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Valorisation of Wastewater Treatment Plants and Aggregates Processing Sludge for Lightweight Aggregates Production

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Abstract

The potential valorization of two types of wastes, sewage sludge from a wastewater treatment plant and washing aggregate sludge from a gravel pit, has been evaluated as raw materials for the manufacturing of lightweight aggregates. These sludges were chemically, physically and mineralogically characterized. They were mixed, milled, formed into pellets, pre-heated for 2 min and sintered in a rotary kiln at temperatures between 1225ºC and 1275 ºC for heating times ranging between 2 and 30 min. The effects of heating temperatures and dwell time on basic properties of the produced aggregates have been investigated. The obtained products were lightweight aggregates (LWAs) in accordance with the standard EN 13055-1 (2002). They showed water absorption values between 23.54% and 27.85% and compressive strength values between 0.82 MPa and 1.07 MPa. The possible applications of the obtained LWAs was similar to those of *Arexpan E07*® (a special variety of LWAs produced by the Spanish company ARCIRESA), which are mainly used for the manufacture of structural concretes, mortar and grout. The manufacturing of artificial LWAs from wastes is considered to be a very satisfactory environmental alternative for dealing with them, since a product with many and very important applications is obtained from raw materials with no value (sludges).

Keywords: Aggregate extraction process, concrete, lightweight aggregate, sludge, wastewater treatment plant

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Introduction

Waste minimization is one of the main environmental problems in mining and industrial activities. Aggregate extraction process sludge (hereinafter referred as *AEPS*) is a waste material that is frequently accumulated on rafts; in spite of its numerous disadvantages, currently, it is the most economical method. Sludge from waste water treatment plants (hereinafter referred as *WWTPS*) is a very important problem in the European Union since the generated amount increases every year. This fact has consequences, like the increase of this waste in dumps and the potential associated disadvantages (odor, leachates,…).

Manufacturing of lightweight aggregates (LWAs) from mineral and organic wastes is considered to be a satisfactory alternative, since a starting material with no value becomes a product with important industrial applications.

A lightweight aggregate (LWA) is a granular material with a loose bulk density not exceeding 1.20 Mg/m^3 or with a particle density not exceeding 2.00 Mg/m^3 (EN 13055-1, 2002). Artificial LWAs are produced by a rapid heating at high temperatures of materials that have the ability to expand.

Concrete formulations based on LWAs have been successfully used since the second half of the 20th century (Guneyisi et al., 2015). They are an important component in building materials such as lightweight thermo-acoustic insulation concrete and lightweight structural concrete, as well as coating road along with bituminous materials or other geotechnical applications. Moreover, their inert and porous character makes them highly suitable for agricultural applications.

Some of the raw materials used to manufacture artificial LWAs have been mining wastes (González-Corrochano et al., 2014; Yang et al., 2015), *WWTPS* (González-Corrochano et al., 2009; Hu et al., 2013; Huang and Wang, 2013), *AEPS* (González-Corrochano et al., 2009), different types of ash (Bialowiec, et al., 2014; Yang et al., 2015), masonry rubble (Mueller, et al., 2015) and natural materials (De'Gennaro et al., 2007).

The objective of this paper is to demonstrate the viability of the recycling of two types of sludge, i.e. one coming from the aggregate extraction process and another coming from waste water treatment, in order to obtain lightweight aggregates with adequate properties to their use in the construction and infrastructure sectors.

Raw Materials

Washing aggregate sludge was taken from a representative gravel pit located in the central area of Asturias (Norther Spain). Aggregates are extracted by a dry method from the flood plain and the lower terrace deposits. The sediments are made up of conglomerates and sandstones with small amounts of silt and clay in the matrix. The <5 mm sieve undersize is washed in a washing trammel and several hydro-cyclones. The suspension of silt and clay in water is transported to settling ponds where solids settle out by gravity. The thickened material is pumped to a filter press that will create a semi-dry clay/silt filter cake which is then stockpiled in safe storage areas.

The sewage sludge came from a wastewater treatment plant located in Cáceres (West Spain). This city generates large amounts of *WWTPS*, which are currently deposited in a landfill without reuse. Samples were dried at room temperature for approximately one week and then dried at 60ºC in an oven.

Methods

Characterization of Raw Materials

The particle size distribution was analysed by laser diffraction with a Coulter® LS™ 230 equipment. The carbon content was determined using a Shimadzu® TOC-V_{CSH} analyser. The chemical composition was analysed using inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Thermo Electron 6500 ICAP) after the sample fusion with lithium metaborate flux at 1000 ºC and acid dissolution (Ingamells, 1970). Loss on ignition (LOI_{rm}) was evaluated as a percentage weight difference in the heated samples at 1000°C for 45 minutes. The composition was plotted in the ternary Riley diagram (Riley, 1951) in order to evaluate the bloating behaviour of the samples.

Bulk mineralogy was determined using the polycrystalline disoriented powder method. Clay fraction mineralogy was studied through the preparation of oriented aggregates after grain size fractionation obtained by sedimentation. X-ray diffractograms were performed with a Panalytical X´Pert MPD diffractometer. The measurements were done with an accelerating voltage of 45 kV and a current of 40 mA.

LWA Manufacturing

On the basis of the results obtained in the characterization, a mixture (*A75W25*) composed by 75% (wt) of *AEPS* and 25% (wt) of *WWTPS* was chosen for the LWA production.

The mixture was milled to a grain size of less than 200 μm (Yasuda, 1991) using an arm mill. The determination of the melting behaviour of the base powder was carried out using a Misura ® heating microscope. Heating was done in air until 1400ºC using a rate of 20ºC/min. Plasticity has been studied by calculation of the Atterberg´s limits (UNE-103-104, 1993 and UNE-103-103, 1994). A controlled amount of water was added to the ground mixture and then extruded using a pneumatic extruder. Cylinders of 1.5 cm of length were cut and rolled by hand to obtain approximately spherical 8-10 mm diameter pellets.

The sintering of pellets was carried out in a tubular rotary kiln, which has a Al_2O_3 refractory tube with a length of 120 cm and an internal diameter of 6 cm. Depending on the temperature reached, three zones can be differentiated in the tube: a central area (*z2*), where the temperature is selected by the user, and two lateral areas, one located at the entrance of the tube (*z1*) and one situated at the exit (*z3*), where temperatures are lower than that of the central zone.

Two steps were differentiated in the heating of the lightweight aggregates:

- *Burnout*. In order to avoid their explosion, a pre-treatment in *z1* of rotary kiln was maintained for 2 minutes, with temperatures between 1225ºC and 1275ºC in *zone 2*. The temperature in *z1* ranges between 425ºC and 500ºC, depending on the selected temperature for *z2*.
- *Sintering*. Next to pre-treatment, the granules of the mixture were introduced in *zone 2* of the rotary kiln using temperatures in the range 1225ºC-1275ºC ($T_{heating}$) for 2-30 minutes. The rotation speed was fixed at 2.2 rpm.

LWA Physical Properties Tests

The Bloating Index (*BI*) was expressed as the volume changes after firing (Fakhfakh et al., 2007).

The Lost bulk density (ρ_b) was calculated as *W/V* ratio, where *W* is the weight of the aggregates contained in a recipient with a volume *V*, established by Standard EN-1097-3 (1998).

Particle density (*dry* and *apparent*) and water absorption after 24 hours (WA_{24h}) was determined using an established procedure described by EN-1097-6 (2000). In accordance with this standard:

- *Dry particle density* (ρ_d) is the relationship between the mass of a sample of aggregates dried in an oven and the volume that these aggregates occupy in water, including the watertight internal pores and the available pores to water.
- *Apparent particle density* (ρ_a) is the relationship between the mass of a sample of aggregates dried in an oven and the volume that these aggregates occupy in water, including the watertight internal pores and excluding available pores to water.

Compressive strength (*S*) is given by (Yashima et al., 1987):

$$S= (2.8\, Pc) / (\pi X^{2})$$

where *Pc* is the fracture load occurring rupture and *X* is the distance between the loading points. The fracture force of a single aggregate was measured in a Nannetti ® FM 96 press.

Results and Discussion

Characterization of Raw Materials and Mixture

Grain size analyses indicates that the aggregate extraction process sludge was relatively fine (average size of 9.71 µm). The waste water treatment plant sludge had a more coarse grain size (average size of 42.46 µm) and a lower percentage of clay and silt (5.83% and 63 %, respectively) than the previous one (15.55% and 69.20%, respectively).

Silica and alumina oxides were the main constituents of *AEPS* (Table 1). They mostly correspond to quartz and phyllosilicates present in the sample (Table 2). Na_2O and K_2O contents are mainly attributed to the interlayer of clay minerals (Fakhfakh et al., 2007). These mineralogical compounds are commonly presented in this type of waste and also in other raw materials/wastes used to manufacture artificial lightweight aggregates (González-Corrochano et al., 2009; Velis et al., 2014).

In *WWTPS*, the main compound was Al_2O_3 (Table 1) which comes from the coagulant (aluminum sulfate) added in the water treatment process. *WWTPS* is mainly formed by amorphous compounds and/or organic matter.

The loss on ignition values of *AEPS* and *WWTPS* are 4.00% and 73.70%, respectively (Table 1). As a consequence, they could release a great amount of gas at high temperatures, a necessary condition to produce expanded LWAs. The high loss on ignition of the *WWTPS* is mainly attributed to the oxidation of the organic matter.

Table 1. *Chemical Composition of the Raw Materials (% Dry Weight)*

Samples	SiO_2	Al_2O_3	FeO + Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	TiO_2	LOI[a]	OC[b]	IC[c]
AEPS	79.47	12.18	0.90	0.06	0.39	1.40	0.06	0.07	0.65	4.00	0.05	0.00
WWTPS	6.41	9.85	0.63	2.25	0.61	0.37	0.15	5.26	0.15	73.70	35.47	0.00

[a] Loss on ignition; [b] Organic carbon content; [c] Inorganic carbon content

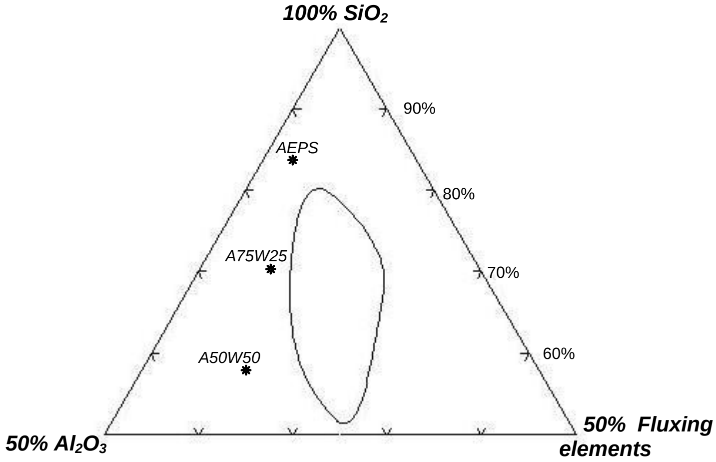
Table 2. *Mineralogical Composition of* AEPS. *+ = 1%-20%; ++ = 21%-40%; +++ = 41%-60%; ++++ = 61%-80%; +++++ = 81%-100%*

Bulk mineralogy		Clay fraction	
Quartz	**Philosilicates**	**Kaolinite**	**Illite**
++	++++	++++	++

The chemical data of the raw materials and potential mixtures (recalculated to 100%) were plotted on the SiO_2-Al_2O_3-fluxing elements (CaO + MgO + K_2O + Na_2O + FeO + Fe_2O_3) diagram (Riley, 1951). No raw materials or potential mixture of them are located within the “bloating area” (Figure 1), hence it is possible that they do not form a sufficient viscous phase to be able to trap a significant amount of gas to produce expanded LWAs.

Table 1 also presents the organic and inorganic carbon contents. Presence of carbon in the sludges is necessary since an adequate carbon/iron ratio to manufacture LWAs is necessary (although it has not been determined yet, Qi et al., 2010). *AEPS* shows very low content of carbon since it does not contain carbonate minerals (Table 2) and/or organic matter. *WWTPS* had more than 35% organic carbon because it is mainly composed of organic matter (faeces, waste paper, etc.).

Figure 1. *Representation of Raw Material Composition and Potential Mixtures of them in Riley´s Ternary Diagram (1951) Showing the Area of Bloating*



The selected mixture presented a liquid limit value of 49.30, a plastic limit value of 38.09 and a plasticity index of 11.20. This means that it exhibits an appropriate behaviour for extrusion (Casagrande 1932, 1948).

The results of the heating microscope test showed that the selected mixture presented a sintering temperature of 1265ºC and a soften temperature of 1275ºC. At 1400ºC, fusion had not started. No expansion was observed.

The above results suggest that some of the potential sources of gas evolution in the artificial aggregates could be the clay mineral itself (absorbed as well as chemically held water) of *AEPS* and/or the organic matter of *WWTPS* (González-Corrochano et al., 2009).

Testing of Physical Properties

The characterization of the produced artificial aggregates showed that expanded aggregates (BI>0) were not obtained. A suitable chemical composition that influences the "softening" at high temperature, as well as the presence of substances able to develop gas (such as water, carbon dioxide, oxygen, etc.), are required to obtain a thermally expandable material (Riley, 1951). In addition, these substances should release the gas above the pre-treatment temperatures and/or should form intermediate phases capable to retaining the gas and releasing it around the swelling temperature (Fakhfakh et al., 2007). Although LOI_{rm} values showed that gas release is possible (Table 1), the selected mixture did not present good chemical characteristics (inside the swollen area of Riley's diagram, Figure 1) to trap the potential gases released at the heating temperatures. Another possibility would be that the majority of the gases escaped during the preheating or even in the central zone of the kiln before an adequate viscosity was reached.

Aggregates with low *BI* or those non-expanded often result in artificial aggregates with relatively high densities (Huang and Wang, 2013) but this fact does not mean that they cannot be defined/classified as lightweight aggregates (González-Corrochano et al., 2009).

The bulk density was less than 1.20 g/cm^3 in all cases (Table 3); therefore, all the artificial aggregates were LWAs (EN-13055-1, 2002) although the expansion did not occur. The obtained bulk density values were comparable to those of the so-called expanded clays, which are in the 0.35-0.75 g/cm^3 range (ARCIRESA, 2014).

Table 3. *Heating Temperatures (*$T_{heating}$*), Heating Dwell Times (*$t_{heating}$*), Bulk Density (*ρ_b*), Dry Particle Density (*ρ_d*), Apparent Particle Density (*ρ_a*), Water Absorption after 24 hours (*WA_{24}*), Compressive Strength (*S*) Values and Compressive Strength: Dry Article Density Ratios (*S/ρ_d)

$T_{heating}$ (ºC)	$t_{heating}$ (min)	ρ_b (Mg/m^3)	ρ_d (Mg/m^3)	ρ_a (Mg/m^3)	WA_{24h} (%)	*S* (MPa)	S/ρ_d
1225	10	0.68	1.39	2.29	27.85	2.33 (0.82) *	1.68
1225	20	0.71	1.44	2.31	26.04	2.83 (1.00) *	1.97
1225	30	0.72	1.48	2.27	23.54	3.03 (1.07) *	2.05
1240	10	0.70	1.43	2.28	26.18	2.71 (0.96) *	1.90
1260	7.5	0.73	1.45	2.26	24.59	3.01 (1.06) *	2.07
1270	4	0.72	1.45	2.22	23.92	2.81 (0.99) *	1.94
1275	2	0.71	1.40	2.21	26.11	2.71 (0.96) *	1.94

(*)Values in bracket would be the obtained values according to Norm EN 13055-1 (2002)

The dry particle density values obtained for all the artificial aggregates were less than 2 g/cm^3 (Table 3); therefore, all the artificial aggregates manufactured in this study are classified as lightweight aggregates in accordance with the standard EN-13055-1 (2002). The dry particle density values decreased as the dwell time and temperature decreased. Bernhardt et al. (2014) attributed the decrease in density to the accelerated expansion caused by gas generation. However, in this study, the decrease in density cannot be attributed to expansion, because *BI* was negative in all the artificial aggregates.

In this study, it has been observed that the ρ_b and ρ_d values of the obtained LWAs at:

- the highest heating time (at the same heating temperature) is higher than that of the produced LWAs at the lowest heating time (Table 3).
- the highest temperature (at the same heating time) is higher than that of the produced LWAs at the lowest temperature (Table 3).

As a consequence, shorter heating dwell times and lower heating temperatures are more adequate to obtain lighter artificial LWAs.

The water absorption of the aggregates decreased as the heating temperature increased (Table 3). Since the presence of a vitrified surface was not observed, this fact could be attributed to the presence of more sintered material and/or a higher proportion of isolated pores (González-Corrochano et al., 2010 and 2011).

The compressive strength is affected by interrelated factors, such as surface features (cracking or fractures due to thermo-stress produce low *S* values), the density and shape of the aggregates (high density values and more spherical shapes produce high *S* values), pore size and distribution, water absorption (low WA_{24h} values produce high *S* values), densification effects due to sintering, bloating index, mineral species present in the LWAs and the nature of the newly formed phases (Fakhfakh et al., 2007). In this study, the compressive strength ranged from 2.33 MPa to 3.03 MPa. The highest value, i.e. 3.03 MPa, was obtained for aggregates heated at 1225ºC for 30 minutes. As it can be observed in the Table 3 and it has been previously reported (González-Corrochano et al., 2009), those values were related to the dry particle density and the water absorption of the pellets.

The fact that the best compressive strength values were achieved with the highest dry particle density values leads to the creation of a parameter that represents a measure of the compromise between the need for high resistance and low density for certain applications (De'Gennaro et al., 2007). This parameter is the S/ρ_d ratio: the best LWAs would be those that show the highest values. The S/ρ_d ratios of the aggregates are shown in Table 3. Thus, the artificial aggregates heated at 1260ºC for 7.5 min and at 1225ºC for 30 min were the best LWAs taking into account the parameter S/ρ_d.

Comparison of the Laboratory Produced and Commercial LWAs

Table 4 shows the most marketed varieties of *Arexpan*®, their main features and applications. *Arexpan*® is the trademark of the only produced LWA in Spain since 2008. The company that manufactures this LWA is Arcillas Refractarias, S.A. (ARCIRESA).

Table 4. *Some Varieties of Arexpan®, Features and Applications (Data Source:* ARCIRESA, 2014, *the Producer)*

Variety	Bulk density (Mg/m^3)	Water absorption (%)	Compressive Strength (MPa)	Applications
E07-Fino	0.75 ± 15%	4.77	N.P.D	Production of concrete, mortar and grout used in civil engineering, construction, roads and prefabricated concrete products
Estándar-Fino	0.40 ± 15%	18.05	N.P.D	
Estandar-Grueso	0.35 ± 15%	17.70	N.P.D	

N.P.D: Not performance determined

The LWAs obtained in this research may be used for different applications, depending on the desired property (i.e. bulk density, WA_{24h} or *S*). Specifically, if the bulk density values are taken into account, it is possible to establish that all the obtained LWAs may be used for the same applications as *E07-Fino* variety: production of concrete, mortar and grout used in civil engineering, construction, roads and prefabricated concrete products.

Therefore, manufacturing of artificial LWAs from wastes can be considered to be a satisfactory environmental alternative for dealing with them, since a product with important applications is obtained from raw materials with no value.

Conclusions

Aggregate extraction process sludge and wastewater treatment plant sludge have been evaluated for use in the production of lightweight aggregates. The physical, chemical and mineralogical analyses of the raw materials, the laboratory-scale manufacturing of artificial aggregates and the technological characterization of the products show that obtaining LWAs from mining and industrial wastes is possible.

The following conclusions have been deduced from this study:

- Potential mixtures of these wastes show the bloating potential taking into consideration the gases released at high temperatures. Despite this, they are not located within the bloating area with a proper chemical composition to trap the gases.
- Due to the growth of industrial and mining activities and, consequently, the continuous production of these types of wastes, the supply of raw materials such as those used in this study is guaranteed.
- All the products obtained in this study are lightweight aggregates, in accordance with Standard EN-13055-1 (2002), although they are not expanded LWAs.
- The expansion largely depends on the formation of a proper viscous phase that traps the released gases. If the gases are not released when a sufficient viscous phase has been formed, they will escape and bloating will not occur.
- The evaluation of the effects of different heating temperatures and dwell times on the main properties of the LWAs is very important since these properties are strongly influenced by those parameters.
- LWAs that show the lowest bulk density and dry particle density are manufactured at 1225ºC for 2 min of preheating and 10 min of sintering. Those with the lowest apparent particle density are produced at 1275 ºC for 2 min of preheating and 2 min of sintering. The lowest water absorption is presented by LWAs manufactured at 1225 ºC for 2 min of preheating and 30 min of sintering. This type of aggregates has the highest compressive strength.
- The possible applications of the synthesized LWAs, taking into consideration the bulk density values are the production of concrete, mortar and grout used in civil engineering, construction, roads and prefabricated concrete products
- The manufacturing of artificial aggregates through waste recycling is not only a useful alternative to the extraction of natural aggregates, but it also offers a feasible solution to waste management and contributes to the recycling of wastes.

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