obtained by MIP for MPR and MCEM IV samples, respectively. Regardless the binder used, mortar samples containing BG exhibit the highest porosity for pore size $\geqslant$0.1 μm according to the lowest workability detected (Fig. 2). Cumulative pore volume data of MPR samples are lower than those of relevant samples prepared with CEM IV. The mean pore radius for MPR ranges from 0.05 to 0.07 μm, whereas for MCEM IV values between 0.09 and 0.13 μm are detected. The more refined capillary porosity suggests a faster hydration at 28 day of curing for PR based binder compared to the commercial pozzolan binder. Indeed, PR works as a very active SCM promoting a more compact matrix in standard curing condition and inhibiting/moderating ASR.

Fig. 10 shows microstructure and EDX analysis of the fracture surfaces of MCEM IV and MPR samples after 28 days of curing. No significant differences in matrixes morphology can be highlighted. EDX results on hydration products show an average Ca/Si weight ratio lower than 1.5, in particular 1.3–1.4 for MCEM IV and 1.1–1.3 for MPR. The determined ratios are comparable and confirm that PR particles react in a very similar way to natural pozzolan, even though low amounts (<3%) of Mg, K and Na are present in MPR.

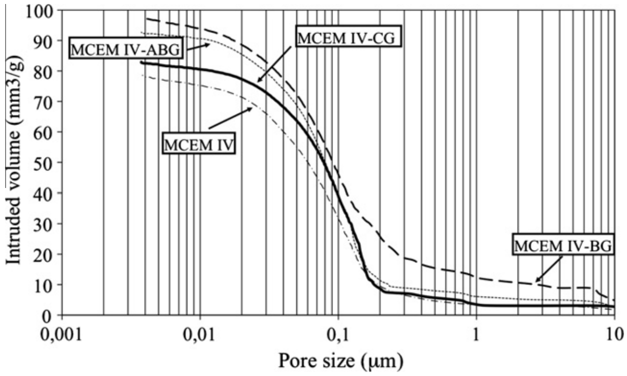

Fig. 9. Pore size distribution of MCEM IV samples after 28 days of curing in standard condition.

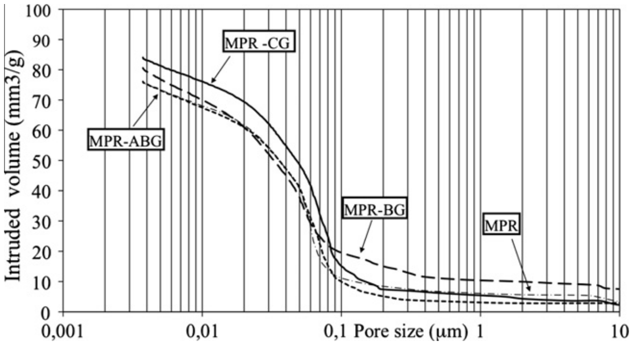

Fig. 8. Pore size distribution of MPR samples after 28 days of curing in standard condition.

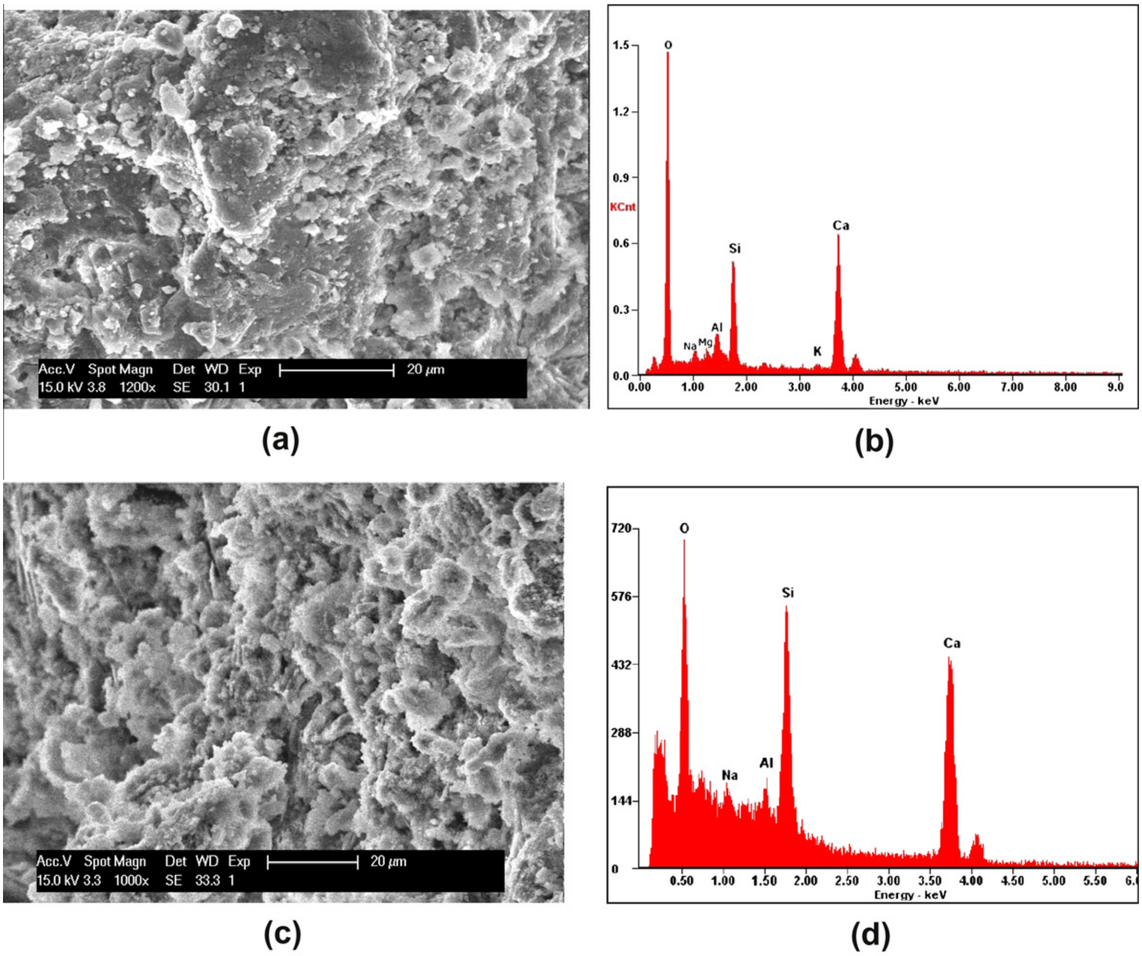

Fig. 10. Microstructure and EDX analysis of PR matrix (a and b) and CEM IV matrix (c and d) after 28 days of curing in standard condition.

4. Conclusions

Based on the results above described, the following conclusions can be drawn by the present study:

- Polishing residue (PR) coming from porcelain stoneware tiles polishing sludge is effective in reducing/suppressing alkali silica reaction promoted by the use of glass aggregate;
- Mortar samples prepared with PR based binder and boron or amber-boron silicate glass show good dimensional stability, high mechanical properties and compact microstructure thus ensuring their use for sustainable building;
- PR based binder is a valid alternative to commercial pozzolan cement as it is able to sustain a safe use of cullet coming from many sources (separated collection, industrial waste, fluorescent lamps, etc.) potentially containing fractions of expanding glass.

It has been highlighted that a tailored combination of different types of ceramic waste can be successful for designing green building materials. As raw materials safeguard and waste exploitation create environmental benefits and represent the milestones for a sustainable development, political authorities should endorse such issues with the aim to promote innovative and ecological attitudes in the construction industry.

Finally, this research promotes the use of supplementary cementing material for ASR suppression thus limiting such deleterious reaction that generally creates severe and dangerous damages in the building construction. Potentially active aggregates as glass or amorphous silica rich natural minerals could thus be safely used for mortar and concrete preparation.

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