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Use of screen glass and polishing sludge in waste-based expanded aggregates for resource-saving lightweight concrete

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ABSTRACT

The purpose of this study is to evaluate different lightweight concretes for the first time formulated with lightweight expanded aggregates produced only with industrial waste. The motive is that a lack of knowledge still exists in the literature about how to integrate these waste-based aggregates in lightweight concrete. To achieve the desired bloating and aggregates physical properties, PC-TV screen glass and ceramic tile polishing sludge were selected as suitable raw materials. Both were characterized by mineralogical and chemical analyses and the effect of different combinations was pointed out. Hot-stage microscopy was used to determine the bloating rates and firing behavior. Lightweight expanded aggregates were obtained using both static laboratory kiln and rotating pilot kiln, by firing at maximum temperatures between 1150 and 1200 °C, to simulate the industrial production process and favor scaling up. The so obtained aggregates were characterized from the physical-mechanical point of view, highlighting an important bloating attitude and bulk density lower than 700 kg/m³ for all the test conditions. Bulk density, water absorption and mechanical properties are fully comparable to commercial counterparts. The best material was used as coarse aggregate in lightweight structural concrete and cellular concrete prepared at pilot scale (for structural application and thermal/acoustic insulation, respectively). The technical properties are consistent with standard requirements of compressive strength (>25 MPa for lightweight structural concrete) and thermal conductivity (18-24 W/m*K for cellular concrete). These results demonstrate the technological feasibility of using waste-based aggregates into lightweight concrete design, according to a circular economy vision.

1. Introduction

The systematic switch-off of the analogical broadcasting system and the consequent passage to digital terrestrial broadcasting, is accompanied by huge replacement of old CRT (Cathode Ray Tube) apparels with new TV units equipped with LCD/LED screens (Hermans et al., 2001). Unfortunately, despite increasing efforts, few advances were made on the treatment process of e-wastes so that the preferred choice is still limited to landfilling (Baldé et al., 2017).

As reported by "The Global E-waste Monitor 2014: Quantities, flows and resources" (from the United Nations University), 41.8 million tons

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of electronic wastes and millions tons of cathode ray tube residues were produced in 2014 worldwide (Baldé et al., 2017). This trend is not expected to decrease in next years (Yao et al., 2018). The presence of hazardous elements, i.e. Pb in the tube and funnel glass and high amount of Sr and Ba in the screen glass (SG), generates problems related to old screens disposal and waste management (2000/532/ EC, 2000). The disposal of such component represents only among the various critical issues common to many other wastes from industrial activities that should be considered according to concepts of circular economy and reuse For this reason, several recent investigations (Behera et al., 2021; Camana et al., 2021; Chinnu et al., 2021; Lim et al., 2019; Liu and Coffman, 2016; Ma et al., 2021; Mpatani et al., 2021; Upadhyay et al.,

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| List of u | used abbreviations |
|-----------|---|
| С | Cellular concrete experimental specimen |
| CCAT | Charge Compensated Aluminium in Tetrahedral |
| | coordination |
| CRT | Cathode Ray Tube |
| DPM | Dried Polishing Mud |
| EDS | Energy Dispersive Spectroscopy |
| GNF | Glass Network Formers |
| GNM | Glass Network Modifiers |
| HSM | Hot Stage Microscope |
| LCD | Liquid Crystal Display |
| LEA | Lightweight Expanded Aggregates |
| LED | Light Emitting Diode |
| LS | Lightweight Structural concrete experimental specimen |
| LoI | Loss on Ignition |
| NBO/T | Non-Bridging Oxygens per Tetrahedrally-coordinated |
| | cations |
| SEM | Scanning Electron Microscope |
| SG | Screen Glass waste |
| SiC | Silicon Carbide |
| XRPD | X-Ray Powder Diffraction |
| XRF | X-Ray Fluorescence |
| | |

2021; Wan et al., 2021) proposed a plenty of solutions aimed at using municipal or industrial wastes in the ceramic or building sectors, including Lightweight Expanded Aggregates (hereafter LEA) giving them a sort of second life as secondary raw materials (Dondi et al., 2009, 2016). This increased interest in LEA production is also boosted by a growing number of studies on the use of a wider range of urban and industrial wastes as materials for LEA production (Cáceres et al., 2019; Dondi et al., 2016).

According to standard definition, lightweight aggregates are mineral granular, with a bulk density not higher than 2000 kg/m³ this latter considered as the key classifier parameter that distinguishes lightweight from dense aggregates (UNI EN 13055, 2016; Bush et al., 2006; de Gennaro et al., 2004). Lightweight aggregates have a lot of applications: e.g., as loose material, in back wall fillers and in the agronomic field or, once mixed with a binder, in the manufacture of plaster, asphalt and lightweight structural concrete, in particular in the production of thermo-acoustic insulators in lightweight concrete (Mindness et al., 2002). Due mainly to the increasing demand for lightweight structural and insulating (thermal and acoustic) mortars and concretes, LEA are currently the object of a revitalized interest (Kurpińska and Ferenc, 2020; Li et al., 2020; Oktay et al., 2015).

Artificial lightweight expanded aggregates are usually produced from natural raw materials (such as clay, shales or zeolites-rich rocks) able to bloat by themselves (Bush et al., 2006; de Gennaro et al., 2009; Dondi et al., 2016) through a quick heating at high temperature given by gases released during hating. If the ceramic matrix exhibits a suitable permeability and viscosity of the liquid phase, the formed gases can be then entrapped and the desired expansion is achieved (Molinari et al., 2020; Moreno-Maroto et al., 2020). If the ceramic matrix exhibits a suitable permeability and viscosity of the liquid phase, the formed gases can be then entrapped and the desired expansion is achieved (Molinari et al., 2020; Moreno-Maroto et al., 2020). When these conditions do not occur, the addition of an expanding agent is necessary. An industrial waste can be selected as gas-forming substance (Dondi et al., 2016). Dried Polishing Mud (hereafter DPM) from stoneware tile polishing process is known as bloating enhancer, given by silicon carbide contamination (about 1% SiC) derived by lapping tools abrasion (de Gennaro et al., 2007, 2009; Zanelli et al., 2021). At high temperature, SiC oxidises leading to dissociation and gas production (Molinari et al.,

2021; Opila and Nguyen, 1998).

Overall, LEA are currently the object of a revitalized interest, due mainly to the increasing demand for lightweight structural and insulating (thermal and acoustic) mortars and concretes (Kurpińska and Ferenc, 2020; Li et al., 2020; Oktay et al., 2015; Xie et al., 2019).

This interest is also fueled by a growing awareness about environmental sustainability, which is stimulating various studies on the use of urban and industrial wastes as possible raw materials for LEA production (Amin et al., 2020; Cáceres et al., 2019; Dondi et al., 2016).

Such new generation of raw materials is often characterized by a much wider range of chemical and mineralogical composition with respect to clays and shales. For this reason, this new kind of aggregates often shows a different technological behavior, imposing some adjustments to the manufacturing process (shaping and firing treatments) in order to meet the expected product specifications.

Glass-based batches represent a test-bench of chemical composition and operative conditions, in order to appraise the role of the physical properties of LEA in binder hardening, such as water absorption, bulk density, and mechanical strength. Another goal is to go further the state of the art for waste recycling: in literature there is a certain number of papers dealing with expansion of waste glass (Adhikary et al., 2021; Bernardo et al., 2010; Ducman et al., 2002; Kourti and Cheeseman, 2010; Li et al., in press; Molinari et al., 2021; Mueller et al., 2008; Pascual et al., 2021; Tuan et al., 2013; Wei et al., 2011; Yio et al., 2021) but no one exist, at best of our knowledge, using glass waste from PC and TV screen and moreover on concretes based on these LEA.

Unlike other papers, this research:

- aims to demonstrate the technological feasibility of different lightweight concretes for the first time formulated with waste-based aggregates;
- Evaluates the full production chain: from the formulation of suitable LEA batches by utilizing only waste (PC-TV screen and SiC-containing porcelain stoneware tile polishing sludge), to the upscaling of LEA manufacturing to a pilot line, to eventually formulate, produce and characterize the lightweight concretes containing waste-based LEA.

The paper is so structured: after the description of experimental activities (section 2), the results are illustrated and discussed, first about waste characterization and mix formulation (section 3.1) then moving to LEA production and characterization (section 3.2) and finally production and characterization of lightweight concretes (section 3.3).

2. Materials and methods

Two wastes were selected from same number Italian waste management facilities: a glass from TV-PC screen (SG) and a mud from the polishing of ceramic tiles (DPM). Both starting materials and fired products were characterized by:

2.1. Mineralogical composition

Mineralogical qualitative and quantitative analysis (Table 1) were performed by X-ray powder diffraction (XRPD) using a Panalytical X'Pert Pro diffractometer, equipped with a RTMS X'Celerator detector with Cu-K α radiation, operating at 40 kV and 40 mA. Scans were collected in the range 5–80 °20 using a step interval of 0.017 °20, with a step counting time of 120 s. Mineral phases were identified by the Panalytical Highscore Plus 3.0c software and PDF-2/ICSD mineral databases and quantitative phase Rietveld refinement were performed using Topas software (version 5.0, Bruker, Germany) (Bish and Howard, 1988; Bish and Post, 1993; Rietveld, 1969). Crystalline and vitreous phases were calculated by means of internal standard method (20 wt% of Al₂O₃, 1 µm, Buehler Micropolish).

Table 1

Chemical and mineralogical composition of waste and their characteristic temperatures by hot-stage microscopy. ^aExpansion <100 vol% is assumed for samples that did not reach the initial volume after the first sintering phase.

| Components | Units | Ceramic tile polishing sludge (DPM) | TV and PC screen glass waste (SG) |
|--|-------|-------------------------------------|-----------------------------------|
| SiO ₂ | wt% | 66.37 | 63.78 |
| TiO ₂ | | 0.55 | 0.41 |
| Al ₂ O ₃ | | 19.35 | 2.34 |
| Fe ₂ O ₃ | | 0.81 | 0.10 |
| MgO | | 2.64 | 0.31 |
| CaO | | 1.64 | 1.02 |
| SrO | | 0.05 | 7.73 |
| BaO | | 0.04 | 8.55 |
| Na ₂ O | | 3.47 | 7.53 |
| K ₂ O | | 1.81 | 7.22 |
| P ₂ O ₅ | | 0.26 | - |
| Loss on ignition | | 3.00 | 1.02 |
| Quartz | wt% | 18 | 2 |
| K-feldspar | | 3 | - |
| Plagioclase | | 3 | - |
| Vitreous phase | | 72 | 98 |
| Others (mullite, zircon, SiC, etc.) | | 3 | tr |
| Sintering temperature (T _s) | °C | 1185 ± 5 | 695 |
| Softening temperature (T _r) | °C | 1220 ± 5 | 855 |
| Melting temperature (T _f) | °C | 1330 ± 5 | 1080 |
| Temperature of maximum expansion (T _{me}) | °C | 1240 ± 5 | 1215 |
| Temperature of max expansion rate (T _{mve}) | °C | 1220 ± 5 | 1200 |
| Maximum volume expansion at T _{me} | vol% | 310 ± 0.1 | $<\!100^{a}$ |
| Isothermal expansion after 30 min at T _{mve} | vol% | 250 ± 0.1 | <100 ^a |

2.2. Chemical composition

Chemical analyses of raw materials were carried out, on pressed powder pellets, using an Axios Panalytical X-ray fluorescence (XRF) spectrometer, equipped with six analyzer crystals, three primary collimators and two detectors (flow counter and scintillator). Analytical uncertainties are 1–2% for major elements and 5–10% for trace elements (Cucciniello et al., 2017). The weight Loss on Ignition (LoI) was determined by gravimetric techniques, firing at 1000 °C powders previously dried at 110 °C overnight.

2.3. Firing behavior and LEA production

Firing behavior was evaluated by a Hot-Stage Microscope (HSM -Expert System Solutions - Misura 2) on cylindrical specimen heated with a heating rate of 10 °C/min until melting (2 mm diameter, 3 mm height) (Dondi et al., 2001). The laboratory scale batches were designed with the goal of maximizing the amount of wastes that allow to comply with the required physical and technological properties of the products. The mix design and the obtained chemistry are reported in Table 2. Laboratory simulation of LEA manufacturing process was conducted on three mixes by preparing about 8 g of powder and pressing at 40 MPa (pellets with a diameter of 10 mm). The pellets were fired in electric chamber static kiln (Nannetti mod. CV) at maximum temperatures of 1150 and 1200 °C and 5 min well time. Moreover, the best mixture of was chosen to a larger scale production (35 L) and in rotating inclined kiln (Lab scale prototype from Nannetti) for about 40 min, with 5 min of soaking time and at the maximum temperature of (1225 °C) (de Gennaro et al., 2009).

2.4. Composition and properties of the vitreous phase

Based on bulk chemistry and mineralogical properties of the fired bodies, chemical composition of vitreous phase was determined to evaluate glass structure parameters (Table 3). The chemical composition

Table 2

| Mix c | lesign | and o | chemical | composition | of | the | LEA | batches. |
|-------|--------|-------|----------|-------------|----|-----|-----|----------|
|-------|--------|-------|----------|-------------|----|-----|-----|----------|

| Mix design (wt %) | Mix 1 | Mix 2 | Mix 3 |
|--------------------------------|-------------------|-------|-------|
| DPM | 40 | 50 | 60 |
| SG | 60 | 50 | 40 |
| Chemical composition of the | LEA batches (wt%) | | |
| SiO ₂ | 66.03 | 66.43 | 66.83 |
| TiO ₂ | 0.47 | 0.49 | 0.50 |
| Al ₂ O ₃ | 9.40 | 11.16 | 12.92 |
| Fe ₂ O ₃ | 0.40 | 0.47 | 0.54 |
| MgO | 1.27 | 1.51 | 1.75 |
| CaO | 1.29 | 1.36 | 1.42 |
| SrO | 4.71 | 3.93 | 3.16 |
| BaO | 5.20 | 4.34 | 3.48 |
| Na ₂ O | 5.99 | 5.59 | 5.19 |
| K ₂ O | 5.12 | 4.58 | 4.04 |
| P ₂ O ₅ | 0.11 | 0.13 | 0.16 |
| Loss on Ignition | 1.80 | 2.00 | 2.21 |

Table 3

Phase composition together with the chemical composition and estimated shear viscosity of the vitreous phase.

| Samples | Mix1 | | Mix2 | | Mix3 | |
|------------------------------------|------------|------------|-------|-------|-------|-------|
| Firing temperature (°C) | 1150 | 1200 | 1150 | 1200 | 1150 | 1200 |
| Bulk density (kg/m ³) | 710 | 770 | 740 | 730 | 740 | 770 |
| Phase composition (wt%) | | | | | | |
| Quartz | 2 | 1 | 2 | 3 | 3 | 3 |
| K-feldspar | 9 | 8 | 12 | 10 | 18 | 14 |
| Plagioclase | 5 | 5 | 5 | 5 | 2 | 2 |
| Vitreous phase | 84 | 86 | 81 | 82 | 77 | 81 |
| Chemical composition of t | he vitreou | s phase (v | vt%) | | | |
| SiO ₂ | 65.52 | 65.91 | 66.04 | 65.59 | 66.11 | 66.04 |
| TiO ₂ | 0.56 | 0.55 | 0.60 | 0.60 | 0.65 | 0.62 |
| Al ₂ O ₃ | 7.86 | 7.89 | 9.64 | 9.97 | 11.90 | 12.22 |
| Fe ₂ O ₃ | 0.48 | 0.47 | 0.58 | 0.57 | 0.70 | 0.67 |
| BaO | 6.19 | 6.05 | 5.36 | 5.29 | 4.52 | 4.30 |
| SrO | 5.61 | 5.48 | 4.85 | 4.79 | 4.10 | 3.90 |
| MgO | 1.51 | 1.48 | 1.86 | 1.84 | 2.27 | 2.16 |
| CaO | 1.28 | 1.25 | 1.42 | 1.40 | 1.73 | 1.65 |
| Na ₂ O | 6.58 | 6.42 | 6.32 | 6.25 | 6.50 | 6.18 |
| K ₂ O | 4.28 | 4.38 | 3.15 | 3.52 | 1.30 | 2.07 |
| P ₂ O ₅ | 0.13 | 0.13 | 0.16 | 0.16 | 0.21 | 0.20 |
| $NBO/T^{a}(1)$ | 0.26 | 0.26 | 0.25 | 0.25 | 0.23 | 0.23 |
| GNF ^b (% atom) | 34.79 | 34.98 | 35.98 | 35.94 | 37.20 | 37.33 |
| CCAT ^c (% atom) | 4.16 | 4.18 | 5.10 | 5.28 | 6.30 | 6.47 |
| GNM ^d (% atom) | 18.21 | 17.85 | 15.38 | 15.29 | 12.33 | 11.95 |
| Viscosity (Log ₁₀ Pa s) | 3.04 | 2.77 | 3.30 | 2.98 | 3.55 | 3.24 |

^a (NBO/T) Degree of polymerization of the melt - From the composition of the liquid phase: Number of Non-Bridging Oxygens (NBO) per Tetrahedrally-coordinated cations (Si,Al) as atomic percentage.

 $^{\rm b}$ (GNF) Glass network formers - From the composition of the liquid phase: GNF (atom%) = Si + CCAT, corresponding to Al^{3+}charge compensated by alkali or alkaline earths.

 $^{\rm c}$ (CCAT) Charge compensated aluminium in tetrahedral coordination - From the composition of the liquid phase: Al^{3+} charge compensated by alkali and alkaline earths: CCAT(atom%) = Na + K+2Ca+2 Mg (up to max value = Al).

 d (GNM) Glass network modifiers - From the composition of the liquid phase: alkali and alkaline earths exceeding CCAT: GNM (atom%) = Na + K+2 Mg+2Ca–CCAT.

of the vitreous phase was calculated by subtracting from the bulk chemistry of the fired body the contribution of mineralogical phases, assuming their stoichiometric compositions weighted on the quantitative phase analysis. Physical properties at high temperature were estimated by predictive models based on the chemical composition of the liquid phase (Fluegel, 2007). The vitreous phase contains different elements that affect both structure and properties of the glass network. In order to facilitate data interpretation, some parameters were used to express specific structural features of the melt:

- Degree of depolymerization of the melt (NBO/T) defined as the number of Non-Bridging Oxygens (NBO) per tetrahedrallycoordinated cations (Si, Al) as atomic percentage and calculated from the composition of the vitreous phase;
- Glass network formers (GNF) From the composition of the liquid phase: GNF (atom%) = Si + CCAT, corresponding to Al^{3+} charge compensated by alkali or alkaline earths.
- Charge compensated aluminium in tetrahedral coordination (CCAT) From the composition of the liquid phase: Al^{3+} charge compensated by alkali and alkaline earths: CCAT(atom%) = Na + K+2Ca+2 Mg (up to max value = Al).
- Glass network modifiers (GNM) From the composition of the liquid phase: alkali and alkaline earths exceeding CCAT: GNM (atom%) = Na + K+2 Mg+2Ca-CCAT.

2.5. Microstructure

Micro-textural observations were carried out by Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS; JEOL JSM-5310 coupled with Oxford Instruments Microanalysis Unit equipped with an INCA X-act detector) and by using the digital microscope (HIROX RH-2000).

2.6. LEA and concrete characterization

Physical and technological properties of LEA were tested by means of European Standards: bulk density (kg/m³) (Archimede's principle), loose bulk density (kg/m³), strength of particle (MPa) (Control Test equipment - load rate 1 MPa*s-1, 20 samples), particle size distribution (mm) and water absorption (%) (UNI EN 13055, 2016). As regards concretes, four different batches were designed and produced following standard indications (UNI 11013, 2002) in order to test LEA-containing concretes and regular ones (as reference) for each feature. For this reason, two kind of lightweight structural concretes (LS1-LS2) and cellular concretes (C1–C2) were cured in a controlled temperature chamber (20 °C for three days) then in water filled tanks (20 °C) until final maturation (28 days) (UNI EN 12390-1, 2012; UNI EN 12390-2, 2019).

Lightweight structural concretes were tested by means of mass volume and compressive strength, on cubic specimens ($15 \times 15 \times 15$ cm) (UNI EN 12390-3, 2019; UNI EN 12390-4, 2019; UNI EN 12390-7, 2021).

Cellular concretes were tested by means of mass volume (UNI EN 12390-7, 2021) and thermal conductivity, on tile shaped specimens (20 \times 20 \times 3 cm). Thermal conductivity evaluation followed experimental procedures reported in literature (Buonanno et al., 2003; Dell'Isola et al., 2012; Frattolillo et al., 2005; ISO/IEC Guide 98-1, 2009). The latter based on a one-dimensional steady state comparative method and expanded uncertainty with a level of confidence of 95%. In particular, a temperature gradient is established across the sample using electrical heaters and a thermostatic bath. The sample is bounded above and below by two reference structures, each comprising a glass plate between two isothermal copper parallel plates. Under the hypothesis of one-dimensional (vertical) heat flux and negligible boundary thermal losses, the effective thermal conductivity (λ) can be evaluated as the arithmetic mean of the thermal conductivity values obtained by equating the heat fluxes measured at the top, middle and at the bottom of the sample. That is, the heat flow through the sample can be measured by measuring the temperature gradient across reference Pyrex® glass plates, whose thermal conductivity and its corresponding uncertainty is known.

3. Results and discussion

3.1. Waste characterization and mix formulation

Both wastes were selected with a particle size distribution of less than 200 μ m, which falls within the general trends identified to obtain the best behavior in terms of both expansion and technological properties of LEA (Dondi et al., 2016).

Both ceramic sludge and screen glass have a high silica content (about 65%), in accordance with literature data (Méar et al., 2006) and about 23% of fluxing content (i.e., the sum of $Fe_2O_3+CaO + MgO + Na_2O + K_2O + SrO + BaO$). The ceramic tile polishing sludge (DPM) has a composition close to the porcelain stoneware, being mainly constituted by glassy phase plus crystalline components like quartz, plagio-clase and minor quantity of mullite, zircon and synthetic silicon carbide (Table 1).

SG waste is characterized by high amounts of SrO and BaO (~8 wt %), confirming the not-recycling attitude of this waste according to European Directives (2000/532/ EC, 2000, 2001/118 /EC, 2001, 2001/119 /EC, 2001, 2001/573/ EC, 2001, 2014/ 955/EU, 2014) and indirectly the need to provide alternatives to landfill disposal. The only crystalline phase present is a 2 wt% of quartz, probably due to a contamination during recycling operations (Table 1).

The thermal behavior of the two wastes is completely different: the glass sample exhibits characteristic temperatures much lower than those of DPM with no evident expansion (Table 1). In contrast, DPM exhibits an isothermal expansion of 310% after firing. On the basis of this peculiar behavior, the only way to use the SG waste is to mix it with DPM, known to play a bloating-enhancer role (de Gennaro et al., 2007, 2009; Monteiro et al., 2004). Formulation and composition of the selected mixtures are reported in Table 2.

3.2. LEA production and characterization

Firing conditions were set to be consistent with previous experimental researches (de Gennaro et al., 2007, 2009) and following suggested temperatures from Hot Stage Microscope (HSM) results.

Bulk density values were evaluated for samples fired at 1150 and 1200 °C (Fig. 1) with 5 min of well time and are reported in Table 3. All samples show bulk density values below 1000 kg/m³ and, in particular, sample Mix1 has the lowest density value (710 kg/m³) after firing at 1150 °C.

The mineralogical composition of waste-based LEA consists in K-feldspar, plagioclase, quartz, and abundant vitreous phase. (Table 3). Changes of the mineralogical composition of the bodies is mirrored in the chemical composition of the vitreous phase which in turn determines the degree of polymerization and physical properties, like shear viscosity at high temperature (Table 3). It is known that for a vitreous phase content higher than 75 wt%, both bulk viscosity and bloating index are mainly controlled by the chemical composition of the vitreous phase (Kaz'mina, 2010). In this study, the vitreous phase is always higher than 75% (77–86 wt%) therefore, the effect of bloating index, in relation to the chemical composition of the vitreous phase and its physical properties, was investigated (Table 3). Growing the percentage of DPM waste (both at 1150 and 1200 °C) the contents of SiO₂, Al₂O₃, TiO₂, Fe₂O₃, MgO increased in the vitreous phase, while the amounts of Na₂O, K₂O, BaO and SrO decreased.

The progressive increase of DPM brought about a gradual shift in the structural features of the melt, which turned slightly more polymerized (Number of Non-Bridging Oxygens per Tetrahedrally-coordinated cations (Si,Al) as atomic percentage NBO/T passing from 0.26 to 0.23). This

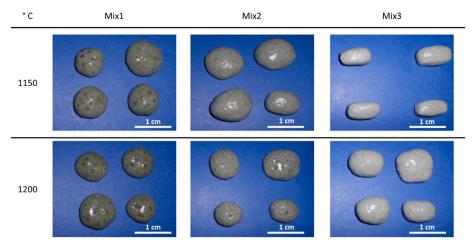


Fig. 1. Appearance of LEA fired at 1150 °C and 1200 °C.

trend agrees with the strong decrease of glass network formers and the growth of network formers (Table 3). This latter is mainly due to the incorporation of tetrahedrally-coordinated aluminum in the network charge compensated by alkali.

Such a chemical composition of the liquid phase reflects in a decreasing fluidity at high temperature, as suggested by the increase of viscosity with the increase of the DPM content.

As reported in literature (Petersen et al., 2017), the viscosity window for obtaining a low-density glass foam, observed for bottle glass with SiC as foaming agent, is between 3.3 and 4.0 \log_{10} Pa s range, that can be compared with the values calculated for the studied mixtures. In particular, these values correspond to the viscosity data calculated for these bodies (Mix1, 2, 3). At firing temperature of 1150 °C, the sample Mix1 is the less dense (710 kg/m³) with a viscosity value of 3.04 \log_{10} Pa s. Temperature rising reduces glass viscosity, both favoring SiC oxidation and allowing gas expansion and bloating. At the same time, progressive decrease of glass viscosity favors bubbles coalescence and gas leaching, so reducing expansion efficiency and leading to higher bulk density (Molinari et al., 2020; Wang et al., 2018).

The presence of solid particles boosts the effective viscosity of the glass-crystals system (Giordano, 2019; Giordano et al., 2008; Proussevitch et al., 1993). At the same time, foam stability and bloating efficiency are limited (Dondi et al., 2016). Given the presence of the same crystalline structures in similar amounts, it is believed that the effect of the solid load is the same and therefore can be overlooked. Based on the main physical and chemical properties discussed, all the tested mixes are suitable for the use as coarse aggregate in concrete manufacturing (Molinari et al., 2020; Wang et al., 2018).

However, Mix1 was chosen for the pilot scale production (35 L – Mix1L) as the best compromise between the largest amount of waste glass used in mix design and good LEA technical features (firing

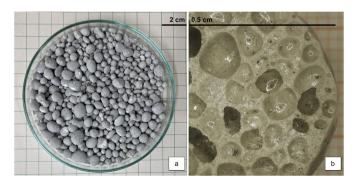


Fig. 2. Appearance of large-scale LEA production fired at 1150 °C.

temperature = 1150 °C; particles density = 710 kg/m³). LEA produced with the rotary kiln have a glassy but rough external surface (Fig. 2a) which reasonably will favor the adherence between aggregate and cement.

Proceeding with a refining process for operative conditions for a large-scale production, thermal behavior of Mix1L was tested and results are reported in Table 4. Indeed, based on T_{mve} (1125 °C) and considering the gap between the inner part of the rotary tube and the value detected by the thermocouple (external to rotating tube) of the oven, a production temperature of 1225 °C was chosen (de Gennaro et al., 2009). By the technical viewpoint, Mix1L aggregates have similar mineralogical composition and viscosity, once compared to the samples prepared at the small laboratory scale (Mix 1 LEA), keeping close to the viscosity window for obtaining a low-density glass foam reported in literature (Petersen et al., 2017).

In the Mix1L, also the microstructure was evaluated in terms of quantity and quality of both pores and septa constituting the expanded structure (Figs. 2b and 3). Micrographs show that bubbles produced by bloating are characterized by a bimodal size distribution: a first population, with bubble diameters ranging from 100 to 500 μ m (Fig. 3a) and

Table 4

Phase composition of Mix1L (large-scale production in rotary kiln at 1225 $^{\circ}$ C). Chemical composition and viscosity of the vitreous phase. Characteristic temperatures of LEA evaluated by hot stage microscope (HSM).

| Phase composition (wt%) | Unit | Mix1L |
|--|--------------------------|-------|
| Quartz | wt% | 2 |
| K-Feldspar | | 7 |
| Plagioclase | | 3 |
| Vitreous phase | | 88 |
| Chemical composition of the vitreous phase (wt%) | | |
| SiO ₂ | wt% | 64.52 |
| TiO ₂ | | 0.60 |
| Al ₂ O ₃ | | 7.40 |
| Fe ₂ O ₃ | | 0.51 |
| MgO | | 1.63 |
| CaO | | 1.17 |
| SrO | | 6.04 |
| BaO | | 6.67 |
| Na ₂ O | | 6.60 |
| K ₂ O | | 4.72 |
| P ₂ O ₅ | | 0.14 |
| Viscosity | (Log ₁₀ Pa s) | 2.97 |
| Sintering temperature (T _s) | °C | 1010 |
| Softening temperature (T _r) | °C | 1018 |
| Melting temperature (T _f) | °C | 1323 |
| Temperature of maximum expansion (T _{me}) | °C | 1233 |
| Temperature of max expansion rate (T _{mve}) | °C | 1125 |
| Maximum volume expansion at Tmve | % | 220 |
| Isothermal expansion after 30 min at the $\mathrm{T}_{\mathrm{mve}}$ | % | 160 |

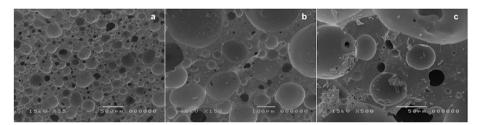


Fig. 3. Micrographs of the internal microstructure of the Mix1L aggregates in section: the scale bar is 500 µm (a), 100 µm (b) and 50 µm (c).

a class of smaller bubbles, ranging from 10 to 50 μ m, mainly present in walls (septa) between larger bubbles (Fig. 3c) as already reported for this type of LEA (de Gennaro et al., 2009).

The physico-mechanical features of experimentally produced LEA were evaluated according to standard procedure (UNI EN 13055, 2016) and compared to those of some commercial products with a similar bulk density value (Table 5). As commercial products, in general, also waste-based LEA offer, as whole, a remarkable range of physical characteristics (e.g., bulk density and water absorption) and processing conditions (e.g. firing behavior). Leaching tests were performed in previous investigations on ceramic materials containing PC-TV glass (Raimondo et al., 2007; Dondi et al., 2009) or polishing sludge (García-Ten et al., 2016; Sarani et al., 2018). No mobilization of elements of concern (Ba, Sr, transition metals) was observed in vitrified products (Raimondo et al., 2007; García-Ten et al., 2016) at variance of the few mg/kg of Ba and Sr found in leachates from porous products (Dondi et al., 2009; Sarani et al., 2018).

3.3. LEA in lightweight concretes

Mix-designing concrete composition must start from an initial prevision of required concrete performances (workability, mechanical resistance, durability, etc.) and from the characteristics of the available raw materials (cement, aggregates, additives). According to this consideration, mix-design is based on some experimental correlations between the composition of the concrete, on one hand, and the performance of the hardened concrete by means of the characteristics of the used materials, on the other (Collepardi et al., 2016; Lydon, 1972; Neville, 2012).

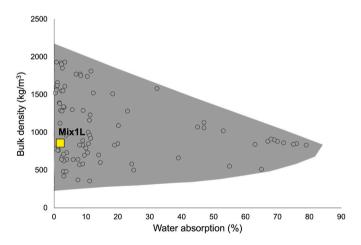
Briefly summarizing the fundamental experimental correlations among components, is possible to assume that:

- 1 The water amount (in kg/m³) influences the workability of the fresh mix (and hardened concrete performances) and can depend by the type of aggregate (rounded or crushed), its size (maximum diameter) and presence of additives (water reducers and aerating agents).
- 2 The water/cement ratio (w/c) represents the relationship between the total amount of water in the mix by weight, including the humidity of the aggregates, and the amount of cement. It is a

fundamental parameter for the quality of concrete, capable of influencing numerous important characteristics and performances such as mechanical strength of the hardened conglomerate, permeability, the extent of shrinkage, etc. The optimal w/c value to produce a hardened concrete with high compressive strength and low permeability is 0.4 (Collepardi et al., 2016; Lydon, 1972; Neville, 2012)

- 3 The volume of inert material is calculated by difference through a balance of volume by subtracting from the volume of concrete, those of the other ingredients (i.e., the volumes of water, cement and air);
- 4 Volume of the total aggregate amount is divided by contributions of two main classes of aggregates (commonly sand and gravel) based on the granulometric curves with respect to the optimal curve chosen (Fuller, Bolomey, Cubic).

From the above reported consideration the role of water amount is a key for a correct mix-design and a very important contribution is due water absorption of aggregates. In fact, while an unsaturated aggregate removes water from the mixture, one with a wet surface provides water



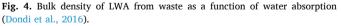


Table 5

Physical-mechanical features of waste-based aggregates compared to those of some commercial products with a similar bulk density value.

| | Sample | Particle size distribution | Bulk density | Loose bulk density | Water absorption (24h) | Strength of particle |
|----------------|----------------------------------|----------------------------|----------------------|----------------------|------------------------|----------------------|
| | | (mm) | (kg/m ³) | (kg/m ³) | (%) | (MPa) |
| commercial LEA | Knauf | 3–8 | <1000 | 440 (±15%) | <13 | <2 |
| | Granulati Zandobbio ^b | 3–8 | <1000 | 450 (±15%) | >10 | >1 |
| | Termolite ^c | 3–8 | <1000 | 380 (±15%) | >10 | >1 |
| | Leca ^d | 3–8 | <1000 | 380 (±15%) | >10 | >1 |
| | Leca più ^d | 3–8 | <1000 | 380 (±15%) | <10 | >1,5 |
| | Mix1L | 3–10 | 860 | 670 | 2.05 | 1.54 |

^a www.knauf.it.

^b www.granulati.it.

^c www.termolite.info.

^d www.leca.it.

to the mixture. In order to respect a w/c ratio close to the optimal value (0.4), the use of an aggregate giving small water absorption coefficient is encouraged (Collepardi et al., 2016; Lydon, 1972; Neville, 2012).

Comparing technical features achieved on Mix1L, with literature data (Fig. 4), waste-based aggregates fall within the field of materials with low density and low water absorption (Dondi et al., 2016). The latter feature is due to the glassy surface created during firing.

Aggregate size plays another key role to produce a concrete with the maximum possible density, or with the lowest number of voids between individual granules. The particle size curve of the solid system (cement + aggregate) must follow specific equations that guarantee the maximum dimensional sorting and the right compromise between density and workability (Fennis and Walraven, 2012).

If large size aggregates are in excess, the mixture would be hardly workable without the addition of water (that can be considered detrimental to mechanical characteristics) on the contrary, if fine aggregate is in excess, a greater amount of water is required to wet the entire surface, determining, again, a high water/cement ratio. For this reason, three particle size distribution curves were commonly used to have the best particle size distribution: the Fuller curve, the Bolomey curve and the Cubic curve (Collepardi et al., 2016). As clearly visible in Fig. 5, Mix1L lot, was sieved and selected to be consistent with Fuller curve trend for an optimal particle size packing.

To test the possible use of Mix1L aggregate in concrete manufacturing, two different types of batches were prepared following common experimental recipes and using mix design reported in Table 6:

- 1) Lightweight Structural concretes (LS specimens).
- 2) Foamed Cellular concretes (C specimens).

The lightweight structural concrete is useful to build elevations of existing buildings that are not strong enough to bear the weight of ordinary concrete structures and which would therefore require complex adaptation interventions to increase their bearing capacity. The use of a structural lightweight concrete reduces, also, the inertia forces that arise when the structure is subject to seismic movements, allowing a decrease in the reinforcement with the same section, or a decrease in the resistant section with the same reinforcement (Collepardi et al., 2016; Neville, 2012).

The foamed cellular concrete, instead, is produced by mixing, in a foaming equipment, a cement grout with a protein-based foam obtained with specific foaming agents. In this way, a closed air cell structure coated with cement is formed inside the cement mixture, giving high insulating power and considerable lightness to the material.

As regards "LS", mix design were experimentally conceptualized with two different ratios (LS1 and LS2) between fine and coarse aggregates (to better estimate waste-based aggregates contribution to concrete properties), both with the same w/c ratio (0.46). For "C" mix

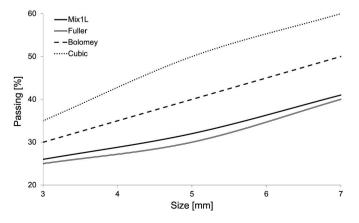


Fig. 5. Particle size distribution of Mix1L.

design, a common recipe without coarse aggregate addition was considered (C1 - reference) along with a waste-based aggregates addition recipe (C2) for a direct comparison.

Accordingly, to their final application, the two types of concrete, containing experimental Mix1L aggregates, have been tested for compressive strength (LS specimens) and thermal conductivity (C specimens) (Table 6). Standard values are reported for reference (UNI EN 1745, 2020; UNI EN 206, 2016). Standard values of thermal conductivity for C2 sample have to be intended as cross-class concrete value between "aerated concrete units" and "other aggregates concrete unit" as reported in standard (UNI EN 1745, 2020).

By experimental results analysis (Table 6) is evident that LS1 and LS2 concretes show typical features of lightweight structural concretes as indicated by standard definition (UNI EN 206, 2016) and in literature (Pacenti, 1980) (Mass volume between 1400 and 2000 kg/m³ and compressive strength, for structural purposes, substantially higher than 20 MPa).

Thermal insulation represents the main field of application for cellular concretes, for this reason is very important to achieve values of thermal conductivity similar to those of commonly used foamed concretes, usually these values range from about 0.14 to 0.24 W/(m K) (Bumanis et al., 2013; UNI EN 1745, 2020), lower is the value better is the insulation.

From comparison between C1 (assumed as reference) and C2 is possible to notice that experimental specimen (C2) seems to improve the insulating properties of a "regular" foamed concrete (C1) showing a measured thermal conductivity value of 0.18 W/(m K).

Comparing experimental results with literature data (Samson et al., 2017) (along with references therein) (Fig. 6 a,b) is clearly visible that innovative concretes produced in this work and containing waste-based aggregates can be considered as trend-followers of other, commercial or not, products.

4. Conclusions

A proof-of-concept of lightweight concrete manufacturing by using waste-based lightweight aggregates was demonstrated all along the production chain.

As a case that has received less attention, we tested possibility to go further the state of the art for glass waste recycling, using this secondary raw material in LEA-based concretes.

Novelty of this work can be summarized as follows:

- PC-TV screen waste glass and ceramic polishing sludge are suitable secondary raw materials to achieve the desired bloating under industrial production conditions. An optimal ratio was found as a compromise between the 40 wt% of ceramic tile polishing sludge and TV and the 60 wt% of PC screen glass waste.
- The selected formulation allows to produce LEA with physical and mechanical properties fully comparable with commercial aggregates. This paves the way to bending the LEA supply chain towards a circular array by replacing to a large extent ordinary expanded clay with waste-based aggregates.
- Both lightweight structural and cellular concretes manufactured with waste-based LEA exhibit a technological behavior fully complying with the performances required by regulations in the building sector.

The achieved results disclose interesting opportunities for waste application in LEA production and related lightweight concrete formulation, rising the circular economy quote in the building sector. This possibility becomes even more appealing considering the growing demand for sustainable building products.

Next steps will be focused on both the life cycle assessment of the waste-based LEA production and the evaluation of hazardous elements stabilization. Further improvements concern a widening of the waste

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Table 6

Batch formulation and physical features of concretes. Std val: standard values ^a(UNI EN 206, 2016), ^b(UNI EN 1745, 2020).

| Properties | Unit | Lightweight Structural | | Cellular | Cellular | | |
|---|-------------------|------------------------|---------------|----------------------|-----------------------------------|-----------------------------------|----------------------|
| | | LS1 | LS2 | | C1 | C2 | |
| Portland 42.5R | kg/m ³ | 350 | 350 | | 300 | 300 | |
| Sand (0-4 mm) | kg/m ³ | 833 | 500 | | 600 | 600 | |
| LEA (Mix1L) | L/m ³ | 400 | 550 | | - | - | |
| | L | - | - | | - | 440 | |
| W/C ratio | 1 | 0.46 | 0.46 | | 0.66 | 0.66 | |
| Additive Sika ViscoCrete-5380 | L/m ³ | 4.55 | 4.55 | | - | - | |
| Additive CaO dry | kg/m ³ | _ | _ | | 6 | 6 | |
| Foaming additive | kg/m ³ | - | - | | 37 | 37 | |
| Water | L | 161 | 161 | | 200 | 200 | |
| | | | | Std val ^a | | | Std val ^b |
| Mass volume (density-curing time 28 days) | kg/m ³ | 1800.5 ± 10 | 1661.5 ± 12 | 1400 < D < 2000 | 1082.6 ± 5 | 1007.7 ± 6 | 800 < D < 1400 |
| Compressive strength | MPa | 26.6 ± 1 | 25.2 ± 1 | R _{ck} >20 | _ | _ | |
| Thermal conductivity and expanded uncertainty with a 95% level of confidence | W/m·K | - | - | | $\textbf{0.24} \pm \textbf{0.05}$ | $\textbf{0.18} \pm \textbf{0.03}$ | 0.14-0.24 |

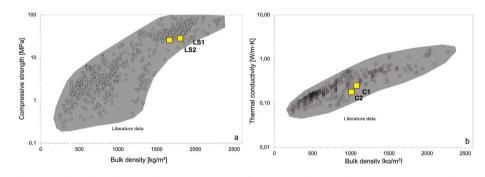


Fig. 6. Compressive strength (a) and thermal conductivity (b) as a function of bulk density for lightweight concretes containing different kind of aggregates. (data after Samson et al., 2017).

compositional range and solutions to lower the firing temperature to approach that applied in the manufacturing of expanded clay.

CRediT authorship contribution statement

Sossio Fabio Graziano: Conceptualization, Methodology, Formal analysis, Investigation, Writing – original draft, Writing – review & editing. Chiara Zanelli: Methodology, Formal analysis, Investigation, Writing – review & editing. Chiara Molinari: Formal analysis, Investigation, Writing – review & editing. Bruno de Gennaro: Conceptualization. Gaspare Giovinco: Methodology, Formal analysis, Investigation. Cecilia Correggia: Methodology, Formal analysis, Investigation. Piergiulio Cappelletti: Conceptualization, Supervision, Writing – review & editing. Michele Dondi: Conceptualization, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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