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Valorization of washing aggregate sludge and sewage sludge for lightweight aggregates production

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HIGHLIGHTS

- The artificial aggregates manufactured can be classified as *lightweight aggregates*.
- High pre-firing times are preferred to manufacture lighter artificial aggregates.
- Low firing times and temperatures are preferred to produce lighter aggregates.

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ABSTRACT

Washing aggregate sludge (*WAS*) from a gravel pit and sewage sludge (*SS*) from a wastewater treatment plant (*WWTP*) were analysed in terms of their physical, mineral and chemical characteristics. Both waste materials were mixed, milled and made into granules, pre-heated for 2 and 5 min and sintered in a rotary kiln at temperatures between 1175 °C and 1275 °C for different dwell times ranging from 1 to 30 min. The effects the raw material characteristics, pre-heating and heating temperatures and dwell times had on loss on ignition (*LOI*), mineralogy, bloating index (*BI*), loose bulk density (ρ_b), apparent and dry particle density (ρ_a, ρ_d), water absorption after 24 h (WA_{24h}) as well as the compressive strength (*S*) of the aggregates were all studied. The products obtained were classified as *lightweight aggregates* (LWAs) in accordance with the Standard EN-13055-1 (loose bulk density $\leqslant 1.20\ g\ cm^{-3}$ or particle density $\leqslant 2.00\ g\ cm^{-3}$). Their water absorption values were between 23.54% and 38.36% and compressive strength values between 1.23 MPa and 3.03 MPa. These properties were affected by the heating temperature, pre-firing and firing dwell times. The compressive strength values obtained were compared to those typically found for commercially available expanded clay aggregates (*EXCAs*) manufactured in Spain. According to these, it was possible to establish two groups of LWAs. The LWAs obtained in this research could potentially be used for the following commercial applications or for similar uses: horticulture, insulation, gardening, lightweight insulating concretes and/or prefabricated lightweight structures.

Abbreviations: BI, bloating index; *BM*, bulk mineralogy; *CM*, clay mineralogy; *EXCAs*, expanded clay aggregates; *IC*, inorganic carbon content; *I.C.*, identification code; *ICP-AES*, inductively coupled plasma-atomic emission spectroscopy; *LOI*, loss on ignition; LOI_{rm}, loss on ignition of raw materials; LOI_{total}, total loss on ignition of granules; LOI_1, loss on ignition of granules in *zone 1*; LOI_{1+2}, combined loss on ignition of granules in *zone 1* and *zone 2*; *LWA*, lightweight aggregate; *OA*, oriented aggregates; *OC*, organic carbon content; *R*, correlation value; *S*, compressive strength; $Si/\sum F$, $SiO_2/\sum$Fluxing ($CaO + MgO + K_2O + Na_2O + FeO + Fe_2O_3$) ratio; *SS*, Sewage sludge; *TC*, total carbon content; *Tsl*, temperature selected by the user in *zone 2*; *WAS*, washing aggregate sludge; *WWTP*, wastewater treatment plant; WA_{24h}, water absorption after 24 h; *W25S75*, a mixture of washing aggregate sludge and sewage sludge 25:75 w/w; *W50S50*, a mixture of washing aggregate sludge and sewage sludge 50:50 w/w; *W75S25*, a mixture of washing aggregate sludge and sewage sludge 75:25 w/w; *XRD*, X-ray diffraction; *z1*, *zone 1* (pre-firing area); *z2*, *zone 2* (firing area); *z3*, *zone 3* (cooling area); ρ_a, apparent particle density; ρ_b, loose bulk density; ρ_d, dry particle density; $\sum F$, $\sum$Fluxing ($CaO + MgO + K_2O + Na_2O + FeO + Fe_2O_3$).

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1. Introduction

Washing aggregate sludge (*WAS*) is a silt-clay waste produced when classifying sand and gravel. Although *WAS* accumulation on rafts has numerous limitations and disadvantages, it is the method most frequently used due to its low cost [1].

Sewage sludge (*SS*) is the final waste from wastewater treatment processes. Currently, sewage sludge management is the most pressing issue in the field of wastewater engineering due to the high amount produced and difficulties in valorizing it. In order to reduce volume, odors and health hazards, sewage sludge is dewatered and stabilized in the wastewater treatment plants (*WWTPs*) and it often undergoes further treatment for disposal and/or valorization.

In mining and industrial activities, waste minimization has been one of the main environmental problems in recent years. Therefore, it is very important to develop new technologies that allow these wastes to be recycled into added-value products.

A *lightweight aggregate* (LWA) is a granular and porous material with a loose bulk density (ρ_b) that does not exceed 1.20 g/cm^3 or with a particle density that does not exceed 2.00 g/cm^3 [2]. Artificial LWAs are formed by rapid heating of materials that have the ability to expand at very high temperatures.

LWAs have become of interest due to their many advantages, namely, reduction of dead load, higher thermal and acoustic insulation and sound fire resistance properties. Gardening, insulating and geotechnical applications, production of lightweight concrete and precast structural units are some applications LWAs have [3,4]. In many constructing fields, they have replaced natural sand and stone [5,6] as since the second half of the 20th century, concrete formulations based on lightweight aggregates have been successfully used [7]. The potential applications LWAs have depend on what chemical and physical properties they possess which are a result of selecting the composition of the raw materials and the sintering methods applied [8,9].

Producing artificial LWAs from wastes is deemed to be a very satisfactory environmental alternative for dealing with them, since previously worthless raw materials thereby become a product with important applications.

Mining wastes [10,11], sewage sludge [12,13], washing aggregate sludge [8,12], different types of ash [8,14] and natural materials [15] are some of the raw materials which have been used to manufacture artificial LWAs.

The objective of this paper is to demonstrate the viability of recycling two types of wastes, i.e. one coming from the aggregate extraction process and another generated in wastewater treatment, in order to develop artificial lightweight aggregates that have adequate properties which can be used in different sectors such as construction, infrastructures and agriculture.

2. Materials and methods

2.1. Raw materials selected to produce LWAs

Two types of raw waste materials were used:

- Washing aggregate sludge (*WAS*). Bulk samples of *WAS* were taken from a gravel pit located in the centre of Asturias (Northern Spain). At this quarry, the aggregates were extracted from the flood plain and the lower terrace deposits by a dry method. The sediments were made up of conglomerates and sandstones with small amounts of silt and clay in the matrix. The <5 mm sieve aggregates were washed using a washing trommel and several hydro-cyclones. The suspended silt and clay in water was transported to settling ponds where solids were separated by gravity. The thickened material was pumped to a filter press that produced a semi-dry clay/silt filter cake which was then stockpiled in safe storage areas. Samples collected from the filter press were transported to the laboratory in plastic containers and dried at room temperature for approximately 3–4 days and afterwards in an oven at 60 °C for several days (until the mass remained constant).
- Sewage sludge (*SS*) came from a wastewater treatment plant located in Cáceres (Western Spain). This city generates large amounts of *SS*, which at present is deposited in a landfill without being reused or recycled. Samples were dried at room temperature for approximately one week and then dried at 60 °C in an oven for 4–5 days (until the mass remained constant).

2.2. Characterization of the raw materials/wastes

Qualitative mineralogical analysis of *WAS* were carried out by X-ray diffraction (*XRD*) [16,17]. Semi-quantitative analysis was made following Schultźs method [18], with this method the uncertainty in the quantification can be up to 15%. Bulk mineralogy (*BM*) was determined using the polycrystalline disoriented powder method after sample grinding and homogenizing in an automatic agate mortar and sieving to <0.053 mm. Clay mineralogy (*CM*) was determined in oriented aggregates (*OA*) of the <0.002 mm fraction obtained by sedimentation from an aqueous suspension and put on glass slides. The *OA* were subjected to thermal treatment at 550 °C for 2 h and to solvation with ethylene glycol at 60 °C for 48 h. X-ray diffractograms were carried out with a *PANalytical®* diffractometer, *X'Pert Pro* model, an Ẋcelerator detector, using CuK_α radiation, a 45 kV accelerating voltage and a 40 mA current. The slit system (soller-mask-divergence-anti-scatter slits) was made up of 0.04 rad-10 mm-1/8°-1/4° slits. The step size was 0.016 (°2θ) and the time per step was 55 s (with a scan speed of 0.038°2θ). We used a Kyowa petrographic optical microscope with a 50–100× magnification level to take images using thin slides. The chemical composition was determined using inductively coupled plasma-atomic emission spectroscopy (*ICP-AES*, *Thermo Electron 6500 ICAP*) after fusion with lithium metaborate at 1000 °C and acid dissolution [19]. Loss on ignition (LOI_{rm}) during fusion was then calculated as being the weight difference (percentage) in heated samples at 1100 °C over 12 h.

Their different compositions were plotted on the ternary Riley diagram [20] to evaluate the bloating behavior of the raw material and the different potential mixtures that it could have. Also, the SiO_2/Σ Fluxing (CaO + MgO + K_2O + Na_2O + FeO + Fe_2O_3) ratio ($Si/\Sigma F$) was established since it shows the ability of the material to form a mass viscous enough to trap released gases [21].

The total, organic and inorganic carbon contents (*TC*, *OC* and *IC*, respectively) were determined using a *Shimadzu® TOC-VCSH* analyzer.

2.3. LWA manufacturing

A mixture made up of 75% (wt) of *WAS* and 25% (wt) of *SS* (*W75S25*) was chosen based on the previously obtained results of the physico-chemical characterization of the wastes. The main reason for selecting this particular mixture was that its composition was closest to the expansion area defined by the Riley diagram [20] and nearer than the chemical compositions which corresponded to the 50% *WAS*: 50% *SS* (*W50S50*) and the 25% *WAS*:75% *SS* (*W25S75*) mixtures (Fig. 1). The mixture selected was milled to a grain size of less than 200 μm [22] and homogenized using a *Restch® SK 100/C Spezialstahl* arm mill with a filter of 200 μm coupled to the exit.

The plasticity of *W75S25* was determined by calculating the Atterberg's limits, using the procedure described in the Spanish standards Norm (Regulation) UNE-103-103 [23] and Norm UNE-103-104 [24]. Water was added to the ground mixture to a level which was 5–6% (plasticity equivalent to Index/2) above the plastic limit and then extruded using a *Nannetti®* pneumatic extruder. Next, cylinders which were 1.5 cm long were manually cut and rolled to produce spherical granules that had a diameter of approximately 8–10 mm.

The green pellets were dried for 48 h at room temperature and then placed in an oven at 105 °C for 48 h.

Determination of the melting behavior of the LWA base powder (*W75S25*) was carried out using a *Misura®* heating microscope. Deformation of the sample during heating (ε%) was checked by microphotographs, taken every 5 °C from 550 °C to 1400 °C, and by a final picture, with the objective of evaluating the most relevant temperatures (sintering, softening and fusion). The heating rate was 20 °C/min in an atmosphere of air.

A tubular rotary kiln (*TOR 120-14, Nannetti®*) with an alumina based refractory lining, with an internal diameter of 6 cm and 120 cm long, was used for sintering the pellets. Depending on the temperatures reached, three areas could be differentiated in the tube: a central area (*zone 2* or *firing area*), where the highest temperature value was reached (this parameter was set by the user), and two lateral areas, with lower temperatures, one located at the inlet zone (*zone 1* or *pre-firing area*) and one situated at the discharge area (*zone 3* or *cooling area*). The temperature in these lateral areas depend on how far they are from the firing area and the temperature set for the latter (Fig. 2).

Two stages were differentiated on heating the LWAs:

- *Pre-firing or pre-heating*. In order to determine the effects of the pre-firing time on the properties of the LWAs, to prevent the rupture of the granules subjected to rapid "flash heat" and to prevent black core formation during the final firing process, dry pellets of the *W75S25* mixture underwent pretreatment in *zone 1* of the rotary kiln in groups of 25 granules, at about 18 cm from the entrance. They were placed in *zone 1* for different dwell times (2 min and 5 min, Table 1), with the temperature in *zone 2* remaining constant. The temperature in the *pre-firing*